CoRncrete
A bio-based construction material

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Challenge the future

CoRncrete
A bio-based construction material

By

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University of
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Preface

'I never wanted to do a successful thesis, but an effective one'

This thesis present research on a material called 'CoRncrete' that I have carried out in past 8 months as a part of the master program in Civil Engineering and Geosciences. Research on CoRncrete combines the field of Geo-Engineering and construction materials, making it a multidisciplinary project. This thesis is an outcome of passion and enthusiasm in trying something new.

The thesis has been carried out mainly in Microlab, Stevin II lab and Geotechnical lab at the Delft University of Technology. I would like to express my gratitude towards my supervisors - Prof. Erik Schlangen, Dr. Henk Jonkers, Dr. Phil Vardon and Dr. Leon van Paassen for their guidance and support. All supervisors agreed to my research proposal instantly and the thesis committee was formed in a single day. The formation of the committee in a single day is definitely a rare event. I am really thankful to my supervisors who gave me the freedom to carry out the research without worrying about the resources.

I am grateful to Renee Mors for motivating me to carry out the initial experiments and to help me improve my technical writing skills. I am also thankful to Virginie Wiktor who showed confidence in my idea and motivated me to interact with professors in material and environment section. I would like to thank Damian Palin for his motivation, guidance and assistance in experimental work. I am thankful to Gerrit Nagtegaal, Arjan Thijssen, John van den Berg from Microlab; Maiko van Leeuwen and Ton Blom from Stevin II; Arno Mulder and Wim Verwaal from geotechnical lab and Ben Norder from polymer materials and engineering section for guiding and assisting me in the experiments. The time spent in these labs was full of fun and knowledge.

I express my gratitude towards Prof. Stephen Picken for his constructive discussion on my thesis. I also thank Jure Zlopasa for discussion on this research.

Finally, I would like to thank my family and friends who kept me motivated throughout the two-year journey of master study. I made some of the awesome friends in delft whom support and company have been phenomenal and highly influential in my personal and professional growth. I am avoiding writing the names of my friend because of this thought in my mind right now, "each and every friend is crucial in defining the shape of life or in some cases thesis, so it's not a very good idea to differentiate, at least on paper!"

This thesis has been crucial in my development as a researcher and I wish to pursue a career in research and academia in future. I would like to conclude this preface with a quote:

"Life should be such that your museum is interesting"

Yask Kulshreshtha
Delft, July 2015

Abstract

CoRncrete is a corn starch based bio-material formed by mixing corn starch with water and sand, and heating the mix in a microwave or oven. This heating process results in the formation of a hardened material. A constant corn starch to sand proportion of 1:5 has been adopted in this study.

The transformation of CoRncrete from a semi-solid to a hardened material is caused by modification of corn starch that occur through a ‘gelatinization’ process. Heating of corn starch in the presence of water results in the formation of a gel that binds with sand grains and hardens. The temperature at which gel formation or gelatinization in CoRncrete occurs, was studied by DSC (Differential Scanning Calorimetry). To understand the microstructure of CoRncrete, ESEM (Environmental Scanning Electron Microscope) tests were carried out. ESEM scans revealed the effect of water content, heating source and heating duration on the gelatinization process that affects the development of microstructure of CoRncrete.

The physical properties of CoRncrete was studied by analysing consistency behaviour of fresh CoRncrete and compressive strength of hardened CoRncrete. It was found that the corn starch in fresh CoRncrete changes its phase from solid to plastic/liquid at a specific water content. This water content was determined by cone penetrometer and proctor tests. This change in phase of corn starch was observed at an optimum water content. At optimum water content, the maximum dry density of fresh CoRncrete and maximum density and strength of hardened CoRncrete is also achieved. The fresh CoRncrete was heated to form hardened CoRncrete with the compressive strength found to be comparable to that of red clay bricks. The factors influencing the strength of hardened CoRncrete such as water content, size of the sand particle and heating duration (in a microwave) has been studied and discussed. A strong positive correlation between density and strength of hardened CoRncrete has been observed. Degradation of CoRncrete was studied by immersing specimens in water. Specimens were degraded partially to fully within a day, raising durability issues.

The sustainability of CoRncrete was studied by carrying out LCA (Life Cycle Assessment) and compared with LCA results of Portland cement concrete and fired clay brick. The analysis indicated that the CoRncrete has comparatively higher environmental impact. Finally, the limiting factors in application of a CoRncrete and their remedial measures have been discussed.

This research work addresses CoRncrete as a potential precursor to a new class of construction materials which gain strength rapidly by heating at relatively low temperature (~100^{o}C).

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Introduction

“There is no secret ingredient. To make something special you just have to believe its special
– Mr.Ping,

1.1. What is CoRncrete?

CoRncrete, as the name suggest, is a bio-based material which uses corn starch as a binder. Corn starch, also known as maizena or corn flour, is a common food ingredient used for thickening soup and sauces. It is a natural polymer derived from a renewable source; Maize crop. In recent years corn starch has been widely used as a ‘green’ material in the production of bio-plastic and bio-ethanol. Corn starch is a remarkable material that has been fascinating everyone with its unusual properties when mixed with water. Suspension of corn starch in water forms a shear thickening fluid, affectionately referred as magical fluid on which one can run without sinking. A lesser known heat-induced transformation of corn starch is explored in this project and it has been applied in the development of a novel construction material, CoRncrete.

CoRncrete is formed by mixing corn starch with sand and water, and heating the mix in a microwave or oven. A video on ‘How to make CoRncrete’ is also available on YouTube. This video was prepared in the nascent stage of the project. The link and QR code is shown in figure 1.1.

Figure 1.1. Screenshot and QR code of ‘How to make CoRncete’, video available on YouTube. The video can also be seen via visiting this link: https://www.youtube.com/watch?v=hqff5Iq7iCs or by Searching “how to make CoRncrete” in YouTube search bar. Please note that the vegetable oil shown in this video is no longer used to make CoRncrete.

Dry corn starch is mixed with sand in a specific weight proportion. The weight proportion of dry corn starch to sand is kept 1:5 throughout this research study based on preliminary test results (discussed in 1.3). Water is added to mix and stirred well. This mixing process results in the formation of fresh CoRncrete. Fresh CoRncrete is then poured into silicone rubber mould and heated in microwave or convection oven. The CoRncrete sample is heated in a microwave for 3-10 minutes (based quantity of CoRncrete and frequency of microwave) and 12-24 hours in an oven at temperature exceeding 100^{o}C. The heating process results in the formation of a hardened material whose compressive strength is comparable to that of red clay bricks (15-25 MPa). Figure 1.2 show schematic diagram on the preparation of CoRncrete.

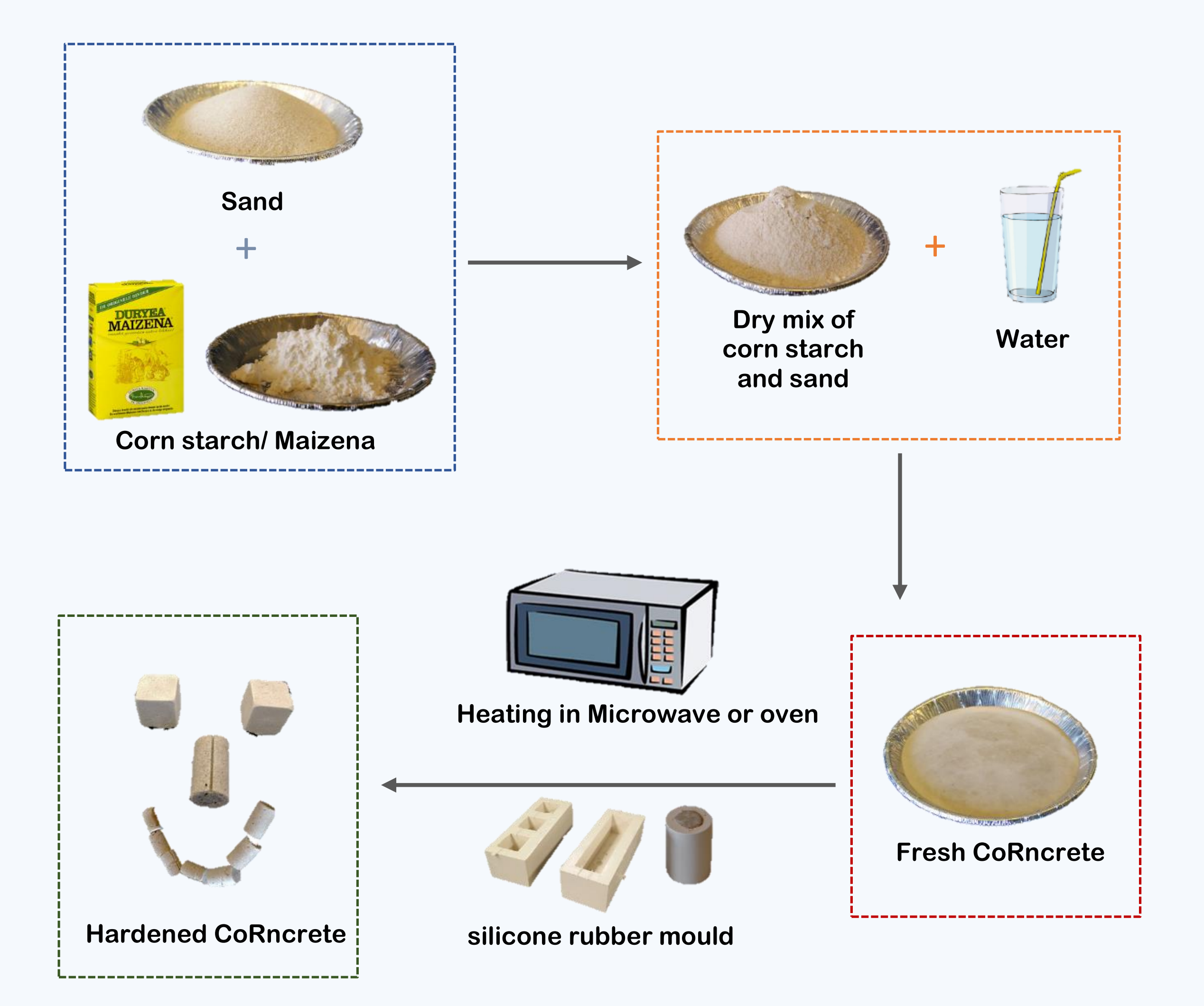

Figure 1.2. Preparation of hardened CoRncrete by mixing corn starch and sand in 1:5 weight proportion, and heating the mix in microwave or oven. The resulting material has compressive strength comparable to red clay brick (15-25 MPa).

1.2. Genesis of Idea

CoRncrete is a ‘fiction movie’ inspired material. This concept may sound bizarre at first instance, but ‘fiction movies’ has inspired the development of technology like advanced 3D printer. A faster technique of 3D printing has recently been developed that has derived its inspiration from Terminator 2 movie (DeSimone, TED 2015). An interesting video on ‘Incredible Hulk’ has also been published by Stanford (Sebastian Alvarado, 2014) recently that addresses the biological science behind origin of hulk and its transform from a human to monstrous green creature.

CoRncrete is inspired from sand man, a character from movie Spiderman 3. Sandman is an interesting character which possess a dual level transformation power: from human to sand structure and, from a loose sand structure to rigid sand structure and vice versa. Sandman can be thought of as a smart flexible material which can change its shape and stiffness based on his wish. Sandman travels as a cluster of flowing sand grains, but when encountered by his enemies, he transforms himself into rock solid sand structure of high stiffness. His strength is then enough to crush a car into a sheet of metal. It seems that this transformation is unlikely with sand grains alone as the material in Sandman’s body. There might be an additional material in between sand grains which can reversibly bind sand grains. This reversible glue like material might be activated by some sort of mechanical, electric, thermal or chemical signal which results in physical transformation. One of the interesting materials that may perform the task of reversible material can be corn starch. It is known that the corn starch with water act as a smart material that can change its stiffness and viscosity upon mechanical, thermal and electric stimulation.

In an attempt to make a sandman like smart material, sand was mixed with corn starch and water. This mix showed impact activated stiffness at certain water content. One of the issues encountered in this material was inability to attain a definite shape. Apparently, Corn starch mixed with sand and water is a viscous fluid that has no fixed shape. In order to provide a definite shape to this high viscosity material, it was heated in a microwave. It was expected that a soft yet well-defined shaped material showing impact activated stiffness will form in a microwave. Unfortunately or rather fortunately, a harder and strong material came out of microwave having no impact activated stiffness property. This material was later named CoRncrete.

Figure 1.3 represent the inspiration derived from Sandman pictorially. Sandman is basically a human which can transform himself in sand structure. The sand structure or Sandman in this image is shown as a composite material with sand grains and corn starch particles. This material was expected to behave like sandman upon heating but resulted in CoRncrete. The image of sandman destroying the building with corn starch roof is an original work by author and it captures the essence of this research; interaction of sand with corn starch.

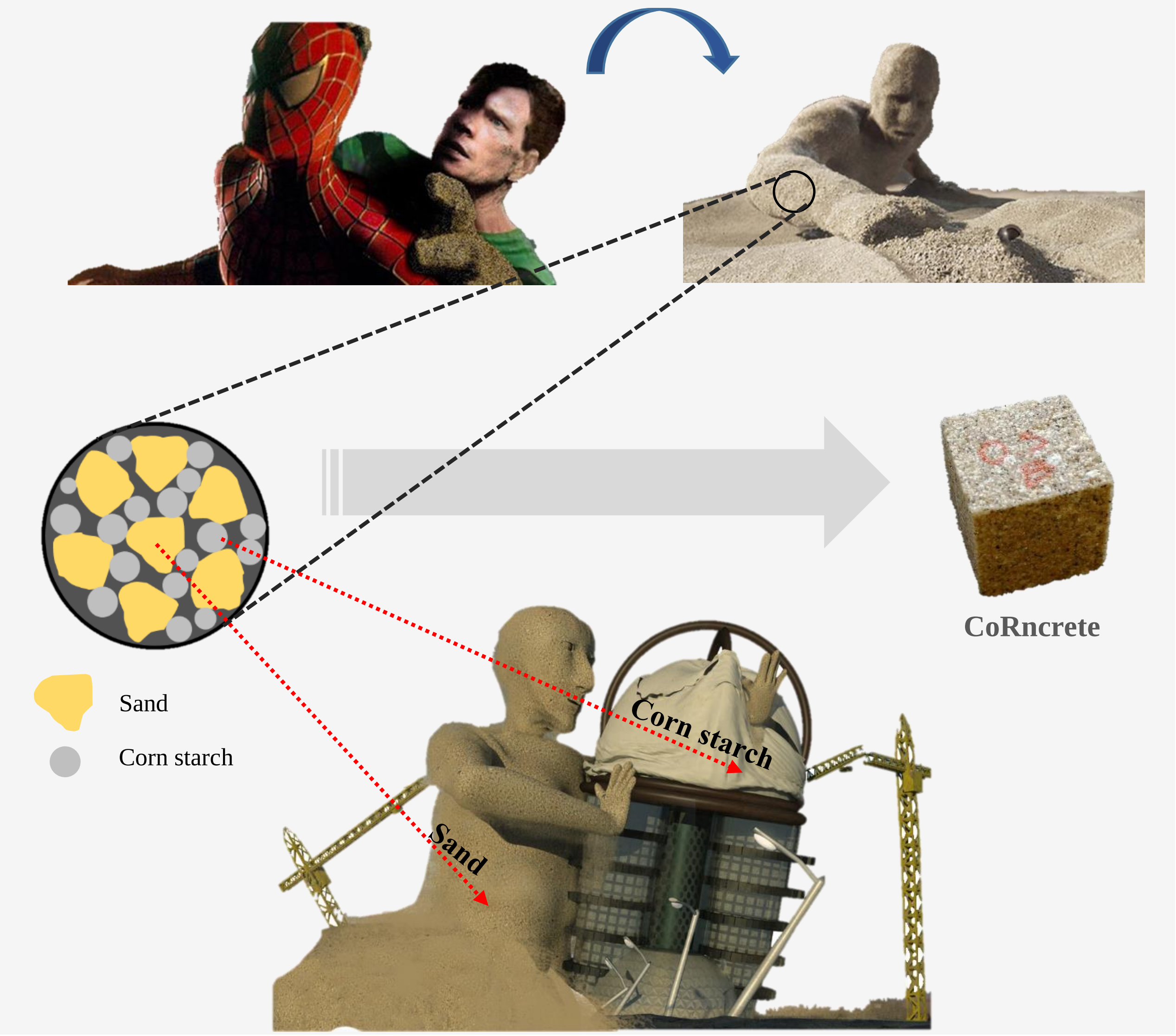

Figure 1.3. A representation of inspiration Behind CoRncrete - Sandman. Sandman, a character from Spiderman 3, can be thought as a smart material which has a 'glue' like material in between sand grains that give it the flexibility to change its stiffness. In an attempt to make a sandman type material, corn starch together with water and sand was heated in the microwave. Contrary to the expectation of a flexible material, hardened CoRncrete was formed.

1.3. Preliminary testing

The preliminary testing of CoRncrete has been influential in defining the framework of this research work. The preliminary testing was aimed at understanding the influence of corn starch, sand and water on the compressive strength of CoRncrete. The first set of testing was carried out on cylindrical specimens (height: 60mm and width: 33mm, two longitudinal notches: 2mm width). The specimens were prepared in silicone rubber mould and each specimen was heated for 3 minutes in a microwave. Specimens were also heated in an oven for 24h at 105^{o}C. The proportion of corn starch, water and sand was varied randomly and its effect on the compressive strength of CoRncrete was studied. The following observation were made during first set of testing:

1. CoRncrete specimens popped out during heating in a microwave. (Also shown in video on 'how to make CoRncrete')
2. Specimens prepared by heating in a microwave were significantly deformed.
3. The compressive strength results of CoRncrete prepared through microwave heating had a significant deviation from mean.
4. Specimens prepared by oven heating had deposition of corn starch at the top.
5. Specimens heated in oven dissociated faster in water as compared to microwave heated specimens.

In the second set of testing, prismatic specimens (40×40×40mm) were casted. In order to prevent pop up of specimens while heating, surcharge load was kept over it. A different combination of the heating mode was tried to control pop-up of the specimen. Finally, a regime of 5min, first 1.5min in thaw mode and next 3.5min in full power mode, was selected for heating 3 fresh CoRncrete specimens simultaneously. Specimens were heated with different corn starch to sand ratio. A corn starch to sand ratio of 1:5 was selected for further testing based on compressive strength results. The influence of sand particles on the strength of CoRncrete was also observed. Test results and extra information about preliminary testing is shown in Appendix A.

Preliminary testing was helpful in determining factors that may influence the strength of CoRncrete. Although, the preliminary testing lacked scientific accuracy, the results played an important role in defining the objectives and test schemes.

1.4. Research questions and objective

Main objective:

To understand and study physical property of CoRncrete, and to analyze its sustainability with respect to traditional building material (concrete/brick)

Sub objectives/ research questions:

1. To understand the formation of microstructure of CoRncrete by studying mechanism of heating in microwave and oven, observing the microstructure of CoRncrete at varying water content, degree of heating and source of heating, and formulating a hypothesis on strength development of CoRncrete based on these observations.

2. To study the physical property of fresh and hardened CoRncrete, initially by finding out an optimum water content in fresh CoRncrete which result in maximum compressive strength of hardened CoRncrete and later, testing the factors that may affect the strength of CoRncrete.

3. To study degradation of CoRncrete in a wet environment by analyzing the effect of wetting at a microscopic level, and measuring reduction in strength of CoRncrete specimens that are submerged in water.

4. To examine the environmental impact of CoRncrete by performing life cycle analysis and comparing results on selected parameters with traditional construction material of same volume, strength and application, and suggest recommendation for better environmental performance.

5. To discuss the limitations in the application of CoRncrete and suggest remedial measures.

1.5. Thesis outline

The thesis outline is shown as a flow chart in figure 1.4. Chapter 1 introduces CoRncrete, its genesis and the research objectives. Chapter 2, background study describes the state of the art non Portland cement based materials and focuses on gelatinization process in starches. Shear thickening and gelatinization properties of corn starch are discussed which may have an influence on the physical transformation of CoRncrete. Chapter 3 is focused on heat induced transformation of CoRncrete. The potential heating mechanism is explained and the hypothesis on physical modification of CoRncrete is formulated based on the microscopic and thermal analysis. Chapter 4 is the core topic of this research work and focuses on the strength of CoRncrete which is influenced by several factors. In this chapter, proctor and cone penetrometer test were carried out on fresh concrete to determine optimum water content that results in CoRncrete of higher strength. Chapter 5 discusses the degradation of CoRncrete when submerged in water for 1, 3 and 7 days. Equotip non-destructive testing is used for indicating the reduction in hardness of specimen upon wetting. The sustainability of CoRncrete is compared to fired clay brick and Portland cement concrete by performing LCA in chapter 6. Recommendation for a better environmental performance of CoRncrete is also discussed in this chapter. Based on results and recommendations in all previous chapters, application of CoRncrete and limiting factors in its application are discussed in chapter 7. Recommendation for further research and conclusion drawn from this study are mentioned in chapter 8 and 9 respectively.

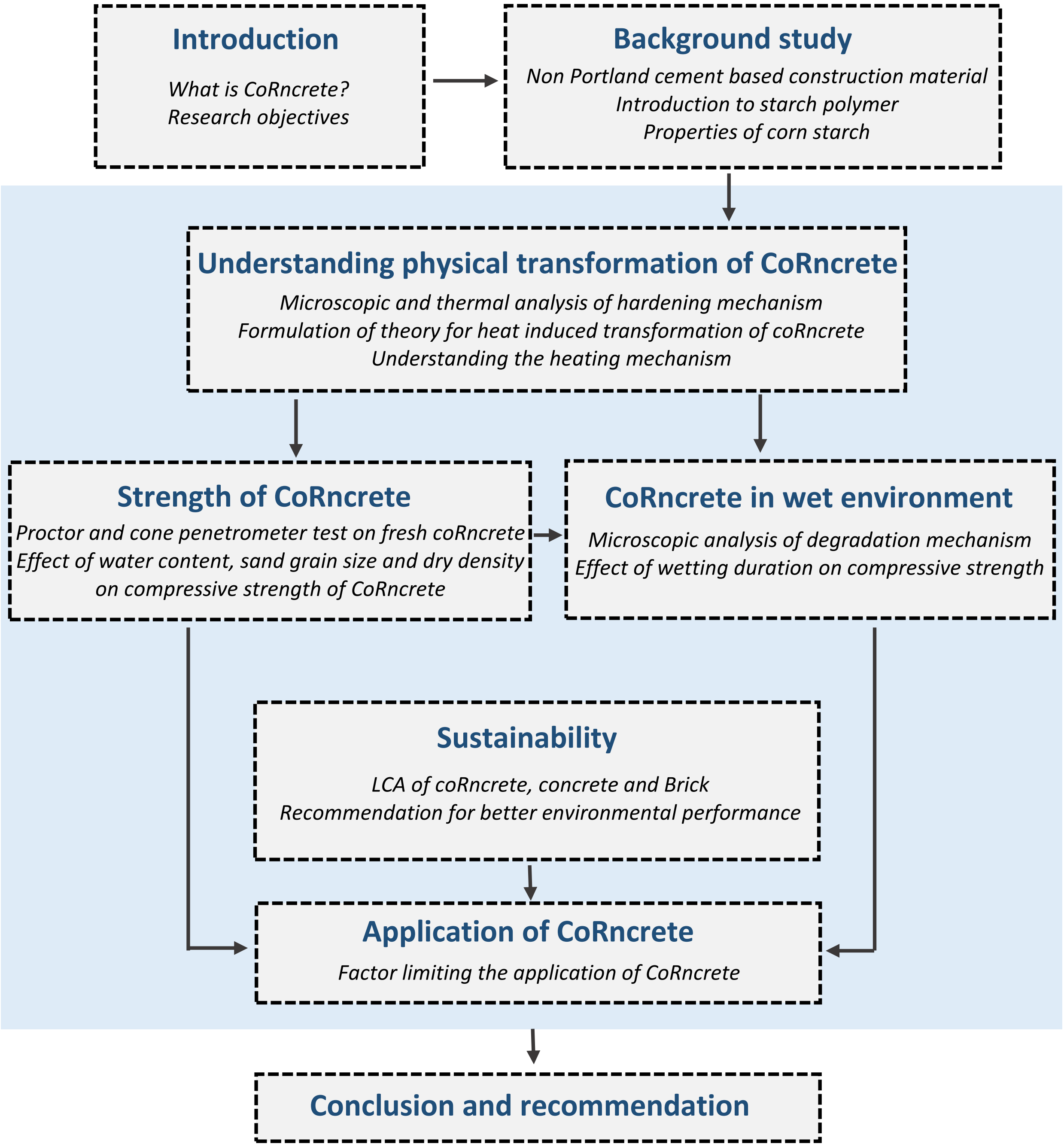

Figure 1.4. Thesis outline of CoRncrete: A bio-based construction material

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Background study

2.1. Introduction

Portland cement concrete is the most common construction material used worldwide. Due to the increase in awareness towards higher carbon footprint of Portland cement concrete, research on non-Portland based construction material is progressing. CoRncrete is a non-Portland cement based material. Other materials in the same category such as CO_2 structure, Bio bricks and geo-polymer concrete are discussed in section 2.2. In order to understand the behavior of fresh and hardened CoRncrete, the thermal and rheological property of corn starch have to be understood well. Introduction to starch polymer and properties of corn starch are discussed in section 2.3 and 2.4 respectively.

2.2. Non Portland cement based materials

2.2.1 CO_2 structure

CO_2 structure, also known as CO_2 eco-structure, is a novel construction material developed by Prof. Norihide Imagawa of Tokyo Denki University in partnership with TIS & Partners Co.ltd, Tokyo, Japan. The CO_2 structure is eco-friendly, rapidly hardening and high compressive strength material. It can be formed in a desirable shape. The fabrication process is also claimed to be eco-friendly as CO_2 is absorbed during the process. Hardener such as Epoxy resin, urethane or lacquer can be added to the CO_2 structure for better heat resistance, chemical stability, waterproofing and tensile strength. The tensile property of CO_2 structure (modified with hardener) is comparable to reinforced cement concrete (RCC). (Imagawa, 2013)

The CO_2 structure is manufactured by a simple process as shown in figure 2.1. Silicone-oxide rich sand is placed in a mould of desired shape (a) (b). The mould is sealed and CO_2 is pumped in (c). CO_2 reacts with silicone oxide rich sand and forms silicon carbide instantly (d). This hardening process is completed within a minute. This process of formation of silicon carbide can be represented in a simplified chemical equation as $SiO_2+CO_2 \rightarrow SiC+2O_2$. At this moment, the material (classified as Type 1 CO_2 structure) is good in compressive strength but fails easily under tensile loading. To improve its tensile property, the CO_2 structure is soaked in epoxy resin (e). The material gains its final strength in 24 h. This CO_2 structure (classified as type 2 CO_2 structure) shows compressive and tensile strength upto 50MPa and 20 MPa respectively. (iC02 lab, 2015). The ICO2 lab claims that the CO_2 structure has a major application in ground recovery and construction of habitat in earthquake hit areas due to a possibility of faster construction of buildings. More information about the fabrication process can be accessed via this video, made by Diginfo TV, Tokyo, Japan: https://www.youtube.com/watch?v=Ppq34lD6d5o

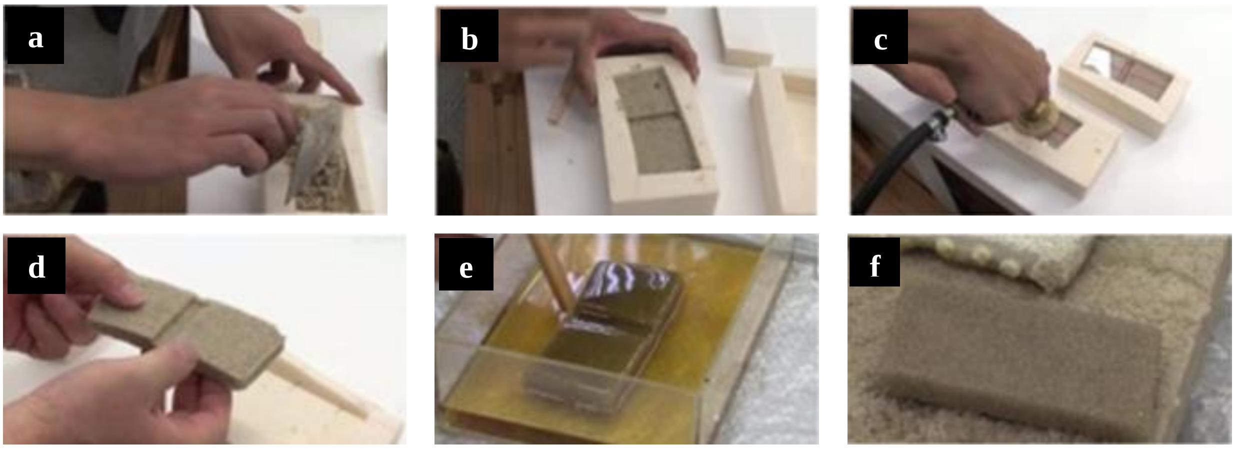


Figure 2.1. The process of making the CO_2 structure. (a) Placing silicone oxide rich sand in mould, (b) giving desirable shape, (c) Injection of CO_2, (d) Formation of silicone carbide (Type 1 CO_2 structure), (e) Soaking in epoxy and (f) Type 2 CO_2 structure ready for application. Images are taken from iCO2 lab website.

2.2.2 Geo-polymer concrete

Geo-polymer concrete is a trending construction material which is considered as third generation cement after lime and ordinary Portland cement (Singh et al., 2015). Geo-polymer is a synthetic alkali aluminosilicate which is formed by reaction of solid aluminosilicate with a highly concentrated aqueous alkali hydroxide or silicate solution. (Davidovits, 1991).

A variety of aluminosilicate materials have been utilized in geo-polymerization technology, but material such as fly ash and metakaolin are the focus of several research work in the formation of geo-polymer concrete. Alkali activators such as sodium hydroxide (NaOH), potassium hydroxide (KOH), Sodium silicate (Na_2SiO_3) and potassium silicate (K_2SiO_3) are used to activate alumninosilicate materials (Duxon et al., 2007). Schematic representation of Geopolymer concrete is shown in figure 2.2.

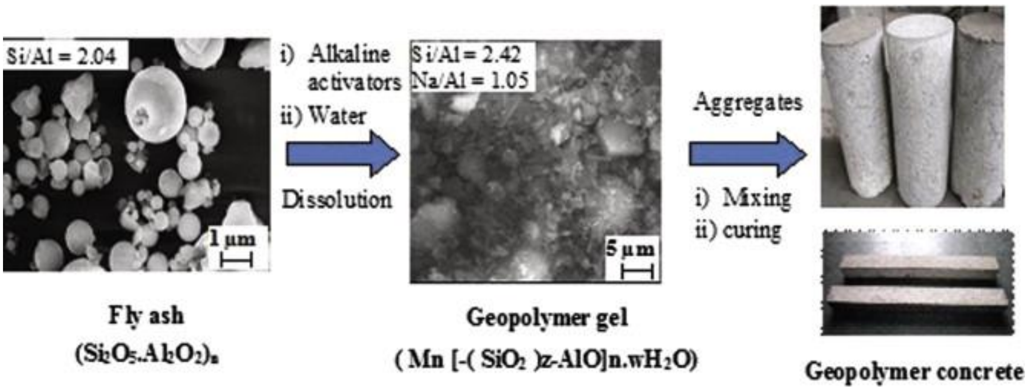


Figure 2.2. Conversion of fly ash into Geo-polymer concrete. (Image taken from Singh et al., 2015**)**

Geopolymer concrete can provide comparable performance to a traditional cementitious material with added advantage of a significant reduction in greenhouse gases. (Gartner, 2004)

2.2.3 Bio-Brick

Microbially induced mineral precipitation (MICP) has been utilized for ground improvement technique (van Paassen et al., 2010, DeJong et al., 2013) and to increase durability of concrete (Jonkers.et al., 2010). A bio-mediated process such as MICP utilizes bacteria to metabolize a carbon or nitrogen source, precipitating minerals in the process.

Recently, Bernadi et al. (2014) utilized MICP for manufacturing of Bio-Brick. In the process of manufacturing bio-brick, high silica sand was initially placed in the mould. The bacterial solution was prepared by growing Sporosarcina paseurii in an ammonium yeast extract media. Bacterial solution was added to sand by percolation (flushing of fluid from top to bottom). This treatment was repeated 3 times a day in order to ensure bacteria attachment at particle contact within a permeable sand matrix. After 28 days of curing, the hardened bricks were heated in an oven at 77°C. Compressive strength in the range of 2-2.5 MPa was achieved. Figure 2.3 show the process of MICP for manufacturing of bricks.

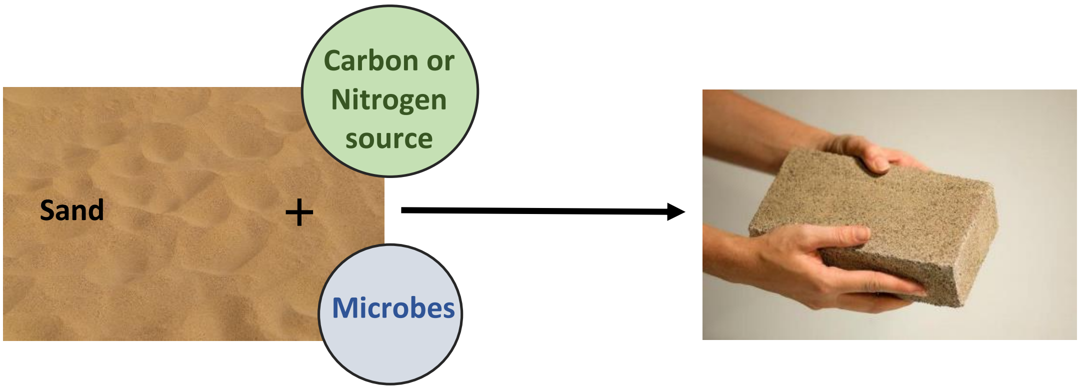


Figure 2.3. MICP (Microbially induced mineral precipitation) in manufacturing of bricks. High silica sand is flushed with a bacterial solution, with added nutrients, multiple times. The final outcome is a hard sand brick.

Biomason, a 2012 startup, has recently started manufacturing bricks based on similar process. They have claimed that their production process finishes in 5 days, releases no CO2 and the bricks have compressive strength comparable to traditional fired clay bricks.

2.3. Introduction to starch polymer

Starch is one of the most abundant natural polymer that is stored in plants. Starch is widely available, cheap, degradable and renewal which makes it an attractive material for food and non-food applications. Starch is extracted from agricultural raw materials and it is present in many food products. Due to its renewability and biodegradable, it is a perfect raw material as a substitute for fossil-fuel components in numerous chemical applications such as plastics, detergents, adhesives (Starch Europe). It is also widely used in papermaking and manufacturing of corrugated boards. In Europe, starch is mainly extracted from potatoes, wheat and maize. According to Starch Europe association, European Union (EU) starch production has increased from 8.7 million tonnes 2004 to 10 million tonnes in 2013. The EU consumes 9 million tonnes of starch (excluding starch bi-products totalling around 5 million tonnes), of which 61% in food, 1% in feed and 38% is used in non-food applications, primarily paper making. 48.2% starch product are based on corn starch.

Starch is known as a semi-crystalline material (Ranayake and Jackson, 2006). The starch molecule consists of a large number of glucose units. “The composition of naturally occurring starch is more or less universal with a major component of amylopectin (75%) and a minor component of amylose (25%) irrespective of its source” (Regina et al., 2014). “When native starch granules are heated in water, their semi-crystalline nature is gradually eliminated, resulting in the phase transition from an ordered granular structure into a disordered state in water which is known as gelatinization” (Lelie`vre and Liu, 1994). The temperature at which gelatinization occurs is called gelatinization temperature. With further heating in excess water, a starch paste is formed due to granule swelling beyond a reversible point. This stage following gelatinization of starch is known as pasting (Quiang, 2005). “When cooled, the dispersed “amorphous” starch molecules undergo slow reassociation forming a tightly packed structure. This process is commonly called retrogradation” (Shrestha and Halley, 2014).

The thermal properties of starch related to gelatinization, pasting and retrogradation are of high importance in order to understand the transformation of CoRncrete.

2.4. Properties of corn starch

Corn starch is the most commonly available starch with wide application in food and non-food industries. The most interesting aspect of corn starch is its behavior when mixed with water. When corn starch is mixed with water it forms a bizarre material that can resist rapid deformation but when left unperturbed, it becomes a thin liquid. The suspension of corn starch particles in water is a non-Newtonian fluid (stress is not directly related to strain). This property allows people to run and bike across a pool of corn starch. In the recent study by Waitukaitis and Jaeger (2012), the reason behind this unusual behavior was found to be dynamic jamming. Rheological and thermal property of corn starch suspension in water is interesting and relevant in order to address the objectives.

Rheology is the study of the flow of matter. The rheological property of corn starch particles in water is important to understand the role of water to corn starch ratio on the consistency of fresh CoRncrete. Crawford et al. (2013) studied the rheological property of corn starch suspension in water by measuring viscosity using a rheometer, at varying shear rate. The viscosity results were translated to thickening stress. A plot of thickening shear stress versus weight percentage of corn starch is shown in figure 2.4. Maximum shear stress is observed at a weight percentage of corn starch 52.5 % (or 90.5% water content; water in corn starch,). The maximum shear stress may relate to the density of corn starch particles in water and hence affect the consistency of fresh CoRncrete. Chapter 4 will elucidate the effect on the consistency of fresh CoRncrete on the strength of hardened CoRncrete.

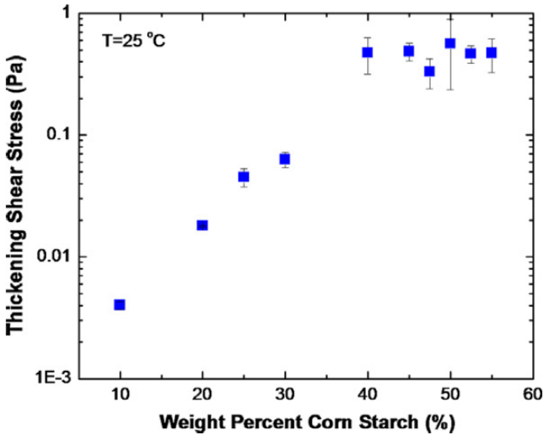


Figure 2.4. Thickening shear stress as a function of corn starch concentration (weight %). The amount of corn starch in the corn starch water mix (corn starch/(corn starch+ water)) is indicated on the horizontal axis. The research on CoRncrete instead uses an amount of water in corn starch (water/corn starch), water content. At 52.2% corn starch concentration or 90.5% water content, maximum shear thickening is observed. (Image taken from Crawford et al. (2013))

The thermal property of corn starch such as Gelatinization is important to understand the transformation of fresh CoRncrete into hardened CoRncrete. Gelatinization process in starch is discussed in section 2.3. Gelatinization temperature may play a crucial role in determining the temperature required for heating of CoRncrete, especially in an oven. Lui et al. (2006) studied the gelatinization temperature of corn starch with different water content (water/corn starch) using DSC (differential scanning calorimetry). Slow heating of 5°C/min was adopted to measure the endothermic heat flow. Figure 2.5 shows the DSC curve for corn starch at varying water content.

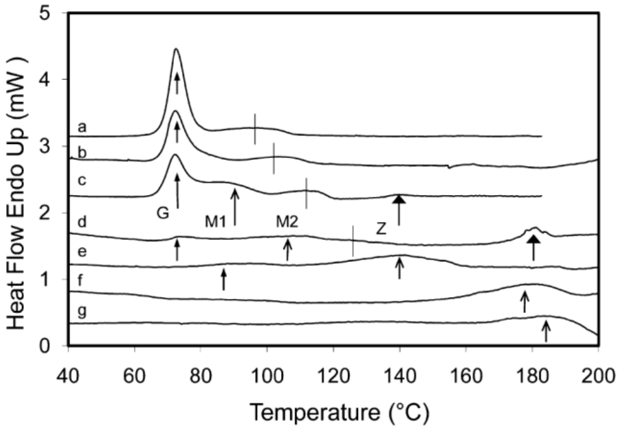


Figure 2.5. DSC endotherms of maize starch with different water content. The moisture content (from top) is: (a) 74.57%; (b) 65.3%; (c) 51.8%; (d) 40%; (e) 29.9%; (f) 16.15%; (g) 9%. The gelatinization temperature increases with decrease in water content (Image taken from Lui et al. (2006))

With a decrease in water content, the gelatinization temperature increases. In this study, water to corn starch percentage adopted in the preparation of fresh CoRncrete range from 60% to 120%. At water content greater than 60%, the peak gelatinization temperature is more or less constant, 72.5°C.

3

Understanding physical transformation of CoRncrete

3.1. Introduction

The transformation of CoRncrete from a semi-solid material to a hardened material is caused by heating that results in physical modification of starch. In order to understand the physical transformation of CoRncrete, microstructural and thermal property are studied. Thermal property: gelatinization temperature of CoRncrete with varying water content (15% and 18%) was studied with differential scanning calorimetry (DSC). To study the microstructure of CoRncrete, samples were imaged on an Environmental scanning electron microscope (ESEM). Cornstarch, partially heated CoRncrete, completely heated CoRncrete, oven heated CoRncrete and two CoRncrete formed in the DSC test were studied. Afterwards, the underlying potential heating mechanism of CoRncrete in oven and microwave is discussed. Finally, DSC and ESEM test results are coupled in order to understand the gelatinization process and hypothesis on physical transformation and strength development of CoRncrete is formed.

3.2. Materials and method

3.2.1 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) is a thermal the analysis technique to measure the heat capacity of material with increasing temperature. In this technique, amount of heat require to raise the temperature of a material at constant temperature rate is measured in comparison to a reference sample. DSC is considered an ideal technique to measure glass transition and gelatinization temperature of the polymeric material.

Sample preparation

The fresh concrete sample was prepared by adding water in 10g dry corn starch- sand (1:5) mix. Two CoRncrete sample with 15% water content (sample A and sample B) and one with 18% water content (sample C) were prepared by adding tap water in dry corn starch-sand mix using a plastic pipette. The mix was stirred well. 18.85mg of sample 1, 17.62mg of sample 2 and 23.37mg of sample 3 was taken and placed in a golden pan for DSC analysis.

Test procedure

To measure gelatinization temperature of fresh CoRncrete at water content 15% and 18% respectively, Perkin Elmer DSC 7 (Netherlands) was used (figure 3.1 (a)). In order to measure the gelatinization temperature of CoRncrete, it was necessary to prevent loss of moisture from the sample. This was ensured by using a high-pressure golden pan (figure 3.1 (b)). An empty stainless steel pan was used as the reference sample (figure 3.1 (b)). The sample pans were heated in air at 5°C/min from 25°C to 200°C and later cooled at 5°C/min from 200°C to 25°C. A slow heating rate was adopted in order to prevent pressure built up and temperature lag due to the high conductivity of golden pan. The heating was limited to 200°C as corn starch burns at 290°C which was indicated by Thermogravimetric analysis (TGA) of corn starch and fresh CoRncrete (refer appendix B for TGA test results). The thermal transition of CoRncrete sample was defined as T_o (onset temperature), T_p (peak gelatinization temperature) and T_c (conclusion temperature).

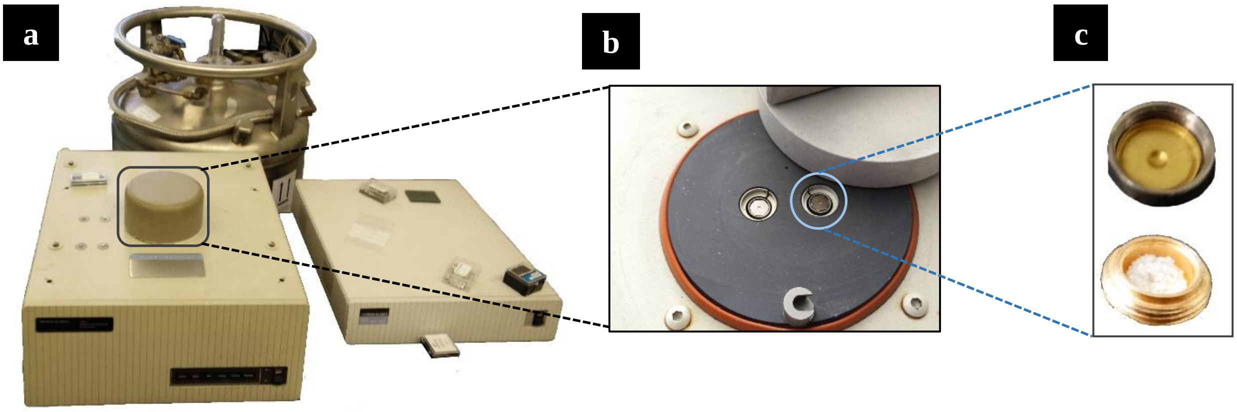


Figure 3.1. DSC test setup (a) DSC equipment, (b) Sample holder with fresh CoRncrete sample pan and a stainless steel empty pan as reference (c) High pressure golden pan filled with fresh CoRncrete (bottom) and its cover (top).

3.2.2 Environmental Scanning Electron Microscope (ESEM) testing

ESEM is an ideal method for characterization of the microstructural geometry of a solid material. ESEM image provides 2-D information about the sample which can be considered sufficient for the purpose of formulating the hypothesis on strength development of CoRncrete. ESEM acquires an image of the sample by scanning it with a beam of electron. The emitted electron are accelerated by an electric field up to an energy of 1-30KV. By mean of the lens, the beam is focused at the surface of the sample which is kept under vacuum. The image is acquired with a backscattered electrons (BSE) detector. In BSE detector, the incident electron back-scatter by interaction with the nucleus of the atom. The number of back scattered electron increases with increasing atomic number. (Guang, 2003).

Sample preparation

The Six samples imaged under ESEM are listed in table 3.1.

Table 3.1. List of samples imaged in ESEM

Sample	Sample number	Water content [%]	Description
Corn starch	1	10.8	Water content of commercially available Maizena (Unilever, Netherlands)
CoRncrete	2	16.66	Partially heated in microwave [1.5 (thaw)]
	3		Complete heating in microwave [1.5 (thaw)+3.5(high power)]
	4		Heated in oven at 105ºC for 24h.
	5	15	DSC sample A heated in confined chamber
	6	18	DSC sample C heated in confined chamber

Commercially available corn starch (sample 1) was directly used from its pack. CoRncrete sample 2 and 3 were prepared by heating fresh CoRncrete with 16.66% water content in a microwave (Model: RFS511 MP, 2450MHz, Amana refrigeration Inc., USA). The fresh CoRncrete was prepared by mixing dry corn starch and sand in 1 to 5 ratio and then adding water. This fresh CoRncrete was mixed well in an electric mixer (Model N50CE, Hobart corporation, US). Details on mixing duration and filling the fresh CoRncrete in silicone rubber mould is given in chapter 4. Sample 2 was heated for 1.5min resulting in partial heating of the sample. Sample 3 was heated in a microwave for 5min. Sample 4 was prepared by heating fresh CoRncrete with 16.66% water content in a convection oven (Model 800, Memmert GmbH, Germany) for 24h at 105°C. Sample 5 and 6 represent the samples that were heated in DSC equipment in order to measure gelatinization temperature.

Test procedure

The CoRncrete samples were imaged under Philips XL-30 (Netherlands), ESEM at Microlab. A schematic image of ESEM is shown in figure 3.2. The images were acquired from split quadrant backscattered electrons (BSE) detector. The sample were kept in ESEM and imaged under vacuum (1 torr) at 20°C. Images at 125x and 1000x magnification were captured.

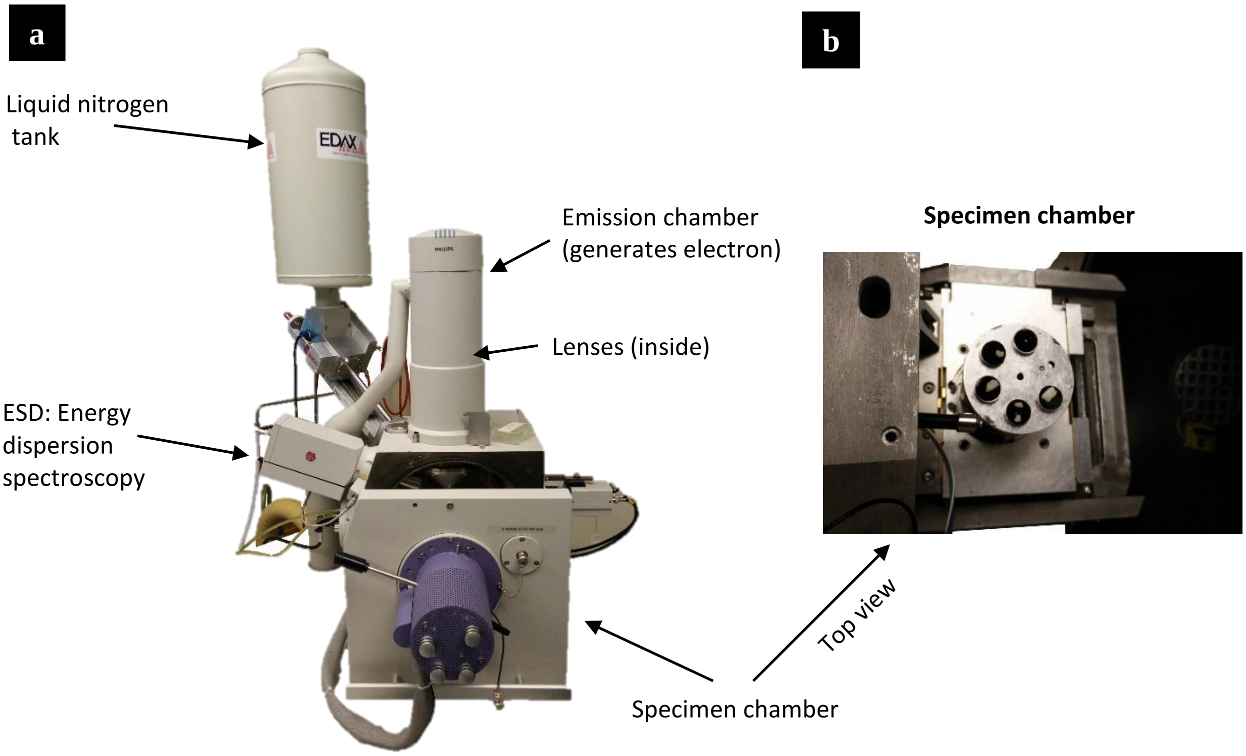


Figure 3.2. (a) Schematic image of Philip XL-30 ESEM and (b) Specimen chamber

3.3. Results and discussion

The physical transformation of CoRncrete is a heat depended modification of corn starch. Before analyzing DSC and ESEM results to formulate the hypothesis on strength development of CoRncrete, underlying heating mechanism should be understood. The heating mechanism plays an important role in the development of strength of hardened CoRncrete.

3.3.1 Heating in microwave oven

Microwave oven or Microwave is a commonly used kitchen appliance for heating and cooking food. A higher heating rate is achieved in microwave ovens in comparison to conventional ovens. Microwaves (MW) are electromagnetic waves of frequency: 300 MHz to 300 GHz. Domestic microwave appliances operate generally at a frequency of 2.45 GHz, while industrial microwave systems operate at frequencies of 915 MHz to 2.45 GHz. Microwave heating is caused by the ability of a material to absorb microwave energy and convert it into heat. The ability of a material to convert microwave energy to heat depends on its dielectric properties, represented by the dialectic constant of the material. Based on the microwave absorption, materials are classified into (i) Absorbers, which are strong absorber of microwave (water) (ii) Transparent, which allow microwave energy to pass through them with little attenuation (dry sand, silicone rubber) and (iii) Opaque which reflects the microwaves (metals). (Chandransekaran et al., 2013). The dielectric constant of water, corn starch, dry sand, saturated sand and silicone rubber at 20°C- 25°C is 80.4, 3 -5, 5, 20-30 and 3.2-9.8 respectively (Clipper controls). The dialectic constant for water decreases with increase in temperature. Therefore, during the heating process, MW absorption by water decreases.

The heating in microwave oven depends mainly on the dielectric property of a material. The presence of water causes heating due to the dipolar nature of water. "When an oscillating electric field is incident on the water molecules, the dipolar molecules try to realign in the direction of the electric field. Due to the high frequency of the electric field, this realignment occurs at a million times per second and causes internal friction of molecules resulting in the volumetric heating of the material". Other factors influencing the heating are: water content, size and geometry of microwave oven, microwave frequency, placement, density and composition of food material. (Chandransekaran et al., 2013).

The transport of water in case of porous/bio material is complex as compared to non-porous material heated in a microwave (Datta, 2007). CoRncrete is a porous biomaterial and water play an important role in heating mechanism. In case of fresh CoRncrete, solid phase consists of corn starch and sand, liquid phase consists of water and the air is present in voids. The behavior of CoRncrete is assumed to be governed by the corn starch. At low water content, corn starch particles adsorb water and exist as a solid particle. Above a certain water content (known as optimum water content or water content at phase change (explained in chapter 4)), cornstarch forms a suspension in water and exist as liquid phase (figure 3.3). The experiments supporting this statement will be elucidated in chapter 4.

Based on phase of corn starch in CoRncrete, the postulated heating mechanism in microwave can be categorized in two parts:

a) Heating mechanism where corn starch is a part of solid phase
b) Heating mechanism where corn starch is a part of liquid phase

The CoRncrete is heated in Silicone rubber mould which does not absorb microwave and is stable upto 200^{o}C

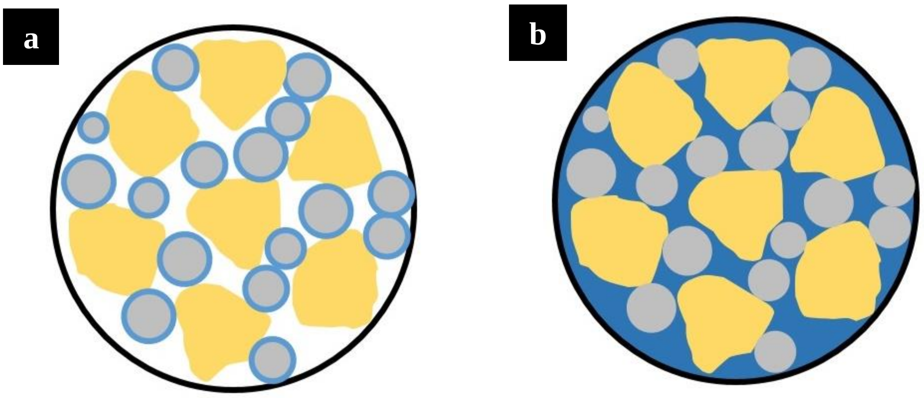


Figure 3.3.Change of phase of corn starch with the addition of water. (a) Low water content: corn starch particles adsorb water and exist as a solid particle and (b) High water content: Cornstarch forms a suspension in water and exist as the liquid phase.

(a) Heating mechanism in microwave where corn starch is a part of solid phase

The proposed heating mechanism of CoRncrete at low water content, where corn starch exists as part of the solid phase is shown in figure 3.4. Water added to CoRncrete is absorbed/adsorbed by the corn starch. This water is physically bounded to corn starch particles (corn starch is hygroscopic). Due to the unavailability of free water, CoRncrete sample allows microwaves (MW) to penetrate through it (a). MW penetrate through the sample and excite water molecules. These molecules oscillate and produce an internal friction which heats up the CoRncrete matrix (b). The temperature of CoRncrete rises and water leaves the sample in the form of vapors. According to Datta (2007), " a hygroscopic material in microwave suffers shrinkage In a porous media, liquid will be attracted or hold more tightly when there is less of it". The decrease in water content results in increased particle attraction also known as capillary suction, leading to shrinkage. CoRncrete shrinks and attains a denser configuration (c). In experiments, Shrinkage of CoRncrete is observed when it is heated with low water content. This observation supports the postulated heating mechanism.

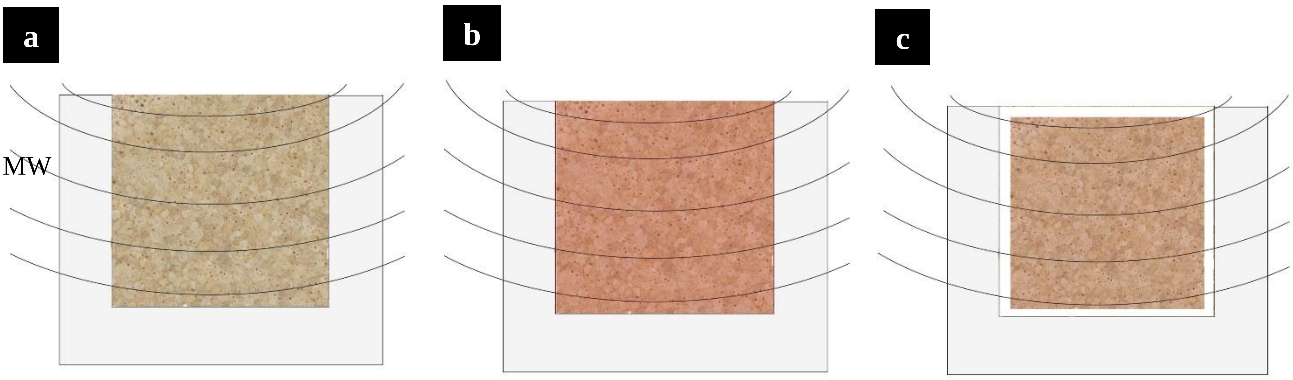


Figure 3.4. Heating mechanism in a microwave where corn starch is a part of the solid phase. (a) MW penetrates through CoRncrete sample kept in a silicone rubber mould, (b) CoRncrete Sample is heated up and (c) CoRncrete sample shrinks due to capillary suction

(b) Heating mechanism in microwave where corn starch is a part of liquid phase

The proposed heating mechanism of CoRncrete at high water content, where corn starch exists as part of the liquid phase is shown in figure 3.5.Above a certain water content, corn starch is believed to become a part of the liquid phase. Corn starch particles at this stage are suspended in water. Due to a higher water content in the sample (high dielectric constant), MW doesn't penetrate in the sample and only the surface of sample heats up (a). Due to evaporation of water, the dielectric property of material changes (dielectric constant decreases). The material becomes hotter and dryer, allowing more MW to penetrate in the sample (b) (Chandransekaran et al., 2013). According to Ni.et al. (1999), "in wet and quite impermeable material, the combination of high heating rates and low permeability is likely to generate high internal pressures, depending on the heating rates and material structure" (c). High pressures are likely to cause strong filtration flows (d). It is possible that the filtration flow in CoRncrete originates from the core and reaches the surface. The high pressure filtration flow may create cracks in the sample (e). Cracks in CoRncrete sample heated with high water content was observed during experiments, shown in chapter 4. Ni.et al. (1999) also report the moisture accumulation on the surface of a sample, which is also observed in CoRncrete immediately after it is taken out of a microwave. The coherence with literature supports the postulate on heating mechanism in a microwave with higher water content.

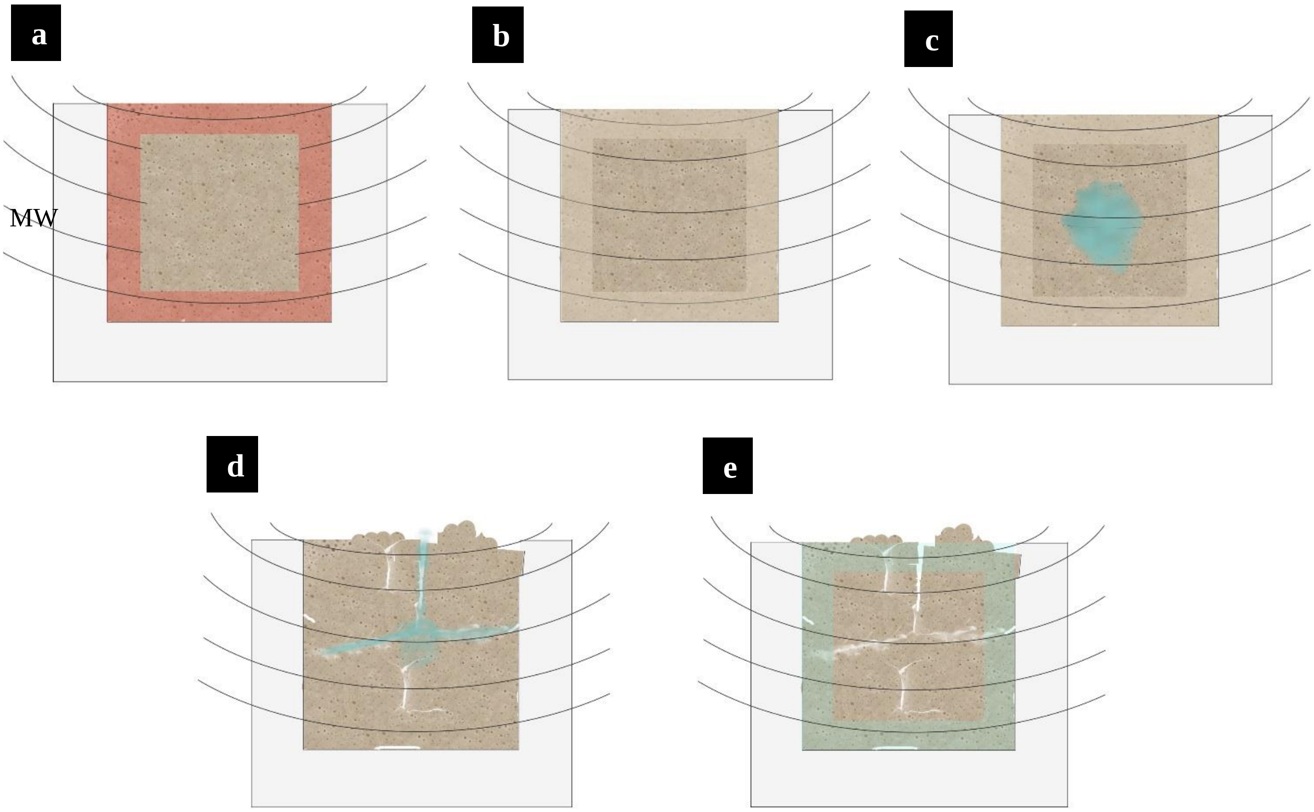


Figure 3.5. Heating mechanism in a microwave where corn starch is a part of the liquid phase. (a)High water content in sample doesn't allow MW to penetrate in and only the surface of sample heats up (b) Sample becomes hotter and dryer allowing more MW to penetrate in the sample, (c) High internal pressure is built up, (d) filtration flow caused due to high pressure and (e) sample is cracked and moisture accumulate on the surface.

3.3.2 Heating in convection oven

The oven is widely known cooking device that is also used for industrial heating of materials. A material is heated in oven by convection or radiation, or a combination of both (Mistry et al., 2006). Radiative heating in oven is based on radiative energy coming from a heat source or filament. In convection ovens, hot air is circulated with help of fans. A combination of both is believe to perform better and efficiently. The heating rate in oven is low as compared to microwave heating thus heating takes longer time.

The assumed heating mechanism in an oven is shown in figure 3.6. CoRncrete is placed (in a silicone rubber mould) in an oven (a). Silicone mould is expected to have a low thermal conductivity than CoRncrete. Hence, the heat front from the top will be faster than heat front from sides and bottom (b). The heat front is related to temperature and removal of water in a specific region of CoRncrete. As, the removal of water is high from the top region of CoRncrete, the heat induced transformation of corn starch is different in the top region of CoRncrete in compared to other regions. This makes CoRncrete sample heterogeneous. Shrinkage cracks are formed on the side faces of CoRncrete due to differential heating (c). Shrinkage cracks and heterogeneity is observed in CoRncrete specimen prepared during the experiment, hence supporting the postulated heating mechanism in a convection oven.

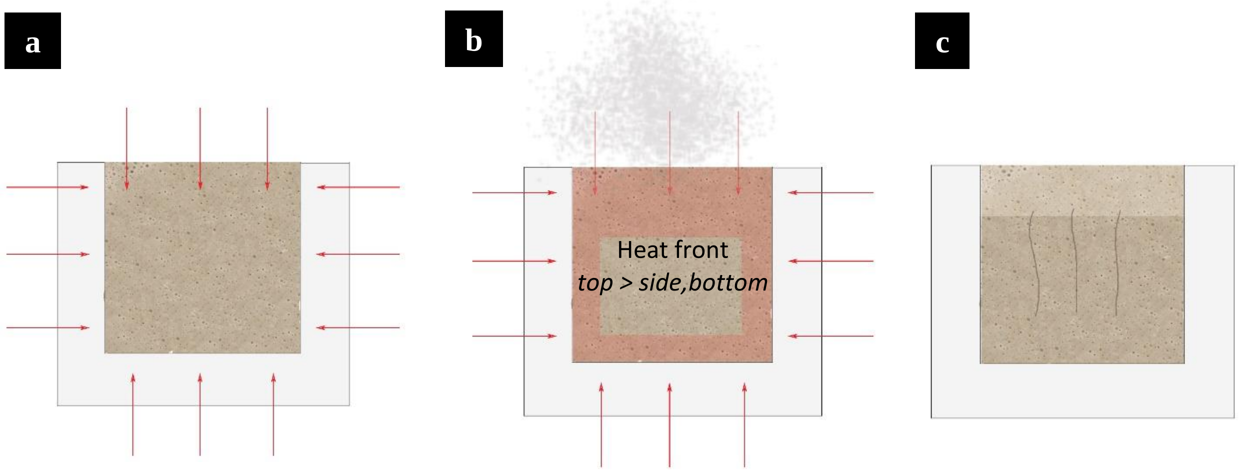


Figure 3.6. Heating mechanism in a convection oven. (a) Heating of sample from all directions, (b) Heat front at top faster than side and bottom, resulting in removal of more moisture from top region of sample and (c) transformation of top layer different than other region and shrinkage cracks are formed.

3.3.3. Transformation of CoRncrete (DSC and ESEM test results)

Transformation of CoRncrete is dependent on physical modification of corn starch. The process which is believed to play an important role in this transformation is gelatinization. “When the starch granules are heated in the presence of water, a phase transition occurs. When sufficient water is present, this transformation, called ‘gelatinization’, results in near-solubilization of the starch.” (Donavan, 1977). Gelatinization is an irreversible process that includes in time-temperature sequence, granular swelling, native crystalline melting, and molecular solubilization (Xie.et al., 2014)

DSC test result

The temperature at which gelatinization occurs is determined by heating fresh CoRncrete sample with 15% and 18% water content in DSC. The heat flow with increasing temperature is measured and shown in figure 3.7. The onset, peak and conclusion gelatinization temperatures observed are listed in table 3.2. Sample A and B with water content 15% (water/starch =0.9) , have a peak gelatinization temperature of 65.7°C and 65.5°C respectively. Sample C with water content 18% (water/starch=1.08) has peak gelatinization temperature of 66°C. The peak gelatinization temperature observed in fresh CoRncrete is lower as compared to cornstarch-water mix studied by Liu.et al., 2006 and Ratnayake and Jackson, 2006. Liu.et al., 2006 measured peak gelatinization temperature of 72.5°C for corn starch with water/starch= 0.75. Gelatinization in CoRncrete is achieved at a lower temperature potentially due to the role of sand grains in storing heat energy and transferring it to corn starch particles and water during the heating process. Factors like, type of corn starch, test equipment, sample preparation and storage, and the heating regime may also influence the peak gelatinization temperature of CoRncrete samples. A lower jump in heat flow is also observed in the case of CoRncrete as compared to starch-water mix, potentially due to the role of sand grains in storage and transfer of heat energy. A study by Liu.et al., 2006 also reveals that the peak gelatinization temperature of corn starch with water to starch ratio greater than 0.5 has a consistent values (72.2°C - 72.5°C). A consistent peak gelatinization temperature might be expected in the case of CoRncrete over 8.33% water content (or starch/water ratio 0.5). The minimum water content adopted in preparing CoRncrete is 10% (or water/starch ratio 0.6). The increase in heat influx with increasing water content is also observed in DSC curves.

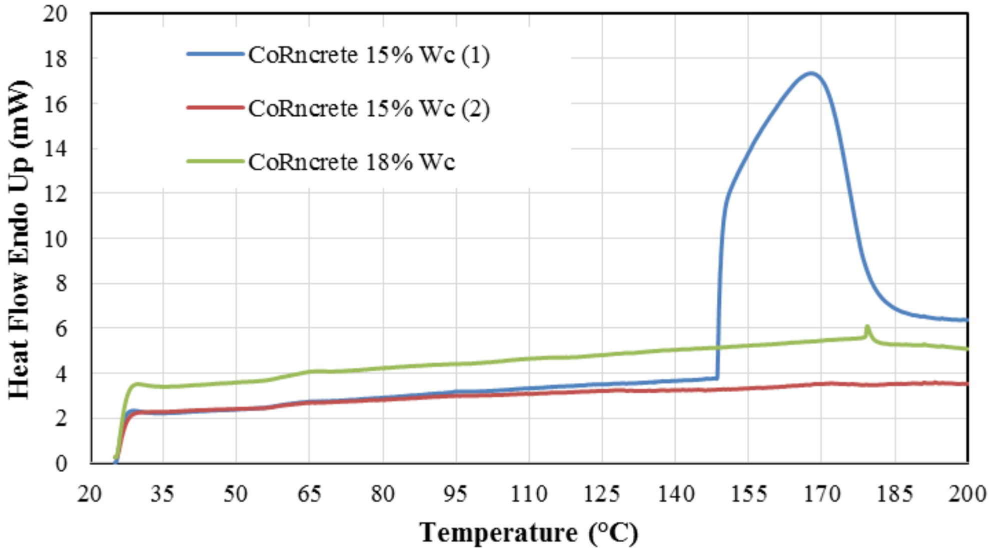


Figure 3.7. DSC curve of the fresh CoRncrete sample with different water content (Wc) heated at 5°C/min from 25°C to 200°C.

Table 3.2. The onset, peak and conclusion gelatinization temperature of fresh CoRncrete.

Sample	**Water content [%]**	**T_o[°C]**	**T_p[°C]**	**T_c[°C]**
A	15	57.3	65.7	69.93
B	15	57.2	65.5	not clearly defined
C	18	57.4	66.1	not clearly defined

In figure 3.7, a large peak in observed in sample A at 150°C. This is due to leakage of moisture from the golden pan. 76.4% water was lost while heating the sample A. A small peak around 180°C is also seen in sample C (CoRncrete with 18% water content) due to 11.9% water loss at 180°C. No peak is observed in sample B as the water loss throughout the heating process was less than 1%. It should be noted the sample B was prepared in order to verify the result of sample A. Significant water loss from sample A could have affected the measurement of gelatinization temperature.

ESEM test results

The microstructural transformation of CoRncrete was studied through ESEM. Different samples with varying extent/degree of gelatinization were imaged under ESEM. Figure 3.8 shows the ESEM image of corn starch particles at 1000x magnification. The average size of corn starch particle was measured as 15µm.

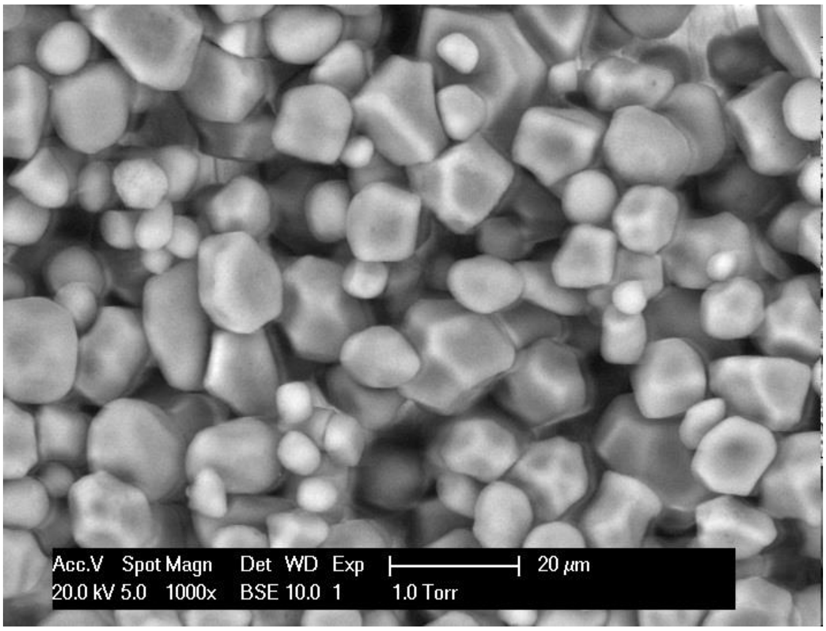


Figure 3.8. Corn starch particles (1000x). The size of corn starch particles varies from 3µm to 26 µm.

When water is added to corn starch, the particles of corn starch swells and forms a stringy network. A video on addition of water to corn starch particle generated by overlapping ESEM images (taken at 4°C under 4.3 torr vacuum) can be seen via this link or searching 'Addition of water to corn starch': https://www.youtube.com/watch?v=kdRGY9YzG9Q. The addition of water while imaging is elucidated in chapter 5.

According to Linke (1981), "when starch is heated in water, there is initially a slight swelling of the granules. Starch granular swells until a plateau is reached. Only after the plateau stage the granules become fully swollen to their maximum size and they burst. The starch is no longer granular in form but is a stringy mass. At this stage, the starch is said to be completely gelatinized. If the swelling is arrested in the plateau prior to maximum swelling or bursting of the granules, the starch is said to be partially gelatinized".

ESEM imaging was done in order to capture changes in starch granule and to analyze factor affecting gelatinization process. ESEM image of DSC sample A (Wc =15%) and sample C (Wc =18%) is shown in figure 3.9. The lighter shade represent the sand grain.

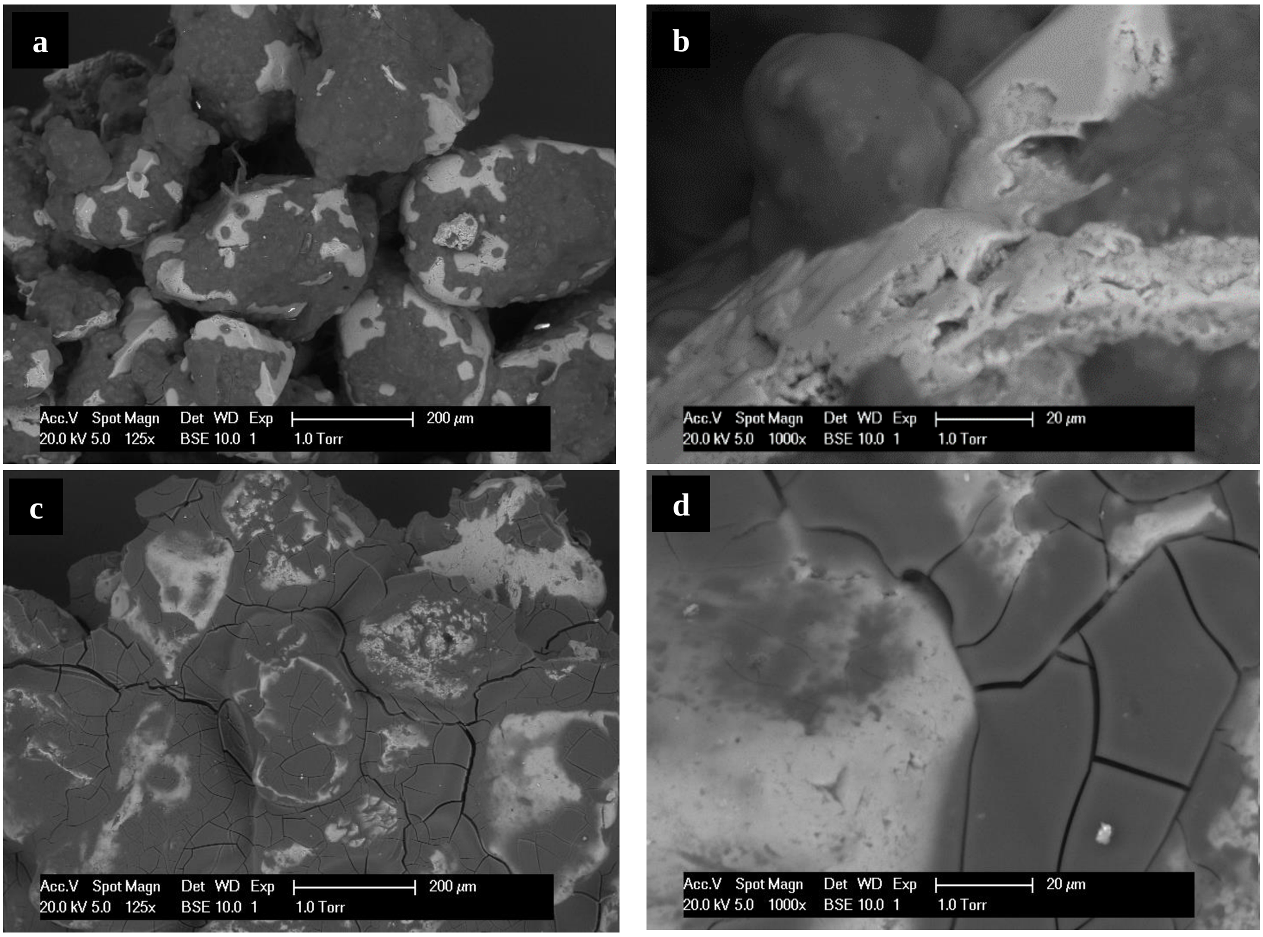


Figure 3.9. ESEM image of CoRncrete heated in DSC at (a) 15% water content (125x), (b) 15% water content (1000x), (c) 18% water content (125x) and (d) 18% water content (1000x). Effect of water content on gelatinization process and gelatinization product can be seen.

ESEM Image of DSC CoRncrete sample A with 15% water content shows the grainy texture of corn starch which may indicate partial gelatinization (a). A single grain of corn starch (25 µm) is seen at a 1000x magnification that appears to be swollen but not completely burst (b). The corn starch particle with low water content may not completely gelatinize. The swollen starch granule bonds with sand particle and forms a network of sand bonded together with partially gelatinized corn starch which act as glue. Due to the polar nature of starches, they bond with other materials (D'Amico, et al., 2010). In figure 3.9 (c), DSC sample with 18% corn starch seems completely gelatinized. The grains of corn starch are not visible. Cracks can be visualized in CoRncrete with high (18%) water content. The crack might form due to shrinkage of corn starch matrix or escape of water from CoRncrete when it was placed in a vacuum. It may be possible that excess water (if existed) didn't take part in the gelatinization process and was arrested in the voids. Later, under vacuum, it escaped. On the other hand, cracks are not observed in DSC sample A (15% Wc) possibly due to loss of 76.4% water during the heating process. DSC curve indicate leakage of water at much above gelatinization temperature, hence the gelatinization process in sample A should not be affected by moisture leakage. It is also speculated that the microstructure of CoRncrete depends on retrogradation process which occurs while cooling of the sample. The loss of moisture from sample A (image a) makes comparison with sample C (image c) difficult. Further research work is recommended on imaging the sample with different water content that are heated and cooled under same condition with minimum loss of water. Although, the comparison is not possible, but the effect of water on gelatinization process can be clearly seen. With the addition of more water, the amount of gelatinized corn starch increases. The water content may also affect the binding and power of starch glue. The quantity of gelatinized product may not be as important as the affinity of partially or fully gelatinized corn starch to the filler material. Nevertheless, Water content has shown a significant effect on gelatinization process, hence on the microstructure of hardened CoRncrete.

Figure 3.10 shows the ESEM image of partially (a,b) and fully(c,d) heated CoRncrete sample prepared with 16.66% water content. In partially heated CoRncrete, regions with different extent of gelatinization are visualized.

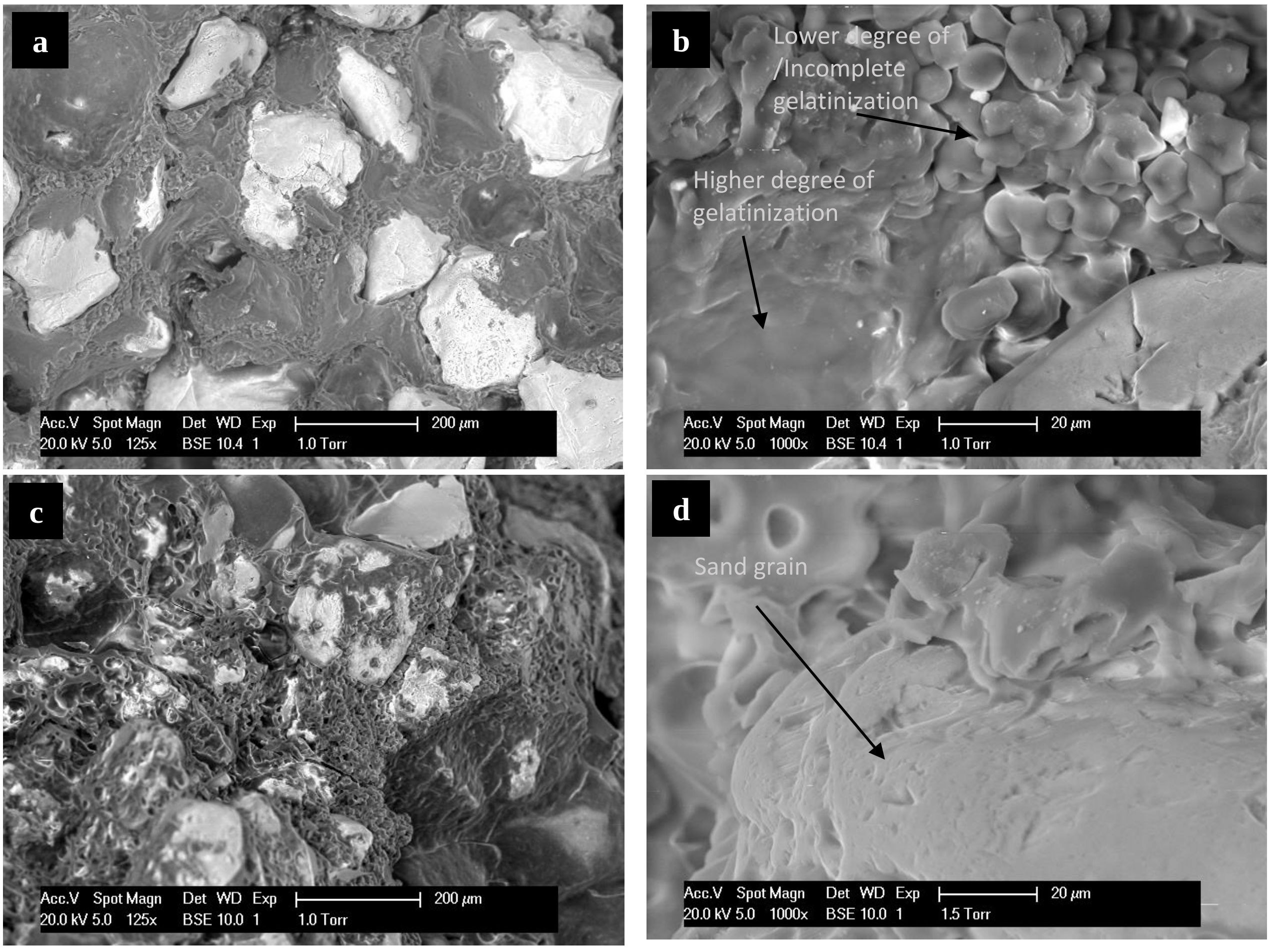


Figure 3.10. ESEM image of CoRncrete specimens heated in microwave at (a) 16.66% water content (partial heating) (125x), (b) 16.66% water content(partial heating) (1000x), (c) 16.66% water content(complete heating) (125x) and (d) 16.66% water content(complete heating) (1000x). Effect of heating duration on gelatinization process can be seen.

The degree of gelatinization seen near the edge of a sand grain is lower as compared to gelatinization seen away from the edge. The amount of water while heating and the temperature of regions may significantly affect the extent of gelatinization within CoRncrete matrix. In partially heated CoRncrete, the interface of sand and corn starch matrix is not completely formed and a gap between sand and matrix is seen. (Fig. 3.10 (a)).

In completely heated CoRncrete sample ((c), (d)), the gelatinization appears complete and no starch granules are visible. The gelatinized corn starch appears like stringy mass. In image d, the bonding of gelatinized corn starch with sand grain can be seen clearly. This bonding is expected due to the polar nature of starch. A comparison between partially and completely heated CoRncrete samples indicate the effect of heating duration on gelatinization process, extent of gelatinization and gelatinization product.

Figure 3.11 shows a CoRncrete specimen heated in an oven. Some powder type of material (the white patches) can be seen on sample (a). This might be impurity or dust accumulated in the sample due to improper storage and handling. Partial gelatinization can be seen in the sample heated in an oven. This indicates that the source or mode of heating also affects the gelatinization process significantly.

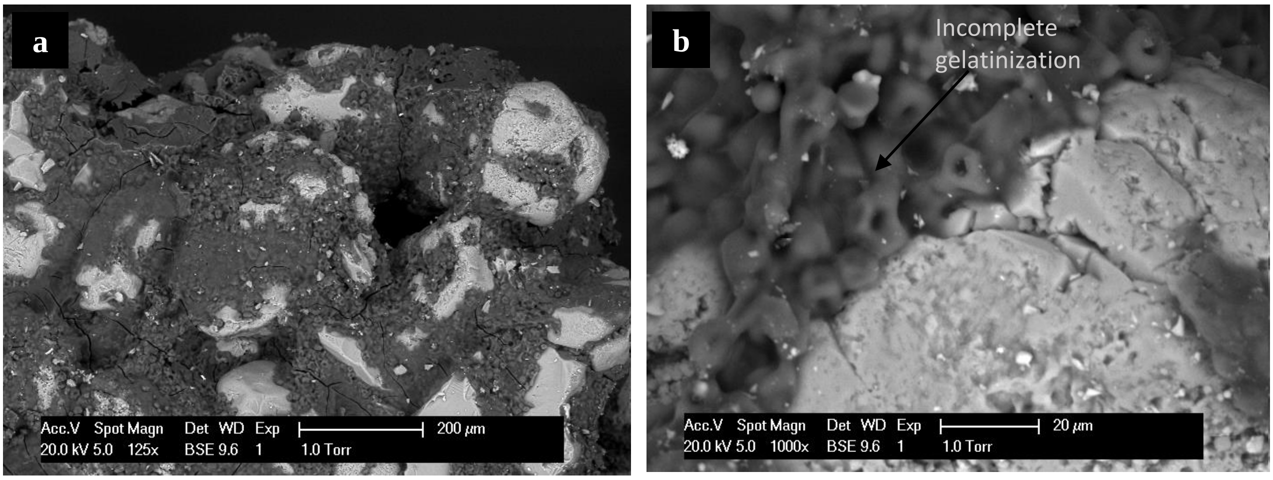


Figure 3.11. ESEM image of oven heated (24h at 105°C) specimens at (a) 16.66% water content (125x) and (b) 16.66% water content (1000x). Impurities on the sample can be seen due to improper storage and handling of sample. Effect of heating source/mode can be seen on gelatinization process.

In this research work, gelatinization process is considered to be most crucial factor that result in the transformation of CoRncrete. The binding of corn starch particles with sand in CoRncrete can be related with a study by D’Amico, et al., 2010 which focus on the use of wheat starch as an adhesive for wood. In both research work, starch act as a binder. Their research focuses on gelatinization coupled with pasting process (that follows gelatinization). In CoRncrete research, the pasting process seems to exist, but the lack of scientific evidence from experiments (on pasting property), makes it improbable to include it in this study.

Retrogradation process (crystallization of gelatinized corn starch while cooling) may also play and important role in the formation of the microstructure of CoRncrete and its strength development. Gelatinization, pasting and retrogradation, all three processes combined are believed to affect the development of microstructure of CoRncrete.

3.4. Chapter conclusion

This chapter focuses on the heating mechanism in microwave and convection oven, and formation of the microstructure of CoRncrete studied with DSC and ESEM technique.

It is postulated that above a certain water content in CoRncrete, the underlying heating mechanism in microwave oven changes which affects the shape and structure of hardened CoRncrete. The heating mechanism in microwave and oven is different due to different heating rate.

The Peak gelatinization temperature measured for CoRncrete sample with 15% and with 18% water content is 65.6°C and 66.1°C respectively. The peak gelatinization temperature of CoRncrete is lower as compared to that of corn starch with similar water content. The data of corn starch gelatinization is derived from literature.

The heat induced transformation of CoRncrete is potentially caused due to the gelatinization of corn starch. In the presence of water, starch granules swells, crystalline region melts and viscous gel is formed. The ESEM scan indicated an influence of water content, heating source (heating rate) and duration of heating on gelatinization of the corn starch, thus on microstructure and strength development of CoRncrete. CoRncrete heated at low water content (15%) seems to partially gelatinize as compared with fully gelatinized corn starch matrix in higher water content CoRncrete (16.66%). In partially heated CoRncrete, regions with different extent of gelatinization are visualized. The degree of gelatinization seen near the edge of sand grain is lower as compared to gelatinization seen away from the edge. Also, the interface of sand and corn starch matrix is not completely formed in CoRncrete due to partial heating resulting in a low degree of gelatinization. A comparison between partially and completely heated CoRncrete samples indicate the effect of heating duration on gelatinization process and bonding between corn starch matrix and sand. Partial gelatinization can also be seen in the sample heated in an oven which indicate the effect of heating source on gelatinization process.

4

Strength of CoRncrete

4.1. Introduction

The transformation of CoRncrete from a semi-solid material to a hardened material is caused by heating that results in physical modification of starch. The heat-induced transformation of CoRncrete is potentially caused due to the gelatinization of corn starch. The ESEM scan in previous chapter indicated an influence of water content, heating source (heating rate) and duration of heating on gelatinization of corn starch that affects the microstructure and strength development of CoRncrete. This transformation is sensitive to the water content in fresh CoRncrete. The water content in fresh CoRncrete also influences the heating mechanism. Water content where the phase of corn starch changes from solid particle to suspension in water, is indicated by performing cone penetrometer test. The cone penetrometer test is carried out on the sand, corn starch and fresh CoRncrete. Although a quick test, the compaction of the sample for cone penetrometer test is not quantifiable, resulting in non-reliability of results. In order to support or confirm the observations of phase change in cone penetrometer test, proctor test was carried out on fresh CoRncrete. The result of fresh CoRncrete was compared with sand. Proctor test show relation between water content and dry density, giving further insight on behavior and consistency of CoRncrete.

While, the effect of water content and duration of heating in the microwave were considered important for strength development (through ESEM results), the effect of sand grain size was considered significant based on preliminary study result. Thus, specimens of 40×40×40mm were prepared in silicone rubber mould and heated in microwave and oven. The specimen were tested in triplicate in order to study the effect of aforementioned parameters on compressive strength of CoRncrete.

4.2. Materials and method

4.2.1. Cone penetrometer test

Cone penetration is a liquid limit test carried generally on clay and silty soils to determine the water content at which soil start behaving as a liquid. This test also indicates the consistency and the undrained shear strength of soil. The liquid limit values are often used in the classification of soils. The liquid limit, for cone penetrometer test, is defined as the water content in soil where a penetration of more than 20mm is measured. Cone penetrometer test is designed for the fine-grained soil like clay and silt. CoRncrete behaves quite differently from these soil. In case of fine-grained soil, the cone penetration stops within few second but in CoRncrete a dynamic penetration is expected due to its shear thickening behavior.

Cone penetrometer test was performed on sand (Source: river), corn starch (Duryea Maizena, Unilever, Netherlands) and fresh CoRncrete (1 corn starch: 5 sand), based on recommendation in BS 1377 part 2 :1998. The test equipment consists of cone penetrometer with a standard cone of mass 80g, and a sample cup of internal diameter 55mm and 40mm height (capacity: 100ml). The test equipment is shown in figure 4.4 (a).

Sample preparation and test procedure

The sample were prepared by adding water in an increment of 3.3% (of the initial mass of material) for each trail. In case of corn starch an equivalent (20%) amount of water was added to ensure similar water to corn starch ratio, as in CoRncrete. The sample was poured in a metal bowl and weighted. The sample was taken to the mixer (Model: KMM760, Kenwood, UK) and mixed for 2 minutes at 50 RPM. After completion of mixing, the bowl was taken to cone penetrometer equipment. The sample cup was filled with material in 3 layers, each tamped 10 times with silicone rubber capped steel rod (Figure 4.4 (b)). Excess material was trimmed off with a spatula to form an even surface with no trapped air bubbles. The sample cup was placed on designated spot on cone penetrometer equipment. The point of the cone was brought to the surface of sample. The rack was lowered down to bring it in contact with the top of the cone. The dial gauge was set to 0mm (penetration). The cone was released by pressing the release button. The reading on dial gauge was taken to the nearest of 0.1mm at 5, 10, 20 and 30 sec. The cone was lifted and cleaned carefully. Approximately 30-50g sample from the middle of sample cup was taken in glass crucible for determination of water content in the sample. The water content was determined by calculating loss in mass of the sample (kept in crucible) when it was heated in an oven at 105°C for 24h. The remainder of the material was added to metal bowl and taken to the mixing machine. The whole process of mixing and cone penetration was repeated. The increment of water was continued until the material became too liquid that the complete (40mm) penetration was achieved within 5sec.

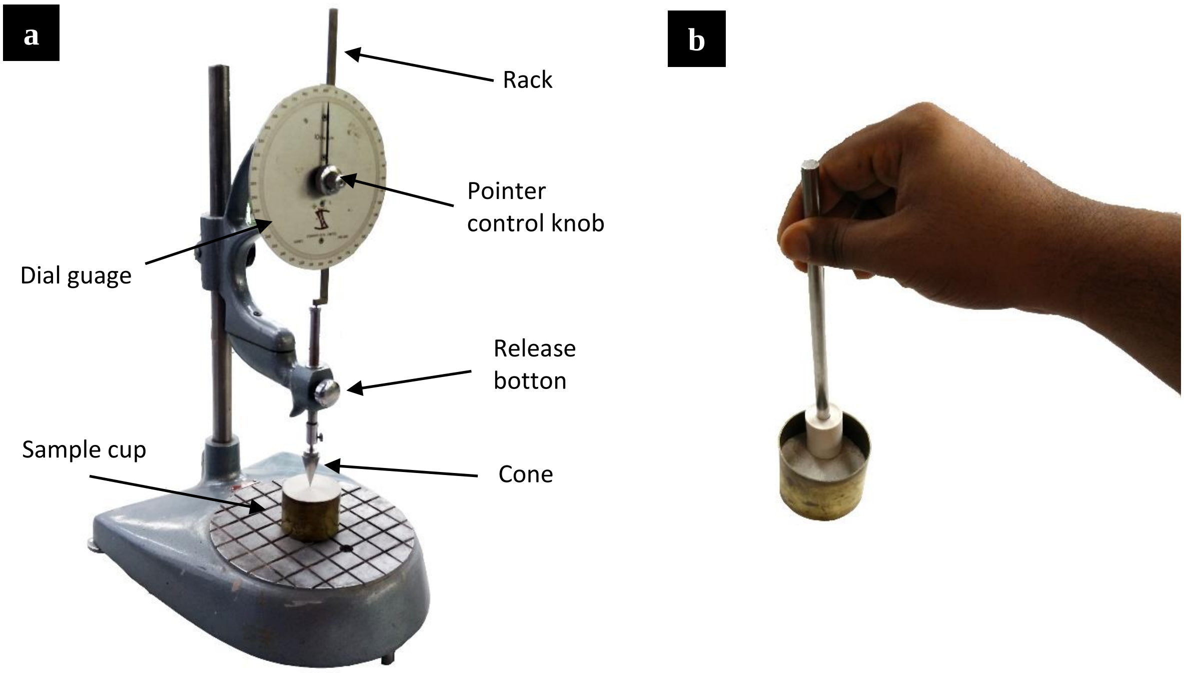


Figure 4.4 Cone penetrometer with sample cup. (a) Schematic image of Cone penetrometer and (b) Compacting the sample with silicone rubber capped steel rod

4.2.2. Proctor test on fresh CoRncrete

Proctor test is a compaction test developed by Ralph R Proctor in 1933. This test is used extensively in the field of geotechnical engineering to determine dry density- water content relationship for geo-materials such as clay, silt and sand. In this test, the soil particles are packed more closely together usually by mechanical means, thereby increasing the dry density of soil. The dry density depends on the degree of compaction applied and the amount of water present in the soil. For a soil compacted at given energy, there is an optimum water content at which the dry density reaches a maximum value. Similarly, an optimum water content for fresh CoRncrete might exist which may/may not result in highest strength of hardened CoRncrete.

A compaction mould (figure 4.5) was used in the experiment as per the guidelines of BS 1377:part4:1990. This cylindrical compaction mould of capacity 1 Liter has an internal diameter of 105mm and internal effective height of 115.5mm. The mould has a detachable base plate and a removable extension collar. Two rammers were used in the experiment: a metal rammer and a plastic rammer of 350gm and 150gm respectively. A guide tube was used to drop rammer vertically from a height of 200mm on a layer of fresh CoRncrete (fig. 4.5. (b)). The guide tube has an internal diameter of 52mm and height of 350mm. The two rammers used in the experiment corresponds to different compaction energies. The compaction energy of falling rammer is dependent on the weight of rammer, height of drop, the number of layers, the number of drop per layer, mould dimension and mould volume. The compaction energy for 350 gm and 150 gm rammer were calculated as 51.5 KJ/m3 and 22.1 KJ/m3 respectively. Refer appendix C for calculation of compaction energy.

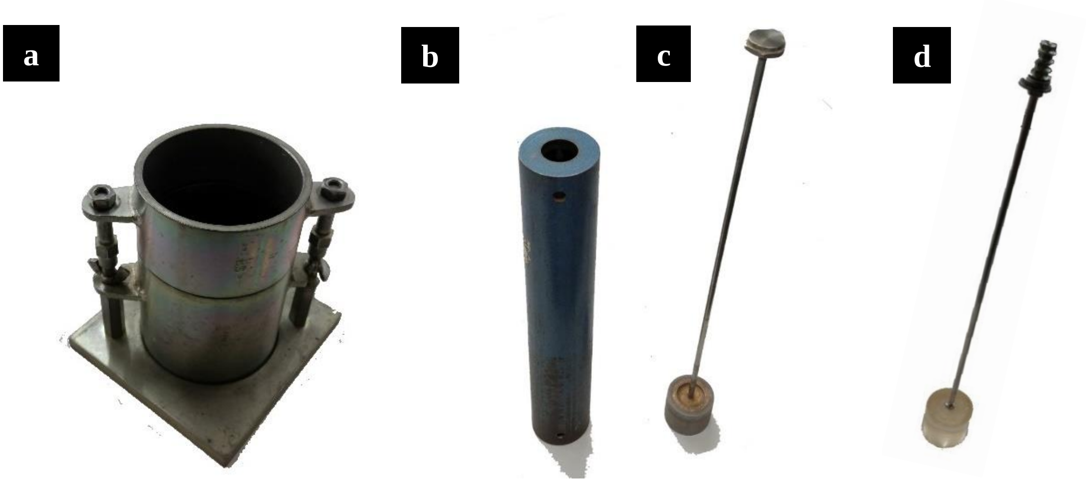


Figure 4.5. Apparatus used in proctor test- (a) Compaction mould, (b) guide tube for controlling height of drop of rammer to 200mm, (c) 350gm metal rammer (Energy: 51.5 KJ/m3) and (d) 150gm plastic rammer (Energy: 22.1 KJ/m3)

Sample preparation

The fresh CoRncrete sample for proctor test was prepared by mixing 375gm oven dried (105 ^{0}C for 24 hours) cornstarch and 1875gm 0.125mm-0.25mm sand fraction (kept at 20 ^{0}C, measured moisture content: 0.03%). Thus, the proportion of cornstarch to sand was kept 1:5. The sample was mixed well for 2 minutes at low speed in a mixer (Model: KMM760, 1200W, Kenwood, UK). The water was added in an increment of 3.3% after each test. The increment of water was changed to 1.5% when a sudden jump in bulk density was observed. The sand was chosen as a reference material for comparison with fresh CoRncrete. 2250 gm 0.125mm-0.25mm sand fraction (kept at 20 ^{0}C, measured moisture content: 0.03%) was taken and similar process of adding water in increment of 3.3% was adopted as in case of fresh CoRncrete

Test procedure

The initial weight of compaction mould (without extension collar) was taken. Extension collar was attached to the mould. Loose fresh concrete was added in mould. The guide tube was kept on it gently and held vertically. The rammer was dropped 25 times, distributed uniformly over the whole area to compact the layer. A second and third approximately equal layer was placed and compacted similarly. The compacted surface in the extension collar was about 6 mm above the level of the mould body. The extension collar was removed carefully and the excess fresh CoRncrete was cut away. The top of the mould was levelled off. The weight of fresh CoRncrete with mould was measured. The sample was broken on a tray with the help of chisel. A representative sample (about 50gm) was taken from the center in a glass crucible for determination of water content by drying sample at 105^{0}C in an oven for 24 hours. The leftover of compacted sample was mixed with the remainder of prepared sample. The water was added in an increment of 3.3% after each test. A similar test with different compaction energy was performed on fresh CoRncrete to compare the results. Proctor test was also performed on the sand as a reference material. The test on sand was also executed with two different compaction energies same as the one used for fresh CoRncrete.

The dry density (ρdry) of fresh CoRncrete or sand can be calculated from the density (ρ) and water content (wc) of sample using the relation:

$$\rho\mathrm{dry} = \frac{\rho}{1+\mathrm{wc}} * 100 \qquad (1)$$

Where water content (wc) is calculated by measuring loss of water (m_w) in oven drying and mass of sample/solid (ms) after oven drying from the relation:

$$\mathrm{wc} = \frac{\mathrm{mw}}{\mathrm{ms}} * 100 \qquad (2)$$

4.2.3. Preparation of silicone rubber mould

CoRncrete specimens of size 40mm×40mm×40mm were casted in silicone rubber mould. Silicone rubber can withstand temperature upto 200ºC (Poly-service website). Silicone rubber has a low dielectric constant, hence they allow the microwave to pass through them with little or no energy absorption. Silicone rubber is flexible and easy to cast in desired shape. This makes silicone rubber ideal choice for mould which has to withstand high temperature in microwave and oven. Other materials such as PVC and wood were tested but performed unsatisfactorily in a microwave. They were burnt and distorted when heated for prolong duration (> 5min).

The silicone rubber moulds were prepared in Microlab, M&E, CiTG . The steps followed for preparation of silicone mould are:

Step 1. Preparation of moulding setup

Wooden master moulds were selected to cast silicone rubber mould. Two rectangular wooden master mould of thickness 175mm and internal dimension 19mm×7mm×5.5mm were prepared. This master mould consisted of five wooden elements which were assembled together. Prefabricated PVC block of size 40mm×40mm×40mm(cubic) and 160mm×40mm×40mm (prismatic) were taken and attached to a wooden beam in the configuration shown in figure 4.1 (a). The blocks attached to the wooden beam were placed over the master mould such that space for mould (silicone rubber) wall of 15mm was formed all around PVC blocks. The surface of PVC block should be smooth and free of impurities. The master moulds (with PVC blocks) were kept on a straight surface. Unused silicone rubber cylindrical mould kept in Microlab were taken and chopped off in small pieces and filled in the master mould as shown in figure 4.1 (b),(c). This was done to save fresh silicone rubber required for preparing moulds for CoRncrete. Two moulds, one with space for 3 cubic CoRncrete specimen and other with space for a prismatic CoRncrete were casted simultaneously.

Figure 4.1. Preparation of moulding setup. (a) Image of PVC block attached with wooden beam (225mm×15mm×17.5mm) kept in the wooden master mould. The wooden master mould consist of 5 elements: 1- 225mm×105mm×17.5mm, 2 - 225mm×15mm×17.5mm and 2 - 105mm×55mm×17.5mm, (b) Silicone rubber chopped in small pieces and (c) chunks of silicone rubber filled in the master mould.

Step 2. Preparation of silicone rubber mix

Silicone rubber is commercially available in two components that have to be mixed before use. The two components were silicone rubber (Siliconen rubber PS 81020, POLY-SERVICE B.V, Netherlands) and hardener (Harder, PS 81020, POLY-SERVICE B.V, Netherlands) as shown in figure 4.2 (a). Silicone rubber and hardener were mixed in 20:1 proportion (by weight) as recommended by the manufacturer. The mixing was done manually with wooden Popsicle stick at room temperature 20°C. The components were stirred until they appear to mix thoroughly (figure 4.2 (b)). The mixing process was completed in 5min. Prepared silicone rubber was poured in a wooden master mould and left for 24h to harden (figure 4.2 (c),(d)). It should be noted that the silicone rubber start setting in 90mins.

Figure 4.2. Preparation of silicone rubber mix. (a)Silicone rubber and hardener, (b) Mixing silicone rubber and hardener, (c) Pouring silicone rubber in master mould and (d) Silicone rubber left to harden for 24h.

Step 3. Hardening of silicone rubber mould

After 24h of curing, the surface of silicone rubber was inspected and it was found to be hardened (figure 4.3 (a)). The master mould was demolded with the help of hand drill (figure 4.3 (b)). The wooden elements were removed one by one (figure 4.3 (c)). The silicone rubber moulds were removed from their respective master mould. Finally, PVC blocks were pulled out. The silicone rubber moulds were ready to use (figure 4.3 (d)). 4 silicone rubber moulds with space for 3 cubic specimens each, and 3 silicone rubber moulds with space for 1 prismatic specimen were casted, to be used in the preparation of hardened CoRncrete.

Figure 4.3. The final step for preparation of silicone rubber mould (a) Hardened silicone rubber after 24h curing at 20ºC, (b) (c) removal of wooden element in order to extract silicone rubber mould (d) Ready to use Silicone rubber mould.

4.2.4. Compressive testing

The specimens of 40×40×40mm were prepared to study the effect of water content, size of sand grain and duration of heating (in a microwave) on compressive strength of CoRncrete. The composition of samples was different for each set of testing. To study the effect of water content on CoRncrete, the water content in fresh/wet CoRncrete sample was gradually increased from 10% to 20%. A total of 12 (6 microwave+6oven) samples were prepared with varying water content. To study the effect of sand grain size on strength, 4 samples were prepared with varying sand fraction: 0.125-0.25mm, 0.25-0.50mm, 0.50-1.0mm and 1-2mm. To study the effect of duration of heating on the strength of CoRncrete in a microwave, 5 samples with a same composition were prepared. The duration (in combination with mode) of heating was different for each sample. 3 cubical specimen for each sample were prepared and tested in order to determine average compressive strength of the material.

Sample preparation

Corn starch and sand were weighted on a digital scale and poured in a metal bowl in 1:5 ratio. They were mixed together manually for 30s. This was done to prevent blowing of corn starch due to its low density while actual machine mixing. The mixing was done based on NEN-EN 196-1:2005 with some modifications. The bowl was fitted in an electric mixer (Model N50CE, Hobart corporation, US) and water was added. As soon as water was in contact with the mix of corn starch and sand, the machine was started and time was recorded. The mixing was continued for 90s at low speed (140 rpm). It should be noted that the mixing at high speed was not performed (which was a recommendation in NEN-EN 196-1:2005) to avoid damage to the machine due to shear thickening property of corn starch with water. The machine was stopped for next 60s and corn starch stuck at the bottom of the bowl was scraped out with hand and poured to the top of sample. The mixing was continued for final 60s. The bowl was detached. Silicone rubber moulds were already dried and cleaned. Fresh CoRncrete was placed in the mould in three layers. Each layer was tamped 10-15 times with a silicone rubber capped steel rod as shown in figure 4.6 (a). The extra material was trimmed off and the samples were taken immediately to a microwave and an oven.

In case of microwave heating method, the sample was placed inside a microwave (Model: RFS511 MP, 2450MHz, Amana refrigeration Inc., USA) and a surcharge of 2kg (sand in silicone mould as shown in figure 4.6 (b)) was placed over it. The surcharge was placed to prevent popping up of CoRncrete while heating. Silicone rubber and dry sand, both have low dielectric constant hence they are assumed not to affect the heating process of CoRncrete significantly. The specimens were heated for 5min, first 1.5 min in thaw mode (19.8KJ energy) and next 3.5 min in high power mode (231KJ energy). This heating scheme was selected based on trails in the preliminary study.

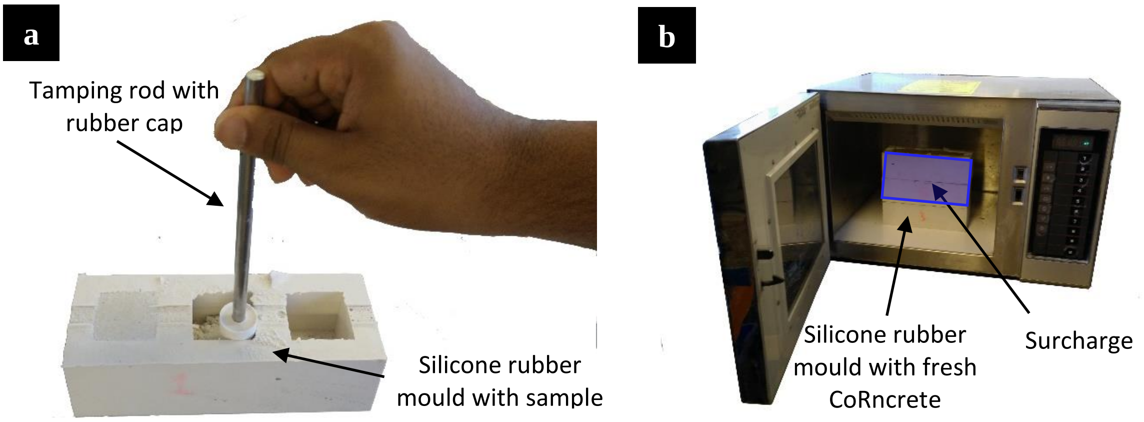


Figure 4.6. Preparation of sample in silicone rubber mould and heating. (a) Filling the mould in 3 layers and compacting each layer with silicone rubber capped steel rod and (b)Mould kept in microwave with surcharge

In oven heating method, the specimens were heated at 105°C for 24h in an industrial oven (Model 800, Memmert GmbH, Germany). No surcharge was placed over the specimens. Once the heating of specimen was finished, the mould was taken out. The specimens were detached and kept at room temperature (20 °C) to cool. After 30 minutes, the specimens were sealed in a plastic bag to prevent further interaction with the environment. After 24h, the specimens were taken out and their dimensions were measured with a digital caliper to the nearest of 0.01 mm.

Test procedure

The specimens were tested in a compression testing machine as shown in figure 4.7 (Model: E161PN114, Matest, Italy) with loading parameter as recommended in NEN-EN 196-1:2005. The loading parameter were:-Loading rate=2.4kN/sec, start Load=0.1kN and stop load=25%.

Figure 4.7. Strength testing machine generally used for concrete specimens

4.3. Result and discussion

4.3.1. Water content at phase change

Cone penetrometer test was performed in order to determine the water content where the phase of corn starch particle in fresh CoRncrete changes from solid to liquid. The test also indicate the undrained shear strength of CoRncrete and cornstarch giving insight about the behavior or consistency of fresh CoRncrete. The result of cone penetrometer test on the sand, corn starch and CoRncrete is shown in figure 4.8. The undrained shear strength is calculated based on the recommendation in Geotechnical investigation and testing, NPR-CEN-ISO/TS 17892-6. The complete experimental data with undrained shear strength values in given in appendix C. It is important to note that a low cone penetration indicate a higher undrained shear strength of the material.

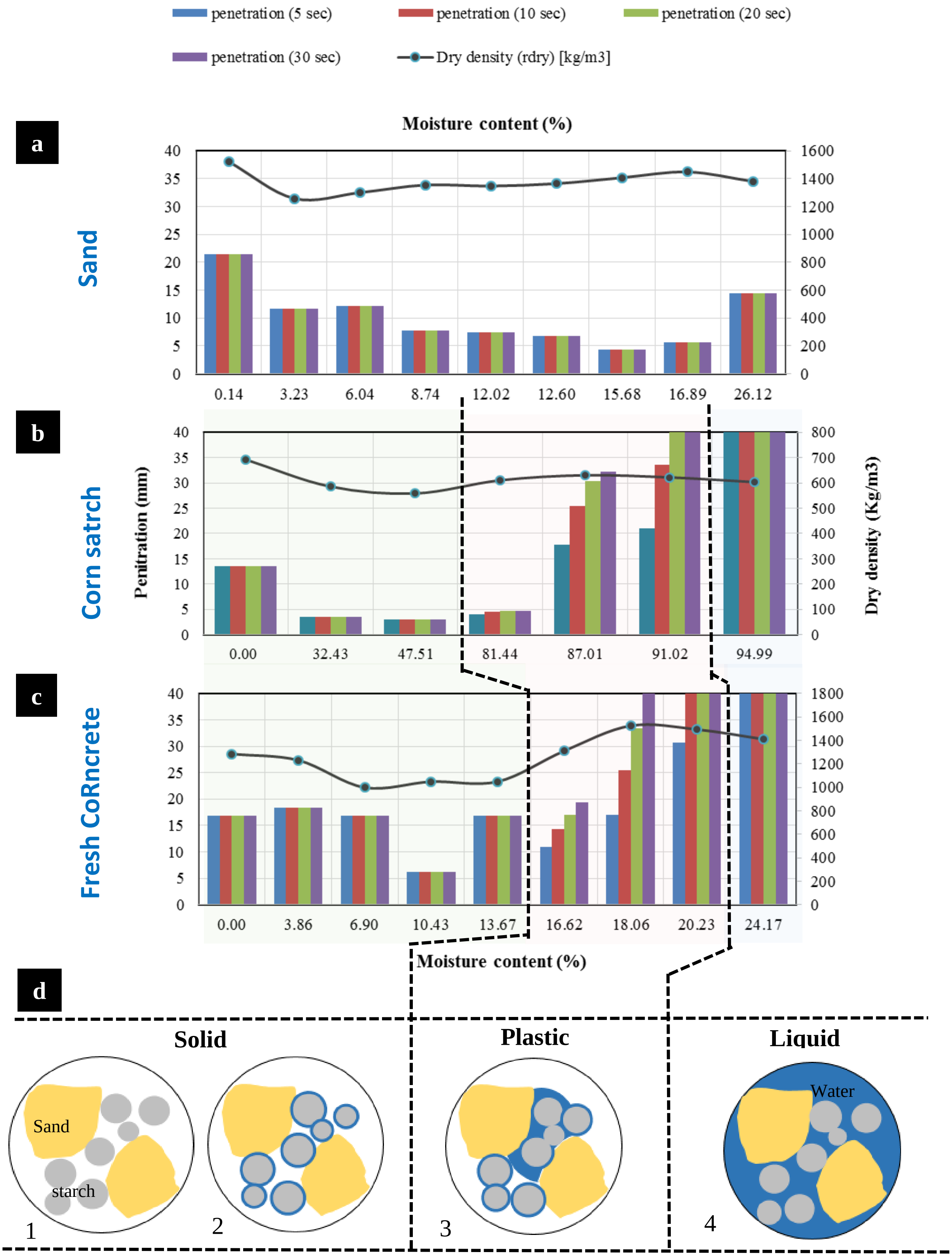


Figure 4.8. Cone penetrometer test result of (a) sand, (b) corn starch and (c) fresh CoRncrete. The phase of corn starch sketches is shown in (d).

According to the recommendation in BS 1377 part 2:1998, the water content at which the penetration recorded is greater than 20 mm should be considered as a liquid limit (material start behaving as liquid).

This is valid for fine grain soil but not for sand. This is evident by the penetration recorded in the dry sand which exceed 20mm (a). The sand particles are cohesion less thus allows cone to penetrate until the surface friction of sand overcomes that of the cone. With the addition of water, the penetration reduces due to increasing undrained shear strength of sand. The increase in undrain shear strength of sand is potentially due to the presence of incompressible water in between grains. The dry density increases with increasing water content. When the sand is fully saturated with water, the dry density decreases. No dynamic penetration is observed in the case of sand.

Corn starch is a fined grained material. A lower undrained strength (3.4kPa.) or a higher penetration is measured in dry corn starch (b). With an increase in water content upto 47.5%, starch particles may swell and behave as cohesive material, thus resisting the impact of falling cone, resulting in very low penetration. At this stage lumps of corn starch particles are formed resulting in increase in air voids, thus lower dry density. No dynamic penetration is visible. From water content 81.44% to 91%, a rapid decrease in undrained shear strength is observed. This indicates that a phase transition might have occurred in this range of water. The dynamic penetration in corn starch indicates its plastic behavior in this range of water content. The penetration at 87.04% water content crosses 20mm, indicating a potential change in phase of corn starch particle from solid to suspension. In the study carried out by Crawford.et al. (2013), the corn starch achieve its maximum thickening stress at 90.5% water content. At 90.5%, the dense cluster of corn starch is formed resulting in high viscosity. Their results are in a coherence of the observation at similar water content 87.04%, where formation of corn starch particle dense cluster in water is indicated by a higher dry density value. With further increase in water content upto 95%, the dense cluster of corn starch loosen up and thinning is observed resulting in full cone penetration or zero shear strength of the water-corn starch mix.

Fresh CoRncrete is a mix of corn starch and sand (1:5). Dry corn starch has no cohesive force, thus starch particles are free to move relative to sand (d1). An undrained shear strength (2.2kPa) value, higher than sand but lower than cornstarch, is observed. With increasing water content from 0 to 6.9%, no significant difference in cone penetration or undrained shear strength is noticed, possibly due to absorption of water and resultant swelling of corn starch particles. Corn starch particles are still free to move with respect to each other and sand. When water is increased from 6.9% to 10.3%, a rise in undrained shear strength is observed. The corn starch particles at this stage may start behaving as cohesive mass (d2). They may be glued to sand particles and form a rigid network of corn starch particles in between sand grains, allowing CoRncrete to resist shear force. The reading at 13.67% is inconsistent with the current hypothesis and is unexplainable. At 16.62% water content, a dynamic penetration is observed, indicating a possibility of phase change of corn starch in fresh CoRncrete (d3). At this water content, some corn starch particle may start forming a dense cluster, indicated by rise in dry density. As corn starch particles are glued to sand, sand also becomes part of this cluster. With further increase in water content to 18.06%, the dynamic penetration at 30 sec results in complete penetration or zero undrained shear strength, confirming the liquid phase of corn starch. A higher dry density is also observed at this water content, further indicating the formation of the dense cluster with all corn starch particle involved. Above this water content, the dense cluster is loosened up (d3) and decrease in dry density is observed. The undrained shear strength value approaches zero.

Figure 4.8 does indicate a similarity between the behavior of cornstarch and fresh CoRncrete. Corn starch plays a major role in governing the behavior of fresh CoRncrete. The range of water content from 16.62%

to 18.06% indicate a dynamic penetration and plastic behavior, which could be an indication of phase change of corn starch. The cone penetrometer test is an indicative test with a higher chance of manual error due to uneven compaction of layers. To capture the water content at phase change, proctor test may indicate better result due to quantifiable compaction. It may also support the results and hypothesis formed on the behavior of fresh CoRncrete based on cone penetrometer test. Phase change is potentially marked by the formation of a dense cluster, hence higher dry density. It might be possible that the optimum water content achieved through proctor test at maximum dry density falls within the range of 16.62% to 18.06% water content.

4.3.2. Relation between maximum dry density and moisture content

The proctor test was performed on fresh CoRncrete and sand. Each material was compacted with compaction energy of 51.5 KJ/m3 and 22.1 KJ/m3. A total of 4 test series were carried out. The results are presented in table 4.1. Fresh CoRncrete show an unusual trend of water content – dry density relationship as compared to sand.

The proctor test shows that the compaction energy has no significant affected on the density of sand in the dry state. A significant difference in dry density of sand (with moisture) at different compaction energy is observed. The general trend of compaction curve for sand is similar for both compaction energy (figure 4.9). The higher compaction energy results in a more compacted sand, hence higher dry density. At saturation or at the point where excess water can be seen in the sample, the sand is unable to compact further. This is caused due to excessive water in the sample which is incompressible. The energy is transferred from rammer to incompressible water resulting in no further compaction. The dry density for sand compacted with different energies reaches a similar value at saturation. No clear optimum water content can be determined from the data. The variation in dry density of sand is low and it is difficult to define a clear peak. The low variation resulting in unclear peak can be due to lower energies used in this test.

Table 4.1. Results of proctor compaction test on fresh CoRncrete and sand. Two rammers representing two different compaction energies were used in the test.

Fresh CoRncrete - 150gm rammer		*Fresh CoRncrete - 350gm rammer*		*Sand - 150gm rammer*		*Sand - 350gm rammer*	
Water content (%)	**Dry density (ρ_{dry}) [kg/m3]**	**Water content (%)**	**Dry density (ρ_{dry}) [kg/m3]**	**Water content (%)**	**Dry density (ρ_{dry}) [kg/m3]**	**Water content (%)**	**Dry density (ρ_{dry}) [kg/m3]**
0.00	1550	**0.00**	1589	**0.00**	1557	**0.10**	1568
2.77	1375	**2.75**	1440	**3.22**	1407	**2.88**	1491
6.15	1271	**5.70**	1344	**6.81**	1415	**6.26**	1504
9.26	1223	**8.95**	1351	**9.60**	1397	**9.80**	1479
13.00	1189	**12.58**	1285	**13.36**	1403	**13.35**	1495
16.64	1589	**16.16**	1470	**18.95**	1402	**18.01**	1487
18.11	1554	**17.94**	1547	**19.86**	1394	**19.03**	1500
19.46	1532	**19.40**	1529	**22.66**	1435	**21.22**	1487
22.36	1447	**20.86**	1534	**22.98**	1506	**22.03**	1514

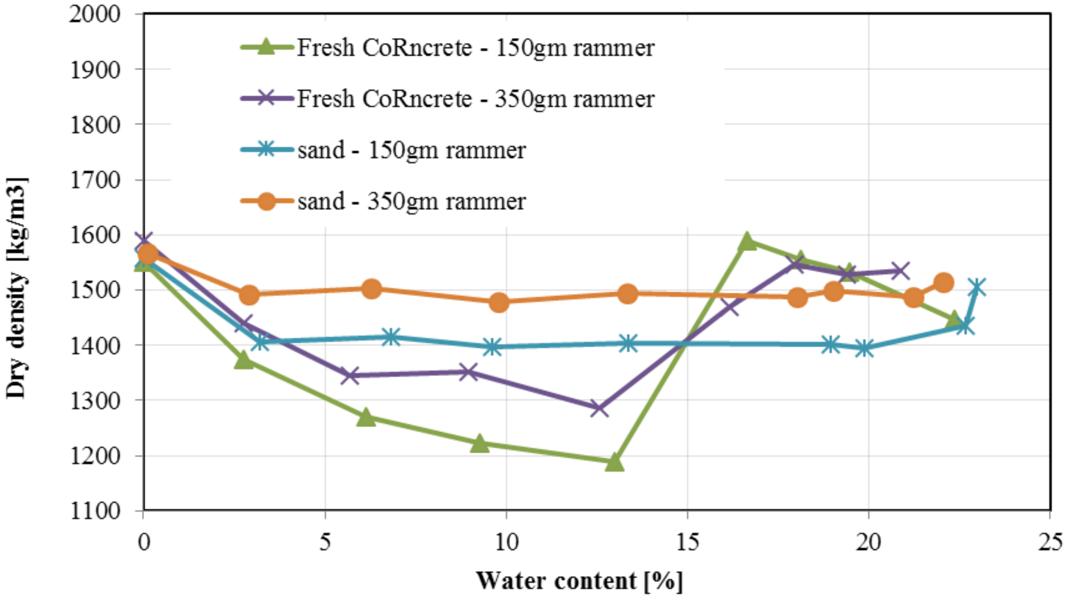


Figure 4.9. The plot of dry density versus water content in percentage. The comparison between wet CoRncrete and sand compacted with 2 different energies are shown. The wet CoRncrete showed an unusual behavior with comparison to sand or any geo material.

In case of fresh CoRncrete, the compaction achieved by both rammers (energies) shows a similar trend. The dry density initially decreases with increasing water content (fig. 4.9). The decrease in dry density might be caused due to the formation of lumps (figure 4.10, a-f). The compaction by rammer is not sufficient to break the lumps, causing inclusion of air in the sample. At water content 13.3%, the dry density shoots up and reaches its maximum irrespective of compaction energy provided. The maximum dry density achieved at 16.64% water content with lighter rammer is 1589kg/m3. The dry density of 1547 kg/m3 is achieved at 17.94% when compacted with a heavy rammer. The optimum water content for light rammer compaction falls in the range of 16.62% and 18.07%, which was speculated to be the range where the phase transition occurs. A light rammer compaction is more realistic to the actual compaction performed on fresh CoRncrete while preparing the sample for cone penetrometer test. At optimum water content, maximum dry density value indicates the formation of a dense cluster, which is in coherence with our hypothesis formed in the previous section. The change of phase can be seen visually at optimum water content where material start flowing like a viscous liquid (Fig. 4.10 (g)). At the optimum water content, CoRncrete also shows its impact activated stiffness. With further increase in the quantity of water from the optimum water content, the dry density decreases and fresh concrete flows easily (Fig. 4.10 (i)).

Ideally, the higher compaction energy should result in higher densification. Whereas, at water content 15% to 20%, the dry density measured for low energy compaction is higher as compared to high energy compaction. The stiffening behavior of wet corn starch under impact load can be a plausible explanation for this complicated behavior. The behavior of fresh CoRncrete assumes to govern by the amount of corn starch in it. With higher compaction energy, fresh CoRncrete behaves stiffer and resist deformation, allowing air to entrap within the sample. With low compaction energy, the response of CoRncrete is softer hence it does not resist that much deformation as in the case with high energy compaction. Thus, allowing air to escape and achieving higher densification.

Figure 4.10. Pictures of fresh CoRncrete (compacted with 350gm rammer) at water content (a)0%, (b)2.75%, (c)5.7%, (d)8.95%, (e)12.58%, (f)16.16%, (g)17.94%, (h)19.40% and (i)20.86%. The pictures shows fresh CoRncrete collected from the center of the sample from proctor mould for water content determination. The picture was clicked after heating the sample for 24hours in an oven at 105 ^{0}C.

It should also be noted that a higher densification of CoRncrete is achieved in comparison to sand (Fig. 4.9). Moreover, a lower energy is required in CoRncrete for higher densification. This indicates the self-compacting or self-densifying behavior of fresh CoRncrete. This materials behavior can be seen as a method of densifying sand. The addition of corn starch with sand at optimum moisture content can result in densification of sand with little or no compaction required.

The proctor test on CoRncrete support insights about fresh CoRncrete that were observed through cone penetrometer test. This result in better understanding of this material. An optimum water content is determined from proctor test where the fresh CoRncrete achieves its maximum density. This optimum water content falls in the range of water content determined in cone penetrometer test and in fact it is close to the lower value of 16.62%. By coupling the results of cone penetrometer and proctor test, It may be deduced that at optimum water content (16.64%) phase transition of corn starch particle occurs from solid to liquid. A water content of 16.66% (Cornstarch: Water: Sand:: 1:1:5) is adopted as the optimum water content for fresh CoRncrete for further testing. At this water content phase transition is expected to occur and it is marked by a maximum dry density of fresh CoRncrete.

4.3.3. Effect of water content on compressive strength of CoRncrete

Water content in the fresh CoRncrete is supposedly the most crucial factor influencing the strength of CoRncrete. In general, with an increase in water content, the strength of CoRncrete increases until an optimum water content is reached. With further increase in water beyond this optimum water content (OWC), the compressive strength reduces. The results of compressive strength of CoRncrete with varying water content is shown in table 4.2 and figure. 4.11.

It is observed that the compressive strength is directly related to the density of hardened CoRncrete. The density of hardened CoRncrete is believe to dependent on the water content in fresh CoRncrete. Water content in fresh CoRncrete effect its dry density. The maximum dry density of fresh CoRncrete results in the maximum bulk density of hardened CoRncrete. The maxima are achieved at optimum water content. In hardened CoRncrete, 95% water is removed in the heating process, hence the bulk density can be considered comparable to its dry density.

Table 4.2. Results of compressive strength test testing on hardened CoRncrete prepared with varying water content.

Heating mode	Water content [%]	Mix proportion [weight]	Density of wet CoRncrete [kg/m3]	Density of solid CoRncrete [kg/m3]	Volume change [%]	Compressive strength [MPa]
Microwave	10.00	1:0.6:5	1246.88	1256.02	10.77	**1.82**
Microwave	13.33	1:0.8:5	1333.33	1409.44	16.13	**7.28**
Microwave	15.00	1:0.9:5	1510.42	1552.29	13.13	**26.67**
Microwave	16.66	1:1:5	1808.85	1569.49	-1.52	**21.64**
Microwave	18.33	1:1.1:5	1771.35	1495.14	-2.19	**15.25**
Microwave	20.00	1:1.2:5	1788.54	1356.34	-11.40	**-**
oven	10.00	1:0.6:5	1258.85	1230.63	-	**-**
oven	13.33	1:0.8:5	1318.75	1168.75	-	**-**
oven	15.00	1:0.9:5	1533.33	1302.06	-0.82	**1.29**
oven	16.66	1:1:5	1829.17	1609.85	1.51	**13.70**
oven	18.33	1:1.1:5	1758.33	1592.07	4.96	**10.66**
oven	20.00	1:1.2:5	1790.10	1521.99	1.27	**9.17**

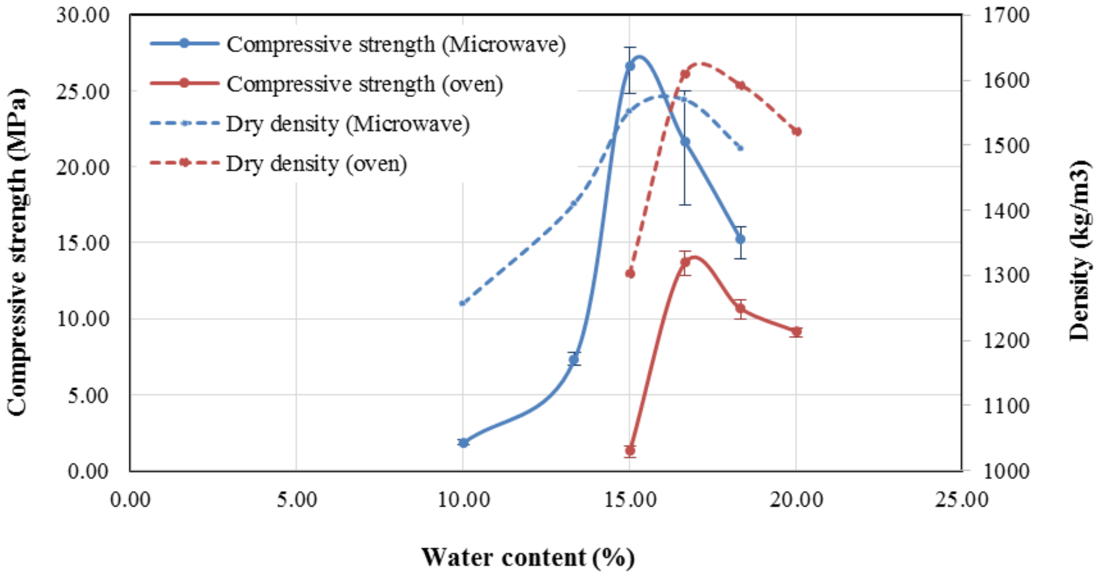


Figure 4.11. The plot of compressive strength of hardened CoRncrete with varying water content. The dry density of hardened CoRncrete is also indicated on the plot. With an increase in water content strength of hardened CoRncrete increases until a certain water content. Thereafter, it decreases.

The density of hardened CoRncrete can be related to its packing density. In dense CoRncrete, sand grains are expected to be closer to each other as compared to low density CoRncrete (figure 4.12). Thus, for the same volume, quantity of sand grains is higher in denser CoRncrete. The higher amount of stiff material within the matrix may result in higher strength. Also, with increase in the density of material, the interfacial area between sand grain and corn starch matrix increases. If the interface is assumed as the weakest link in hardened CoRncrete, the interfacial area will have an effect on bonding of sand particle and corn starch matrix. With high interfacial area, higher strength may be required to fail the specimen.

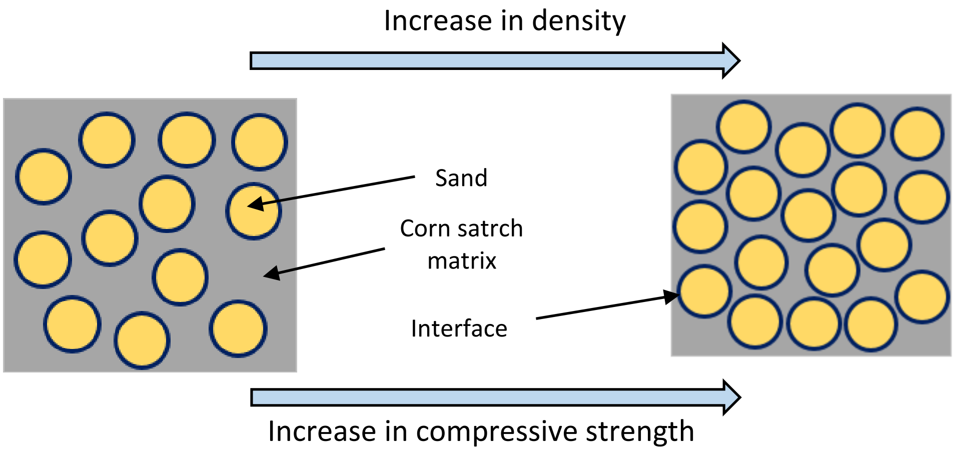


Figure 4.12. Effect of increases in the density of hardened CoRncrete (which is a function of water content in fresh CoRncrete) on compressive strength.

In chapter 3, the gelatinization process is discussed. The gelatinization process is important in the formation of microstructure in hardened CoRncrete. The gelatinization process is affected by the amount of water in fresh CoRncrete. With increases in water content, the extent or degree of gelatinization increases (figure

4.13). The extent of gelatinization affects the bonding of corn starch matrix and sand. Greater bonding is expected in completely gelatinized corn starch matrix. Moreover, the quantity of gelatinized product appears higher with greater water content. It is observed that after optimum water content is reached, the strength reduces. This may be possible due to a decrease in concentration of corn starch particle in the matrix, affecting the bonding power of ‘starch glue’ or gelatinized corn starch matrix. This may also be possible due to increasing shrinkage crack or the segregation of corn starch particle making the CoRncrete heterogeneous with addition of more water beyond optimum water content. The effect of water content on the binding capacity of the corn starch matrix and sand grain is unknown which makes it difficult to make any strong conclusions.

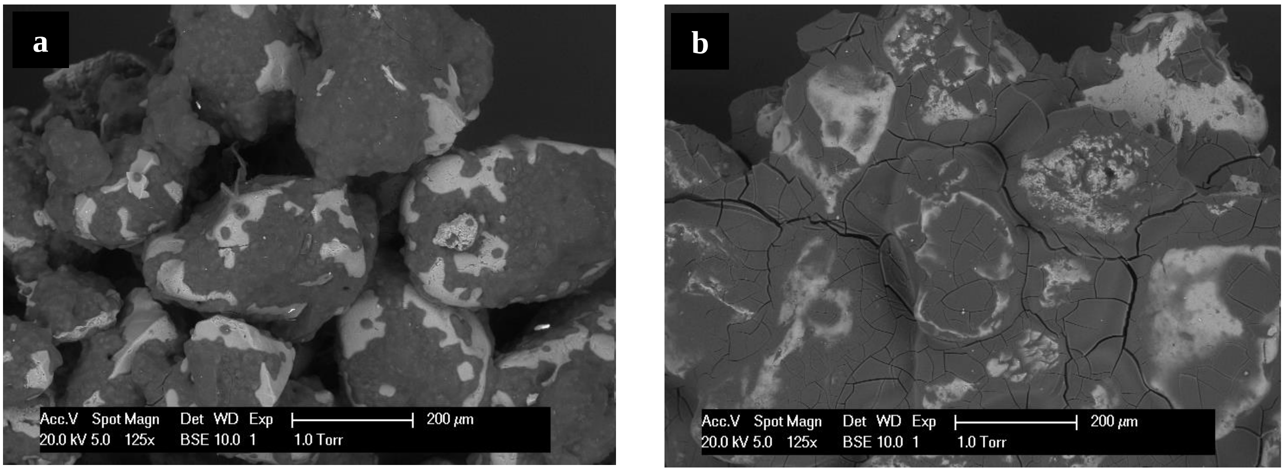


Figure 4.13. ESEM image of hardened CoRncrete heated in DSC equipment with the water content (a) 15% and (b) 18%. The water content has effect on gelatinization and degree of gelatinization achieved in 15% water content is lesser than 18%. The gelatinized product appears higher in 18% water content specimen.

In case of CoRncrete formed by heating in a convection oven, the strength increases until optimum water content. With further increase in water content, the strength decreases. The image of CoRncrete specimens heated in a convection oven is shown in figure 4.14 Very weak specimens were formed with water content 10% and 13.33% possibly due to a low degree of gelatinization (a,b). At 16.66% optimum water content, maximum compressive strength of 13.7 MPa is achieved. As discussed in the previous chapter, complete gelatinization is not achieved at this water content. With further increase in water, segregation of water in fresh CoRncrete was observed, resulting in different extent of gelatinization in a different region, making hardened CoRncrete specimen heterogeneous (e,f). The increase in water beyond optimum water content may also affect the shrinkage crack, gelatinization process, the binding power of gelatinized corn starch, all resulting in a decrease in strength.

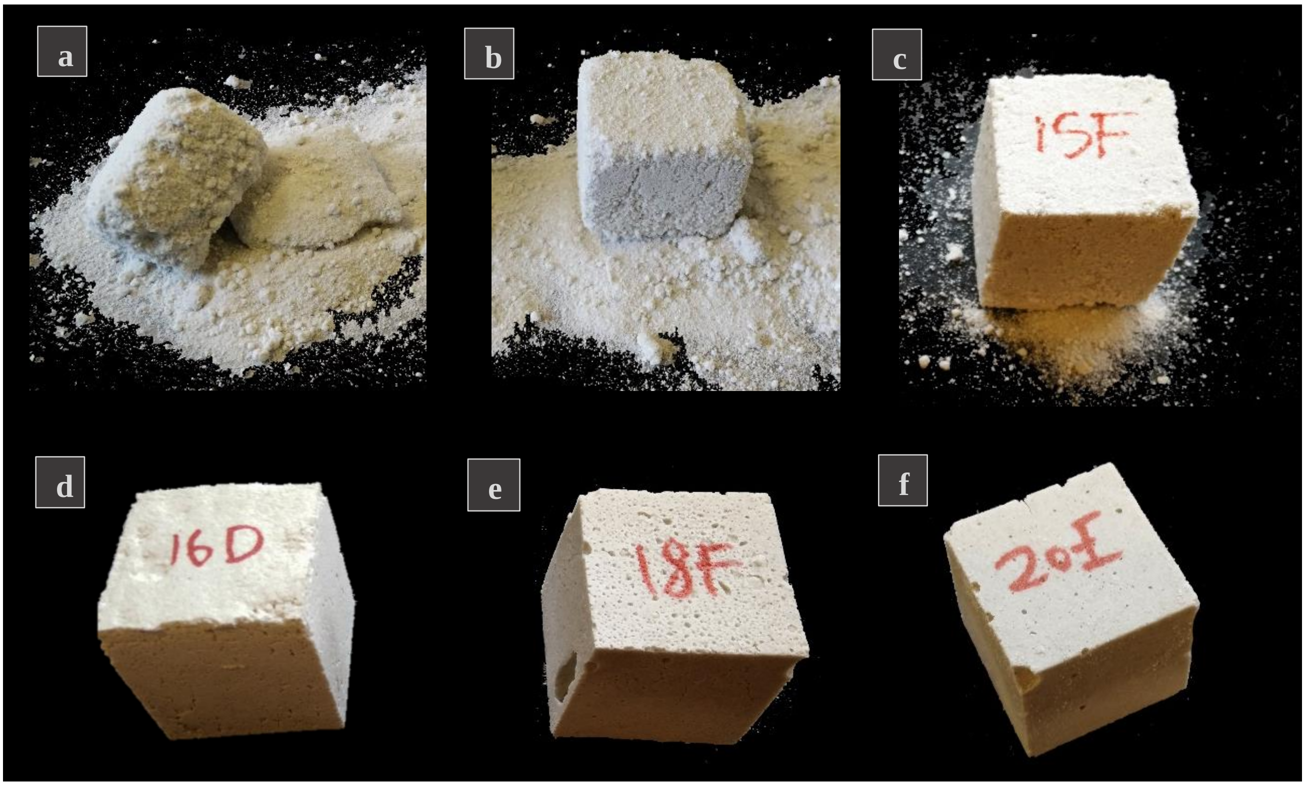


Figure 4.14. CoRncrete specimen heated in convection oven with water content (a) 10%, (b) 13.33%, (c) 15%, (d) 16.66%, (e) 18.33% and (f) 20%.

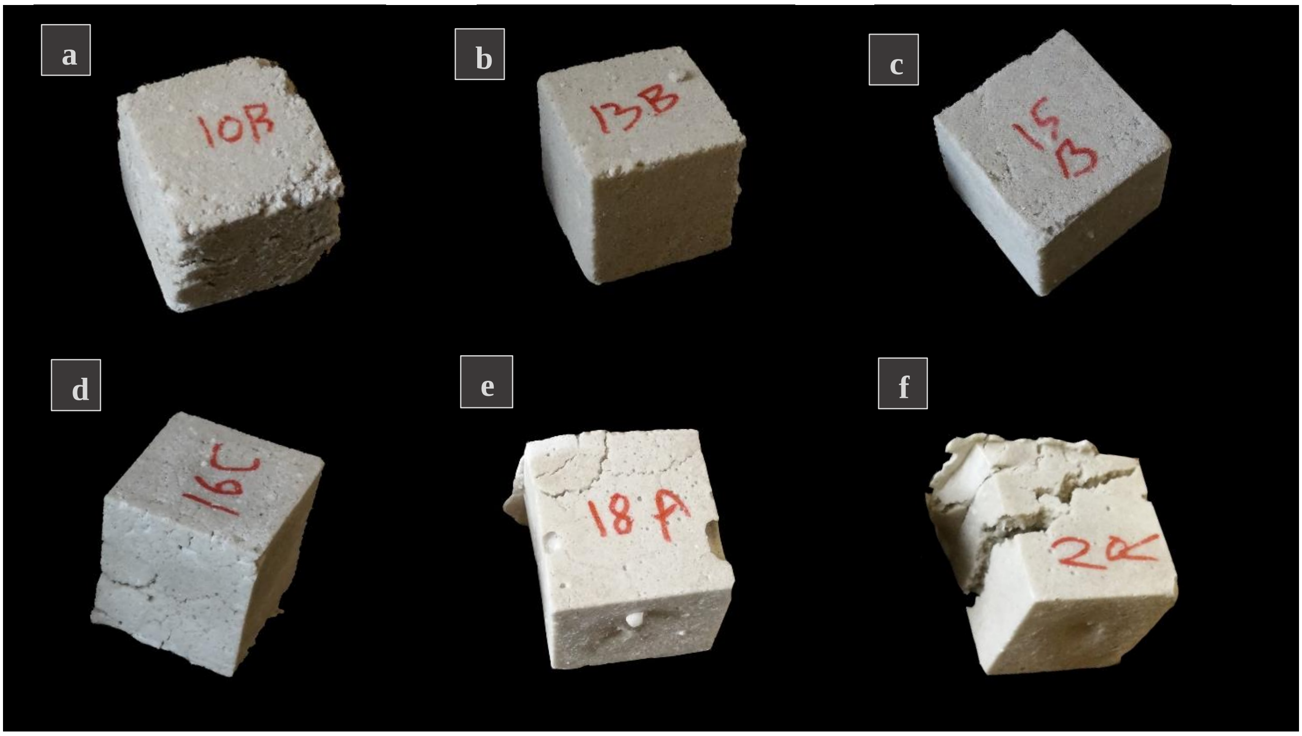


Figure 4.15. CoRncrete specimen heated in microwave with water content (a) 10%, (b) 13.33%, (c) 15%, (d) 16.66%, (e) 18.33% and (f) 20%.

In case of CoRncrete prepared by heating in a microwave, an increase in strength is observed until 15% water content. With further increase in water, the strength decreases. The image of CoRncrete specimens heated in microwave is shown in figure 4.15. The underlying principle of a microwave heating plays an important role in the formation of CoRncrete specimens. The specimen formed with quantity of water less than the optimum water content results in shrinkage and attains a denser configuration. The shrinkage affects the density and hence the compressive strength. An average compressive strength of 10.77MPa is achieved at 10% water content. At this water content, the shape of CoRncrete is not regular (a). With an increase in water content to 15%, the strength increases rapidly and maximum compressive strength of 26.67MPa is achieved. A regular shaped specimen is formed at this water content (c). Shrinkage upto 12% of the volume is measured at 15% water content (shrinkage represented as positive volume change). It is believed that the gelatinization process at this water content is not completed, resulting in partially gelatinized corn starch granules. However, these granules still bond with sand particle and forms a dense configuration. At 16.66% or optimum water content, a higher strength is expected. At this water content, corn starch is believed to be completely gelatinized based on ESEM image of hardened CoRncrete shown in the previous chapter. Contrary to the expectation, a lower strength is observed potentially due to the formation of crack in the specimen as a result of high pressure filtration flow during heating process. The underlying heating mechanism in CoRncrete is assumed to change at and above optimum water content which results in the formation of crack (e,f). With an increase in water content, crack width and deformation in specimen increases, and the compressive strength decreases. A significant deviation in results is observed with higher water content due to different cracking pattern in each CoRncrete specimens

It should be noted that the strength of CoRncrete formed by heating in a microwave is higher than CoRncrete heated in a convection oven. In a microwave, the rapid heating and close boundary condition increases the temperature of CoRncrete instantly and gelatinization temperature is achieved instantly. While in an oven, the rate of heating is slow and top boundary is open. Water evaporates before gelatinization temperature is reached. Thus gelatinization occurs at relatively lower water content, resulting in a low degree of gelatinization as compared to microwave heated specimen.

4.3.4. Effect of sand grain size on compressive strength of CoRncrete

The effect of size of sand grain size on compressive strength can be seen in table 4.3 and figure 4.16. Irrespective of the source of heating, with an increase in sand grain size, compressive strength decreases. The specimens with 16.66% water content were prepared in an oven. The strength of oven heated CoRncrete reduces from 10.7MPa to 2.64MPa with the use of coarser aggregate (1.0-2.0mm). The specimen prepared in a microwave with the regular heating regime of 5min (1.5 thaw+3.5 high power) does not follow the trend of decreasing strength with increasing sand grain size. The strength of 0.5-1.0mm sand aggregate CoRncrete is higher than 0.25-0.5mm sand aggregate CoRncrete, both heated with 15% water content in a microwave. This is possibly due to burning 0.25-0.5mm sand grain specimen. While performing the heating task, a burning smell was sensed from one of these specimens. It was decided to redo the test with a different heating regime of 4.5min (1.5 thaw+3 high power) in order to prevent the influence of burning on test results. The compressive strength result of these specimens followed the trend of decreasing strength with increasing sand grain size. This indicates that the ideal heating scheme for each type of CoRncrete is different. These result also formed the basis of finding the influence of heating duration in microwave on compressive strength of hardened CoRncrete. It can also be seen in figure 4.16 that with an increase in the size of sand grain, the shrinkage of CoRncrete reduces (shrinkage represented as positive volume change). Specimen with 1.0-2.0 sand aggregate swells. Shrinkage, which possibly depend on the porosity of fresh CoRncrete may play an important role in the development of strength of CoRncrete.

Table 4.3. Results of the effect of sand grain size on the compressive strength of hardened CoRncrete.

Heating mode	Sand fraction [mm]	Mode thaw(min)+ max. Heating(min)/ oven heating [hour]	Density of wet CoRncrete [kg/m3]	Density of solid CoRncrete [kg/m3]	Volume change [%]	Compressive strength [MPa]
Microwave	0.125-0.25	1.5+3.5	1510	1552	13.1	**26.67**
Microwave	0.25-0.5	1.5+3.5	1507	1493	10.4	**10.61**
Microwave	0.5-1.0	1.5+3.5	1678	1564	5.0	**11.43**
Microwave	1.0-2.0	1.5+3.5	1825	1588	-1.8	**6.32**
Microwave	0.125-0.25	1.5+3	1521	1495	9.6	**23.02**
Microwave	0.25-0.5	1.5+3	1504	1505	7.9	**14.94**
Microwave	0.5-1.0	1.5+3	1678	1529	3.6	**11.91**
Microwave	1.0-2.0	1.5+3	1825	1567	-3.6	**8.28**
oven	0.125-0.25	24	1829	1610	1.5	**13.70**
oven	0.25-0.5	24	1858	1683	3.9	**9.27**
oven	0.5-1.0	24	1852	1663	3.6	**4.67**
oven	1.0-2.0	24	1924	1686	1.2	**2.64**

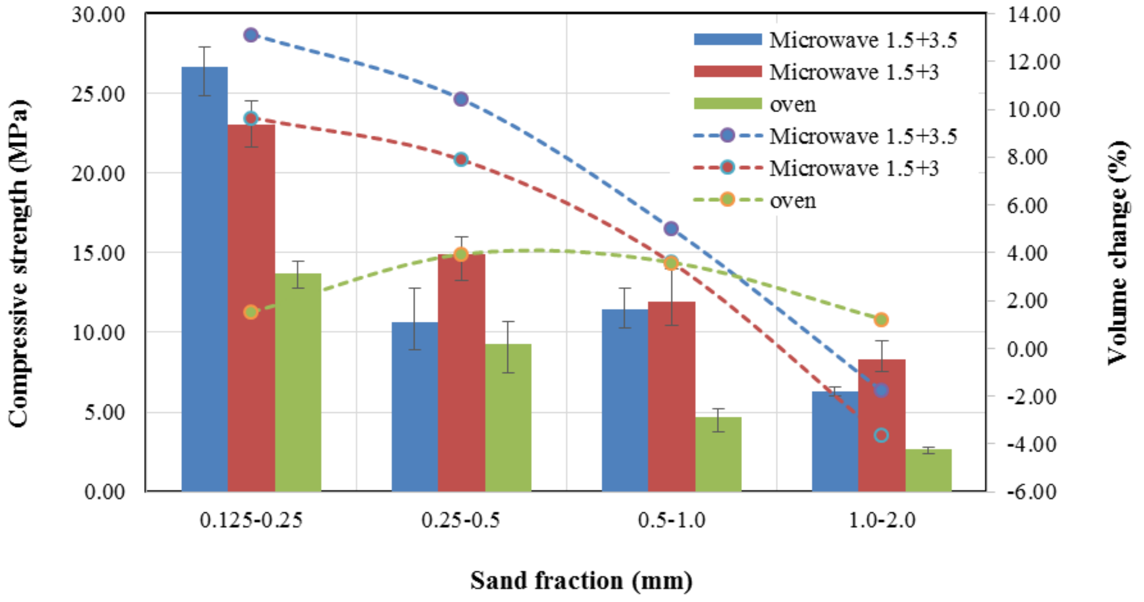


Figure 4.16. The plot of compressive strength of hardened CoRncrete with increasing sand grain size. The specimen in a microwave was prepared with 15% water content whereas in an oven with 16.66%. The strength of CoRncrete reduces with increasing sand grain size. A relation between volume change (shrinkage or swelling) and sand grain size is also observed. shrinkage is represented as positive volume change. With an increase in sand grain size, shrinkage decreases.

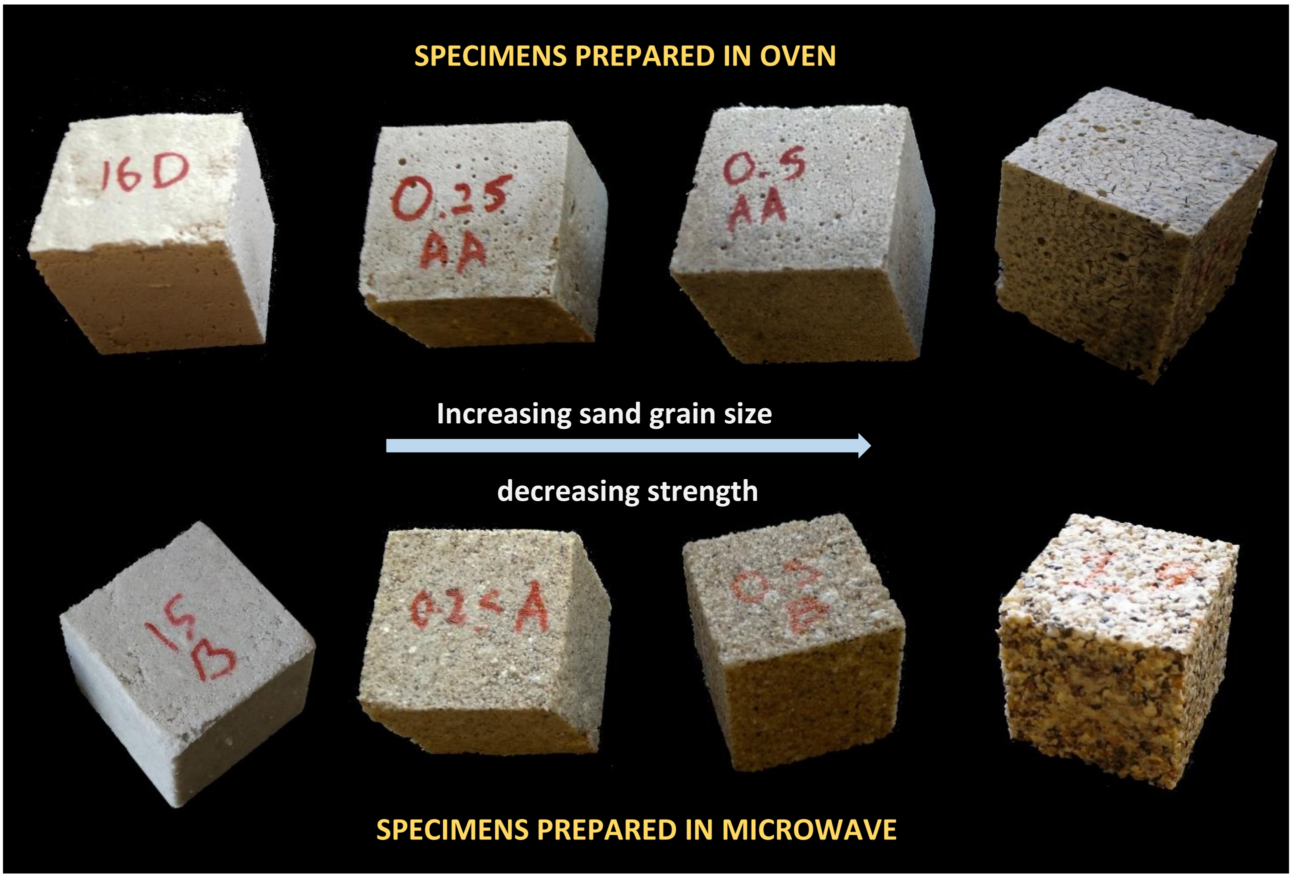


Figure 4.17. CoRncrete specimen with varying sand grain size heated in microwave and oven at 15% and 16.66% water content respectively.

The image of specimens heated in microwave and oven is shown in figure 4.17. Factors that might influences the strength of hardened CoRncrete with increasing sand grain size must be understood. Although, the weakest link in CoRncrete is unknown, an assumption of the interface being the weakest link might explain the effect. With an increase in sand size, the surface area of sand grain reduces (figure 4.18). If the interface is assumed as the weakest link, decrease in surface area may result in the reduction of sand-starch bonding area. A lower bonding may result in lower strength.

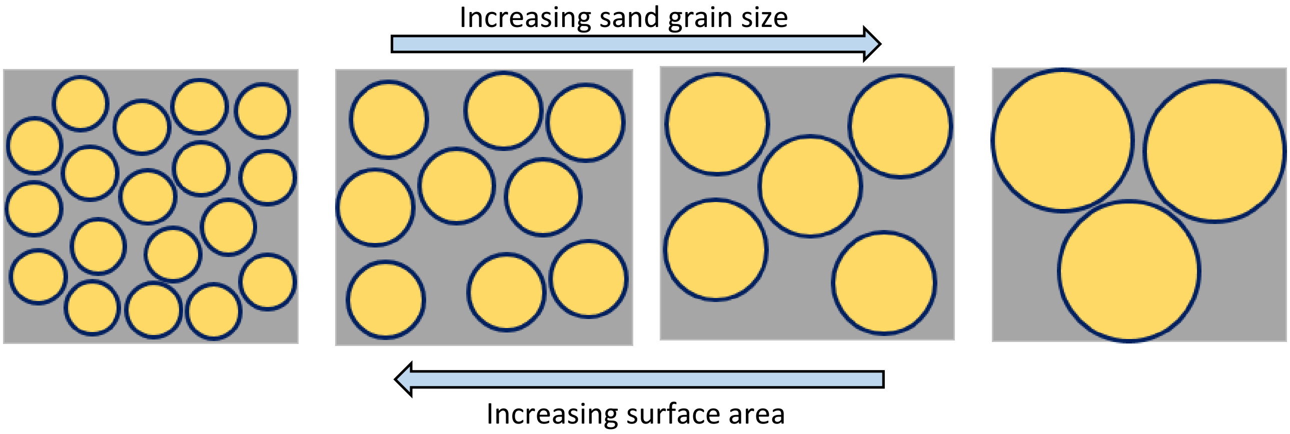


Figure 4.18. Influence of increasing particle size on surface area.

Another possibility is the reduction in strength due to higher tensile stress generated in corn starch matrix. With an increase in the size of sand grain, the average distance between sand grains is expected to increase. Due to shrinkage, the higher tensile stress may appear in the matrix of coarse grained CoRncrete. Higher tensile stress can result in lower strength. In this postulate, the failure will not occur through the interface of sand and corn starch matrix but the matrix itself. The two postulates put forward are speculation and further testing is required to comment on the observed behaviour.

4.3.5. Effect of heating duration on compressive strength of CoRncrete in microwave

The heating duration or heating energy has a significant effect of gelatinization process as discussed in chapter 3. The effect of heating duration on CoRncrete specimen prepared with 15% water content is shown in table 4.4 and figure 4.19. With an increase in duration of heating, the compressive strength increases until the water content in specimen reduces significantly. With further heating, compressive strength reduces. The moisture content in specimen reduces with increase in heating.

Table 4.4. Effect of heating duration in microwave on compressive strength of hardened CoRncrete.

Mode thaw[min]+ Max. Heating [min]	Time in microwave [sec]	Energy [KJ]	Density of wet CoRncrete [kg/m3]	Density of solid CoRncrete [kg/m3]	Volume change [%]	Moisture in dry CoRncrete [%]	Compressive strength [MPa]
1.5+1	150	165	1381	1477	9.05	74	**0.97**
1.5+2	210	231	1469	1480	11.99	10	**21.08**
1.5+3	270	297	1521	1495	9.64	7	**23.02**
1.5+3.5	300	330	1489	1552	13.13	4	**26.67**
1.5+4	330	363	1572	1512	8.19	4	**17.55**
1.5+4.5	360	396	1494	1480	11.06	2	**11.95**

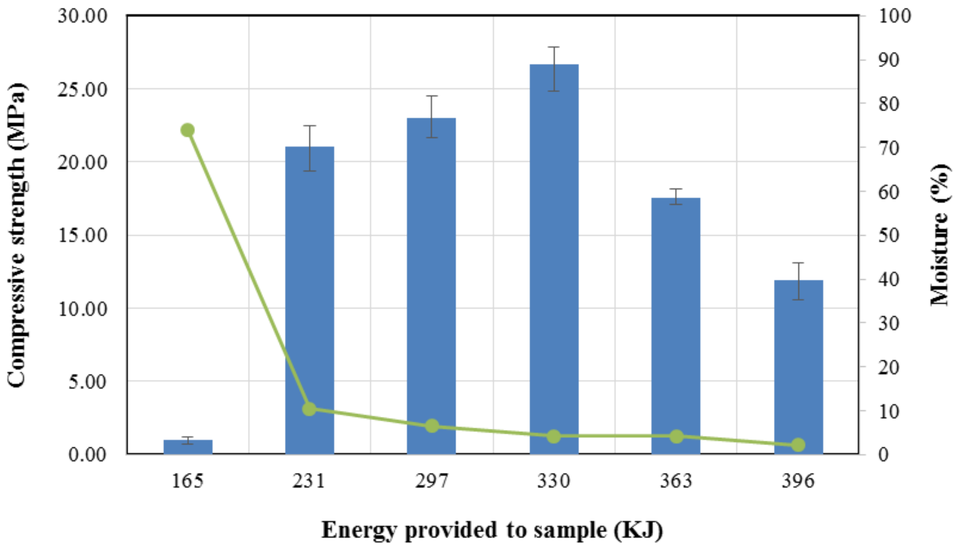


Figure 4.19. Effect of heating duration in microwave on the compressive strength of CoRncrete. The heating time is represented in Kilo Joules of energy. The change of moisture content with heating time in CoRncrete specimens prepared with 15% water content is also shown.

The heating duration has a significant effect on gelatinization process, thus the strength of CoRncrete (figure 4.20). When CoRncrete is heated for 1.5min in thaw mode, the specimen is still soft. The gelatinization process is incomplete as shown in figure 4.20. At this moment, the moisture loss from the specimen is low. With an increase in heating duration from 1.5min to 3.5 min (1.5min thaw+2min high power) or 231 KJ, the gelatinization process is significantly advanced and results in a rapid increase of compressive strength. A significant reduction in moisture is also observed at this energy. Beyond this heating duration, the change in compressive strength and moisture content is low. The maximum compressive strength of 26.66 MPa is noticed at 5min or 330KJ energy. With further heating, the removal of moisture becomes difficult as it is held tightly by gelatinized corn starch matrix. This may cause a rise in temperature beyond 290^{o}C and corn starch starts burning. The burning of corn starch reduces the compressive strength. The burning mark in specimens were observed originating from the core of specimen. CoRncrete heated in 2450MHz frequency, 1100watt microwave with 15% water content has an optimum heating duration of 5min for the given heating regime (1.5 thaw+ (x) min high power). However, the brand, size and age of microwave oven may significant effect on the optimum heating duration.

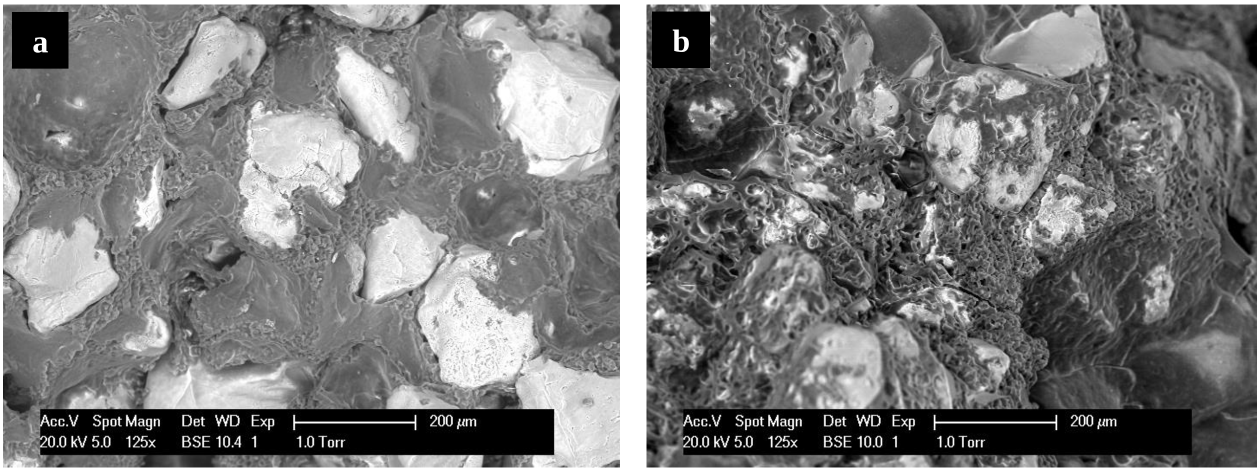


Figure 4.20. ESEM image of CoRncrete specimens with 16.66% water content (a) partially (1.5min) and (c) completely (5min) heated in a microwave. The heating affect the gelatinization process and the bonding of gelatinized corn starch to sand.

4.4. Chapter conclusion

The behavior of fresh CoRncrete was studied through cone penetrometer and proctor test. The results of cone penetrometer test gave indication of undrained shear strength of CoRncrete at varying water content and an approximate water content range (16.62% - 18.06%) where phase of corn starch particles changes from solid particles (with adsorbed water) to particles in suspension of water. The behavior or consistency of fresh CoRncrete was also indicated by cone penetrometer. To support and verify the observation of change in phase of corn starch particle from solid to suspension in water, proctor test was carried out. Optimum water contents from proctor test (corresponding to maximum dry density) were found to be 16.64% and 17.94% at compaction energy of 22.1 KJ/m3 and 51.5 KJ/m3 respectively. This optimum water content falls in the range of water content determined in cone penetrometer test and in fact it is close to the lower value of 16.62%. A water content of 16.66% (Cornstarch: Water: Sand:: 1:1:5) was adopted as the optimum water content for fresh CoRncrete. At this water content phase transition is expected to occur and it is marked by the maximum dry density of fresh CoRncrete.

Factors such as water content, size of sand aggregates and duration of heating (in a microwave) affects the strength of CoRncrete significantly. In general, with an increase in water content, the strength of CoRncrete increases until an optimum water content is reached. With further increase in water beyond this optimum water content (OWC), the compressive strength reduces. A co-relation between the density of hardened CoRncrete and its compressive strength is found. In an oven, Increase in water beyond optimum may affect the extent of gelatinization, cause segregation, increase shrinkage cracks and decrease the binding power of gelatinized corn starch, all resulting in a decrease in strength. In case of microwave heating, the strength increases until 15% water content. Above this water content, crack formation result in reduction of strength. With an increase in size of the sand aggregate, decrease in compressive strength was observed. This might be related to a decreases in the surface area with increase in size of sand grain. If the interface is assumed to be weakest link, less bonding area will result in a weaker specimen. With an increase in the size of sand grain, the average distance between sand grains is expected to increase. Due to shrinkage, the higher tensile stress may appear in the matrix of coarse-grained CoRncrete. Higher tensile stress can result in lower strength. Irrespective of the source of heating, with an increase in sand grain size, compressive strength decreases. With increase in duration of heating, the compressive strength increases until the water content in specimen reduce significantly. With further heating, compressive strength reduces. The moisture content in specimen reduces with increase in heating.

Compressive strength upto 26.67 MPa was achieved in the case of microwave heating (water content 15%). Whereas, it was limited to 13.70 MPa (water content (optimum): 16.66%) in oven heating method. This difference may be explained on the basis of ESEM scans which indicate that the extent of gelatinization achieved in microwave heating was higher than oven heating at similar water content. In a microwave, the rapid heating and close boundary condition increases the temperature of CoRncrete instantly and gelatinization temperature is achieved instantly. While in an oven, the rate of heating is slow and top boundary is open. Water evaporates before gelatinization temperature is reached. Thus gelatinization occurs at relatively lower water content, resulting in a low degree of gelatinization as compared to microwave heated specimen.

A co-relation between optimum water content, dry density of wet CoRncrete, the density of hardened CoRncrete and strength of hardened CoRncrete was found. At optimum water content, the phase transition of corn starch occurs, maximum dry density in fresh CoRncrete and hardened CoRncrete is achieved and maximum compressive strength is observed. Exception in case of microwave heating method was observed where the underlying heating mechanism believes to affect the structure and strength of CoRncrete.

5

CoRncrete in a wet environment

5.1. Introduction

Strength, durability and sustainability are three key factors that define the performance of any material. CoRncrete is a strong material with compressive strength comparable to fired clay brick. The durability of CoRncrete is studied in this chapter by visualizing the degradation of CoRncrete specimen in a wet environment. 3 category of CoRncrete specimens were selected based on compressive strength result in previous section: Microwave heated CoRncrete specimen with 15% water, microwave heated specimen with 16.66% water and oven heated specimen with 16.66% water content. The hardness of CoRncrete is measured using Equotip (non-destructive testing) before and after submerging specimens in water for 1,3, and 7 days. The Equotip results give an indication of degradation of CoRncrete under wet environment. The test results are more relied on visuals (photograph and video) which are successful in capturing the degradation process. Two videos included in this chapter covers the majority of work related to degradation of CoRncrete in a wet environment.

5.2. Material and method

5.2.1. Environmental Scanning Electron Microscope (ESEM) testing

ESEM image provides 2-D information about the sample which is useful in visualizing the change in the microstructure of a material. An advantage of ESEM over SEM's is possibility to wet the sample while imaging. The ESEM technique is already elucidated in chapter 3. An introduction to the wetting process of the sample is included in the section.

Sample preparation

CoRncrete specimens with 16.66% water content were prepared by heating in a microwave. The specimen was heated in a microwave for 5min [1.5 (thaw) + 3.5(high power)] and later stored in plastic foil for 20days. Detailed preparation of specimens is already explained in section 4.2.4. One specimen was directly taken, chopped off and tested in ESEM. Another specimen was submerged in water for 7 days and then dried for 2 days at 20°C. A portion of the specimen was chopped off and taken for ESEM testing.

Test procedure

CoRncrete sample was imaged under Philips XL-30 (Netherlands), ESEM at Microlab. The sample was kept in ESEM and imaged under vacuum (1 torr) at 20°C. The pressure in the specimen chamber was increased to 4.3 torr. With a help of special system water was introduced into the chamber. At 20°C, the water exists in gaseous phase within the chamber. The temperature of specimen chamber was lowered to 1°C.At this temperature, the water vapour in specimen chamber condensed on the sample, thus the sample was wetted. This condensation process is often experienced in winters when a person with spectacles enters a moist kitchen and his glasses are covered with water vapours. The wetting mechanism of sample in ESEM is somewhat similar. CoRncrete images in a wet environment were captured at 500x magnification until 18min. The specimen submerged in water for 7 days was imaged in ESEM at 20°C. Images at 125x and 1000x were taken.

5.2.2. Equotip hardness test

Equotip is a non-destructive test to measure the hardness of metallic material. It is a battery operated spring loaded device with a 3mm diameter spherical tungsten carbide tip. The tip is made to fall on a hard surface and the impact and rebound velocity are measured. This velocity is represented in Hardness value (L). According to Verwaal and Mulder (1993), Equotip can also be used as an index test to estimate the rock strength. The test conducted by Equotip is found reliable for a homogenous rock material with strength greater than 10MPa. It is recommended to use the sample with a volume of 200 cm^3 however, smaller sample can be used.

Sample preparation

CoRncrete specimens of 40×40×40mm (64 cm^3) were prepared by following the procedure explained in section 4.2.4 of chapter 4. The three categories of specimens prepared for degradation test are shown in table 5.1.

Table 5.1. Specimens for degradation study

Specimen	Source of heating	Water content in fresh CoRncrete [%]	Heating regime	Number of specimens
M16.66	Microwave	16.66	5 min (1.5 thaw +3.5 high power)	9
M15	Microwave	15	5 min (1.5 thaw +3.5 high power)	9
O16.66	Oven	16	24h at 105°C	9

Test procedure

The hardness of CoRncrete specimens was measured using Equotip (figure 5.1). 10 individual test were conducted on a face of specimen perpendicular to casting side. It was ensured that test location was separated by at least 5mm. The test location of the specimen is shown in figure 5.1.

In the next step, the specimens were submerged in water such that 1 cm of water head was available on the top of specimen. The specimens were kept at 20°C. The degradation process was also recorded by a mobile phone camera. The specimens were taken out of the bucket on 1, 3 and 7 days. They were dried for 48h and the change in hardness was measured with Equotip at the same testing locations.

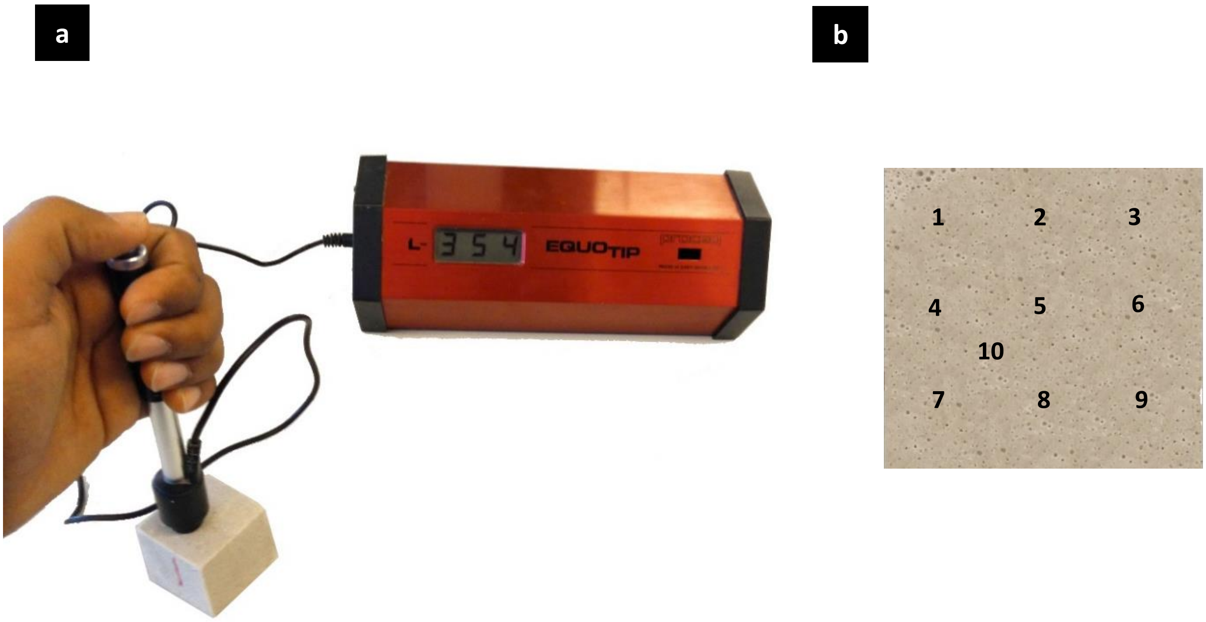


Figure 5.1. Equotip testing. (a) Testing the surface hardness of CoRncrete specimens with Equotip, (b) 10 test location shown on a CoRncrete specimen.

5.3. Result and discussion

5.3.1. Microscopic analysis of degradation mechanism

The microstructural degradation of CoRncrete heated in a microwave with 16.66% water content was studied through ESEM. The ESEM images are shown in figure 5.2. A rapid degradation can be visualized. CoRncrete gains strength due to the gelatinization of starch. Under the wet condition, the gelatinized starch starts degrading and forms loose gel. The interface between sand and gelatinized starch may loosen up. Complete softening of corn starch is visualized after 18 minutes. This indicates that CoRncrete degrade rapidly in wet environment supplied in ESEM test. The test condition in ESEM includes vacuum and high voltage electrons beam incident on specimen's surface. An important question arises here: Does CoRncrete behave in the same way under normal atmospheric condition? The answer is perhaps No, due to a special property of corn starch with water called magnetorheology. Corn starch with water forms a magnetorheological fluid that changes its viscosity under high voltage. Under ESEM condition, gelatinized corn starch with water degrades at an accelerated pace due to the magnetorheological behaviour of corn starch. If this holds true, the specimen (Microwave, Wc=16.66%) should degrade slower in atmospheric condition.

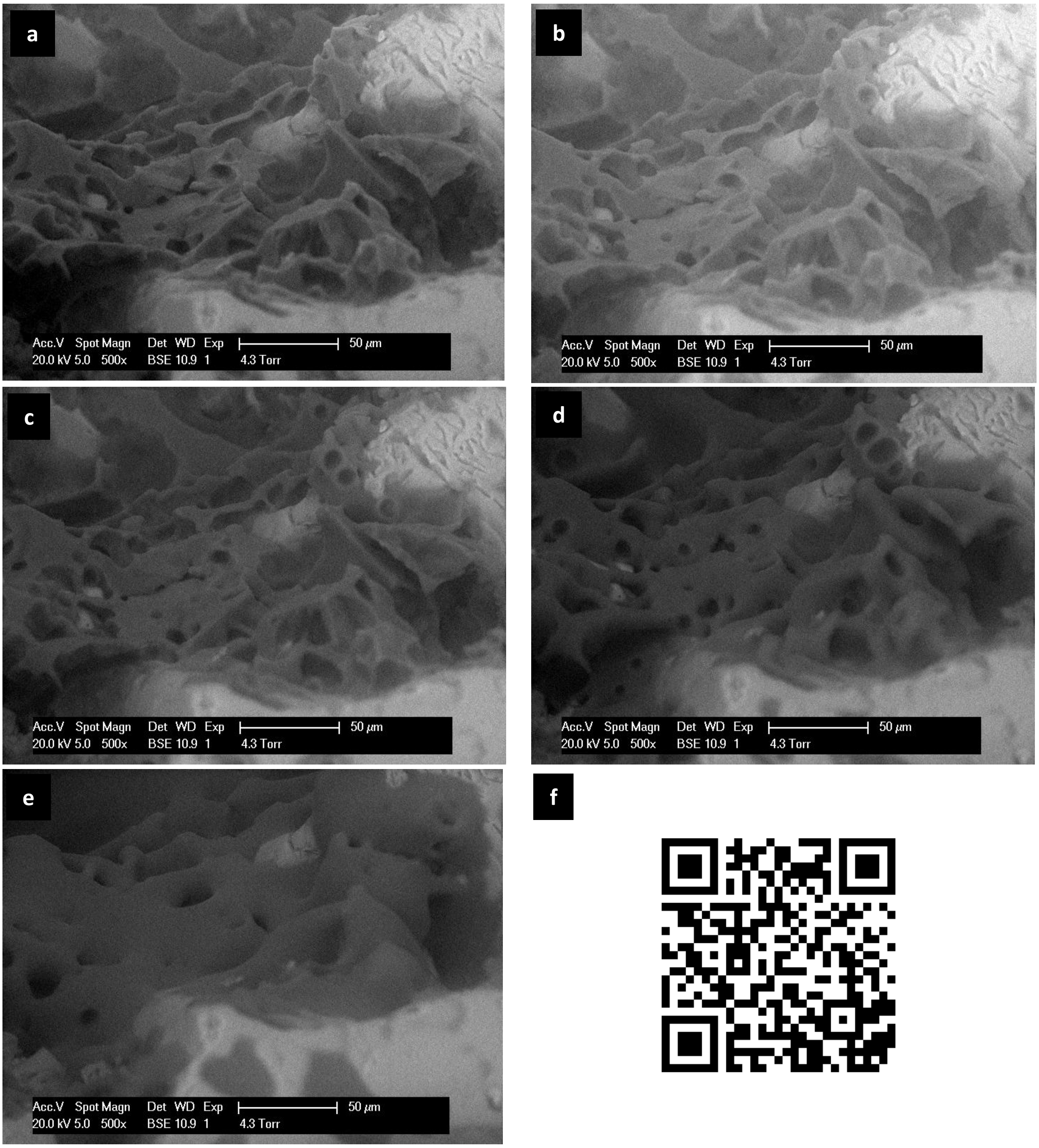


Figure 5.2. ESEM image of degradation of CoRncrete at (a) 0min (20°C), (b) 6 min (1°C), (c) 9 min (1°C), (d) 13 min (0.5°C), (e) 13 min (0.5°C), (f) QR code video https://www.youtube.com/watch?v=2fBX-KjEJuc . The video is made by overlying images captured by ESEM.

ESEM image of a degraded CoRncrete specimen (heated in a microwave, Wc =16.66%) submerged in water for 7 days and dried for next 2 days at 20°C, is shown in figure 5.3. Cracks are visible on the specimen. The cracks might form due to wetting and drying cycle in atmospheric conditions or drying under vacuum condition. At 1000x magnification, a gap at sand- corn starch interface is visible.

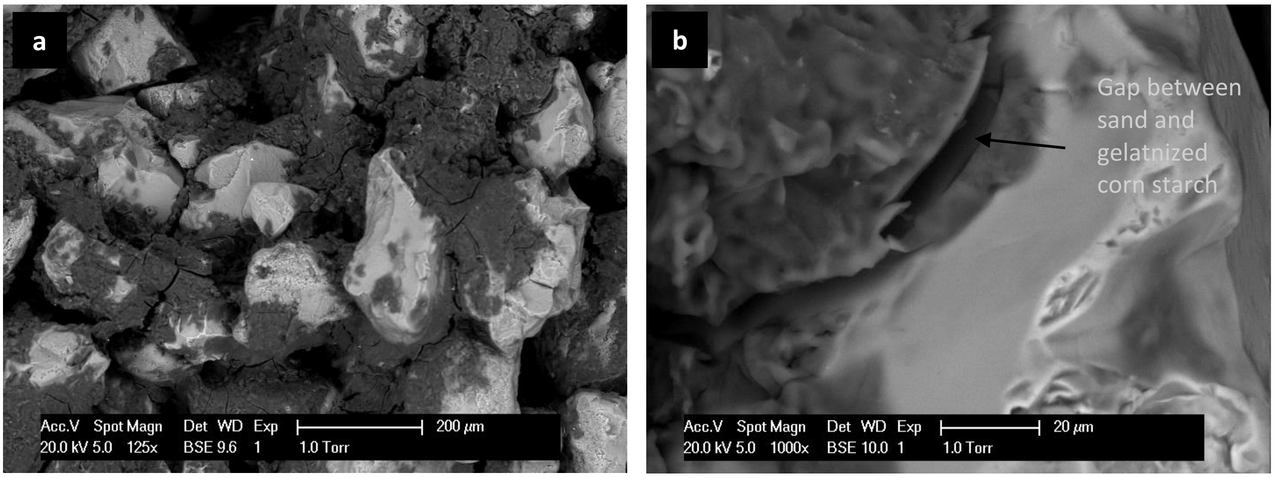


Figure 5.3. ESEM image of microwave heated specimens kept submerged in water for 7 days and dried for next 2 days (a) 125x and (b) 1000x.

5.3.2. Effect of wetting duration on degradation of CoRncrete

The effect of wetting duration on degradation was studied using hardness value measured by Equotip. However, the degradation of CoRncrete specimens heated in a microwave with 15% water content and CoRncrete specimens heated in an oven at 16.66% was so rapid that the visuals were sufficient for observation. The degradation of a single CoRncrete specimen of each category is shown in figure 5.3 and figure 5.4. The actual test was performed on a triplicate and a better (and highly recommended) visualization of degradation can be seen via following the QR code in figure 5.5. The degradation in specimens (Microwave: Wc=15% and oven: Wc= 16.66%) started as soon as specimens were kept in water. A complete degradation is visible in 1 day.

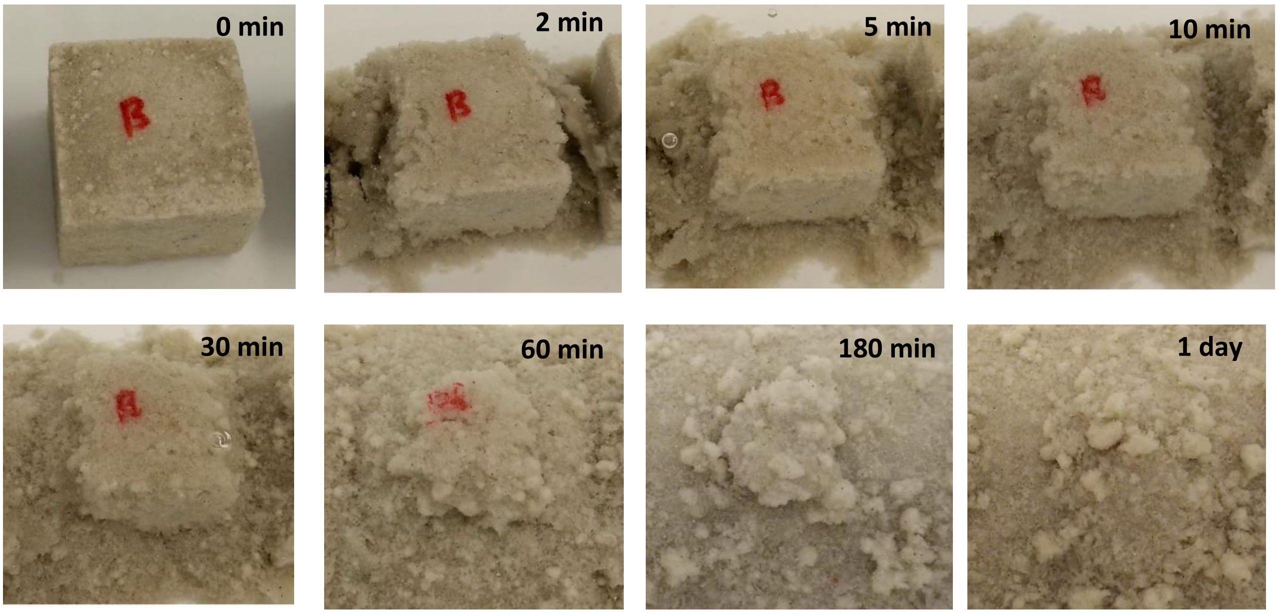


Figure 5.3. Degradation of CoRncrete specimen heated in microwave with 15% water content

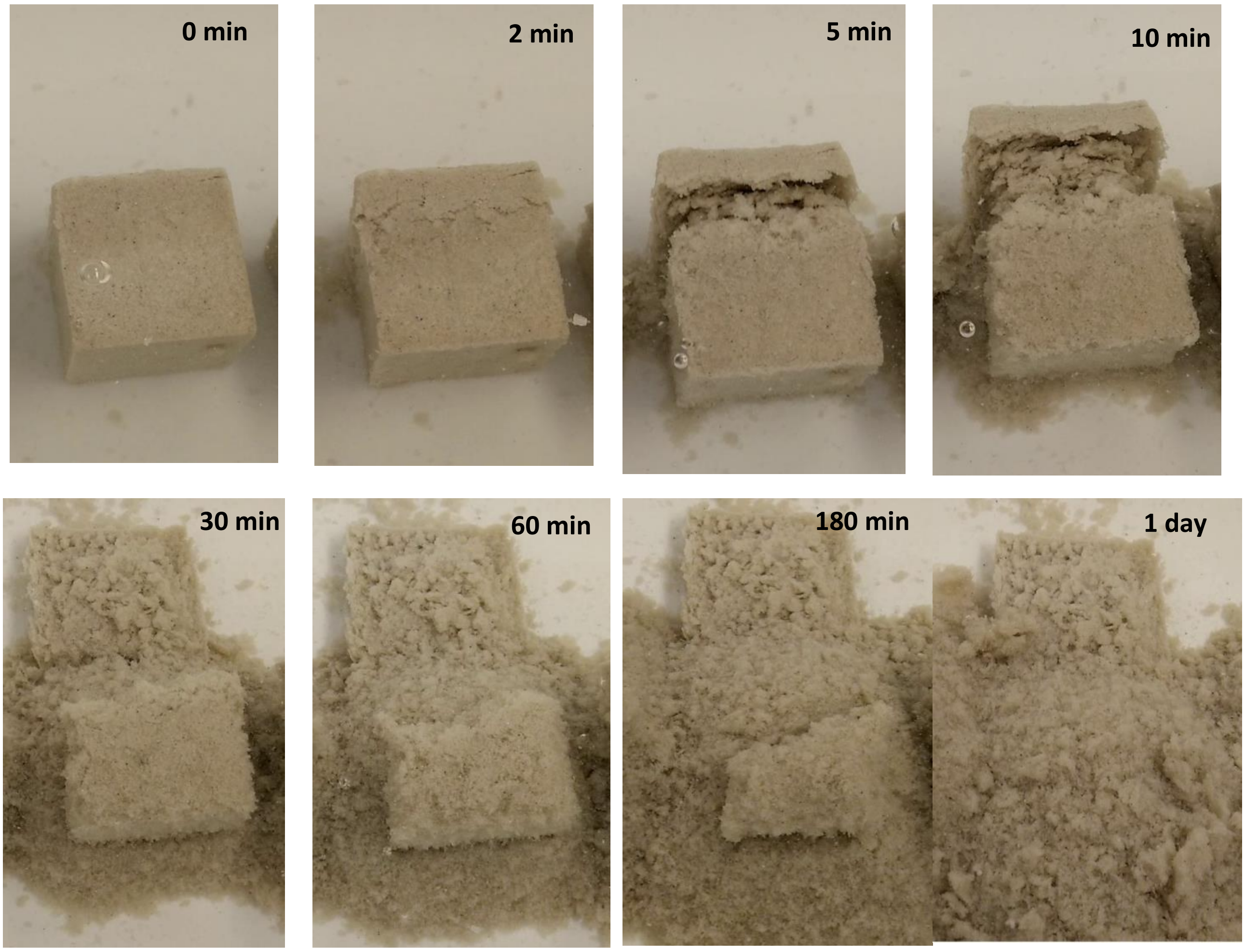


Figure 5.4. Degradation of CoRncrete specimen heated in oven (105ºC,24h) with 16.66% water content

Figure 5.5. QR code for video on the degradation of CoRncrete heated in microwave and oven with 15% and 16.66% water content respectively. Video link https://www.youtube.com/watch?v=hvnxkZBCsA4

The specimen heated in a microwave with 15% water content degraded rapidly. A similar observation was expected for CoRncrete specimen heated in a microwave with 16.66% water content. However, Specimen with higher water content (16.66) shows better performance. Figure 5.6 shows degradation of CoRncrete specimen heated in a microwave with 16.66% water content. Few cracks were formed on the specimen during the heating process. When submerged in water, the cracks appear to fill in until 1 day, potentially due to swelling and degradation of corn starch at the face of crack. On the 3rd day, large crack is observed in specimens. The number of crack increases with time and on the 7th day, CoRncrete is degraded sufficiently.

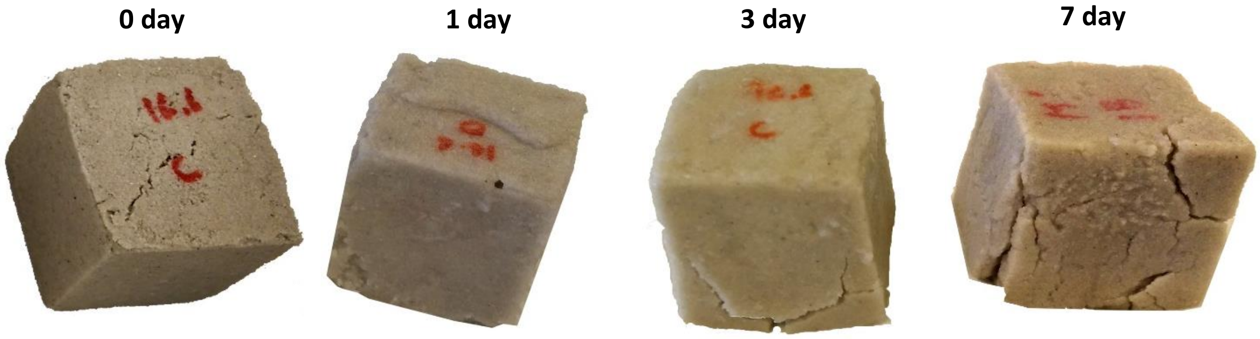
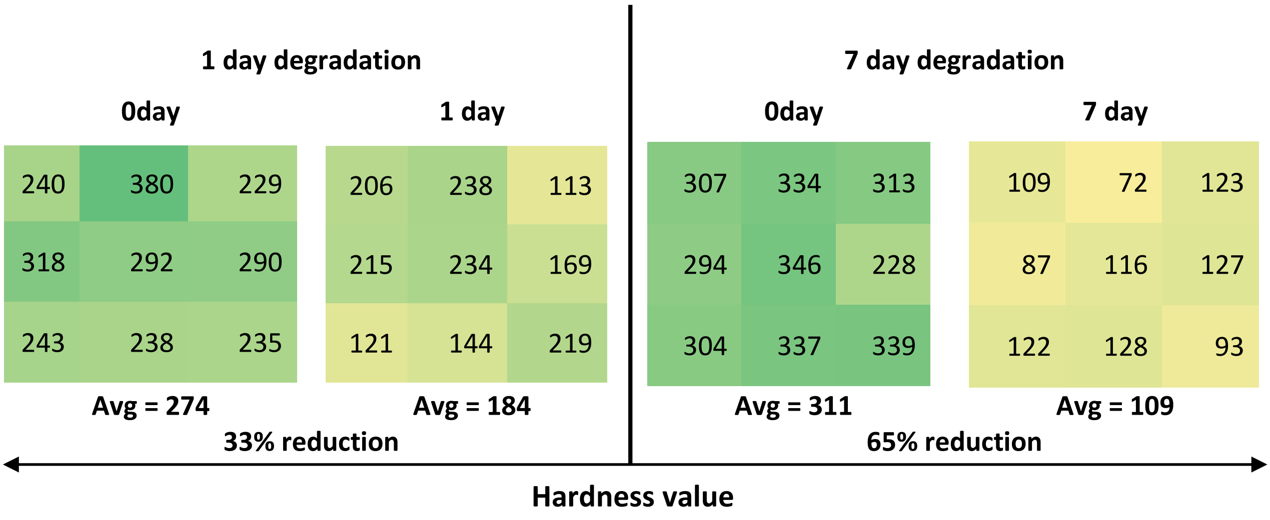


Figure 5.6. Degradation of CoRncrete specimen heated in microwave with 16.66% water content

To quantify the degradation, Equotip hardness number of specimens were measured with time. Figure 5.7 shows a reduction in hardness of 2 specimens, one submerged in water for 1 day and another for 7 days. 9 hardness values on the specimen are averaged to indicate a reduction in hardness. Although, Equotip hardness number is useful to determine compressive strength of a uniform material, compressive strength was not measured for CoRncrete due to unavailability of young modulus value (which is required to calculate compressive strength) and heterogeneity in CoRncrete specimen as a result of crack formation while heating. The crack formation while heating is typical for specimens heated with greater than or equal to 16.66% water content.



1 day degradation

0day

240	380	229
318	292	290
243	238	235

Avg = 274

1 day

206	238	113
215	234	169
121	144	219

Avg = 184

33% reduction

7 day degradation

0day

307	334	313
294	346	228
304	337	339

Avg = 311

7 day

109	72	123
87	116	127
122	128	93

Avg = 109

65% reduction

Hardness value

Figure 5.7. Hardness value indicating the reduction in surface hardness of CoRncrete after degradation in water for 1 and 7 days. In Green scale, the greenish and yellowish region signify high and low hardness value respectively.

The average reduction in hardness value for 1 day and 7 day is 36% and 64% respectively. The hardness value of all specimen is shown in appendix D. Unfortunately, readings of 3rd day degradation are not available.

The Equotip hardness measurement seems not a reliable method to quantify the reduction in strength of CoRncrete in wet environment, but it is successful in indicating the extent of reduction in hardness of the surface of the material.

Based on the results of degradation test of all category of the specimen, degradation seems a function of water content and source of heating. A higher water content in CoRncrete potentially leads to better performance in wet environment. It is already known from chapter 3 that water content affects the gelatinization process of starch. Fully gelatinized starch (expected in case of microwave heating with 16.66% water content) indicate a slower degradation than partially gelatinized starch (expected in case of oven heated specimen with 16.66% water content and microwave heated sample with 15% water content). The water content and heating source significantly affect the gelatinization process, hence the degradation in the wet environment.

5.4. Chapter conclusion

CoRncrete degrades rapidly in a wet environment that was supplied in ESEM test. Under ESEM condition, gelatinized corn starch together with water may result in accelerated degradation potentially due to the magnetorheological behaviour of corn starch in CoRncrete.

CoRncrete specimens heated in a microwave and oven with 15% and 16.66% water content respectively, show degradation as soon as they were placed in water. A complete degradation is visible in 1 day.

For CoRncrete specimens heated in a microwave with 16.66% water content, the average reduction in hardness value measured by Equotip in 1 day and 7 days is 36% and 64% respectively. The Equotip hardness measurement seems not a reliable method to quantify a reduction in strength of CoRncrete in a wet environment, but it is successful in indicating the extent of reduction in hardness of material's surface.

The specimen with 15% and 16.66% water content heated in a microwave and oven respectively degrades completely in 24 hours. Whereas, CoRncrete heated in a microwave with 16.66% water content shows a partial but significant degradation (based on Equotip hardness value) in 7 days. The water content and method of heating affect the gelatinization process, resulting in partial gelatinization at low water content (15%) in a microwave and at optimum water content in oven heating. The extent of gelatinization potentially relates to degradation rate.

6

Sustainability analysis

6.1. Introduction

Traditional building material such as Portland cement concrete and fired clay brick exert a negative impact on the environment in form of higher carbon footprint. CoRncrete is a corn starch based material which is derived from a renewable source. The renewability of raw material in CoRncrete may make it sustainable than concrete and bricks. The sustainability analysis is performed using Life cycle assessment (LCA) study. Environmental performance of CoRncrete, Portland cement and fired clay brick is assessed and compared. LCA is executed by following 4 steps and recommendations to improve the environmental performance of CoRncrete is suggested in the end.

6.2. Production process of corn starch, Portland cement and fired clay bricks

6.2.1. Corn starch

Corn starch is made from corn kernel or seeds of corn/maize. Corn starch can be produced from two methods: Wet milling and dry milling method. Wet milling method is most adopted technique for manufacturing of corn starch. In the production of corn starch by wet milling, corn kernel is converted into slurry which is later processed to corn starch and other starch based materials. The corn starch production process is shown in figure 6.1. The maize crop is a warm season crop that matures in 2-3 months (veggieharvest). Once the maize if fully grown, it is harvested and the kernels from the cob are separated and processed to make starch. The production process of corn starch from corn kernel as reported by Robson et al. (1994), is briefly explained here:

1. **Receiving and steeping**: Corn kernel contains starch, gluten and germ (an embryo of seed which is responsible for reproduction). Shelled and cleaned kernels are placed in steep tanks and soaked in water containing a small quantity of sulphur dioxide for 24 to 48 hours at a temperature of 10^{o}C. This process allows extraction of soluble material from kernel. The sulphur dioxide prevents fermentation and help in separation of starch and protein. After steeping is completed, the steep water is drained from kernels.

2. **Degermination:** Kernels are grounded in attrition mills to loosen the hull. Water is added to mills, creating a mixture of slurry and whole germ. The slurry is placed in hydroclone separator which remove the lighter germ. The germ is then dried and sold in the further process of corn oil and germ meal.

3. **Grinding and steeping:** The remainder of the kernel, including the hull, gluten and starch component is sent through additional series of grinding and screening process. After passing through grind mill hull particles are caught on screen while gluten and starch pass through.

4. **Starch gluten separation:** The remaining of gluten and starch is then separated in centrifugal. The gluten is dried or sold as corn gluten feed. The starch slurry is washed and dewatered using filters and centrifuges.

5. **Production of corn starch:** The starch slurry is passed through a starch dryer resulting in production of dry corn starch. Starch slurry is also used in manufacture of materials like dextrin and modified starch.

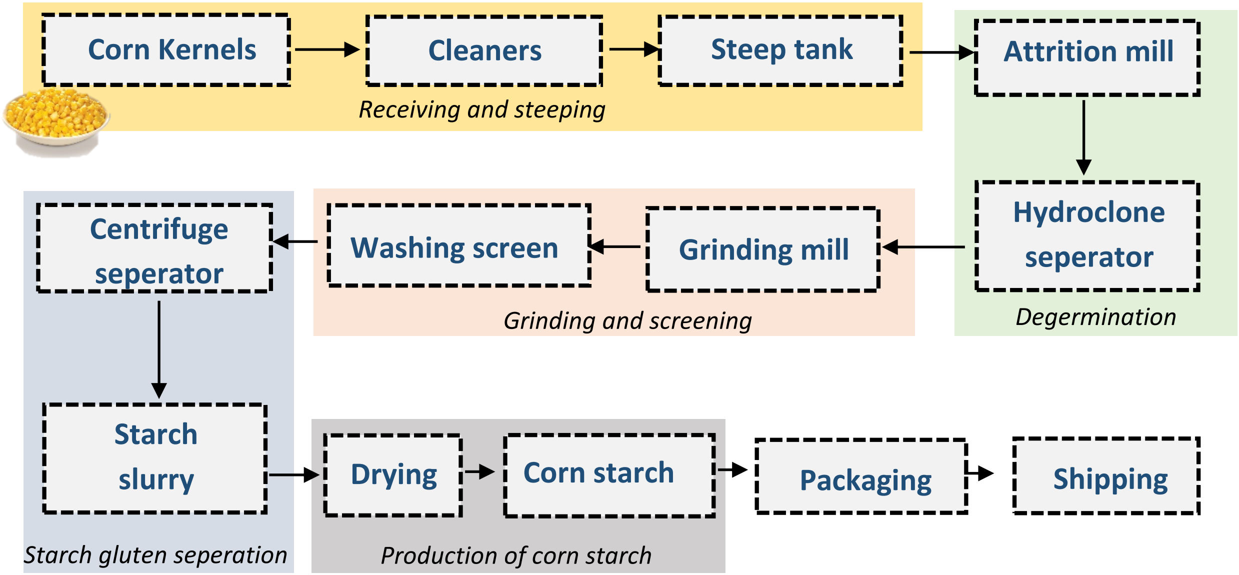


Figure 6.1. Process flow diagram for the production of corn starch. Adopted from Robson et al. (1994)

The amount of gaseous and particulate emission from the whole process is low. However, some amount of sulphur dioxide and odorous vapour emission come out from the steeping tanks, the gluten, feed and germ dryers. The wet milling process explained here is derived from a literature published in 1994. However, Van Ziest, W.J. et al (2012) report similar process indicating no significant change in the overall process of producing starch over the last two decade.

6.2.2. Portland cement

The production of Portland cement is considered energy intensive and it releases gaseous and particulate emissions that are harmful to the environment. The production process of Portland cement is shown in figure 6.2. In brief, the raw material of cement like coral stone, shale, bauxite and iron ore are quarried and processed. The materials are crushed, mixed and later heated in a rotary kiln at greater than 1400^{o}C (Huntzinger, D.N. and Eatmon, T.D. (2009)). The clinker is cooled and gypsum is added to it. The mix is milled into a very fine powder and later packaged for storage and transport.

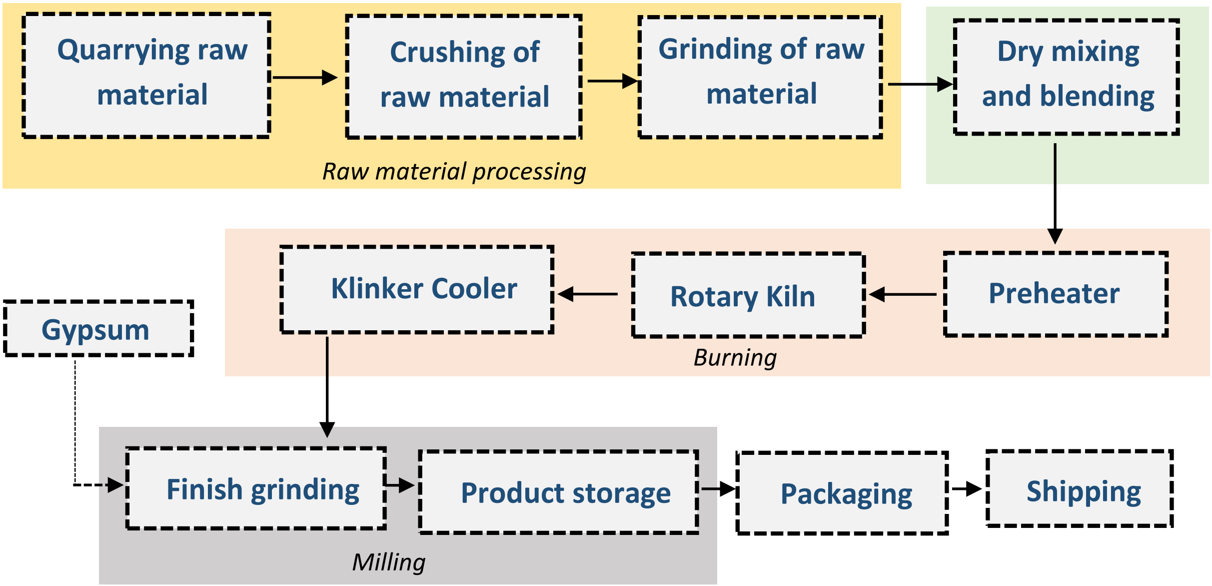


Figure 6.2. Process flow diagram for the production of Portland cement. Adopted from Huntzinger, D.N. and Eatmon, T.D.(2009)

6.2.2. Fired clay brick

Fired clay brick has a relatively simple manufacturing process as compared to corn starch and Portland cement. The production process of brick is shown in figure 6.3. Clay is the major raw material used in manufacturing of brick. According to Kumbhar et al. (2014), Clay with other materials like coal powder or rice husk is mixed with water to form a plastic material. This material can be moulded in different shape manually or automatically and left to dry under sunlight for 3-4 days. Afterwards, the bricks are fired in a kiln at 1200-1300 °C for 24h .The bricks are fired generally with coal or natural gas as fuel.

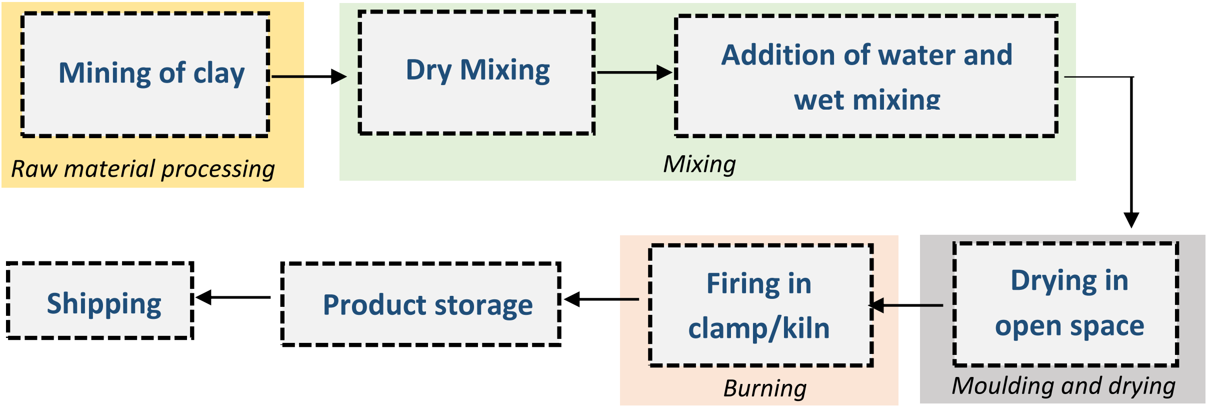


Figure 6.3. Process flow diagram for the production of fired clay brick. Adopted from Kumbhar et al. (2014)

6.3. Life cycle assessment

The life cycle assessment is executed in 4 steps as recommended in ISO 14040:2006, Environmental management - Life cycle assessment – Principles and framework.

6.3.1 Definition of the goal and scope of the LCA

The goals of LCA analysis are:

1. ***To compare the environmental effect of CoRncrete with traditional building material like Portland cement concrete and fired clay bricks, all used as a structural block for indoor application.***

2. ***To identify the life cycle stage of CoRncrete that is contributing most to the environmental impact.***

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The application, functional unit, strength and life selected for CoRncrete, Portland cement concrete and fired clay brick are:

- Application: Structural block for indoor use
- Functional unit: $1m^3$
- Strength: 25MPa
- Life: 50 years

The intended application of all materials is restricted to structural block for indoor use due to rapid degradation of CoRncrete in a wet environment. CoRncrete specimens tested in this research have shown an average compressive strength of 26.66MPa. Although, the size of specimens tested in this research is smaller in comparison to selected functional unit, it is assumed that the size effect does not exist. The fired clay bricks are reported to have a compressive strength greater than 20MPa (Bernadi et al. (2014)). It is important to note that unlike concrete, CoRncrete and brick cannot be manufactured in a larger block of $1m^3$ but in a smaller block of $64cm^3$ and $2000cm^3$ (in general) respectively. The life span of CoRncrete is not known thus it is assumed to survive dry environment for 50years.

The scope or system boundary for LCA is shown in figure 6.4. The blue area defines the boundary of LCA which is limited from processing of raw material ('cradle') to manufacturing of material ('gate').

The LCA study is carryout out based on the product life cycle inventory (LCI) database from Eco-costs V3.3 (Vogtländer, 2012). A list calculated with Simapro 8.01 based on ecoinvent v3 LCI data, is selected for the study. A total of 12 environmental categories are evaluated in this LCA. The environmental categories are expressed as 'cost to environment' in Euros per m^3 of material.

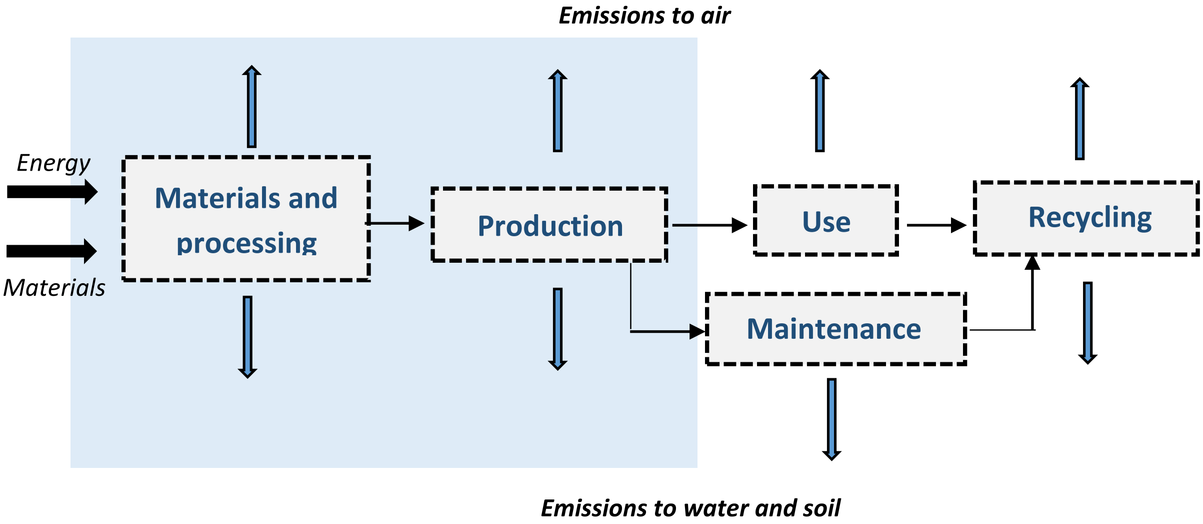

Figure 6.4. Process flow diagram for a material and definition of system boundaries marking the parts targeted in LCA study. Adopted from Vogtländer ,2012

6.3.2 Life cycle inventory analysis

The life cycle inventory analysis (LCIA) is the listing of all environmental relevant input and output in various life cycle stages. This information is already included in Ecoinvent data. The quantity of the various ingredients of CoRncrete and Portland cement concrete is shown in table 6.1. The information on ingredients is important to calculate the eco-cost of various environmental category. The quantity of CoRncrete and concrete is calculated based on the design strength of 25MPa . Ingredients for Portland cement concrete is calculated using ACI method shown in Appendix D. The LCIA for CoRncrete also includes electricity (generated from coal powered thermal plants) required for heating 1m^3 CoRncrete.

Table 6.1. Quantity of various ingredient in CoRncrete, Portland cement concrete and fired clay brick

Ingredients	**Quantity of material(kg/m^3)***		
	CoRncrete	**Concrete**	**Brick**
Corn starch	255	-	-
cement	-	298	-
water	230	185	-
coarse aggregate	-	992	-
fine aggregate	1277	882	-
Bulk density of fresh material	1763	2357	N.A
Density of hardened material	1552	2357	1920
Air %	N.A	2	N.A

6.3.3 Life cycle impact assessment

Life cycle impact assessment of CoRncrete, Portland cement concrete and fried clay brick is shown in figure 6.5. Environmental impacts are expressed in Eco-cost (Euros). Environmental categories; human heath, eco toxicity and resource depletion are further categorized into several environmental categories which sum up to give an aggregated value. The carbon footprint is calculated based on the midpoint environmental category, not presented in the chart.

The environmental categories mentioned are self-explanatory. However, photochemical oxidation, acidification and eutrophication potential are explained here (Sustainability reader by Jonkers, 2015):

Photochemical oxidation potential: Airborne pollutant such as volatile organic compounds (VOCs), carbon monoxide (CO) and nitrogen oxides (NOx) released particularly from diesel vehicle, reacts with sunlight and form reactive compounds such as ozone. These reactive compound are harmful to human health.

Acidification potential: Chemical compound such as SO_2 (sulphur dioxide), NO_x (nitrogen oxides, NO and NO_2), and NH_4^+ (ammonium) are acidic or produce acid when reacting with water. The acid has detrimental effect on soil, ground and surface water, ecosystem and buildings.

Eutrophication Potential: Fertilizers are used in agriculture which are rich in nitrogen and phosphorous compounds. These compound are washed out and end up in ground or surface water source. The water plants and algae are often limited by these nutrients, so they may start to grow in excess quantities, restricting other types of living. This process of excess nutrient loading in the environment is called 'eutrophication'. Due to eutrophication the biodiversity is affected. Sometimes eutrophication causes no oxygen situation in water that result in the death of living organism.

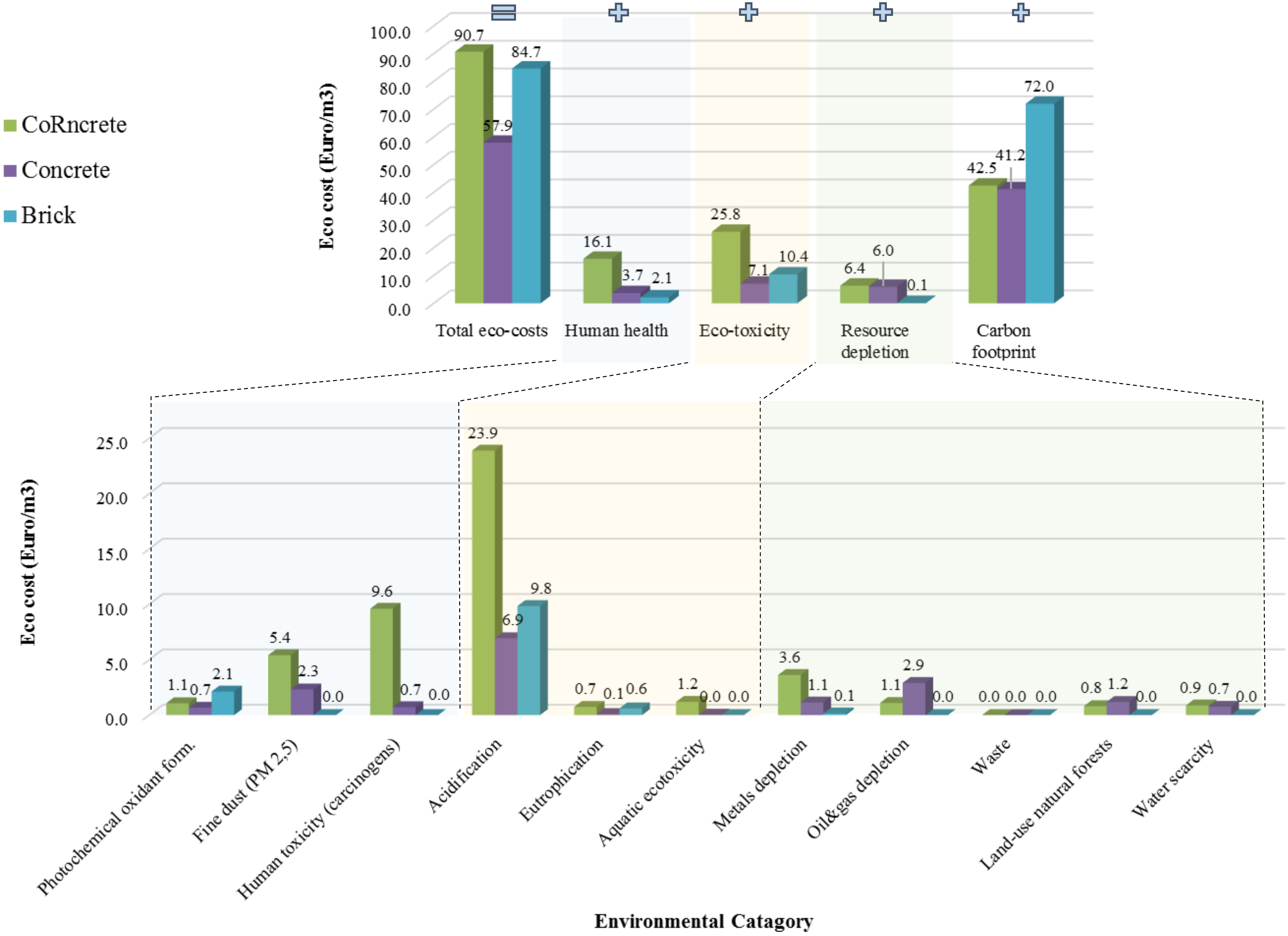

Figure 6.5. Life cycle impact assessment of CoRncrete, Portland cement concrete and fired clay brick

6.3.4 Life cycle interpretation phase

The environmental impact of CoRncrete was expected lower than concrete and brick before the LCA due to its renewable source and low heating temperature, hence low CO_2 emission. Contrary to expectation, LCA shown in figure 6.5 indicates that the environmental impact of CoRncrete is higher than Portland cement concrete and fired clay brick, for given boundary conditions. The total eco-cost of CoRncrete, concrete and brick is 90.7, 57.9 and 84.7 Euro/m^3 respectively. The extended data in Appendix D shows that the corn starch is responsible for 95% of eco-cost of CoRncrete. Other ingredients like sand, water and heating of CoRncrete have negligible impact. The major environmental category responsible for higher eco-cost of CoRncrete is human health and eco-toxicity. CoRncrete has 5 times higher impact on human health and 3 times higher eco-toxicity as compared to concrete. Acidification potential, human toxicity and fine dust are three main environmental categories impacting human health and eco-toxicity. Acidification potential of CoRncrete is 2-3 times higher than that of concrete and brick. Whereas, human toxicity due to the production of CoRncrete is 13 times higher than that of concrete.

The higher eco-cost in these categories is potentially due to chemical fertilizers that are used in production of maize crop (source of corn starch). Low environmental profile of CoRncrete relative to concrete and brick can be due to higher energy, water and nutrients input (fertilizers) during crop growth stage. In this stage, significant energy is used in an overall production process of the crop, and fertilizers are used to increase the yield of crop. Toxic chemical such as nitrogen and phosphorous present in fertilizer have a bad impact on the environment (Sharpley and Menzie, 1987). Kim and dale (2008) studied the LCA of corn starch based ethanol. According to them, use of fertilizer could result in Nitrogen loss from soil (eg. NO_x and N_2O) and might contribute to greenhouse gas emission, acidification, eutrophication and photochemical smog. Higher greenhouse emission and acidification are also observed in LCA of CoRncrete.

It is important to note that the acidification potential is significantly higher as compared to eutrophication (figure 6.5). The use of fertilizer is expected to affect eutrophication potential significantly, which is not observed. Another possibility which may result in higher eco-cost in acidification and human toxicity environmental category is the release of SO_2 during the production of corn starch from the corn kernel. A detailed analysis on the production process and inventory is recommended to confirm the speculations.

One of the major limitation of this LCA is its restricted scope or boundary. The gate to grave phase (use, maintenance and recycling) may significantly affect the environmental performance of the materials. CoRncrete has a potential to be recycled and used again as a structural block. This may give it an advantage and positive environmental points against concrete and brick, which are generally recycled and used as aggregates. Transportation of material from the production centre to application site may play an important role too. In a deserted area, if maize crop growth is possible or it can be transported over short distances, CoRncrete can turn out to perform environmentally better than other materials. Soil property, climatic condition and crop management practice in corn farming also affects the environmental performance (Kim and dale, 2008).

Recommendation for better environmental performance:

Use of green fertilizer can reduce the environmental impact of production of corn starch, making CoRncrete more sustainable in comparison to traditional building materials. Environmental scientists are concerned about use of corn starch in industrial application, especially for the production of ethanol because of its effect on rising price of corn starch. The use of corn starch in such application has resulted in an increase of corn starch price from 0% to over 80% (Condon,et al., 2015). Corn starch is the main ingredient in CoRncrete and use of CoRncrete may significantly increase the price of corn starch in future. Hence, **production of CoRncrete from corn starch is a competition to food and food industry**. One approach to prevent this competition is to **utilize biomass/feedstock from maize crops in the production of starch**. This will prevent the impact on increasing corn starch prices. Additionally, use of biomass will result in positive environmental points or negative eco-cost, making CoRncrete sustainable. The feedstock can be processed in bio-refineries to manufacture starch (Naik et al., 2010).

6.4 Chapter conclusion

The environmental impact of CoRncrete is higher than Portland cement concrete and fired clay brick, for given boundary conditions (cradle to gate). The total eco-cost of CoRncrete, Portland cement concrete and fired clay brick is 90.7, 57.9 and 84.7 Euros/m^3 respectively. The major environmental category responsible for higher eco-cost of CoRncrete is human health and eco-toxicity. CoRncrete has 5 times higher impact on human health and 3 times higher impact on eco-toxicity as compared to concrete. Acidification potential, human toxicity and fine dust are three main environmental categories impacting human health and eco-toxicity. Acidification potential of CoRncrete is 2-3 times that of concrete and brick. Whereas, human toxicity due to the production of CoRncrete is 13 times higher than that of concrete.

The higher eco-cost in these categories is potentially due to chemical fertilizers that are used in production of maize crop (source of corn starch). Another possibility which may have resulted in higher eco-cost in acidification and human toxicity environmental category is the release of SO_2 during the production of corn starch. Other factors like recyclability and transportation may give CoRncrete positive environmental points against concrete and brick. Also, the carbon footprint of CoRncrete is lower than bricks and comparable to concrete.

Use of green fertilizer can reduce the environmental impact of the production of corn starch, making CoRncrete more sustainable in comparison to traditional building materials. Production of CoRncrete from corn starch is a competition to food and food industry. One approach to prevent this competition is to utilize biomass/feedstock from maize crop in the production of starch.

7

Application of CoRncrete

CoRncrete is a novel bio-based material intended to be used in construction as a structural block (like a brick). It can be an attractive construction material due to its light weight (as compared to traditional concrete and fired clay bricks), the rapid gain in strength, good compressive strength and its biodegradability. Moreover, the binding agent, corn starch, is based on a renewable source which can be grown on any fertile land.

Although CoRncrete seems to be an interesting material, its application is mainly limited due to:

1. Rapid to fast degradation in water causing durability issue.
2. High environmental cost making it unsustainable.
3. Competition with food

The aforementioned limitations will be discussed in detail and solution to these limitation will be suggested in following text.

The degradation of CoRncrete in water is a major challenge to overcome. The degradation mechanism of CoRncrete in water is discussed in chapter 5. CoRncete heated in an oven at optimum water content (OWC) shows low water resistance and degrades completely within 24 hours. CoRncrete heated in a microwave with water lower than optimum water content (16.66%) degrades completely within 24 hours, while CoRncrete heated in microwave at OWC has a better resistance to water. The degradation study indicate a possibility to increase the resistance of CoRncrete in water by adopting a higher water content in fresh CoRncrete. However, at higher water content, segregation of constituents in fresh CoRncrete and formation of cracks in hardened CoRncrete leads to increase in heterogeneity, resulting in lower strength. If a method to prevent the aforementioned problems is developed, the durability of CoRncrete in a wet environment can be improved significantly. It is also possible to include hydrophobic additives such as oil in fresh coRncete which may provide water resistant property inherently. External treatment of CoRncrete by soaking in epoxy resin or urethane as adopted in Co2 eco-structure (Imagawa, 2013) can improve its water resistance along with improving strength properties significantly. This option has a setback of increased production cost.

LCA indicate that the CoRncrete has a higher environmental impact than traditional concrete and red clay. This is due to higher eco cost in environmental categories such as eco-toxicity and human health. The higher eco cost in these categories is potential due to the use of chemical fertilizer in production of maize, the source of corn starch. Use of green fertilizer can reduce the environmental impact of the production of corn starch, making CoRncrete more sustainable in comparison to traditional building materials. As explained in chapter 6, use of corn starch in CoRncrete may significantly increase the price of corn starch in future.

Hence, the production of CoRncrete from native corn starch is a competition to food and food industry. One approach to prevent this competition is to utilize biomass from maize in the production of starch.

Production cost of concrete, which is not evaluated in this study, will also play a major role in the marketability of CoRncrete. Upscaling the specimen size to the size of a standard brick is a challenge due to the formation of shrinkage crack in prismatic CoRncrete specimen. Figure 7.1 represent these limitation and possible solutions pictorially.

Future application of CoRncrete (at its present development state) is limited to small sized cubical structural block for indoor use and production of CoRncrete bricks to build houses in deserted areas. CoRncrete may also prove reliable for ground improvement. Its flow ability at optimum water content and transformation at relatively low temperature makes it a potential material for 3D printing of building. A survey carried out as a part of this project indicate that the market for CoRncrete is limited in Netherlands in comparison to India. This is due to special subsidies to contractor and designer in India for using green material (assuming that CoRncrete will be greener in future). Moreover, architects showed interest in this material due to customization of CoRncrete blocks in a variety of shapes and colour.

Figure 7.1. Pictorial representation of limitation of CoRncrete and its possible solutions.

8

Conclusions

"In the end I realized it's not how cool stuff is, but the meaning behind it"
– Dragon warrior

The following conclusions are drawn from this study:

1. The heat induced transformation of CoRncrete is caused due to the gelatinization of corn starch. This gelatinization occurs at 65.6°C and 66.1°C in fresh CoRncrete with 15% and 18% water content respectively. Other processes such as pasting and retrogradation may also play an important role in the formation of CoRncrete.

2. The ESEM scan indicated a possible influence of water content, heating source (heating rate) and duration of heating on gelatinization of corn starch, affecting the microstructure of CoRncrete.

3. The results of cone penetrometer test gave indication of undrained shear strength of CoRncrete at varying water content and an approximate water content range (16.62%-18.06%) where phase of corn starch particles is expected to changes from solid particles (with adsorbed water) to particles in suspension of water. Optimum water content from proctor test (corresponding to maximum dry density) were found to be 16.64% and 17.94% at compaction energy of 22.1 KJ/m^3 and 51.5 KJ/m^3 respectively.

4. A co-relation between the dry density of wet CoRncrete, the density of hardened CoRncrete and strength of hardened CoRncrete was found. Exception in case of microwave heating method was observed where the underlying heating mechanism believe to affect the structure and strength of CoRncrete.

5. Compressive strength upto 26.67 MPa was achieved in the case of microwave heating (water content 15%). Whereas, it was limited to 13.70 MPa (water content (optimum): 16.66%) in oven heating method.

6. Factors such as water content, size of sand aggregates and duration of heating (in a microwave) affected the strength of CoRncrete significantly.

7. When submerged in water, CoRncrete specimen show complete to partial degradation in a day to a week. The degradation process depends on the water content of fresh CoRncrete and source of heating. The specimen with 15% and 16.66% water content heated in a microwave and oven respectively degrades completely in 1 day. Whereas, CoRncrete heated in microwave with 16.66% water content shows a partial but significant degradation (based on Equotip hardness value) in 7 days. The water

content and method of heating affect the gelatinization process which potentially affect degradation rate.

8. CoRncrete has a higher environmental impact as compared to red clay brick and Portland cement concrete. The eco-cost calculated by LCA for per m^3 of CoRncrete, brick and concrete (of 25 MPa strength, life: 50 years), used as a structural block, is 91, 81 and 58 euros respectively. The higher eco-cost of CoRncrete is potentially due to the use of fertilizer that results in higher eco-toxicity and impact human health adversely. The chemical from fertilizer enters the food chain and affects flora, fauna and human health.

9. CoRncrete is an attractive construction material due to its light weight, the rapid gain in strength, good compressive strength, biodegradability and its derivation from renewable sources. However, factor such as durability, high environmental cost and competition with food are the suggested limitations in its present application.

9

Recommendations

CoRncrete is a novel bio-based material whose properties are explored in this research work. CoRncrete rapidly gains strength, has a good compressive strength and is biodegradable. This makes it an interesting material for further exploration. The recommendation for the future study of CoRncrete can be divided into two parts: Recommendations or challenges to realize CoRncrete as a future construction material and recommendation for scientific study and improvement over current research.

Recommendations or challenges to realize CoRncrete as a future construction material

1. CoRncrete has shown a poor environmental performance as compared to traditional construction material potentially due to use of fertilizers and high energy input during the growth period of maize crop. The use of CoRncrete may also result in competition with food, raising cost of corn starch in future. Starch production is also possible with biomass of maize crop. Use of second generation starch in CoRncrete production may result in better environmental performance. An alternative material with low environmental impact and similar heat induced transformation property may also be used in the future study.

2. CoRncrete degrades in a wet environment which raises durability issues. Use of hydrophobic additives is recommended in the future study to improve its performance in wet environment.

3. Upscaling of CoRncrete to a regular size structural block is a major challenge due to the appearance of crack while heating fresh CoRncrete. Some technique needs to be developed in order to prevent the cracking while heating. Use of additive, different heating regime or construction of a special mould are few options that can be tried in future.

Recommendations for scientific study and improvement over current research

Chapter 3. Understanding physical transformation of CoRncrete

1. In order to understand the microstructure and development in the strength of CoRncrete, more ESEM scans can be produced. ESEM imagining of CoRncrete specimens, heated in microwave and oven at various water content, could confirm the hypothesis formed in this work. It is important to maintain similar heating and cooling boundary conditions for the specimen in order to compare the ESEM scans.

2. This study is restricted to gelatinization process in the formation of the microstructure of CoRncrete. It is speculated that pasting and retrogradation may also play an important role. Experiments to investigate pasting and retrogradation properties should be carried out for understanding the complete picture of the microstructural development of CoRncrete.

Chapter 4. Strength of CoRncrete

3. This research utilizes a constant corn starch to sand ratio of 1:5. Various corn starch to sand ratio can be tested and an optimum proportion can be found out experimentally. The corn starch to sand proportion in which all the voids of sand is filled with corn starch can be calculated theoretically and verified with experimental results.

4. The thickness of silicon rubber mould was a limiting factor in casting of the prismatic specimen (160mm×40mm×40mm). The mould should be prepared with sufficient thickness to prevent the lateral deformation of CoRncrete specimens.

5. The tensile strength of CoRncrete is not explicitly addressed in this research. The tensile property of CoRncrete is important in determining its functionality. The tensile strength of CoRncrete could be studied with varying water content.

6. Temperature, duration of heating and amount of fresh air circulation in convection oven may have a strong impact on the gelatinization and compressive strength of CoRncrete. These effects could be studied.

7. The heating scheme for a microwave in this research is selected based on random experiments. The heating scheme can be optimized to find an ideal heating scheme which results in the maximum strength of specimen at the minimum energy required.

8. For better understanding of fracture process of CoRncrete, the weakest link in the specimens could be evaluated experimentally.

9. Cone penetrometer test is carried out on the sand, corn starch and CoRncrete and results are reported based on single reading. Results should be accurately reported based on an average of three readings.

Chapter 5. CoRncrete in wet environment

10. Degradation of CoRncrete is quantified using Equotip which appears less reliable due to heterogeneity in CoRncrete. The degradation should be quantified using a better technique. Hydrophobic additives can be mixed in CoRncrete and their degradation can be compared with normal CoRncrete.

Chapter 6. Sustainability analysis

11. Life cycle assessment (LCA) should be carried out over the complete life cycle of CoRncrete and traditional construction material for better reflection on environmental performance.

Others

12. Corn starch contains glucose which may attract the insect. A study should be carried out in order to study the growth of microbes and attraction of insect towards CoRncrete.

13. Use of waste material like plastic may improve the performance of CoRncrete in water. The addition of waste material may give added value to CoRncrete and also make more sustainable.

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Appendix A

Results of preliminary test

Optimum water content setup

It was speculated through results of first set of experiments that an optimum mix proportion may exist which results in the higher strength of CoRncrete. A setup to determine the optimum mix was prepared (figure A.1). This setup consisted of a metal rod capped with a rubber that was dropped on fresh CoRncrete sample from a specific height. This setup was based on an assumption that the optimum water content is the water content where fresh CoRncrete start showing up impact activated hardening with no excess water on top of sample. The optimum water content for CoRncrete sample with varying water and sand content was found out using this technique. Use of this setup was debatable due to non-scientific measurement approach.

Figure A.1. (a) Cylindrical CoRncrete specimens and (b) optimum water content setup. Optimum water content setup is helpful to indicate the optimum water content of CoRncrete. Water content where the sample shows impact activated hardness and no excess water on top, is the optimum water content.

Effect of corn starch - sand ratio on compressive strength of CoRncrete

The corn-starch sand ratio is one of the important parameter affecting the consistency of fresh CoRncrete and strength of hardened CoRncrete. In this study, a constant corn starch to sand ratio was adopted in order to reduce the variables in testing of CoRncrete. The effect of corn starch to sand ratio on compressive strength of CoRncrete is shown in table A1 and figure A2. The test was carried out on CoRncrete specimen of 40×40×40 mm. It is important to note that the water content in the mix was kept 1.1 which corresponds to 18%. This water content is high as compared to optimum found using cone penetrometer and proctor test. Initially, the optimum water content was found using the setup explained in previous section. The setup overestimated the optimum water content. Thus, the results shown here are not so accurate and the test could have been carried out with water content 15% or 16.66% for better results. Nevertheless, a corn starch to sand ratio of 1:5 was adopted which has resulted in CoRncrete of good strength.

Table A.1. Effect of corn starch - sand ratio on compressive strength of CoRncrete

Sample	Mix proportion (by weight)			Strength (MPa)
	Corn starch	water	Sand	
A	1	1.1	4.75	15.09
B	1	1.1	5	15.38
C	1	1.1	5.3	13.21
D	1	1.1	5.6	11.67

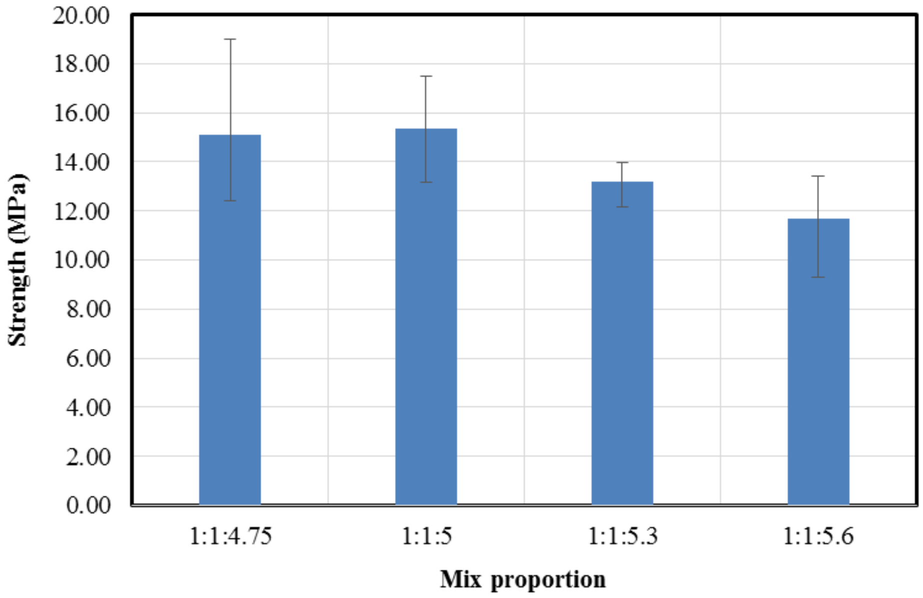

Figure A.2. Effect of corn starch - sand ratio on compressive strength of CoRncrete

Effect of sand grain size and heating mode on compressive strength of CoRncrete.

The effect of sand grain size can be seen in table A.2 and figure A.3. The test was carried out on CoRncrete specimen of 40×40×40 mm. In this testing, a different water content was adopted for mix of different grain size. This was due to measurement of different optimum water content by falling rod setup. With increase in sand size, the compressive strength decreases. A comparison between specimen heated in microwave and oven is also made. Microwave heated sample resulted in higher compressive strength as compared to oven heated sample. It is important to note that the results for effect of sand grain size and heating source are not so comparable due to different water content adopted in making CoRncrete. However, the results indicated that the grain size and heating mode has a significant effect of compressive strength of CoRncrete.

Table A.2. Effect of sand particle size and heating mode on compressive strength of CoRncrete.

Sample	Sand fraction (mm)	Mix proportion (by weight)			Strength (MPa)
		Corn starch	water	Sand	
Microwave	0.125-0.25	1	1.1	5	15.38
	0.25-0.5	1	1.1	5	17.52
	0.5-1.0	1	1	5	13.46
	1.0-2.0	1	1	5	9.48
Oven	0.125-0.25 (0ven)	1	0.95	5	7.69

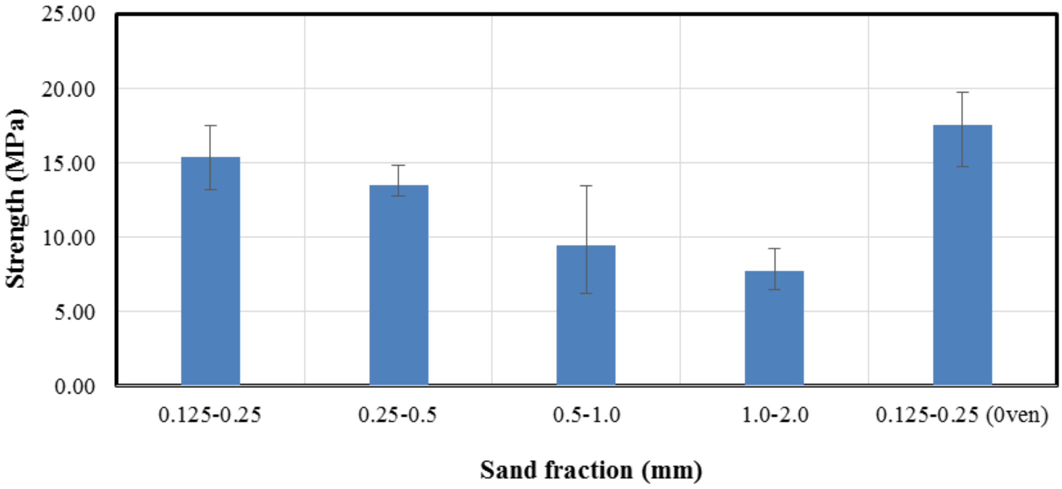

Figure A.3. Effect of sand particle size and heating mode on compressive strength of CoRncrete.

Appendix B

TGA, DSC and ESEM

TGA analysis of corn starch and fresh CoRncrete

TGA analysis of dried corn starch and dry fresh CoRncrete was performed by STA F3, Netzsch, Germany. The result of TGA analysis of corn starch and dried fresh CoRncrete is shown in figure B.1. The heating rate adopted for corn starch was 5°C/min ranging from 25°C to 900°C. The heating rate adopted for (almost dry) fresh CoRncrete was 4°C/min ranging from 25°C to 900°C. The sample were kept in a metal pan and they were heated in Argon environment. The burning temperature of corn starch and CoRncrete was found to be 290°C.

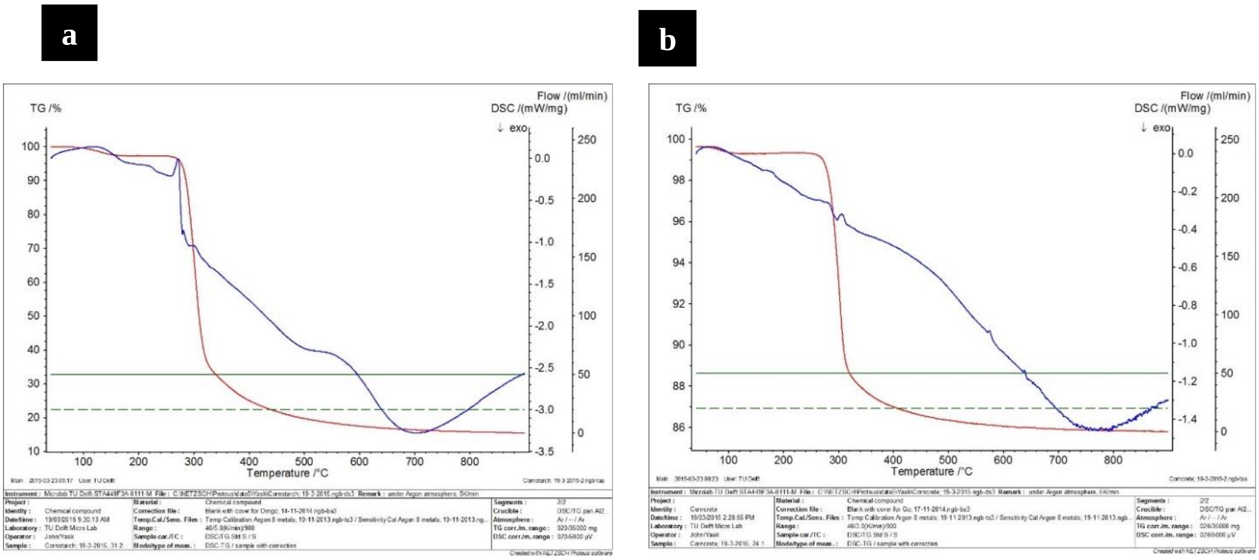

Figure B.1. TGA analysis of (a) corn starch (b) fresh CoRncrete. The burning point of corn starch and CoRncrete was measured to be 290°C.

DSC data of fresh CoRncrete and retrogradation plot

Table B.1 show DSC data for 3 samples and figure B.2 show the plot for retrogradation of fresh CoRncrete sample heated at varying water content. The cooling trend of CoRncrete sample was similar for specimen with different water content. Exception in sample A can be seen as the golden pan in this case was leaking and endothermic energy was provided initially as rate of cooling was faster than the required rate. With increase in water content, exothermic heat flow increases.

Table B.1. Data for samples tested in DSC.

Sample	Water content [%]	Mass before [mg]	Mass after [mg]	Loss [mg]	Loss [%]	Water loss [%]
A	15	18.85	16.69	2.16	11.46	76.39
B	15	17.62	17.60	0.02	0.11	0.75
C	18	23.37	22.87	0.5	2.14	11.89

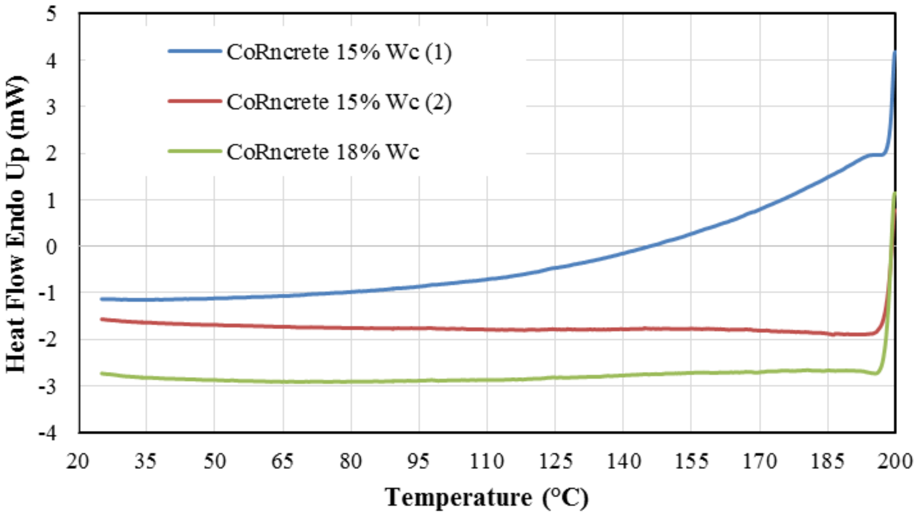

Figure B.2. Retrogradation curve of fresh CoRncrete at varying water content measured from DSC. Cooling rate: 5°C/min from 200°C to 25°C.

Other ESEM Images

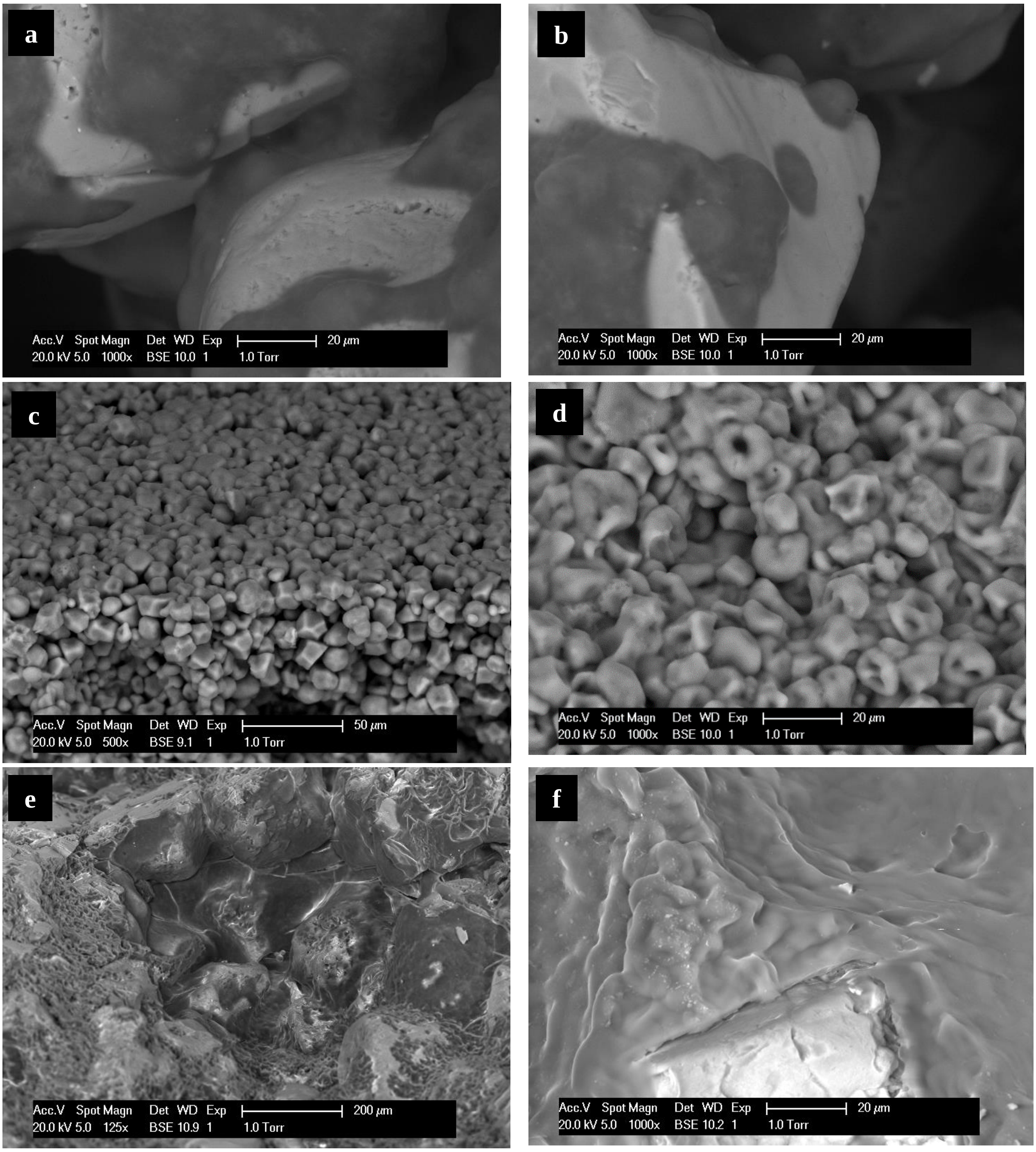

Figure B.3. ESEM image of CoRncrete specimen. (a), (b) DSC specimen A (water content 15%) at 1000x, (c) corn starch with water content 90% heated in oven for 24h at 105oC (500x), (d) corn starch with water content 90% heated in oven for 24h at 105oC (1000x),(e) CoRncrete specimen heated in microwave with 16.66% water content and (f) partially microwave heated sample heated with 16.66% water content.

Appendix C

Cone penetrometer, Proctor test and tensile strength of CoRncrete

Cone penetrometer test on sand, corn starch and CoRncrete and calculation of undrained shear strength

Experimental data of cone penetrometer test on sand, corn starch and CoRncrete is shown in table C.1, C.2 and C.3. The undrained shear strength is calculated by:

$C_{ur} = c \times g \times m / i^2$

C_{ur} is the undrained shear strength of remoulded soil, in kPa;
c is the constant, dependent on the state of the soil and the tip angle of the cone:
c = 0.80 for cones with 30° tip;
g is the acceleration at free fall, in m/s2
m is the mass of the cone, in g;
i is the cone penetration, in mm.

Table C.1. Cone penetrometer test on sand and calculation of undrained shear strength

Mass empty basket [g]	Mass basket + wet sand [g]	Mass basket + dry sand [g]	Moisture content (%)	Density [kg/m^3]	Dry density [kg/m^3]	penetration (5 sec) [mm]	penetration (10 sec) [mm]	penetration (20 sec) [mm]	penetration (30 sec) [mm]	Undrained shear strength (30 sec) [kPa]
48.02	62.33	62.31	0.14	1524	1522	21.5	21.5	21.5	21.5	1.4
44.92	66.03	65.37	3.23	1299	1258	11.6	11.6	11.6	11.6	4.7
43.25	68.90	67.44	6.04	1378	1300	12.2	12.2	12.2	12.2	4.2
45.66	70.05	68.09	8.74	1473	1355	7.8	7.8	7.8	7.8	10.3
46.10	76.11	72.89	12.02	1509	1347	7.5	7.5	7.5	7.5	11.2
45.56	76.92	73.41	12.60	1536	1364	6.8	6.8	6.8	6.8	13.6
49.56	83.21	78.65	15.68	1628	1407	4.4	4.4	4.4	4.4	32.4
47.22	78.85	74.28	16.89	1693	1448	5.7	5.7	5.7	5.7	19.3
45.58	93.34	83.45	26.12	1740	1380	14.4	14.4	14.4	14.4	3.0

Volume of cup: 0.1 L
Empty mass of cup: 0.1147 Kg

Particle density of sand: 2650 kg/m^3

Table C.2. Cone penetrometer test on corn starch and calculation of undrained shear strength

Mass empty basket [g]	Mass basket + wet corn starch [g]	Mass basket + dry corn starch [g]	Moisture content (%)	Density [kg/m^3]	Dry density [kg/m^3]	penetration (5 sec) [mm]	penetration (10 sec) [mm]	penetration (20 sec) [mm]	penetration (30 sec) [mm]	Undrained shear strength (30 sec) [kPa]
42.89	52.10	52.23	0.00	683	693	13.5	13.5	13.5	13.5	3.4
42.82	56.88	53.55	32.43	768	586	3.5	3.5	3.5	3.5	51.2
35.42	45.40	42.25	47.51	816	559	3	3	3	3	69.7
45.06	68.88	58.29	81.44	1100	611	4	4.5	4.7	4.7	28.4
44.79	67.77	57.17	87.01	1171	631	17.8	25.5	30.4	32.2	0.6
48.55	71.59	60.70	91.02	1180	622	21	33.6	40	40	0.4
43.67	74.84	59.77	94.99	1168	604	40	40	40	40	0.4

Volume of cup: 0.1 L
Empty mass of cup: 0.1147 Kg
Particle density of corn starch (calculated from ultra pycnometer test): 1522 kg/m^3

Table C.3. Cone penetrometer test on CoRncrete and calculation of undrained shear strength

Mass empty basket [g]	Mass basket + CoRncrete [g]	Mass basket + CoRncrete [g]	Moisture content (%)	Density [kg/m^3]	Dry density [kg/m^3]	penetration (5 sec) [mm]	penetration (10 sec) [mm]	penetration (20 sec) [mm]	penetration (30 sec) [mm]	Undrained shear strength (30 sec) [kPa]
45.77	69.67	69.71	0.00	1282	1284	16.8	16.8	16.8	16.8	2.2
44.80	65.86	65.11	3.86	1276	1231	18.3	18.3	18.3	18.3	1.9
43.65	77.28	75.16	6.90	1066	999	16.8	16.8	16.8	16.8	2.2
47.60	74.24	71.76	10.43	1157	1049	6.2	6.2	6.2	6.2	16.3
35.41	64.16	60.74	13.67	1191	1049	16.8	16.8	16.8	16.8	2.2
48.56	91.67	85.58	16.62	1526	1310	11	14.4	17	19.3	1.7
50.23	95.88	88.95	18.06	1797	1525	17.1	25.4	33.3	40	0.4
44.87	107.00	96.62	20.23	1792	1493	30.6	40	40	40	0.4
45.67	104.46	93.08	24.17	1749	1410	40	40	40	40	0.4

Volume of cup: 0.1 L
Empty mass of cup: 0.1147 Kg
Particle density of corn starch (calculated by mean averaging): 2460 kg/m3

Calculation of compaction energy of hammer used in Proctor test

The compaction energy of hammer used in proctor test is shown in table C.4.

Table C.4. Calculation of compaction energy

Rammer type	Weight of Hammer(g)	Height of Drop (cm)	Number of drops per layer	Number of layers	Mould diameter (m)	Mould height (m)	Mould Volume (dm3)	Calculated energy (kJ/m3)
Heavy	350	20	25	3	0.105	0.1155	1.0001	51.5
Light	150	20	25	3	0.105	0.1155	1.0001	22.1

Result of proctor test expressed in weight percentage of corn starch

The result of proctor test is shown in table C.5. The water content in corn starch is expressed in water/corn-starch ratio and weight percentage of corn starch. The results indicate that in case of low weight hammer compaction, at maximum dry density, the weight percentage of corn starch is 50%. This value is close to 52.5% weight percentage of corn starch as measured by Crawford et.al (2013) in their experiments with corn starch and water.

Table C.5. Result of proctor test expressed in weight percentage of corn starch

Fresh CoRncrete - 150gm rammer				**Fresh CoRncrete - 350gm rammer**				**sand - 150gm rammer**		**sand - 350gm rammer**	
Moisture content [%]	**m_water/ m_starch [%]**	**m_starch/(m_ water+ starch) [%]**	**Dry density $[kg/m^3]$**	**Moisture content (%)**	**m_water/m_starch [%]**	**m_starch/(m_ water+ starch) [%]**	**Dry density $[kg/m^3]$**	**Moisture content (%)**	**Dry density $[kg/m^3]$**	**Moisture content (%)**	**Dry density $[kg/m^3]$**
0.00	0%	100%	1550	**0.00**	0%	100%	1589	**0.00**	1557	**0.10**	1568
2.77	17%	86%	1375	**2.75**	17%	86%	1440	**3.22**	1407	**2.88**	1491
6.15	37%	73%	1271	**5.70**	34%	75%	1344	**6.81**	1415	**6.26**	1504
9.26	56%	64%	1223	**8.95**	54%	65%	1351	**9.60**	1397	**9.80**	1479
13.00	78%	56%	1189	**12.58**	75%	57%	1285	**13.36**	1403	**13.35**	1495
16.64	100%	50%	1589	**16.16**	97%	51%	1470	**18.95**	1402	**18.01**	1487
18.11	109%	48%	1554	**17.94**	108%	48%	1547	**19.86**	1394	**19.03**	1500
19.46	117%	46%	1532	**19.40**	116%	46%	1529	**22.66**	1435	**21.22**	1487
22.36	134%	43%	1447	**20.86**	125%	44%	1534	**22.98**	1506	**22.03**	1514

Tensile strength of CoRncrete

The tensile strength result of CoRncrete is shown table C.6. Tensile strength was carried out on 160×40×40mm prismatic beams. The results are not reliable due to formation of cracks in the CoRncrete while heating. A significant volume loss while heating was measured for oven heated and microwave heated sample (water content 15%). A higher deviation in results is seen in microwave heated specimen due to formation of shrinkage crack in specimen with 15% water content and various crack formed due to infiltration flow in 16.66% water content specimen. The tensile strength of CoRncrete measured for specimen heated with16.66% water content in microwave is higher than specimen heated in microwave with15% water content. Whereas in compressive strength results, 15% water content specimen showed higher strength than 16.66% water content. The tensile strength of specimen heated in microwave is higher than that of heated in oven. Image of one specimen from each category is shown in figure C.1.

Table C.6. Tensile strength test result of CoRncrete

Source of heating	Water content [%]	Density[kg/m^3]	Volume loss [%]	Strength [MPa]	Average tensile strength [MPa]
Oven	16.66	1550	-19.13	1.17	1.18
		1605	-2.04	1.10	
		1650	-5.14	1.27	
Microwave	15	1868	24.85	3.73	3.97
		1773	11.86	1.90	
		1829	18.72	6.29	
	16.66	1644	-8.61	4.80	6.65
		1518	-31.16	8.17	
		1547	-26.35	6.98	

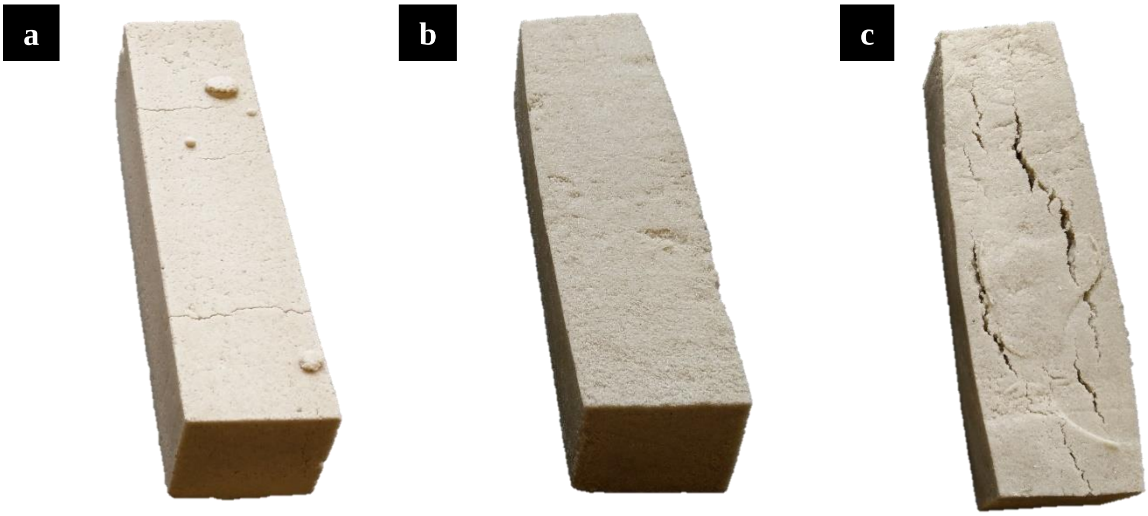

Figure C.1. CoRncrete prismatic specimen (a) Specimen with 16.66% water content heated for 24h at 105ºC in oven, (b) Specimen heated in microwave with 15% water content and (c) Specimen heated in microwave with 16.66% water content

Appendix D

Degradation and sustainability of CoRncrete

Equotip measurement of reduction in hardness of CoRncrete submerged in water

The result of degradation test of CoRncrete specimen heated in microwave with 16.66% water content is shown in table D.1. The specimens were submerged in water for 1, 3 and 7 days. Unfortunately, the results of 3 day is not available.

Table D.1. Equotip measurement showing reduction in hardness of CoRncrete specimen in wet environment, expressed in hardness number.

Source of heating	Water content in sample	Day	Initial [Hardness number]	After [Hardness number]	Reduction in hardness [%]	Average Reduction in hardness [%]
Microwave	16.66%	1	274	228	33	
			309	153	26	36
			305	109	50	
		3	305			
			287			
			323			
		7	311	109	65	
			324	135	58	64
			303	97	68	

Mix design of concrete according to ACI (American concrete institute) method 211.9-91

Desired strength = 25MPa,
Since OPC is used, from table of relation between w/c ratio and average compressive strength of concrete
The estimated water/ content ratio: 0.62

For a slump of 50mm, 20mm maximum size of aggregate, for a non-air entrained concrete, Mix water content is 185Kg/m^3
Weight of cement = 185/0.62 = 298.4 Kg/m^3

For 20mm course aggregate of finess modulus of 2.80, the dry roded bulk volume of coarse aggregate is 0.62 per unit volume of concrete

Weight of coarse aggregate = 0.62*1600=992Kg/m^3

The estimated density of fresh concrete for 20mm maximum size of aggregate for non-air entrained concrete = 2355kg/m^3

Weight calculation of Fine aggregate is shown here:

Ingredients	Specific gravity	volume of ingredients [cm^3]
cement	3.15	94726.06
water	1.00	185000.00
coarse aggregate	2.70	367407.41
Air (2%)	-	20000.00
total		667133.47
fine aggregate	2.65	332866.53

For specific gravity of 2.65,
Weight of fine aggregate = 332866.53*2.65/1000 =882.10 Kg/m^3

Finally, list of materials with respective quantity:

Ingredients	Quantity [kg/m^3]	ratio
cement	298.39	1.00
water	185.00	0.62
coarse aggregate	992.00	3.32
fine aggregate	882.10	2.96

Eco-cost calculation of CoRncrete, Portland cement concrete and fired clay brick

Table D.2 show the eco-cost calculation of CoRncrete, Portland cement concrete and fired clay brick. The eco-cost of individual ingredient is calculated and summed together for final values. It is observed that corn starch is responsible for 95% of total eco-cost in CoRncrete. Also, Portland cement is responsible for 84% of total eco-cost in concrete.

Table D.2. Eco-cost calculation of CoRncrete, Portland cement concrete and fired clay brick

Material	Ingredient	Code	Quantity	unit	Material as mentioned in Eco-invent	Total eco-costs [Euro]	Human health [Euro]	Eco-toxicity [Euro]	Resource depletion [Euro]	Carbon footprint [Euro]
CoRncrete	corn starch	A.010.06.219	255.5	kg	Maize starch {GLO}| market for | Alloc Def, S	85.9	15.7	25.4	4.1	40.7
	water	A.150.01.202	229.9	kg	Tap water, at user {Europe without Switzerland}| market for | Alloc Def, S	0.1	0.0	0.0	0.1	0.0
	sand	A.040.09.105	1277.3	kg	Idemat2014 Sand	4.0	0.3	0.2	2.2	1.2
	electricity	B.030.01.103	13	MJ	Idemat2014 Electricity UCTE coal	0.7	0.0	0.1	0.0	0.5
			1	**m3**		**90.7**	**16.1**	**25.8**	**6.4**	**42.5**
Concrete	cement	A.040.01.102	298.4	kg	Idemat2014 Cement (Portland)	48.4	2.9	6.1	1.2	38.1
	water	A.150.01.202	185	kg	Tap water, at user {Europe without Switzerland}| market for | Alloc Def, S	0.1	0.0	0.0	0.1	0.0
	fine aggregate	A.040.09.105	882.1	kg	Idemat2014 Sand	2.8	0.2	0.2	1.5	0.8
	coarse aggregate	A.040.09.104	992	kg	Idemat2014 Gravel	6.6	0.6	0.8	3.1	2.2
			1	m3	Concrete	**57.9**	**3.7**	**7.1**	**6.0**	**41.2**
Brick	Brick	A.040.03.101	1920	kg	Idemat2014 Red Clay Brick, for housing and roads, packed	**84.7**	**2.1**	**10.4**	**0.1**	**72.0**