A Fundamental Study on Mechanical & Physical Properties of Polymer-modified Self-healing Mortars Using Bacteria

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Abstract

A polymer-modified self-healing mortar is a mixture of Portland cement, water, sand, polymer and self-healing material. The polyacrylic ester emulsion (PAE) is used as polymer and as self-healing material bacterial spores from genus Bacillus + calcium lactate is used. The reason why this type of bacteria is used as a component of self-healing material is because of the high alkaline environment inside the mortar. A mortar has a pH value of between 9 and 11. As far as known, only this kind of bacteria can be used as a self-healing agent for mortars. Why a polymer-modified self-healing mortar? This polymer-modified self-healing mortar has an autonomous self-healing mechanism with, a high workability, a high flexural strength and also a high adhesion.

The experiments that has been performed in this study show that a polymer-modified self-healing mortar is a strong material with and active self-healing. This bacterial self-healing mechanism can heal cracks up to 0.46mm in contrast to the autogenous self-healing (of conventional mortar) of cracks up to 0.18mm. Beside this the fresh mortar showed a high workability. These properties make the polymer-modified self-healing mortar a strong and a more durable mortar. This concept can also be used to produce polymer-modified self-healing concrete.
## Contents

1 Introduction 

2 Theory 
   2.1 What is Cement? 
   2.2 What is Mortar? 
   2.3 Polymer-modified Mortar 
      2.3.1 Principles of Polymer Modification 
      2.3.2 Mechanism of Polymer-cement co-matrix Formation 
      2.3.3 Advantages of Polymer-modification 
   2.4 Properties of Fresh Polymer-modified Mortar 
      2.4.1 Workability 
      2.4.2 Bleeding and Segregation 
      2.4.3 Setting Behavior 
   2.5 Properties of Hardened Polymer-modified mortar 
      2.5.1 Adhesion 
   2.6 Self-healing for Cementitious Materials Using Bacteria 
   2.7 Polymer-modified self-healing mortar 
   2.8 What is Concrete? 
   2.9 Summary 
   2.10 Hypothesis (Expectations) 

3 Experiments 
   3.1 Materials 
   3.2 Specimens 
   3.3 Test Methods & Results 
      3.3.1 Flow Test 
      3.3.2 Three Point Flexural Test 
      3.3.3 Compression Test 
      3.3.4 Pull-out Adhesion Test 
      3.3.5 Water-permeability test 

4 Discussion 
   4.1 Workability 
   4.2 Flexural Strength 
   4.3 Compression Strength 
   4.4 Adhesion 
   4.5 Crack Healing 

5 Conclusion
1 Introduction

Concrete is a mixture of cement, water, sand and aggregate. It is a very strong, relative cheap construction material and it is among few building materials produced directly on the job [13]. This is why concrete is a popular and universally used construction material [13]. At this moment there are a lot of concrete structures like bridges, roads, (highrise) buildings, tunnels etc. These concrete structures are reinforced with steel. The reason why concrete structures are reinforced is because concrete has a low tensile strength.

Another disadvantage of concrete is that it cracks. These cracks ensure that the concrete becomes water-permeable [12]. In wet environment there is a risk of ingress of aggressive substances which can corrode the reinforcement [12]. This can reduce the durability of the structure [12]. To prevent the corroding of the reinforcement it is necessary to repair the cracks. The cracks can be repaired with cement mortar. The problem with the existing mortars is that the durability and the adhesion of the mortars are not sufficient. This means that after repairing the cracks the mortar will not remain for a long time. Because of the poor adhesion the mortars can fall out of the cracks after a while. If this happens the cracks has to be repaired again. This is very expensive and also labor intensive.

Mortar with a high durability en adhesion can be possibly made using polymers and bacteria. This is called the polymer-modified mortar with bacteria. The polymers are used for a better workable paste with a high adhesion [9]. The polymers make the mortar also more durable. The bacteria is used for the durability, in the sense that it gives the mortar self-healing properties [12],[7]. With this self-healing property the mortar will last very long and because of the high adhesion the mortar will bind very well with concrete.

This concept can also be used to make self-healing polymer-modified concrete. With this type of concrete it is possible to build structures instead of with ordinary concrete. The advantage of self-healing polymer-modified concrete structures is that the service life of the structure can be enhanced and more important, this type of concrete structures does hardly need maintenance. A self-healing polymer-modified concrete structure in moist environment is ideal because of the self-healing property of the concrete.

The goal of this thesis is to understand the fundamental mechanical and physical properties of a self-healing polymer-modified mortar using bacteria. To understand this, it is necessary to perform experiments with these type of mortars. Through this thesis there will be a continuously comparison between a conventional mortar and polymer-modified self-healing mortar.

Before beginning with the experiment it is important to know things about the following subjects:
- What is cement?
- What is concrete?
- What is mortar?
- What is a polymer-modified mortar? What are the advantages of using polymers?
- PAE polymer.
- Self-healing material for cementitious materials.

These subjects are treated in the first section called Theory. The aim of this section is to provide theory about the just named subjects in order to understand the goal of this thesis. The goal of this thesis is once again to
understand the fundamental mechanical and physical properties of a self-healing polymer-modified mortar using bacteria.

After the theory section begins the experimental work, in this section the theory is tested. Everything that is told in the theory section will be tested, the experimental results will confirm (or not) what is told in the theory section. Then there will be a discussion about the results and finally comes the conclusion. At the end of the thesis the references can be found that is used for this thesis. Figure 4.1 shows the set-up of the thesis.

![Flow-chart of this thesis](image)

Figure 1.1: Flow-chart of this thesis.
2 Theory

To understand this thesis and before beginning with the experiment, it is necessary to know what exactly concrete is. How is concrete made? What are the compositions of concrete? What is cement? It is also important to know what kind of polymers and bacteria are used in a polymer-modified self-healing mortar. In this section it is explained what cement, concrete and mortars are and how they are made. After this the polymers and the self-healing material are treated.

2.1 What is Cement?

Cement is a finely pulverized, dry material. It is obtained by burning a mixture of lime and clay to form a clinker, then pulverizing the clinker into powder [8]. Cement is not a binder by itself but develops the binding property as a result of hydration. Water is responsible for the hydration of cement. When cement is mixed with water it forms a plastics paste which develops stiffness (sets) and then steadily increases in compressive strength (hardens) by chemical reaction with water [6]. The chemical reaction between water and cement causes the paste to harden, this reaction is called hydration [6]. Hydration gives off heat, known as the heat of hydration [6]. So cement is a material which binds together solid bodies (aggregate) by hardening from a plastic state [6]. A cement which increases in strength even when stored under water after setting is called hydraulic [6].

The most used hydraulic cement in the world is Portland cement [6]. There are other kinds of cement, but the word cement in common usage, refers to Portland cement [6].

A Portland cement grain contains a lot of minerals and the four main minerals present in a Portland cement grain are tricalcium silicate, dicalcium silicate, tricalcium aluminate and calcium aluminoferrite [4]. Table 2.1 shows the main components of a cement grain with their chemical formulae.

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical formula</th>
<th>Abbreviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tricalcium silicate (alite)</td>
<td>Ca_3SiO_5</td>
<td>C_3S</td>
</tr>
<tr>
<td>Dicalcium silicate (belite)</td>
<td>Ca_2SiO_4</td>
<td>C_2S</td>
</tr>
<tr>
<td>Tricalcium aluminate</td>
<td>Ca_3Al_2O_6</td>
<td>C_3A</td>
</tr>
<tr>
<td>Tetracalcium aluminoferrite (ferrite)</td>
<td>Ca_4Al_nF_3_2-nO_7</td>
<td>C_4AF</td>
</tr>
</tbody>
</table>

The composition of cement is varied depending on the application. A typical cement grain contains 50% - 70% alite, 15% - 30% belite, 5% - 10% C_3A, 5% - 15% C_4AF and 3% - 8% other additives or minerals (such as oxides of calcium and magnesium) [4]. Figure 2.1 shows a typical cement grain.
The hydration of the calcium silicate, aluminate, and aluminoferrite minerals causes the hardening or setting of cement [5]. The ratio of $C_3\text{S}$ to $C_2\text{S}$ helps to determine how fast the cement will set [5]. With higher $C_3\text{S}$ content cement will set faster. Higher amounts of ferrite lead to slower hydration [5]. The ferrite phase causes the gray color in cement [5].

The addition of water to dry cement results in a thin cement paste. In time, the cement paste sets and develops strength through a series of reactions. Hydration of cement is not linear throughout time [5]. At first the hydration proceeds very slowly, allowing the thin mixture to be properly placed before hardening [5].

Alite is the most important mineral in cement for strength development during the first month, while belite reacts much more slowly and contributes to the long-term strength of the cement [5]. Both the silicate phases react with water to form calcium hydroxide and a rigid calcium-silicate hydrate gel, C-S-H [5].

Aluminate and ferrite phases comprise less than 20% of cement [5]. Their reactions in cement are also very important and affect the hydration of the calcium silicate phases [5]. Figure 2.2 shows the hydration of cement.
Before mixing cement with water it contains alite, belite, C₃A, ferrite, and some additives. When the cement powder is mixed then there will be a hydration reaction. After the hydration of cement there will be other compounds then before the hydration. Table 5.2 shows the compounds before and after the hydration of cement.

<table>
<thead>
<tr>
<th></th>
<th>Before hydration</th>
<th>After hydration</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₃S (alite)</td>
<td></td>
<td>C-S-H</td>
</tr>
<tr>
<td>C₂S (belite)</td>
<td></td>
<td>CH</td>
</tr>
<tr>
<td>C₃A</td>
<td></td>
<td>Ettringite</td>
</tr>
<tr>
<td>C₄AF (ferrite)</td>
<td></td>
<td>Water</td>
</tr>
<tr>
<td>Additives</td>
<td></td>
<td>Porosity</td>
</tr>
<tr>
<td>Water</td>
<td></td>
<td>Unhydrated cement</td>
</tr>
</tbody>
</table>

2.2 What is Mortar?

Mortar is a mixture of cement, water and sand. It is a workable paste that is used to bind together construction blocks (bricks) or fill the gaps between them [11]. Mortar is also used to repair cracks of concrete structures. If concrete cracks, water can go inside the concrete in wet environments and make the reinforcement rust. To prevent this the concrete has to be repaired with mortar.

2.3 Polymer-modified Mortar

Polymer-modified mortar is prepared by mixing a polymer or monomer with a fresh cement mortar [9]. Polymers and monomers are available in dispersed,
powdery or liquid form [9]. Figure 2.3 shows polymers and monomers that are used as cement modifiers.

![Polymers and Monomers for Cement Modifiers](image)

Figure 2.3: Polymers and monomers for cement modifiers [9].

As shown in figure 2.3, there are several types are polymers that can be used to produce several types of mortars. Although polymers and monomers in any form used in mortars, it is important that both hydration of cement and polymer phase formation proceed well to yield a monolithic matrix phase with a network structure in which the hydrated cement phase and polymer phase interpenetrate [9].

### 2.3.1 Principles of Polymer Modification

Polymer modification of cement mortar is done by both cement hydration and polymer film formation processes in their binder phase. The cement hydration process generally precedes the polymer formation process [9]. After the cement hydration process and polymer formation process, a co-matrix is formed by both cement hydration and polymer film formation processes.

### 2.3.2 Mechanism of Polymer-cement co-matrix Formation

The co-matrix phase which consist of cement gel and polymer films is formed according to a three-step model shown in figure 2.4.
**Step one** [9]. When polymers are mixed with fresh cement mortar, the polymer particles are uniformly dispersed in the cement paste phase. In this polymer-cement paste, the cement gel is gradually formed by the cement hydration. The water phase is saturated with calcium hydroxide formed during the hydration. The polymer particles deposit partially on the surfaces of the cement-gel-unhydrated-cement particle mixtures. The calcium hydroxide in the water phase reacts with a silica surface of the aggregates to form a calcium silicate layer. The formation of calcium hydroxide and ettringite in the contact zone between the cement hydrates and aggregates ensure the bond between them. Figure 2.5 shows the formation of polymer film on cement hydrates.
Step two [9]. As the cement hydration proceeds further and the capillary water is reduced, the polymer particles flocculate to form a continuous close-packed layer of polymer particles on the surfaces of the cement-gel-unhydrated cement particle mixtures and simultaneously adhere to the mixtures and the silicate layer over the aggregate surfaces. In this case, the larger pores in the mixtures are found to be filled by the adhesive and autohesive polymer particles. The size of the pores in the cement paste ranges from a few hundred picometers to several hundred nanometers. The polymer particles range from 50 to 500 nanometers.

Step three (Hardened structure) [9]. With water withdrawal by cement hydration, the close-packed polymer particles on the cement hydrates merge into continuous films. The films bind the cement hydrates together to form a monolithic network in which the polymer phase interpenetrates throughout the cement hydrate phase. Such a structure acts as a matrix phase for polymer-modified mortar, and the aggregates are bound by the matrix phase to the hardened mortar.

2.3.3 Advantages of Polymer-modification

It is considered that hardened cement paste mainly has an agglomerated structure of calcium silicate hydrates and calcium hydroxide bound together by the van der Waals forces [9]. Therefore microcracks occur easily in the hardened cement paste under stress. This leads to poor tensile strength and fracture toughness of ordinary cement mortar [9].
In the polymer-modified mortar the microcracks are bridged by the polymer films which prevent crack propagation, and simultaneously, a strong cement hydrate-aggregate bond is developed [9]. This is shown in the scanning micrographs of cross-sections of SBR-and PAE-modified mortars, in figure 2.6.

This effect increases with an increase in the polymer-cement ratio [9]. This leads to higher tensile strength, fracture toughness and adhesion.

The polymer films also provide a considerable increase in waterproofness, resistance to chloride ion penetration, moisture transmission, carbonation and oxygen diffusion, chemical resistance and freeze-thaw durability [9]. Such an effect is promoted with increasing polymer-cement ratio.

### 2.4 Properties of Fresh Polymer-modified Mortar

Polymer-modified mortar is made by using polymers or monomers and has a network structure which consists of cement gels and microfilms of polymers. That is why the properties of polymer-modified mortar are improved over conventional cement mortar [9].

#### 2.4.1 Workability

Generally, polymer-modified mortar provide a good workability over conventional cement mortar [9]. This is because of improved fluidity due to ball bearing action of polymer particles among cement particles and entrained air [9]. This means that the polymer-modified mortar has also an improved water retention over ordinary cement mortar. The water retention is dependent on the polymer-cement ratio.

#### 2.4.2 Bleeding and Segregation

The resistance of polymer-modified mortar to bleeding and segregation is very good in spite of their larger flowability. This is due to the hydrophilic colloidal properties of the polymers, air entraining and water-reducing effects of the substances of the polymers [9].
2.4.3 Setting Behavior

The setting of polymer-modified mortar is delayed to some extent in comparison with conventional cement mortar [9].

2.5 Properties of Hardened Polymer-modified mortar

In general, polymer-modified mortar show an increase in the tensile and flexural strengths [9]. This is because of the polymer films and an overall improvement in cement-aggregate bond [9]. But there is no improvement in the compressive strength as compared to ordinary cement mortar and concrete.

2.5.1 Adhesion

A very useful aspect of polymer-modified mortars is their improved adhesion to different substrates compared to ordinary mortar [9]. The development of adhesion is because of the high adhesion of polymers. The adhesion of most polymer-modified mortars increases with rising polymer-cement ratio [9].

2.6 Self-healing for Cementitious Materials Using Bacteria

In 2006 researchers at Technical University Delft started a project aiming to use bacteria as a component of self-healing agent incorporated in mortar or concrete where it could act as an autonomous repair system. An autonomous repair system means a repair system that is activated when needed [7], [12]. When there is a crack inside the concrete or hardened mortar, and water goes inside the concrete (or hardened mortar), then the system will activate itself to repair the crack. An autonomous repair system is actually an active crack healing mechanism done by the hardened mortar or concrete itself.

It is also important to know that there is also an autogenous repair mechanism. The autogenous repair system is a passive repair mechanism [1]. The power behind this autogenous repair system is the partially non-hydrated cement particles. If there is a crack inside the concrete/mortar, water gets inside the concrete/mortar in moist environment. These non-hydrated cement particles will react with water and partially seal the crack. Almost every conventional concrete/mortar has this autogenous self-healing system. In a lot of cases, this is of course not enough. To increase this self-healing potential, an autonomous self-healing system is needed. This is the reason why researchers at Technical University Delft started with the project to make an autonomous repair system using bacteria.

The bacteria that is used as a self-healing agent must be capable of surviving in an extreme alkaline environment. Cement and water have a pH value of up to 13 when mixed together. This is a hostile environment for life. Most organisms die in an environment with a pH value of 10 or higher. In a series of studies it was found that strains of the bacteria genus Bacillus can thrive in high-alkali environment [2]. There was also found that only a specific group of the bacteria genus Bacillus can survive inside the mortar or concrete. Those are the ones which can produce spores comparable to plant seeds[11]. These spores have extremely thick cell walls that enable them to remain intact for up 200 years.
while waiting for a better environment to germinate [2]. These spores are not incorporated inside the mortar or concrete alone. The spores are added in the mortar or concrete along with calcium lactate [2]. It is also interesting to know that this bacteria works optimal in temperatures of 20-25 degree celsius and in a pH environment of 9-11. A hardened concrete or mortar has a pH value of 9-11. If the bacteria finds itself in this perfect environment with enough food, then it will grow exponentially and heal the micro cracks.

The principle mechanism of bacterial crack healing is that the bacteria act as a catalyst and transform the calcium lactate to limestone [2]. So when a concrete structure is damaged and water starts to seep through the cracks that appear in the concrete, the spores of the bacteria germinate in contact with the water and calcium lactate [2]. After being activated, the bacteria start to feed on the calcium lactate [2]. As the bacteria feeds, oxygen is consumed and the soluble calcium lactate is converted to insoluble limestone [2]. The limestone hardens on the cracked surface and thereby sealing it up. Figure 2.7 shows the bacteria spores. Figure 2.8 shows the autonomous self-healing mechanism.

![Figure 2.7: Electron micrograph of bacterial spores [2].](image)

![Figure 2.8: Self-healing mechanism [2].](image)

2.7 Polymer-modified self-healing mortar

A polymer-modified mortar is a mortar with polymers inside the mortar mixture. If the self-healing material is mixed with the polymer-modified mortar, then there will be a polymer-modified self-healing mortar with autonomous self-healing properties.
2.8 What is Concrete?

Concrete is a mixture of aggregate, sand, cement and water. A chemical reaction (hydration) between cement and water causes concrete to harden to a stonelike condition. Because hydration hardens concrete, freshly placed concrete submerged underwater will harden [13]. Concrete is at first a plastic paste that can be cast or mold into almost any size or shape.

Aggregate is a granular material, such as sand, gravel, crushed stone or construction and demolition waste. Aggregate make up between 60% to 80% of the volume of normal concrete [13]. All aggregate is screened to ensure good size gradation because concrete differs from other cement-water-aggregate mixture in the size of its aggregate. When cement is mixed with water and aggregate with a diameter of less than 4.00 mm is called a mortar. The mixture is called concrete when cement is mixed with aggregate of more than 6.4 mm, plus fine aggregate and water [13]. Aggregate size and shape influence the amount of water required. Aggregate surface texture influences the bond between the aggregate and the cement paste [13]. In properly mixed concrete, the paste completely surrounds each aggregate particle and fills all spaces between the particles.

The four most important properties of concrete are workability, durability, high compressive strength and ability to protect the reinforcement against rusting. The reason why concrete is reinforced is that concrete has a low tensile strength. Besides the properties just named, concrete is fireproof, watertight, relatively economical and easy to make. Concrete has also disadvantages and the most important disadvantage of concrete is that it cracks.

2.9 Summary

Here follows a short summary of the theory that is treated in this section.

Cement is a fine pulverized material that is used to produce mortar or concrete. Mortar is produced by making a mixture of cement, sand with different particles and water in certain proportions. By adding polymers inside the fresh mortar, the fresh mortar will get a better workability and a higher adhesion. The polymer-modified mortar has also better mechanical properties like a higher tensile strength. The polymer-modified mortar has a higher tensile strength because of polymer films inside the co-matrix of the mortar. The compressive strength of the polymer-modified mortar is not improved over the ordinary cement mortar.

When the self-healing material, bacteria of genus Bacillus plus calcium lactate, is added, the polymer-modified mortar will get self-healing properties. This self-healing polymer-modified hardened mortar will repair the cracks that will occur inside the mortar, by itself.

2.10 Hypothesis (Expectations)

The aim of this thesis is to develop a mortar with an autonomous self-healing mechanism. Beside this the mortar should have a high workability, high flexural strength and a high adhesion. A mortar with these kind of properties is called a polymer-modified self-healing mortar. The just named properties will be tested in the experimental section. The aim of this thesis is also to understand the
It is important to formulate a hypothesis when doing an experiment. It is a theorem that has to be proven with the results of the experiment. For this experiment the hypotheses are formulated below.

1. The fresh polymer-modified self-healing mortars will have a better flow, the workability will be better in comparison with the control mortar. This is expected because of the ball bearing effect of the polymers. The workability will be measured with the flow test.
2. The flexural strength of the polymer-modified self-healing mortar will be higher than the flexural strength of the control mortar. This is expected because of the co-matrix of the polymer-modified mortar. The co-matrix consists of cement gel and polymer films. Because of this co-matrix there is a better bond between the cement hydrates and the aggregates. The three point flexural strength test will be performed for this hypothesis.
3. The compressive strength of the polymer-modified mortar will be more or less the same as the compressive strength of the control mortar. Compressive test will be performed.
4. The adhesion of the polymer-modified self-healing mortar to a concrete substrate will be very good because of the polymer that is used as cement modifier. The polymer-modified mortar will also have a higher tensile strength over the control mortar. The pull-out adhesion test will be performed to measure the bond and the tensile strength of each mortar. 5. The crack-healing of the self-healing mortar will be more than the crack-healing of the control mortar. The bacteria inside the self-healing mortar makes sure that the crack is healed as good as possible. The crack-healing inside the control mortar occurs because of the non-hydrated cement particles. The control mortar has no bacteria inside the mortar that can act as a part of an autonomous repair system. That is why it is expected that the crack-healing with bacteria will be better than the crack-healing without bacteria. To measure the healing, the water-permeability test will be performed.
3 Experiments

This section explains the experimental work that is performed in order to test the hypotheses and better understand the fundamental mechanical and physical properties of the polymer-modified self-healing mortar.

The first subsection explains what kind of materials are used. After this, it is explained what kind of specimens are tested. After that the test methods are treated and finally the results of the tests are shown.

3.1 Materials

Materials used for the experiment are ordinary Portland cement, sand with different sizes, polyacrylic ester emulsion (PAE), self-healing material (SHM = bacterial spores+calcium lactate) and water. These materials are treated in the previous section.

3.2 Specimens

Mortar test specimens were prepared with ordinary Portland cement (CEMI 42.5N), sand with different diameters, polyacrylic ester (PAE), self-healing material (SHM) and water. There were four types of specimens made; control mortar, SHM mortar, PAE-5+SHM and PAE-10+SHM mortars. The control mortar is a normal mortar, it does not have the SHM or PAE. The SHM mortar has only the SHM. PAE-5+SHM and PAE-10+SHM mortars have both the SHM and polyacrylic ester. Of each type, there are two series of specimens made. The first series will harden 14 days and the second series will harden 28 days. Table 3.1 shows the applied mixture proportions.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Control</th>
<th>SHM</th>
<th>PAE-5+SHM</th>
<th>PAE-10+SHM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand (mm)</td>
<td>10882</td>
<td>10882</td>
<td>10882</td>
<td>10882</td>
</tr>
<tr>
<td>0.125 - 0.25</td>
<td>1093</td>
<td>1093</td>
<td>1093</td>
<td>1093</td>
</tr>
<tr>
<td>0.25 - 0.50</td>
<td>2021</td>
<td>2021</td>
<td>2021</td>
<td>2021</td>
</tr>
<tr>
<td>0.50 - 1.00</td>
<td>2327</td>
<td>2327</td>
<td>2327</td>
<td>2327</td>
</tr>
<tr>
<td>1.00 - 2.00</td>
<td>2327</td>
<td>2327</td>
<td>2327</td>
<td>2327</td>
</tr>
<tr>
<td>2.00 - 4.00</td>
<td>3114</td>
<td>3114</td>
<td>3114</td>
<td>3114</td>
</tr>
<tr>
<td>SHM (30 g/dm³)</td>
<td>-</td>
<td>192</td>
<td>190</td>
<td>192</td>
</tr>
<tr>
<td>Cement</td>
<td>2776</td>
<td>2776</td>
<td>2776</td>
<td>2776</td>
</tr>
<tr>
<td>Water</td>
<td>1527</td>
<td>1527</td>
<td>1170</td>
<td>814</td>
</tr>
<tr>
<td>Polymer PAE-5%</td>
<td>-</td>
<td>-</td>
<td>496</td>
<td>-</td>
</tr>
<tr>
<td>Polymer PAE-10%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>992</td>
</tr>
</tbody>
</table>

Figure 3.1 shows a picture of the specimens that is prepared for this experiment.
3.3 Test Methodes & Results

The test methodes that were applied on the specimens are the slump test, compressive and flexural test (strength test), adhesion test and water permeability test.

3.3.1 Flow Test

Before putting the mortars into molds, the flow of the mortars has te be measured. It is necessary to measure the flow of the fresh mortar because the flow tells how workable the mortar paste is. To measure the flow of the fresh mortar the flow test is performed. For the flow test the following is needed: a cone, a scale for measurement and a tamping rod made of steel. Figure 3.2 shows the set-up for a slump test.
Figure 3.2: Set-up of the flow test.

The mold for the flow test is a cone as shown in figure 3.2. The base of the cone is placed on a surface and the container is filled with fresh mortar in two layers. Each layer is tamped 25 times with a standard 16 mm diameter steel rod, rounded at the end. When the mold is completely filled with fresh mortar, the top surface is struck off [3]. The cone must be held against its base during the entire operation so that it could not move due to the pouring of the fresh mortar [3]. Immediately after filling is completed and the mortar is leveled, the cone is slowly lifted vertically. The unsupported mortar will now slump.

According to the European standards table 3.2 is used.

Table 3.2: Slump in mm [10].

<table>
<thead>
<tr>
<th>Degree of workability</th>
<th>Slump (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very low</td>
<td>10 - 50</td>
</tr>
<tr>
<td>Low</td>
<td>50 - 100</td>
</tr>
<tr>
<td>Medium</td>
<td>100 - 160</td>
</tr>
<tr>
<td>High</td>
<td>160 - 210</td>
</tr>
<tr>
<td>Very high</td>
<td>≥ 210</td>
</tr>
</tbody>
</table>

It is now clear what a flow test is and why it is performed. Now the results of the flow test can be interpreted very well. The results of the flow test is shown in Table 3.3.

Table 3.3: Results of flow test and the unity-weight.

<table>
<thead>
<tr>
<th>Control</th>
<th>SHM</th>
<th>PAE-5+SHM</th>
<th>PAE-10+SHM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow (mm)</td>
<td>81-125-141</td>
<td>79-99-119</td>
<td>129-189-201</td>
</tr>
<tr>
<td>Unit-weight (kg/m³)</td>
<td>2248</td>
<td>2205</td>
<td>2115</td>
</tr>
</tbody>
</table>

Table 3.3 shows the flow of each specimen. Below the control mortar the following flows are written: 81 mm, 125mm and 141 mm. The first value shows the flow of the fresh mortar when the cone is slowly removed for the first time. The second value shows the flow of the fresh mortar after the plate, on which the slumped mortar lay, is hit gently 10 times (according to the European standards). The third value is the value of the flow after hitting the plate 15 times (according to the Japanese standards). The results show that the flow of polymer-modified mortar is higher than the control and SHM mortar.
Table 3.3 also shows the unit-weight of each mortar type. The unit-weight of the control mortar is bigger than the unit-weight of the polymer-modified mortar. This is because of the improved air retention of the polymer-modified mortar.

Now knowing the quantitative side of the flow test, it is also nice to see how the slumped fresh mortars look like. The figures below show how the slumped fresh mortars looked like after gently hitting the plate 10 times.

Figure 3.3: Flow of control mortar.

Figure 3.4: Flow of SHM mortar.
Once again, after seeing the results and pictures it can be said that the workability of the polymer-modified self-healing mortar is higher than the control mortar. It is also important to notice that the unit-weight of the polymer-modified self-healing mortar is decreased. This is because of the improved air retention of the polymer-modified self-healing mortars.

### 3.3.2 Three Point Flexural Test

The three point flexural test is a method to measure the behavior of materials subjected to beam loading. Figure 3.7 shows the set-up of the flexural test.
In this set-up the load is applied in the middle of the specimen. Then the load with its associated displacement is measured, until the specimen is broken. The measurements take place computer-controlled.

So with this test method the flexural strength of the specimens can be measured. The flexural strength is a mechanical parameter for brittle material, like hardened mortar or concrete. It is defined as a material’s ability to resist deformation under load. The flexural strength can be calculated with the following equation.

\[ \sigma = \frac{3FL}{2bd^2} \]  

With F=the applied load, L=length of the beam, b=width and d=thickness of the beam. The value that comes out of the equation represents the highest stress experienced within the material at its moment of rupture.

As said before, there were two types made for each mortar. Every type had three replicates. The first types were meant to harden for 14 days and the second types were meant to harden for 28 days. From now on, specimens that are hardened in 14 days will be mentioned as specimen(14). The specimens hardened in 28 days will be mentioned as specimen(28). So there are three 14 days specimens and three 28 days specimen for each mortar type.

The results from this test is plotted in a load-displacement diagram. This is done for the 14 days hardened specimens and 28 days hardened specimens. For each specimen is the flexural strength calculated with the equation 1.

The load-displacement diagrams are shown below.
Figure 3.8: Load-displacement diagram of control mortar.

Figure 3.9: Load-displacement diagram of control mortar.
Figure 3.10: Load-displacement diagram of SHM mortar.

Figure 3.11: Load-displacement diagram of SHM mortar.
Figure 3.12: Load-displacement diagram of PAE-5+SHM mortar.

Figure 3.13: Load-displacement diagram of PAE-5+SHM mortar.
The diagrams show a linear relationship between the load and the displacement. This is because the specimens are brittle and every brittle material show a linear relationship between load and displacement.

For a better comparison Table 3.4 shows the (average) maximum load on each set and the (average) flexural strength of each set of three replicates.
Table 3.4: Maximum load and flexural strength of each set of specimen.

<table>
<thead>
<tr>
<th></th>
<th>Load (kN) 14 days</th>
<th>Load (kN) 28 days</th>
<th>Flex. S. (N/mm²) 14 days</th>
<th>Flex. S. (N/mm²) 28 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>1.95</td>
<td>2.99</td>
<td>7.3</td>
<td>11.2</td>
</tr>
<tr>
<td>SHM</td>
<td>2.31</td>
<td>2.14</td>
<td>8.7</td>
<td>8.0</td>
</tr>
<tr>
<td>PAE-5+SHM</td>
<td>2.48</td>
<td>2.79</td>
<td>9.3</td>
<td>10.5</td>
</tr>
<tr>
<td>PAE-10+SHM</td>
<td>2.87</td>
<td>3.29</td>
<td>10.8</td>
<td>12.3</td>
</tr>
</tbody>
</table>

The average load-displacement diagram of each specimen is given in figures 3.16 and 3.17.

![Figure 3.16: Average load-displacement diagram at 14 days.](image1)

![Figure 3.17: Average load-displacement diagram at 28 days.](image2)
From the test results the flexural strength of the 28 days hardened mortars are higher than the flexural strength of the 14 days hardened mortars. However, the SHM(28) mortar shows a decrease in flexural strength. The SHM(28) mortar has also the lowest flexural strength. PAE-10+SHM mortar has the highest flexural strength in both cases, 14 days hardened and 28 days hardened.

### 3.3.3 Compression Test

A compression test is a method to measure the behaviour of materials under crushing load. The specimen is compressed and deformation at various loads is recorded. In this experiment the deformation under various load is not recorded, only the maximum load is recorded under which the specimen fails. Figure 3.18 shows the set-up of the compression test.

![Figure 3.18: Set-up of the compression test.](image)

The test is performed by pulling the lever up and down, until the specimen is broken. The maximum compression load can be read from the small white box next to the lever. This is done for each specimen.

Table 3.5 shows the maximum compressive load and maximum compressive strength of each specimen.

<table>
<thead>
<tr>
<th></th>
<th>C. Load(kN)</th>
<th>C. Load(kN)</th>
<th>C. S.(N/mm²)</th>
<th>C. S.(N/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>14 days</td>
<td>49.48</td>
<td>58.99</td>
<td>30.9</td>
<td>36.9</td>
</tr>
<tr>
<td>28 days</td>
<td>49.34</td>
<td>50.34</td>
<td>30.8</td>
<td>31.5</td>
</tr>
<tr>
<td>Control</td>
<td>49.34</td>
<td>50.34</td>
<td>30.8</td>
<td>31.5</td>
</tr>
<tr>
<td>SHM</td>
<td>31.88</td>
<td>38.92</td>
<td>19.9</td>
<td>24.3</td>
</tr>
<tr>
<td>PAE-5+SHM</td>
<td>29.06</td>
<td>51.14</td>
<td>18.2</td>
<td>32.0</td>
</tr>
<tr>
<td>PAE-10+SHM</td>
<td>29.06</td>
<td>51.14</td>
<td>18.2</td>
<td>32.0</td>
</tr>
</tbody>
</table>
First it can be said that the compressive strengths of the 28 days hardened specimens are higher in comparison with the compressive strength of 14 days hardened specimens. The compressive strength of SHM(28) mortar is almost the same as the compressive strength of PAE-10+SHM(28) mortar. Further shows PAE-5+SHM(28) mortar the lowest compressive strength and the control(28) mortar shows the highest compressive strength.

3.3.4 Pull-out Adhesion Test

The adhesion test is a method to measure how strong the bond is between two different/same materials. With this test it is also possible to measure the tensile strength of one of the materials. In this experiment the fresh mortar from each type was bonded with an already hardened concrete plate. After 28 days the adhesion test was performed. Figure 3.19 shows the set-up of the adhesion test.

![Figure 3.19: Set-up for the pull-out adhesion test.](image)

Figure 3.19 shows that a metal ring with a diameter of 5 cm is glued to the mortar. The device next to/on the mortar is then connected with this metal ring. After that, the lever of the device is turned until the specimen is pulled out
of the plate. The load at which the mortar fails can be read from the circular meter on the device.

The results of the adhesion test is mentioned in table 3.6.

<table>
<thead>
<tr>
<th>Tensile load (kN)</th>
<th>Tensile strength (N/mm$^2$)</th>
<th>Failure mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>3.9</td>
<td>1.9</td>
</tr>
<tr>
<td>SHM</td>
<td>3.6</td>
<td>1.8</td>
</tr>
<tr>
<td>PAE-5+SHM</td>
<td>3.9</td>
<td>1.9</td>
</tr>
<tr>
<td>PAE-10+SHM</td>
<td>4.0</td>
<td>2.0</td>
</tr>
</tbody>
</table>

The results show that the bond between the mortar and the concrete plate is very good, because in every specimen the mortar failed. Further it can be said that PAE-10+SHM mortar has the highest tensile strength and the plain mortar has the lowest tensile strength. The control mortar and PAE-5+SHM mortar have the same tensile strength.

Figure 3.20 shows how a mortar fail looks like.

3.3.5 Water-permeability test

The water-permeability test is a useful method for measuring how good a crack is healed. One of the objectives of this experiment is to make a comparison between autogenous self-healing and autonomous self-healing using bacteria. The set-up of the test is shown in figure 3.21.
To perform this test, a circular 28 days hardened mortar should be cracked. The circular mortar has a diameter of 10 cm and a thickness of 1.5 cm. The crack is made by using the compression device from figure 17. The cracks that occur have a width between $300\mu m$ and $500\mu m$. After making the cracks, the specimen was glued in a metal ring. Figure 3.22 shows this.

After this, the metal ring with the specimen is set under water for a couple of weeks. This is done to make sure the cracks will heal by the bacteria or non-hydrated cement particles inside the mortar.

After a couple of weeks under water, the specimen should be installed inside the water-permeability device. The metal ring with the specimen will be
installed at the bottom of the cylindrical container. Then the container will be filled with water. If the cracks are not healed then there will be a flow of water through the specimen. This water will end up in the smaller container that lies on the scale. This scale is connected with the computer where the data is stored.

As mentioned before, the metal ring with the specimen should be put under water for a couple of weeks in order to activate the bacteria that heal the cracks. This means that this test takes some time. Because of time shortage, this test is not performed completely. Right now the metal rings with the specimens are under water.

There is also mentioned that one of the objectives of this thesis is to make a comparison between the autogenous and autonomous self-healing. This comparison can not be done without completing the water-permeability test. Fortunately researchers at Technical University Delft, Dr. H.M. Jonkers and Dr. Virginie Wiktor, did this test before (without using polymers). They found crack-healing of up to 0.48mm wide cracks in self-healing mortar (plain mortar) and a crack-healing of up to 0.18mm wide cracks in control mortar. These values are found after submersion of the specimens under water for 100 days. Figure 3.33 shows adjusted stereomicroscope images of the control and plain mortar before and after healing.

Figure 3.33: Stereomicroscope images of before and after crack-healing [12].

The water-permeability test for this thesis will be performed after a couple of weeks and the results will also be published.
4 Discussion

This section discusses the results of the experimental work that is performed in the previous section. First the results of the slump test will be discussed. Then the results of the flexural test. After that the results of the compression test. After discussing the results of the slump test, flexural test and compression test, the results of the adhesion test will be discussed. At the end the self-healing of the cracks will be also discussed. During the discussion the hypothesis, that is made in the beginning of the experimental work, will be kept in mind. This is necessary because it is important to know if the hypothesis (expectations) came out or not. If the expectations came out, then the test results support the hypothesis. If the results do not match the expectations then it is important to know why.

4.1 Workability

The results of the flow test showed that the workability of the polymer-modified mortars are higher than the workability of the control and SHM mortar. The reason why the workability of the PAE-5+SHM and PAE-10+SHM mortar is much higher is because of the improved fluidity due to ball bearing action of polymer particles among cement particles and entrained air.

The test results also show that the unit-weight of the polymer-modified mortars are less than the control mortar. This is because of the improved air retention due to the ball bearing effect of the polymer.

Hypothesis 1 was: the fresh polymer-modified self-healing mortars will have a better flow, the workability will be better in comparison with the control and plain mortar. This is expected because of the ball bearing effect of the polymers.

The results of the slump test confirm hypothesis 1.

4.2 Flexural Strength

The results of the three point flexural test show that PAE-10+SHM mortar has the highest flexural strengths. This is because of the polymer films inside the mortar. The polymer films make sure that there is a strong bond between the cement hydrates and aggregates. This results in a higher flexural strength in comparison with the control mortar. This effect can be increased by increasing the polymer content to a certain level, the results also shows this. PAE-5+SHM mortar has a lower flexural strength in comparison with PAE-10+SHM.

Hypothesis 2 was: the flexural strength of the polymer-modified self-healing mortar will be higher than the flexural strength of the control mortar. The is expected because of the co-matrix of the polymer-modified mortar. The co-matrix consists of cement gel and polymer films. Because of this co-matrix there is a better bond between the cement hydrates and the aggregates resulting in a higher flexural strength.

The results of the flexural strength confirm hypothesis 2.

4.3 Compression Strength

The results show that the control(28) mortar has the highest compressive strength. Then comes PAE-10+SHM(28) mortar with a compressive strength of 31.9
The SHM(28) mortar has a compressive strength of 31.5 N/mm$^2$ and the PAE-5+SHM mortar has a compressive strength of 24.3 N/mm$^2$.

The polymer-modified mortars show a lower compressive strength in comparison with the control mortar. This can be explained because of the air (and water) retention of the polymer. The polymer-modified mortars have more entrained air then the control/SHM mortar. This makes sure that the compressive strength becomes relatively low.

Hypothesis 3 was: the compressive strength of the polymer-modified mortar will be more or less the same as the compressive strength of the control/plain mortar.

The results of the compression test does not completely confirm hypothesis 3 because the control mortar has a higher compressive strength then the polymer-modified mortars. The reason for this is that the polymer-modified mortars have more entrained air in comparison with the control/SHM mortar.

### 4.4 Adhesion

The failure mode in all four specimens was mortar fail. This means that the bond between the mortar and the concrete plate was very good. If the bond is very good, then why did the concrete not fail? The reason for this is that the concrete plate is much older than the mortar and therefore much stronger. This is why the failure occurred in the mortar and not in the concrete plate.

The results show that the PAE-10+SHM mortar has the highest tensile strength. This is because of the polymer films. The polymer films ensure a strong bond between the cement hydrates and aggregates. This effect can be increased by increasing the polymer content to a certain level. Further have the control mortar and the PAE-5+SHM mortar the same tensile strength and the plain mortar has the lowest tensile strength. It is important to notice that the differences between the tensile strengths are small.

Hypothesis 4 was: the adhesion of the polymer-modified self-healing mortar to a concrete slab will be very good because of the polymer that is used as cement modifier. The polymer-modified mortar will also have a higher tensile strength over the control/plain mortar.

The test results show that adhesion and tensile strength of each mortar are more or less the same.

### 4.5 Crack Healing

Hypothesis 5 was: The crack-healing of the self-healing mortar will be more then the crack-healing of the control mortar. The bacteria inside the self-healing mortar makes sure that the crack is healed as good as possible. The crack-healing inside the control mortar occurs because of the non-hydrated cement particles. The control mortar has no bacteria inside the mortar that can act as a part of an autonomous repair system. That is why it is expected that the crack-healing with bacteria will be better then the crack-healing without bacteria.

This hypothesis can not be confirmed right now because the water-permeability test is not performed due to time shortage. But according to Dr. H.M Jonkers and Dr. Virginie Wiktor is the hypothesis right. They showed that after 100 days the cracks from the self-healing mortar were completely healed.
5 Conclusion

The results of this study show that the polymer-modified self-healing mortar has a better mechanical and physical properties over the conventional mortar. The polymer-modified self-healing mortar has a higher workability, higher flexural strength, higher compressive strength, and of course a higher tensile strength. The polymer-modified self-healing mortar has an autonomous self-healing mechanism because of the bacterial spores and calcium lactate. This means that the mortar heals itself when needed. If there is a crack inside the mortar and water goes inside the mortar, the bacteria will become active and heal the crack to prevent corrosion of the reinforcement. So the polymer-modified self-healing mortar is more durable in comparison with conventional mortar and also has better physical and mechanical properties. This concept can also be used to make polymer-modified self-healing concrete.

The polymer-modified self-healing mortar is a good construction material that can be used to repair damaged concrete structures or it can be used to bind bricks together. It is also possible to make concrete structures with polymer-modified self-healing concrete. This type of mortar/concrete is more durable and also very strong.

It is advisable to use the polymer-modified self-healing concrete for building tunnels, bridges or concrete structures in wet environment. The reason for this is because of the self-healing property and durability of this type of concrete. For example, if there is a polymer-modified self-healing tunnel then the tunnel does not need a lot of maintenance. If the tunnel cracks then the cracks will be healed by itself because of the bacterial spores. It is important to notice that repairing damages of a concrete tunnel is very expensive and also very hard and labor intensive. If the tunnel is made of a self-healing concrete then the tunnel will repair itself and there is no external repair needed. In these kind of cases a polymer-modified self-healing concrete is advisable.
References