

**EXAMINING THE RHEOLOGY AND SHRINKAGE PERFORMANCE OF
LOW CARBON CONCRETES**



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ABSTRACT

Construction globally consumes a great number of building materials such as steel, timber and aluminium. Concrete is one of the most commonly used materials. However, the production of concrete releases a significant emission, i.e. carbon dioxide, which has a huge impact on the environment. Some alternatives have been introduced to create low carbon concretes because of their less carbon footprint characteristic.

Three representatives are fly ash, ground granulated blast-furnace slag (GGBS), limestone. The differences between these materials in physical and chemical aspects change the behaviour of rheology and shrinkage at an early age. This Bachelor's thesis outlines the characteristics of each material mentioned previously and considers their influence on the mix design of concrete. Instead of concrete, mortar mixtures were tested to simplify the effect of coarse aggregate on the overall result. In addition, the samples could be handled easily within a relatively small quantity.

It was found that the addition of GGBS and limestone resulted in the reduction of yield stress and viscosity owing to its high fineness in terms of rheometry (rheological behaviour). Fly ash, with a larger particle size than Portland cement, deduced the reverse trend by increasing flow resistance. Regarding the shrinkage results, GGBS itself made the sample shrink more. But when it is combined with fly ash, the result reversed. Limestone with less than a certain percentage of the total weight of the mixture showed a trivial change.

Keywords fresh concrete, rheology, shrinkage, pozzolanic materials

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1 INTRODUCTION

1.1 Background of study

Concrete has often been considered as solid stone functioning as a structural load-bearing element. However, to obtain the designated strength of concrete after curing process, it is crucial to provide dedicated care during setting time. In practice, most of the construction process emphasizes the durability and strength of concrete rather than the workability including satisfying flowability in formworks. Construction contractors often ignore the importance of handling concrete so that concrete can be cured properly. Hence, factors that affect fresh concrete, such as the use of different materials, ambient conditions, and the addition of admixtures, should be taken into consideration for obtaining the required qualities.

It is commonly known that concrete is liquid enough to flow and fill a mould after being mixed. It contains organic or mineral suspended particles in water and becomes solid and gain strength over time. However, unlike water or oil, fresh concrete behaves differently under applied stress. There are several factors influencing the workability and consistency of concrete and this report will mainly focus on the effect of different pozzolanic cements on rheometry and shrinkage.

Concrete, after mixing, cannot level itself when it is poured into a mould (excepting self-compact concrete). The material not only flows by gravitational effect but also by vibration which helps to ensure that the particles of concrete are distributed evenly. This prevents reducing strength by segregation. In the experiments of this report, fresh concretes are able to flow under their own weight without vibration since aggregates contained are of grained sizes.

As the second most consumed substance on Earth after water with the consumption of 3 tonnes per person per year; the industry of concrete, in fact, accounts for around 7% of global carbon dioxide emissions (Harvey, 2018). The manufacture of cement is immensely energy consuming and emission intensive because of the heat required at 1450°C in a kiln (Neville, 2011).

1.2 Aims and Objectives

1.2.1 Overall aim

The main aim of the project is to give revision of the mix design of different cement types and the main characteristics of materials associated with early age properties. Two important properties are rheology and shrinkage. Based on the

results acquired from laboratory tests, the impacts of each material as well as their combination were observed.

1.2.2 Specific objectives

Portland cement (CEM I) was partially replaced by other materials to create a low carbon concrete. This examination was considered due to sustainability concern of construction industry nowadays. The range of materials was also characterised so that the manufacturing process can be executed. The effects of fly ash, limestone and ground granulated blast-furnace slag have been studied for a long time. However, the behaviour of shrinkage and rheology during the early age have been underestimated over the strength and durability, which perform after concrete has hardened. This report gives a general observation of these early properties based on material characteristics.

Mortars were examined as a simple version of concrete where the smaller size of aggregates were introduced. Yield stress, plastic viscosity, and shrinkage performance were demonstrated via empirical data with technical engineering analysis. The tests include 1D laser shrinkage, viscometer and a few tests of material characterisation.

Literature review and material used were studied to identify the essential properties of each cementitious material and then to obtain overall parameters of combined mixtures. The mix design was given according to the standard of cement types. The changes in proportions can demonstrate the effects of supplementary materials in rheological and shrinkage behaviours.

1.3 Outline of thesis

The thesis is structured in 5 chapters including the introduction in Chapter 1. Chapter 2 reviews literature relating to materials used for the tests and briefly discusses the concept of rheology and shrinkage of fresh concrete. Low carbon concrete can be understood as sustainable concrete that differs from ordinary concrete. A comparison of material in physical and chemical properties was included for approachable analysis later on. The factors affecting rheology and shrinkage are the main concern in interpreting test results.

Chapter 3 outlines the work carried out using laboratory tests. These tests include PSD and XRF analysis for individual properties of cement, viscometer for rheology examination, and thin-layer measurement and cone test for shrinkage configuration. The results of each test were discussed simultaneously.

Chapter 4 outlines the results obtained from the tests in chapter 3 and further discussion was added., including a number of graphs and tables.

2 LITERATURE REVIEW

The literature review uses a number of books, articles and digital sources for an overall revision of material characteristics as well as basic concepts of rheology and shrinkage. The scientific features and observations were collected from reliable sources of other studies and research. Low carbon concrete and its early-age properties were described. The results of tests can be explained based on these properties, but the empirical data might be considered for various factors affecting the performances.

2.1 Low carbon concrete

Concrete originally is made by mixing three main materials: cement, water, and aggregate. Although Portland cement has been used widely since Joseph Aspdin took out the patent for this material in 1824 in England (Neville A. M., 2011), other materials are recently being added in the mixture in order to gain certain properties. These materials create less carbon footprint than Portland cement does.

2.1.1 Portland Cement

Pure cement is Portland cement within less than 5% of another inorganic material(s). Therefore, cement consisting of Portland cement and one or more appropriate inorganic materials can be called blended cement. Portland cement contains some compounds created by the interaction of mainly lime, silica, alumina, and iron oxide in the kiln. These compounds are known as C3S (tricalcium silicate- $3\text{CaO}\cdot\text{SiO}_2$), C2S (dicalcium silicate- $2\text{CaO}\cdot\text{SiO}_2$), C3A (tricalcium aluminate- $3\text{CaO}\cdot\text{Al}_2\text{O}_3$), C4AF (tetracalcium aluminoferrite- $4\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_3$). The majority of cement is constituted by C3S which, along with C2S, critically influences the rate of hydration (Britannica, 2019). The initial stage, or so-called dormant period, is extremely important in the placement of cement before hardening.

The main problem of Portland cement is the environmental impact of production process: emission (i.e. carbon dioxide), dust, coal trains, etc. discharged by combustion. The extreme heat at 1450°C in a kiln is generated by burning fossil fuels and limestone. Furthermore, it is difficult to provide appropriate curing conditions and concrete is subjected to cracking.

2.1.2 Pozzolanic Materials

Pozzolanic materials are siliceous or siliceous-aluminous materials which will react chemically with calcium hydroxide at an ambient temperature to form compounds with cementitious properties (according to ASTM Standard C 618-80) (Alonso, J.L. and Wesche, K., 1991). These Portland cement substitutions became widely known because of their advantages in sustainability, durability and financial aspects. There are, for example, pulverized fuel ash (fly ash and bottom ash), ground granulated blast-furnace slag (GGBS), limestone and other pozzolanic material. Their effects on concrete performance have been studied to ensure appropriate properties in the construction process.

Concrete made with GGBS, fly ash or limestone sets more slowly than concrete made with ordinary Portland cement, depending on the proportion of cementitious material, but also continues to gain strength over a longer period in production conditions. This results in lower heat of hydration and lower temperature rises, and makes avoiding cold joints easier, but may also affect construction schedules where the quick setting is required. (Suresh, D. and Nagaraju, K., 2015). **Pozzolanic reaction** is when Portland cement is exposed to water and hydrates, the products include calcium hydroxide (Ca(OH)_2 -portlandite) reacting to pozzolanic materials to form calcium silicate hydrate (C-S-H).

2.1.3 Ground Granulated Blast-Furnace Slag- GGBS

GGBS is a by-product from blast furnaces used for iron production. The iron ore is reduced to iron and the remaining materials form a slag that floats on top of the iron. When the slag is collected periodically as liquid, it has to be quenched in large volumes of water. The quenching makes the optimal cementitious properties and produces granules similar to coarse sand. This granulated slag is then dried and ground to a fine powder called GGBS or Slag cement (Figure 1). It cannot be used alone to make concrete but can contribute the highest proportion among other pozzolanic materials.

GGBS content ranges from 30-70% of total cement in the production of two major types of pozzolanic cement for ready-mixed (mostly) or site-batched durable concrete (Suresh, D. and Nagaraju, K., 2015):

- Portland Blast furnace cement
- High-slag blast-furnace cement

The differences in rheological behaviour between GGBS and Portland cement may enable a small reduction in water content to achieve equivalent consistency class.

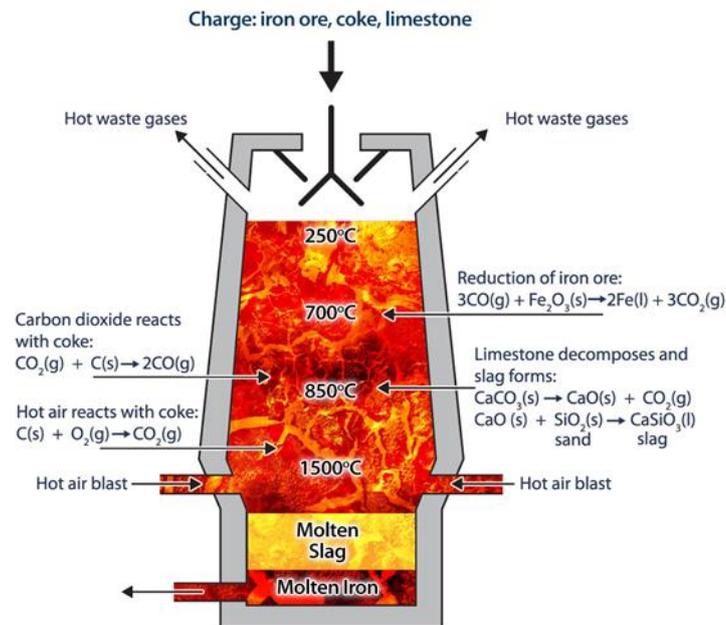


Figure 1. The blast furnace production

Advantages and disadvantages

The use of GGBS brings numerous advantages, such as:

Durability: The strength of concrete does not degrade.

Better **workability:** making placing and compaction easier as well as less risk of cold joints (particularly in warm weather).

Lower early age **temperature rise:** reducing the risk of thermal cracking in large pours.

Chemical attack: elimination of the risk of damaging internal reactions such as alkali silica reaction (ASR) and high resistance to damage by sulfate and reinforcement corrosion.

Environmental benefits: as by-product and waste, GGBS is one of the greenest of construction materials, specifically in the reduction of carbon dioxides and primary energy use as well as quarrying and landfill saving.

On the other hand, there are several problems encountered when choosing GGBS as a supplementary in cement. First of all, the increase in GGBS portion leads to the fact that early **strength** development is slower, but it gains more durability at late stage. Higher **dry shrinkage** at the later stage compared to Portland cement is another drawback of GGBS due to the higher proportion of mesopore. It was also found that high surface area and pore volume (pore sizes of 1–40 nm) contributed to high values of total drying shrinkage (Juenger & Jennings, 2002).

Properties of GGBS

The chemical composition of slag depends on the composition of the raw material in the iron production process. The percentage of GGBS in cement is determined based on the use of concrete in specific purposes such as ground structures (20-30%), underground structures (30-50%) and strict temperature requirement structures (50-65%) (Suresh, D. and Nagaraju, K., 2015).

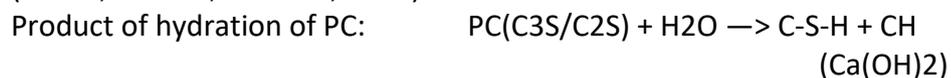
Physical properties

The specific surface area of GGBS is about 400-600m²/kg. It can reach to 600 m²/kg for prestressed concrete bridge superstructures (SAGAWA, Yamamoto, & HENZAN, 2009). The physical properties are specific gravity 2.9, bulk density 1200 kg/m³ and fineness 350 m²/kg. In order to use GGBS as a cementitious material, the granulated slag must be ground finer than cement.

Chemical properties

Typical elements of GGBS are calcium oxide (40%), silica (35%), alumina (13%), and magnesia (8%). A flux containing a mixture of limestone and forsterite or dolomite (in some cases) is combined with silicate and aluminate impurities from the ore and coke (Łukowska & Salih, 2015).

Although GGBS is a hydraulically latent material, in the presence of lime contributed from cement, a secondary reaction involving glass components (Calcium Alumina Silicates) set in. As a consequence of this, cementitious compounds are formed. They are categorized as secondary C-S-H gel. The interaction of GGBS and Cement in the presence of water is described as follows (Mittal, Kaisare, & Shetti, 2006):



2.1.4 Fly Ash

Pulverized fuel ash (PFA) is a solid, fine-grained material resulting from the combustion of pulverized coal in power station furnaces before the flue gases emitted. Large amounts of fly ash and bottom ash are produced during the process of generating electrical power including high-temperature combustion, dry combustion and fluidized-bed combustion (Alonso, J.L. and Wesche, K., 1991). The term of bottom ash, which is collected from the bottom of the boilers, is not applied to fly ash collected in mechanical or electrostatic separators.

Based on the types of production, fly ash can be sub-divided into two categories (other forms of classifications exist too):

- Class F as produced by burning anthracite or bituminous coal
- Class C as produced by burning lignite or sub-bituminous coal

The properties of these two types can be distinguished by the addition of cementitious properties in class C along with the properties of pozzolanic materials in class F.

Fly ash is either produced in a dry state in coal plants for direct use or from stockpiled FA overproduction which is mainly in landfills across the UK and stored in a moist state. FA has several main applications in the cement sector as clinker addition (kiln feed or inter-ground); in concrete sector as cement addition (pre-cast and pre-mix concrete- type II addition); in landfill as soil for woodland or filling for land reclamation; or other smaller-scale sectors such as soil amelioration; the ceramics industry; catalysis; recovery of cenospheres, unburnt carbon and magnetic spheres; and in zeolite synthesis (these applications are out of scope for this research).

Advantages and disadvantages

Fly ash brings numerous advantages over traditional Portland cement as follows (Sacha Alberici, 2017):

Reduction of CO₂ emissions: as FA reduces the amount of clinker used in cement and concrete, the amount of CO₂ discharged will be consequently reduced. It also means a reduction in the environmental footprint of industrial productions.

Reduction in the use of virgin material: the use of raw materials, such as limestone and clay used to produce clinker, are reduced.

Reduction of waste: FA is one of the most abundant industrial by-products on Earth with low embodied energy and this material can be reused or recycled.

Reduction of costs: As a substitute for clinker and as replacement of cement FA reduces the costs of cement since FA is on average less expensive than clinker and cement. According to the stakeholders interviewed, the use of FA can reduce the amount of clinker per tonne of cement from 850 kg to 600 kg.

Increased Quality: the main driver nowadays is the contribution of FA to the hardness of concrete through hydraulic and pozzolanic activity.

However, there are some barriers hindering FA usage in, particularly concrete production. Firstly, the **availability** of FA is not constant during seasons. It has been researched that fly ash is overproduced in winter and underproduced in summer due to sudden changes of wholesale gas prices (UKQAA, 2019) or, in other words, the demand of power consumption. Secondly, fly ash applications may face resistance from traditional builders due to its tendency to effloresce as well as the concern of **freeze/thaw** performance; and small builders may not be familiar with FA product. Additionally, **slower strength** gain, increased need for air-entraining admixtures and salt scaling are the disadvantages of FA worth to consider. Standard specifications for fly ash for use in concrete is stated in BS EN 450-1:2012.

Properties of fly ash

As fly ash is a waste material from burning pulverized coal, its chemical and mineralogical composition depends upon the characteristics and compositions of the coal burned in the power plant. It can either be siliceous or calcareous and consists primarily of glassy spheres with some crystalline matter and unburnt carbon (indicated by loss on ignition).

Physical properties

Fly ash is predominantly made from the high temperature combustion process. The remaining fine particles escape to electrostatic precipitators in form of fly ash. The particle is mainly spherical, irregular or angular, depending on the nature and granulometry of the burnt coal and combustion condition of the power plants, with a small amount of hollow contribution (5% by mass). The range of particle sizes of fly ash is mostly between 1 and 100 μm - silt-sized with some clay-sized fraction; and that of bottom ash is up to 200 μm – sand-sized with some silt-size fraction. Grain sizes over 125 μm are very porous (Alonso, J.L. and Wesche, K., 1991). The specific surface is usually between 250 and 600 m^2/kg (Blaine method) which leads to the high tendency of calcium hydroxide reaction (Neville A. , Cementitious materials of different types, 2011). According to the CEMBUREAU Technical Committee, fly ash must have a specific surface area of not less than 270 m^2/kg with no higher than $\pm 50 \text{ m}^2/\text{kg}$.

Chemical properties

FA is particularly rich in SiO_2 , Al_2O_3 , and Fe_2O_3 , and also contains other oxides such as CaO , MgO , MnO , TiO_2 , Na_2O , K_2O , SO_3 , etc. The high content of CaO (15 to 40%) may cause unsoundness in mortar and concrete, and potentially affect their hydraulic properties (Alonso, J.L. and Wesche, K., 1991).

The carbon content, which is obtained by the presence of carbonates when fly ash is burnt at about 1000°C in fly ash determines the water requirement for mortar and concrete applications. The higher the carbon content, the greater the amount of water necessary to obtain a paste of normal consistency. To state differently, the better fly ash contains less carbon. Class F fly ashes may have a higher value of carbon percentage than those belonging to Class C.

Fly ash does not react with water directly. The silica present in the fly ash reacts with calcium hydroxide (Ca(OH)_2), produced during the hydration of cement, and the principal product of the reaction is calcium silicate hydrate (C-S-H).

Cement Reaction (hydration): $\text{C}_3\text{S} + \text{H}_2\text{O} \rightarrow \text{C-S-H} + \text{Ca(OH)}_2$

Pozzolanic Reaction: $\text{Ca(OH)}_2 + \text{SiO}_2 + \text{H}_2\text{O} \rightarrow \text{C-S-H} (3\text{CaO} \cdot \text{SiO}_2 \cdot 4\text{H}_2\text{O})$
(silica from ash constituents).

2.1.5 Limestone

Limestone is a collective term for a group of sedimentary rocks that consist of at least 50 percent calcite, a mineral formed of calcium carbonate. If some of the calcium becomes replaced by magnesium, the resulting calcium magnesium carbonate rock is called dolomitic limestone. Clastic limestone is made up of biogenic grains, or clasts from shell or bone fragments from dead marine organisms. Non-clastic limestone can be formed by the precipitation of carbonate crystals in either shallow water or groundwater (Kielmas, 2017).

Crushed limestone is often the main raw ingredient in Portland cement clinker manufacture. There is some evidence in the past that lime was used as the main binder in construction due to its hydraulic property. However, the development of cement during the early nineteenth century hindered the use of pure lime.

Advantages and disadvantages

Nowadays, limestone has been gaining its popularity as an environmentally sustainable alternative with several benefits:

Environmental credential: low energy consumption (for grinding) as limestone is softer than clinker and reabsorption of CO₂ during burning. The availability of limestone is more stable compared to other materials.

Although limestone brings some advantages in terms of economic aspects, builders also need to consider some of the disadvantages, namely **slow strength gain** and **deterioration**. Similar to GGBS and fly ash, limestone and cement's formation is relatively slower than cement pastes per se. A certain amount of limestone (up to 15%) as a supplementary increases early strength in accordance to the increase of the rate of cement hydration and calcium carboaluminate, but the higher strength achievement exists limit when limestone percentage reaches to 15-25%.

Properties of limestone

Limestone cement can be divided into four main classes LC 1 to 5 regarding the percentages of limestone content of 0%, 10%, 15%, 20%, and 35% respectively. The first four types have the same 28-day compressive strength equivalent to PC 42.5R, whereas LC5 is similar to PC 32.5R (Tsilivilis, Batis, Chaniotakis, Grigoriadis, & Theodossis, 2000)

Physical properties

The colour of most limestones is varying shades of grey (caused by carbonaceous impurities) and tan (caused by iron). The density is in a range of 2300 and 3000 kg/m³ and less than 1% water absorption. All limestones are crystalline, with average grain sizes ranging from less than 4 µm to about 1000 µm (Oates, 1998).

Chemical properties

Chemically, both limestone and marbles are siliceous calcium carbonate rocks: 38-42% lime (CaO), 20-25% Silica (SiO₂), 2-4% Alumina (Al₂O₃), 1.5 to 2.5% other Oxides like Na, Mg; loss On Ignition (LOI): 30-32%. Limestone, like marble and other calcareous stones, is referred to as acid sensitive, therefore acidic products should not be used on limestone and marbles (Stones, 2019).

2.1.6 Comparison of Different Cementitious Materials

In the following Table 1, different cementitious materials are compared using several sources (Leeuwen, Kim, & Sriraman, 2016); (Neville A. , Cementitious materials of different types, 2011); (SAGAWA, Yamamoto, & HENZAN, 2009); (Aso Cement, 2019); (United Kingdom- Quality Ash Association, 2002).

Table 1. Comparison of properties of PC and PFA, GGBS, and limestone.

Concrete property	PC	PFA	GGBS	Limestone
PSD (µm)	1-50	1-125	0.1-40	4-1000
Density (kg/m ³)	3150	1350-2700	1200	2300-3000
Specific surface area (m ² /kg)	350	355	350	-
Colour	Dark grey to white	Tan to dark grey	Off-white	Grey, yellow to white
Water demand		Decreased	Slightly decreased	Slightly unchanged
Workability		Increased	Increased	Increased
Strength		Increased over time (decreased in initial time)	Increased over time (decreased in initial time)	Decreased (varied by the level of replacement)
Durability		Enhanced	Enhanced	Essentially equivalent
Production method	Clinker (limestone +clay) + gypsum added in a kiln	Combustion in power plants	Tapping liquid in iron production	Sedimentary rock to crush and grind

2.2 Concrete at the early age

2.2.1 Mixing

Concrete mixing time varies with the types of the mixer. The revolutions of the mixer are sufficiently about 20rpm in order to obtain adequate mixing (Neville A. , The Mixing of Concrete, 2011). The average strength of concrete increases with an increase of in mixing time, which is shown in Abram's tests (Abrams,

1918). Within the first minute, the impact of mixing time on strength is considerably important. The exact value of minimum time is, therefore, given by the mixer manufacturer. According to ASTM C 94-99a, after all, solid materials have been put in the mixer, water is required to add not later than after one-quarter of the mixing time (ACI 304.R-89, ACI, 1994).

Adding water, known as retempering, will lower the strength of the concrete but lack of water content results in the dry mix which hinders the flowability of fresh concrete but reduces the rate of shrinkage at the early age. These both scenarios of water consumption should be avoided by considering the water to cement ratio, sand absorption, hydration of cement control, etc. to ensure consistency class as the designated purpose. Superplasticizer is another solution to maintain the designed strength of concrete while reducing the amount of water, but it could lead to the fact that supplement substance affects on overall performance within either beneficial usage or change of properties (Neville A. , The Mixing of Concrete, 2011).

2.2.2 Setting

Concrete starts the setting process after about 30-minute mixing as the material becomes more hardened and reduces its plasticity. Final setting occurs in generally 10 hours and concrete becomes solid and gains strength (Mishra G. , 2018). Chemical compositions, fineness of cementitious materials, water contents of pastes and ambient temperature have been observed to influence the setting time of mortars and concrete, particularly where the high carbon content is concerned.

Both initial and final setting times remain within the limits specified in cement standards. The use of different cement might affect setting time. For example, fly ash-class F generally prolongs concrete setting time while the strength of concrete gain is slower when compared to that of ordinary Portland cement only. Using GGBS gives a similar result by about 30 minutes. This is an advantage of these two materials when concrete mix remains workable for a longer time, and the risk of developing cold joints decreases. This property is helpful for construction in warm weather.

2.3 Rheology of fresh concrete

2.3.1 Introduction

Rheology is a part of workability needed to be considered as the effect on later properties at the stage of hardened concrete. Rheology is a complex study of how a matter flows in an either liquid state or soft solid under a condition in which the deformation is plastic when applying force. The substances considered are mainly Non-Newtonian fluids as neither Newton's law nor Hooke's Law satisfies the mechanical explanation. The **flow** and **deformation** of

these materials cannot be characterized by a constant value of *viscosity*, which is a measure of the resistance of a fluid to an applied stress, while Newtonian fluids are able to store deformational energy and the flowability is proportional to applied pressure. For instance, ketchup becomes “thinner” when being shaken as it can be thought of as an increase of internal friction; but water flow rate remains constant in accordance with the force applied (Roussel, Understanding the Rheology of Concrete, 2012).

Concrete pumping involves the flow of a complex fluid under high pressure in a pipe, and thus predicting flow behaviour of concrete pumping is a challenging research area. For the characterization and prediction of the flow of concrete pumping, the fundamental understanding of various factors is needed, which include rheological properties, dynamic segregation, the stability of constituent materials, the geometry of a pumping circuit, slip layer formed between bulk concrete and a pipe wall, and the relationship between the pressure and flow rate. The flow of concrete in a pipe differs from one of typical viscous fluids like water or oil.

Fresh concrete after being mixed and during the first hours behaves as a **Non-Newtonian** fluid and Tattersall first developed in the concrete and mortar formulations in 1955. More precisely, he assumed a relationship between the thixotropic behaviour of the cement paste and the performance of the vibration process commonly used when the concrete is placed (Rubio-Hernández, 2018). When concrete hardens, all particles stop moving or interacting with each other and consequently becomes less responsive to applied stress. Two factors considered during the earlier stage are **viscosity** and **yield stress**. The emphasis on the flow means that it is concerned with the relationships between stress, strain, the rate of strain, and time. The Deborah number indicates the ratio of relaxation time to observation time which shows the behaviour of a particular material in whether elastic (solid) regime or viscous (liquid) regime. When the value reaches 1, the material is said to be viscoelastic and influenced by dispersions of solid particles in water in concrete paste. Viscoelasticity quantifies the *deformation* or *oscillation* of a material.

Categories of liquid materials

Depending on the change of strain rate versus stress inside a material the viscosity can be categorized as having a linear, non-linear, or plastic response. When a material exhibits a linear response, it is categorized as a Newtonian material. In this case, the stress is linearly proportional to the strain rate. If the material exhibits a non-linear response to the strain rate, it is categorized as Non-Newtonian fluid. There is also an interesting case where the viscosity decreases as the shear/strain rate remains constant. A material which exhibits this type of behaviour is known as thixotropic. In addition, when the stress is independent of this strain rate, the material exhibits plastic deformation (Meyers and Chawla, 1999).

2.3.2 Viscosity

Viscosity (μ) is the measure of the internal friction of a fluid that resists the flow. This friction becomes apparent (or apparent viscosity) when there is an interaction between layers of a fluid which move relatively under an applied force, and apparent viscosity (η) is equal to the shear stress (τ) applied to the fluid over the shear rate ($\dot{\gamma}$):

$$\eta = \frac{\tau}{\dot{\gamma}}$$

For a Newtonian fluid, the apparent viscosity is constant and equal to the Newtonian viscosity of the fluid, but for non-Newtonian fluids, the apparent viscosity depends on the shear rate (Fox, McDonald, & Pritchard, 2012). The SI derived unit is Pa·s (Pascal-second), but the centipoise is frequently used in practice: 1 cP = 1 mPa·s. Table 2 shows several materials with a range of viscosity as non-Newtonian fluids.

Table 2. Viscosities of some non-Newtonian fluids (Michael Smith Engineers, 2019)

Fluid	Viscosity [Pa·s]	Viscosity [cP]
honey	2–10	2,000–10,000
molasses	5–10	5,000–10,000
molten glass	10–1,000	10,000–1,000,000
chocolate syrup	10–25	10,000–25,000
molten chocolate*	45–130 [23]	45,000–130,000
ketchup*	50–100	50,000–100,000
peanut butter*	c. 250	c. 250,000
shortening*	c. 250	250,000

There are various types of non-Newtonian fluids that significantly deviate from time-independent behaviour indicated in Figure 2, including: Shear-thickening liquids, Shear-thinning liquids, Bingham plastics (Alderman, 1997).

Viscosity can be assumed as a slope of line plotted by shear stress against shear strain.

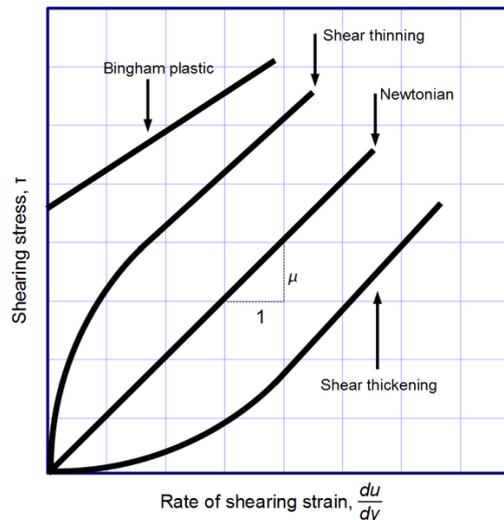


Figure 2. Viscosity regime

In addition, thixotropic and Rheopectic (dilatant) liquids become less and more viscous respectively over time when shaken, agitated or otherwise stressed. These types of liquid were not shown in this graph.

Viscoelasticity

Viscoelasticity includes both viscous and elastic characteristics under deformation. When applying stress on the material, there are critical some time needed for the structure to start deforming. It is assumed that as long as the structure remains its basic structure, the given stress is proportional to the deformation in the solid regime or to state shear rate in the liquid regime. However, the steady state shear rate of material at both states requires a certain period to reach the value. It contrasts with purely elastic material whose rate is independent of time (PA., 2015). The reason is that the supplied energy is stored initially in the form of elasticity. During loading, it is the energy stored in a material, during unloading it is the energy recovered. For concrete materials, viscous effects may be more complex, involving not only viscous dissipations in the interstitial liquid but also in the interactions between the elements of the structure.

Concrete at an early age should be considered as either a viscoelastic product when being **pumped** in the pipe where zero zones of shear stress exists or plastic correspondence when **setting up** in formwork.

Plastic viscosity

When a matter stops deforming elastically and switches to plastic deformation upon reaching yield stress, plasticity appears. Alternatively, while elastic deformation is termed reversible as the stored elastic strain energy helps recover upon load removal; plasticity deformation is considered as permanent and irreversible involving energy dissipation. In other words, more energy is expended to achieve the original state. Plastic deformations or loadings are normally rate independent of the stresses applied, which is in contrast to universal Newtonian fluids. Most engineering materials undergo plasticity,

meaning permanent deformations occur once the stress goes above the elastic limit.

It should be distinguished between plastic materials and material under viscoplastic. The latter one is rate dependent and it starts flowing whenever the yield stress is exceeded (ACI 238, 2008), while the other one yields instead (PA., 2015).

2.3.3 Yield stress fluids

The state in which a material turns from solid to liquid defines by the change of behaviour in response to the applied stress. Some industrial and natural materials keep the shape it has been given until a stress applied exceeds the critical value which the materials start to flow and behave as liquids; and the phenomenon continues as long as the stress is maintained. The critical value is called yield stress of fluids (Roussel, Understanding the Rheology of Concrete, 2012) and at this stage, a material deforms plastically.

A material which essentially behaves as a solid, i.e. mainly elastic with possibly some viscoelastic effects, when the applied stress is smaller than the yield stress τ_c , and as a viscous liquid when the stress is larger than τ_c (Figure 3).

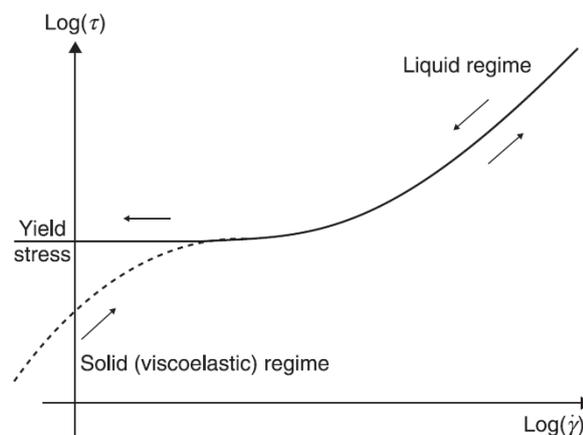


Figure 3. Apparent flow curve of a yield stress fluid

This curve is obtained from an increasing–decreasing stress or shear rate ramp. The increase corresponds to the dotted line and the decrease to the continuous line (Roussel, Understanding the Rheology of Concrete, 2012).

There are various materials used in civil engineering such as ceramics slips, paints, mortars, plasters, cement pastes, and fresh concrete. The primary difference between fresh concrete and typical viscous fluids like water or oil is yield stress is greater than zero and additionally, the centre of the pipe is where concrete is not sheared i.e. a zone around the symmetry axis (Jacobsen, 2009). Fresh concrete begins to flow when the yield stress value is exceeded (the shear

stress exceeds a threshold limit), otherwise, its behaviour resulted in an elastic material; and after applying vibration, the yield stress is reduced to the extent that concrete can flow due to its own weight (Juradin, 2012). Although measurements of plastic viscosity have several practical applications (pumping, casting rates, etc.), yield stress is the most important parameter for formwork filling in practice. Gravity and pressure gradient generate a shear stress and if this value is lower than yield stress (as a complex function of the formwork thickness and the local density of steel reinforcement), concrete stops flowing before it levels or the formwork is entirely filled (Roussel, Rheology of fresh concrete: from measurements to predictions of casting processes, 2007).

Consistency is a term used to qualitatively describe the flow of fresh concrete. However, consistency can be quantitatively characterized by the yield stress (Daoud, 2008). Concrete that has a sticky consistency tends to have problems with the finishing. On the other side, a flowable concrete can create problems with stability. Hence, the consistency of fresh concrete has to be chosen to balance between the placing requirements and finishing conditions. In this essence, the yield stress can be used to control the consistency of fresh concrete.

2.3.4 Bingham plastic model

Many rheological models have been developed to describe the flow of liquids or liquids with solid suspensions (Daoud, 2008). The variations among these models are their description of the onset of flow and the flow behaviour. Materials start to flow when the shear stress exceeds a threshold limit referred to as yield stress. For some materials, such as water and oil, the yield stress is equal to zero and for others, such as fresh concrete, it is greater than zero. There are several basic models (Francisco-José Rubio-Hernández, 2018) that have been used to describe the flow behaviour of liquids or liquids with solid suspension (Table 3).

Table 3. Viscoplastic models used for describing steady flow curve of cement pastes.

Model Name	Equation
Bingham	$\tau = \tau_y + \eta_p \dot{\gamma}$
Modified Bingham	$\tau = \tau_y + \eta_p \dot{\gamma} + c \dot{\gamma}^2$
Herschel–Bulkley	$\tau = \tau_y + K \dot{\gamma}^n$
Robertson–Stiff	$\tau = A(C + \dot{\gamma})^B$
Karam	$\tau = A \exp(k\phi) (\dot{\gamma}_0 - \dot{\gamma})$
Casson	$\tau = \tau_y + \eta_\infty \dot{\gamma} + 2\sqrt{\tau_y \eta_\infty} \sqrt{\dot{\gamma}}$
Modified Casson	$\tau^m = \tau_y^m + \eta_\infty \dot{\gamma}^m$
Papo–Piani	$\tau = \tau_y + \eta_p \dot{\gamma} + K \dot{\gamma}^n$
Vom Berg	$\dot{\gamma} = B \sinh\left(\frac{\tau - \tau_y}{A}\right)$

For a concentrated suspension (e.g. concrete), there are forces acting between the particles. These forces do not merely change the viscosity but actually change the type of flow (Tattersall, 1983). Tattersall and Banfill after carrying out systematic investigations in the rheology of concrete found that there was a linear relationship between torque and the rotation speed of the viscometer after a certain torque had been exceeded (Anal K. Mukhopadhyay and Sehoon Jang, 2009). They stated that concrete flow could be expressed by the Bingham model and can be written as:

$$\tau = \tau_0 + \mu \dot{\gamma}$$

where τ (in Pa) is the shear stress, τ_0 is the yield stress, μ (Pa.s) is the plastic viscosity, and $\dot{\gamma}$ (in s^{-1}) is the shear strain rate.

Or the expression can be written in accordance to torque and impeller speed:

$$T = g + hN$$

Where T (in N) is torque, g is yield value “flow resistance”; N is impeller speed; and h is constant, caters for the effect of shear rate and plastic viscosity. This linear relationship is shown in Figure 4.

Apparent viscosity η or k is $\eta = \frac{g}{N} + h$

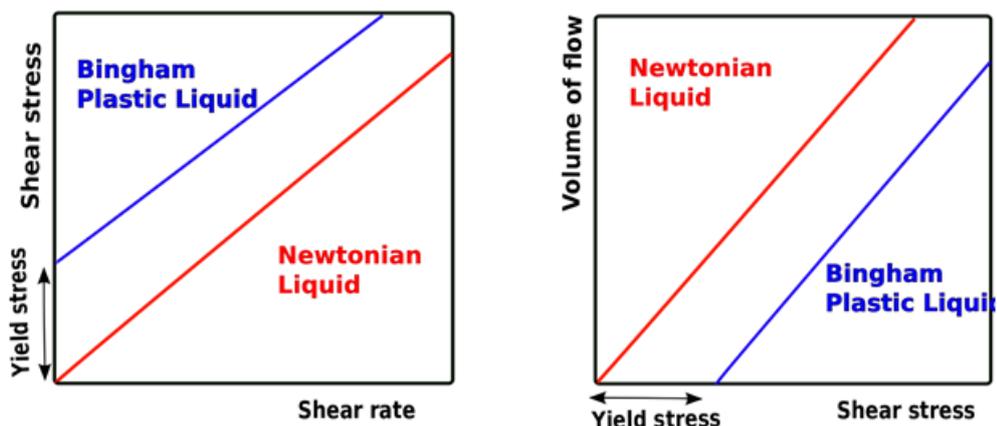


Figure 4. Bingham liquid and Newtonian liquid (Richard, 2011)

2.3.5 Segregation

It is impossible to obtain a full compaction of a workable mix while there is a presence of appreciable segregation. The separation of layers of constituents causes separation so that the distribution of the heterogeneous mixture is no longer uniform. In terms of concrete, the reasons are the difference in the sizes of particles and the specific gravity of mix constituents. These extent can be adjusted by good handling and suitable grading (Neville, 2011).

Segregation might occur in cases where either the mix is too dry or wet. The first form of segregation in a dry mix is that coarse particles move separately out from the finer particles and settle to the bottom, or further along a slope. Additional water could improve the cohesion of the mix. The second form is a

remarkably wet mix as the grout (or cement paste- cement and water) separates from the mix because of the lowest specific gravity.

The importance of handling is determined by the way of storing fresh concrete in a bucket before pouring into formworks as well as the distance of travel. The danger of segregation could be reduced within great care of handling, transporting and placing. Furthermore, dropping concrete from a considerable height, passing along a chute with a direction change, and discharging against obstacles encourage segregation, so a particular cohesive mix should be used for each circumstance.

Concrete with variable grades (gap-grading), segregation is more likely to occur as the coarse aggregates tend to move down from finer ones and the rheometry and shrinkage behaviour are not stable throughout the material. In reality, grout floats upward and that leads to bleeding; or laitance in case of a layer comprised of hydrated cement and fine aggregates which rises to the surface with the addition of too much water. Concrete, furthermore, should be placed and remained in a specific position, as a result, it must not be allowed to flow along the form without initial force or energy from i.e. pumping. Consequently, the use of vibration provides the most valuable means of compacting concrete. However, this method causes segregation in a sense of improper placing and vibrating (overdone according to specification) and coarse aggregates are left especially in a sloppy mix (Neville A. , 2011).

It can be **negligible** in case of mortar which remains the grade of aggregate (sand-sized particles). For instance, the mortar used for up-binding material, such as slab to column bond, avoids segregation so that dropping downward is avoided from a gravity difference.

2.3.6 Factors affecting rheology

Fresh concrete can be considered as a polydisperse system of particles (Farris, 1968). Cementitious materials contain more than one type of particles which differentiate them to monodisperse suspensions of solid particles. The rheology of the matter is affected by the amount of dissipated energy from the interaction between particle and liquid, or particle and particle. Therefore, the physical properties of the particle of the materials play an important role in changing the workability of concrete (Table 4). These properties also indicate the bond of cement paste and aggregates. Aggregates can be fine aggregate and coarse aggregate and many materials are used such as sand, gravel, stone, crushed rock, and sometimes waste materials or recycled fine.

Table 4. Effect of concrete ingredients on concrete rheology

Factors	Yield stress	Plastic viscosity
Cement content	Decrease	Decrease
Water content	Decrease	Decrease
Aggregate volume fraction	Increase	Increase
Sand to aggregate ratio	Optimum value	Optimum value
Microfine content	Mixed	Mixed
Water-reducing admixtures	Decrease	Mixed
Air-entrainment agent	Mixed	Decrease
Viscosity modifying admixtures	Increase	Increase
Fly ash	Decrease	Mixed
Silica fume (Low dosage)	Decrease	Decrease
Silica fume (High dosage)	Increase	Increase
Slag (GGBS)	Mixed	Increase

Table 5 (ACI 238, 2008) gives information about yield stress and plastic viscosity of some materials from the simplest structure as cement paste to the fully developed structure as universal concrete (Banfill, P.F.G., 2003). The increase of particle size provides higher viscosity and yield stress due to the higher resistance of material to deformation. Concrete contains about 70-80% aggregate whose size is much larger than that of mortar or cement paste. Hence, its parameters are higher than other two.

Table 5. Rheology of cement paste, mortar and concrete

Material	Cement paste, grout	Mortar	Flowing concrete	Self-compacting concrete	Concrete
Yield stress (N/m ²)	10-100	80-400	400	50-200	500-2000
Plastic viscosity	0.01-1	1-3	20	20-100	50-100
Structural breakdown	Significant	Slight	None	None	None

Volume fraction

Volume fraction describes the number of particles in a material (Chateau, 2011). It can be called *packing density* or *packing compacity* and it is the ratio of the volume of the particles V_s to the total volume of the packing V_p . The volume of void V_v is, therefore, the remaining volume when subtracting particle volume from the total packing volume ($V_p = V_s + V_v$). The increase in volume fraction leads to the increase in viscosity and the maximum value obtained is when there is the least room for particle movement. Krieger- Dougherty equation illustrates the correlation between viscosity and volume fraction:

$$\frac{\eta}{\eta_{medium}} = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\eta]\phi_m}$$

Where:

η is the viscosity of the suspension;

η_{medium} is the viscosity of the medium;

ϕ is volume fraction of solids in the suspension;

ϕ_m is maximum vol. fraction of solids in the suspension;

$[\eta]$ is the intrinsic viscosity of the medium

(2.5 for spheres or $2.5 + 0.123(R - 1)^{0.925}$ for others).

Unlike solid whose volume fraction is constant with the range of particle size, fresh concrete has the effective volume fraction which is inversely proportional to the **size of the particle**. This can be expressed by the equation below.

$$\phi_{eff} = \phi \left(1 + \frac{L}{2a}\right)^3 \text{ (Hill, 2015)}$$

Where:

ϕ_{eff} is effective volume fraction;

Φ is volume fraction of solids in the suspension;

L is particle separation;

a is the radius (Figure 5).

(Courtesy by Malvern Instruments limited)

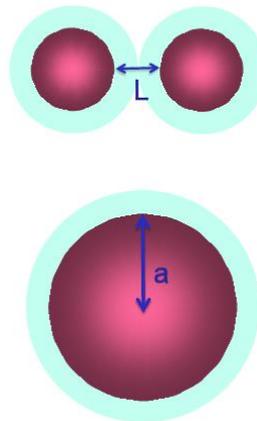


Figure 5. Particle physical parameters

Size ratio

The size ratio of concrete particles can be described by their length (L) and long diameter (D). This is one of the important parameters having an impact on particle packing density (Chateau, 2011). Elongated particles have a high value of size ratio whereas the value reaches to 1 within regular geometry particles.

Shape and texture

According to Chateau, spherical particles have a lower packing density than that of regular particles, but the random packing density shows a reverse trend between these two types of shape. Several values of different shapes are shown in Table 6 (Chateau, 2011).

Table 6. Random close packing density for particles of different shapes

Particle shape	Random close packing density
Spheres	0.60-0.64
Cubes	0.76
Parallelepiped ($4.0 < L/D < 8.0$)	0.51-0.67
Disks ($L/D=1$)	0.63
Spheroids ($0.6 < L/D < 1.3$)	0.58-0.61
Rounded aggregates	0.59-0.63
Crushed aggregates	0.50-0.57
Fibres ($L/D=10$)	0.48
Fibres ($L/D=167$)	0.03

With particles with the size ratio of less than one, their regular geometric shape can give a high value of density because they favourably rearrange locally with respect to the flow direction more easily than particles with irregular shape or rounded boundary. The angularity of particle also influences on packing density. It can be understood as the roundness of particle. However, there is no exact correlation between roundness and size ratio that indicating the role in particle packing density.

With particles with the size ratio of larger than one, it seems like random packing density depends on size ratio with the inversely proportional relationship. Some authors predicted that elongated particles found in fibre, which have high size ratio, obtain low random packing density by fitting empirical data (Nardin, Papirer, & J.Schultz, 1985). To conclude, lower viscosity can result from a more spherical shape.

Particle size distribution

Mixing materials with different sizes of particles lead to an increase in compacity (Bournonville, 2003). It means the composition of mixture affects the packing density, hence, the viscosity of concrete. The reason is that the smaller particles are able to fill up pores between larger-size particles. The action can reach the optimum condition when vibration and mixing are applied to help particles' movement. Therefore, polydisperse close packing increases and the maximum value of 0.74 can be obtained (Hill, 2015) whilst monodisperse suspensions have a lower maximum packing density. In conclusion, wider particle size distribution gives lower viscosity, and, consequently, the liquid is under higher flow rate.

Sand-to-cement ratio

According to some trial tests before finalising the mix proportions, the ratio of sand to cement changed from 3:1 to 2:1 while maintaining the water/cement ratio of 0.6. It can be observed that the increase of cement or reduction of aggregate produces higher workability products. In other words, the smaller size of particles, the more flowable concrete can obtain.

Admixtures

No admixture was involved in this experiment, but its effects are proved by other technical reports. One of the common types of admixture is superplasticiser which is used to either reduce yield stress while keeping the same content of solids or to increase solids content at constant yield stress (Flatt, 2012). The reason is that superplasticiser adsorbs onto particles and yield stress can be reduced by changing the interforce between particles. It means there should be less contact of particles to others by steric hindrance. As a result, concrete with the use of superplasticiser has more workability and ensures the design strength.

2.3.7 Summary

Rheology is a very powerful tool to examine the internal structure of a material. Several factors affect the rheometry through varying viscosity of a matter in liquid state. The most important factors are the size and shape of particles contributing to a mix of cementitious materials and aggregates. To indicate the influence of particles in viscosity, the volume fraction is used as a measurement of particles packing capacity. These parameters might not follow direct correlation, but the observation can be made through empirical data from mixtures of different types of pozzolanic materials and Portland cement, which will be shown in Chapter 3. Furthermore, water as a solvent plays a critical role in a mixture affecting the volume fraction of the suspension system.

2.4 Shrinkage of fresh concrete

There are numerous researches examining early-age shrinkage in concrete, concerned with the effects of varying environmental conditions and materials used. However, there have been developments recently in concrete technology which has shown that changing the mixture of materials used in fresh concrete can minimize the risk of shrinkage. This is because the properties of various types of cement and aggregates perform differently in the rate of hydration and water evaporation on the surface of the concrete.

2.4.1 Introduction

Concrete reduces in volume due to loss of water and the change of volume is called shrinkage, which is either autogenous or induced. As a deleterious effect

for the long-term strength and durability of concrete, shrinkage begins immediately after concrete is mixed, the rate of shrinkage increases for the first 30 minutes. After this time, it remains at this high level, until the concrete has completely hardened. The process continues but at a much decreased rate. Shrinkage causes unsightly cracks in concrete, although making concrete without cracks or shrinkage is highly unlikely. Cracks also occur when using reinforced concrete, even with higher extent, due to stress caused by the reinforcement restraint.

There are several critical types of shrinkage throughout the lifecycle of concrete. At the early stage, plastic shrinkage and autogenous shrinkage are present in fresh concrete; while at the later stage, drying shrinkage and carbonation shrinkage occur at a slow rate in hardened concrete (Table 7 and Figure 6). This report focuses on the first two types of shrinkage. The major factors affecting shrinkage include mix proportion, material properties, curing methods, the types of coarse aggregates and the shape of aggregates. In addition, humidity, water-cement ratio, the hardness of aggregates, moisture movement in concrete, wind velocity and geometry of the specimen can also have an impact.

The measurement of shrinkage is demonstrated by the change in length per unit length- the unit is in **percentage** - and it is time-dependent.

Table 7. Types of shrinkage

Type of shrinkage	Note
Plastic Shrinkage	<ul style="list-style-type: none"> • movement of water (hydration process) or environmental conditions leading to evaporation of water on the surface of wet concrete • proportional to cement content and, inversely proportional to the w/c ratio
Autogenous Shrinkage	<ul style="list-style-type: none"> • due to hydration and not including environmental effects (temperature and moisture changes) • is usually ignored in concretes with w/c more than 0.40
Drying Shrinkage	<ul style="list-style-type: none"> • the ever-lasting process when concrete is subjected to drying conditions • the gel water is lost progressively, and it causes volume change over a long time

Carbonation Shrinkage	<ul style="list-style-type: none"> • by decomposition of calcium, compound in hydrated cement and reduce in volume • increase strength and reduce permeability
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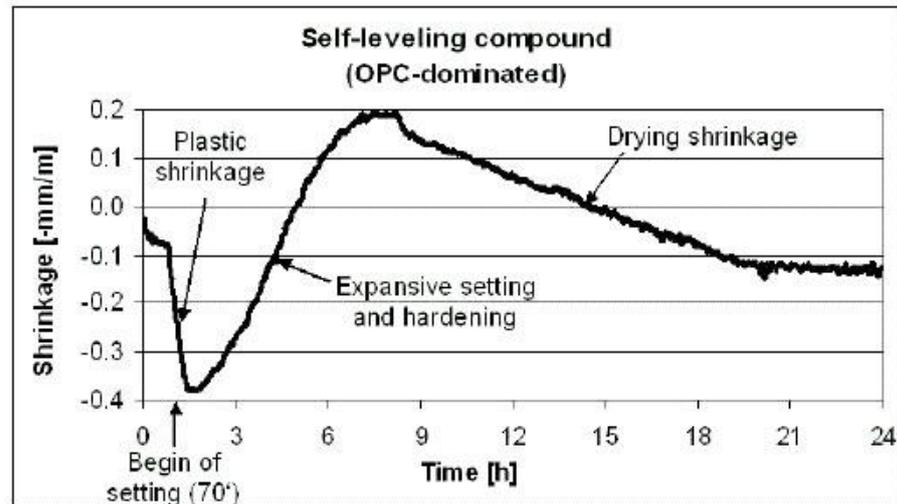


Figure 6. Types of shrinkage over time

2.4.2 Early age shrinkage

It is commonly known that concrete maintains semi-fluid properties during the first 3–4 hours whilst *hydration reactions* are very slow. The process accelerates over the following 8–12 hours when concrete becomes more rigid and hardened, which leads to cracking (CCAA, June 2005). The time period depends on many variables mainly including the constituents of the concrete, the mix proportions, and the ambient conditions. The reduction in volume is of the system cement-plus-water: the cement paste is plastic, and its volumetric contraction constitutes of about one percent of the absolute volume of dry cement (Swayne, April 1942).

Concrete moves from a saturated to a partially saturated state due to the drying process of the concrete surface. This movement causes *capillary* tension in the pore water (bigger pores) and gel pore (smaller pores) so that water begins to dry out (see Figure 7). **Cracking** occurs whenever there is a restraint against the contraction of concrete as capillary tension develops stresses which exceed the tensile capacity of the concrete. A recent research sponsored by Cement Concrete & Aggregates Australia (CCAA) at the University of Queensland (Dux P F, 2000) has shown that strains in the concrete of similar magnitude to those resulting from drying shrinkage can be developed during the early age. Fresh concrete has a small tensile strength and hence cracking is likely. Furthermore, the internal restraint mechanism supplemented by external restraint from formwork, subgrade friction, member geometry and so on, can also lead to early cracking.

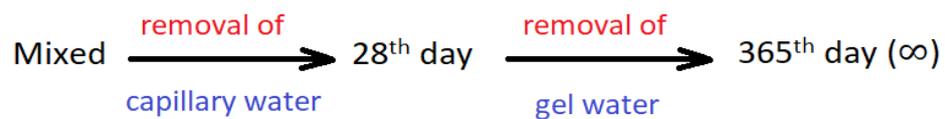


Figure 7. Time line of water removal in concrete

The early-age shrinkage is the sum of plastic shrinkage, plastic settlement and autogenous shrinkage (Newlands, Paine, & Dhir, 2008).

2.4.3 Plastic shrinkage

When concrete is still in plastic state or before setting, water movement leads to the contraction in a volume called plastic shrinkage. In detail, *water evaporation* on the surface on wet concrete causes bleeding which is proportional to the magnitude of plastic shrinkage. Hydration (slowly in the first 4 hours) and the absorption of aggregates (this will be discussed in Chapter 4) are considered relatively small contributions to the total water loss at an early age before *initial setting* (Neville A. , Elasticity, shrinkage, and creep, 2011). Plastic shrinkage, instead, is affected by temperature, ambient relative humidity, and wind velocity. Furthermore, cement content and water/cement ratio will be tested in this report.

The recommended value of shrinkage should not exceed 1 kg/m² per hour (ACI 304.R-89, ACI, 1994). Additionally, it is worth noticing that the rate of loss of water per se is not likely to predict the plastic shrinkage as it depends on the rigidity of the mix.

Plastic shrinkage cracking is known to be introduced when the rate of evaporation exceeds the rate at which the bleeding water rises to the surface. The crack can be either very deep or quite short and difficult to close permanently. In case of a large horizontal area where contraction in the horizontal direction is more difficult than vertical one, several deep cracks of an irregular pattern are formed. This mechanism is also demonstrated in the test using a thin-layer shrinkage measurement which will be discussed in Chapter 3 and the cracks can be called pre-setting cracks. They are usually parallel to one another and spaced 0.3-1 m apart (see Figure 8).



Figure 8. Pre-setting cracks in horizontal surface of mortar mix (CEM I and limestone)

2.4.4 Autogenous shrinkage

Autogenous shrinkage is another type of shrinkage at the early age of concrete at which the *macroscopic volume reduction* is mainly due to **cement hydration** after *initial setting* (setting time of concrete see Chapter 2) when there is no water supply or the moisture movement to and from cement paste is not permitted. Also, this mechanism can explain the withdrawal of water from capillary pores by the hydration of unhydrated cement, or so-called *self-desiccation*. Therefore, the volume changes due to the loss or ingress of substances (e.g. water absorption or chloride ingress), the temperature variation, the application of an external force or restraint are not included in autogenous shrinkage causes. This shows the opposite pattern to plastic shrinkage which is predominantly affected by water evaporation on surface.

To express autogenous shrinkage, it is used by the percentage of volume contraction “autogenous shrinkage ratio” or one-dimensional length change “autogenous shrinkage strain”. Although the shrinkage is essentially in three-dimensional phenomenon, autogenous shrinkage strain is described as linear strain (Ei-ichi Tazawa, 1999). It also contributes to the strain of drying shrinkage strain.

Moreover, autogenous shrinkage is a part of a wider evaluation- *chemical shrinkage* -as when cement minerals react to water, the total volume decreases between two phases: solid and liquid the reduction in volume is caused unhydrated cement has a larger volume than that of hydrated cement. Autogenous shrinkage can be understood as external chemical shrinkage within thermal restraint. Autogenous shrinkage/ chemical shrinkage has been found to be linearly related to the degree of hydration of the cement. Figure 9 illustrates their behaviours in different phases. Although it is *negligible* for conventional concrete with high w/c ratio, it becomes *significant* for high strength concrete with low w/c and high cement content.

After setting of cement, the hardened body shrinks predominantly by hydration which is compensated by the formation of *voids* in microstructure. The formation of *ettringite* (hydrous calcium aluminium sulfate mineral) creates a large volume of fine pores in the hardened body and *C-S-H* will then fill these pores as long as hydration reaction occurs. The rigidity of the structure affects autogenous shrinkage in relation to morphology (shape and form at the microscopic scale) and chemical composition of hydrates (Japan Society of Civil Engineers, 1996).

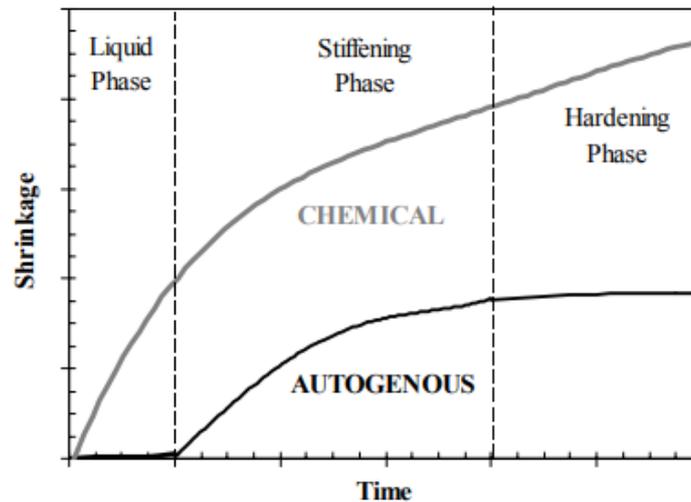


Figure 9. Autogenous shrinkage and chemical shrinkage over time. (Holt, 2001)

2.4.5 Factors affecting early-age shrinkage

Plastic shrinkage results from the surface evaporation of water and the bleeding rate of concrete due to various factors as mentioned below. The depth of concrete covered to reinforcement will not be stated in this research.

Environmental conditions

In a condition where the ambient temperature is high, the demand of water use is increased. This leads to an increased rate of loss of slump and a rapid hydration and evaporation on concrete surface. In hot weather, plastic shrinkage cracking can be confused with ***plastic settlement cracking*** caused by differential settlement of fresh concrete due to obstruction i.e. large particles of aggregate or reinforcing bars to subside.

There will not be any shrinkage if the relative humidity reaches 100 per cent. The evaporation is increased when the temperature of concrete is higher than the ambient temperature, even in a high relative humidity of the air.

Wind velocity is another influence on the magnitude of shrinkage. It can be seen in Table 8 (L'Hermite, 1960) that the wind velocity is directly proportional to the rate of shrinkage.

Table 8. Plastic Shrinkage of Neat Cement Paste Stored in Air at a Relative Humidity of 50% and Temperature of 200C

Wind velocity		Shrinkage 8 hours after placing
m/s	mph	
0	0	10-6
0.6	1.35	1700
1.0	2.25	6000
7 to 8	16 to 18	7300
		14000

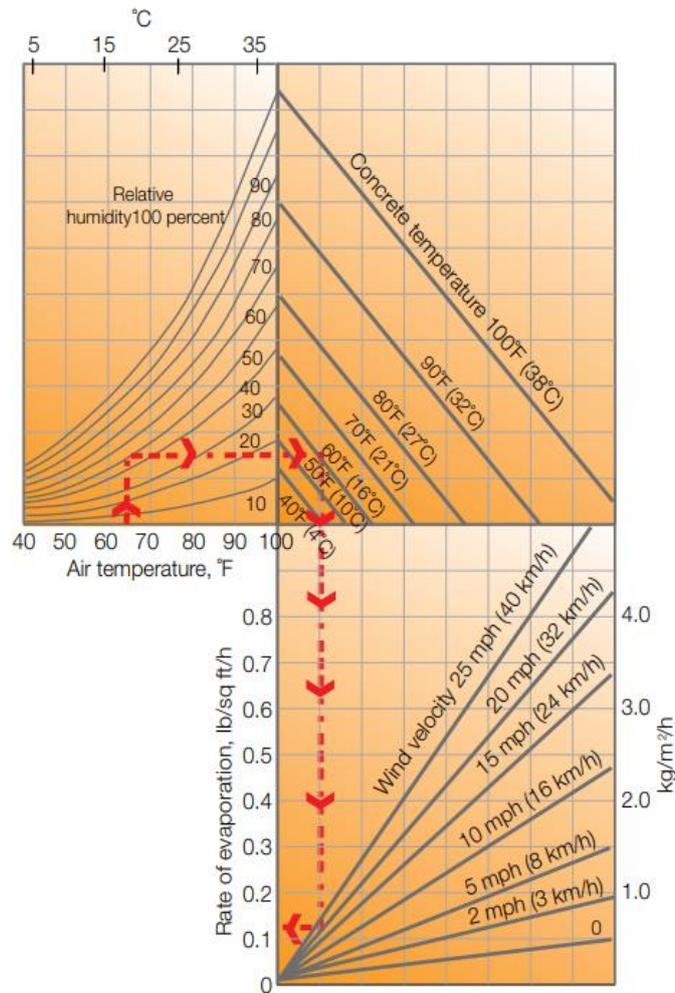


Figure 10. Effect of temperatures, relative humidity, and wind velocity on the rate of evaporation of surface moisture from concrete

The rate of evaporation leads to the variation of plastic shrinkage and Figure 10 shows that the value of rate of evaporation is estimated by considering temperature, humidity to wind velocity (Solutions, 2014).

Cementitious materials

Different aggregated types and sizes may have varying effects on hydration process and surface water evaporation when maintaining the w/c ratio among all mixes. Early-age shrinkage can be controlled by monitoring the mix proportions so that the properties of each constituent contribute to the overall performance. It has been proved that plastic shrinkage is higher when increasing the cement content of the mix and reducing water content.

Fly ash

The higher fly ash content, the more autogenous shrinkage is reduced. However, the increase in fly ash class F does not affect the amount of autogenous shrinkage (Tangtermsirikul, 1999). There is a number of reasons for this result correlating to shrinkage mechanism. Firstly, adding up fly ash to cement results in less hydration. Pozzolanic reaction can only occur when there is Ca(OH)_2 produced from the hydration (see Chemical properties of fly ash previously) and fly ash reacts slowly at the early age. The **shape of particles** of fly ash is spherical, which means that they retain less water than cement particles which are irregular. This results in larger free water in a mixture with fly ash than the one without fly ash, especially when the specimens are made with the same water to binder ratio or similar w/c ratio. As autogenous shrinkage is due to water loss in hydration process, higher free water content means the reduction in shrinkage. Chemical expansion (*chemical composition*) due to special characteristics of high SO_3 in fly ash is another reason. There is a proportional correlation between the content of SO_3 and chemical expansion. Swelling due to water absorption (recoverable process- not affective to autogenous shrinkage) can be negligible. Finally, in term of **particle size**, paste with fly ash having a smaller average size than cement obtains larger autogenous shrinkage; while fly ash having a bigger average size than cement shows a smaller autogenous shrinkage than that of cement paste.

Slag

Autogenous shrinkage increases when part of OPC is replaced by slag, which is a completely contrary scenario. This is because the amount of added slag slightly decreases the extent of shrinkage but generally, it is still much higher than that without slag (Chan, Liu, & Lu, Effects of Slag and Fly Ash on the Autogenous Shrinkage of High Performance Concrete, 1999). When both slag and fly ash supplement Portland cement, it is likely that slag dominates the effect of autogenous shrinkage of concrete. It is because its high **fineness** leads to active pozzolanic reaction and the self-desiccation process is irritated by **refined pore structure**.

The hydration rate of combined GGBS and fly ash cement is higher than that of fly ash alone. The amount of formed C-S-H significantly increases when blast furnace slag is added. Although GGBS reacts more slowly than fly ash; when it is incorporated, GGBS reacts with Ca(OH)_2 to produces C-S compound, and together with ettringite act as nucleation sites (Kirsty, 2012), fly ash therefore hydrates with an increasing rate. The content of Ca(OH)_2 reduces by the reaction

of GGBS and fly ash but the fly ash may contribute to the formation of C_2AH_8 due to the release of Al_2O_3 (Voglis & Kakali, 2005).

Limestone

The amount of limestone in cement under 30% is likely to show a difference in shrinkage. Mixes with more than 40% limestone substitution indicates a significant *reduction* in shrinkage. However, similar results are obtained in mixes with 40%, 50% and even 100% of fine limestone (Carlos, Masumi, Hiroaki, Maki, & Takahisa, 2010). In other words, the increase of limestone in the mixture does not mean a monotonous effect of the behaviour of shrinkage.

Limestone addition slightly enhances the degree of hydration of cement during the first day. This is the so-called filler effect and is related to more available space or water for hydrates growth (i.e. when adding 20% of filler the water-to-binder (w/b) ratio increases from 0.4 to 0.5 if limestone is considered inert at this age) and maybe to more nucleation sites (Bizzozero & Scrivener, 2015). The high amount of SiO_2 in limestone compared to GGBS and FA accounts for the reaction with CaO (crystal powder) started within one hour and formed C–S–H gel. In addition, limestone is the main ingredient in clinker manufacture of Portland cement, so the amount of carbon oxide contributes to the reaction of forming $Ca(OH)_2$ as a hydration reactant.

Admixtures

The chemical admixtures, including water reducing, accelerating, retarding, superplasticiser, and air entraining admixtures, give certain influences on shrinkage performance.

Water reducing admixture helps reduce the extent of water needed while retaining workability of concrete, so shrinkage should become greater due to less excess water and cracks are likely to occur.

Accelerating admixture is used in cold weather curing to increase the rate of hardening and setting of concrete, therefore, less time required possibly decreases the effect of shrinkage.

Opposite to accelerating admixture, *retarding admixture* is used in hot weather curing due to high temperature's effect on the rate of hardening. It decelerates the reaction process which possibly leads to the reduction in shrinkage.

Superplasticiser admixture is similar to water reducing admixture in lessening water usage without interfering with concrete workability. Superplasticiser also enable concrete to cure faster as accelerating admixture does. However, this agent has no impact on shrinkage or creep (Mishra G. , 2014).

When *air entraining admixture*, as an agent used for preventing freeze-thaw effect, is added, air bubbles are formed to reduce bleeding and segregation, hence, it reduces shrinkage (especially plastic shrinkage).

2.4.6 Summary

It is found in shrinkage modelling, that the rate of strength gain is inverse to the shrinkage of the concrete. Consequently, it is possible to conclude that a sample hydrating and developing strength at a faster rate may have less total shrinkage (Christopher and Goulias, 2009). Fly ash and limestone as supplements help to reduce shrinkage while GGBS shows the opposite trend. Their combination might show different behaviour and will be examined during several tests mentioned later in Chapter 4. Researchers have reported that autogenous shrinkage in liquid phase is very little or negligible and it progresses slowly over time in stiffening and hardening phases as compared to chemical shrinkage which increases very rapidly in liquid phase in a very short duration and continues in a faster rate even in hardening phase. It is suggested that if the early age shrinkage magnitude exceeds 1 mm/m there is a high risk of cracking (Holt, 2001).

3 TEST METHODS

3.1 Materials

The various types of each cementitious material can perform differently. In this research, one type of each element in the mix was used in order to maintain the consistency of results. Portland cement is high strength cement of 52.5N; fly ash is of standard FA450 type DFA3; GGBS is a universal type and limestone is crushed clastic material used in Portland cement production. Sand is used as fine aggregate.

Physical and chemical properties of cement constituents were determined by two tests, namely Laser PSD by Mastersizer 2000 and X-ray fluorescence analysis (XRF) respectively. This chapter discusses the results of these analyses in detail.

3.1.1 Mix design

There are **six** designed mixes for the tests of rheology and shrinkage of mortar.

Table 9 shows that the total amount of cement (CEM-I) is maintained to be 500 (kg/m³) of specific density for each mixture. Simultaneously, the amount of sand (fine aggregate) is also the same so that sand to cement ratio is unchanged. Additionally, water/cement ratio was meant to be 0.6. However, the content of water has increased slightly due to water absorption by fine aggregate (1.5% mass of sand) and the contribution of very fine aggregate which functions as cement (3% particles passing through $\varnothing 0.08 \mu\text{m}$ sieve).

Table 9. Mix design

Mix no.	Mortar Mix	Constituents, kg/m ³					Water	w/c
		CEM-I	GGBS	FA	LS	Sand		
1	CEM-I	500	0	0	0	1000	300	0.60
2	CEM-II/A-L	450	0	0	50	1000	300	0.60
3	CEM-II/C-M (V-L)	300	0	150	50	1000	300	0.60
4	CEM-II/C-M (S-L)	300	150	50	0	1000	300	0.60
5	CEM-VI/(S-L)	200	250	0	50	1000	300	0.60
6	CEM-VI/(S-V)	200	250	50	0	1000	300	0.60

Portland cement was replaced by no more than two other materials. The maximum of limestone used was no more than 10% of the total cement weight and remained while supplementing GGBS and fly ash (mix 2,3 and 5). GGBS dosage was changed in two mixes (4&6) with the same of FA content. All tests of shrinkage and rheology were done at the same time due to limited time before the samples start hardening which cause false measurement and calibration.

No admixture was used for all mixes. Sand/cement ratio, instead, was changed to examine the workability of the mix. It is observed that ratio of 2:1 gives a better result than that of 3:1. The samples were all flowable enough without the use of superplasticiser even though it could be recommended with the dosage of 0.25% of cement content by DG51.

3.1.2 Particle size distribution (PSD)

Mastersizer 2000 is a tool used to help determine the size distribution of particles. It is done by running dispersant through so that a portion of sample follows the flow and passes the measuring area where laser detector is used to determine the accurate particle size. Pump speed and ultrasonic displacement should be cautiously adjusted to obtain proper results. Each material was tested several times to ensure the data taken is reliable and with an appropriate technical skill. Graphs show the particle size distribution in the total volume of material run (Figures 11-14).

Portland cement

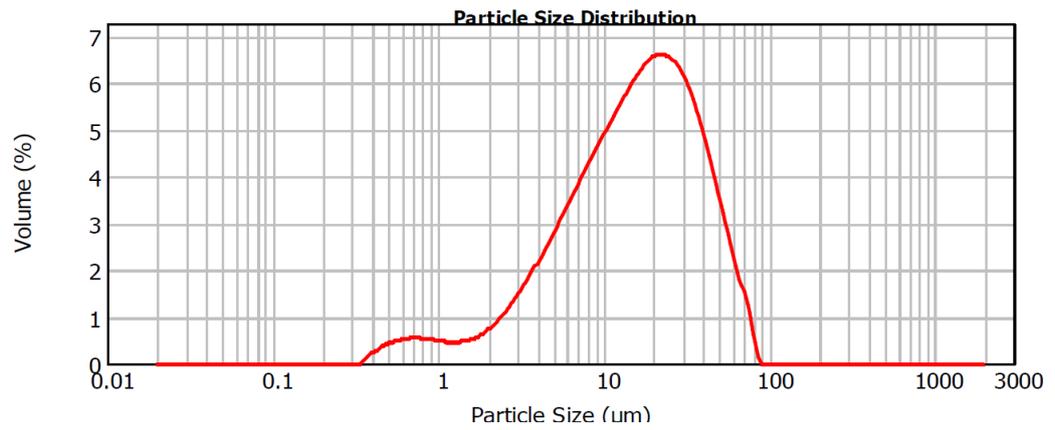


Figure 11. Particle Size Distribution of PC

Fly ash

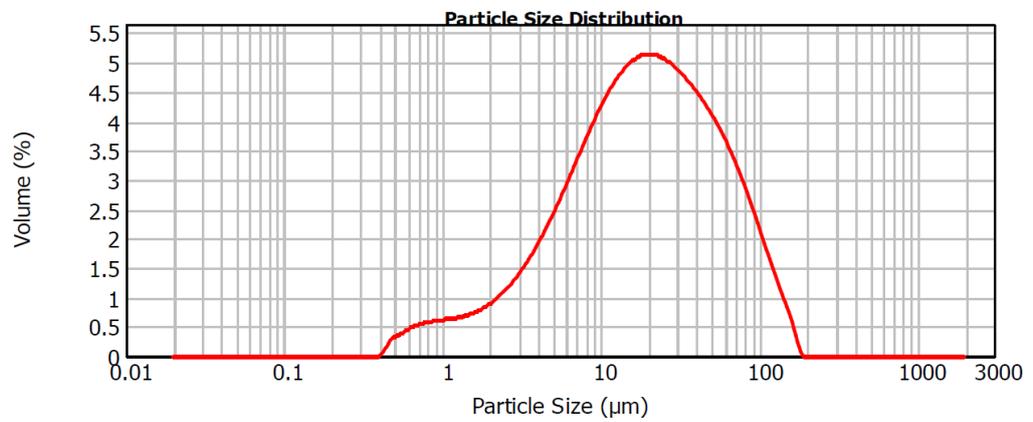


Figure 12. Particle Size Distribution of fly ash

GGBS

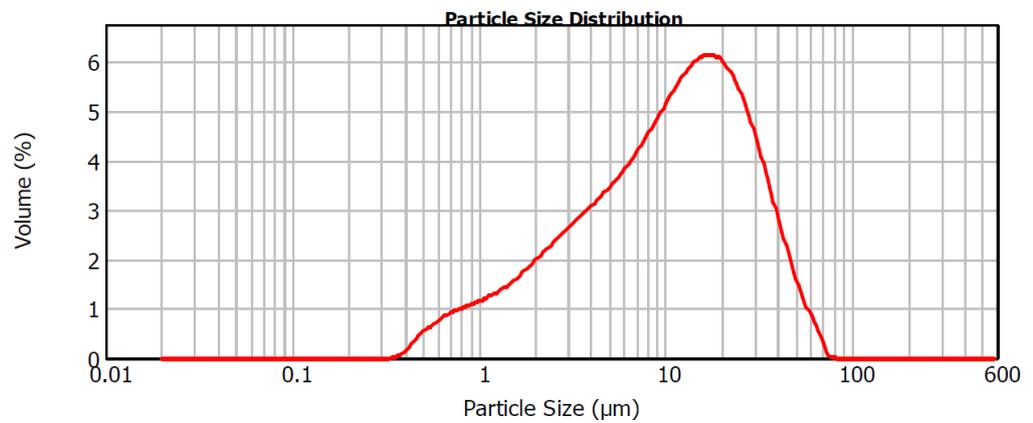


Figure 13. Particle Size Distribution of GGBS

Limestone

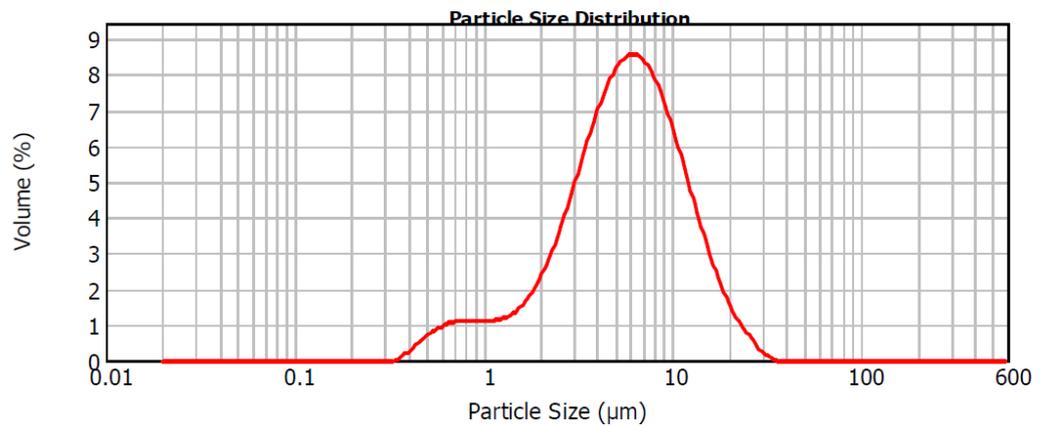


Figure 14. Particle Size Distribution of limestone.

Comparison of four materials

From data recorded in four graphs of PSD mentioned previously and especially from the data log from the Mastersizer software, we can summarise the physical properties of each material (Table 10) and make some comparisons where relevant.

Table 10. Physical property comparison between PC, GGBS, FA, and limestone.

Properties	Materials			
	PC	GGBS	FA	Limestone
Particle RI	1.73	1.65	1.65	1.65
Size Range	0.3-79.4	0.4-69.2	0.4-158.4	0.3-30.2
Weighted Residual (%)	1.1	0.9	0.8	1.2
Specific Surface Area (m ² /g)	0.3	0.5	0.3	0.6
d(0.1) (µm)	3.6	1.8	3.4	1.7
d(0.5) (µm)	16.0	11.0	18.6	5.6
d(0.9) (µm)	43.8	32.7	72.8	13.2

The dispersant used was water with Dispersant RI of 1.330 except with PC, Propan-2-ol was used with RI value of 1.390 due to reaction between cement and water.

As can be seen in Table 10 limestone has the smallest value of mean size as indicated by d(0.5) and, consequently, the tightest size distribution range. In contrast, fly ash has the largest size range which passes through \varnothing 158.489 µm sieve; its mean value of size is also the largest among these **four** materials. PC seems to have least porous or larger size compared with other **three** types due to the modest value of a specific surface area. As the results, the interparticle forces is higher; and rheology and shrinkage become greater.

3.1.3 X-ray fluorescence (XRF) analysis

X-ray fluorescence is an analytical technique used for determining the elemental composition of materials. XRF is a non-destructive method that utilises an X-ray source for the detection of certain chemical elements and their amounts. When X-ray energy causes electron-transfer, a graphic of X-ray intensity peak can be recorded based on energy peak. This spectrum identifies the elements and their quantity (Table 11).

Table 11. Oxide composition by mass (%) of PC, GGBS, FA, limestone

Composition	PC	GGBS	FA	Limestone
CaO	63.4	37.92	3.1	69.34
SiO ₂	19.2	34.66	50.1	2.31
SO ₃	3.2	1.59	1.2	0.6
LOI	3.5	0.73	5.6	0.16

As mentioned earlier (see 2.4.5), the amount of CaO, SO₃ and SiO₂ are critical for shrinkage behaviour due to a chemical reaction after mixing concrete. The other constituents are present in relatively smaller or negligible amounts and do not react and lead to water loss and consequent shrinkage.

3.2 Rheometry test

3.2.1 Introduction

Rheometry tests aim to provide information on the flow under specific shear stress. About a hundred tests are often based on empirical approach instead of **rheological principles**. An American Concrete Institute (ACI 238-1R-08, 2008) report gives the most extensive list and description of these tests including confined flow tests, free flow tests and vibration tests energy (Ferraris, C.F.; Martys, N.S., 2012). One of the widely used approaches is the cone slump which mainly gives the decrease in height of a cone of concrete under gravitational force.

Tattersall, however, applied **rheological principles** to concrete by using the Two-point Test involving the measurement of shear stress under various shear rates along with calculatable plastic viscosity and yield stress. The rheometer is named after Tattersall and operated by rotating an impeller at different speeds while measuring torques. In case the geometry is simple and meets certain criteria, the rotation speed and torque can be converted into a shear rate and a shear stress respectively. The fact is that Two-point rheometer is designed for

concrete with a slump larger than 100mm which is equivalent to medium-to-high workability concrete. This rheometer for concrete encounter is a challenge due to the size of the aggregates. While a fluid rheometer usually has gaps between the shearing planes of a few millimeters or less, the gap, in case of concrete, needs to be at least five times the size of the largest aggregates i.e. 50–100 mm for aggregate sizes of 2-20mm diameter. The material used in this report is mortar whose aggregate sizes range from 0.063 mic-4mm so the issue can be negligible.

An impeller is rotated by a rotor with an adjustable speed knob and submerged in a container of fresh concrete. To avoid sedimentation, the impeller can have either blades or parallel plate or T-bar with vertically fixed movement. The quantity of concrete should be large enough to be representative as well as be small enough to be handled easily. In other words, the bucketful sample brings the satisfactory result with a simple and robust apparatus.

The measurement regime consists of increasing and then decreasing the speed (in predetermined discrete steps) and measuring the torque at each speed. Torque versus speed measurements are plotted and the yield stress and plastic viscosity determined via a regression analysis.

3.2.2 Problem

The non-Newtonian nature of fresh concrete can make it difficult to simply scale measurements using rheometers calibrated with a Newtonian fluid using a simple correction factor. There are further complications in that concrete is a very complex granular fluid (basically particles of different shapes and size embedded in a non-Newtonian fluid matrix) making a continuum approximation of the fluid properties difficult.

To lessen this complexity, the idea is to have as input the properties of the embedding matrix fluid (paste), obtained from accurate experimental measurements, along with information about the size and shape distribution of the aggregates. The model output would be the rheological properties of the mixture. Obviously, the model has to be validated by experimental tests. The power of this approach is that it allows for a detailed study of the general flow patterns in a rheometer, the interaction of the granular fluid with the impeller or the container, and the degree of sedimentation or segregation of aggregates within the rheometer.

3.2.3 Apparatus

Description

There are several models of viscometers (LV, RV, HA and HB) produced by Brookfield specialist (Brookfield Engineering Laboratories, Inc., 2019)

The Brookfield Dial Viscometer RVT of Helipath consists of four main parts:

1. A volume-determined **beaker** (1) contains a tested liquid limit to the capacity of 600ml;
2. A **T-bar spindle** (2) rotated with designated speeds is attached to a rotor powered by a precision synchronous motor;
3. A **Dial Reading** (3) shows the values of torque relatively taken with factors;
4. A **stand** (4) allows the spindle to move vertically in a range of distance adjusted by two ring fasteners.

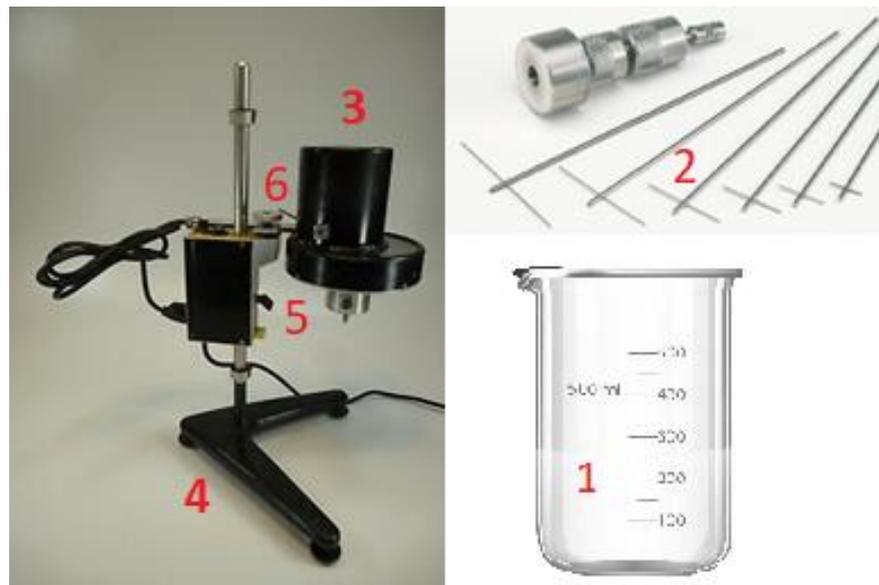


Figure 15. Viscometer- Brookfield model RVT

Speed changes are affected by a gear train having either four or eight speeds. The maximum speed (RPM) will be set at full clockwise rotation and the minimum speed at full counter-clockwise rotation. There is a square speed control knob (5) with two numbers on each face in the ratio of 20:1.

The T-bar spindles have different sizes of diameter. The longer T bar should be, the more liquid the material becomes and vice versa. The one used in this report is type D (or no. 4) with a diameter of 20mm.

The clutch lever (6) located at the back of the Dial Reading is depressed to raise the pointer and hold the instrument's reading. The action should be done dedicatedly and required much practice so that the result taken is more accurate. Each control on Viscometer, including a motor switch, speed change knob, and clutch, is preferably operated independently.

Methodology

The spindle is attached to the viscometer lower shaft by lifting the coupling screw slightly in left-hand thread.

Two ring fasteners on the post of the stand are moved to the points where the spindle can be lifted up and down inside the fluid at the proper depth (to avoid creating void).

Mortar after being mixed is poured into a beaker until it reaches 400ml line (larger volume of material is recommended with longer T-bar spindle) and let the liquid level itself until the surface is visually even.

The spindle is lowered and centred in the material until the meniscus of the fluid is at the centre of the immersion groove on the spindle's shaft.

The motor is switched on and allowed time for the indicated reading to stabilize. It depends on the speed at which the viscometer is running and the characteristics of the sample fluid as at a certain case when the spindle requires time to be fully affected by the material i.e. after at least two revolutions.

The reading is recorded manually at each speed as no digital recorder is used and multiplied by the factor (see Table 12). For the maximum accuracy, readings below 10.0% torque (dial reading) should be avoided.

(note: it is suggested to record data twice by changing speeds from 20 to 0.5rpm and inversely)

Calculation

The reading is multiplied by factors of specific speeds and types of spindle used.
Dial reading x Factor = Viscosity in cP (mPa•s)

Example:

RVT viscometer with spindle 4 (D) at 2.5.rpm factored by 800

Dial reading: 50 so the plastic viscosity is $50 \times 800 = 4000$ (mPa•s) with 1% tolerance.

Table 12. Factors of spindle speed of RVT Viscometer

RV													
1		2		3		4		5		6		7	
.5	200	.5	800	.5	2M	.5	4M	.5	8M	.5	20M	.5	80M
1	100	1	400	1	1M	1	2M	1	4M	1	10M	1	40M
2	50	2	200	2	500	2	1M	2	2M	2	5M	2	20M
2.5	40	2.5	160	2.5	400	2.5	800	2.5	1.6M	2.5	4M	2.5	16M
4	25	4	100	4	250	4	500	4	1M	4	2.5M	4	10M
5	20	5	80	5	200	5	400	5	800	5	2M	5	8M
10	10	10	40	10	100	10	200	10	400	10	1M	10	4M
20	5	20	20	20	50	20	100	20	200	20	500	20	2M
50	2	50	8	50	20	50	40	50	80	50	200	50	800
100	1	100	4	100	10	100	20	100	40	100	100	100	400

☐ = Spindle

■ = Spindle Speed

☐ = Factor

3.3 Shrinkage test: Thin layer measurement

Some experiments have been done to analyse the relationship between shrinkage of fresh concrete over time and types of cementitious materials as well as other ambient factors. As concrete starts shrinking immediately from the point it is set in the formwork, so the equipment measuring shrinkage should record the data in the first important hours and simulate environmental conditions. Mortar is likely to change its volume more quickly than concrete due to the different properties of aggregates used. The tests on mortar can illustrate a similar pattern of shrinkage behaviour on concrete, which will be discussed in Chapter 4.

Thin-layer test and cone test are two of the approaches considering 1D measurement including surface-volume effect and cylindrical effect respectively.

3.3.1 Introduction

Thin layer measurement illustrates the shrinkage process of mortar in terms of horizontal measurement. The sample is poured on a rectangular formwork with Styrofoam fixed along the edges. Building materials, which are capable of self-levelling, will “flow” under certain initial force applied on it (gravitational force by pouring action). A fast setting mortar sets and hardens within a couple of hours and the drying process is generally terminated after the first 24 hours. This basically allows the investigation on different stages of the early shrinkage and expansion, namely the plastic shrinkage, setting expansion and drying shrinkage. The factors that affect the behaviours are both external (climate) and internal (formulation) according to the manual of thin-layer measurement apparatus by a German producer (Schleibinger Geräte, 2015). Figure 16 below shows free shrinkage over the time in a general way in which there are three ranges using different measurement techniques, namely fluid (F), starting of setting (S) and hardened material (H).

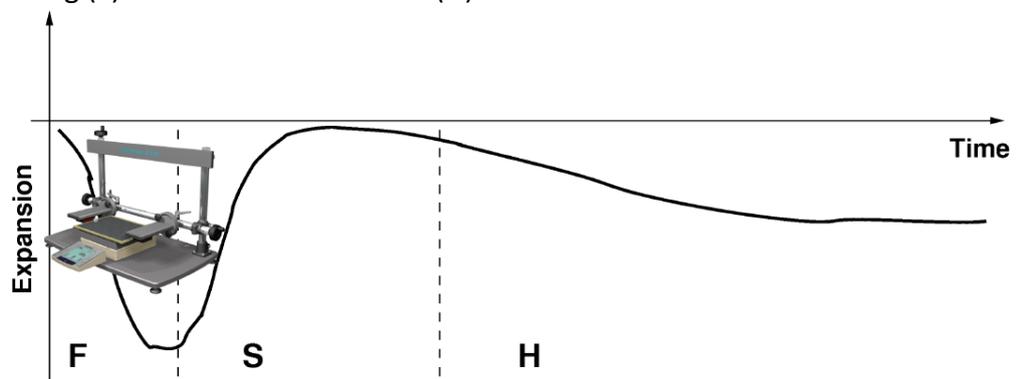


Figure 16. Shrinkage over time (Brookfield Engineering Laboratories, Inc., 2019)

3.3.2 Problem

The layer of mortar in this test is moderately 6mm thick, so consequently, the critical concern is that the **high ratio** of surface area over total volume accelerates **water loss**. In other words, the evaporation is the dominating mechanism for fast and intense physical shrinkage, whereas the hydration reaction leads to a pronounced chemical shrinkage. In case of Ettringite formation, it becomes a strong expansion. However, as long as a material is in the fluid state shrinkage is not causing a problem until mortar starts hardening. The only thing needed to keep in mind is that a length change on each side of a 1 cubic meter cube of 1/1000 is already a volume change of about 3 litres.

When the material is setting and/or is in contact with a material that has no shrinkage or expansion (plastic foil underneath), strain inside the material or in the contact zone will appear. As soon as this tension will be over the actual tensile strength of the material, the material structure will be damaged, usually by the occurring of cracks. It can be related, in practice, when pouring concrete on top of each other.

3.3.3 Apparatus

Description

The equipment includes two main parts (see Figure 17 and Figure 18):

Two lasers (1) project to reflectors (10x10x10mm) made of Styrofoam cube attached to a small piece of dull aluminium foil on the sides. They are adjusted by handwheels (2) on both sides and aligned horizontally to the formwork so that this 1D measurement of the sample provide the data of shrinkage in only one direction.

A **formwork** (3) (700x375mm), consisting of plastic foil and Styrofoam rim (6mm), is placed on a scale (4) as a support which measures the change in weight of the sample over time. The formwork is also fixed to a glass panel and the two light-weight reflectors are put on top of the surface of mortar.



Figure 17. Thin-layer shrinkage measurement apparatus

The data logger monitors the data from the machine including displacement of two cube reflectors, temperature, humidity and/or weight.

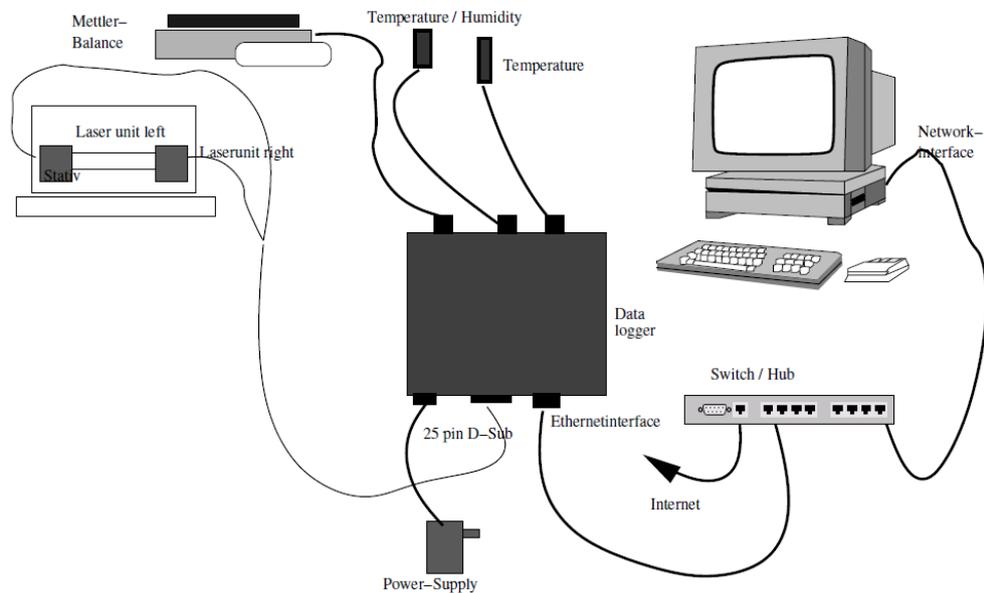


Figure 18. Components of thin-layer measurement

Methodology

The specimen is filled in the formwork and the reflectors are set on the surface about 10mm away from the formwork border.

Two handwheels adjusting two lasers both sides are moved until LED indicators on the lasers turn to green/yellow which means the measurement is into the range and the raw value of about 0.500 is obtained in terms of Online-Data Numerical. It is ensured that the height of the laser beam (adjusted by a middle handwheel) allows the beam to hit the reflectors in the middle.

Before starting the test, it is required to offset height and time to zero as well as clear the old data-set for all channels. A single command can be done to conclude all previous steps by choosing option "Quick-Start". The temperature and mixing time should be constant. Humidity changes over time due to evaporation, which should be controlled and recorded by the data logger.

Data obtained

All data taken can be illustrated by tables and graphs from software run by a web browser (Mozilla Firefox- as used in this experiment) and then transferred into Excel for further usage. Several channels can be displayed in the same plot, but it is preferable to show critical values for further comparisons of different designed mixes. Time set up is 30s interval. No further interpretation of data is needed.

3.4 Shrinkage test: Cone test

3.4.1 Introduction

Similar to the thin-layer measurement, shrinkage cone test measures cement and concrete shrinkage and expansion but in a vertical direction of specimen in a shape of a cone. This method of measurement differs from the out-fashioned technology with the use of a rigid cylindrical mould which cannot showcase appropriate results due to the existence of horizontal shrinkage/ expansion. It is worth to notice that the sample, under isotropic shrinkage and expansion, is expanding both in vertical and horizontal directions. The cone test as 1D method solves this problem of undefined relocation of the material by the presumption that with an isotropic form volume change, the angles of a body keep the same smaller cone is only changing its height (see illustrated explanation in Figure 19 below).

Under the prerequisite of an isotropic shrinkage (expansion) the radius r and the height h of a cone shrink (expand) the same percentage:

$$h' = k \cdot h \text{ and } r' = k \cdot r \text{ (k for example 80\%)} \quad V'/V = 0.8^3 = 0.512$$

or mathematically

$$\frac{h'}{h} = \sqrt[3]{\frac{V'}{V}}$$

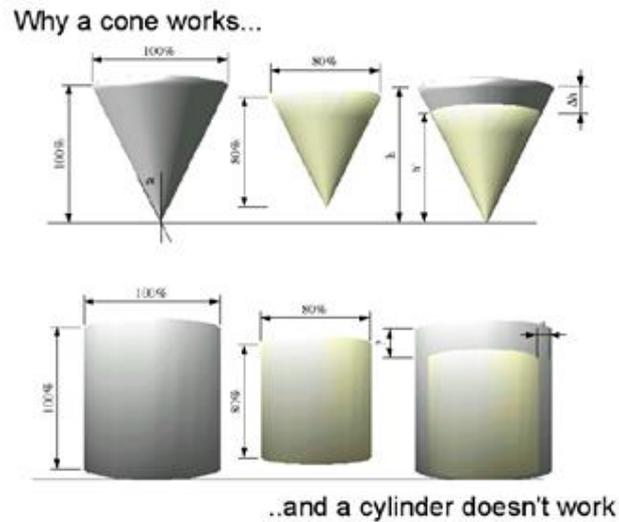


Figure 19. Cone shrinkage and cylinder shrinkage

As mentioned in the session of thin-layer measurement system, the fast setting mortars set and harden within a couple of hours and the drying process is generally terminated after the first 24 hours. The process is affected by several factors including external (climate) and internal (formulation). Since the two shrinkage tests- cone test and thin-layer test- are executed at the same time with a specific sample, it is ideally considered to obtain similar environmental conditions.

3.4.2 Problem

When mortar is poured into the cone formwork, without cautious handling and stable pour, it can cause layer separation. Several observations were made to indicate that water is unlikely to fully bond with all particles and segregation might occur (Figure 20).



Figure 20. Sample after being poured (a) and after 24 hours (b)

3.4.3 Apparatus

Description

The machine consists of two main components (Figure 21):

Cone formed vessel (1) which can contain 349cm³ mortar with 100mm high. The parameters of the vessel are 682 cm³ in volume and 125mm in height.

The container is under a **laser unit (4)** projecting to light-weight reflector placed on top of specimen (3) after being poured.

The Shrinkage cone is designed with a double wall metal vessel for easy heating and cooling using an external liquid temperature control unit. A polypropylene foil (2) helps avoid wall friction. A laser beam is mounted on a high-performance boom stand. A handwheel at the boom stand (5) is used to adjust the distance from the reflector to the laser beam into a measuring range of 4mm.

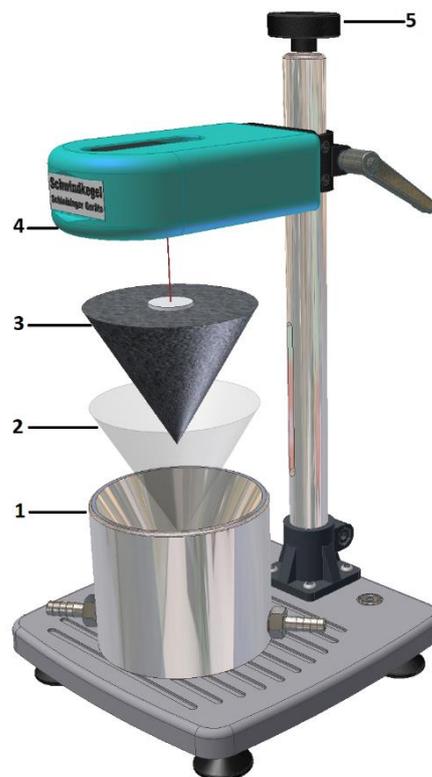


Figure 21. Shrinkage cone apparatus

The laser beam measures the very early shrinkage and expansion in the first minutes and hours after mixing: touch free and with 1-micron resolution. The length change is registered with a resolution of 1/10 micron and data of synchronous length change, temperature, relative humidity is stored by a data logger transferring to a web browser (Mozilla Firefox). There is no **mechanical coupling** between the fluid and the sensor.

Methodology

Mortar is filled into the cone formed container. The handwheel is moved to a certain position where a LED indicator shows green/yellow colour (see Appendix A.2) which means it is in a measuring range with a raw value of about 0.6 (in Online-Data Numerical tab of web-browser software's interface).

Data logger records the data via software and the displacement can be seen along with the control of environmental conditions such as temperature, relative humidity in at least first 24 hours.

Before starting the test, it is required to offset height and time to zero as well as clear the old data-set for all channels. Single command can be done to conclude all previous steps by choosing option "Quick-Start".

Data obtained

The result showcases the change of height of the cone shape sample. Volume change can also be obtained using the relationship between height and volume mentioned in the introduction part. All data taken can be illustrated by tables and graphs from software run by web browser (Mozilla Firefox- as used in this experiment) and then transferred into Excel for further usage. Several channels can be displayed in the same plot, but it is preferable to show critical values for further comparisons of different designed mixes. Time set up is 30s interval.

4 RESULTS

4.1 Shrinkage test results

4.1.1 Thin-layer measurement test result

The total amount of shrinkage during the first 24 hours was measured to determine how six mixtures with different constituent proportions changed shrinkage behaviour under horizontal restraint (Figure 22). The displacement (μm) can be equivalent to shrinkage (mm/mm or unitless) over the horizontal length of 210mm of sample.

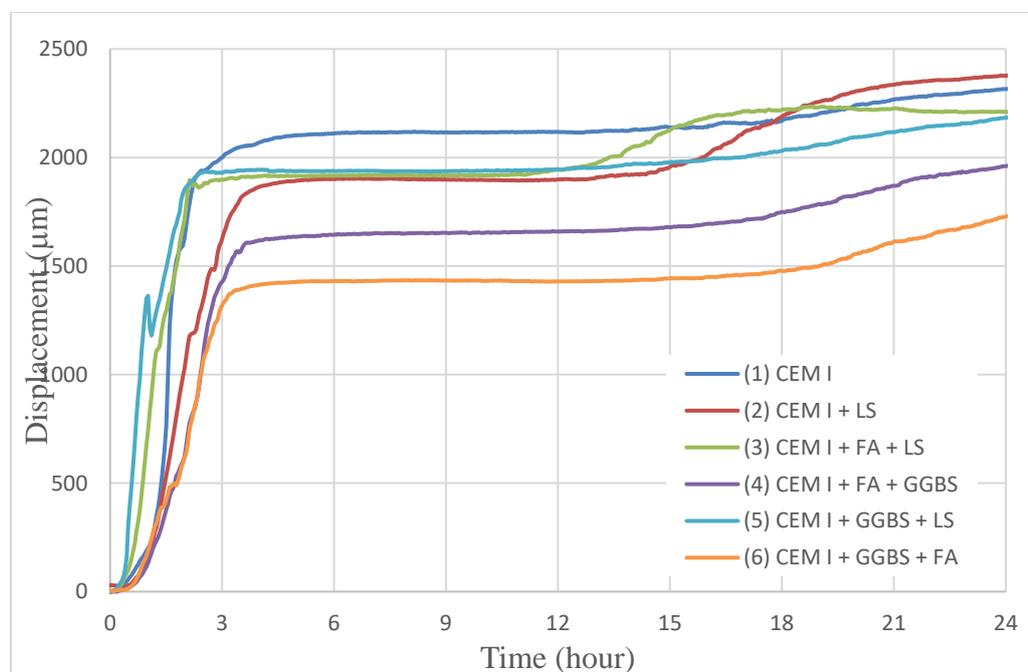


Figure 22. Thin-layer measurement: total displacement of six mixes with combined cement

The partial replacement of 10% by limestone resulted in the tendency for a smaller displacement (mix 1 and 2), but at the end of the period the total displacement is approximately equal. It could be explained as mentioned in the effect of limestone on shrinkage (see 2.4.5). Although limestone slowed down the rate but the proportion of limestone less than 30% showed no significant change overall. Fly ash with a bigger particle size than Portland cement had been expected to shrink less than that of Portland cement per se, and this expectation was met in the recorded data where the total shrinkage was less than the mix containing only limestone (mix 3 and 2).

It can be pointed from the graph that the increase of GGBS in the mixes within the same FA content (i.e. mix 4 and 6) reduces the amount of shrinkage. Therefore, GGBS is likely helping to reduce the shrinkage when combining with FA. However, when GGBS and FA were used individually (i.e. mix 3 and 5), the higher amount of GGBS showed a result similar to that of the mix with fly ash in a lower proportion.

Another approach considers the amount of shrinkage at critical points of time as demonstrated in Table 13.

Table 13. Thin-layer measurement: Shrinkage of six mixes at critical time points (10^{-3} mm/mm)

Time	MIX					
	1	2	3	4	5	6
0.5	0.3	0.1	0.5	0.1	1.5	0.1
1	1.0	0.8	3.5	0.6	6.5	0.9
2	7.8	4.9	8.2	3.0	8.8	2.9
3	9.5	7.8	9.0	6.8	9.2	6.3
4	9.9	8.9	9.1	7.7	9.3	6.7
6	10.1	9.0	9.1	7.8	9.2	6.8
16	10.2	9.6	10.4	8.1	9.5	6.9
24	11.0	11.3	10.5	9.3	10.4	8.2

Most of the mixes show a low rate of shrinkage after being mixed until the first 30 minutes. Also, it is clear that the most shrinkage happens in the first 4 hours. Water evaporation occurs quickly before setting. The amount of shrinkage marginally changed over six mixes during the period starting from the 4th hour to the 16th hour. This is due to setting process taking place. As mentioned in Chapter 2 for setting, the length of setting time (between initial and final setting) should roughly be the same for all materials. However, it can be seen in Figure 4.1 that the mix with only limestone supplement (mix 2) slowed the initial setting time the most nearly at the hour of 4. Furthermore, mixtures with limestone and GGBS or FA (i.e. mix 3 and 5) indicated the shortest time to initial setting, which is also equal to that of pure cement mixture (i.e. mix 1).

Humidity variation

During the tests, relative humidity was measured to display the relation to shrinkage.

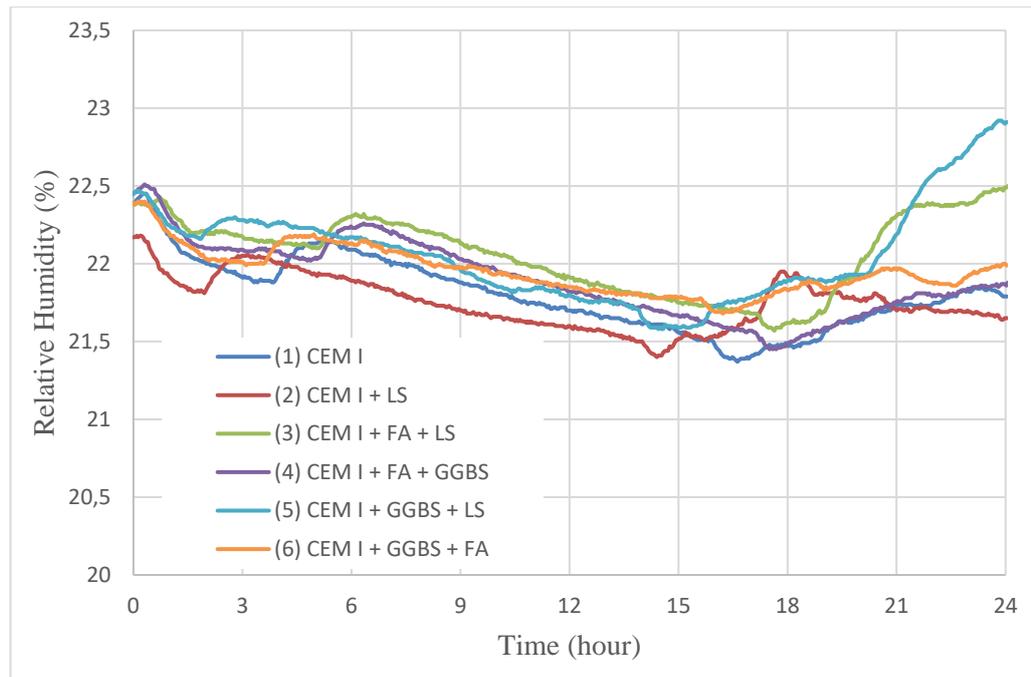


Figure 23. Thin-layer measurement: relative humidity changes of six mixes

Most of the humidity of all mixes sketched the same patterns. Nevertheless, mix 2 seemed more severe change at first 2 hours. It could be because of the quality control of ambient conditions, so the value of humidity did not start similar to other tests. There is some fluctuation between the 15th -18th hour. This happened when mortar started to harden after final setting. Hydration process consequently speed up.

The mean value of humidity of all mixes is shown in Figure 24.

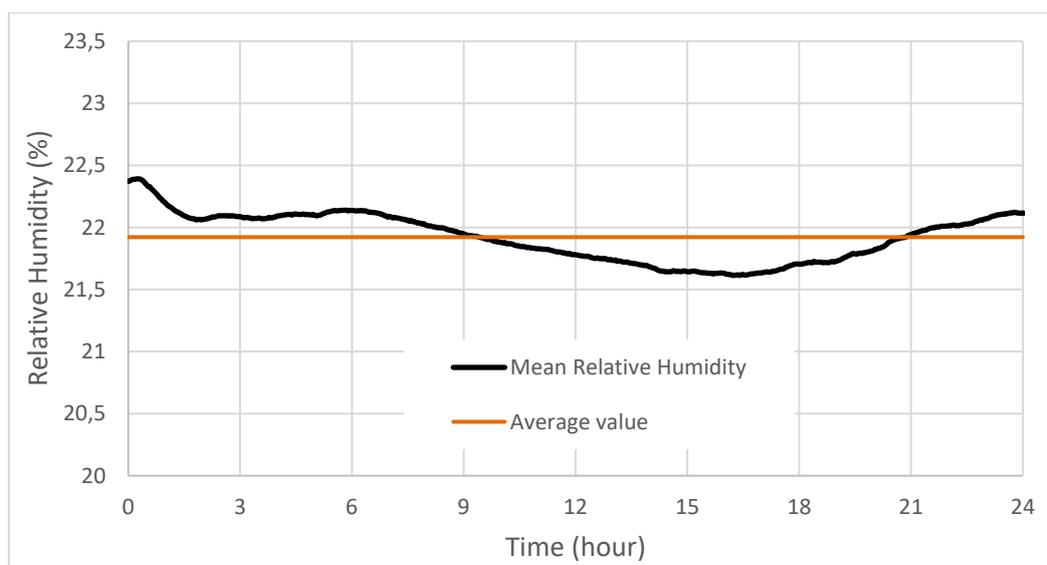


Figure 24. Thin-layer measurement: Average humidity of all tests

Overall, humidity varies from 21.6% to 22.4%. The mean value of 21.9% shows that the ambient condition is considered to be dry for the test lower than 30% for comfortable zone. This condition contributes remarkable effect to shrinkage extent.

Weight loss due to hydration

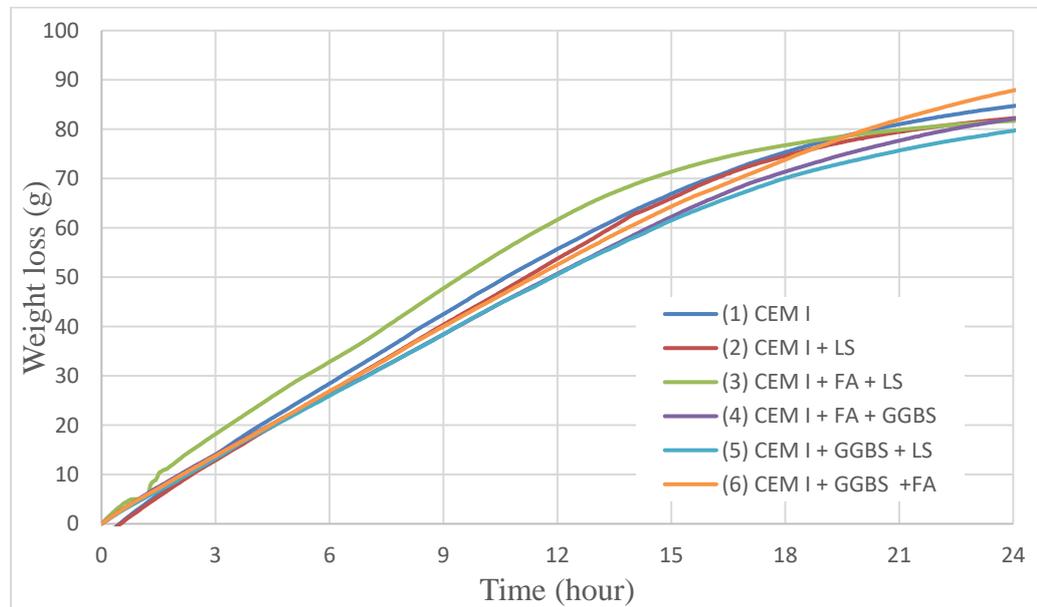


Figure 25. Thin-layer measurement: weight loss of six types of combined cement in mortar

All six combined cement mixes seem to show similar behaviour of water loss which increases steadily over a 24-hour period. It should be noticed that the primary weight of each sample was not the same. However, they share the same surface area as spread and compacted in a thin formwork. Figure 25 illustrates that all mixes started hydrating at almost the same ambient conditions. Cement with the supplementary of fly ash and limestone (mix 3) shows a remarkably higher loss in weight during the first 18 hours by 77g loss but then a lower rate takes place and the sample ends up with the second smallest amount of water lost with about 82g weight loss. The mix with the present of GGBS (dominantly) and FA (increase potential shrinkage- mix 6) shows the highest amount of water evaporation of approximately 90g. The mix of CEM I, GGBS and limestone accounts for the lowest number of weight loss during the whole period with 80g at the 24th hour.

It can be assumed that after 24 hours, water evaporation on surface and water loss due to hydration also encounter the effect of drying shrinkage as long as mortar/concrete starts hardening, and water movement seems to occur more slowly.

4.1.2 Cone test result

Similar to the thin-layer shrinkage measurement, cone test was to determine shrinkage behaviour under vertical restraint. The sample placed in a cylindrical formwork would manage the problem of bi-direction shrinkage which meets in case of cylinder formwork. Thin-layer measurement and cone measurement always started to control ambient condition and the consistency of the mixes.

The displacement (μm) can be equivalent to shrinkage (mm/mm or unitless) over the height of 140mm of sample.

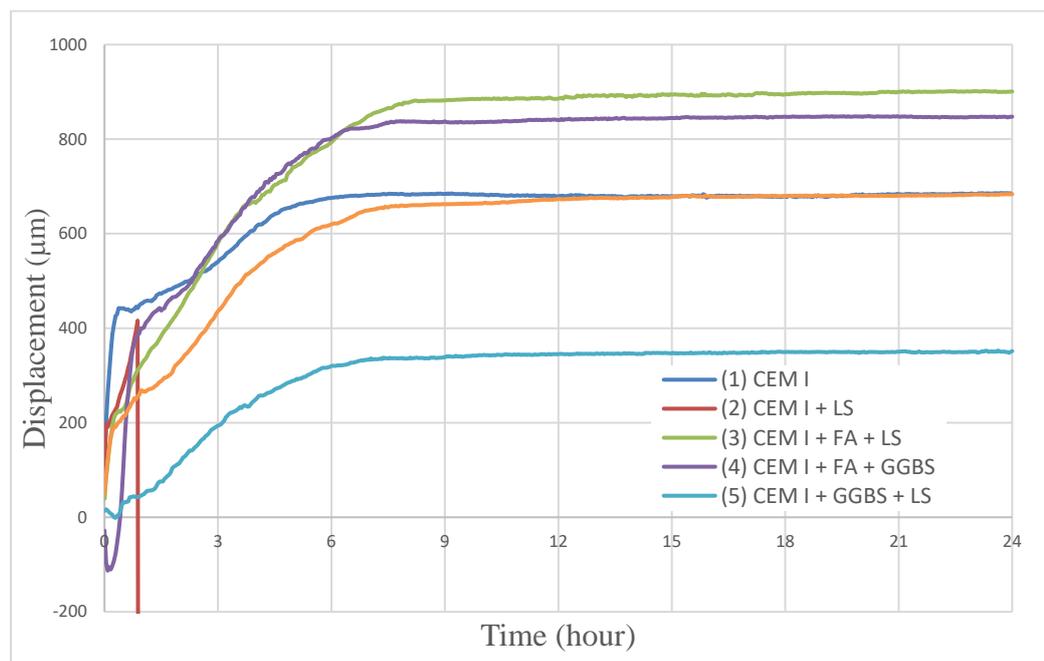


Figure 26. Cone test: total displacement of six types of combined cement in mortar.

Mix containing only Portland cement (mix 1) ended at the same point as that of GGBS and FA combination (mix 6). Similar to thin-layer measurement, the increase of GGBS for PC replacement in the mix with FA reduced shrinkage (mix 4 and 6). However, the alternation of limestone instead of fly ash in mix (5) and (6) was unexpectedly the lowest figures of shrinkage of all mixes.

Mix 2 consisting 10% limestone encountered a technical problem, so the data was recorded for less than an hour. It should have been predicted to have a slightly lower shrinkage than that of pure cement mix.

Alternatively, the shrinkage extent can be described in Table 14 with the main time points.

Table 14. Cone test: Shrinkage of six mixes at critical timepoints (10^{-3} mm/mm)

Time (hour)	MIX					
	1	2	3	4	5	6
0.5	3.2	2.0	1.6	0.8	0.2	1.5
1	3.2	-	2.4	2.9	0.3	1.9
2	3.5	-	3.2	3.4	0.9	2.4
3	3.8	-	4.1	4.2	1.4	3.1
7	4.9	-	6.1	5.9	2.4	4.6
24	4.9	-	6.4	6.1	2.5	4.9

Mix (4) with limestone supplement only received data for 50 minutes due to a technical problem. It is worth noticing that this mixture evaporated speedily and slightly less slowly than mix (1) containing Portland cement did initially. Then, the mixture's shrinkage extent accelerated to reach approximately $3.0 \cdot 10^{-3}$ before the machine stopped recording.

Most of the mixes show the highest rate of shrinkage after being mixed until the first hour. There is an exception of Portland cement mix (1) that its rate was accelerated during the first 30 minutes then reduced to the lowest rate among other four mixes until the 5th hour when it starts hardening. On the other hand, the other four mixes remained almost the same value of shrinkage after 7 hours placing in formworks. The mix of fly ash and limestone constituted the largest amount of shrinkage over 24-hour testing.

Relative humidity variation

During the tests, relative humidity was measured to display the relation to shrinkage.

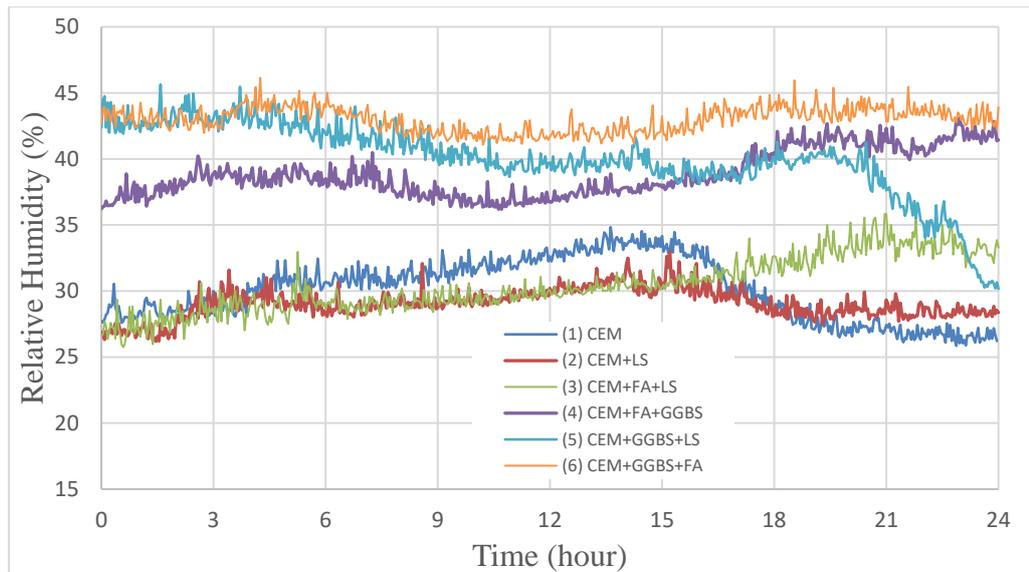


Figure 27. Cone test: Relative humidity changes of 6 types of combined cement in mortar

The combination of these humidity lines from Figure 27 can be acquired to get the mean value for all mixes in Figure 28.

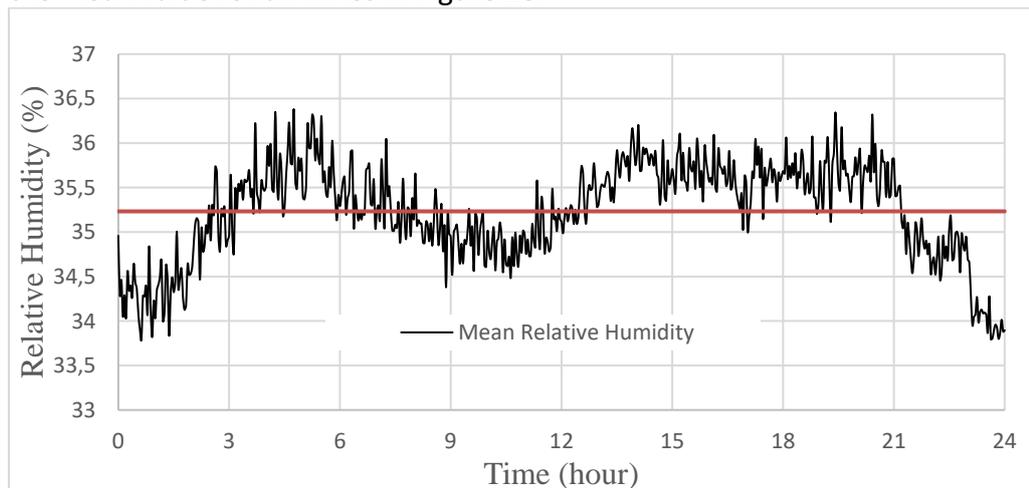


Figure 28. Cone test: Average humidity of all tests

Compared to thin-layer measurement, the relative humidity is much higher within the same samples used. It also shows an opposite pattern when generally the value nearly returned to the original figure of under 34%.

4.1.3 Overall shrinkage result

As the cement used is a combination of different materials with design proportions, the equivalent parameter of the mix can be obtained based on the percentage of each constituent included. The effect of physical properties is demonstrated via particle size distribution, whilst the effect of chemical properties is via chemical composition mainly of CaO , SO_3 and SiO_2 due to their

reaction after mixing. Each property will be analysed in Tables 15-18 and Figures 29-32.

Effect of particle sizes on shrinkage

Table 15. Shrinkage figures of all mixes in relation to particle size

Parameter	MIX					
	1	2	3	4	5	6
Mean Particle Size (μm)	16	14.96	15.74	14.76	12.46	13.76
Shrinkage TLM	11.0	11.3	10.5	9.3	10.4	8.2
Shrinkage Cone	4.9		6.4	6.1	2.5	4.9

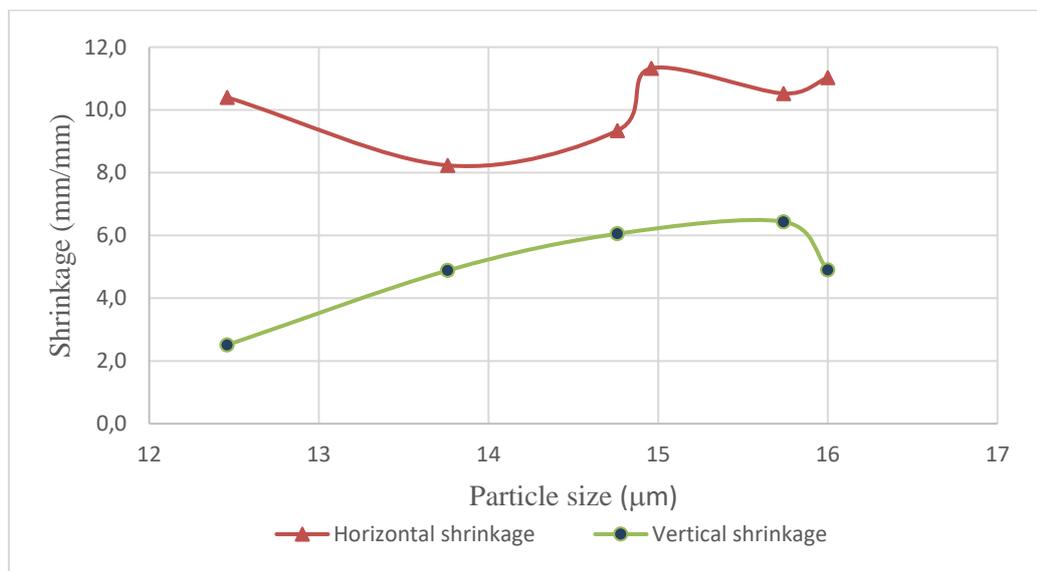


Figure 29. The relation between shrinkage and particle size

Vertical shrinkage was directly proportional to the size of particle, except in the case when only Portland was used. In terms of horizontal shrinkage, the trend was quite similar. Although, the effect of GGBS (e.g. mix of PC, GGBS and limestone) in changing the particle size was dominated by its high fineness leading to active pozzolanic reaction, it seems that the supplement of GGBS showed lower value than that without (i.e. mix of PC and limestone). This resulted in an unreliable behaviour of the mix.

Effect of SO₃ content on shrinkage

Table 16. Shrinkage figures of all mixes in relation to SO₃ content (%)

Parameter	MIX					
	1	2	3	4	5	6
SO ₃	3.2	2.9	2.34	2.52	2.14	2.2
Shrinkage TLM	11.0	11.3	10.5	9.3	10.4	8.2
Shrinkage Cone	4.9		6.4	6.1	2.5	4.9

The increase amount of SO₃ seemingly led to the increase of shrinkage due to chemical expansion horizontally but the trend was unclear when considering vertical shrinkage through cone test (Figure 30).

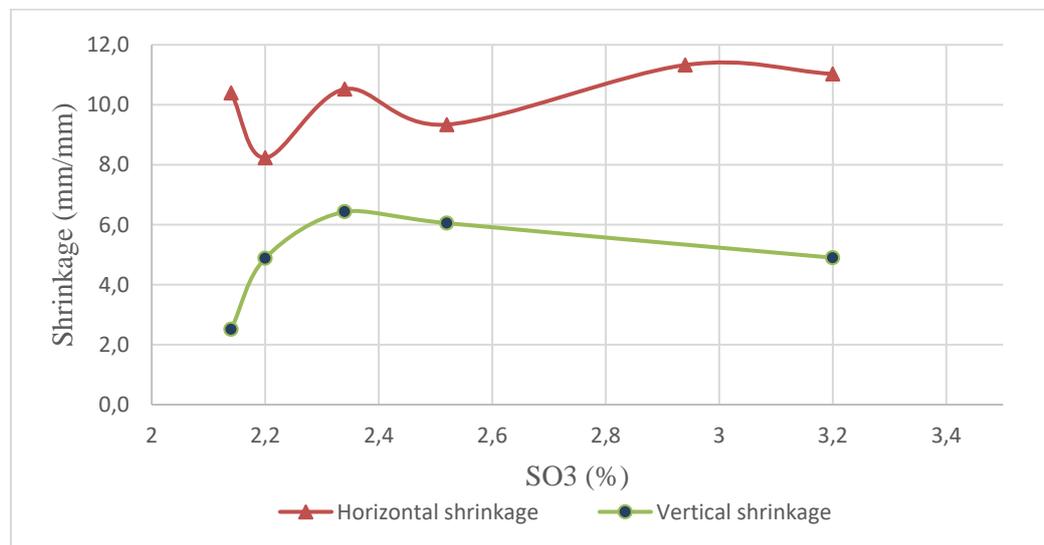


Figure 30. The relation between shrinkage and SO₃ content

Effect of CaO content on shrinkage

Table 17. Shrinkage figures of all mixes in relation to CaO content (%)

Parameter	MIX					
	1	2	3	4	5	6
CaO	63.4	64	45.9	49.7	51.3	44.6
Shrinkage TLM	11.0	11.3	10.5	9.3	10.4	8.2
Shrinkage Cone	4.9		6.4	6.1	2.5	4.9

It can be assumed that the more CaO makes the more material shrinks in horizontal direction. On the other hand, vertical shrinkage showed no general pattern as the curve fluctuates unsystematically.

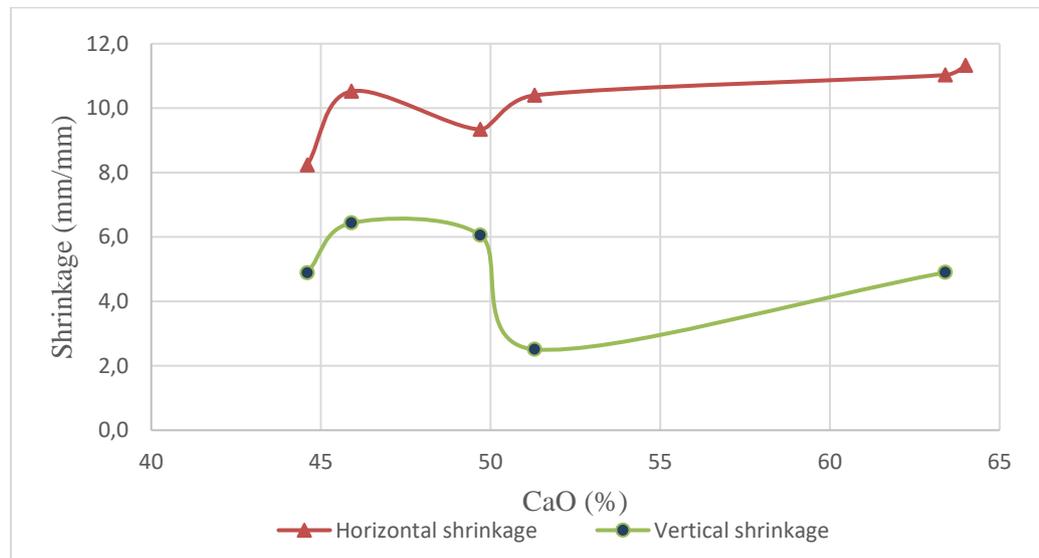


Figure 31. The relation between shrinkage and CaO content.

Effect of SiO₂ content on shrinkage

Table 18. Shrinkage figures of all mixes in relation of SiO₂ content (%)

Parameter	MIX					
	1	2	3	4	5	6
SiO ₂	19.2	17.5	26.8	26.9	25.2	30
Shrinkage TLM	11.0	11.3	10.5	9.3	10.4	8.2
Shrinkage Cone	4.9		6.4	6.1	2.5	4.9

Horizontally the shrinkage reduces as the content of SiO₂ raises. It is also similar to the observation of CaO change that the pattern cannot be found with a methodical approach in terms of cylindrical shrinkage by cone test.

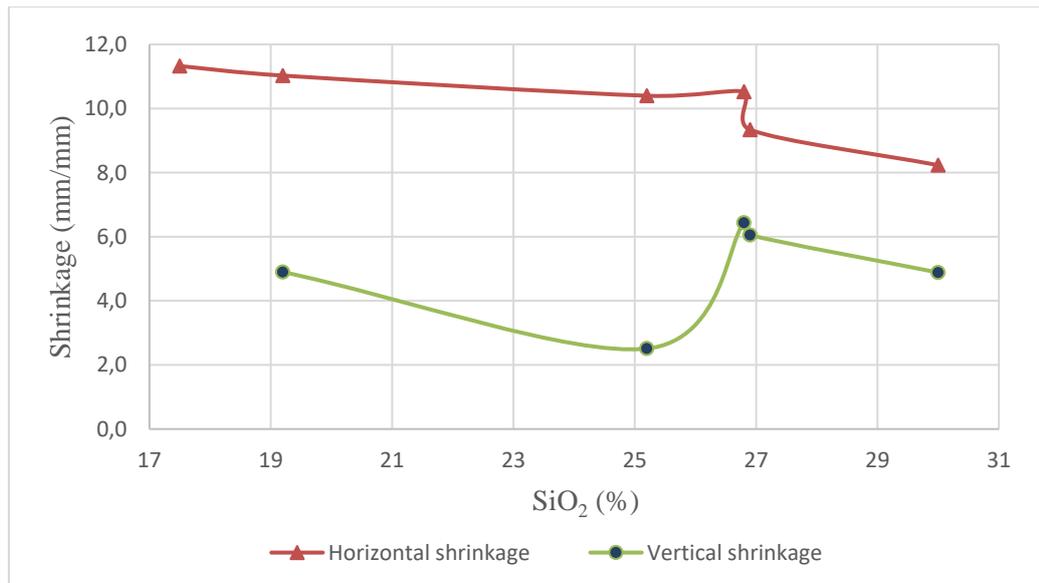


Figure 32. The relation between shrinkage and SiO₂ content.

4.2 Rheometry test results

The Brookfield Dial Viscometer RVT was used to measure the viscosity in relation to the torque and spindle speed with given factors of the calibration. The description and data interpretation can be found in appendix A.1.

It is recommended to record data twice by changing speeds from 20 to 0.5rpm and inversely. However, the Brookfield Dial Viscometer RVT broke down during several test, so the data taken might not be reliable. The procedure can be continued in the future if the machinery is fixed or there is an alternative. Trial measurement had been done before the accident with uncertain data. The steps of testing and data analysis will be considered similarly to the example Table 19.

Table 19. Trial data from a mix containing Portland cement.

Speed (rpm)		0.5	1	2.5	5	10	20
Reading	<i>1st attempt</i>	48	52	72	55	60	52
	<i>2nd attempt</i>	45	58	68	42	56	48
Factor of spindle		4000	2000	1000	400	200	100
Viscosity (cP)		192000	104000	72000	22000	12000	5200
		180000	116000	68000	16800	11200	4800
Mean value (cP)		186000	110000	70000	19400	11600	5000

It can be predicted that a wider particle size distribution gives lower viscosity, and, consequently, the liquid is under higher flow rate (see 2.3.6). The compaction capacity is a significant factor influencing rheological behaviour of the material. Therefore, the mixture of fly ash and limestone seems to be more flowable than other five mixtures. Meanwhile, the combination of GGBS and limestone might be the least workable sample. The prediction is based on combined particle ranges as displayed in Table 20 and particle sizes in Table 21.

Table 20. Combined particle size range of six mixes

	Individual size range (μm)	PC	GGBS	FA	Limestone	Size range (μm)
Mix no.		79.1	68.8	158	29.9	
	1	500	0	0	0	79.1
	2	450	0	0	50	74.2
	3	300	0	150	50	97.9
	4	300	150	50	0	83.9
	5	200	250	0	50	69.0
	6	200	250	50	0	81.8

Also, a larger particle size leads to the higher value of yield stress and viscosity. The mix containing pure Portland cement might give the highest viscosity and yield stress compared with other mixes.

Table 21. Combined particle size of six mixes

	Individual size (μm)	PC	GGBS	FA	Limestone	Mean size (μm)
Mix no.		16	11	18.6	5.6	
	1	500	0	0	0	16.0
	2	450	0	0	50	15.0
	3	300	0	150	50	15.7
	4	300	150	50	0	14.8
	5	200	250	0	50	12.5
	6	200	250	50	0	13.8

The presence of fly ash makes the mix obtain a higher size range, but the overall average size also becomes higher. It can result in higher amount of yield stress and viscosity. Meanwhile, GGBS displays opposite patterns which might lead to higher workability of the mixtures. The effect of limestone is uncertain until the real data is recorded.

Summary observation

4.2.1 Rheology

The prediction of rheological behaviour by changing the proportion of cement constituents was made due to technical problems and unreliable data received. The wider the particle size distribution of the materials, the more compactable the material can become. The reason is that in a disperse system with variation in particle size, the smaller particles fulfil the void between larger particles. This means less void created and, consequently, less water needed to get the more liquid concrete.

4.2.2 Shrinkage

Horizontal shrinkage is likely to indicate more correlation with the variety of physical and chemical properties of polydisperse systems than that of vertical shrinkage. Firstly, horizontal shrinkage increases after 24 hours when increasing the size of particles of material. Also, the chemical composition of CaO and SO₃ also make the samples shrink more horizontally due to their contribution to the loss of water by hydration. The increase of SiO₂, in contrast, reduces the amount of shrinkage because of their reaction with Ca(OH)₂ to create C-S-H gel. Finally, vertical shrinkage only displayed the proportional relationship with the particle size regardless of pure cement's figure. Other factors are unlikely to be influential.

Depending on the direction of the measurement in shrinkage, the result in setting time can vary. In terms of components within a high surface-to-volume ratio such as slab elements, the setting time can start earlier compared to that within a lower ratio such as columns.

5 CONCLUSIONS

The thesis studied how mix design affects the properties of rheology and shrinkage of concrete at the early age. The supplementary materials such as GGBS, fly ash and limestone change the overall parameters of cement. The fresh concrete of these constituents flows and shrinks differently in comparison with concrete made of ordinary Portland cement. The performances of these materials were recorded using 1D laser shrinkage and viscometer. Conclusions are stated below considering the aspect of using sustainable cement and their influences on rheology and shrinkage of fresh concrete.

5.1 Low carbon concrete, rheology and shrinkage

5.1.1 Low carbon concrete material

Pozzolanas as by-products or recycled materials can be collected from other industrial manufacturing processes. This potentially reduces 40-80% emission extent compared with Portland cement (Harvey, 2018). Although pozzolanic materials can only partially replace the content of Portland cement, it produces greener concrete with a lower carbon footprint. The concern of sustainability has been raising up as one of critical criteria in the construction industry.

5.1.2 Rheology

Rheology is an essential study about how concrete flows to formworks after being mixed. Regardless of self-compacting concrete, concrete is unable to level itself without vibration. In order to have a high workability of concrete, yield stress and viscosity of material should be taken in account. Several approaches have been stated but Bingham modelling is the most suitable method in terms of simplifying the correlation between shear stress and viscosity.

Physical properties of constituent materials are considerably more important than chemical properties. Specifically, volume fraction, size ratio, shape and texture, and sand-to-cement ratio are the main factors of rheological behaviour. The addition of admixtures has become a concern nowadays since ambient conditions and other factors might cause hindrance to the process of making concrete. The admixtures reduce yield stress so that the mix can be poured more easily. Although testing was postponed due to a mechanical failure, there are several predictions based on particle size and size distribution. GGBS addition would give lower results of yield stress and viscosity, while fly ash consisting of larger particles than Portland cement would show the opposite scenario. Further examination should be continued to clarify the prediction to a certain extent.

5.1.3 Shrinkage

Shrinkage is an inevitable issue for all concrete products and the control of shrinkage by using sustainable materials was examined in this report. This is important since shrinking to a certain extent may cause cracking. Cracks occur when the tensile strain of concrete beyond its tensile capacity in case there is no embedded material within concrete and externally imposed conditions applied.

GGBS has been proved to increase shrinkage of concrete at both its early age and after that. However, the tests on shrinkage showed that when GGBS and fly ash are all partially replaced by Portland cement content, they helped to reduce shrinkage. The increase of GGBS of these mixtures also indicated shrinkage reduction and resulted in the lowest figure among the other mixes during the first 24 hours. Limestone with less or more than critical contents shows no momentous change in shrinkage behaviour.

5.2 Problems/ Limitations

In practice, the interference of reinforcement affects both workability and shrinkage of concrete. In terms of workability, the pumping concrete might be struggled due to hindering space by *reinforcement bars* installed. These bars interact with particles of fresh concrete and create temporary disjunction. In addition, vibration is likely to be difficult over non-spacious area and insufficient vibration can cause segregation. On the other hand, reinforcement elements affect the tensile strength between metal and particles and increase the degree of shrinkage, which can lead to cracking at the early stage.

Sand absorption is another factor needed to consider. The sand used was not standardized according to CEN Reference sand (see Appendix A.3). The number of particles passing through sieve of 0.08 in diameter was remarkably high that contributed greatly in water consumption. Some mixes without considering very-fine sand resulted in less workability or even become un-flowable. In this case, it can be that the number of small-size particles was added to cement content. In detail, 3% (mean value) was the addition to cement quantity.

5.3 Recommendations

5.3.1 Rheology

The use of viscosity-enhancing admixture has been proved to improve the workability of mixture during fresh state. Where the option of using smaller particles is limited due to strength requirement, concrete workability can be adjusted by the addition of this admixture within dosage recommended by a competent engineer or expert.

5.3.2 Shrinkage

The most common solution to reduce early age volume changes is to avoid drying by proper handling of the concrete for the first few hours after placement. Covering the surface of concrete with polyethylene sheeting after setting can reduce plastic shrinkage or fog spraying to keep the surface moist are also other remedies.

Some materials can be added or adjusted in the mix to reduce shrinkage. According to Chan, et al, introducing fibres or low shrinkage cement in the concrete helps (1999). To avoid risks of cracking in magnificent concrete buildings, CaO based expansive agent had been used in combination with SRA(Shrinkage Reducing Agents). This chemical product is based on calcium sulphoaluminates or CaO which reacts with water, to hinder expansion in reinforced concrete structures (Kar & Sanjay, 2016).

In terms of reinforcement concrete, the rebar design should be taken into consideration such as the increase of cover to reinforcement to reduce restraint. When concrete is still in plastic state, revibration is likely sufficient to avoid settlement cracking.

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APPENDICES

Appendix 1. Brookfield manual of test results

Table 1	Column	Description
	J	Enter model and serial number of your instrument where indicated above data table.
	A	Enter the actual value of your specific calibration fluid. This value can be found on the bottle label or on the accompanying calibration certificate.
	B	No entry required. Calculated 1% of fluid value in column A
	C	No entry required. Shows instrument model.
	D	Enter spindle code for spindle used in calibration check
	E	Enter RPM used for calibration check
	F	No entry required, FSR is calculated based upon spring, spindle and speed
	G	No entry required. Calculated 1% of FSR value in column F
	H	No entry required. % torque value calculated from viscosity in column I
	I	Enter viscosity reading from instrument display

Table 2	Column	Description
	Low Limit	No entry required. Calculated value for low limit of acceptable range
	Reading	No entry required. Value copied from column I in table 1
	High Limit	No entry required. Calculated value for high limit of acceptable range
	Passed?	TRUE = Passed. FALSE = Failed

Figure 33. Explanation of data interpretation from the Brookfield Dial Viscometer **Invalid source specified..**

CALIBRATION TEMPLATE FOR ANALYZING CALIBRATION RESULTS										
<i>INSTRUCTIONS : ENTER VALUES IN BOLD FIELDS ONLY</i>										
<i>Enter value of calibration fluid in column A</i>			<i>Enter spindle code in column D</i>			<i>Enter instrument model: RV serial number:</i>				
				<i>Enter in numerical order the RPMs used in column E</i>			<i>Enter viscosity reading in column I</i>			
A	B	C	D	E	F	G	H	I	Low Limit	High Limit
Actual Fluid Value (cP) from Label or Certificate	1% of Fluid Value (cP)	Instrument Model	Spindle Code	RPM	FSR cP	1% of FSR cP	% Torque (must be >10%)	Viscosity Reading in cP		
-	-	RV	4	0.5	400000.00	4000.00	46.5%	186000	180140	191860
-	-	RV	4	1	200000.00	2000.00	55.0%	110000	106900	113100
-	-	RV	4	2.5	80000.00	800.00	87.5%	70000	68500	71500
-	-	RV	4	5	40000.00	400.00	48.5%	19400	18806	19994
-	-	RV	4	10	20000.00	200.00	58.0%	11600	11284	11916
-	-	RV	4	20	10000.00	100.00	50.0%	5000	4850	5150

Figure 34. Spreadsheet for viscosity reading by AMETEK Brookfield **Invalid source specified.**

Appendix 2. Led colours indicate the range of laser measurement

At the top side of the Laser-head there are a muticolor LED.

color	meaning
green	Everything is ok. You in the measurement range
red	Max or Min range is exceeded !
yellow	Everything is ok. Position of the target in the middle of the measurement range

Figure 35. Cone test: Colour indication of laser.

Appendix 3. Standard sand used

CEN standard sand is a natural, siliceous sand. The particles are mostly rounded and at 98% of silica content.

Table 22. Particle size distribution of the CEN Reference sand

Square mesh size (mm)	2.00	1.60	1.00	0.50	0.16	0.08
Cummulative sieve residue (%)	0	7±5	33±5	67±5	87±5	99±1

Moisture content of sand should be less than 0.2%. the sieving process will be stopped until less than 0.5g/min of particles passing through each sieve.

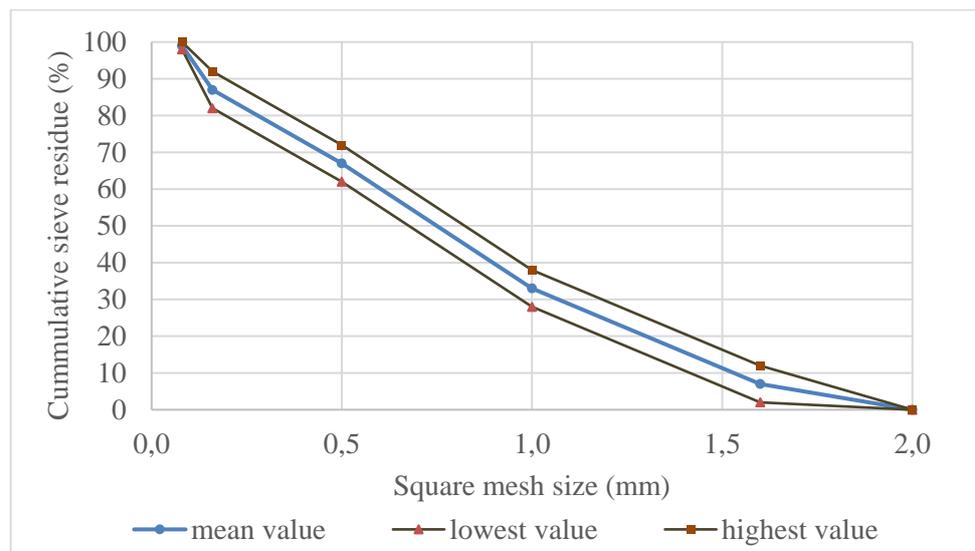


Figure 36. Particle size distribution of sand used