Improving the quality of recycled aggregates by biodeposition of CaCO$_3$ in the pore structure

Brecht Vandevyvere, Sam Vanhessche

Supervisor: Prof. dr. ir. Nele De Belie
Counsellors: dr. ir. Jianyun Wang, dr. Joris Schoon

Master's dissertation submitted in order to obtain the academic degree of Master of Science in Civil Engineering

Department of Structural Engineering
Chair: Prof. dr. ir. Luc Taerwe
Faculty of Engineering and Architecture
Academic year 2015-2016
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$ in the pore structure

Brecht Vandevyvere, Sam Vanhessche

Supervisor: Prof. dr. ir. Nele De Belie
Counsellors: dr. ir. Jianyun Wang, dr. Joris Schoon

Master's dissertation submitted in order to obtain the academic degree of Master of Science in Civil Engineering

Department of Structural Engineering
Chair: Prof. dr. ir. Luc Taerwe
Faculty of Engineering and Architecture
Academic year 2015-2016
The authors give permission to make this master dissertation available for consultation and to copy parts of this master dissertation for personal use. In the case of any other use, the limitations of the copyright have to be respected, in particular with regard to the obligation to state expressly the source when quoting results from this master dissertation.

Brecht Vandevyvere
Sam Vanhessche

Ghent, June 2016
Acknowledgement

This master dissertation is a part of the Master program in Civil Engineering at the University of Ghent.

During one year, we tried to improve the quality of recycled aggregates by a calcium carbonate biodeposition. In this research period, we could count on the cooperation of numerous people. We would like to take this opportunity to thank these people.

First, we would like to thank Prof. dr. ir. Nele De Belie, dr. ir. Jianyun Wang and dr. Joris Schoon for facilitating the subject and observing our progress. Especially we would like to thank dr. ir. Jianyun Wang for her support in the chemical and biological part of this master dissertation and dr. Joris Schoon for his assistance in the aggregate and concrete part.

Besides our supervisor and counsellors, we would also like to thank some companies: Sagrex nv (HeidelbergCement group), Sika Belgium nv and AC Materials nv. Sagrex and AC Materials provided us the different aggregates for our research. Sagrex also offered us their laboratory and employees to execute some of our tests. Sika gave us advice and information about the use of admixtures and also provided us a sample of superplasticizer. In particular we would like to thank Mark Jonckheere (AC Materials), Kristof Weyns (Sika) and Bastian Bicher (Sika). A word of thanks is also designated towards the employees of Labo Magnel (Ghent University), which assisted us during different tests. Besides this, we also would like to thank LabMET (Ghent University), which gave us the opportunity to work in their chemical and biochemical lab. Without the collaboration of these firms and people, it would have been very hard to finish our master dissertation.

This thesis is not only the result of a year-long research, it is also the end-piece of two intense years at the University of Ghent. Therefore we like to address our final word of thanks to our friends and relatives for their support throughout this period. Special thanks goes to our parents, for giving us the opportunity to start and complete these studies.

Brecht Vandevyvere
Sam Vanhessche

Ghent, June 2016
Abstract

In this research, the quality of recycled aggregates was improved by a calcium carbonate biodeposition. To obtain a dense and qualitative good calcium carbonate precipitation, different treatment procedures were examined on two types of recycled aggregates: recycled concrete aggregate (RCA) and mixed recycled aggregate (MA). Based on the results of the weight increase, the water absorption decrease, the SEM-images and the ultrasonic pulse test, the optimal treatment procedure was selected. Once the optimal treatment procedure was selected, some other aggregate properties such as the porosity, the resistance to wear and fragmentation, etc. were examined. Furthermore, the effect of the bio-treated aggregates on concrete properties was analysed. Therefore, two concrete mixtures were produced with untreated aggregates (RCAC/U and MAC/U) and two mixtures with treated ones (RCAC/T and MAC/T). Besides these mixtures, also a natural aggregate concrete (NAC) was made, as a reference for the performance and properties of concrete. After the hardening process of the concrete, the water absorption by submersion, the compressive strength, the modulus of elasticity, etc. for the different concrete mixtures was examined. Due to this, also a relative comparison between treated and untreated concrete could be made. Both the results of the aggregate as the concrete research showed promising effects of the biodeposition treatment.
Improving the quality of recycled aggregates by biodeposition of CaCO₃

Brecht Vandevyvere – Sam Vanhessche

Supervisor(s): Prof. dr. ir. N. De Belie (UGent), dr. ir. J. Wang (UGent), dr. J. Schoon (Sagrex)

In this research, the quality of recycled aggregates was improved by a calcium carbonate biodeposition. To obtain a dense and qualitative good calcium carbonate precipitation, different treatment procedures were examined on two types of recycled aggregates: recycled concrete aggregate (RCA) and mixed recycled aggregate (MA). Based on the results of the weight increase, the water absorption decrease, the SEM-images and the ultrasonic pulse test, the optimal treatment procedure was selected. Afterwards also some other aggregate properties such as the porosity, the resistance to wear and fragmentation, etc. was determined for the optimal treatment procedure. Once the optimal treatment procedure was selected, the influence of the biodeposition was examined on the concrete properties. Therefore, a concrete mixture was produced with untreated aggregates (MAC/U) and a mixture with treated ones (MAC/T). Besides these mixtures, also a natural aggregate concrete (NAC) was made, that will serve as a reference for the performance and properties of the concrete. After the hardening process of the concrete, the water absorption by submersion, the compressive strength, the modulus of elasticity, etc. for the different concrete mixtures was examined. Due to this, also a relative comparison between treated and untreated concrete could be made. Both the results of the aggregate as the concrete research showed promising effects of the biodeposition treatment.

Keywords: Recycled aggregates, biodeposition, Bacillus sphaericus

I. INTRODUCTION

At this very moment, materials from construction and demolition waste are recycled, resulting in recycled aggregates. 95% of them is used in low quality application (foundations of roads, rip-rap,...). This is caused by a questionable attitude towards the performance of these aggregates. Increasing active research is being conducted for the application of these materials in higher grade application. This is done by actively viewing for new possible applications. Also research is done on the improvement of the properties of the material, such that a higher performance quality aggregate is obtained [1]. The main issue with recycled aggregates designated for concrete applications, is the high water absorption. When concrete is produced, the aggregates will thrive in a water-rich environment. Because of the increased water absorption, a certain amount of water will unwantedly be removed or added to the mixture. This way, the recycled aggregate can have undesired influences on the properties and the quality of the concrete. One of the possible methods to improve the quality of recycled aggregates is by precipitation of calcium carbonate (CaCO₃). As such, the water absorption could be lowered and the undesired consequences could be avoided. This method is also known as the so-called ‘biodeposition method’.

II. BIODEPOSITION

The hydrolysis of urea is one of the few biologically occurring reactions that can generate carbonate ions without an associated production of protons. If the hydrolysis of urea $\text{CO(NH}_2\text{)}_2$ starts, urea is degraded to ammonia $\text{NH}_3$ and carbonate acid $\text{H}_2\text{CO}_3$ (Eq. (1) and Eq. (2)). These products subsequently equilibrate in water to form carbonate $\text{CO}_3^{2-}$ and 2 moles of ammonium $\text{NH}_4^+$ (Eq. (3) and Eq. (4)). The $\text{OH}^-$ ions that are formed during this process will result in an increase of the pH. [2]

\[
\text{CO(NH}_2\text{)}_2 + \text{H}_2\text{O} \rightarrow \text{NH}_4\text{COOH} + \text{NH}_3 \quad (1)
\]

\[
\text{NH}_2\text{COOH} + \text{H}_2\text{O} \rightarrow \text{NH}_3 + \text{H}_2\text{CO}_3 \quad (2)
\]

\[
2 \text{NH}_3 + \text{H}_2\text{O} \leftrightarrow 2 \text{NH}_4^+ + 2\text{OH}^- \quad (3)
\]

\[
2\text{OH}^- + \text{H}_2\text{CO}_3 \leftrightarrow \text{CO}_3^{2-} + 2\text{H}_2\text{O} \quad (4)
\]

The global reaction of Eq. (1) – (4), can be written as follows:

\[
\text{CO(NH}_2\text{)}_2 + 2 \text{H}_2\text{O} \rightarrow 2 \text{NH}_4^+ + \text{CO}_3^{2-} \quad (5)
\]

When the hydrolysis of urea occurs in a calcium-rich environment (e.g. in and around recycled aggregates) and if bacteria are present in the environment, the bacteria will attract the calcium ions ($\text{Ca}^{2+}$). These ions will react with the carbonate ions ($\text{CO}_3^{2-}$) to form a dense CaCO₃ layer in the pores of the aggregates. This
results in an increased durability of the aggregate. The corresponding biochemical reaction for the calcite precipitation is given in Eq. (6).

\[ CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3 \downarrow \] (6)

Figure 1: Events during the microbial induced carbonate precipitation [3]

III. MATERIALS AND METHODS

A. Materials

In this research, three natural aggregates were used: sand 0/4 and limestone 4/6.3 and 6.3/20. The sand originated from the site of Kallo, while the limestone originated from the site of Cimescaut and Quenast. All of these are Belgian aggregate production sites. Additionally, two types of recycled aggregates were used. The first type is a rather high quality crushed concrete aggregate (RCA; 6.3/20), while the second type is a mixed recycled aggregate (MA; 6.3/20). The densities and the water absorption of the different aggregates are given in table 1.

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>( \rho_{\text{d}} ) [kg/m³]</th>
<th>( \rho_s ) [kg/m³]</th>
<th>( \rho_{\text{std}} ) [kg/m³]</th>
<th>WA₂₅ [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand 0/4</td>
<td>2670</td>
<td>2740</td>
<td>2700</td>
<td>0.94</td>
</tr>
<tr>
<td>Limestone 4/6.3</td>
<td>2680</td>
<td>2730</td>
<td>2700</td>
<td>0.73</td>
</tr>
<tr>
<td>Limestone 6.3/20</td>
<td>2650</td>
<td>2700</td>
<td>2670</td>
<td>0.63</td>
</tr>
<tr>
<td>RCA 6.3/20</td>
<td>2350</td>
<td>2660</td>
<td>2460</td>
<td>4.98</td>
</tr>
<tr>
<td>MA 6.3/20</td>
<td>2120</td>
<td>2480</td>
<td>2270</td>
<td>6.77</td>
</tr>
</tbody>
</table>

The biodeposition treatment for the recycled aggregates consisted of a growth medium and a biodeposition medium was needed. The bacteria used were *Bacillus sphaericus*. The growth medium for the *B. sphaericus* was an urea yeast extract medium (UYE-medium) which consisted of 20 g/l yeast extract (YE) and 20 g/l urea (VWR International, Belgium). In the growth medium 2V% *B. sphaericus* was added, which were incubated at 28 °C on a shaker at 100 rpm for 24 hours until a concentration of \( 10^8 \) cells/ml was reached. The biodeposition medium in this research was a 0.5M urea/Ca-nitrate solution.

To examine the effect of the bio-treated aggregates on the concrete properties, three concrete mixtures were made. Concrete mixtures with untreated mixed aggregates (MAC/U) and with treated ones (MAC/T) were produced. During the research also a natural aggregate concrete (NAC) was made. This concrete served as a reference for the performances and properties of conventional concrete. For each mixture, 4 cubes with a side of 150 mm were made. An overview of the concrete compositions for the different concrete mixtures is given in table 2.

Table 2: Concrete compositions

<table>
<thead>
<tr>
<th></th>
<th>NAC [kg/m³]</th>
<th>MAC/U [kg/m³]</th>
<th>MAC/T [kg/m³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM III/A 42.5 LA</td>
<td>320</td>
<td>320</td>
<td>320</td>
</tr>
<tr>
<td>Water</td>
<td>165</td>
<td>215</td>
<td>204</td>
</tr>
<tr>
<td>Sand 0/4</td>
<td>722</td>
<td>659</td>
<td>659</td>
</tr>
<tr>
<td>Limestone 4/6</td>
<td>456</td>
<td>412</td>
<td>412</td>
</tr>
<tr>
<td>Limestone 6/20</td>
<td>786</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>RCA 6/20</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>MA 6/20</td>
<td>-</td>
<td>820</td>
<td>820</td>
</tr>
</tbody>
</table>

B. Methods

In the first stage of this research, the optimal treatment procedure to form a dense calcite precipitation was determined. The different treatment procedures are graphically shown in figure 2. In the spray method (a), four replicates of each type of the recycled aggregates (± 200 g) were sprayed once, twice or three times with the growth culture (10^8 *B. sphaericus* cells/ml). Between the different spray actions, the aggregates were sprayed with a 0.5 M Ca-Nitrate biodeposition medium. Afterwards, the recycled aggregates were sprayed for three days with this biodeposition medium. In the immersion method (b), the four samples of each type of recycled aggregate were firstly immersed for 12 hours in the growth culture (10^8 cells/ml). After this time period, the bacterial suspension was removed and the aggregates were immersed for 3 days in the 0.5 M Ca-Nitrate solution.

![Figure 2: Treatment procedures](image)

The efficiency of the different treatment methods was examined on four parameters: the weight increase, the water absorption (WA₂₅), decrease, the cohesion strength (CEM III/A 42.5 LA), and the SEM-images of the aggregates. The cohesion strength was tested by the ultrasonic pulse test (UP-test). In this test 4 samples (± 70 g) of each recycled aggregate and each treatment procedure were placed on a 1 mm sieve and were submerged in the demineralized water recipient of the Haver USC 200-76. Afterwards, the aggregates were sonicated with ultrasonic waves for 5 minutes at a frequency of 46 Hz. Subsequently, the weight loss of each sample (formula (7)) could be determined.

\[ \text{Mass loss} \% = \frac{m_2 - m_1}{m_2} \times 100 \] (7)

Where: \( m_2 \) = mass of aggregate before UP-test
\( m_1 \) = mass of aggregate after UP-test

The weight loss was used as a parameter for the cohesion strength of the CaCO₃ precipitation. After the optimal treatment procedure was selected, three other aggregate properties of the treated and
untreated aggregates were tested. These parameters were the water absorption in function of time, the porosity and the resistance to fragmentation. The water absorption of the aggregates in time was determined with the hydrostatic weighing method [4]. For this, a test proportion of about 500 g was used. The sample was brought into a wire basket, which was submerged in water. The water absorption of the aggregates was measured at regular time intervals. The porosity of the aggregates was examined by the mercury intrusion porosity test (MIP) on 3 aggregates samples (1.4 – 1.5 g) of each recycled aggregate. The instrument used for this test was the Pascal 140 and 440 series by Thermo scientific. It consists of a low and high pressure module, which will indicate the aggregate porosity. The resistance to fragmentation was determined conform NBN EN 1097-2:2010. For each aggregate type (5000 ± 5) g was tested. The sample consisted of 65% of the 10/12.5 fraction and 35% of the 12.5/14 fraction.

On the different concrete mixtures, four parameters were examined: the porosity, the water absorption by submersion, the compressive strength and the modulus of elasticity. The porosity of the concrete mixtures was determined with the MIP test. The water absorption of the hardened concrete was determined by submerging the 4 cubical samples in water until a constant mass was obtained. This test was examined according to NBN B 15-215:1989. After this test the E-modulus of the different cubes were determined with an ultrasonic device. Also the compressive strength was determined (NBN EN 12390-3:2009).

IV. RESULTS AND DISCUSSIONS

Due to the precipitation of CaCO₃, there was a weight increase of the aggregates. The weight increase for both recycled aggregate types in case of treatment procedure (a) and (b) are given in figure 3.

As can be seen from figure 3, the highest weight increase was found for immersed aggregates. The weight increase for this method fluctuated between 0.41% and 0.89% for the RCA. Due to the higher porosity of the MA, the weight increase for these aggregates was much higher. It fluctuated between 1.21% and 1.47%.

If a closer look is taken to the spray methods, it could been observed that there is no significant difference between the weight increase for each one of the spray methods and the references. This indicates that by applying the spray method, almost no CaCO₃ precipitation was formed.

Due to the precipitation of CaCO₃ some pores of the recycled aggregates were partially or completely blocked. This caused not only an increase of weight, but also a decrease in water absorption for the bio-treated aggregates. In figure 4, the water absorption decrease for the aggregates is given. This graph indicates that there is no significant difference in the water absorption decrease for the different treatment methods. Despite the high weight increase, no significant decrease in water absorption could be observed.

To improve the water absorption decrease, the recycled aggregates (RCA and MA) were treated two times with the submersion method. As can be seen in table 3, the water absorption of the RCA did not significantly change, while for the MA, a high additional water absorption decrease was observed. The decrease changed from 0.04% to 1.18%.

Not only the amount of CaCO₃ precipitation and the decrease of the water absorption must be taken into account to determine the optimal treatment procedure. Another more important parameter is the cohesion strength of the CaCO₃ precipitatin. The cohesion was evaluated with the ultrasonic pulse test. Figure 6 represents the relative mass losses of the treated aggregates from this test. The graph indicates that the lowest mass loss was obtained for dual-treated recycled aggregates. The mass loss for the dual-treatment was about 0% for RCA and between 0.046% and 0.080% for MA. Based on these results, it can be
concluded that the highest cohesion strength is obtained by applying two times the immersion method. To get a better insight in the absorption behaviour of the treated and untreated aggregates, the water absorption in function of time was determined (see figure 8). As indicated in figure 8, it can be concluded that the full water absorption of the recycled aggregates (RCA and MA) was respectively 5.49% and 6.69%. After treating the RCA 1x and the MA 2x according the immersion method, the full water absorption of the aggregates was 6.31% and 5.17%. This indicates a decrease of 0.32% for the RCA and 0.38% for the MA. Besides this, if the water absorption of the bio-treated MA was analysed, it was noticed that the water absorption increased more smoothly compared to the untreated ones.

The treatment procedure is a reliable method only if a homogeneous CaCO₃ layer is formed around the recycled aggregate. If not, no significant water absorption decrease will be obtained. In figure 7, the SEM-image of the dual-treated MA is given. A homogeneous CaCO₃ layer can be observed. Similar pictures were obtained for the treated RCA.

Due to the treatment of the recycled aggregate, the pores of the RCA and the MA were (partly) filled with the CaCO₃ precipitation. The formation of these crystals resulted in a lower pore volume and logically changed the pore distribution. In figure 7, the total pore volume of the treated and untreated recycled aggregates are given. As can be seen, the pore volume changed from 7.79 % to 7.03 % by treating (1x) the RCA. This corresponds with a decrease of 9.8 %. For the MA, a decrease of 40,7% was obtained by treating two times the MA. This corresponds with a decrease of the total pore volume from 15.66 % to 9.27 %. Based on these results, it can be concluded that the decrease of porosity for the MA was 4 times higher compared to the RCA. Due to this porosity decrease, a higher performance aggregate was obtained.

Due to the treatment of the aggregates, not only the water absorption and the porosity changed. The treatment procedure also influenced the resistance to fragmentation. As can be seen in figure 9, the Los Angeles coefficient (LA) of the recycled aggregates were higher than those of the natural aggregates. For the untreated recycled aggregate (RCA/U) and the untreated mixed aggregate (MA/U), a LA-coefficient of respectively 30% and 40% was observed, while the limestone 6.3/20 (NA) had a LA-value of 17%. This indicates that the natural aggregate had the highest resistance to fragmentation, while the MA/U had the lowest one. If the recycled aggregates were treated, it can be observed that for the RCA a LA-decrease of 1% was visible. For the MA, the LA-coefficient decreased by 4%. The reduction of the LA-coefficient indicates that due to the treatment, the aggregates had a higher resistance to fragmentation. A possible reason for this is that due to the formation of CaCO₃, a higher aggregate strength was obtained.

**Figure 6: Mass loss for the different treatment procedures**

**Figure 5: SEM image of MA/T (2x) with 100x magnification**

**Figure 7: Total pore volume for the recycled aggregates**

**Figure 8: Water absorption of the different aggregates**

**Figure 9: LA - coefficient of the different aggregates**

Improving the quality of recycled aggregates by biodeposition of CaCO₃.
The treatment of the MA (2x) did not only influence the aggregate properties, but also some concrete properties. The main observation was that the treated aggregates seemingly influenced the effective water-cement ratio (W/C ratio). A lower porosity of the concrete mixture with bio-treated aggregates was observed. The porosity of the concrete mixtures with untreated MA was 11.78% (MAC/U), while for the mixtures with treated MA a porosity of 5.77% (MAC/T) was measured. This corresponds with a respective decrease of 51.0% (figure 10). Other property changes, as the water absorption, compressive strength and modulus of elasticity, also indicated a change in the W/C ratio.

![Figure 10: Porosity of the different concrete mixtures](image)

The water absorption of hardened concrete is mainly influenced by the porosity of the aggregates and the continuity of the pore structure in the concrete. Therefore the water absorption of recycling concrete is in general higher than the one from conventional concrete. This was also observed in this research (figure 11). The water absorption of the MAC/U mixture was 6.66%, while those of the NAC/U was only 3.78%. By treating the MA two times with the immersion method, the water absorption of the aggregates was reduced. This reduction in the aggregate water absorption (together with the decrease of the concrete porosity) logically resulted in a water absorption decrease of the concrete. The water absorption of the concrete decreased with 1.3% if biotreated MA were used. This corresponds with a water absorption of 5.36% for the MAC/T.

![Figure 11: Water absorption by submersion of the different concrete mixtures](image)

The most important property of concrete is the compressive strength. This property is influenced by different parameters. One of this is the use of recycled aggregates in the concrete skeleton. Due to the higher porosity of these aggregates, the aggregate strength will be lower which logically results in a decrease of the compressive strength. In figure 11, the cubical characteristic compressive strength of the different concrete mixtures are given. The characteristic compressive strength of the reference concrete mixture (NAC/U) was 56.3 N/mm², while the MAC/U showed a value of 37.7 N/mm². This corresponds with a decrease of 32.1%. The decrease is due to the fact of the lower strength of the MA in the concrete. Due to the treatment of the MA (2x), the porosity of the aggregates and concrete decreased which resulted in a higher compressive strength of the concrete (figure 12). The characteristic compressive strength (cubical) of the concrete mixture with treated MA was 40.1 N/mm², which corresponds with an increase of 5%.

![Figure 12: Compressive strength of the different concrete mixtures](image)

The modulus of elasticity indicates the stiffness of the material. The E-modulus of concrete is largely determined by those of the aggregates which are present in the concrete. Due to the lower strength of the recycled aggregates, the E-modulus of recycled concrete is in general lower than conventional concrete. This was also observed in this research (figure 13). The E-modulus for the NAC mixture was 40.6 GPa, while for the recycled concrete (MAC/U) only a E-modulus of 27.8 GPa was observed. This corresponds with a E-modulus decrease of 31.6%. If the MA were treated twice with the immersion method, the concrete porosity changed, which resulted in an increase of the E-modulus with 27.4% for the MAC mixture. This corresponds with an E-modulus of 35.4 GPa for MAC/T.

![Figure 13: Modulus of elasticity of the different concrete mixtures](image)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
V. CONCLUSION

By improving recycled aggregates with a calcium carbonate precipitation, the weight of the aggregates increased. This is caused by the formation of CaCO$_3$ crystal in and around the aggregates. This resulted in a decrease of the aggregate porosity, hence a decrease in water absorption. The highest weight increase occurred if the aggregates were firstly submerged in the growth medium and afterwards submersed in the biodeposition medium (method (b)). Beside this, it was observed that by treating the MA twice according the immersion method, an additional decrease in water absorption occurred. The CaCO$_3$ precipitation did not only influence the weight and the water absorption of the aggregates, also some other aggregate properties changed. It was observed that by treating the MA, the LA-value decreased with a value of 4%. This indicates that the treated MA had a higher resistance to fragmentation.

If treated recycled aggregates were used in concrete mixtures, it influenced the effective W/C ratio. Due to this, the concrete porosity decreased which resulted in a higher performance concrete. This was clearly visible from the water absorption, the compressive strength and the modulus of elasticity of the concrete.

The water absorption test indicated a decrease in absorption of 1.3%, while the compressive strength and the E-modulus increased with respectively 5% and 27.4% by using two times treated MA. Note however that from the current research it could not be determined how or if the bio-treated affected the concrete properties.

Generally, it can be concluded that a CaCO$_3$ precipitation affected the aggregate properties in a positive way. The biodeposition treatment showed promising results for the improvement of aggregates designated for concrete applications.

REFERENCES

Table of contents

Chapter 1 Literature review ............................................................................................................. 1

1. Recycled aggregates ....................................................................................................................... 1
   1.1 The origin of recycled aggregates ......................................................................................... 1
   1.2 Composition of construction and demolition debris ............................................................ 1
   1.3 Classification of recycled aggregates ..................................................................................... 3
   1.4 Regulations concerning the use of recycled aggregates in concrete .................................... 4
   1.5 Properties of recycled aggregates ......................................................................................... 6
      1.5.1 Resistance to wear and abrasion .................................................................................... 6
   1.6 Particle density ....................................................................................................................... 7
      1.6.1 Water absorption and adsorption .................................................................................... 8
      1.6.2 Other properties ............................................................................................................. 9
      1.6.3 Conclusion ..................................................................................................................... 10

2. Concrete with recycled aggregates ............................................................................................. 10
   2.1 Importance of water in concrete ............................................................................................. 11
      2.1.1 Functions of water in concrete ....................................................................................... 11
      2.1.2 Mixing water ............................................................................................................... 12
   2.2 Influences of recycled aggregates on concrete properties .................................................... 12
      2.2.1 Workability of fresh concrete ....................................................................................... 12
      2.2.2 Density of fresh and hardened concrete ...................................................................... 16
      2.2.3 Water absorption of concrete ....................................................................................... 16
      2.2.4 Modulus of elasticity ..................................................................................................... 17
      2.2.5 Compressive strength .................................................................................................... 19

3. Biodeposition treatment .............................................................................................................. 24
   3.1 Ureolytic activity and carbonate biomineralization .............................................................. 24
   3.2 Types of bacteria ..................................................................................................................... 25
   3.3 Biodeposition methods ............................................................................................................ 26
      3.3.1 Calcite Bioconcept (France) ............................................................................................ 26
      3.3.2 University of Granada (Spain) ........................................................................................ 27
      3.3.3 Biobrush consortium (UK) ............................................................................................. 27
      3.3.4 University of Ghent ....................................................................................................... 27

4. Effects of carbonate biomineralization ...................................................................................... 28
   4.1 Effects on the aggregate properties ....................................................................................... 28
   4.2 Effects on the properties of concrete ..................................................................................... 28
      Improving the quality of recycled aggregates by biodeposition of CaCO₃
4.2.1 Improvement in compressive strength of concrete ........................................ 28
4.2.2 Reduction in permeability ........................................................................... 29
4.2.3 Other effects ............................................................................................... 29

5. Applications of microbial concrete .................................................................. 30
  5.1 Microbial concrete in crack remediation ....................................................... 30
  5.2 Microbial concrete in restoration of stone buildings ..................................... 30

Chapter 2 Materials ............................................................................................. 31
  1. Aggregates .......................................................................................... 31
     1.1 Natural aggregates ....................................................................... 31
     1.2 Recycled aggregates ................................................................ 31
  2. Cement .................................................................................................. 31
  3. Superplasticizer ...................................................................................... 32
  4. Biodeposition treatment ........................................................................... 32
     4.1 Bacillus sphaericus ........................................................................ 32
     4.2 Growth medium of bacteria ............................................................ 32
     4.3 Biodeposition medium ................................................................ 33

Chapter 3 Methods .............................................................................................. 34
  1. Characterization of aggregates ................................................................ 34
     1.1 Sieve analysis ............................................................................... 34
     1.2 Particle density and water absorption .............................................. 34
        1.2.1 Aggregate particles passing the 31.5 mm and retained on the 4 mm sieve .......... 34
        1.2.2 Aggregate particles passing the 4 mm and retained on the 0.063 mm sieve .......... 35
     1.3 Water absorption in time ................................................................ 36
     1.4 Resistance to wear and fragmentation .................................................... 37
     1.5 Mercury intrusion porosimetry ............................................................. 37
     1.6 Scanning electron microscope .............................................................. 38
  2. Concrete ................................................................................................. 39
     2.1 Concrete mixtures .......................................................................... 39
     2.2 Fresh concrete .................................................................................. 40
        2.2.1 Workability ......................................................................... 40
        2.2.2 Air content and mass density of fresh concrete ......................... 40
     2.3 Hardened concrete ........................................................................ 40
        2.3.1 Water absorption by submersion ................................................ 40
        2.3.2 Mass density ........................................................................ 41
        2.3.3 Modulus of elasticity ............................................................... 41

Improving the quality of recycled aggregates by biodeposition of CaCO₃
2.3.4 Compressive strength ................................................................. 42

3. Biodeposition treatment .................................................................. 43

3.1 Concentration of the bacteria ...................................................... 43

3.2 Optimization of biodeposition medium ........................................ 43

3.2.1 Light wave spectrometer: colorimetric method ...................... 43

3.2.2 pH-meter .................................................................................. 44

3.3 Optimization of treatment procedure ......................................... 44

3.3.1 Treatment procedure I ............................................................. 44

3.3.2 Treatment procedure II .......................................................... 45

3.3.3 Treatment procedure III ......................................................... 46

3.3.4 Ultrasonic pulse test ............................................................... 46

Chapter 4 Research and results ......................................................... 47

PART I: BIODEPOSITION TREATMENT .................................................. 47

1. Optimization of biodeposition medium ........................................ 47

1.1 Biodeposition medium I: urea & calcium nitrate ...................... 47

1.2 Biodeposition medium II: Urea & Ca-Formate ......................... 49

1.3 Conclusion ................................................................................. 51

2. Optimization of treatment procedure ......................................... 52

2.1 Weight increase of the aggregates ............................................. 52

2.2 Water absorption of the aggregates ......................................... 54

2.3 Ultrasonic pulse test ................................................................. 56

2.4 Scanning electron microscope .................................................. 58

2.5 Conclusion ................................................................................. 64

PART II: AGGREGATE PROPERTIES ...................................................... 65

1. Sieve analysis .............................................................................. 65

2. Particle density and water absorption ....................................... 65

3. Water absorption in time ............................................................ 67

4. Los Angeles and micro-Deval ..................................................... 70

5. Mercury intrusion porosimetry ................................................. 71

6. Conclusion .................................................................................. 74

PART III: CONCRETE PROPERTIES ..................................................... 75

1. Consistency ............................................................................... 75

1.1 Slump ....................................................................................... 75

1.2 Flow ......................................................................................... 75

2. Air content .................................................................................. 76

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
List of figures

Figure 1: Composition of C&DW (Oikonomou, 2005) ................................................................. 2
Figure 2: Effect of moisture state aggregate on water content of concrete (Majid, 2000) ............... 8
Figure 3: Max. degree of hydration as a function of W/C ratio (Belgische Betongroepering, 2009) ... 11
Figure 4: Structure of PCE (Sika, 2016) .................................................................................... 13
Figure 5: Possible functions of PCE on water reduction in fresh concrete (Sika, 2016) ................. 13
Figure 6: Changes of slump of concrete mixtures (Poon et al., 2004) ......................................... 14
Figure 7: Effect of initial free water content on initial slump of concrete (Poon et al., 2004) ......... 15
Figure 8: Slump in time with SSD aggregates for different replacement ratios (Mefteh et al., 2013) 15
Figure 9: Slump in time with OD aggregates for different replacement ratios (Mefteh et al., 2013) .. 15
Figure 10: Influence of replacement ratio on water absorption (Salomon & Paulo, 2004) .......... 17
Figure 11: Initial tangent modulus of elasticity ............................................................................ 18
Figure 12: Secant modulus of elasticity ....................................................................................... 18
Figure 13: Compressive strength as a function of W/C ratio (Corinaldesi, 2010) ......................... 21
Figure 14: Compressive strength (replacement/hardening time) (Kwan et al., 2012) .................... 22
Figure 15: Compressive strength (replacement/hardening time) (Safiuddina et al., 2013) .......... 22
Figure 16: Influence of moisture state on the compressive strength (Poon et al., 2004) .............. 23
Figure 17: Influence of moisture state on the compressive strength (Mefteh et al., 2013) .......... 23
Figure 18: Events during microbial induced carbonate precipitation (Hammes & Verstreeta, 2002) 24
Figure 19: Gram-negative bacterium (left) vs gram-positive bacterium (right) (Dreamtime, 2015) .. 26
Figure 20: Bacillus sphaericus ..................................................................................................... 26
Figure 21: Water absorption of recycled concrete aggregate (Grabiec et al., 2012) ...................... 28
Figure 22: Drawing conventional concrete (A–C) versus bacteria-based self-healing concrete (D–F). 29
Figure 23: Schematization of test setups for water absorption in time ....................................... 36
Figure 24: Pascal 140 series: low pressure module ..................................................................... 38
Figure 25: Pascal 440 series: high pressure module ..................................................................... 38
Figure 26: Treatment procedure I (spray methods) ..................................................................... 45
Figure 27: Treatment procedure I (immersion method) ................................................................. 45
Figure 28: Treatment procedure II .............................................................................................. 45
Figure 29: Treatment procedure III ............................................................................................ 46
Figure 30: Decomposed urea of the Ca–Nitrate solution (10^9 B. sphaericus cells/ml) .................. 47
Figure 31: pH-values of the Ca–Nitrate solution (10^9 B. sphaericus cells/ml) ............................. 48
Figure 32: Decomposed urea of the Ca–Nitrate solution (2x10^9 B. sphaericus cells/ml) .......... 48
Figure 33: pH-values of the Ca–Nitrate solution (2x10^9 B. sphaericus cells) ............................ 49
Figure 34: Decomposed urea of the Ca–Formate solution (10^9 B. sphaericus cells/ml) ............ 49
Figure 35: pH-values of the Ca–Formate solution (10^9 B. sphaericus cells/ml) ....................... 50
Figure 36: Decomposed urea of the Ca–Formate solution (2x10^9 B. sphaericus cells/ml) ........ 50
Figure 37: pH-values of the Ca–Formate solution (2x10^8 B. sphaericus cells/ml) ................. 51
Figure 38: Weight increase for treatment procedure I ................................................................. 52
Figure 39: Comparison weight increase for the different treatments ........................................... 53
Figure 40: Comparison weight increase for one or two times treatment I .................................. 53
Figure 41: Water absorption for treatment procedure I (RCA) .................................................... 54
Figure 42: Water absorption for treatment procedure I (MA) .................................................... 54
Figure 43: Water absorption decrease for treatment procedure I ............................................... 55

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
List of tables

Table 1: Composition of C&DW (Vision on technology (VITO), 2013) .......................................................... 2
Table 2: Classification of recycled aggregates (COPRO, 2015) ........................................................................ 3
Table 3: Explanation symbolic display constituents of C&DW (NBN EN 12620, 2013) ............................... 4
Table 4: Maximum percentage of replacement of coarse aggregates (% by mass) ........................................ 5
Table 5: Consistency classes .......................................................................................................................... 12
Table 6: Conversion coefficients for compressive strength ........................................................................... 20
Table 7: Overview of different applications of microbial concrete (Kaur Dhami et al., 2012) ............... 25
Table 8: Concentrations of Ca-Nitrate medium ............................................................................................. 33
Table 9: Concentrations of Ca-Formate medium .......................................................................................... 33
Table 10: Concrete compositions ............................................................................................................... 39
Table 11: Compressive strength classes (NBN EN 206-1, 2001) ............................................................... 42
Table 12: Values for $k_n$ for the % characteristic value (NBN EN 1990, 2002) ............................................. 42
Table 13: Weight increase [%] for treatment procedure I ............................................................................... 52
Table 14: Comparison weight increase [%] for the different treatments .................................................... 53
Table 15: Comparison weight increase for one or two times treatment I .................................................. 53
Table 16: Water absorption decrease [%] for treatment procedure I .......................................................... 54
Table 17: Comparison water absorption decrease [%] for the different treatments ................................ 55
Table 18: Comparison water absorption decrease [%] for one or two times treatment procedure I ........ 56
Table 19: Mass loss [%] for the different treatment procedures ................................................................. 56
Table 20: Particle density and water absorption of the different aggregates .............................................. 65
Table 21: Total pore volume [%] of the aggregates ....................................................................................... 71
Table 22: Dry density [kg/m$^3$] of the different concrete mixtures ............................................................ 78
Table 23: Water absorption by submersion [%] of the different concrete mixtures .................................. 78
Table 24: Cubical compressive strength $f_{cub}$ [N/mm$^2$] of the different concrete mixtures ................ 79
Table 25: Modulus of elasticity [GPa] of the different concrete mixtures .................................................... 81

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃

\[ V \]
1) Volume of recipient
2) Volume of concrete
3) Ultrasonic velocity

VeBe
VeBe-test

VITO
Vlaamse Instelling voor Technologisch Onderzoek

\[ V_x \]
Coefficient of variation

\[ W/C \]
Water-cement ratio

\[ W/C_{eff} \]
Effective water-cement ratio

\[ WA_{24} \]
Water absorption after 24 hours

\[ WA_t \]
Water absorption at the considered time step

\[ X \]
cohesive materials (e.g. clay and soil), miscellaneous: metals (ferrous and non-ferrous), non-floating wood, plastic or rubber, gypsum plaster

\[ X0 \]
Exposure class of concrete without reinforcement or embedded metal where there is no significant freeze/thaw, abrasion or chemical attack

\[ XA1 \]
Exposure class of concrete in a slightly aggressive chemical environment

\[ XC1 \]
Exposure class of dry or permanently wet concrete

\[ XC2 \]
Exposure class of wet, rarely dry concrete

\[ XC3/4 \]
Exposure class of concrete with moderate humidity or cyclic wet and dry conditions

\[ XD1 \]
Exposure class of concrete in a moderate humidity

\[ XF1 \]
Exposure class of concrete with moderate water saturation without de-icing agent

\[ Y \]
Cumulated sieve passing of a sieve

\[ YE \]
Yeast extract

**Greek characters**

\[ \alpha_h \]
Degree of hydration

\[ \varepsilon \]
Deformation

\[ \nu \]
Poisson ratio

\[ \rho \]
Density of concrete

\[ \rho_a \]
Absolute particle density

\[ \rho_p \]
Pre-dried particle density

\[ \rho_{rd} \]
Oven-dried particle density

\[ \rho_{ssd} \]
Saturated and surface-dried particle density

\[ \rho_w \]
Density of water

\[ \sigma \]
Stress

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Framework of the investigation

The protection of the environment is a fundamental factor, directly related to the survival of the human race. Our luxurious lives are endangered by the depletion of natural resources. These are finite in nature and their extensive use pressurizes ecosystems. As such, the lives of future generations is compromised. From the viewpoint of environmental preservation and the overall human welfare, an effort should be made for the efficient utilization of resources.

In our daily lives, construction materials are of vital importance. A large amount of our time is spent in buildings or at construction infrastructures (home, office, roads, bridges, etc.). The construction industry is the thriving heart of our modern life. In contrast, due to its importance, it could also be responsible for our downfall. 50% of all exploited natural raw materials and 40% of all energy is consumed by the construction industry. In addition, 50% of all waste is produced by it (Oikonomou, 2005). As such, the industry sets high demands on our surroundings, with all possible negative consequences arising therefrom.

Concrete is the construction material of our era. It offers high structural strength, stability, resistance, durability, easy production and painless placement, all of these for a relatively low cost. Due to this, it has become the most widely used construction material in the world. However, the resources needed to produce concrete are depleting. On the other hand, there is a large volume of demolition waste. Many buildings constructed after World War II and during the financial boom now reached their design lifetime and are demolished. This produces enormous amounts of waste material, but there are not enough disposal sites, which causes a logistic and environmental nightmare. The large quantities of construction and demolition waste, the shortage of natural resources and the problems concerning the deposition of concrete and demolition waste have encouraged the use of this waste for new construction applications.

At this very moment, construction and demolition waste is recycled. The easiest way of recycling is breaking the waste material into small pieces. This process results in so-called recycled aggregates. This method of recycling enables to reuse the waste for aggregate applications, such as concrete. Unfortunately, 95% of the produced recycled aggregates are used in low quality applications (foundations of roads, rip-rap, ...). This is caused by a questionable attitude towards the performance of these aggregates. More and more active research is being conducted to find ways to use these materials in higher grade applications. This is firstly done by actively viewing for new possible applications. Secondly, attempts are made to improve the properties and performances of these materials. As such, instead of ‘downcycling’, the material can be properly recycled. (Vision on technology (VITO), 2013).

The technical progress and the ability to reuse waste in applications which can suffice our basic needs, can diminish our dependence on natural (primary) resources. Because of this, it is not preferred to refer to the construction and demolition waste as ‘waste’. It gives a rather negative connotation to a material which can replace a primary raw material. Therefore, it is better to refer to it as construction and demolition ‘debris’ and the produced recycled material originating from it is often referred to as secondary raw material.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
**Problem statement**

When recycled aggregates are used, careful attention must be paid to the way they behave in a concrete mixture. These materials have a different composition than natural aggregates, mainly due to their origin and the processes by which they are obtained. This gives rise to a range of potential issues in concrete applications.

A major issue is the porosity of the material. This causes an increased water absorption of the recycled aggregates. When concrete is produced, these aggregates will thrive in a water-rich environment. Because of the increased water absorption, a certain amount of water will unwantedly be removed or added to the mixture. This way, the recycled aggregate can have undesired influences on the properties and the quality of the concrete. Therefore, it is of great importance to investigate the effect and behaviour of this absorption.

Furthermore, the increased porosity and composition of the aggregates itself will have its repercussions on the properties of concrete. It can cause a lower strength and durability. In addition, the modulus of elasticity can decrease. In general, it increases the porosity of the concrete itself, with all its consequences.

On the issue of porous materials, some research has already been conducted. A biodeposition treatment improved the durability of these materials by precipitation of calcium carbonate. This method could possibly be applied to recycled aggregates. The effect of such treatment on the aggregate properties is relatively unknown. Neither are the influences of bio-treated aggregates on concrete properties. But, it seems that a biodeposition treatment can provide a solution for the issue of porous materials in concrete applications.
Chapter 1  Literature review

1. Recycled aggregates

1.1  The origin of recycled aggregates

Numerous processes and actions in the construction industry result in residues. These residues originally had no meaningful purpose. Therefore, they were treated as waste and remained unused. They were often just dumped at a landfill in the past. But, nowadays construction materials are increasingly judged by their ecological characteristics. Furthermore, it is not even allowed to dump building waste anymore in most developed countries. Prohibition laws and special taxes for the creation of waste areas were brought into practice to enforce this. As such, recycling and closed loop processes gained increasingly more interest. These residues are no longer discarded and it is tried to give these residues a new function. Reusing them not only protects natural resources but also eliminates the need for disposal sites.

Construction and demolition waste (C&DW) is the collective term for waste materials generated by constructing, renovating and demolition of buildings. The waste originating from construction and demolition of roads and pavements is also included. Excavated soil that is released by this work, is not considered as construction and demolition debris.

From the viewpoint of sustainable resource management, concrete and demolition waste is sorted and reduced to applicable sizes. From there on the material can be given a meaningful function. One of the most often used applications is as an aggregate. The granular material obtained from the crushing of construction and demolition waste is called a recycled aggregate (RA). Due to its origin, it is often referred to as a secondary raw material.

Recycled aggregates can be used to replace a primary raw material (sand, limestone, etc.). In theory this can be done for fine and coarse materials. In practice however, when they are used in concrete, it is mostly limited to only the coarse fractions. For fine fractions the aggregate has to go through a much more intensive breaking process. This is not only more expensive, it also seems to have a negative influence on the properties of the aggregates. Finer recycled particles seem to have a higher porosity than larger ones. The porosity may undesirably affect the properties of the concrete. The use of fine recycled aggregates is therefore not recommended (Marinkovic et al., 2010; Gómez-Soberón, 2002).

1.2  Composition of construction and demolition debris

A large variety of materials are present in construction and demolition debris. The most common materials are concrete, masonry, ceramic, natural stones, asphalt, wood, plastics, scrap metal, paper, gypsum and lime. These materials can be divided over the two general fractions, stone and non-stone fraction, as followed (Boehme et al., 2011):

- Stone fraction
  - inert fraction: concrete rubble, masonry, ceramic and natural stone
  - asphalt rubble
- Non-stone fraction
  - Recoverable fraction: wood, plastics, scrap metal, paper and cardboard
  - Difficult to recover: gypsum, lime, aerated,...

Improving the quality of recycled aggregates by biodeposition of CaCO₃
The exact share of these materials in the total portion of construction and demolition debris is variable. Decisive factors are the sample size, the location and the timing of the sampling. For sample size and location one can for example consider the total amount of waste material: worldwide, for a specific country or for a specific project. The timeframe can also be of importance, when a certain area is considered. For example: It can occur that a lot of asphalt roads were torn up over a given time period. The asphalt fraction will thereby be larger than average for this given location and period.

Figure 1 illustrates the heterogeneity of the construction and demolition waste. It also gives an idea about the share of the different fractions. The figure shows the results of a Greek research by Oikonomou (2005). Note that this is not a precise distribution, valid for all C&DW, but just the results of a single research for a specific region (Greece) and time period (2004).

![Figure 1: Composition of C&DW (Oikonomou, 2005)](image)

To illustrate the fact that the composition is time and location dependent table 1 is given. This table shows the composition of the total amount of C&DW for certain countries and regions (Vision on technology (VITO), 2013).

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Concrete</td>
<td>40%</td>
<td>41%</td>
<td>33%</td>
<td>12%</td>
<td>70%</td>
</tr>
<tr>
<td>Masonry</td>
<td>25%</td>
<td>43%</td>
<td>-</td>
<td>54%</td>
<td></td>
</tr>
<tr>
<td>Other minerals</td>
<td>2%</td>
<td>-</td>
<td>-</td>
<td>9%</td>
<td>-</td>
</tr>
<tr>
<td>Asphalt</td>
<td>26%</td>
<td>12%</td>
<td>-</td>
<td>5%</td>
<td>27%</td>
</tr>
<tr>
<td>Wood</td>
<td>2%</td>
<td>2%</td>
<td>41%</td>
<td>4%</td>
<td>-</td>
</tr>
<tr>
<td>Metal</td>
<td>1%</td>
<td>0,2%</td>
<td>14%</td>
<td>3%</td>
<td>-</td>
</tr>
<tr>
<td>Gypsum</td>
<td>-</td>
<td>0,3%</td>
<td>-</td>
<td>0,2%</td>
<td>0%</td>
</tr>
<tr>
<td>Plastic</td>
<td>-</td>
<td>0,1%</td>
<td>-</td>
<td>2%</td>
<td>-</td>
</tr>
<tr>
<td>Miscellaneous</td>
<td>7%</td>
<td>2%</td>
<td>12%</td>
<td>12%</td>
<td>3%</td>
</tr>
</tbody>
</table>

The data stated earlier (figure 1 and table 1), confirms the statement that the composition of C&DW is quite variable. In addition, it shows the heterogeneous nature of the material. These findings are one of the reasons why recycled aggregates are not trusted much. The materials present have other properties than natural aggregates and their heterogeneity and unreliable composition causes large fluctuations in aggregate properties. Therefore, C&DW is often sorted and divided into its different components. As such, the composition and properties of the material can be controlled and a reliable secondary material can be obtained. More about this in section 1.3. Consequences of the composition on aggregate properties are described in section 1.5.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
1.3 Classification of recycled aggregates

Certain materials are not desired in particular construction applications. Institutions oversee the quality of materials used to ensure the quality of construction application. Regulations differ in each country and therefore each country has its own or multiple institutions. More about these regulations and institutions is given in section 1.4.

One of the Belgian institutions that oversees the control of products in the construction sector is COPRO. They prescribed a classification for recycled aggregates and their application domains. These prescriptions are summarized in the PTV 406 document. The following categories are defined over there (COPRO, 2015):

- Concrete aggregate
- Asphalt aggregate
- Mixed aggregate
- Masonry aggregate
- Mixed- and asphalt aggregate
- Concrete- and asphalt aggregate
- High quality concrete aggregate

The share of particular materials present in the recycled material, define the aggregate class. PTV 406 imposes criteria for the maximum and minimum percentages of a certain material. These criteria are tabulated in table 2. The symbols mentioned in this table are explained in table 3.

<table>
<thead>
<tr>
<th>Classification of recycled aggregates (COPRO, 2015)</th>
<th>( R_c )</th>
<th>( R_{cug} )</th>
<th>( R_b )</th>
<th>( R_a )</th>
<th>( R_g )</th>
<th>( X )</th>
<th>( FL )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concrete aggregate</td>
<td>( \geq 70 )</td>
<td>( \geq 90 )</td>
<td>( \leq 10 )</td>
<td>( \leq 5 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>Asphalt aggregate</td>
<td>-</td>
<td>( \leq 30 )</td>
<td>( \leq 10 )</td>
<td>( \geq 70 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>Mixed aggregate</td>
<td>-</td>
<td>( \geq 50 )</td>
<td>( \leq 50 )</td>
<td>( \leq 5 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>Masonry aggregate</td>
<td>-</td>
<td>( \leq 40 )</td>
<td>( \geq 60 )</td>
<td>( \leq 5 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>Mixed- and asphalt aggregate</td>
<td>-</td>
<td>( \geq 30 )</td>
<td>( \leq 50 )</td>
<td>( \leq 30 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>Concrete- and asphalt aggregate</td>
<td>-</td>
<td>( \geq 70 )</td>
<td>( \leq 10 )</td>
<td>( \leq 30 )</td>
<td>( \leq 2 )</td>
<td>( \leq 1 )</td>
<td>( \leq 5 )</td>
</tr>
<tr>
<td>High quality concrete aggregate</td>
<td>( \geq 90 )</td>
<td>( \geq 95 )</td>
<td>-</td>
<td>( \leq 1 )</td>
<td>( \leq 0.5 )</td>
<td>( \leq 0.5 )</td>
<td>( \leq 2 )</td>
</tr>
</tbody>
</table>

Different classes of aggregates may be used in different types of application. However, it is not always allowed to replace the natural aggregates completely by recycled ones. The allowed amount of replacement depends on the aggregate class. For concrete applications in Belgium, the use of recycled aggregates is limited to 20% of the coarse aggregate volume (HeidbelbergCement Group, 2014).

If the future application of the recycled aggregate is already known, it might be interesting to ensure the criteria of a certain class are fulfilled. In this manner the material can be used in an optimal way. To do this the materials are often sorted out during the demolition of buildings (selective demolition). This is a first, crude way of separation. Various other separation techniques can be applied (washing, magnets, optical, X-ray, etc.). In the end, it is possible to get a pure fraction for practically each material present. How accurate this separation needs to be done depends on the desired application and costumer needs. It can also be possible that just one material is not desired in the final product and needs to be filtered out of the demolition waste (Kathy, 2010).
Table 3: Explanation symbolic display constituents of C&DW (NBN EN 12620, 2013)

<table>
<thead>
<tr>
<th>Category</th>
<th>Description</th>
</tr>
</thead>
</table>
| \( R_c \) | Concrete, concrete products, mortar  
|          | Concrete masonry units |
| \( R_{cug} \) | Unbound aggregates  
|          | Natural stone  
|          | Hydraulically bound aggregate |
| \( R_b \) | Clay masonry units (e.g. bricks and tiles)  
|          | Calcium silicate masonry units  
|          | Aerated non-floating concrete |
| \( R_a \) | Bituminous materials |
| \( R_g \) | Glass |
| \( FL \) | Floating materials in volume |
| \( X \) | Other:  
|          | Cohesive materials (e.g. clay and soil  
|          | Miscellaneous: metals (ferrous and non-ferrous)  
|          | Non-floating wood, plastic or rubber  
|          | Gypsum plaster |

1.4 Regulations concerning the use of recycled aggregates in concrete

For the use of aggregates in concrete, several guidelines exist. In Europe, Eurocode forms the basis of these guidelines. In addition every country has its own set of national annexes (NBN, NF, NEN, DIN,...). As a result, every country has its own collection of national quality labels. This often leads to complications, especially with the open-border and free traffic policy in Europe. To resolve this issue the CE-marking (Conformité Européenne) was established. It is a mandatory conformity marking for products sold within the European economic area. The CE-mark only certifies that the product complies with the minimum requirements imposed by the European Union. This implies that the mark is neither a quality mark, neither an admission to freely use the product for any application (Boehme et al., 2011; CE marking, 2015).

For the free application of products in concrete, the national quality labels exist. Before the CE-marking these were independent of each other and could differ a lot. Now all European quality labels must meet the legally required CE-mark criteria. Quality labels are not mandatory; it is not obligated to use products with a quality label. However, in just about all cases, contractors specify the required quality labels to ensure the product fulfils certain quality requirements.

In short there is the Eurocode with different national annexes for every country. To allow fair competition in an open market the CE-mark imposes minimum requirements for products. These requirements are based on the standards described in the Eurocode and the national annexes. At last, different quality labels define the use of products in certain applications, based on the requirements imposed by the CE-mark.

On the topic of aggregates, the European standard EN 12620 defines the specifications, components and test methods. No distinction is made between primary or secondary aggregates. At the level of concrete, the European standard EN 206 defines most requirements for manufacturing, specifications and quality assurance. NBN B 15-001 is the Belgian annex to this European standard. Neither of these normative references exclude the use of recycled aggregates (Boehme et al., 2011).

Improving the quality of recycled aggregates by biodeposition of CaCO\(_3\)
In Belgium the most import quality brand is BENOR. It belongs to the Belgian bureau of normalisation. BENOR applies to a wide range of building materials, including concrete products. The label also applies for the materials used to produce concrete (aggregates, admixtures, cement, fly ashes, rebar, ...). Consequently, it can also be used for the certification of recycled aggregates. The instances that specialize in the control of certified recycled aggregates in Belgium are COPRO and Certipro. They verify if the recycled material complies with all technical requirements and standards. If this is the case they can approve the BENOR label, which is distributed by BE-CERT, or give their own certification label, respectively the COPRO and QUAREA certificate (Boehme et al., 2011; BENOR, sd).

Aggregates for concrete must meet the criteria described in NBN EN 12620. Additional requirements need to be met when recycled aggregates are used. No recommendations for the use of fine recycled aggregates are given in the standards. As such, only recommendations for the use of coarse recycled aggregates are given. The following recommendations for the use of recycled aggregates exist according to the European standard (NBN EN 206, 2014):

- **Aggregate distribution:** d ≥ 4mm and D ≥ 10mm
- **Composition categories:**
  - Type A: Rc90, Rcu95, Rb10, Ra1, XRG1, FL2
  - Type B: Rc50, Rcu70, Rb30, Ra5, XRG2, FL2
- **Properties criteria:** F1, f1,5, LA40, SS0,2, A40
- **Minimal oven dried particle density (ρrd):**
  - Type A: 2100 kg/m³
  - Type B: 1700 kg/m³

A distinction is made between type A and type B recycled aggregates. Type A recycled aggregates are aggregates from a known source. This source is mostly a concrete source. Small percentages of impurities are allowed. This type may be used in exposure classes to which the original concrete was designed with a maximum percentage of replacement of 30% (*), unless indicated otherwise in table 4. Type B recycled aggregates originate from an unknown source or are relatively heterogeneous. They do not necessarily originate from a concrete source. This type should not be used in concrete with a compressive strength class higher than C30/37. The maximum share of recycled aggregates for both types are tabulated in table 4 (NBN EN 206, 2014).

<table>
<thead>
<tr>
<th>Recycled aggregate type</th>
<th>X0</th>
<th>XC1, XC2</th>
<th>XC3, XC4, XF1, XA1, XD1</th>
<th>Other exposure classes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type A</td>
<td>50%</td>
<td>30%</td>
<td>30%</td>
<td>0 - 30% (*)</td>
</tr>
<tr>
<td>Type B</td>
<td>50%</td>
<td>20%</td>
<td>0%</td>
<td>0%</td>
</tr>
</tbody>
</table>

NBN B 15-001 imposes additional requirements for the application of recycled aggregates in concrete in Belgium (NBN B 15-001, 2012):

- **Composition categories:** Rc90, Rcu95, Ra1, XRG0.5, FL2.
- **Properties criteria:** F1, f1,5, LA40, SS0.2, A10
- **Minimal particle density (ρrd) of 2200 kg/m³**
- **Maximal water absorption of 10%, with a maximal variance of 2%**

Improving the quality of recycled aggregates by biodeposition of CaCO₃
The required composition categories are clarified in NBN EN 12620. Rc90 and Rcu95 indicate the main components of the aggregates. If the material is only composed of concrete or mortar components Rc90 is applicable. Then a minimum of 90% of the material needs to consist of these respective components. If the material is composed of unbound material and/or natural aggregates Rcu95 is applicable. Then a minimum of 95% of the material needs to consist of these components. Additional requirements are imposed for the remaining materials: A maximum of 1% of the material may consist of bituminous material (Ra1). Furthermore, a maximum of 2% of the components may be floating elements (FL2). At last, only 0.5% of the material may consists of other elements (XRg0.5) (NBN EN 12620, 2013).

The required physical and geometrical requirements are also clarified in NBN EN 12620. First of all, the amount of fines may only be 1.5% of the total (f1.5). Furthermore, the flakiness index is limited to 20 (FI20). The water-soluble sulphates may not exceed 0.2% (SS0.2). The settlement during a test for organic materials according to EN 1744-1 may not exceed 10 mm (A10). At last the Los Angeles value is limited to a maximum of 40 (LA40) (NBN B 15-001, 2012).

As mentioned above, the water absorption must be lower than 10%. To determine the water absorption the method describes in annex C of EN 1097-6 must be used. The value after 24 hours must be considered (WA24). However, the full water absorption can be higher for recycled aggregates. On the other hand, the amount of water absorbed during the mixing process will probably be lower than WA24. Therefore, for concrete design, the WA24 of recycled aggregates must be reduced with 4%. The remaining water absorption needs to be accounted for in the mixing water. This water will be absorbed from the concrete mix and will no longer contribute to the effective water-cement ratio (W/C\text{eff}, cfr. section 2.1) (Boehme et al., 2011; NBN B 15-001, 2012).

For Belgium, NBN B 15-001 states that recycled aggregates meeting the criteria from NBN EN 12620 and the ones stated above, may be used for concrete of exposure classes X0 and XC1 and environment classes E0 and EI. The compressive strength class of these climate and environment classes is limited. For use in other classes the suitability for use needs to be proven. NBN B 15-001 also states that the recycled aggregates may only occupy 20% of the total coarse aggregate volume (NBN B 15-001, 2012).

As mentioned in section 1.3, BENOR and COPRO use PTV 406 to classify recycled aggregates. According to this classification the criteria imposed by NBN B 15-001 correspond to a high quality concrete. However, this contradicts with the fact that there are also concrete aggregates mentioned in PTV 406.

1.5 Properties of recycled aggregates

For use in concrete, the properties of recycled aggregates are usually inferior to their natural equivalent. This will have implications if they are used for concrete production. The difference in properties can be attributed to the presence of materials other than natural stones. In the following sections some aggregate properties are examined and the factors that can influence them.

1.5.1 Resistance to wear and abrasion

The Los Angeles abrasion value (LA-value) is a measure for the resistance to crushing of the aggregate. NBN EN 1097-2 specifies the test method to determine this value. In short, the mass loss of the aggregates is measured during a standardized test. The lower the LA-value, the lesser mass is lost and the more the aggregate is resistant to crushing (NBN EN 1097-2, 2010).

Improving the quality of recycled aggregates by biodeposition of CaCO3
It can be stated that in general, the LA-value of recycled aggregates is higher than the one of natural aggregates. This can be attributed to the composition and nature of the material. The materials present usually have a lower resistance to crushing. Secondly, the higher value can be attributed to the attached mortar (Boehme et al., 2011; Juan & Gutiérrez, 2009).

Marta Sanchez de Juan and Pilar Alaejos Gutiérrez (2009) conducted a study on the influence of the quantity of mortar attached to the properties of recycled concrete aggregates. The influence on the LA-value was included in this study. They found that, when the amount of attached mortar increased, the LA-value also increased. The attached mortar has hardly any resistance to crushing. During a Los Angeles abrasion test it is therefore almost completely separated from the aggregates. In addition the remaining natural aggregate also partly shatters. The total mass loss will therefore be higher, hence a higher LA-value. These results were confirmed during other researches (Safiuddina et al., 2013; Wang et al., 2008).

1.6 Particle density

The particle density of an aggregate depends on the material of which it is composed. In addition, the pore structure plays an important role. More pores result in a less dense structure, hence a lower density. The particle density of aggregates can be determined with the methods described in NBN EN 1097-6. A distinction is made between the following particle densities (NBN EN 1097-6, 2013):

- **Absolute particle density** ($\rho_a$):
  Density obtained by dividing the oven-dried mass of an aggregate sample by the volume it occupies in water, including the volume of any internal sealed voids but excluding the volume of water in any water accessible voids. In order to form an impression of the concept, the following definition is given; it is the density of the material that would be obtained when the particle is crushed to a very fine powder.

- **Oven-dried particle density** ($\rho_{od}$):
  Density obtained by dividing the oven-dried mass of an aggregate sample by the volume it occupies in water including the volume of any internal sealed voids and the volume of any water accessible voids.

- **Pre-dried particle density** ($\rho_p$):
  Density obtained by dividing the pre-dried mass of an aggregate sample by the volume it occupies in water including the volume of any internal sealed voids and the volume of any water accessible voids.

- **Saturated and surface-dried particle density** ($\rho_{ssd}$):
  Density obtained by dividing the sum of the oven-dried mass of an aggregate sample and the mass of water in any water accessible voids by the volume it occupies in water including the volume of any internal sealed voids and the volume of any water accessible voids.

For most natural aggregates such as limestone and sand, the values obtained for the different densities will be quite the same. After all, these materials contain little or no pores. The only difference between oven dry density and the absolute density is the volume of the pores accessible for water. Hence, there will be little difference between these two densities. Due to the small amount of pores, there will be little to no water absorption. Thus, the pre-dried and saturated but surface dry densities will vary little from the other densities.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
For recycled concrete aggregates, there is a fraction of foreign materials present which have a high pore content. In addition attached mortar also has a porous structure. Thus, the overall pore content for recycled aggregates is higher compared to natural aggregates. This results in lower values for the densities.

The porous structure also gives cause to larger differences between the different densities. The pore volume is sometimes included and sometimes not, in the considered volume. This will result in lower densities. Different researches established that the densities decreased when the amount of attached mortar, and thus the porosity, increased. (Safiuddina et al., 2013; Wang et al., 2008; Juan & Gutiérrez, 2009).

For natural aggregates the variance in density measurements will be small. The material is homogenous and no large variations occur. At the other hand, the composition of recycled aggregates is heterogeneous. Different materials are present and their share can vary for different samples. The amount of attached mortar can also vary. These variations and the heterogeneity will cause a larger variance for density measurements of recycled aggregates.

For a concrete design, the accounted density of the natural aggregates is the absolute density. Sand and limestone absorb little water thus this value will be close to the actual density of these aggregates in the concrete mixture. On the other hand, recycled aggregates absorb a considerable amount of water. The density of these aggregates in a concrete mixture will be close to the saturated and surface-dried particle density. It is therefore advised to use this density in the concrete design, when recycled aggregates are applied (Boehme et al., 2011).

1.6.1 Water absorption and adsorption

Water absorption is the capacity of an aggregate to absorb an amount of water. This is possible due to the pore structure of the aggregate. The water absorption factor is the ratio of the mass of water absorbed to the dry weight of the aggregate. Recycled aggregates usually have a higher water absorption factor than natural aggregates. For a natural aggregate this absorption has a maximum around 5%. Recycled aggregates can have absorption ratios reaching up to 10% and higher (Safiuddina et al., 2013).

1.6.1.1 Effect of increased water absorption

The higher water absorption of recycled aggregates can cause difficulties for concrete design. It can cause difficulties to control the properties of fresh concrete because it can change the water content sensibly.

<table>
<thead>
<tr>
<th>Absorb water from concrete mix</th>
<th>Add water to concrete mix</th>
</tr>
</thead>
</table>

*Figure 2: Effect of moisture state aggregate on water content of concrete (Majid, 2000)*

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Dry aggregates absorb water from the concrete mixture. Fully saturated aggregates with some adsorption water can add water to the concrete mixture. This phenomena is illustrated by figure 2. It can result in a change of the effective water-cement ratio. Ultimately, the strength and durability of the hardened concrete can be affected.

### 1.6.1.2 Reasons for increased water absorption

Natural rock and the aggregates originating from them have an internal pore structure. As mentioned, these pores allow the aggregate to absorb a certain amount of water. The magnitude of this absorption depends on the number, size and continuity of the pores (Tegguer, 2012; Katz, 2003).

Recycled aggregates consist of natural aggregates, some attached mortar and other materials originating from the C&DW. The attached mortar has its own pore structure. It is a very porous structure. Hence, an increase in mortar content will result in an increase in water absorption (Juan & Gutiérrez, 2009; Wang et al., 2008; Katz, 2003). The other fractions present, such as brick and wood, mostly have a large capacity to absorb water. This will contribute to the water absorption factor of the aggregate. The combination of the attached mortar and the impurities results in higher water absorptions for recycled aggregates, compared to natural aggregates (Juan & Gutiérrez, 2009; Safiuddina et al., 2013; Wang et al., 2008).

In addition to the factors stated above, it appears that the aggregates size is of interest. Finer aggregates generally have a higher absorption factor than larger ones. They have gone through a more intense crushing process. The possibility of gaps and cracks in the aggregate is therefore greater. These cracks can retain water, resulting in an increase of the water absorption. Furthermore, the surface area in immediate contact with the water in proportion to the total volume of the aggregate is much larger.

Finally, the amount of attached mortar to fine aggregates tends to be larger. The specific surface of fine aggregates is larger and therefore more cement attaches to these particles. All these factors result in an increased absorption capacity of fine aggregates compared to coarser aggregates (Juan & Gutiérrez, 2009; Katz, 2003; Ramamurthy et al., 2009).

When (part of) the material present in the recycled aggregates originates from old concrete, it seems that the properties of the original concrete also play a role. The attached mortar crushes more difficult when the original concrete is stronger. Hence, for higher strength concrete the amount of attached mortar will be higher. This will result in a higher water absorption, as explained above. Furthermore, when the original concrete has a poor quality and contains a lot of pores this will also increase the water absorption of the recycled material (Ramamurthy et al., 2009; Torrijos et al., 2008; Poon et al., 2004).

### 1.6.2 Other properties

In addition to the properties stated above, the following properties of recycled aggregates differ notably from the ones of natural aggregates:

- Modulus of elasticity (E-modulus)
- Porosity
- Shape of the aggregates
- Compressive strength of the aggregates
- Micro-Deval value (resistance to wear)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
These properties of recycled aggregates are inferior to the ones of natural aggregates designated for concrete applications. As for the properties mentioned earlier, again the explanation is to be found in the attached mortar and the presence of materials other than natural rock.

The porous structure of the attached mortar results in a larger porosity of the aggregates. This results in the larger water absorption, as mentioned earlier. The porous composition also resulted in a lower resistance to fragmentation (LA-value) and wear (MD-value).

The same goes for the compressive strength of the aggregates. The cement stone breaks quicker which results in a lower strength of the aggregates. The deformations at a given pressure are bigger compared to natural aggregates. Thus, the E-modulus is lower. There is in fact a direct link between the porosity of aggregates and their modulus of elasticity (Mehta & Monteiro, 2001). A thorough description can be found in section 2.2.4, where the E-modulus of concrete is described.

Finally, it appears that recycled aggregates have a rougher shape compared to natural aggregates. The rougher structure will have an impact on the workability of fresh concrete. The aggregates slide more difficult and the workability drops (Safiuddina et al., 2013; Mefteh et al., 2013; Leite et al., 2013).

1.6.3 Conclusion

The properties of recycled aggregates differ from the ones of natural aggregates. When the aggregates are intended to be used in concrete most differences in properties are disadvantageous. As mentioned several times the difference is caused by the nature and the heterogeneity of the materials present, the attached mortar and the uncertainty of composition.

In this context Marta Sanchez de Juan & Gutiérrez (2009) did a research about the influence of the amount of attached mortar for recycled concrete aggregates. The following conclusions were made:

- Quality of recycled concrete aggregate is lower than natural aggregate quality, due to the mortar that remains attached to the natural aggregate.
- The amount of mortar attached to fine fraction is higher than to coarse fraction.
- The main properties unfavourable affected by mortar content are absorption, density, Los Angeles abrasion and sulphate content.
- The attached mortar results in a higher sulphate and alkyne content.

Other researches came to the same conclusion. Properties like modulus of elasticity, abrasion, porosity, shape and compression strength of the recycled aggregates were all qualitatively lower than those from natural aggregates (Juan & Gutiérrez, 2009; Katz, 2003; Ramamurthy et al., 2009). This will unfavourably affect the properties of concrete produced with recycled aggregates.

2. Concrete with recycled aggregates

The properties and behaviour of concrete produced with recycled aggregates can differ from those of conventional concrete. As stated before the high water absorption is an important factor in this story. The physical properties of the aggregates can also cause these differences. In the following sections a brief summarization of the various influences and their causes are given. Only the properties which were investigated during this research are considered.
2.1 Importance of water in concrete

2.1.1 Functions of water in concrete

Water in concrete is mainly used for the hydration reaction. Hydration is the reaction between a hydraulic binder, mostly cement, and water. During this process cement stone and hydrates are formed. These compounds provide a cohesive concrete frame and interconnect the aggregates. The course of the hydration process is an important factor in the properties and durability development of concrete (Belgische Betongroepering, 2009; Simons & Verduyn, 2011).

The hydration process requires a continued presence of water. In case a lack of water occurs, the process will (partially) stop. To ensure full hydration of all cement a minimal water-cement ratio (W/C ratio) of 0.25 is required. However, part of the water in concrete is bound as gel water. This quantity is approximately 15% of the cement paste. Thus, to ensure a sufficient hydration and a minimum of pores a W/C ratio of 0.4 is advised (Belgische Betongroepering, 2009).

A distinction can be made between free water, chemically bonded and physically bonded water. At first all water is free. As soon contact is made with the cement, some of this water is chemically bonded by the hydration reaction. Another part is physically bonded to the cement paste. If enough water is present, some water can remain free. The free and physically bonded water after the hydration process define the pore structure of the concrete (Belgische Betongroepering, 2009).

The end product of the hydration reaction is cement stone. It consist of the hydrates produced during the reaction. In addition, unhydrated cement and pores, whether or not filled with water, are part of it. The hydration of cement never happens completely. The degree of hydration ($\alpha_h$) defines the extent of which the cement is hydrated. Complete hydration of cement, corresponding to a hydration degree of 1, does never occur. This is shown in figure 3. Due to this uncompleted hydration there will always remain some hydraulicity. The unhydrated cement can still react with water. This can be of importance when concrete is crushed and reused as recycled aggregate (Belgische Betongroepering, 2009; Neville & Brooks, 2010).

![Figure 3: Max. degree of hydration as a function of W/C ratio (Belgische Betongroepering, 2009)](image)

As long as water is free and not bonded it contributes to the workability. This is a secondary function of the water in concrete. The workability might be important during the execution of concrete works. In order to provide sufficient workability the W/C ratio is often higher than the advised minimum of 0.4. Note that increasing the W/C ratio results in worse concrete properties. Therefore, admixtures are often used to increase workability or to lower the W/C ratio.
2.1.2 Mixing water

Mixing water is the water that is added during the production of a concrete mixture. One can distinguish two types: the effective mixing water and the mixing water. Where the effective mixing water is the part that is used for the hydration reaction and the workability. This definition leads to a distinction between W/C ratio (W/C) and effective W/C ratio (W/C\text{eff}). The definitions are comparable to each other. The difference between both lies in the absorption of the aggregates.

Water absorbed by aggregates cannot take part in the hydration reaction, nor can it contribute to the workability. Hence, this part of the mixing water is not accounted in the effective mixing water, nor the effective W/C ratio.

Note that initially all of the mixing water is free. One could state that initially there is no difference between the mixing and the effective mixing water. As long as the water is not absorbed it can contribute to the workability and it can be used for the hydration reaction. However, after hydration there can be a notable difference between the mixing and the effective mixing water and thus the W/C ratio and effective W/C ratio. The influence of this absorption is the most crucial aspect in this master dissertation. As this absorption can have a serious effect on the properties of the concrete, it is important to (partially) compensate the absorbed water of aggregates with high water absorption.

2.2 Influences of recycled aggregates on concrete properties

2.2.1 Workability of fresh concrete

The workability of concrete is the ability of concrete to flow without losing stability or homogeneity. It can be separated into consistency and cohesion. Consistency is the possibility to flow, where cohesion is the ability of the concrete to remain stable without segregation or bleeding.

Consistency of fresh concrete is defined by various methods: slump, flow, VeBe and compaction degree of Walz. Each of these methods has their own testing apparatus and defines specific consistency classes. An overview of consistency classes is given in table 5. The respective normative reference for the test method is also given in this table. The compaction degree of Walz is left out because it is rarely used in practice (Belgische Betongroepering, 2009).

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>S1 10-40 mm</td>
<td>F1 ≤ 340 mm</td>
<td>V0 ≥ 31 s</td>
</tr>
<tr>
<td>S2 50-90 mm</td>
<td>F2 340-410 mm</td>
<td>V1 30-21 s</td>
</tr>
<tr>
<td>S3 100-150 mm</td>
<td>F3 410-480 mm</td>
<td>V2 20-11 s</td>
</tr>
<tr>
<td>S4 160-210 mm</td>
<td>F4 490-550 mm</td>
<td>V3 10-6 s</td>
</tr>
<tr>
<td>S5 ≥220 mm</td>
<td>F5 560-620 mm</td>
<td>V4 5-3 s</td>
</tr>
<tr>
<td></td>
<td>F6 ≥ 630 mm</td>
<td></td>
</tr>
</tbody>
</table>

NBN EN 206 states that the different consistency classes are not related with each other. There is no relationship between them. This is because the principle for each test is different. Where for the flow and VeBe test mechanical energy is added, the slump test does not add any energy at all (Belgische Betongroepering, 2009; NBN EN 206, 2014).

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
2.2.1.1 Influencing parameters

A first parameter that can influence the consistency is the free water content. As described in section 2.1, water can contributed to the workability as long as it is free. It is the dominant parameter concerning the workability of concrete. Free water can form a water film around the aggregate particles which reduces the cohesion and increases the consistency. Too much adsorption water can cause segregation, which is negative. In addition it also raises the W/C ratio.

To increase workability it is not advisable to add more water since this will change the W/C ratio. This can cause negative influences on other concrete properties. Therefore, admixtures were created. The function of admixtures is to lower the W/C ratio, to increase the workability of concrete or enable both effects simultaneously. As such, the amount of admixtures logically has an influence on the workability (Belgische Betongroepering, 2009; Sika, 2013).

Different types of admixtures exist. Nowadays, the most common used plasticizers are polycarboxylate ether superplasticizers (PCE). These consist of a backbone structure where carboxyl groups and side chains can be added upon (figure 4). Depending on which type of groups are added and the amount of them the PCE can have a different impact on the workability. They can work as water reducing, slump retaining, slump controlling or a combination of these effects. The difference between the functions is illustrated by figure 5 (Sika, 2016).

![Figure 4: Structure of PCE (Sika, 2016)](image)

At last the angularity and surface roughness of the aggregates also has it influence on the workability. More angular or rougher aggregates result in a more difficult concrete to process. The consistency drops because there is more energy needed to let the aggregates flow over each other due to inter particle resistance (Belgische Betongroepering, 2009; Sika, 2013).

2.2.1.2 Influence of recycled aggregates

The presence of recycled aggregates influences the parameters described in the previous section. First of all, the high water absorption results in a compensation and thus more water in the mixture. This can increase the workability. On the other hand, insufficient compensation of the absorption can cause a decrease in consistency. A detailed description of this effect is given in the next section. Secondly, as a result of the breaking process, recycled aggregates are often more angular and rougher. In general it can be stated that the consistency of concrete decreases, when the amount of recycled aggregates increases (Leite et al., 2013; Safiuddina et al., 2013).
2.2.1.3 Influence of moisture state aggregates on workability

The moisture state of aggregates can have some effects on the properties of concrete. In this section the influences on the workability are defined. The distinction is made between oven-dry (OD), air-dry (AD) and saturated-surface-dry (SSD) aggregate moisture states.

A first research was executed by Poon et al. (2004). They used natural and recycled aggregates. In their study three moisture state of the coarse aggregates were applied: OD, AD and SSD. To maintain the design mix proportion unchanged, the amount of water added was adjusted according to the actual moisture contents of the aggregates. They studied the workability in time by using the slump test. The results of their research are shown in figure 6 (a)-(d).

![Figure 6: Changes of slump of concrete mixtures (Poon et al., 2004)](image)

Poon et al. (2004) found that, when only natural aggregates were used, the initial slump values were about the same (100-110 mm). Difference in moisture state did not seem to affect the initial slump significantly. This can easily be explained by the fact that the water absorption of the aggregates is small. Therefore, no large difference in water content will arise, thus no large influence on the workability. Note that the concrete with OD aggregates had a slightly higher workability but the workability decreased to zero faster. This can be explained by the higher initial water content but this water gets absorbed by the aggregates over time.

The situation is different for the concrete prepared with recycled aggregates. In this case, the water absorption of the aggregates is significant. Mixtures made with OD aggregates showed higher initial slump values. The amount of initial free water was higher and this affected the workability. Mixtures
with AD and SSD aggregates did not differ much from the reference initial slump (100 mm). The amount of free water is about the same for both. In case of SSD aggregates, any difference between the reference concrete and recycling concrete is caused by the angularity and roughness of the aggregates, as there is no difference in free water content (Leite et al., 2013; Poon et al., 2004).

From this previous results Poon et al. (2004) showed that the initial slump is proportional with the initial amount of free water. This relation is illustrated in figure 7. The amount of initial free water is normally constant. However, when the water absorption of the aggregates is compensate it can vary. For natural aggregate this is never accounted for but, due to the high water absorption of recycled aggregate, this is important to reduce negative effects on concrete properties.

![Figure 7: Effect of initial free water content on initial slump of concrete (Poon et al., 2004)](image)

Mefteh et al. (2013) conducted a research on the effect of moisture state and pre-wetting on the workability of concrete. However, they did not accounted the absorption water of the aggregates. The results of their research are shown in figure 8 and figure 9.

![Figure 8: Slump in time with SSD aggregates for different replacement ratios (Mefteh et al., 2013)](image)

![Figure 9: Slump in time with OD aggregates for different replacement ratios (Mefteh et al., 2013)](image)

For the SSD aggregate concrete mixtures the slump does not seem to be affected much by the amount of recycled aggregates. Nevertheless, the slump values of the concrete mixture with no replacement has lower slump values. In case of the OD aggregate concrete mixtures the slump is largely affected by the replacement ratio. Higher replacement ratios result in lower slump values. This can be explained by the fact that the absorption water is not accounted for. The aggregates absorb water from the

Improving the quality of recycled aggregates by biodeposition of CaCO₃
mixture, which originally was supposed to be used for the hydration and workability of the concrete. This results in lower slump values and W/C ratios. For full replacement, there is almost no consistency left. The slump is far off the intended value. This illustrate the importance of the compensation of absorption water (Mefteh et al., 2013).

2.2.2 Density of fresh and hardened concrete

On average the wet density of concrete most commonly used in practice in Belgium is ± 2400 kg/m³. The density of concrete can vary depending on the amount of entrained air, the amount and density of the aggregates and the cement and water content. A decrease in density for a conventional concrete is usually caused by a higher water content. However, in the case of concrete with recycled aggregates the amount and density of these aggregates are important influencing factors (Belgische Betongroepering, 2009; Lima et al., 2013).

The density of concrete is inversely proportional to the air content. If the air content increases, the density will decrease. This is one of the main influencing parameters for the density of concrete. Freshly poured concrete contains about 15 to 20% entrained air. Too large amounts of air are undesirable. The concrete will be too porous, which will affect the properties of the concrete (strength, durability, water absorption, density,...). For this reason concrete is compacted. Due to the compaction, air is driven out. This will lower the air content and increase the density of the concrete. However, too intensive compaction can cause segregation, which is negative for the concrete properties. On average the air content in recycling concrete is larger than in conventional concrete (Belgische Betongroepering, 2009; Katz, 2003).

Since the density of concrete also depends on the amount and density of the aggregates present, the density of recycled concrete will usually be lower than a comparable conventional concrete. The larger the replacement ratio, the lower the density. This trend is confirmed by several researches, for both fresh and hardened concrete (González-Fonteboa et al., 2008; Iker Bekir & Selim, 2004; Lima et al., 2013; Katz, 2003).

Another influencing parameter is the water content. The behaviour of water during the mixing and hydration process has already been described in section 2.1. The free and chemically bonded water define the pore structure. Obviously, the pores can be filled with water or air. For hardened concrete this can vary the density, free and chemically bond water evaporate in an oven (± 105 °C). Therefore, NBN EN 12390-7 defines an oven-dry density, a saturated density and a density as received (NBN EN 12390-7, 2009). In general the density decreases when the water content increases because water has a lower density than the other materials present in concrete (Belgische Betongroepering, 2009; Katz et al., 2013).

2.2.3 Water absorption of concrete

Water absorption of hardened concrete is the amount of water that can be absorbed by a dry specimen. It depends on the porosity, the structure, the size and the distribution of the pores. The pore structure is a product of the hydration reaction and the mixing water. The water absorption in concrete is mainly enabled by the mortar fraction and partially by the pore structure of the coarse aggregate fraction (Katz, 2003).
The porosity of recycled aggregates is mostly higher. Therefore the water absorption of recycling concrete is often higher compared to conventional concrete. In case recycled aggregates originate from crushed concrete, they also contain attached mortar. Thereby the mortar content of the concrete produced with these aggregates increases. In general it can be stated that the water absorption of recycling concrete increases with increasing amount of recycled aggregates (Salomon & Paulo, 2004).

Figure 10 shows the result of a research conducted by Salomon & Paulo (2004). They used recycled coarse aggregates obtained from masonry and concrete. The water absorption clearly increases when the replacement ratio increases. Note however the drop in water absorption for the 20% replacement by recycled concrete aggregate, compared to the reference concrete (0% replacement). They explain this behaviour by considering the changes in the aggregate grading between the different mixes.

Figure 10: Influence of replacement ratio on water absorption (Salomon & Paulo, 2004)

2.2.4 Modulus of elasticity

The modulus of elasticity (E-modulus) is a material dependent property. It provides an insight into the deformation under load. Large deformation at a low load results in a small E-modulus. Small deformation under large loads leads to a high E-modulus of the material. The E-modulus is the ratio between the magnitude of the stress (σ) caused by an external load on the material, and the elastic deformation caused by this deformation (ε). It is a measure of the stiffness of the material. In a stress-strain diagram, the elastic modulus is represented as the slope of the tangent to the curve (σ-ε-curve) (Belgische Betongroepering, 2009; Mehta & Monteiro, 2001; Vanhooymissen et al., 2002).

For concrete the stress-strain behaviour is non-linear. The tangent slope is constantly changing and so the modulus of elasticity varies, decreasing with increasing stresses. A distinction is made between two different elastic moduli: the initial tangent ($E_{c0}$) and the secant modulus of elasticity ($E_{cm}$). The tangent modulus of elasticity corresponds to the tangent line to the stress-strain curve at the origin.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
This is also known as the dynamic modulus of elasticity and is the value which can be found by means of measuring the resonance frequency of the test piece or by means of ultrasonic measurements. The secant modulus of elasticity is also called the static modulus of elasticity. This modulus is used to calculate the long-term behaviour of structures. It is the slope of a line that connects the origin with 40% of the value of the maximum stress ($f_{cm}$) in the stress-strain curve (Mehta & Monteiro, 2001).

\[
E_{cm} = 9.500 \left( \frac{f_{cm}}{3} \right) = 9.500(f_{ck} + 8)^{1/3}
\]

Where:
- $E_{cm} = \text{secant modulus of elasticity [N/mm}^2]\]
- $f_{cm} = \text{mean compressive strength [N/mm}^2]\]
- $f_{ck} = \text{characteristic compressive strength [N/mm}^2]\]

\[
E_{c0} = 10.000 \left( \frac{f_{cm}}{3} \right) = 10.000(f_{ck} + 8)^{1/3}
\]

Where:
- $E_{c0} = \text{initial tangent modulus of elasticity [N/mm}^2]\]
- $f_{cm} = \text{mean compressive strength [N/mm}^2]\]
- $f_{ck} = \text{characteristic compressive strength [N/mm}^2]\]

When recycled aggregates are used to produce concrete the modulus of elasticity will decrease. The $E$-modulus of concrete is largely determined by those of the aggregates which are used to construct the concrete. Safiuddina et al. (2013) bundled research about recycled aggregates and recycled concrete. They found that in these researches concrete produced with crushed recycled aggregate had a modulus of elasticity up to 45% lower than that of a conventional concrete with crushed natural aggregates.
The modulus of elasticity of aggregates is directly related to their porosity. Porous aggregates offer less resistance to deformation, hence a lower E-modulus (Etxeberria et al., 2007; Mehta & Monteiro, 2001). Recycling aggregates usually have a porous structure. Consequently they are more susceptible to deformations, thus a lower E-modulus compared to conventional aggregates (Etxeberria et al., 2007; Mehta & Monteiro, 2001; Ramakrishnan & Bang, 1998).

### 2.2.5 Compressive strength

The compressive strength \( f_c \) of concrete is defined as the maximum stress which can be applied without failure. It is considered as the most important property of concrete. Several correlations exist between the compressive strength and numerous other concrete properties (e.g. equation (1) and (2) from the previous section). In the concrete industry, a sample is taken and the compressive strength is determined for each concrete mixture produced. It is an essential part of the quality control.

Concrete is a multiphase material. The strength of each material present in such a material can be strength limiting. The strength of a given solid material is inversely proportional to its porosity. Hence, the strength of concrete is determined by the porosity of the materials it consists of. This was already partially suggested in equation (1) and (2), as the E-modulus is inversely proportional to the porosity of the material. The main components of concrete are the cement matrix, coarse aggregates and the bonding zone between these two. The latter is usually the weakest link and is also known as the interfacial transition zone (ITZ). Aggregates are usually not determining the concrete strength. They are usually quite dense and strong. However, if low quality recycled aggregates or high porosity aggregates are used they can decrease the compressive strength. Also, the higher the strength class, the more the strength of the aggregate can be determining and eventually strength limiting (Mehta & Monteiro, 2001; Poon et al., 2004).

During the mixing process, coarse aggregates will be surrounded by a water film. This film is located exactly at the location of the ITZ. The film causes a local increase in W/C ratio. This will result in cement stone with more and larger pores. This phenomena is mainly present in the ITZ and causes a local decrease in strength. The strength in this zone may increase by a slow chemical interaction between the cement matrix and the aggregates. During this process, new hydration products can form in the porous structure of the ITZ. To enable this mechanism, products need to be present that facilitate a slow hydration. Examples are latent-hydraulic materials like fly ashes and blast furnace slag or specific admixtures (Mehta & Monteiro, 2001).

Despite the presence of latent-hydraulic materials or admixtures, the ITZ can still be the weakest link. Micro-cracks occur in this zone due to the discontinuities in the cement matrix, caused by the coarse aggregates. These lead to crack initiation. Once the cracks originating from the ITZ interconnect, the concrete will reach its ultimate strength and collapse. This is the most common failure condition of concrete (Mehta & Monteiro, 2001).

#### 2.2.5.1 Test dependent parameters which may influence the compressive strength

Compressive strength is determined conform NBN EN 12390-3. Specimens are placed between pressure plates and a hydraulic machine applies a compressive force to it. The compressive strength is the ratio of the maximum load to the surface area on which this load was applied. There are however some parameters that can change the result of this test. These are described in this section.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
First parameter is the rate of load increase. It is specified in the normative reference and depends on the surface size on which the load is applied. This is because the rate of load application can influence the strength of the concrete. Faster load increase will result in a lower ultimate strength (Belgische Betongroepering, 2009; NBN EN 12390-3, 2009; Vanhooymissen et al., 2002).

A second parameter is the shape of the test specimen. The smaller the sample, the greater the strength seems to be. The compressive strength of concrete is usually defined as the breaking strength of a cylindrical test piece with a height of 300 mm and diameter of 150 mm. However, in practice mostly cubical samples (150 mm x 150 mm x 150 mm) are used. Therefore concrete is often defined by a certain strength class: C X/Y. Where X equals the characteristic strength of the cylinder and Y equals the characteristic strength of the cube. Literature provides conversion coefficients and formulae to convert test results obtained with other test specimens shapes or dimensions. One of these formulae is equation (3) (Vanhooymissen et al., 2002).

\[
\frac{f_{cX}}{f_{c\text{ cub 200}}} = 0.65 + \frac{0.7}{\left(1 + \frac{\sqrt{A_c}}{200}\right)^{1.05}}
\]

Where:
- \(f_{cX}\) = compressive strength of specimen with cross section \(A_c\) [N/mm²]
- \(A_c\) = cross-sectional area of the specimen [mm²]
- \(f_{c\text{ cub 200}}\) = compressive strength of cube with edges of 200 mm [N/mm²]
- \(h\) = height of the test specimen [mm]

By applying equation (3), conversion coefficients can be calculated. The results of this calculation for some common test pieces are tabulated in table 6. The final compressive strength \(f_c\) is the product of the conversion coefficient \(a\) and the obtained strength of the respective specimen \(f_{cX}\).

<table>
<thead>
<tr>
<th>(f_{cX})</th>
<th>shape</th>
<th>Edge distance [mm]</th>
<th>(a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(f_c)</td>
<td>cylindrical</td>
<td></td>
<td>1.000</td>
</tr>
<tr>
<td>(f_{c\text{ cub 200}})</td>
<td>cubical</td>
<td>200</td>
<td>0.829</td>
</tr>
<tr>
<td>(f_{c\text{ cub 150}})</td>
<td>cubical</td>
<td>150</td>
<td>0.789</td>
</tr>
<tr>
<td>(f_{c\text{ cub 100}})</td>
<td>cubical</td>
<td>100</td>
<td>0.742</td>
</tr>
</tbody>
</table>

A third parameter is the time between pouring and testing of the concrete. The strength of concrete is developed during the course of time. Fresh concrete has little to no strength. As time goes on, the concrete develops more strength. Note that the temperature and humidity of the environment also have an influence on the strength development. NBN EN 12390-2 defines some regulations to store test specimens which will be used to test the mechanical properties of concrete. It is advised to store them under water or in a climate with a relative humidity higher than 95%. The temperature during storage should be around 20 °C. Storing the specimen in a humid environment is mainly important the first days after pouring. The hydration reaction can come to a standstill when there is not enough water present. This can be caused by premature dehydration. In addition, dehydration can cause micro-cracks due to shrinkage. Both influence the strength of the final concrete. Hence the storage in a high humidity environment, to prevent dehydration. Furthermore, the temperature is defined because higher temperatures result in faster strength development (NBN EN 12390-2, 2009).

Improving the quality of recycled aggregates by biodeposition of CaCO₃
2.2.5.2 Test independent parameters which may influence the compressive strength

As stated before concrete is a multiphase material. Its strength depends on the strength of the materials present. Mainly the porosity of these materials are of great importance. Assume a specific granular skeleton and ignore the influences of the aggregates on the strength. The only two other materials present in the concrete are water and cement. Their influence is briefly issued in this section.

Water and cement are directly interconnected with each other by the W/C ratio. The importance of water was already described in section 2.1. It was already stated that free water and physically bonded water defined the pore structure of the cement stone. More water results in more pores, which will cause a lower strength. Note however that not the absolute volume of water is important, but its relative ratio with the amount of cement. If more cement is available, more water can chemically bind. Thus, there is a direct relation between strength and W/C ratio. This dependency is shown in figure 13.

![Figure 13: Compressive strength as a function of W/C ratio (Corinaldesi, 2010)](image)

The absolute volume of cement also has an influence. The more cement present, the more pores can be filled. However this is limited. At a certain limit more cement causes a greater distance between the coarse aggregates, lowering the strength. The type of cement also has an influence. Different types of cement have different mechanisms and speeds of strength development. Furthermore, higher W/C ratios can cause slower strength development (Belgische Betongroepering, 2009).

2.2.5.3 Influence of recycled aggregates

The parameters described in the previous section are still in play. The influences described in this section are purely inflicted by the recycled aggregates. When concrete is produced with recycled aggregates a certain amount of the natural aggregates is replaced by them. In general the compressive strength of concrete decreases, when the replacement ratio increases.

Figure 14 and figure 15 show the results of researches by respectively Kwan et al. (2012) and Safiuddina et al. (2013). Both did experiments on recycled concrete by (partly) replacing the coarse aggregates by recycled ones. Both came to the same conclusion: The compressive strength decreases for increasing replacement ratios. Both tested the strength development in time. Their results show that the lower strength accounts for all the evaluated time steps. In addition, it showed that the strength development is about the same for concrete with recycled aggregates, as for concrete without recycled aggregates. These phenomena can clearly be observed in the graphs.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
The explanation for the lower strength can be found in the porosity of the material. This causes a weaker zone. As stated before porosity is strength determining. Furthermore, fracture patterns of recycled concrete show that cracks are larger and run through the weaker zone. For higher replacement ratios, it is more likely that the weaker zones interconnect with each other. This results in increasingly lower compressive strengths (Gómez-Soberón, 2002; Kwan et al., 2012).

When recycled concrete aggregates are used, the compressive strength also seems to be depending on the original concrete. When aggregates are used originating from a low strength concrete in a high strength concrete, the strength will be significantly lower than expected. Influencing parameters for this are the W/C ratio and type of cement used in the original concrete. The attached mortar gives cause to a weak zone in the concrete. Fracture patterns can develop from this zone and propagate through the whole mortar. Note that this phenomenon is particularly important when the original concrete is weaker than the new concrete (Tabsh & Abdelfatah, 2009).

In general, when recycled aggregates are used, the strengths decreases for higher replacement ratios. The relative decrease in strength depends on the used aggregates. When concrete is used where the natural coarse aggregates are fully replaced by recycled ones the strength can be up to 40% lower. Many studies have been conducted and all came to this same conclusion (Marinkovic et al., 2010; Gómez-Soberón, 2002; Kwan et al., 2012; Iker Bekir & Selim, 2004; Tabsh & Abdelfatah, 2009; Paine & Dhir, 2010; Lima et al., 2013).

### 2.2.5.4 Influence of moisture state aggregates on compressive strength

The moisture state of aggregates can have some effects on the properties of concrete. In this section the influences on the compressive strength are defined. The distinction is made between oven-dry (OD), air-dry (AD) and saturated-surface-dry (SSD) aggregate moisture states.

A first research was executed by Poon et al. (2004). They used natural and recycled aggregates. In the study three moisture state of the coarse aggregates were used: OD, AD and SSD. To maintain the design mix proportion unchanged the amount of water added was adjusted according to the actual moisture contents of the aggregates. The results from their research is shown in figure 16.

Poon et al. (2004) found that, when only natural aggregates were used, mixtures with AD and SSD aggregates had similar compressive strengths. While the mixture with OD aggregates had a lower strength. They explain this difference by the effect that the moisture content has on the development of biodeposition of CaCO$_3$. 

### Figure 14: Compressive strength (replacement/hardening time) (Kwan et al., 2012)

![Compressive strength (replacement/hardening time) Kwan et al., 2012](image)

### Figure 15: Compressive strength (replacement/hardening time) (Safiuddina et al., 2013)

![Compressive strength (replacement/hardening time) Safiuddina et al., 2013](image)
of the ITZ. Wong et al. (1999) already found that SSD aggregates had a better bond than OD ones. This may explain the lower strength. In addition the high initial W/C ratio in the OD mix, due to the compensation of the absorption water, might also decrease the strength of the cement matrix (Poon et al., 2004; Wong et al., 1999).

The situation is different for the concrete prepared with recycled aggregates. The effect on the strength development is just the opposite. Lower compressive strength were obtained for concrete prepared with SSD recycled aggregates. This phenomenon can be attributed to the bleeding of the aggregates. During vibration the water inside the aggregates may move toward the cement matrix. As such a local increase in W/C ratio in the vicinity of the particles can occur. This weakens the bond between the aggregates and the cement matrix and increases the porosity of the ITZ (Poon et al., 2004).

On the contrary, when OD recycled aggregates are used, water may move from the cement matrix towards the aggregate. As such, fine aggregate and cement particles accumulate around the coarse recycled aggregate. As a result, a stronger bond between the coarse aggregate and cement matrix might be formed. The water displacement towards the aggregate may also cause a local decrease in W/C ratio in the vicinity of the particles. This results in higher strengths of the ITZ. Etxeberria et al. (2007) stated that this can also cause a lower strength.

When the aggregate absorbs to much water, the decrease in W/C ratio is too large and the hydration reaction will be incomplete, resulting in a decrease of the compressive strength (Etxeberria et al., 2007; Poon et al., 2004).

Mefteh et al. (2013) conducted a research on the effect of moisture state and pre-wetting of the aggregates, on the compressive strength of concrete. Despite the fact they did not accounted the absorption water similar conclusions could be made. Mixtures with OD recycled aggregates and low replacement ratios had higher compressive strengths than the reference mixture with natural aggregates. The recycled aggregates absorb water from their vicinity and cause a local increase in strength. However, for higher replacement ratios the compressive strength decreased. This is because the absorption of mixing water lead to an incomplete hydration reaction. The results of this research are shown in figure 17.
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$

3. Biodeposition treatment

A biodeposition treatment is a biological process where bacteria are used to form a deposition of calcium carbonate. This deposition can possibly improve the quality of recycled aggregates. It might counteract the higher porosity, the higher water absorption, etc. of these aggregates by a biodeposition of calcium carbonate (CaCO$_3$). Due to the possible positive effects on the aggregate properties, it could enable an improvement in the quality of concrete produced with bio-treated recycled aggregates. In the following sections more information is given about this biological process.

3.1 Ureolytic activity and carbonate biomineralization

The primary role of the bacteria in the biodeposition treatment process is to precipitate CaCO$_3$. The hydrolysis of urea is one of the few biologically occurring reactions that can generate carbonate ions (CO$_3^{2-}$) without an associated production of protons. If the hydrolysis of urea $CO(NH_2)_2$ starts, urea is degraded to ammonia $NH_3$ and carbonate acid $H_2CO_3$ (Eq. (4) and Eq. (5)). These products subsequently equilibrate in water to form $CO_3^{2-}$ and 2 moles of ammonium $NH_4^+$ (Eq. (6) and Eq. (7)). The $OH^-$ ions that are formed during this process will result in an increase of the pH (De Belie, 2010).

$$CO(NH_2)_2 + H_2O \rightarrow NH_2COOH + NH_3 \quad (4)$$

$$NH_2COOH + H_2O \rightarrow NH_3 + H_2CO_3 \quad (5)$$

$$2NH_3 + H_2O \leftrightarrow 2NH_4^+ + 2OH^- \text{ (pH increase)} \quad (6)$$

$$2OH^- + H_2CO_3 \leftrightarrow CO_3^{2-} + 2H_2O \quad (7)$$

The global reaction of Eq. (4) - (7), can be written as follows:

$$CO(NH_2)_2 + 2H_2O \rightarrow 2NH_4^+ + CO_3^{2-} \quad (8)$$

When the hydrolysis of urea occurs in a calcium-rich environment a biochemical reaction will occur between the calcium and the carbonate ions. Bacteria present in this environment will attract the calcium ions (Ca$^{2+}$). The reaction with the carbonate ions will cause the formation of crystals on the bacterial cell. This could result into a dense calcium carbonate layer in the pores and on the surface of aggregates present in this environment. The corresponding biochemical reaction for the calcite precipitation is given by Eq. (9).

$$CO_3^{2-} + Ca^{2+} \rightarrow CaCO_3 \downarrow \quad (9)$$

Figure 18: Events during microbial induced carbonate precipitation (Hammes & Verstraete, 2002)

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
The chemical calcium carbonate precipitation process in Eq. (9) is determined by four key factors: the calcium concentration, the concentration of dissolved inorganic carbon (DIC), the pH and the availability of nucleation sites (Hammes & Verstraete, 2002). To have CaCO$_3$ precipitation, there must be sufficient calcium and carbonate ions so that the ion activity product (IAP) exceeds the solubility constant ($K_{sp}$). If the IAP is compared with the $K_{sp}$, the saturation ($\Omega$) of the system can be defined (Eq. (10)).

$$\Omega = \frac{a(Ca^{2+})a(CO_3^{2-})}{K_{sp}}$$  \hspace{1cm} (10)

For the saturation state $\Omega>1$, the system is oversaturated and calcium carbonate precipitation takes place Eq. (9) (De Muynck et al., 2010).

The equilibrium reactions and constants governing the dissolution of CO$_2$ in the aqueous media (25 °C; 1 atm) are given in Eq. (11)-(14) (Stumm & Morgan, 1981).

$$CO_2(g) \leftrightarrow CaCO_2(aq.) \hspace{1cm} pK_H = 1.468$$ \hspace{1cm} (11)

$$CO_2(aq.) + H_2O \leftrightarrow H_2CO_3 \hspace{1cm} pK = 2.84$$ \hspace{1cm} (12)

$$H_2CO_3 \leftrightarrow H^+ + HCO_3^- \hspace{1cm} pK_1 = 6.352$$ \hspace{1cm} (13)

$$HCO_3^- \leftrightarrow CO_3^{2-} + H^+ \hspace{1cm} pK_2 = 10.329$$ \hspace{1cm} (14)

With $H_2CO_3^- = CO_2(aq.) + H_2CO_3$

### 3.2 Types of bacteria

Under suitable conditions most bacteria are capable to induce calcium precipitation. The precipitation often occurs as a by-product of common metabolic processes such as photosynthesis, sulphate reduction and urea hydrolysis (Zhong & Yao, 2008). Bacteria also have been reported to influence precipitation by acting as sites of nucleation or calcium enrichment. In earlier research, different types of bacteria were used to contribute to calcium carbonate precipitation (Knorre & Krumbein, 2000). These bacteria are the Bacillus cereus, Bacillus sp. CT-5, Bacillus pasteurii, Shewanella pasteurii, Sporosarcina pasteurii, Bacillus sphaericus, Bacillus pseudofirmus, Bacillus cohnii, etc. An overview of the bacteria for different applications is given in table 7 (Kaur Dhami et al., 2012).

<table>
<thead>
<tr>
<th>Application</th>
<th>Organism</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bacillus sp. CT-5</td>
<td>Ramachandran et al. (2001)</td>
</tr>
<tr>
<td></td>
<td>pasteurii</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Shewanella</td>
<td>Ghosh et al. (2005)</td>
</tr>
<tr>
<td></td>
<td>Sporosarcina pasteurii</td>
<td>Achal et al. (2011a)</td>
</tr>
<tr>
<td>Remediation of cracks in con crete</td>
<td>Sporosarcina pasteurii</td>
<td>Bang et al. (2001)</td>
</tr>
<tr>
<td></td>
<td>Bacillus pasteurii</td>
<td>Ramachandran et al. (2001)</td>
</tr>
<tr>
<td></td>
<td>Bacillus pasteurii</td>
<td>Ramakrishnan (2007)</td>
</tr>
<tr>
<td></td>
<td>Bacillus sphaericus</td>
<td>De Belie et al. (2008)</td>
</tr>
<tr>
<td></td>
<td>Bacillus sphaericus</td>
<td>De Muynck et al. (2008a,b)</td>
</tr>
<tr>
<td></td>
<td>Bacillus pseudofirmus</td>
<td>Jonkers et al. (2007)</td>
</tr>
<tr>
<td>Self-Healing</td>
<td>Bacillus cohnii</td>
<td>Jonkers et al. (2007)</td>
</tr>
</tbody>
</table>

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
From earlier studies (De Muynck et al., 2008a; De Belie et al., 2008b) it appears that, among all microorganisms that were examined, the *Bacillus sphaericus* (*B. sphaericus*) was the most suitable for biodeposition applications. It showed high urease activity and a continuous formation of dense CaCO₃ crystals. Based on these researches, the *B. sphaericus* is selected to be used in this master dissertation.

The *B. sphaericus* is a gram positive bacterium (figure 19), with rod shaped cells that form spherical endospores (figure 20). A gram-positive bacteria have a very thick cell wall made of a protein called peptidoglycan, whereas, gram-negative bacteria have a very thin peptidoglycan layer that is sandwiched between an inner cell membrane and a bacterial outer membrane. Several genera of endospore-forming bacteria are distinguished on the basis of cell morphology, shape and cellular position of the endospore, relationship to O₂ and energy metabolism. The *B. sphaericus* is an endospore-forming bacteria. They are ecologically related because they are found in nature, primarily in soil (Madigan et al., 2012).

### 3.3 Biodeposition methods

In the past different biodeposition methods were determined to precipitate a CaCO₃ layer. Each of these methods have their own advantages and disadvantages. In this section a short overview of the different biodeposition treatments are given (De Muynck et al., 2010b).

#### 3.3.1 Calcite Bioconcept (France)

The Calcite Bioconcept was examined by a collaboration between the University of Nantes, the Laboratory for the research of historic monuments (LRMH) and the company Calcite Bioconcept (Le Metayer-Levrel et al., 1999). In this research, different bacteria were isolated from natural carbonate producing environments. They were screened for their carbonatogenic yield, i.e. ratio of the weight of calcium carbonate produced to the weight of organic matter input (OM). Also an optimal nutrient medium was examined.

Laboratory tests indicated that the optimal nutrient media was a media which contained a source of proteins. Additionally, a fungicide was added in the media to prevent the unwanted growth of fungi. (Orial et al., 2002). SEM images indicated that the highest performance was obtained with the *B. cereus*, which showed a carbonatogenic yield of 0.6 g CaCO₃/OM (Castanier et al., 1999).

Based on this conclusion, an in situ test was carried out. This was done on an area of 50 m² of Tuffeau limestone on the tower of the Saint Médard Church. It could be concluded that the presence of the biocalcin decreased the water absorption rate to a significant extent (5 times lower). Furthermore, no influence on the aesthetic appearance could be observed (Le Metayer-Levrel et al., 1999).

Improving the quality of recycled aggregates by biodeposition of CaCO₃
3.3.2 University of Granada (Spain)

On the Calcite Bioconcept, different limitations could be observed. Rodriguez-Navarro et al. (2003) concluded that the thickness of the bioconsolidating cement was limited to only a few microns and so the Calcite Bioconcept should be ineffective for an in-depth consolidation. Besides this, another drawback was based on the use of bacillus in stones. The formation of endospores by the bacteria may lead to germination and uncontrolled biofilm growth under appropriate conditions (i.e. temperature, humidity and nutrient availability).

Based on these limitations, the University of Granada did some laboratory tests with the *Myxococcus xanthus* (= an abundant gram-negative, non-pathogenic aerobic soil bacterium) for the creation of a consolidating carbonate matrix in the porous system of limestone. These tests, indicated that there was no uncontrolled bacterial growth and that the carbonate cementation to a depth of several hundred micro meters (>500 µm) by using the *M. xanthus*. So the latter will have better effects than the use of the *B. cereus* in the Calcite Bioconcept.

3.3.3 Biobrush consortium (UK)

The basic aim of the Biobrush (Bioremediation for Building Restoration of the Urban Stone Heritage) project was to integrate the existing knowledge on the application of microorganisms for the remediation of damaged stones. They tried to remove nitrates, sulphates and organic matter present on the surface of artworks (Gauri et al., 1992; Ranalli et al., 1999). As such, this method could be considered as a special kind of biodeposition treatment.

In the Biobrush project, bacteria were isolated from a stream in Somerset (UK) and bacteria from culture collections that had been reported to have calcifying activity, were screened for their ability to deposit calcite in solid and liquid modified B4media. From the 10 isolates that were retained and assessed for their ability to deposit calcite on stone surfaces, *Pseudomonas putida* was chosen for further study in field trials (May, 2005).

In these field trials, bacteria were applied to the stone by brushing. Subsequently, the bacteria were covered with moistened Japanese paper, above which a 1–1.5 cm thick layer of carbogel prepared with modified B4 was applied. Tris–HCl buffer was added to the Carbogel to adjust the low pH of this carrier. Finally, the gel was covered with a polyethylene sheet. As a result of this treatment a decrease of the water absorption and open porosity by 1 % and 5 %, respectively, was obtained. In order for this treatment to be effective as a consolidant, a 2 weeks treatment was observed to be necessary.

3.3.4 University of Ghent

At the Ghent University an alkaliphilic spore-forming bacteria is used to precipitate a calcium carbonate layer. They propose the microbial hydrolysis of urea as a strategy to obtain a restoring and protective calcite layer on recycled aggregates according to Eq. (4) - (7). The hydrolysis of urea presents several advantages over the other carbonate generating pathways. These main advantages are (De Belie N., 2010):

- the hydrolysis process can be easily controlled;
- it has the potential to produce high amounts of carbonate within a short period of time.

This treatment procedure is also used in this master dissertation by using the *B. sphaericus*. 

Improving the quality of recycled aggregates by biodeposition of CaCO₃
4. Effects of carbonate biomineralization

The precipitation of calcium carbonate in and around aggregates will not only influence the properties of the aggregates, but also the properties of the concrete. In the following sections, the most important effects of the calcium carbonate precipitation is given. First a closer look is taken to the effects on the aggregate properties. Afterwards on the concrete properties. The effects described are results from earlier researches. Note that these researches did not always aimed at the improvement of aggregates.

4.1 Effects on the aggregate properties

One of the main aggregate properties that will be influenced by the deposition of CaCO$_3$ is the water absorption of these aggregates. In the research of Grabiec et al. (2012) two types of recycled aggregates were treated with the *S. pasteurii*. The first aggregate was coming from concrete with a W/C–factor of 0.70 and 28-day compressive strength of 17.4 MPa, while the other aggregate was coming from concrete with W/C-factor of 0.45 and a 28-day compressive strength of 42.2 MPa. Figure 21 shows that results of the water absorption before and after treatment of these aggregates. It can be seen that the water absorption will decrease more for the aggregates from the concrete with a lower compressive strength. This offers the opportunity to improve the quality and increase the use of lower quality recycling aggregates. Better water absorption was also observed in the case of aggregates of finer grain size, confirming an average of relatively 30–35% lower water absorption than before biomodification and a relative decrease of 50% in water absorption for aggregates with particle size 12/16 mm. The general trend to lower water absorption of aggregates through the use of *S. pasteurii* shows that bacteria have a positive impact on this property of aggregates.

![Figure 21: Water absorption of recycled concrete aggregate (Grabiec et al., 2012)](image)

A: W/C = 0.45, fraction 6/8 mm  
B: W/C = 0.45, fraction 12/16 mm  
C: W/C = 0.70, fraction 6/8 mm  
D: W/C = 0.70 fraction 12/16 mm

4.2 Effects on the properties of concrete

4.2.1 Improvement in compressive strength of concrete

One of the most important characteristics of a concrete structure is his compressive strength. Recent researches show that by using microbial concrete, the compressive strength can be increased significantly (depending on the type of the bacteria). Jonkers & Schlangen (2007) concluded that the compressive strength was increased with 10% by using bacterial spores of the *B. pseudofirmus* and the *B. cohnii*. Achal et al. (2009) observed an increase of compressive strength of 17% by using *S. pasteurii* in mortar cubes. Park et al. (2010) observed an increase of 22% by using *Arthrobacter crystallopoietes*.

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
4.2.2 Reduction in permeability

An important parameter that influences the concrete durability is the permeability of concrete. The higher the permeability of concrete, the more aggressive gasses and/or liquids can penetrate into the concrete. Once they get into the concrete structure, the structure will no longer maintain its structural integrity and the lifespan will be reduced (Nolan et al., 1995). Due to these shortcomings, different researchers started to examine the effect of microbial concrete on permeation properties of the concrete.

Achal et al. looked at the influence of the *S. pasteurii* on cement mortar cubes (Achal et al., 2011). They reported a decreased water permeability on the bioremediated cubes, but the penetration of water at the sides was higher than at the top. This is due to better compaction and closing of pores at the top by the calcite precipitation. The same research group also studied the effect of *S. pasteurii* on water impermeability in concrete cubes and found similar results (Achal et al., 2010). Six times reduction in absorption of water was reported upon treatment of mortar cubes with *Bacillus sp. CT-5* as compared to untreated specimens (Achal et al., 2011).

4.2.3 Other effects

De Belie et al. (2008a) studied the effect of biodeposition of calcite by the *B. sphaericus* on mortar specimens. They observed a significant differences in carbonation depth between the treated and untreated specimens after two weeks. Due to this, it was found that the treated specimens have a better resistance towards chloride penetration as compared to untreated mortar specimens. The increased resistance was similar to that of the acrylic coating and the water repellent silanes and silicones and larger than in the case of the silanes/siloxanes mixture (Basheer et al., 1997; Ibrahim et al., 1997).

Due to the self-healing effect of biomineralized concrete, there will be a reduction of the corrosion of the reinforcement (see figure 22). Mukherjee et al. (2010) reported a fourfold reduction in corrosion of reinforced concrete specimens upon application of *Bacillus sp. CT-5*. The same research group also observed a reduction in water and chloride ion permeability upon use of calcite by *S. pasteurii*. Qian et al., (2010) used *B. pasteurii* to check its effect on permeability resistance and acid attack. They reported that bacterial calcite improves surface permeability resistance and resist the attack of acid (pH > 1.5).

Figure 22: Drawing conventional concrete (A–C) versus bacteria-based self-healing concrete (D–F) (Jonkers H., 2007)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
5. **Applications of microbial concrete**

In the past, a lot of research was done to use bacteria in concrete structures to improve the quality and life span of these constructions. This is called ‘microbial concrete’. In the following section a short overview is given of microbial concrete in practical applications (Kaur Dhami et al., 2012).

5.1 **Microbial concrete in crack remediation**

A common problem in concrete as construction material is its liability to cracking. Due to these cracks, the deterioration of concrete structures usually involves movement of aggressive gasses and/or liquids that penetrate from the surrounding into the structure. This reduces the durability of the concrete structures. Crack problems in concrete are mostly solved by impregnation with epoxy, latex binding agents, polyvinyl acetate, etc. to fill the cracks and as such, improve the durability of the structure.

These traditional repair systems also have many disadvantages such as a different thermal coefficient compared to concrete, weak bonding etc. To counteract these negative effects, many researchers try to use CaCO$_3$ deposition to repair the cracks in a natural way (Bang et al., 2001; De Belie et al., 2008a; De Belie et al., 2008b; Ramachandran et al., 2001).

5.2 **Microbial concrete in restoration of stone buildings**

Due to the calcite leaching processes, the porosity of buildings which are built with durable carbonate stones (such as the pyramids of Egypt) will increase in time. Consequently, the lifetime of the construction will be reduced. Several conservative treatments are available with inorganic and organic products, which can slow down the deterioration process of monuments (Lazzarini, 1986).

However, these products have several drawbacks. The two main drawbacks are the differences in their chemical composition and thermal expansion coefficient. So their efficiency is inconsistent. Due to the shortcomings of these conventional techniques, researchers have focussed on bacterially induced carbonate precipitation to reduce the permeation properties and thereby, enhancing the durability (Le Metayer-Levrel et al., 1999; Rodriguez-Navarro et al., 2003; Dick, et al., 2006; Tiano, 1995; Jimenez-Lopez et al., 2007).
Chapter 2  Materials

During this research the quality of recycled aggregates were improved by a treatment with a biodeposition of calcium carbonate. Concrete was made with and without the treated aggregates to investigate the effect of the treatment on the properties of recycling concrete. In addition, a concrete with a comparable granular skeleton composed of natural aggregates was made as a control.

The materials used to make concrete mixtures and the materials used for the biodeposition are described concisely in this chapter.

1.  Aggregates

Aggregates are base components of concrete. They determine the granular skeleton. Primary (natural) aggregates used in this research were provided by Sagrex, which is part of the Heidelberg Cement Group. Sagrex consists of several quarries and processing sites spread across different countries. The aggregates used in this study were all from the sites located in Belgium. The secondary (recycled) aggregates were provided by AC materials.

1.1  Natural aggregates

Natural aggregates were selected to construct a regular concrete. For the fine fraction a rounded sand 0/4 was used. This sand originated from the site of Kallo. For the coarse aggregates a 6.3/20 limestone was used. This limestone originated from the site of Cimescaut. To obtain a continuous granular skeleton a second limestone of fraction 4/6.3 was selected. This limestone originated from the site of Lemay. The product info sheets of these aggregates are given in Annex.

1.2  Recycled aggregates

Two types of recycled aggregates were chosen. First of all, a crushed concrete aggregate (RCA). According to NBN EN 12620 and Copro the composition of these aggregates result in rather high quality properties. Therefore, a second recycled material was selected with other properties and another composition; a mixed recycled aggregate (MA). This aggregate contains particles other than crushed concrete such as masonry, glass, wood, ... . The properties of these aggregate type are usually slightly worse for concrete production, hence more room for improvement exists.

Both recycled aggregates originate from the processing site of AC materials. They were crushed and sieved by AC materials such that a fraction 6.3/20 remained. This enables to replace the limestone 6.3/20 fraction from the regular concrete and retain a similar granular skeleton. As such the properties of the recycled concrete could be compared with the conventional one.

2.  Cement

The cement used in this research was a CEM III/A 42.5 N LA. The cement was produced by CBR, which is also part of the Heidelberg Cement Group. This type of cement is the most commonly used type for normal ready-mixed concrete applications in Belgium. It is a blast furnace cement where the main component is Portland clinker and the secondary component is blast furnace slag. The cement is conform NBN EN 197-1 and is a CE and Benor certified. The product description and composition can be found in Annex.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
3. Superplasticizer

The superplasticizer used in this research was provided by Sika NV. They provided a sample of a commonly used superplasticizer in the Belgian concrete industry, ViscoCrete®-1068. It is composed of Ligno and polycarboxylate ether (PCE) components. This is done to provide high performant concrete. The Ligno works as a sacrificing component to counteract small differences in concrete mixtures, cement type and absorption rates. The PCE does the actual work.

This superplasticizer can be used for conventional and recycled concrete. It prolongs the workability time and is designed for concrete with a slump class of S3 or higher. The superplasticizer is conform with EN 934-2:2002 and fulfils the specific requirements for water reducing/plasticizing admixtures and high range water reducing/super plasticizing admixtures. It also is certified by Benor and CE.

Note that PCE superplasticizers do no work optimally with cement type III. The carboxyl groups cannot attach to the slag components of the cement. They attach to the clinker but once there is no clinker left the slump retaining function of the plasticizer cannot longer be fulfilled (Sika, 2016). The choice of VC®-1068 is therefore ideal.

4. Biodeposition treatment

4.1 Bacillus sphaericus

In this study the B. sphaericus LMG 22257 (Belgian co-ordinated collections of micro-organisms at Ghent University) is used. The selection of this bacteria was based upon earlier studies (De Belie et al., 2008a), which show that these bacteria create a dense calcium carbonate deposition.

4.2 Growth medium of bacteria

The liquid culture medium for B. sphaericus is an urea yeast extract medium (UYE-medium) which consists of 20 g/l yeast extract (YE) and 20 g/l urea (VWR International, Belgium). This UYE medium has a high source of proteins which enables sufficient support for microbial growth of bacteria (De Muynck et al., 2013). Moreover this medium has already proven its success in obtaining dense highly ureolytic cultures of the B. sphaericus (De Muynck et al., 2010). This is why this medium was used.

The manufacturing of this UYE-media is done per liter. Whereby, 20 g yeast extract was dissolved in 900 ml demineralized water and 20 g urea in 100 ml demineralized water. After these two solution were made, the yeast extract solution (900 ml) was sterilized by autoclaving for 20 min at a temperature of 120 °C. The urea solution (100 ml) was separated and stored in two falcons of 50 ml. This was done by means of filtration through a sterile 0.20 µm Millipore filter (Millipore, USA).

After autoclaving the yeast extract, the solution was cooled down to room temperature. Then, both the yeast extract and urea solution were mixed together under sterile condition. This solutions serves as the growth medium for the bacterial strain.

This growth medium was used to produce larger quantities of bacteria. To do this, 2 V% B. sphaericus was added to the medium. The cultures were incubated at 28 °C on a shaker at 100 rpm for 24 hours until 10⁸ cells/ml were obtained. After this time period, the growth medium (with B. sphaericus) was ready to start the treatment of the recycled aggregates.
4.3 Biodeposition medium

The biodeposition medium was composed of urea and a calcium source. The precipitation of a dense calcium carbonate layer in and around the recycled aggregates are mainly influenced by the amount of calcium ions (Ca\(^{2+}\)) and carbonate ions (CO\(_3^{2-}\)). The latter are obtained by the microbial hydrolysis of urea in a so-called biodeposition medium (biodeposition method of the University of Gent).

In the scope of this research, biodeposition media with different Ca sources and concentrations were tested: an urea – calcium nitrate medium and an urea calcium formate medium. In each of these media, the concentration of the urea is fixed (1 M), but the concentration of the Ca-Nitrate or the Ca-Formate was varied. In table 8 and table 9 the different tested biodeposition media are mentioned. From these media, the optimal one was selected. The medium used for the treatment of the recycled aggregates had a lower concentration of urea (0.5 M).

<table>
<thead>
<tr>
<th>Table 8: Concentrations of Ca-Nitrate medium</th>
<th>Table 9: Concentrations of Ca-Formate medium</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Urea [M]</strong></td>
<td><strong>Ca(NO(_3))_2 \cdot 4 \text{H}_2\text{O} [M]</strong></td>
</tr>
<tr>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>1</td>
<td>0.50</td>
</tr>
<tr>
<td>1</td>
<td>0.75</td>
</tr>
<tr>
<td>1</td>
<td>0.90</td>
</tr>
</tbody>
</table>

Improving the quality of recycled aggregates by biodeposition of CaCO\(_3\)
Chapter 3  **Methods**

1. **Characterization of aggregates**

Characterization of the aggregates was needed for the concrete design. In addition other aggregate properties were determined to evaluate the effect of the biodeposition treatment. Sampling of the aggregates for the tests was done in accordance with EN 932-1:1996. In case reduction was needed this was done in accordance with EN 932-2:1999.

1.1  **Sieve analysis**

The particle size distribution was determined according to NBN EN 933-1:1997. The minimum mass of test proportion for normal weight aggregates is prescribed in table 1 of this normative reference. The mass of test proportion is a function of the maximum aggregate size D. Interpolation was used if the maximum aggregate size was not mentioned in the table. This resulted in minimum test proportions of 4.45 kg for the 6.3/20 fraction aggregates (limestone and recycled aggregates), 0.43 kg for the 4/6.3 fraction (limestone) and 0.2 kg for the 0/4 fraction (sand).

None of the materials were washed before testing. Dry sieving was used for all sieving analysis. In case of natural aggregates samples were dried at (110 ± 5) °C until constant mass. In case of recycled aggregates, samples were dried at (75 ± 5) °C. The sieving column existed of standardized sieves with following sieving apertures: 31.5 – 20 – 16 – 10 – 8 – 6.3 – 4 – 2 – 1 – 0.5 – 0.25 – 0.125 – 0.063 mm.

To avoid overloading of sieves, the tests samples were divided in multiple smaller samples. The dry mass before every test was recorded. The sample was put in the sieving column and was shaken mechanically. The mass retained on each sieve was recorded, in addition the material remaining in the pan was measured.

The sieving analysis was checked for overloading. Furthermore it was validated if the sum of retained masses did not differ more than 1 % from the dry mass before the test. If one of these two checks was not satisfied the test was repeated.

1.2  **Particle density and water absorption**

The particle densities and water absorption were determined according to EN 1097-6:2013. For all aggregates, the pycnometer method was used. Note that there is a difference in testing method for aggregates with particles passing the 4 mm test sieve and ones where most of the particles are retained on the 4 mm sieve.

1.2.1  **Aggregate particles passing the 31.5 mm and retained on the 4 mm sieve**

This method applies for the concrete aggregate 6.3/20, mixed aggregate 6.3/20, limestone 6.3/20 and limestone 4/6.3. Samples were taken and washed on the 4 mm sieve to remove finer particles. After washing, the material was dried until constant mass. The minimum required mass of test portions was determined by using table 2 from EN 1097-6:2013. Interpolation was used when the aggregate size was not mentioned in the table. This resulted in minimum test portions of 2.77 kg for the 4/20 fractions and 0.3 kg for the 4/6.3 fraction aggregate.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
The samples were divided over multiple pycnometers in such a way that they occupied about half of the pycnometer. The results of each individual pycnometer were added together at the end of the test. The aggregates were submerged in the pycnometer for 24 hours. The entrapped air was removed on a regular basis by gently shaking the pycnometer. After 24 hours the pycnometers were weighed. Then the aggregates were removed and their saturated surface dry mass was determined by removing the surface moisture with dry cloths. At last the materials were dried until constant mass to determine the dry weight. The natural aggregates were dried at a temperature of (110 ± 5) °C. The recycled aggregates samples were dried at (75 ± 5) °C.

From the recorded masses during the test one can calculate the particle densities and water absorption by using the following formulae:

\[
\rho_a = \rho_w \frac{M_4}{M_4 - (M_2 - M_3)} \quad (15)
\]

\[
\rho_{rd} = \rho_w \frac{M_4}{M_1 - (M_2 - M_3)} \quad (16)
\]

\[
\rho_{ssd} = \rho_w \frac{M_1}{M_1 - (M_2 - M_3)} \quad (17)
\]

\[
WA_{24} = \rho_w \frac{100 \times (M_1 - M_4)}{M_4} \quad (18)
\]

Where:
- \(M_1\) = mass of the saturated and surface-dried aggregate in air
- \(M_2\) = mass of the pycnometer containing the sample of saturated aggregate and water
- \(M_3\) = mass of the pycnometer filled with only water
- \(M_4\) = mass of the oven-dried aggregate
- \(\rho_w\) = density of water at the test temperature

The test was done in a controlled environment. The air temperature was always around 21 °C. The water temperature was also checked on a regular basis to ensure it was constant during the test duration.

1.2.2 Aggregate particles passing the 4 mm and retained on the 0.063 mm sieve

This method applies for the sand 0/4 used during this research. In general the procedures is the same as for the previous pycnometer method. Only here, the aggregates were not washed before testing. The tests sample proportion for this should not be less than 300 g according to EN 1097-6:2013. Hence, one single test sample of 300 g was used.

In addition, the aggregates were not made surface dry by using dry cloths but by using a flow of hot air. To assess whether the surface dry state has been achieved, a standardized metal cone is used. The cone is filled with some of the test material. The material is tamped as described in the normative reference. The cone is then lifted. If the material collapses after removing the cone, one can consider the material as surface dry. The rest of the test procedure is the same as for the method described in the previous section. Also, the same calculations formulae can be used.
1.3 Water absorption in time

The methods to measure the water absorption in time are based on the methods to determine the water absorption prescribed in EN 1097-6:2013. This normative reference prescribes methods for water absorption by using a pycnometer or by hydrostatic weighing.

The first method is based on the pycnometer method (Tam et al., 2008). The method is approximately the same as the one described in the previous section. Only now the weight of the pycnometers filled with water and aggregates were measured at regular time intervals. Each time the water was refilled until the level mark. This method is also known as the real-time assessment of water absorption (RAWA). The test proportion for this test was the same as for the determination of the particle densities and WA24. The test sample was again divided over different pycnometers, each of them about half full.

The second method is based on the hydrostatic weighing method (Tegguer, 2012). A test proportion of about 500 g was used for this test. The sample was brought into a wire basket which was submerged. The weight of the basket and aggregate sample was measured at regular time intervals.

For both methods the measurements were continued until no significant weight increase was noticed. These tests were executed for all coarse aggregates. The saturated aggregates were dried until constant mass at (75 ± 5) °C. The data collected during these tests was then translated to water absorption rate curves according to the following formula (Bendimerad et al., 2015):

\[
WA_t = \frac{M_t - M_{end} + m_{WA, end}}{M_4}
\]

Where:

- \(WA_t\) = water absorption at the considered time step
- \(M_t\) = mass of the container with the sample (and water) at the considered time step
- \(M_{end}\) = mass of the container with the sample (and water) at the final time step
- \(m_{WA, end}\) = mass of the water absorbed by the aggregates during the test
- \(M_4\) = mass of the oven-dried aggregate

Figure 23: Schematization of test setups for water absorption in time
The water absorption in time was also examined for aggregates during the mixing process. For each coarse aggregate type one replicate of about 12 kg was made to simulate the aggregate behaviour during concrete production. The aggregates were added into the mixer separately with some of the mixing water. They were mixed for 20 minutes. Every minute a sample of ± 250 g was taken out of the mixer. Any adsorbed water was removed by using dry cloths. As such the saturated-surface dry weight \( M_T \) was determined. The aggregates were then dried in an oven at \((75 \pm 5) \, ^\circ\text{C}\), until constant mass to determine the dry weight \( M_4 \). The WA at each time step was determined using equation (18). As such the absorption rate of the aggregates during mixing could be evaluated.

### 1.4 Resistance to wear and fragmentation

The resistance to wear is determined for all coarse aggregates according to NBN EN 1097-1:2011. The resistance to wear is characterized by the micro-Deval coefficient \( M_{DE} \). To determine this coefficient the micro-Deval apparatus is used.

In addition to the resistance to wear, the resistance to fragmentation is determined for all coarse aggregates. To determine this, the Los Angeles test method conform NBN EN 1097-2:2010 is used. The resistance to fragmentation is then defined by the Los Angeles coefficient \( LA \). A standardized Los Angeles testing machine was used.

The sample for both tests was prepared conform NBN EN 1097-1:2011. The following sieving apertures were used: \(14 \rightarrow 12.5 \rightarrow 10\) mm. A modified laboratory sample was obtained by mixing 65% of the \(10/12.5\) fraction with 35% of the \(12.5/14\) fraction. This sample was then reduced according to the requirements of EN 932-2:1999 to obtain 4 test samples of \((500 \pm 2)\) g for the micro-Deval test and a test sample of \((5000 \pm 5)\) g for the LA test.

The \( M_{DE} \)-value and \( LA \)-value were then determined according to the corresponding normative reference. The formulæ used are respectively (20) and (21). For both coefficients the weight loss was determined by measuring the retained mass on 1.6 mm grading sieve.

\[
M_{DE} = \frac{500 - m}{5} \quad (20)
\]

\[
LA = \frac{5000 - m}{50} \quad (21)
\]

Where:
- \( M_{DE} \) = the micro-Deval coefficient
- \( LA \) = the Los Angeles coefficient
- \( m \) = oven-dry mass of the oversize fraction retained on a 1.6 mm [g]

### 1.5 Mercury intrusion porosimetry

The pore size, porosity and pore size distribution was analysed using mercury intrusion porosimetry (MIP). This was examined for the recycled aggregates to examine the influence of the treatment on the porosity of the material.
Mercury does not spontaneously penetrate pores by capillary action. By applying external pressure the mercury is forced into the pores. The required pressure is inversely proportional to the pore size. One can determine the pore size distribution from the pressure versus intrusion data.

The instrument used for this test was the Pascal 140 and 440 series by Thermo scientific. It consist of a low and high pressure module, respectively the 140 and 440 series. The data acquired by these devices immediately showed the pore size distribution.

To execute the MIP-test, 3 samples were taken from every recycled material (treated and untreated). Due to the limitations of the MIP-equipment the aggregate size needed to be smaller than 10 mm. Therefore the material was sieved on a 10 mm sieve. In addition also fines were washed out. Each time a sample of 1.4 – 1.5 g was used to perform the test.

Samples for this test needed to be dried. If moisture is still present the mercury cannot penetrate the pores correctly. The samples were freeze-dried. They were submerged in liquid nitrogen for a few seconds and then they were stored in a vacuum chamber. The samples were considered dry when the mass did not differ more than 0.1 % for two successive measurements.

### 1.6 Scanning electron microscope

A scanning electron microscope (SEM) apparatus was used to investigate the surfaces of the coarse aggregates. These pictures were created for the treated and untreated aggregates. By making SEM pictures of the treated recycled aggregates, the calcium carbonate precipitation on the aggregate surface could be observed.

For the SEM-test, different samples were handpicked. The selection criteria for this was to get an aggregate with at least one rather flat side. After this selection, the different aggregates were coated under low vacuum mode with a low nitrogen pressure at 10-20 kV of acceleration voltage.

After this preparation, the SEM-pictures were made from the flat sides for each aggregate. This was done by the JEOL JSM-7600F SEM. In addition the energy dispersive spectroscopy (EDS) spectra were used to verify the chemical compositions of the crystals on the aggregate surface.
2. Concrete

2.1 Concrete mixtures

The compositions of the concrete mixtures were based on the optimal grain size distribution according to Fuller. Fuller assumes a continuous granular distribution and tries to reduce the amount of air voids between the grains. Equation (22) is based on the distance in between the aggregates, which is minimally required in order that all the grains would be able to slide in their ideal location. Thus leading to the maximum achievable filling ratio (Belgische Betongroepering, 2009). For each concrete composition an optimal granular distribution was determined.

$$Y = \sqrt{\frac{d}{D}}$$

Where:
- \(d\) = mesh width of a random sieve [mm]
- \(D\) = maximum grain size of the aggregates \(D_{\text{max}}\) [mm]
- \(Y\) = cumulated sieve passing of a sieve with mesh width \(d\) [%]

To examine the influences of the biodeposition treatment on the concrete properties, a relative comparison between treated and untreated concrete was made. Therefore, concrete was produced with untreated recycled concrete aggregates (RCAC/U) and with treated ones (RCAC/T). The same was done with the mixed aggregates (MAC/U & MAC/T). During the research also a natural aggregate concrete (NAC) was produced. This concrete would serve as sort of a reference for the performances and properties of concrete. An overview of the concrete compositions is given in table 10. The aim was to create a concrete which met the criteria for exposure class EE3. The minimal cement content for this class is 320 kg/m³ and the maximum W/C ratio is 0.5.

<table>
<thead>
<tr>
<th>Table 10: Concrete compositions</th>
</tr>
</thead>
<tbody>
<tr>
<td>NAC [kg/m³]</td>
</tr>
<tr>
<td>CEM III/A 42.5 LA</td>
</tr>
<tr>
<td>Water</td>
</tr>
<tr>
<td>Sand 0/4</td>
</tr>
<tr>
<td>Limestone 4/6</td>
</tr>
<tr>
<td>Limestone 6/20</td>
</tr>
<tr>
<td>RCA 6/20</td>
</tr>
<tr>
<td>MA 6/20</td>
</tr>
</tbody>
</table>

To minimize the variable parameters, each concrete was produced in a similar way. The coarse 6.3/20 aggregates were pre-wetted before mixing. The other aggregates were air-dried by spreading them on the floor. The water absorption of the coarse aggregates was accounted for in the mixing water. Pre-wetting was done by submerging the coarse aggregates. Therefore, 90% of the mixing water was used. This water and the aggregates were stored in a container. The container was covered to prevent evaporation. After 24 hours these aggregates were used to produce concrete. Sediments of fines were flushed from the container by using the remaining 10% of the mixing water.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
2.2 Fresh concrete

2.2.1 Workability

The consistency of the fresh concrete was tested using the slump test as prescribed by NBN E258/N 12350-2:2009. For each mixture a sample of $\pm 6$ l was taken directly after the mixing process. Samples were taken according to NBN EN 12350-2:2009. A minimum slump class S3 ($100 - 150$ mm) was intended for each concrete. If the intended consistency was not achieved, superplasticizer was added and the concrete was mixed for another minute. A new slump test was then executed. This process was repeated till a slump class of $S3$ was achieved.

When a S3 slump was achieved the consistency was also tested by using the flow test following NBN EN 12350-5. The flow was determined directly after the slump test.

2.2.2 Air content and mass density of fresh concrete

The air content of fresh concrete was tested after the intended consistency was reached. A sample was taken according to NBN EN 12350-1:2012 and tested using the manometer method conform NBN EN 12350-7:2009. Based upon the law of Boylle-Mariotte one can determine the air volume in a closed container. The manometer directly shows the air content and no further calculations needs to be done.

The density of fresh concrete was measured at the same time as the air volume. As NBN EN 12350-6:2009 describes a method where concrete is poured into a recipient with known volume. The volume of the manometer was determined with water as described in NBN EN 12350-6:2009. As such the volume is known and both tests can be executed at the same time. The wet mass density can then be calculated using the following formula:

$$D = \frac{m_2 - m_1}{V}$$  \hspace{1cm} (23)

Where:

- $D =$ mass density of concrete [kg/m³]
- $m_2 =$ mass of the recipient [kg]
- $m_1 =$ mass of the recipient filled with concrete [kg]
- $V =$ volume of the recipient [m³]

2.3 Hardened concrete

For every concrete composition, four 150x150x150 mm³ cubes were made to determine the compressive strength, water absorption, density and elastic modules. These samples were produced conforming NBN EN 12390-2:2009 and stored in a climate controlled room for 14 days. The temperature in this room was always $(20 \pm 2)$ °C and the relative humidity was over 95%.

2.3.1 Water absorption by submersion

To determine the water absorption of the concrete, the four specimens were submerged in water for 7 days after their 14 days of storage in the climate room. Then, the saturated mass of the concrete was determined conform NBN B 15-215. Specimens were then stored in a ventilated oven at $(105 \pm 5)$ °C.
for another 7 days. As such, the dry mass of the concrete could be determined. It was checked if the mass remained constant. If not, this would be noted.

The water absorption by submersion can be calculated with the following formula:

\[ A = \frac{M_1 - M_2}{M_2} \times 100 \]  

(24)

Where:
- \( A \) = water absorption of concrete by submersion [m%]
- \( M_1 \) = saturated weight of concrete [g]
- \( M_2 \) = oven-dry weight of concrete [g]

2.3.2 Mass density

The mass density of the hardened concrete was determined for all four cubical samples. The height, width, and depth of every sample was measured using a digital calliper. For each dimension three measurements were executed. From these measurements the volume was calculated. The weight was also measured using a scale. The mass density of the hardened concrete was then calculated using the following formula:

\[ D = \frac{M}{V} \]  

(25)

Where:
- \( D \) = mass density of concrete [kg/m³]
- \( M \) = mass of concrete [kg]
- \( V \) = volume of concrete [m³]

The obtained density is the oven-dry density since all samples were oven-dry as a result of the water absorption by submersion test.

2.3.3 Modulus of elasticity

The modulus of elasticity of the hardened concrete was determined by the ultrasonic device 0-4500μs from Macben. The measurements were executed on all four cubical samples by the method described in NBN EN 12504-4:2004. The ultrasonic device measures the time needed for an ultrasonic wave to travel through the concrete. From this, the modulus of elasticity can be calculated using equation (30) (Olympus, sd).

\[ E = \frac{\rho(1-\nu)(1-2\nu)}{1-\nu} \times V^2 \]  

(26)

Where:
- \( E \) = modulus of elasticity [N/m²]
- \( V \) = ultrasonic velocity [m/s]
- \( \rho \) = density of concrete [kg/m³]
- \( \nu \) = Poisson’s ratio [-], assumed to be 0.20 (Afsar, 2012)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
2.3.4 Compressive strength

The compressive strength of the hardened concrete was determined on all four cubical samples. This was done according to NBN EN 12390-3:2009, after 28 days of hardening. The compression apparatus by MFL system used to execute this test is conform NBN EN 12390-4. Samples were placed in this apparatus with their upper mould surface turned to the observer. As such, the compressive force was exerted on two completely flat sides. The load was increased at a ratio imposed by NBN EN 12390-3 until failure. The compressive strength can be calculated using the following formula:

\[ f_{c\text{ cub }150} = \frac{F}{A_c} \]  (27)

Where:

- \( f_{c\text{ cub }150} \) = compressive strength of concrete for a cube with sides of 150 mm [N/mm²]
- \( F \) = load at failure [N]
- \( A_c \) = loaded area [mm²], here 150 x 150 mm²

As described in the literature review, the compressive strength depends on the shape of the sample. Therefore, the results from equation (27) are multiplied by a factor 0.789 to obtain the cylindrical compressive strength \( f_c \). Both \( f_c \) and \( f_{c\text{ cub }150} \), can be used to check if the intended compressive strength class is obtained. The strength classes and respective characteristic compressive strengths are given in table 11.

<table>
<thead>
<tr>
<th>Class</th>
<th>C8/10</th>
<th>C12/15</th>
<th>C16/20</th>
<th>C20/25</th>
<th>C25/30</th>
<th>C30/37</th>
<th>C35/45</th>
<th>C40/50</th>
</tr>
</thead>
<tbody>
<tr>
<td>( f_c )</td>
<td>8</td>
<td>12</td>
<td>16</td>
<td>20</td>
<td>25</td>
<td>30</td>
<td>35</td>
<td>40</td>
</tr>
<tr>
<td>( f_{c\text{ cub }150} )</td>
<td>10</td>
<td>15</td>
<td>20</td>
<td>25</td>
<td>30</td>
<td>37</td>
<td>45</td>
<td>50</td>
</tr>
</tbody>
</table>

The characteristic value of the compressive strength was determined using equation (28). This is conform EN 1990 (Annex D7), taking into account the standard variation. The characteristic value depends on the number of samples and the prior knowledge on the coefficient of variation (\( V_x \)).

\[ f_{cx} = \bar{x} - k_n \times s \]  (28)

Where:

- \( f_{cx} \) = characteristic value of concrete compressive strength [MPa]
- \( \bar{x} \) = mean value [MPa]
- \( k_n \) = multiplication factor [-], here 2.63
- \( s \) = standard deviation of the compressive strength for \( n \) samples [MPa]

The value for \( k_n \) is derived from table 12 which corresponds to table D1 form EN 1990.

<table>
<thead>
<tr>
<th>( n )</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>8</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>( \infty )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( V_x ) known</td>
<td>2.31</td>
<td>2.01</td>
<td>1.89</td>
<td>1.83</td>
<td>1.80</td>
<td>1.77</td>
<td>1.74</td>
<td>1.72</td>
<td>1.68</td>
<td>1.67</td>
<td>1.64</td>
</tr>
<tr>
<td>( V_x ) unknown</td>
<td>-</td>
<td>-</td>
<td>3.37</td>
<td>2.63</td>
<td>2.33</td>
<td>2.18</td>
<td>2.00</td>
<td>1.92</td>
<td>1.76</td>
<td>1.73</td>
<td>1.64</td>
</tr>
</tbody>
</table>

Improving the quality of recycled aggregates by biodeposition of CaCO₃
3. Biodeposition treatment

3.1 Concentration of the bacteria

After growing the *B. sphaericus* in the conditions as explained in “Chapter 2: Materials”, the concentration of the bacteria was checked by use of an optical density spectrophotometer (Dr. Lange ISIS 9000). Firstly, a cuvette of 3 ml was filled with demineralized water, to calibrate the optical density spectrophotometer. Afterwards, a dilution of the UYE-medium was made by filling a cuvette with 2.9 ml demineralized water and 100 µl bacteria. This cuvette was put in the optical density meter to determine the absorbance measurements at 610 nm (Abs$_{610}$). Based on this value, the colony-formit unit/ml (CFU/ml) in the diluted solution was determined with formula (29) (Wang J., 2013).

\[
CFU/ml = 10^{(0.87 + Abs_{610} + 7.381)}
\]  
(29)

Afterwards, the dilution factor was taken into account to know the bacteria concentration in the UYE-medium. If this concentration was lower than $10^8$ cells/ml, the bacteria was further incubated until a concentration between $10^8$ cells/ml and $10^9$ cells/ml was obtained.

3.2 Optimization of biodeposition medium

The optimal biodeposition medium was determined by testing two media: an urea/Ca-Nitrate biodeposition medium and an urea/Ca-Formate biodeposition medium. For each of these media, different concentrations of Ca-Nitrate or Ca-Formate were tested. These concentrations were already tabulated in table 8 and table 9. Besides different Ca-Nitrate or Ca-Formate concentrations, also the amount of the bacteria was changed. This was done by making a sodium chloride (NaCl) solution, with a bacteria concentration of $10^{10}$ cells/ml. Afterwards 2 or 10 ml of the NaCl-solution was solved in 100 ml of each biodeposition medium. This corresponds with a bacteria concentration of respectively 2 x $10^8$ cells/ml and $10^9$ cells/ml. For each biodeposition medium concentration, with a certain amount of bacteria, three replicates were tested.

3.2.1 Light wave spectrometer: colorimetric method

The amount of decomposed urea was measured after the *B. sphaericus* was added to the different biodeposition media. The measurements were executed after three and five days by using the colorimetric method of the light wave spectrometer. This method defines the amount of decomposed urea by measuring the total ammoniacal nitrogen (TAN) in a liquid sample.

For each biodeposition medium (and each concentration of bacteria), three replicates were made with the same dilution factor ($f$). The dilution was made by pipetting 0.5 ml in a flask of 100 ml and afterwards pipetting 1 ml of the 100 ml flask in a 50 ml flask. This corresponds with a dilution of 10000. After the preparation of every flask of 50 ml, 1 ml of the potassium sodium tartrate solution (KNaC$_{4}$H$_{4}$O$_{6}$·4H$_{2}$O) and 1 ml of the Nessler reagent was added. After mixing and waiting for 10 minutes, the obtained colour was measured at 425 nm and a value for the concentration ammoniacal nitrogen was given. The concentration of decomposed urea was determined with equation (30).

\[
conc(urea) = \frac{conc(N) * M(urea)}{M(2N)} * f
\]  
(30)
3.2.2  pH-meter

After performing the colorimetric method, also the pH-values of the different media are measured. This value is an indicator for the amount of decomposed urea. The pH was measured by the Metrohm 744 pH Meter with a 6.0228.000 electrode from Metrohm (Herisau, Switzerland). Before measuring, the equipment was calibrated by standardized solutions with pH-values of 4 and 7.

3.3  Optimization of treatment procedure

Based on the efficiency of the biodeposition treatment, the optimal treatment procedure to form a dense calcite layer was determined. The efficiency of the treatment methods was examined on four replicates (± 200 g) of each type of the recycled aggregates.

The samples used for these tests were first washed on the 4 mm sieve to remove fine particles. Before the treatment of these aggregates could be started, the water absorption after 24 hours (WA_{24}) for each replicate was determined. After the treatment, the WA_{24} of the replicates were determined again to see if there was any reduction in the water absorption due to the treatment. Besides this, also the weight increase due to the treatment was checked.

The WA_{24} was determined with a simplified method. Instead of using a pycnometer or the wire-basket method the aggregates were simply submerged for 24 hours by pouring water into the recipient. After 24 hours the water was discarded. The aggregates were removed from their recipient and any adsorbed water was removed by using dry cloths. As such the saturated-surface dry weight (M_1) was determined. The aggregates were then dried in an oven at (75 ± 5) °C, until constant mass to determine the dry weight (M_4). The WA_{24} was determined using equation (18).

To determine the weight increase, the washed aggregate samples were first dried in an oven at (75 ± 5) °C. If a constant mass was obtained, the aggregate samples were treated according a certain treatment procedure. After the treatment procedure, the aggregates were again dried in the same oven. If a constant mass was obtained, the weight increase could be determined by subtracting the dry masses before and after the treatment.

In this research, three treatment procedures were used. More explanation about these different treatment procedures are given in the following sections. Based on the results of the water absorption before and after treatment and the weight increase, the optimal treatment procedure and application method is determined. Those will be used to treat the aggregates designated for the concrete tests.

3.3.1  Treatment procedure I

Spray

A fist method of application is the spray method. Four replicates of each type of the recycled aggregates (± 200 g) were sprayed with the growth culture (10^6 cells/ml – 10^9 cells/ml). It was chosen to spray one, two or three times with these bacteria. Between the different spray actions, the aggregates were sprayed with a 0.5 M Ca-Nitrate/urea biodeposition medium. Afterwards, the aggregates were sprayed for three more days with this medium. The time intervals between spraying the Ca-Nitrate/urea medium was determined by the time that the aggregates needed to become surface dry. The three spray methods that were described above, are schematically given in figure 26.
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$

### Methods

#### Treatment procedure I (spray methods)

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>a)</td>
<td>Spray (1x): UYE medium ($10^7$ – $10^8$ cells/ml)</td>
</tr>
<tr>
<td>b)</td>
<td>Spray (2x): UYE medium ($10^7$ – $10^8$ cells/ml)</td>
</tr>
<tr>
<td>c)</td>
<td>Spray (3x): UYE medium ($10^7$ – $10^8$ cells/ml)</td>
</tr>
</tbody>
</table>

**Figure 26: Treatment procedure I (spray methods)**

#### Immersion

A second method of application is the immersion treatment method. Four samples of each type of recycled aggregate were firstly immersed for 12 hours in an 24h growth culture ($10^8$ – $10^9$ cells/ml). After this time period, the bacterial suspension is removed and the aggregates are immersed for 3 days in the 0.5 M Ca-Nitrate/urea biodeposition medium. A schematic overview of this treatment procedure is given in figure 27.

**Figure 27: Treatment procedure I (immersion method)**

#### Treatment procedure II

After the incubation of the *B. sphaericus* culture, the bacteria were centrifuged and were suspended in a 8.5 g/l NaCl-solution. By dissolving the bacteria in the NaCl-solution, it was intended to increase the amount of the bacteria until a concentration of $10^{15}$ cells/ml was obtained. After this preparation, the treatment of the aggregates can start. Firstly, four replicates of each type of the recycled aggregates were immersed for 12 hours in the NaCl-solution. Afterwards the bacterial suspension is removed and the aggregates are immersed for 3 days in the 0.5 M Ca-Nitrate/urea biodeposition medium. A schematic overview of this treatment procedure is given in figure 28.

**Figure 28: Treatment procedure II**
3.3.3 Treatment procedure III

In this treatment procedure, the two recycled aggregates were firstly immersed for 12 hours in the UYE-medium with $10^8$ cells/ml – $10^9$ cells/ml. Afterwards, the same samples were immersed in a biodeposition medium. This medium consisted of 0.5 M Ca-Nitrate/urea. 5 g/l yeast extract (YE) was added to the biodeposition medium, to improve the precipitation of CaCO$_3$. A schematic overview of treatment procedure III is given in figure 29.

![Figure 29: Treatment procedure III](image)

3.3.4 Ultrasonic pulse test

The amount of the CaCO$_3$ precipitation is not the only parameter that will improve the quality of the recycled aggregates. A more important parameter is the cohesion strength of the CaCO$_3$ precipitation to the aggregates. This cohesion was examined by the ultrasonic pulse test.

In the ultrasonic pulse test, 4 samples (± 70 g) of each recycled aggregate and each treatment procedure were tested. These samples were placed on a 1 mm sieve and were submerged in the demineralized water recipient of the Haver USC 200-76. The samples were sonicated for 5 min at a frequency of 46 kHz with ultrasonic waves. Samples were collected and dried until constant mass (weight change of less than 0.1% between two measurements with a 24 h interval). Subsequently, the weight loss of each sample was measured. The weight loss was used as a parameter for the cohesion strength of the CaCO$_3$ precipitation.
Chapter 4  Research and results

The aim of this research was to verify if the quality of recycled aggregates could be improved by use of a biodeposition treatment. The aggregate properties with regard to concrete production were of vital importance. Especially the reduction of the water absorption.

To verify this the research was split up into three separate stages. In a first stage the biodeposition treatment itself was investigated. The optimal medium and concentration were determined. In addition the optimal aggregate treatment application method was examined.

Once the biodeposition medium and the treatment procedure was optimized, the next stage could start. There the influences on the recycled aggregate properties were examined. At the same time the influences of the treatment on concrete properties was studied in a third research part. For both parts the optimal biodeposition medium and treatment procedure was applied.

The results of these research phases are documented in this chapter. The materials and methods applied during this research are described in chapter 2 and 3.

**PART I: BIODEPPOSITION TREATMENT**

1. Optimization of biodeposition medium

As mentioned in chapter 2, two types of biodeposition media were examined. The optimal biodeposition medium will be used in the further course of the research. To determine this optimum the amount of decomposed urea in time was measured.

1.1 Biodeposition medium I: urea & calcium nitrate

For a first biodeposition medium a calcium nitrate solution was used. Figure 30 represents the relative percentages of decomposed urea (in time) for different concentrations of the Ca-Nitrate. To increase the decomposition rate, $10^{11}$ cells of the *B. sphaericus* is added to this biodeposition medium (100 ml). This corresponds with a concentration of $10^9$ cells/ml. Figure 30 indicates that the highest decomposition rate occurs for the highest Ca-Nitrate concentration. Beside this, it can be concluded that after one day almost 80-100% of the urea is decomposed. This was valid for all Ca-Nitrate concentrations.

![Figure 30: Decomposed urea of the Ca-Nitrate solution ($10^9$ B. sphaericus cells/ml)](image-url)

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
During the decomposition of urea, the pH-value changes (cfr. chapter 1 section 0). The pH-values in function of time for the different Ca-Nitrate solutions are shown in figure 31. The pH-values were always higher than 8 and increased in time. The highest increase occurred within the first day. This corresponds with a high decomposition rate of the urea (see figure 30). Figure 31 also indicates that the higher the concentrations of Ca-Nitrate was, the lower the pH-values measured at a certain time were.

![Figure 31: pH-values of the Ca-Nitrate solution (10^9 B. sphaericus cells/ml)](image)

For lower concentrations of *B. sphaericus*, the decomposition of urea will go much slower. This is visualized by figure 32, where only 2x10^{10} bacteria cells (instead of 10^{11}) was added to the different Ca-Nitrate solutions (100 ml). This corresponds with a concentration of 2x10^8 cells/ml. It can be seen that for the 0.25 M and the 0.9 M Ca-Nitrate solution, the decomposition after one day was respectively only 33.27% and 6.83%. Furthermore, it can be concluded that only a small amount of the urea was decomposed if the concentration of Ca-Nitrate was too high (> 0.50 M), even after 5 days. As such, more time was needed to have a sufficient amount of decomposed urea. The decomposed urea after 5 days for the 0.25 M and 0.50 M Ca-Nitrate solutions respectively were 76.94% and 63.31%, while the 0.75 M and 0.90 M solutions showed only 24.04% and 9.71% decomposed urea. Another difference that can be observed when using 2x10^8 *B. sphaericus* cells/ml (instead of 10^9 cells/ml) was that for higher Ca-Nitrate concentrations, the amount of the decomposed urea was smaller (the opposite was observed for 10^9 cells/ml).

![Figure 32: Decomposed urea of the Ca-Nitrate solution (2x10^8 B. sphaericus cells/ml)](image)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Research and results

Figure 33 represents the pH-value during the decomposition of urea for the case of $2 \times 10^8 B.\ sphaericus$ cells/ml. It can be seen that in the first days, there was a decrease in the pH-value, due to the slow decomposition rate of the urea. At the fifth day, the pH-value increased again due to the increased decomposition of urea. Again it can be stated that the higher the Ca-Nitrate concentration, the lower the pH-value was.

![Figure 33: pH-values of the Ca-Nitrate solution (2x10^8 B. sphaericus cells)](image)

### 1.2 Biodeposition medium II: Urea & Ca-Formate

For the second biodeposition medium, calcium formate was used instead of calcium nitrate. Again the amount of decomposed urea (in function of time) was determined for different concentrations of calcium source. $10^{13} B.\ sphaericus$ cells were added to the biodeposition medium (100 ml). This again corresponds with a concentration of $10^9$ cells/ml. Similar as for Ca-Nitrate, it can be seen that after one day already a high amount of the urea (> 80%) was decomposed. After the first day, only a small further increase was witnessed. Figure 34 indicates that the decomposition of urea is larger for higher concentrations of Ca-Formate.

![Figure 34: Decomposed urea of the Ca-Formate solution (10^9 B. sphaericus cells/ml)](image)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
The pH-values corresponding with this decomposition of urea is given in figure 35 (10^9 B. sphaericus cells/ml). The pH-values were always higher than 8. For all concentrations of Ca-Nitrate, there was an increase in the pH-value in time. The highest increase was observed after the first day. This indicated a large amount of decomposed urea within the first day (similar as for the Ca-Nitrate biodeposition medium). Besides this, it can be witnessed that for an urea/Ca-Formate solution, the pH-value was higher for lower concentrations of Ca-Nitrate. This was the opposite observation from the Ca-Nitrate biodeposition medium.

![Figure 35: pH-values of the Ca-Formate solution (10^9 B. sphaericus cells/ml)](image)

Again the concentration of the B. sphaericus was adjusted to 2x10^8 cells/ml. As such, the decomposition of urea went much slower. This can be observed in figure 36, where the decomposed urea in function of time is given. Figure 36 also indicates that after one day only a small amount of urea was decomposed. So similar as in figure 32 (Ca-Nitrate with 2x10^8 B. sphaericus cells/ml), more time was needed to have a high amount of decomposed urea. If the concentration of Ca-Formate was too high (≥ 0.50 M), it can be detected that after 3 days just a small amount of the urea (less than 50%) was decomposed. This was the case for the 0.50 M, 0.75 M and 0.9 M Ca-Formate solution. Even after 5 days the amount of decomposed urea was just 44.70% (0.75 M), 42.33% (0.50 M) and 38.73% (0.90 M). The decomposition rate for these three concentrations was in the same range, but the 0.25 M Ca-Formate solution had a higher decomposition rate. Similar as for 2x10^8 B. sphaericus cells/ml in a Ca-Nitrate solution, the amount of the decomposed urea was smaller for higher Ca-Formate concentrations.

![Figure 36: Decomposed urea of the Ca-Formate solution (2x10^8 B. sphaericus cells/ml)](image)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Figure 37 represents the pH-value during the decomposition of urea with $2 \times 10^8$ B. *Sphaericus* cells/ml in a Ca-Formate biodeposition medium. It can be seen that for the biodeposition medium with 0.25 M Ca-formate, there was an immediate increase in the pH-value after one day. For higher concentrations of Ca-Formate there was a decrease in pH-value in the first three days. After these days, the pH-value increased, due to the higher amount of decomposed urea. It can also be concluded that the higher the Ca-formate concentration, the lower the pH-value will be. This is similar as the $2 \times 10^8$ B. *sphaericus* cells in the Ca-Nitrate biodeposition medium.

![Figure 37: pH-values of the Ca-Formate solution (2x10^8 B. sphaericus cells/ml)](image)

1.3 Conclusion

Figure 30 until figure 37 show that the amount of decomposed urea is mainly influenced by 3 parameters: the concentration of the bacteria, the concentration of Ca$^{2+}$ and the duration of the test.

For a high concentration of the bacteria ($10^9$ cells/ml), there was already a high amount of decomposed urea after just one day (Ca-Nitrate: 77-100%; Ca-Formate: 80% - 100%). Furthermore, it can be observed that for higher concentrations of Ca-Nitrate or Ca-Formate, there will be more urea decomposed.

When the concentration of the B. *sphaericus* was adjusted to $2 \times 10^8$ cells/ml, it can be concluded that more time was needed to acquire the same amount of decomposed urea. For $2 \times 10^8$ cells/ml, a lower concentration of Ca-Nitrate or Ca-Formate indicated a higher amount of decomposed urea. This was the opposite observation as the one noticed for the $10^9$ B. *sphaericus* cells/ml.

Based on the results, it can be concluded that the concentration of the bacteria may not be too high to have a sufficiently slow CaCO$_3$ precipitation. Due to this, it was chosen to work with a growth medium with $10^8$ B. *sphaericus* cells/ml and a 0.5 M Ca-Nitrate biodeposition medium (with 0.5 M urea), in the next stages of the research. The measurements indicated that for this biodeposition medium a decent amount of urea was decomposed after 3 days. Based on this observation, it was also concluded that the duration of the biodeposition treatment for the recycled aggregates must be at least 3 days.

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
2. Optimization of treatment procedure

In this section an optimal biodeposition treatment procedure was evaluated. Originally the application method was analysed with the optimal medium from the previous section. As will become clear from the results, the effect of the treatment was small. Therefore some adjusted media were used in an effort to further improve the biodeposition medium. Details about the different application methods and used biodeposition media can be found in chapter 2 and 3 (materials and methods).

2.1 Weight increase of the aggregates

A first evaluation of the effectiveness of the biodeposition treatment was done by means of the weight increase of the specimens. This weight increases is directly related with the amount of CaCO\(_3\) precipitation. Results for treatment procedure I with different processes of application (immersion and spray method) are tabulated in table 13. A graphical overview of these results is given in figure 38.

Table 13: Weight increase [%] for treatment procedure I

<table>
<thead>
<tr>
<th></th>
<th>Immersion</th>
<th>3 x Spray</th>
<th>2 x Spray</th>
<th>1 x Spray</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>RCA</td>
<td>0.71 ± 0.07</td>
<td>0.63 ± 0.26</td>
<td>0.39 ± 0.04</td>
<td>0.82 ± 0.07</td>
<td>0.21 ± 0.51</td>
</tr>
<tr>
<td>MA</td>
<td>1.33 ± 0.06</td>
<td>0.39 ± 0.02</td>
<td>0.47 ± 0.02</td>
<td>0.40 ± 0.05</td>
<td>0.13 ± 0.12</td>
</tr>
</tbody>
</table>

As can be seen from figure 38, the highest weight increase was observed for the immersed aggregates. The weight increase for this method fluctuates between 0.41% and 0.89% for the RCA. The weight increase for the mixed recycled aggregate was much higher. They vary between 1.21% and 1.47% relative weight increase. This was probably caused by the higher porosity of these aggregates.

If a closer look was taken to the spray methods, it could be observed that there was no significant difference in weight increase between the different methods, nor was there a remarkable difference compared to the reference. This indicated that just a small amount of CaCO\(_3\) precipitation was formed with these methods.

From these results it was decided to use immersion as standard treatment procedure in the remaining research. This method resulted in the best effect, with the most significant weight increase, thus the best precipitation, for both aggregate types.
In table 14 and figure 39, the weight increase for the different treatment procedures (I, II and III) with immersion are given. It can be seen that the lowest weight increase was caused by treatment procedure I. On the other hand, the highest weight increase was caused by treatment procedure III. The weight increase for the latter lied between 1.48% and 2.32% for the RCA. For the mixed recycled aggregate an even higher increase was noticed, varying between 2.95% and 3.20%. By applying treatment procedure II, it could be seen that the weight increase of the RCA was about the same as for treatment procedure I. For the MA, the weight increase of treatment II was between the values of treatment I and III: 1.71% and 2.81%.

<table>
<thead>
<tr>
<th>Table 14: Comparison weight increase [%] for the different treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment</td>
</tr>
<tr>
<td>Treatment I</td>
</tr>
<tr>
<td>Treatment II</td>
</tr>
</tbody>
</table>

Not only the absolute weight increase is of importance, but also the quality of the precipitation. Therefore the cohesion strength of the CaCO$_3$ is of great interest. It was observed that the cohesion for treatment procedure I was best. More about this in section 2.3 of this chapter. Based on this observation, it was decided to analyse the effect of multiple treatments. As such, in addition to the different treatments procedures, batches of aggregates were also treated two times with procedure I. Results from this are shown in table 15 and figure 40. A significant weight increase for both types of aggregates could be witnessed. In case of RCA an increase from 0.72% - 0.81% to 2.05% - 2.19% was observed. For the MA, the weight increase changed from 1.21% – 1.47% to 2.18% – 2.54%.

<table>
<thead>
<tr>
<th>Table 15: Comparison weight increase for one or two times treatment I</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment</td>
</tr>
<tr>
<td>Treatment I (1x)</td>
</tr>
<tr>
<td>Treatment II</td>
</tr>
</tbody>
</table>

Figure 39: Comparison weight increase for the different treatments

Figure 40: Comparison weight increase for one or two times treatment I

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
2.2 Water absorption of the aggregates

The precipitation of CaCO\textsubscript{3} does not only induce a weight increase, but also a change in water absorption. This is because the CaCO\textsubscript{3} precipitation will partly or completely block and/or fill the pores of the aggregate. Figure 41 and figure 42 give an overview for the water absorption (WA\textsubscript{24}) of the aggregates (RCA & MA) before and after treating them according to the different application methods used with treatment procedure I.

![Figure 41: Water absorption for treatment procedure I (RCA)](image1)

![Figure 42: Water absorption for treatment procedure I (MA)](image2)

To get a better insight in the change in WA\textsubscript{24}, figure 43 was made. This graph indicates that there was no significant difference in the water absorption changes for the different application methods of procedure I. An overview of these results is given in table 16.

<table>
<thead>
<tr>
<th>Treatment Method</th>
<th>RCA</th>
<th>3 x Spray</th>
<th>2 x Spray</th>
<th>1 x Spray</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Immersion</td>
<td>0.24 ± 0.11</td>
<td>0.40 ± 0.26</td>
<td>0.11 ± 0.18</td>
<td>0.33 ± 0.25</td>
<td>0.12 ± 0.26</td>
</tr>
</tbody>
</table>
Improving the quality of recycled aggregates by biodeposition of CaCO₃

Research and results

In table 17 and figure 44, the average decrease in WA₂₄ for the different immersion methods of treatment I, II and III are given.

Table 17: Comparison water absorption decrease [%] for the different treatments

<table>
<thead>
<tr>
<th></th>
<th>Treatment I</th>
<th>Treatment II</th>
<th>Treatment III</th>
</tr>
</thead>
<tbody>
<tr>
<td>RCA</td>
<td>0.24 ± 0.11</td>
<td>0.12 ± 0.09</td>
<td>0.05 ± 0.18</td>
</tr>
<tr>
<td>MA</td>
<td>0.04 ± 0.20</td>
<td>0.04 ± 0.04</td>
<td>0.70 ± 0.04</td>
</tr>
</tbody>
</table>

For treatment procedure I, only a decrease between 0.06% and 0.33% was visible for the RCA. For the MA, almost no decrease was observed. Even some increases could be observed. For treatment procedure II no significant decrease at all could be monitored. If 5 g/l YE was added to the biodeposition medium (treatment III), a more convincing decrease of the water absorption could be witnessed in case of MA (0.58% - 0.76%). But again, no significant decrease was visible for the RCA.

From the previous results, it can be concluded that the decrease in water absorption was relatively low. Especially when one considers that the water absorption of the aggregates was one of the main parameters monitored in this research to demonstrate the quality of the recycled aggregates. This
rather low decrease was one of the main reasons to investigate multiple treatments. As stated before, it was chosen to use procedure I two times. The effect of this double treatment on the water absorption is summarized in figure 45 and table 18. It can be concluded that no significant supplementary decrease in absorption of the RCA could be observed. But, for the MA the WA24 decrease increased greatly, to 0.80% - 1.52% after a second treatment. This is quite the success, given the fact there was just an average decrease of 0.04% after a first application of the treatment.

| Table 18: Comparison water absorption decrease [%] for one or two times treatment procedure I |
|---------------------------------|---------------------------------|
| Treatment I (1x) | Treatment I (2x) |
| RCA | 0.24 ± 0.11 | 0.28 ± 0.01 |
| MA | 0.04 ± 0.20 | 1.18 ± 0.21 |

Figure 45: Comparison water absorption decrease for one or two times treatment procedure I

2.3 Ultrasonic pulse test

As stated before, not only the amount of precipitation and the decrease of the water absorption are of importance. To select the optimal treatment procedure another, more important parameter is the cohesion strength of the CaCO₃ precipitation. This cohesion strength was analysed by applying the ultrasonic pulse test. The weight loss during this test was considered as a representation of this cohesion strength. This was determined for the different (immersion) treatment procedures.

Table 19 and figure 46 represent the relative mass losses of the treated aggregates. The graph indicates that the lowest mass loss was obtained for treatment procedure I. This was the main motive to use this treatment procedure as standard treatment during the remainder of the research. When procedure I was applied a second time, the mass losses were even lower (RCA: about 0%, MA: 0.046 % - 0.080 %).

Figure 46 also indicates that the mass loss for the reference MA was higher than those of the RCA (respectively 0.35% - 0.49% compared to 0.14% - 0.20%). Furthermore, it can be observed that the mass loss for the reference samples was larger than those of the treated ones.

| Table 19: Mass loss [%] for the different treatment procedures |
|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
|                                | Reference | Treatment I (1x) | Treatment I (2x) | Treatment II | Treatment III |
| RCA                             | 0.16 ± 0.02 | 0.12 ± 0.03    | 0.00 ± 0.00      | 0.21 ± 0.02 | 0.20 ± 0.01   |
| MA                              | 0.41 ± 0.04 | 0.10 ± 0.01    | 0.07 ± 0.01      | 0.34 ± 0.06 | 0.19 ± 0.03   |
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
2.4 Scanning electron microscope

As mentioned in “Chapter 3: Methods”, the CaCO₃ precipitation on the recycled aggregates (RCA & MA) was observed by use of a scanning electron microscope (SEM). Pictures were taken for each type of bio-treated aggregate (application method: submersion). The results were compared with the images of the respective untreated aggregates. The SEM-images of the untreated RCA and MA are given in figure 49 and figure 50.

![Figure 49: RCA 200x magnification (a), 1000x magnification (b)](image)

![Figure 50: MA 200x magnification (a), 1000x magnification (b)](image)

For treatment procedure I (with 1x immersion), it was observed that a homogeneous CaCO₃ layer was formed on the surface of both recycled aggregates (RCA and MA). This homogeneous CaCO₃ layer is clearly visible on the 200x magnification SEM-images (figure 51 and figure 52). From these images it can also be concluded that a larger amount of CaCO₃ crystals were formed on the MA surface, than on those of the RCA. This is visible on the 1000x magnification images in figure 51 and figure 52. These images indicate a hexagon CaCO₃ crystal shape with a diameter of around 7.5 μm on the MA, while on the RCA the diameter is only around 4 μm. For dual treated aggregates it can be seen that a bigger amount of CaCO₃ crystals were formed, compared to the singly treated ones (treatment procedure I). The 500x magnification SEM-images of the dual-treated aggregates are given in figure 53 and figure 54. Same as for single-treated aggregates, a larger amount of CaCO₃ crystals could be noticed on the SEM-picture of the MA, compared to the one of the RCA. Due to the double treatment not only more crystals are formed, also a bigger crystal diameter is observed. The crystal diameter (hexagon) for both aggregate types is around 15 μm.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Research and results

TREATMENT PROCEDURE I (immersion; 1x)

Figure 51: RCA/T-I 200x magnification (a), 1000x magn. (b) and EDS spectra at point 1 (c)

TREATMENT PROCEDURE I (immersion; 2x)

Figure 53: RCA/T-I (2x) 200x magnification (a), 500x magn. (b) and EDS spectra at point 1 (c)

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO\textsubscript{3}

Treatment procedure I can be considered as a reference procedure for the biodeposition treatment. As stated before, the other procedures used an increased amount of bacteria or yeast extract. The impact of these adjustments could not be properly determined from previous results. The results did contradict each other to a certain degree. The cause of this contradictions, and the real influences of these adjustments could be well observed from the SEM analysis.

In treatment procedure II, the concentration of the \textit{B. sphaericus} was increased by three compared to procedure I and III. It was observed that in certain areas of the aggregate surface, a high amount CaCO\textsubscript{3} precipitation was formed. But, on other places almost no CaCO\textsubscript{3} was formed. The reason for this can be found in the higher concentration of the bacteria, as described in section 1 of this research part. The increased decomposition rate of urea will result in a non-uniform precipitation of CaCO\textsubscript{3}. Figure 55 and figure 57 represent the SEM-images at a location where a high amount of CaCO\textsubscript{3} was formed on the aggregate surfaces. On the other hand, figure 56 shows SEM images at a location on the RCA surface where the amount of CaCO\textsubscript{3} precipitation was much lower. These images also indicate that a round crystal shape is formed on the aggregates. The diameter of the crystals on the MA surface is around 20 μm, while on the RCA surface the crystal diameter is only around 7.5 μm.

In treatment procedure III, the concentration of the \textit{B. sphaericus} was not increased, but 5 g/l YE was added in the biodeposition medium. This will increase the precipitation speed. The concentration of yeast extract is mainly growth related. An increased amount of yeast extract will cause an increasing amount of microorganisms. This is accompanied with an increased amount of ureolytic bacteria and thus an elevated urease activity (De Muynck et al., 2008b). The faster CaCO\textsubscript{3} precipitation will once more result in a less uniform CaCO\textsubscript{3} layer on the aggregate surfaces. This could be well observed for the RCA. Figure 55 shows a location on the surface where a high amount of precipitation was noticed. Figure 56 represents the SEM-images at a location where a low amount of CaCO\textsubscript{3} was formed. The results for MA were different. There a more uniform CaCO\textsubscript{3} was observed (figure 57). These images again indicate that a round crystal shape is formed on the aggregates. The size of the crystals has the same magnitude as for treatment procedure II.
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of $\text{CaCO}_3$
The general conclusion that can be made from the SEM-images is that an increased precipitation speed does not necessarily results in a better treatment. The aim of the treatment is to obtain a continuous CaCO$_3$ layer. As such the pores are blocked and water absorption of the aggregates would decrease. But elevating the urease activity by increasing the amount of bacteria or yeast extract did not cause a uniform precipitation (only another crystal shape with a bigger CaCO$_3$ crystal diameter was observed). If a closer look is taken to the crystals, it can also be seen that the bacteria imprints are visible on the CaCO$_3$ crystals. (see figure 61)

To analyse the impact of the double treatment and the ultrasonic bath, SEM-pictures were made before and after the ultrasonic pulse test. Figure 62 and figure 63 show the treated RCA respectively before and after this test (treatment procedure I; 2x). From these pictures, it can be concluded that the homogeneous CaCO$_3$ crystal field remained after undergoing sonication. For the MA, the same conclusion can be made (figure 64 and figure 65). Hence, it can be stated that procedure I results in a homogenous and firm precipitation of CaCO$_3$.
2.5 Conclusion

This part of the master dissertation research concerned the search for an optimal treatment procedure for the recycled aggregates. The biodeposition treatment results in precipitation of CaCO$_3$ in the pore structure and on the surface of the recycled aggregates. Due to this, there will be an increase in weight and a possible decrease in water absorption.

Research concerning different methods of application (immersion, spraying) indicated that the most increase of weight was obtained by submerging the aggregates in the different biodeposition media. As a larger increase in weight indicates more precipitation of CaCO$_3$, this method was considered as the most effective application method.

In addition to the various application methods, different treatment procedures (I, II and III) were examined. The lowest weight increase was obtained for treatment procedure I. The highest weight increase was noticed for treatment procedure III. This indicates that increasing the concentration of yeast extract in the biodeposition medium, results in more CaCO$_3$ precipitation. The weight increase for procedure II was also higher compared to procedure I, especially for MA. This indicates that more precipitation is also formed when a higher concentration of bacteria is used.

The results for the decrease in water absorption ($WA_{24}$) showed comparable developments as the weight increase. The highest decrease in water absorption was obtained with treatment procedure III, especially for the MA. On the contrary, the decrease in absorption for the RCA was minimal, despite the relatively high weight increase. From the SEM-images it became clear that this was caused by a non-uniform precipitation of CaCO$_3$. This was also the fact for treatment procedure II, resulting in small water absorption decreases for these treatment procedures.

Based on the weight increase data one could state that procedure III is the optimal one. However, as results from water absorption and SEM showed, this is not the fact. Not only the amount of precipitation is important but also the distribution over the aggregate surface. In this context procedure I is the most optimal one, with the most uniform distribution of the CaCO$_3$ precipitation.

Another important parameter is the cohesion strength of the CaCO$_3$. From the results of the ultrasonic pulse tests it could be concluded that the best cohesion was also obtained with treatment procedure I. The larger cohesion strength can be attributed to the slower hydrolysis rate of urea, followed by a steady deposition of the CaCO$_3$, as could be observed from the SEM-images.

Based on all these observations and results, it can be concluded that the most optimal treatment procedure is procedure I (growth medium: 20 g/l YE, 20 g/l urea, $10^8$-$10^9$ B. sphaericus cells; biodeposition medium: 0.5 M urea and 0.5 M Ca-Nitrate). The quality improvement of the aggregate is relatively low, but steady. In this light a second application of this treatment was executed. SEM-pictures before and after the ultrasonic pulse test with a secondary treatment concluded that the homogeneous CaCO$_3$ crystal field was retained. The added value was mainly visible for MA, where a notable supplementary decrease in water absorption was observed. For the RCA the additional decrease was minimal. Therefore it was decided that the optimal treatment for RCA was a single immersion treatment (procedure I) while for MA a double immersion treatment (procedure I) was optimal. These methods will be applied in the next research stages.
PART II: AGGREGATE PROPERTIES

1. Sieve analysis

For all aggregates used in this master dissertation the sieve grading was determined. These were used to design the concrete composition. The results of these tests are given in figure 66.

The different coarse aggregates (6.3/20) are located in the same range. This is beneficial for the concrete composition because this will result in comparable granular skeletons. MA contains some more fines compared to the other coarse aggregates. The limestone (4/6.3) has a steep grading curve, which indicates a pure and narrow range of grain diameters. This is a logical result of the fact that these were used to complete the continuous granular distribution.

2. Particle density and water absorption

The particle densities and water absorption after 24 hours (WA_{24}) of the different aggregates are given in table 20. The particle densities which are shown in this table, are: the oven-dried particle density ($\rho_{\text{rd}}$), the saturated surface-dried particle density ($\rho_{\text{sdd}}$) and the apparent particle density ($\rho_a$).

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>$\rho_{\text{rd}}$ [kg/m$^3$]</th>
<th>$\rho_a$ [kg/m$^3$]</th>
<th>$\rho_{\text{sdd}}$ [kg/m$^3$]</th>
<th>WA_{24} [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand (0/4)</td>
<td>2670</td>
<td>2740</td>
<td>2700</td>
<td>0.94</td>
</tr>
<tr>
<td>Limestone (4/6.3)</td>
<td>2680</td>
<td>2730</td>
<td>2700</td>
<td>0.73</td>
</tr>
<tr>
<td>Limestone (6.3/20)</td>
<td>2650</td>
<td>2700</td>
<td>2670</td>
<td>0.63</td>
</tr>
<tr>
<td>RCA/U (6.3/20)</td>
<td>2350</td>
<td>2660</td>
<td>2460</td>
<td>4.98</td>
</tr>
<tr>
<td>RCA/T (6.3/20)</td>
<td>2320</td>
<td>2620</td>
<td>2440</td>
<td>4.83</td>
</tr>
<tr>
<td>MA/U (6.3/20)</td>
<td>2120</td>
<td>2480</td>
<td>2270</td>
<td>6.77</td>
</tr>
<tr>
<td>MA/T (6.3/20)</td>
<td>2150</td>
<td>2440</td>
<td>2270</td>
<td>5.42</td>
</tr>
</tbody>
</table>
To get a better insight in the difference between the different aggregates figure 67 and figure 68 were made. These figures represent the densities and the water absorption of the aggregates in a graphical way. In figure 67, the aggregate properties for all the untreated aggregates are given, while figure 68 shows a relative comparison between the treated and untreated recycled aggregates.

**Figure 67: Particle density and water absorption of the untreated aggregates**

**Figure 68: Particle density and water absorption of the treated and untreated aggregates**

Figure 67 indicates that the highest density (for all aggregate types) is the apparent particle density (ρ<sub>a</sub>). Pores are not included in the volume for the determination of ρ<sub>a</sub>. As such, the volume will be smaller, which logically results in a higher density. The apparent particle densities of the recycled aggregate types are lower than the those from the natural aggregates. Recycled aggregates normally are more porous. Although the apparent density does not account these pores, the test method used cannot determine all pores. Some pores are not accessible by water and are not excluded from the particle volume. Due to the higher porosity, the probability of inaccessible pores is bigger. Beside this, some materials with lower densities are present in the recycled aggregates. These two aspects result in a lower apparent particle density for recycled aggregates. As indicated in figure 67, the apparent density of the RCA and the MA are respectively 2660 kg/m³ and 2480 kg/m³, while the limestone (6.3/20) has an apparent density of 2700 kg/m³. The apparent particle density of RCA is just slightly lower than natural aggregates because it originates from concrete.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Research and results

The lowest particle density witnessed, is the oven-dried particle density \( (\rho_{rd}) \). The pores of the particle are included in the volume for the determination of \( \rho_{rd} \). Hence, the density will decrease. \( \rho_{rd} \) for recycled aggregates is lower due to higher porosity of these materials.

The saturated surface dried particle density \( (\rho_{ssd}) \) includes the pore volume, but filled with water. \( \rho_{ssd} \) always lies between the values of \( \rho_{rd} \) and \( \rho_{a} \). This is fairly obvious as the density of water is lower than stone material and higher than air. Again, \( \rho_{ssd} \) of recycled material is lower than the natural materials because of the higher porosity.

The higher porosity of recycled aggregates can also be observed in the water absorption of the aggregates. The water absorption for the RCA and the MA is respectively 4.98% and 6.77%, while the water absorption for limestone (6.3/20) is only 0.63%.

A higher the water absorption will result in larger differences between the different particle densities (NBN EN 1097-6, 2013). This results from the fact that the only difference between the various densities is the volumes of the pores (whether or not filled with water). This trend can be noticed in figure 67. The different densities of the natural aggregates are close together, while those of the recycled aggregates do vary a great deal.

The biodeposition treatment of the aggregates resulted in some changes in the aggregate properties (cfr. figure 68 for a relative comparison between treated and untreated aggregates). Due to the CaCO₃ precipitation, the amount of pores accessible by water reduced. This resulted in lower water absorptions and a smaller variation between the different particle densities. The reduction in water absorption of the RCA and the MA is respectively 0.15% and 1.35%.

3. Water absorption in time

The water absorption in time for the different aggregates was determined with the pycnometer method and the hydrostatic method. In addition the absorption rate for aggregates in the mixer was also analysed as described in “Chapter 3: Methods”.

The results for the different test methods in the first 20 minutes are shown in figure 69 (limestone 6.3/20), figure 70 (RCA 6.3/20) and figure 71 (MA 6.3/20). From these graphs it can be detected that the water absorption with the pycnometer method was higher than those of the hydrostatic method. The water absorption difference for the two methods after 20 minutes was 0.02% for the natural aggregates, 1.15% for the RCA and 2.42% for the MA. These differences are possibly caused by the testing method, as will be explained in “Chapter 5: Discussions” (section 1.2.1).

The water absorption in time during mixing of the aggregates in the concrete mixer was the highest of all, for all time steps and all aggregates. To make a comparative analysis the absorption during mixing was tested for washed material. In a first phase, the intention was to pre-wet the aggregates in the mixer. Therefore, also unwashed materials were tested. As exhibited by the results, the water absorption test in the mixer went rather difficult. No decent conclusion could be made from these results and they were not considered in the remainder of the research.

The though and inconclusive measurements of the absorption rate in the mixer was one of the reasons why the pre-wetting method as described in “Chapter 3: methods” was used, instead of pre-wetting in the mixer. A further discussions of the results is given in “Chapter 5: Discussions” (section 1.2.1).

Improving the quality of recycled aggregates by biodeposition of CaCO₃.
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Besides the absorption of the untreated aggregates, the water absorption (in time) of the treated aggregates were examined. To minimize the influence of the executor, the water absorption of the treated aggregates was determined with the hydrostatic method. Afterwards, a comparison could be made with the water absorption of the untreated aggregates (by applying the hydrostatic method). Results are shown in figure 72 (72 hours) and figure 73 (2 hours).

As indicated in figure 72, the full water absorption of the recycled aggregates (RCA and MA) is respectively 5.49% and 6.69%. After treating the recycled aggregates the full water absorption of the aggregates was respectively 6.31% and 5.17% which correspond to a decrease of 0.32% for the RCA and 0.38% for the MA.

The course of the water absorption curve changed for MA (figure 72). The bio-treated MA behave more like RCA, with a fast absorption rate in the start and a gradual transition to a relatively slow absorption rate during the remaining course. The untreated ones have a faster absorption rate in the beginning (fast absorption of the high porous material) and a sharp transition to a rather continuous absorption rate. Even after 72 hours, the aggregates are not yet fully saturated (but close). No notable difference is noticed in the course of the curve between RCA and RCA/T. Obviously, the curve for limestone is much lower and a constant value is reached way earlier then the recycled aggregates.

As has been repeatedly mentioned, an important parameter during the concrete production is the amount of free water. This water can influence the effective W/C-factor and consequently the durability and properties of the concrete. Notable absorption of mixing water due to the aggregates is expected to happen in the first moments after contact (± 1 to 2 hours). Therefore, a closer look was taken to the water absorption during the first two hours (figure 73). As can be seen, the water absorption by treating the aggregates results in a water absorption decrease of 0.39% for the RCA and 0.30% for the MA.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$

4. Los Angeles and micro-Deval

In this section the influence of the biodeposition treatment on the resistance of the aggregates to wear (MDE) and fragmentation (LA) is examined. In figure 74, the LA-coefficients of the different aggregates are given, while figure 75 shows the corresponding MDE-coefficients.

The results show higher LA-coefficients for recycled aggregates than those for natural aggregates. The untreated recycled aggregate (RCA/U) and the untreated mixed aggregate (MA/U), have a LA-coefficient of respectively 30% and 40%, while the limestone 6.3/20 (NA) only has a LA-value of 17%. This indicates that the natural aggregate has the highest resistance to fragmentation, while the MA/U has the lowest one.

When recycled aggregates were treated a decrease in LA-value could be noticed. For the RCA only a decrease of 1% is visible, while for the MA, the LA decreased by 4%. The reduction indicates that the aggregates will have a larger resistance to fragmentation, as a result of the biodeposition treatment. A possible reason for this is that due to the formation of CaCO$_3$ a higher aggregate strength is obtained.

If a closer look is taken to the resistance against wear (figure 75), it can be concluded that the recycled aggregates have a higher MDE-coefficient compared to the natural aggregates. Bio-treated aggregates show no significant difference in MDE-coefficient compared to untreated ones. This indicates that the treatment has no great influence on the resistance against wear.

In general it can be concluded that the treatment has no significant influence on the resistance against wear and fragmentation. The MA do show some decrease in LA, but despite this, the values are still relatively high compared to natural aggregates.
5. Mercury intrusion porosimetry

By treating the recycled aggregates, the pores of the RCA and the MA will (partly) be filled with CaCO$_3$ precipitation. Some of the pores are also blocked due to the CaCO$_3$ layer covering the aggregates. The influence on the (for mercury accessible) pores was analysed with MIP.

In table 21 and figure 76, the porosity of the treated and untreated recycled aggregates are given. As can be seen, the porosity changes from 7.79 % to 7.03 %, which corresponds with a decrease of 9.8%. For the MA, a decrease of 40.7% was obtained by applying the biodeposition treatment. This corresponds with a decrease of the total pore volume from 15.66 % to 9.27 %. Based on these results, it can be concluded that the decrease of porosity for the MA was 4x larger compared to the RCA. These decreases indicate that higher quality aggregates can be obtained, especially for the MA.

<table>
<thead>
<tr>
<th>Sample</th>
<th>RCA/U</th>
<th>RCA/T</th>
<th>MA/U</th>
<th>MA/T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>10.40</td>
<td>9.94</td>
<td>18.61</td>
<td>7.78</td>
</tr>
<tr>
<td>Sample 2</td>
<td>8.71</td>
<td>4.20</td>
<td>12.71</td>
<td>10.76</td>
</tr>
<tr>
<td>Sample 3</td>
<td>4.26</td>
<td>6.95</td>
<td>/</td>
<td>/</td>
</tr>
</tbody>
</table>

|            | 7.79 ± 1.83 | 7.03 ± 1.66 | 15.66 ± 2.41 | 9.27 ± 1.22 |

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of \( \text{CaCO}_3 \)

The \( \text{CaCO}_3 \) precipitation does not only influence the total porosity. The formation of \( \text{CaCO}_3 \) crystals also changes the pore distribution. Figure 77 and figure 78 show the total pore distribution of respectively RCA and the MA. In figure 77, it can be seen that for the RCA two peaks occurs. The highest peak is observed for a pore diameter of 0.010-0.100 \( \mu \text{m} \), while a lower peak occurs around 1 \( \mu \text{m} \). For the MA only one peak can be observed around 1 \( \mu \text{m} \) (figure 78). This indicates that for MA, more larger pore diameters occur compared to RCA, which explains the higher porosity of this aggregate type. The pore distribution of the untreated MA, shows a higher pore volume than RCA. This conclusion was also obtained from figure 76.

If a closer look is taken to the pore distribution (figure 77 and figure 78), it can be observed that not all the pore diameters are influencd by the \( \text{CaCO}_3 \) precipitation the same way. Apparently, the pore diameter must have a certain minimum dimension to allow the \( \text{Ca}^{2+} \) and the \( \text{CO}_3^{2-} \) ions to form \( \text{CaCO}_3 \) crystals. This could mainly be noticed by the fact that the peak around 1 \( \mu \text{m} \) was moved and decreased for the bio-treated samples. In figure 79 and figure 80, a close-up of the pore distribution of the RCA and the MA are given. For the RCA, it can be seen that the treatment mainly influence the pore diameters of 1-13 \( \mu \text{m} \). For the MA, the same conclusion can be made (see figure 80).
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
6. Conclusion

This part of the master dissertation research concerned the properties of the used aggregates and the influence of the biodeposition treatment on these properties.

As could be concluded from the first phase of the research, a biodeposition treatment caused precipitation of CaCO₃ on the aggregate surface and in the pores. As marked before, this resulted in a weight increase. Accompanied with these weight increase a decrease in water absorption was observed. From the particle densities results it can be concluded, that the precipitation of CaCO₃ does inhibit the water absorption by blockage of the water accessible pores. This conclusion is based on the fact that the range of different particle densities becomes smaller.

These observations are a first indication of an actual quality improvement for the recycled aggregates. The water absorption curves do however weaken this statement. The full water absorption (72 hours) does not change a lot. The short term (2 hours) difference is also rather small. This indicates that the pores are somehow still partially accessible for water. The result from LA and MD tests also weaken this statement. No substantial difference can be detected between the untreated and treated aggregates. Only for the mixed aggregates a notable improvement in LA-coefficient could be observed.

Then again, the results from MIP showed a substantial improvement due to the treatment. The bio-treated aggregates did show a significant decrease in pore volume compared to the untreated ones. This strengthens the statement that a biodeposition treatment can improve the quality of aggregates. Note however the large variance on the results from MIP. Maybe the most important conclusion that could be made here is that the biodeposition treatment is limited to a certain range of pore sizes. Especially larger pores seem to be affected by the biodeposition treatment.
1. Consistency

1.1 Slump

As indicated in chapter 3, the slump test was executed to check if the anticipated consistency class (S3 – slump 10 to 15 cm) was obtained. If not, superplasticizers (SP) was added and mixing was continued. The slump results and the added amount of SP are summarized in figure 81.

Figure 81 indicates that for the concrete mixtures: NAC and the MAC/U the intended consistency class was directly achieved after the mixing process. The slump was even a bit more than intended. The added amount of the superplasticiser was respectively 35 ml and 30 ml. In contradiction, RCAC/U had an initial slump of only 3 cm. Due to this, the mixing process was continued until a sufficient slump was achieved. This was the case when 60 ml superplasticiser was added to the concrete mixture.

If a closer look is taken to the concrete mixtures with treated aggregates, it can be observed that relative large amounts of SP needed to be added for low slump values. For the RCAC/T mixture a slump of only 2 cm (S1) was measured, after adding 70 ml superplasticiser. Also for the MAC/T, a slump of merely 7 cm was measured, with 30 ml SP. The same amount of superplasticizer was deliberately added to the concrete with untreated and corresponding treated ones. As such, a relative comparison could be made. These show a considerable decrease in slump after bio-treatment (RCA: 8 cm, MA: 9 cm lower slump). The concrete with bio-treated aggregates were not even able to achieve an S3 class.

![Figure 81: Slump test of the different concrete mixtures](image)

1.2 Flow

When a S3 slump was achieved (if possible - figure 81), or a comparable amount of SP was added, the consistency was tested with the flow test. Due to the testing principle, the result of this test are more accurate. Hence, a more precise indication of the workability can be given and a better evaluation can be made. Therefore, flow test are gaining more importance over the slump test in practice (Sika, 2016).
In figure 82 an overview of the results is given. It is observed that for all the mixtures, except the RCAC/U an consistency class F3 (42 - 48 cm) was achieved. This is to some extend comparable to a consistency class S3. Note however that there is no relation between slump and flow values. But, in contrary to the slump results, the consistency of the mixtures was in the same range. And, although the slump test indicated the consistency of the RCAC/T and the MAC/T was too low (figure 81), still a sufficient consistency was achieved according to the flow test, for all concrete mixtures.

Based on these results, not much can be concluded from the effect of the treatment. Obviously, the consistency of recycled aggregates concrete is lower than natural concrete, even for higher amounts of SP. For the effect of the treatment on RCAC not much could be concluded. RCAC/T had a higher consistency but also more SP was used, so it is unsure if this was caused by the SP or by the treatment. Due to the substantial amount of SP, the first possibility seems the most plausible one. However, a large contrast with the slump results is observed, which showed a large decrease in consistency for the concrete with bio-treated aggregates. MAC/T showed a slight decrease in flow compared to MAC/U.

2. Air content

The air content of fresh concrete has important repercussions on the durability of the concrete. The air content of the concrete mixtures was measured before casting. In figure 83, an overview of the results is given. As indicated in this figure, the air content of the different concrete mixtures was always lower or equal to an air content of 2.5%. No excessively large air contents could be noticed, nor any large variations. All air contents were about the same and around the intended value of 2%.
3. Density

In the following sections some more information is given about the wet and dry density of the different concrete mixtures (table 10). Not only the results are discussed, also a reference to the literature is made where possible, to clarify the observed outcomes.

3.1 Wet density

In figure 84, an overview of the measured wet density is given. This figure indicates that the highest value is obtained for the reference concrete without recycled aggregates (NAC). The wet density for this mixture was 2420 kg/m³. As indicated in table 20, the recycled aggregates had a lower density than the natural aggregates. As stated in “Chapter 1: Literature review”, the density of concrete depends on the particle density of the aggregates. As such, the wet density of the RCAC/U and MAC/U should be lower than for the NAC mixture. The wet density for the RCAC/U and MAC/U were respectively 2330 kg/m³ and 2260 kg/m³. This corresponds with a decrease of 3.9% and 6.6% compared to the NAC mixture.

The wet density for the RCAC/U and the MAC/U were 2330 kg/m³ and 2260 kg/m³, while the RCAC/T and MAC/T had a wet density of 2420 kg/m³ and 2300 kg/m³. This corresponds with an increase of 3.9% and 1.8%. For the MAC this could be attributed to the increase in particle densities (cfr. “Part II: Aggregate properties”, section 2). For the RCAC the increase in density can originate from the lower air content of the respective fresh concrete.

![Figure 84: Wet density of the different concrete mixtures](image)

3.2 Dry density

In table 22, the measured dry density for the different concrete cubes is given. Figure 85 was made, to get a better insight in the differences for the various concrete mixtures. This graph indicates a similar trend as for the wet densities. The concrete mixtures with recycled aggregates again had a lower density compared to the natural concrete. This can be attributed to the lower particle densities of the recycled aggregates. Beside this, again a higher densities of concrete mixtures with bio-treated aggregates were noticed, compared to their untreated associate. The dry density of the RCAC/T and MAC/T were respectively 2330 kg/m³ and 2160 kg/m³, while those of the RCAC/U and MAC/U were only 2240 kg/m³ and 2140 kg/m³. This corresponds with an increase of respectively 3.6% and 1.0%.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Table 22: Dry density [kg/m³] of the different concrete mixtures

<table>
<thead>
<tr>
<th></th>
<th>NAC</th>
<th>RCAC/U</th>
<th>RCAC/T</th>
<th>MAC/U</th>
<th>MAC/T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube 1</td>
<td>2370</td>
<td>2240</td>
<td>2320</td>
<td>2150</td>
<td>2180</td>
</tr>
<tr>
<td>Cube 2</td>
<td>2340</td>
<td>2230</td>
<td>2340</td>
<td>2110</td>
<td>2170</td>
</tr>
<tr>
<td>Cube 3</td>
<td>2360</td>
<td>2260</td>
<td>2330</td>
<td>2150</td>
<td>2160</td>
</tr>
<tr>
<td>Cube 4</td>
<td>2330</td>
<td>2250</td>
<td>2320</td>
<td>2130</td>
<td>2140</td>
</tr>
</tbody>
</table>

Table 23: Water absorption by submersion [%] of the different concrete mixtures

<table>
<thead>
<tr>
<th></th>
<th>NAC</th>
<th>RCAC/U</th>
<th>RCAC/T</th>
<th>MAC/U</th>
<th>MAC/T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube 1</td>
<td>3.65</td>
<td>5.17</td>
<td>3.73</td>
<td>6.41</td>
<td>5.23</td>
</tr>
<tr>
<td>Cube 2</td>
<td>3.76</td>
<td>5.14</td>
<td>3.68</td>
<td>6.91</td>
<td>5.29</td>
</tr>
<tr>
<td>Cube 3</td>
<td>3.65</td>
<td>5.02</td>
<td>3.69</td>
<td>6.37</td>
<td>5.32</td>
</tr>
<tr>
<td>Cube 4</td>
<td>4.03</td>
<td>5.07</td>
<td>3.75</td>
<td>6.94</td>
<td>5.60</td>
</tr>
</tbody>
</table>

Figure 85: Dry density of the different concrete mixtures

4. Water absorption by submersion

The water absorption of hardened concrete is mainly influenced by the porosity and the continuity of the pore structure in the concrete. It can serve as an indication for the durability of the concrete as pores accessible by water make the concrete vulnerable for harmful substances.

Results are summarized in table 23 and figure 86. The water absorption of the concrete with untreated recycled aggregates was larger compared to NAC. The water absorptions of the RCAC/U and MAC/U mixtures were respectively 5.10% and 6.60%, while those of NAC was 3.78% on average.

Water absorption of the concrete with treated aggregates was lower than the respective untreated ones. A decrease of 1.39% (RCAC) and 1.30% (MAC) was observed, by using bio-treated recycled aggregates instead of untreated ones (figure 86). This corresponds with a water absorption of 3.71% for the RCAC/T mixture and 5.36% for the MAC/T.

These results indicate a less porous structure for concrete with bio-treated aggregates, hence a more durable concrete. This can be caused by lower porosity of the aggregates or by a change in the effective W/C ratio. An elaborate discussion on this topic is given in “Chapter 5: Discussions”.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$

5. Compressive strength

The most important property of concrete is the compressive strength. This property is influenced by different parameters as discussed in “Chapter 1: Literature review”. One of them is the use of recycled aggregates in the concrete skeleton. Another is the shape of the concrete sample. The compressive strength was determined with cubical samples but the cylindrical concrete strength was also calculated.

In table 24, the cubical compressive strength of the different concrete cubes are given. Based on this table, figure 87 and figure 88 were made, which represent the cubical and cylindrical characteristic compressive strength. The characteristic compressive strength (cubical) of the reference concrete mixture (NAC/U) was 56.3 N/mm$^2$, while the RCAC/U and MAC/U indicated lower values, respectively 49.8 N/mm$^2$ and 37.7 N/mm$^2$. This corresponds with a decrease of 10.7% and 32.1% compared to the reference.

The concrete with treated aggregates showed higher compressive strengths (figure 87 and figure 88). The characteristic (cubical) compressive strength of the corresponding concrete mixtures increased by 32% (RCAC) and 5% (MAC). This corresponds with a $f_{ck,cub}$ value of 65.7 N/mm$^2$ for the RCAC/T mixture and 40.1 N/mm$^2$ for the MAC/T mixture.

As for the water absorption of concrete these results can indicate a lower porosity of the concrete. Possible explanations are lower porosities of the aggregates or a change in water content. The relatively high compressive strength for RCAC/T makes it doubtful whether the observed changes are caused purely by the treatment. More about this in “Chapter 5: Discussions”.

Table 24: Cubical compressive strength $f_{ck,cub}$ [N/mm$^2$] of the different concrete mixtures

<table>
<thead>
<tr>
<th></th>
<th>NAC</th>
<th>RCAC/U</th>
<th>RCAC/T</th>
<th>MAC/U</th>
<th>MAC/T</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube 1</td>
<td>63.6</td>
<td>54.7</td>
<td>71.7</td>
<td>40.4</td>
<td>53.2</td>
</tr>
<tr>
<td>Cube 2</td>
<td>61.0</td>
<td>52.7</td>
<td>79.3</td>
<td>45.1</td>
<td>51.6</td>
</tr>
<tr>
<td>Cube 3</td>
<td>59.0</td>
<td>56.1</td>
<td>75.1</td>
<td>45.3</td>
<td>53.3</td>
</tr>
<tr>
<td>Cube 4</td>
<td>61.1</td>
<td>52.7</td>
<td>81.0</td>
<td>45.3</td>
<td>44.8</td>
</tr>
</tbody>
</table>

|        | 61.2 ± 0.9| 54.1 ± 0.8| 76.8 ± 2.1| 44.0 ± 1.2| 50.7 ± 2.0 |

Figure 86: Water absorption by submersion of the different concrete mixtures
6. Modulus of elasticity

The modulus of elasticity indicates the stiffness of the material. The E-modulus of concrete is largely determined by those of the aggregates which are present in the concrete. In general recycled aggregates have a lower strength and a higher porosity. Therefore, the E-modulus of recycled concrete is expected to be lower than the natural concrete. This is also indicated by the results of the ultrasonic elasticity measurements shown in table 25 and figure 88. The E-modulus for the NAC mixture was 40.6 GPa, while for the recycled concrete (RCAC/U and MAC/U) only an E-modulus of 33.2 GPa and 27.8 GPa was observed. This corresponds with a decrease of 18.2% and 31.6%.

Results from “Part II: Aggregate properties” suggested that treated aggregates have a reduced porosity. As such, concrete with these aggregates was expected to have a higher E-modulus than the respective concrete with untreated ones. The results indicated that this statement was true. An increase in E-modulus of 32.6% (RCAC) and 27.4% (MAC) was witnessed. The E-modulus of the RCAC/T mixture and the MAC/T mixture was respectively 44.1 GPa and 35.4 GPa.

Note that another probable cause of these observations can be a change in W/C ratio. As for the compressive strength, a sceptical attitude needs to be taken towards the huge E-modulus observed for RCAC/T.
7. Broken concrete parts

After performing the compressive test, some small concrete parts of the different concrete mixtures were collected for further evaluation.

7.1 Visual inspection

In figure 90 till figure 93, some of the collected concrete parts are shown. If a closer look is taken to these pictures, it is visible that for some aggregates present in these parts were split in half. This indicates that these aggregates were part of the failure crack pattern. This observation was not only visible for the concrete mixtures with pure recycled aggregates (figure 90 and figure 92), but also for the ones with treated aggregates (figure 91 and figure 93). Based on these observations, it can be stated that the compressive strength is partially determined by the strength of the aggregates. As such, the compressive strength of the concrete might tell something about the aggregate strength, thus the influence of the bio-treatment on the aggregate strength.
Improving the quality of recycled aggregates by biodeposition of CaCO₃

7.2 Porosity

Besides the aggregate porosity, also the porosity of the cement matrix was evaluated by using some of the collected broken concrete parts. The results are shown in figure 94. The porosities of the concrete mixtures with untreated recycled aggregates were 7.21% (RCAC/U) and 11.78% (MAC/U), while for the mixtures with treated aggregates porosities of 6.10% (RCAC/T) and 5.77% (MAC/T) were measured. This corresponds with a decrease of 15.4% and 51.0% respectively.

In figure 95, the total pore distribution of the different concrete mixtures is given. It can be seen that two significant peaks occurred. The highest peak is observed for a pore diameter around 0.100 μm, while a lower peak occurred around 10 μm. The first peak of the MAC/U mixture was much higher than the one of the MAC/T mixture. For the RCAC/U and RCAC/T mixtures barely any difference in pore size distribution could be observed.
These results indicate a positive effect for concrete with bio-treated aggregates. The observations are in line with previous ones. Although, one should be cautious with these results. They show a decrease in porosity of the cement matrix. This might indicate a change in effective W/C ratio, which was not intended. It hast to be pointed out that still some aggregate particles were present in the mortar. Since these were split, cfr. figure 90 till figure 93, the full pore structure of the aggregates is accessible for mercury. This potentially influenced the test result, which makes it difficult to draw well-founded conclusions with regard to the true effect of the biodeposition treatment.

8. Conclusion

The examined concrete properties showed a positive development with respect to quality and durability. Water absorption and MIP tests indicated a decrease in porosity for the concrete with bio-treated aggregates, despite the relatively constant air content. In addition, the density increased. Furthermore, the modulus of elasticity and compressive strength increased.

The measurements showed similar trends as for the aggregate properties, which can possibly explain some of the changes. However, one has to be cautious before drawing conclusions. It might be possible that not all observed effects are caused by the treatment. As can be witnessed, some changes are out of proportion, which make them questionable. There are some indications that the effective W/C ratio is different for the mixtures with treated aggregates compared to those with the untreated ones. The concrete mixtures with treated recycled aggregates showed a lower porosity. Also other properties like compressive strength, E-modulus and water absorption by submersion indicated a change in porosity. All these changes could be linked to a decrease in effective water-cement ratio. However, how the bio-treated aggregates affect the effective W/C ratio, or if they are the genuine cause for the change, is still unknown (see discussion).

Further research will need to be executed to establish if the variations are caused by the biodeposition treatment or other influencing parameters. Some results do indicate a positive effect which could be attributed to the change in aggregate properties. As such, it can be concluded that the treatment surely shows potential for concrete applications.
Chapter 5 Discussion

1. Discussion on research and results

In this section observations and results from the research executed in this master dissertation are discussed. Mostly things that were expected but which could not be proven or observations which need to be looked at with some scepticism are described.

1.1 The biodeposition treatment

Before this master dissertation a decent amount of studies were executed on the topic of biodeposition treatments. However, none were executed on the field of aggregate improvement for concrete production, some studies did concern concrete applications. From these previous studies B. sphaericus seemed to be the optimal choice. These bacteria were also used in this research, without any questions or analysis on the topic.

1.1.1 Application method

Different application methods were evaluated: spray and submersion method. The one where aggregates were immersed seemed to be the best. It must however be mentioned that the spraying method also looked promising. Despite the test results did not show quite the effect, a lot of precipitation was visible. Firstly, some of this precipitation did not took place on the aggregate. Secondly, these tests were executed in a ventilated room. This did cause premature drying out of the different treatment media. Optimizing this method and the environmental conditions could result in larger amounts of precipitations in the aggregate pore structure. This optimization could cause a homogeneous CaCO$_3$ precipitation on the aggregate surface which could result in a sufficient decrease of the water absorption.

The submersion method was also not close to optimal. The effectiveness is terribly low. Firstly, this could be caused by the little amount of bacteria which were absorbed in the aggregate pores. This logically results in a low CaCO$_3$ precipitation in the aggregate pores. Secondly, it is also possible that the bacteria absorption in the aggregates was relatively good, but by submerging the aggregates in the biodeposition medium, the bacteria could have come back into suspension. These two aspects, or a combination of both, could cause the low effectiveness. Beside this, it was observed that a large amount of the calcium carbonate precipitation was found on the bottom of the containers used for these submersion treatments. Furthermore the precipitation, which did happen on the aggregate surface, was mainly formed on the upper side of the top aggregates in the containers. These observations could be caused by gravity and the insufficient absorption of bacteria in the pore structure.

For the spray and immersion method, also environment conditions could cause the relatively low water absorption decrease. Some more research need to be examined to determine the influence of the humidity, temperature, etc. on the CaCO$_3$ precipitation.

It also has to be pointed out that recycled aggregates contain a large amount of fines. These have a relatively high absorption ratio, which is partially at the root of the problem statement. Sedimentation of fine particles makes treatment by submersion rather complicated if one wants to retain these fines.
After all, only two applications methods were tested. Other, untested, methods could result in better CaCO$_3$ precipitation. For instance, the immersion method could be executed the other way around. In this way, the aggregates are firstly submersed in the biodeposition medium and afterwards in the growth media. This way the probability of precipitation in the pore structure might increase. Another example is submersing the aggregates firstly in the biodeposition medium and afterwards spraying the aggregate surface with the bacteria. As such the biodeposition media is located in the pore structure and on the aggregate surface. This might be more effective than the case where it was tried to absorb the bacteria in the aggregate pores. Various other methods which combine submerging and spraying or which alter the sequence or duration of application could all have their influences on the effectiveness of the CaCO$_3$ precipitation.

Generally, it can be concluded that further, extensive research in terms of application of the biodeposition treatment is required to obtain the best method and the highest increase in aggregate quality.

1.1.2 Field of application

As results suggested, the biodeposition treatment seems to affect mostly the larger pore structures. This might raise some questions about the possible fields of application. In the case of large porous materials, as the MA used in this research, the treatment does decrease the porosity of the material significantly. These material contain a decent amount of large pores, hence the effectiveness. But for relatively low porous material, as the RCA used in this research, no significant in decrease could be noticed.

A possible explanation for this observations are the size of the bacteria. These might be too big to penetrate the smaller pores. Possibly other bacteria have better effects but as mentioned B. sphaericus was used without questioning. As such one must consider that the treatment mostly affects materials with a large pore structure and the field of application is limited.

Another possibility is that the treatment does reduce the porosity of the aggregates but that the precipitation itself also contains a fine pore structure. As such the problem of water absorption is reallocated from the aggregate to the calcium carbonate precipitation.

It has to be pointed out that this statement is partly based on results from the MIP tests. In contradiction to other tests, these were not executed on an untreated sample and then on the same treated sample. This is quite impossible because the samples are contaminated with mercury after a MIP test. This makes a well-founded comparison and conclusion slightly difficult. Results from the water absorption test do however substantiate this statement.

Based on the executed research we can no more than speculate. The results are al pointing in the same direction, but further research will have to prove if the treatment does indeed affect mostly the large pores and if so, what is the cause for this. Afterwards, one can see how this might be tackled.

1.2 Aggregate properties

The selection and effectiveness of the biodeposition medium was partially based on weight increase and water absorption tests. The aggregate used to verify these characteristics were washed. As such, no solid statement can be made on the topic of quality increase since the fine particles were not

Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
included, while these contribute to a large portion of the water absorption. Nevertheless, relative comparison and conclusion for the specific range of aggregate sizes can be made. It is assumed that the observations and conclusion made for these fractions, can be drawn on to the finer fractions.

1.2.1 Water absorption in time

The water absorption was measured in different ways and on different samples. Not all these tests seemed to be successful and despite the fact the same aggregate types were used, notable differences were present in the test results.

As stated in “Chapter 4: Research & results” the water absorption curves in time for the pycnometer method showed higher values than the curves from hydrostatic measurements. This deviation is most probably caused by the gentle shaking of the pycnometer during the RAWA method. This adds energy, which releases entrained air from the aggregate pores. The higher the porosity of the aggregates, the more interrupted air that is removed this way and the higher the difference between the two methods will be.

Furthermore, the curves for water absorption in the mixer indicated even higher values. This can have some possible causes. Firstly, during the mixing process, the water absorption is also influenced by the dynamic effects of the mixing. In such a way, entrained air can be released from the pores. Secondly, due to the high impact on the aggregates during the mixing process, fines are created. These fines increase the water absorption, but also complicate the measurements. To determine the water absorption, the aggregates are made surface dry with drying-cloths. The finer particles stick to these cloths, which results in a loss of mass and a measurement error. Furthermore, their saturation state should be assessed with a metal cone (as for sand - cfr. chapter 3, section 1.2.2). This was not done because this would mean that the fine and coarse materials would have to be separated someway. However, there seemed to be no good way to carry this out without affecting the results.

Based on these observations and the remarks above, it was decided to only use the hydrostatic measurements during the research, to obtain well-founded comparisons and conclusions. Note that no conclusions can be made on absorption behaviour of aggregates during concrete production based on these curves. It is expected that the absorption behaviour will be different because of the competition among the different components (sand, cement, coarse aggregates, …), the creation of fines and the dynamic effects. Besides, during hydrostatic measurements aggregates are fully submerged, which is not entirely the case during mixing.

Water absorption curves were made for the first 2 hours. This was done because it was expected that water absorption from the aggregates in fresh concrete only occurs in the first few moments. As soon as concrete starts to harden and crystals start to form, it was expected that the absorption would cease. As such, it can be assumed that only a fraction of the total water absorption of the aggregates will be extracted from the mixing water. We can only speculate about the exact amount, because no test were executed to check this and, as mentioned, the absorption behaviour in concrete is unknown.

To exclude the effect of this unknown absorption behaviour on the concrete properties, it was chosen to use pre-wetted aggregates. As such, unwanted absorption from effective mixing water could be avoided. But, in practice pre-wetting by submersion is not used and the absorption curves show no significant decrease within the first two hours, despite the biodeposition treatment. This could be of

Improving the quality of recycled aggregates by biodeposition of CaCO₃
great importance when one tries to obtain a qualitatively better aggregate for concrete production. However, no solid conclusion can be made based on these curves. We therefore propose that a closer look should be taken to the absorption behavior of aggregates in concrete mixtures.

As discussed in “Chapter 4 - Part II: Aggregate properties”, the treatment affected the course of the absorption curve, especially for MA. This can substantiate the statement that the treatment mainly affected the larger pores. The untreated MA had a rapid increase in water absorption in the first few moments. This could be caused by filling of the large pores. The absorption rate of the bio-treated MA was slower. Possibly because the larger pores were partially blocked with CaCO$_3$ precipitation and only smaller pores remained. These take more time to absorb water, hence the slower absorption rate.

### 1.2.2 Aggregate resilience

Results from the ultrasonic pulse tests and Los-Angeles test suggested an increase in aggregate resilience due to the biodeposition treatment. The weight losses after treatment were about the same or smaller compared to the reference for the ultrasonic pulse test. The LA-values were lower after treatment. The results from the micro-Deval test did not quite confirm this statement.

A possible explanation for the increased LA-value is that cracks and large pores were filled with CaCO$_3$. Normally these are the locations where crack initiation occurs. Taking this away makes aggregates less vulnerable for abrasion. This might also explain why the effect is relatively significant for the MA, but small for the RCA. The fact that an increase in resistance was not really noticed for the micro-Deval tests, substantiates this statement. The micro-Deval tests wears the aggregates. The presence of cracks does not really affect the resistance to wear.

The ultrasonic pulse results were also in line with this statement. There aggregates were sonicated with high frequencies ultrasonic waves. The fact that smaller weight losses were observed for treated aggregates compared to the reference, indicated that in some way the aggregates gained some strength. The most plausible explanation for this is that the treatment caused some sort of firm outer layer, which protected the aggregate from disintegration. Another possible explanation is that some of the weaker parts are fixated by the treatment.

The small changes in MDE and LA-values can also be explained. During these test the aggregates were intensively hammered upon. The small amount of CaCO$_3$ cannot make a notable difference with such high impact forces. Therefore its more than logical that the differences were rather small. Originally it was even expected that the resistance to wear and fragmentation would decrease, such as is the case for recycled aggregates with mortar attached to it (cfr. Literature review).

### 1.3 Concrete properties

#### 1.3.1 Consistency

As could be noticed in “Chapter 4 – Part III: Concrete properties”, there were large differences in the consistency results between the slump and the flow test. The slump test indicated a much lower consistency for the concrete with treated aggregates compared to ones with untreated ones. On the other hand, the flow test suggests that the consistency was approximately in the same range for the different mixtures.
The difference between these two test can be explained by the method of measuring. The two test rely on different principles (cfr. Literature review). Furthermore, the slump test is more sensitive to the operation of the executor. In addition, the slump apparatus used made it hard to lift the cone in a fluent motion.

Based on the results not much could be concluded. The results from the slump and the flow test contradicted each other, and not always the same amount of superplasticizer was used. The only relative comparison that could be made was for the MAC. There a decrease in consistency could be observed for the same amount of superplasticizer. This could be caused by a decrease of water content or because of a rougher aggregate due to the treatment. The latter was sensed during the research, but was not tested.

1.3.2 Hardened concrete properties

The hardened concrete properties are discussed here. A possible cause for the measured changes between concrete mixtures with treated and the ones with untreated aggregates is evaluated.

A first possible explanation is that the reduced porosity of the aggregates affected the properties. In case of water absorption, this can surely be the case. The biodeposition treatment resulted in a reduction of water absorption and porosity of the aggregates. This reduction will logically result in a decrease of water absorption for the respective concrete. The increase in density is also in the same line; less pores results in higher densities. Also the modulus of elasticity, porosity and compressive results substantiate this statement. However, for these last three properties the difference before and after treatment was so big that we question if this was completely caused by the decrease in porosity of the aggregate.

Based on these observations a second possible explanation is proposed. Despite the fact effort was done to remain the effective W/C ratio constant, it could be possible that it did change. This could originate from multiple sources.

The first possibility is human error. During the preparation of the aggregates a wrong amount of water could have been added. As such, the water content and the properties changed. Another thing that needs to be pointed out is the amount of water to be added was determined on a sample of the aggregates which were supposed to be used for mixing. This sample could have been unrepresentative and caused a wrong amount of compensation water to be added to the mixing water.

A second and third possibility could still originate from the effect of the biodeposition treatment. Firstly, the adsorption behaviour of the aggregates could have changed due to changes in the surface structure of the aggregate. In specific, the treated aggregates could adsorb more water. As a consequence less water is present in the mortar, decreasing the effective W/C ratio of the concrete. Secondly, the absorption behaviour of the aggregates could have changed. Although aggregates were pre-wetted they were not fully saturated yet. Possibly they still extracted water from the mortar fraction, decreasing the effective W/C ratio.

A fourth possibility could originate from the pre-wetting of the aggregates. As stated in the literature review, fully saturated aggregates can cause an increase in effective W/C ratio due to movement of water from the aggregate toward the mortar. As more water is present in untreated aggregates compared to the respective treated ones, more water could have been added to the mortar fraction.

Improving the quality of recycled aggregates by biodeposition of CaCO₃
More and more we suspect a change in W/C ratio caused the variation in properties. The change in intrinsic properties of the aggregates seems to be unable to initiate such difference in concrete properties. At best, they should ease the concrete design by lowering the water demand. But no other great influences were expected. Further research will have to indicate the cause of these changes in concrete properties.

2. Economic viability

Recycled aggregates originate from construction and demolition waste. These materials have varying properties and compositions. For concrete application, thorough selection and processing measures need to be made to ensure quality. In addition, the materials need to be transported from the demolition site to processing sites and then to the construction site. This makes the production of these aggregates more expensive than comparable natural aggregates.

Furthermore, more actions need to be made to ensure durable and qualitative concrete (e.g. more superplasticizer, more water,...). In this master dissertation the applied action was the use of a biodeposition treatment to increase the quality of the aggregate and as such the concrete.

Although the treatment looks promising, the economical downside must be considered. Earlier studies showed the high costs involved and stated this as one of the main disadvantages of biodeposition treatments. this disadvantage was encountered during this research, despite no economic study was executed. The research was partially limited due to the fact that an immense amount of growth medium and biodeposition medium would have been needed.

A biodeposition treatment by submersion was considered as the best application method. This will increase the cost in two ways. Firstly, more medium is needed to treat aggregates by submersion compared to spraying. Secondly, it will require infrastructural changes in current concrete production plants. More about this in the next section.

An economic study will have to point out if the use of bacteria outweighs the savings it can create. Furthermore, we are confident that improvement in the treatment procedure can reduce the costs.

3. Industrial feasibility and practical implication

The concrete industry in Belgium is quite conservative. Not much companies are willing to use recycled aggregates and lesser companies would like to make large adjustments to their infrastructure for them. Therefore, we want to stipulate a few remarks that could make industrial application of biodeposition treatment less attractive.

It was observed that calcium carbonate precipitation for the treatment by submersion mainly happened at the upward side of the aggregates situated at the top of the immersion bath. Therefore aggregates were well spread and turned over occasionally, when the treatment was applied during the research. If this method would be used in a full scale concrete production plant, this will cause the need for large submersion baths and intensive labour to turn over the aggregates. This makes application in the concrete industry less feasible.

If spraying would be used a more feasible situation occurs. The production of recycled aggregates already includes washing of aggregates in a washing drum. A spraying installation for biodeposition

Improving the quality of recycled aggregates by biodeposition of CaCO₃
treatment could easily be mounted to this drum. Spraying installations could also be added on top of conveyor belts, often used in aggregate and concrete production plants.

The production of growth and biodeposition medium takes some time. In this research the microbiology lab of LabMET (lab for microbial ecology and technology, Ghent University) was used. This lab was no match for the scale of the original test setup. As the name suggest, bacteria at LabMET are produced on a small scale. For a minimal amount of concrete tests (consistency, air content, density and compressive strength) about 60 l growth medium was needed and the same amount of biodeposition medium. The available facilities were limited to a maximum production of 8 l in three days (at best). A lot of the originally planned research needed to be discarded due to this. The production of bacteria and biodeposition medium just was too time consuming. This has to be taken into consideration when the technology is on point and ready for industrial applications.

Cost related and practical issues might make the use of biodeposition treatments in concrete technology less attractive. When the technology is on point a practical study should be made to implement biodeposition for concrete application on an industrial scale.
Chapter 6  General conclusion and perspectives

During this master dissertation, the use of bacteria for biodeposition of calcium carbonate was assessed for the improvement of recycled aggregates in concrete applications. The main problem of recycled aggregates is their high water absorption, due to attached mortar and the presence of materials with high porosities. As such, the improvement of the aggregates and hence the effectiveness of the treatment, was assessed upon the decrease in water absorption.

In the first stage, the biodeposition medium and bacteria were analysed. Earlier studies on the subject indicated that *Bacillus sphaericus* resulted in the most effective calcium carbonate precipitation. Therefore, this bacteria was also used in this research. Firstly, the composition of the deposition medium was optimized. Results showed that the speed and amount of urea decomposition was mainly influenced by the concentration of the bacteria and the amount of calcium in the medium. Earlier research already stated that a high precipitation speed is not beneficial for the effectiveness of the treatment. This was also observed in this study. From the decomposition curves, it was chosen to work with a growth medium of $10^8$ cells/ml *B. sphaericus* and a 0.5 M Ca-nitrate/0.5 M urea biodeposition medium. This composition resulted in a steady decomposition rate and a decent amount of decomposed urea in an acceptable time range. Secondly, a proper treatment procedure was explored. Treatment by immersion showed the largest increase in weight and largest decrease in water absorption of the aggregates. Altered compositions of the media were also evaluated. These media had more bacteria or a higher yeast extract concentration. They showed more weight increase and bigger CaCO$_3$ crystal diameters, but no significant decrease in water absorption. SEM-images indicated that this was caused by a non-uniform deposition of the calcium carbonate. As such, it can be concluded that an increased decomposition and precipitation rate does not necessarily results in a better treatment. Furthermore, the cohesion strength of the precipitation seems to decrease for the treatments with increased precipitation rates. Therefore, also multiple treatments were assessed, which was promising, as a further reduction in water absorption could be noted for the high porous recycled aggregates. However, the effectiveness depended on the type of recycled aggregates, as the low porous recycled aggregates did not show a significant extra decrease in water absorption.

In the second stage, the effect of the optimized treatment on aggregates was thoroughly analysed. The biodeposition treatment led to an increase in weight, and ideally a drop in water absorption. The decrease in water absorption was adopted as a measure for quality improvement of the recycled aggregate. The results were very promising. For both types of recycled aggregates evaluated, a decrease in water absorption could be observed. But, the reduction was not identical for both types. The course of the water absorption for both bio-treated aggregates was approximately the same. First a relatively rapid absorption of water takes place. Thereafter, a gradually transition to a stable and slow absorption of water takes place. The water absorption curves showed a small difference in water absorption between treated and untreated aggregates in short (2h) and long term (72h). Also, the resistance to abrasion and fragmentation seems to be almost unaffected by the treatment. This weakens the statement about the promising quality improvement. Then again, tests on the porosity of the materials showed a positive effect of the treatment. A significant decrease in the porosity can be noticed. Both the water absorption curves, as the porosity measurements suggest that the treatment mainly affected large pores (> 1 µm). The fact that the treatment appears to be more effective for the high porous aggregate supports this statement.
In terms of aggregates, it can be concluded that the treatment lead to a promising improvement for concrete applications. However, it was observed that the effectiveness largely depended on the type and porosity of the targeted aggregates. The margin of improvement is still relatively small. Further research will have to focus on additional development of the treatment and possible types of aggregates that can be addressed by the treatment.

In the last stage, the treated aggregates were used in concrete mixtures. Relative comparisons were made between the mixtures with treated and the ones with untreated aggregates. The results showed positive developments. There were however some indications that a change in effective water-cement ratio took place. The cause of this change could not be found. It is plausible that these were caused by the treatment. Likewise this could also originate from various other parameters which influence concrete properties. Further research will have to stipulate the actual cause. As long as this is not clarified, it can only be concluded that the change in intrinsic properties of the aggregates showed promising results for concrete applications. The effects on the concrete properties could be observed to some degree, but it was unclear to what extent this truly was caused by the biodeposition treatment.

As is clear from these research stages, the biodeposition treatment showed promising effects for the improvement of the quality of recycled aggregates designated for concrete mixtures. The undesirable high water absorption could be altered, which will have it consequences in the concrete design and production. However, further research will have to be executed to increase the effectiveness of the treatment and to obtain properties comparable to natural aggregates.

No previous research did a thorough analysis of the absorption behaviour. The absorption behaviour of aggregates in a concrete mixture is practically unknown. It have been stated that aggregates will probably never be able to reach their fully saturated state. Adsorption and absorption of other materials present in the mixture will prohibit this. The exact quantity of absorbed water and the absorption behaviour is uncharted. This could however be a more than excellent and thought-provoking topic. This would address the problem at the root, instead of searching for a solution. It would also shed light on the exact quantity of the water absorption problem. This would be quite an extensive topic. The absorption behaviour will not only be different for each type of material, but also the saturation state of the aggregates will have an influence. Also the other materials present in the concrete mixture will possibly influence the behaviour. Due to the extent of this topic, the lack of literature and the focus of this master dissertation, this could not be included in the research.

As long as the absorption behaviour of high porous materials in concrete is not fully examined, it will always be difficult to come up with a solution. Particularly when these do not cover the full scale of the problem, which is the case in the current state of this research. If one wants to clear up this problem an exact quantification of the problem or a full coverage needs to be determined.

To provide a full coverage with a biodeposition treatment, more research will have to be executed. As frequently mentioned throughout the course of this dissertation, various topics could be further optimized.

A first topic is the biodeposition medium itself. This research was based on previous studies, which focused on the application of self-healing concrete. Therefore, it could be interesting to start back from the basics and analyse the effect of a wide range of biodeposition medium compositions. It is plausible
that compositions or application methods that did not quite proved their effectiveness in other applications, are more effective for aggregate improvement. At a specific point during this master dissertation, it was however necessary to go to the next stage in the research and the most effective treatment at that point was chosen. In our opinion, a more extensive optimization analysis can greatly increase the effect of the treatment.

Only when the optimization of the treatment procedure is at point, one can continue with the concrete applications. According to our knowledge, an efficient treatment will cause a decrease of the absolute water absorption to values comparable to natural aggregates. From there on, the actual problem disappears. Then, the positive (or possibly negative) effects of the treatment on concrete properties could be studied.

In the current state of the research, it was quite unclear in which way, or if the treatment influenced the concrete properties. It could be of interest to analyse the origin of the properties changes. This could be done by proper analyse of the mortar skeleton and the interfacial transition zone of the aggregates. Also various concrete mixture test set ups could be researched. In this master dissertation only pre-wetted aggregates by submersion were used. One could however also apply other pre-wetting methods or no pre-wetting at all to investigate the effect on the absorption behaviour. Also the consistency over time can be measured to further delve into this topic. This were all part of the original research but was discarded due to the slow production rate of the growth media. Further research on the subject of aggregate properties can also shed light on the changes the treatment can cause. Examples are the roughness, the angularity and shape of aggregates, exact composition of the aggregates, ... .

If all aspects of the biodeposition treatment reached their full potential, the method will have to be fully evaluated. If it can improve the quality to an extent where recycled aggregates become comparable to natural aggregates, is it worth the effort? What will be the benefits? The cost and practical aspect has been mentioned throughout the discussion chapter. An in depth cost analysis and feasibility study should determine whether the use of a biodeposition treatment outweighs the possible disadvantages.

To finalize this master dissertation a global conclusion statement is made. The biodeposition treatment showed promising results for both aggregates and concrete applications. The effectiveness is however still relatively low. But, we are more than confident that further optimization and research will enable high grade quality improvements.
Chapter 7 References


Improving the quality of recycled aggregates by biodeposition of CaCO$_3$


Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of CaCO₃


Improving the quality of recycled aggregates by biodeposition of CaCO₃
References


Sika. (2013). *Sika Concrete Handbook*. Zürich: Sika Services AG.


Improving the quality of recycled aggregates by biodeposition of CaCO₃


Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Annex IV: Product info sheet of cement

Hoogovencement

**CEM III/A**

**Productionsschijving**
Hoogovencement CEM III/A is a lichtgewicht cement dat verkregen wordt door het gemalen van de hoofdcomponenten portlandcementliner en gegranulireerde hoogovencement. Door de juiste verhouding tussen de samenstellende bestanddelen wordt, in combinatie met een bepaalde maatschappij, een cement verkregen in de sterkekleur 32,5, 42,5 of 52,5. Alle cementen worden binnen hun sterkekleur gekenmerkt als een cement met een normale begrensting.

Hoogovencement CEM III/A voldoet aan de eisen zoals gesteld in de Europese gemeenschapsnorm EN 197-1, inclusief wijzigingsblad A1. Deze norm geeft eisen ten aanzien van de samenstelling op bestanddelen, chemische eisen, mechanische en fysische eisen.

**Samenstelling**
De eisen aan de samenstelling zijn uitgedrukt in procenten ten opzichte van de som van alle hoofd- en naverbanddelen. Dit totaal wordt nog vermenigvuldigd met het reëlevante calciumiiFeCaat om het bindingsgedrag te reguleren.

<table>
<thead>
<tr>
<th>Hoofdbestanddelen (in massa %)</th>
<th>Nenematerialen (in massa %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lichte zand (L)</td>
<td>Hoogovencement (H)</td>
</tr>
<tr>
<td>CEM III/A</td>
<td>35 - 45</td>
</tr>
</tbody>
</table>

**Mechanische en fysische eisen**
De sterkekleur van een cement bepaalt de minimale druksterkte gemeten na 28 dagen op normalprimaire. Binnen zijn normsterkekleur heeft dit cement een normale begrensting, aangeduid met N. Het begin van de binding is een maat voor het ophellen van een cementpasta. Aan de eis van vormhoudbareheid moet worden voldaan om aan te tonen dat een cementpasta niet gevonken is voor verhuizing.

<table>
<thead>
<tr>
<th>Sterkekleur</th>
<th>Drukkerkte in MPa</th>
<th>Begins van de binding (dagen)</th>
<th>Vormhoudbareheid (minuten)</th>
</tr>
</thead>
<tbody>
<tr>
<td>32,5 N</td>
<td>≥ 16,5, ≥ 32,5, ≥ 52,5</td>
<td>≥ 75</td>
<td>≥ 50, ≥ 45</td>
</tr>
<tr>
<td>42,5 N</td>
<td>≥ 10,0, ≥ 42,5, ≥ 62,5</td>
<td>≥ 50</td>
<td>5, 10</td>
</tr>
<tr>
<td>52,5 N</td>
<td>≥ 20,0, ≥ 42,5, ≥ 62,5</td>
<td>≥ 20</td>
<td>≥ 45</td>
</tr>
</tbody>
</table>

**Speciale eigenschappen**
Cement dat voldoet aan de in EN 197-1 gestelde eisen is voorzien van een CE-markering. Daarnaas kan cement geassurificeerd worden op aanvullende specifieke eigenschappen. Deze eigenschappen kunnen tot uiting in de naamgeving van het cement. De naamgeving is
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO₃

<table>
<thead>
<tr>
<th>PRODUCTINFORMATIE</th>
</tr>
</thead>
<tbody>
<tr>
<td>VORM</td>
</tr>
<tr>
<td>UITERLIJK / KLEUR</td>
</tr>
<tr>
<td>Vloeibaar, bruinachtig</td>
</tr>
<tr>
<td>VERPAKKING</td>
</tr>
<tr>
<td>Metalen vat van 200 kg</td>
</tr>
<tr>
<td>Container van 1000 kg</td>
</tr>
<tr>
<td>Tankladingen op verzoek</td>
</tr>
</tbody>
</table>

| OPSLAG           |
| OSLAGCONDITIES / HOUDBAARHEID |
| 12 maanden in een gesloten verpakking. |
| Beschermen tegen vocht, intens direct zonlicht en contaminatie. |
| Indien geleverd in bulk, moeten de tanks en reservoires vrij zijn van residuen van vorige leveringen. |
| Bij langdurige opslag moet het product terug opgeroerd worden. |

| TECHNISCHE GEGEVENS |
| CHEMISCHE BASIS |
| Water gebaseerde polymeeroplossing |
| DENSITEIT (20°C) |
| 1,08 kg/l |
| Ph WAARDE |
| Ongeveer 5 |
| ALKALI GEHALTE (NA₂O EQ.) |
| ≤ 1,0 % |
| VASTE STOF GEHALTE |
| 27% |

| SYSTEEMINFORMATIE |
| TOEPASSINGINSTRUCTIES |
| DOSERING |
| 0,2 tot 2,0 % van het cementgewicht |
| TOEPASSINGSMETHODE / GEREEDSCHAP |
| In de betoncentrale raden we aan om Sika® ViscoCrete®-1068 direct de watertoevoeging aan het beton toe te voegen en dit vóór toevoeging van andere toelagstoffen. |
| Om optimaal het vloeibaar makend potentieel te benutten, adviseren wij bij een optimale menging een nat-mengtijd van ten minste 45 seconden. |
| Begin ten vroegste met de fijne dosering van het water na 2/3 van de nat-mengtijd om een te vloeiende consistentie te voorkomen. |
| Bij een nadosering op de werf, raden we het gebruik van dezelfde superplastificerend aan als in de betoncentrale. |
| Indien Sika ViscoCrete®-1068 wordt toegevoegd aan een betonmixervrachtwagen, moet een gelijkmatige verdeling van de superplastificerend gegarandeerd worden. Daarom beveelen we aan het product op het beton te gieten. De mengtijd moet 1 min per m³ bedragen, maar minstens 5 minuten in totaal. |

| OPMERKINGEN BIJ VERWERKING / BEPERKINGEN |
| Voor gebruik is een test volgens EN 206-1 nodig. |
| Er is mogelijkheid van een belangrijke bindingsvertraging in functie van het gebruikte cementtype bij een maximale dosering. |
| Een bindingsvertraging van 21u 55min werd vastgesteld bij het uitvoeren van de compatibiliteitsproeven in combinatie met cement CEM II B 32,5 N LH SR LA bij een maximale dosering. |
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Annex VI: SEM images after the ultrasonic pulse test

To control the cohesion strength of CaCO₃ precipitation, SEM-images of the different treatment procedures were made, after the specimens were sonicated. An overview of these images are given in this annex. If these images were compared with those before the ultrasonic pulse test, no significant difference could be observed.

**TREATMENT PROCEDURE I (immersion; 1x)**

Figure 96: RCA/T-I with 200x and 1000x magnification

Figure 97: MA/T-I with 200x and 1000x magnification

Improving the quality of recycled aggregates by biodeposition of CaCO₃
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
Improving the quality of recycled aggregates by biodeposition of CaCO$_3$
TREATMENT PROCEDURE III

Figure 102: SEM image of RCA/T-III with 200x and 500x magnification

Figure 103: SEM image of RCA/T-III with 200x and 500x magnification

Improving the quality of recycled aggregates by biodeposition of CaCO₃