INTRODUCING CEMENT KILN DUST AS A SUPPLEMENTARY CEMENTITIOUS MATERIAL TO REDUCE CEMENT USAGE

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A project report submitted in partial fulfilment of the requirements for the award of the degree of Bachelor of Engineering (Hons) Construction Management

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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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Specially dedicated to

my beloved family and friends.

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ABSTRACT

Cement Kiln Dust (CKD) is an industrial waste produced by cement manufacturing plant, which is collected using air pollution control devices. As a common practice, CKD is being landfilled or introduced back into cement manufacturing plant as raw feed. As massive cement production has led to severe chronic environmental issues, such as abundant CKD produced, air pollution and greenhouse effects, cement usage has to be reduced. CKD is potentially to be incorporated into cement mix. In order to utilize CKD, a deeper understanding of its characteristics has to be studied. In current research, a series of laboratory tests have been performed for the purpose of identifying the physical, morphological, chemical and early-hydration characteristics of CKD. The tests include PSD, SEM, EDX, XRF, XRD, LOI, TGA, FTIR, SCT and STT. After conducting these tests, it shows that CKD is generally feasible to be used as a SCM. In terms of physical property, CKD is relatively finer than OPC by having average particle sizes of 0.90 µm and 15 µm respectively. Besides, in terms of morphological and chemical properties, CKD is generally feasible to be used as SCM as it possesses similar texture, shapes and comparable chemical constituents to OPC. On the other hand, the early-hydration investigation on CKD concluded that it requires greater water demand which is 52.0 % compared to PCC which is 25.0 %. CKD also possesses longer setting times compared to PCC, in which the initial and final setting times are 80 and 510 minutes respectively. However, the initial and final setting times for PCC are 60 and 170 minutes respectively. In addition, it can be concluded that the strength development in CKD is relatively low compared to OPC.

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LIST OF SYMBOLS AND ABBREVIATIONS

°	Degree
%	Percentage
℃	Degree Celsius
℉	Degree Fahrenheit
g	Gram
keV	Kilo Electron Volt
m	Metre
mm	Millimetre
µm	Micrometre
nm	Nanometre
min	Minute
mW	Milliwatt
MPa	Megapascal
℃/min	Degree Celsius per Minute
cm^2/g	Square Centimetre per Gram
g/cm^3	Gram per Cubic Centimetre
N/mm^2	Newton per Square Millimetre
$\boldsymbol{n}$	An Integer
$\boldsymbol{d}$	Spacing between Layers of Atoms, m
$\boldsymbol{C}$	Weight of Blended-cement Added, g
$\boldsymbol{P}$	Percentage of Water in Weight, %
$\boldsymbol{W}$	Weight of Water Added, g
$\boldsymbol{W_0}$	Initial Weight of CKD sample, g
$\boldsymbol{W_1}$	Final Weight of CKD sample, g
$\boldsymbol{LOI}$	Percentage of Loss On Ignition, %

$\boldsymbol{\lambda}$	Wavelength of the X-ray, m
$\boldsymbol{\theta}$	Angle between Incident Rays and Surface of the Crystal, °
Al	Aluminium
Al_2O_3	Aluminium Oxide
C	Carbon
CO_2	Carbon Dioxide
Ca	Calcium
CaO	Calcium Oxide
CaO_f	Free Calcium Oxide
$Ca(OH)_2$	Calcium Hydroxide
$CaCO_3$	Calcium Carbonate
$CaSO_4$	Calcium Sulphate
Cl	Chlorine
Cr_2O_3	Chromium (III) Oxide
Fe	Iron
Fe_2O_3	Iron (III) Oxide
H	Hydrogen
H_2O	Water
K	Potassium
KCl	Potassium Chloride
K_2O	Potassium Oxide
Mg	Magnesium
MgO	Magnesium Oxide
Mn	Manganese
Mn_2O_3	Manganese (III) Oxide
Na	Sodium
NaCl	Sodium Chloride
Na_2O	Sodium Oxide
NO_x	Nitrogen Oxide
O	Oxygen
P_2O_5	Phosphorus Pentoxide

S	Sulphur
SO_2	Sulphur Dioxide
SO_3	Sulphur Trioxide
Si	Silicon
SiO_2	Silicon Dioxide
TiO_2	Titanium Dioxide
-OH	Hydroxyl
Ca^{2+}	Calcium Ion
Cl^-	Chloride Ion
CO_3^{2-}	Carbonate Ion
OH^-	Hydroxide Ion
C_3A	Tricalcium Aluminate
C_4AF	Tetracalcium Auminoferrite
C_2S	Belite or Dicalcium Silicate
C_3S	Alite or Tricalcium Silicate
APCD	Air Pollution Control Device
C-A-H	Calcium Aluminate Hydrate
C-S-H	Calcium Silicate Hydrate
CEM	Cement
CKD	Cement Kiln Dust
EDX	Energy Dispersive X-ray
EPA	Environmental Protection Agency
ESP	Electrostatic Precipitator
FTIR	Fourier Transform Infrared Spectroscopy
GBFS	Granulated Blast Furnace Slag
GCT	Gas Conditioning Tower
LOI	Loss On Ignition
MS	Marble Sludge
OPC	Ordinary Portland Cement

PCC	Portland Composite Cement
PSD	Particle Size Distribution
SCM	Supplementary Cementitious Material
SCT	Standard Consistency Test
SEM	Scanning Electron Microscopy
STT	Setting Time Tests
TGA	Thermogravimetric Analysis
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

CHAPTER 1

INTRODUCTION

1.1 Background

From the past two centuries, construction industry has developed rapidly due to the advancement in technology and the rapid growth of global population. Moreover, as the standard of living gets higher, people expect a higher level of comfort in their daily life. Thus, the sustainable rise in market demand has stimulated the construction industry to increase their output.

The output of construction activities includes residential building, commercial buildings, industrial buildings and infrastructures such as dams, bridges, highways etc. In most of the construction works, concrete is the most widely used construction material. Since cement is the main constituent of concrete production, this is where the demand of cement comes from. According to the latest research, around 3.6 billion tonnes of cement is produced globally every year, with volume predicted to rise to more than 5 billion tonnes by 2030 (El-Attar, Sadek, & Salah, 2016). However, the increase in production of cement has led to several chronic environmental issues. The main concern of cement production is the consumption of raw materials and the emission gases from the cement production plant. These gases have led to serious air pollution problems and greenhouse effect. Moreover, the production process of cement often creates noise and odours to the surrounding (Stajanča & Eštoková, 2012).

As the production of cement has caused substantial negative impacts to the environment, innovative yet environmental-friendly solutions have to be identified and implemented with the aims to overcome the problem of massive production in cement industry. Generally, there is an effective method to reduce cement production and usage, which is using Supplementary Cementitious Material (SCM). SCM is a material that can be incorporated into concrete mix and contributes to the properties of hardened concrete. Hence, the amount of cement usage can be reduced. One of the most eco-friendliest and pioneering materials that can be used as a SCM is the by-product derived from cement production, which is known as Cement Kiln Dust (CKD).

CKD is a fine particle material removed from the kiln of cement production plant by exhaust gases and collected using APCDs. Generally, CKD composed of silica (SiO_2), calcium carbonate ($CaCO_3$), calcium oxide (free lime, CaO_f), and some minor components such as sulphates and chlorides. According to the Environmental Protection Agency (EPA), CKD is categorized as a non-hazardous solid waste material. (Najim, Mahmod, & Atea, 2014). In the most latest statistical report, roughly 85.9 million tonnes of cement was produced in the United States, in comparison to 4,200 million tonnes produced globally (Stastista, 2016). Consequently, the amount of CKD produced is approximately 15% to 20% from the amount of cement produced which is about 700 million tonnes per year (United States Environmental Protection Agency (US EPA), 1993).

Traditionally, most of the CKD generated is being landfilled. According to Maslehuddin et al. (2008), there are about 200,000 tonnes per year of landfill space that being occupied by surplus CKD in the United Kingdom. Moreover, some of the manufacturer of cement will introduce the CKD back into the cement making process. However, it is not practically effective. This is because the international specifications have restricted the alkaline content in cement to be less than 0.6% to prevent the situation of alkali-aggregate reaction that may cause excessive salt formed in the concrete (Marku, Dumi, Liço, & Çakaj, 2012).

In fact, there are several researchers have carried out their studies regarding the use of CKD as a SCM by incorporating it into concrete mix. Most of the researchers have obtained positive results. However, the properties of CKD are often inconsistent due the different factors in the cement manufacturing system. Hence, with the aim to study the feasibility of CKD to be used as an effective SCM, analyses on CKD properties and its early-age behaviours are carried out in current research.

1.2 Problem Statements

As the national economy is developing rapidly, construction industry is playing an important role in stimulating the economic evolution. When the growth of construction industry is accelerating, it strengthens the process of urbanization and creates a society with intact infrastructures. Meanwhile, the market demand for construction materials begins to increase, especially on cement industry as cement is the primary ingredient for most construction projects. The production of cement continually increases 2.5% per year and is expected to increase to about 3.7 to 4.4 billion tonnes by the year of 2050 (Rubenstein, 2012). Nevertheless, the fast growth of cement industry has caused chronic environmental impacts to the Mother Nature.

Among all the environmental issues arise from cement production, air pollution and greenhouse effects are the most serious issues, which require a greater concern. The pollutants emitted from the cement production comprise of dust, carbon dioxide (CO_2), nitrogen oxides (NO_x) and sulphur dioxide (SO_2) (Stajanča & Eštoková, 2012). According to the report of Rubenstein (2012), approximately 5% of global CO_2 is being produced by the cement industry. Hence, it is the second largest producer of greenhouse gases which cause global warming phenomenon (Najim et al., 2014).

Other than that, natural resources running down is another negative effect caused by massive production of cement. This is because the major raw material used in cement production is limestone, which is extracted from natural large quarries. Typically, 1.65 tonnes of limestone and 0.4 tonnes of clay are quarried for each tonne of cement production (British Geological Survey, 2005). Therefore, an alternative solution must be provided to control the over-usage of cement and to prevent the issue of resources depletion.

On the other hand, there are several scientific evidences which prove that air pollution from the combustion of fossil fuels in cement manufacturing process may lead to various health problems. For instance, itchy eyes, respiratory diseases such as tuberculosis, chest discomfort, asthma, cardio-vascular diseases and even premature death (Mishra & Siddiqui, 2014).

At the same time, CKD produced from the production of cement may also be an environmental pollution problem if it is being treated as a waste instead of a renewable source. In the report of Maslehuddin, Al-Amoudi, Rahman, Ali, & Barry, (2009), UK cement industry estimates that the energy lost in CKD is equivalent to the CO_2 emission per year, which is about 80,000 tons. Yet, this figure increases with the increase in consumption of cement.

Aside from the common understanding of environmental impacts caused by the cement industry, the contemporary issue that is being concerned in current research is the low application of CKD as a SCM in most construction industry. This may be due to the substantial variation in physical and chemical composition of CKDs obtained from different cement plants (Siddique & Rajor, 2012). The variation in the properties of CKDs are due to a plenty of factors such as raw feeds that are being used, type of kiln operation, collection techniques, grade of fuel burnt, CKD collection locations at manufacturing plant, etc. (Gupta, Pandey, & Srivastava, 2015; Naik, Canpolat, & Chun, 2003). Consequently, these factors have hinder most construction industry in including CKD into concrete mix as they could not assure the quality of concrete when blending CKD into the mix due to the uncertain characteristics of CKD.

In order to overcome the environmental issues contributed by cement production industry and the challenges faced when using CKD as a SCM, feasibility of CKD to be used as an effective SCM has been studied. Subsequently, characterization analyses on CKD and determination of early-age behaviours of CKD as a SCM are proposed in detail.

1.3 Aims and Objectives

This research aims to study the feasibility of CKD as a SCM to reduce cement usage by providing a deeper understanding of CKD's properties. To achieve the aim, there are two objectives formulated as follows:

i) To characterize the physical, morphological and chemical characteristics of Cement Kiln Dust (CKD).
ii) To identify the early-age behaviours of Cement Kiln Dust (CKD) as a Supplementary Cementitious Material (SCM).

1.4 Significance of Study

Cement Kiln Dust (CKD), the by-product generated from cement production will become an environmental hazard if it is not being treated in an eco-friendly way. Since CKD possess a cementitious value, it deserves a chance to be reused as a SCM by partially replacing it into cement usage. By using CKD as a partial replacement of cement, the cement usage in construction industry can be reduced significantly. Indirectly, the environmental pollution problems can be lessened.

In order to retain an ecological environment in the society, numerous researches have been conducted regarding the incorporation of CKD as a SCM. Most of the researches have proven that the use of SCM as a partial replacement material will not deteriorate the performance of concrete.

Furthermore, current research aims to improve from previous studies by studying the properties of CKD comprehensively in terms of physical, morphological and chemical characteristics with its early-hydration performance to enhance its potential to be an effective SCM. Meanwhile, the cement industrial waste can be reduced and the environmental issues can be controlled.

1.5 Scope of Study

Current study is carried out to study the feasibility of CKD for the use of SCM. To be more precise, the scope consists of studying the physical, morphological and chemical characteristics of CKD and its early hydration effect when it is being mixed with cement paste. The variable is the percentages of CKD replaced in cement pastes. Sources of CKD may vary in terms of cement plant, method of dust collection (APCDs) and collection point in the system. Controlled sample is prepared in determining the early hydration effect, in which 100% of PCC is being used to make cement paste.

Firstly, the CKD sample collected has gone through a series of experimental tests for characterization purpose. Next, CKD sample is blended with PCC in different proportion to make blended-cement pastes. All the blended-cement mixes are investigated to identify its early-age behaviours. Table 1.1 summarizes all the tests carried out in this research and their respective standards.

Table 1.1: List of Tests and Standards

No.	Test	Standard
1	Particle Size Distribution (PSD)	BS ISO 13320:2009
2	Scanning Electron Microscopy (SEM)	BS ISO 16700:2004
3	Energy Dispersive X-ray (EDX)	BS ISO 15632:2002
4	X-ray Fluorescence (XRF)	BS ISO 29581-2:2010
5	X-ray Diffraction (XRD)	BS EN 13925-3:2005
6	Loss On Ignition (LOI)	BS EN 14346:2006
7	Thermogravimetric Analysis (TGA)	BS EN ISO 11358-1:2014
8	Fourier Transform Infrared Spectroscopy (FTIR)	ASTM E3085 - 17
9	Standard Consistency Test (SCT)	BS 4550-3-3.5:1978
10	Setting Time Tests (STT)	BS EN 196-3:1995

The ultimate objective of conducting these tests is to reduce cement usage by introducing CKD into concrete mix. Nevertheless, the limitation of this scope of study is the engineering and durability properties of concrete when incorporating CKD powder will not be covered due to the limited allowable time in conducting this research.

1.6 Research Flowchart

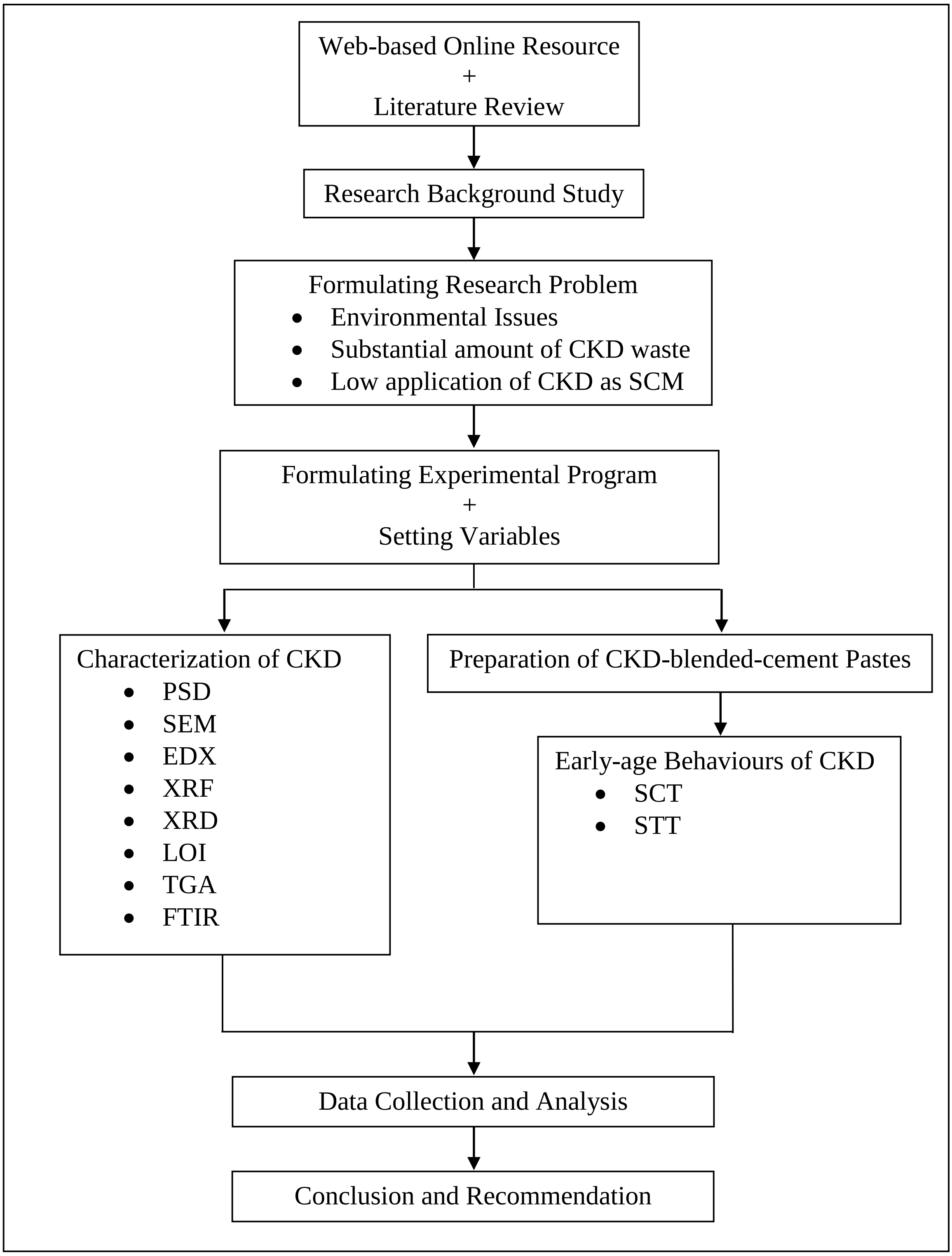

Figure 1.1: General Flowchart of Research

1.7 Thesis Outline

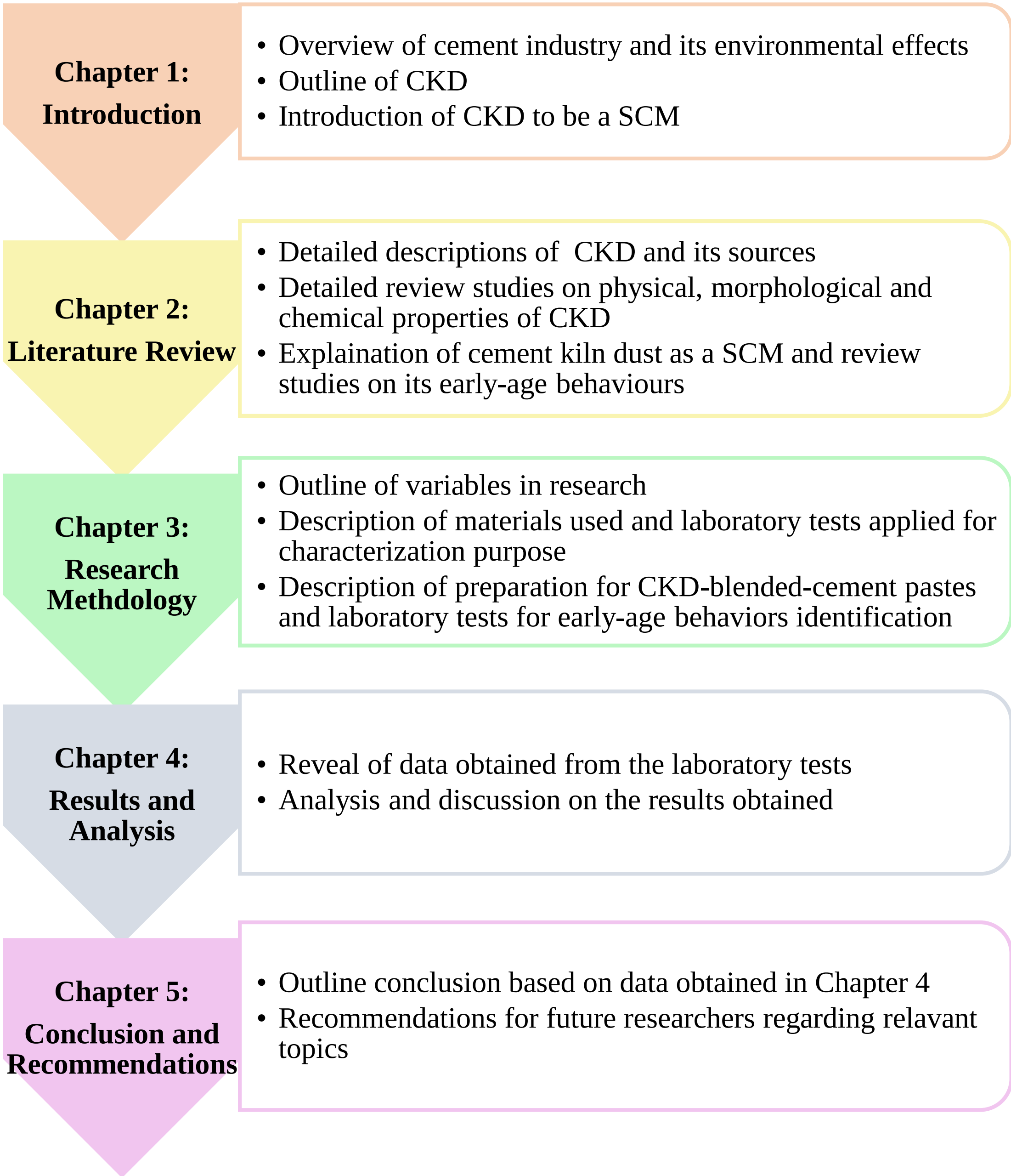

Figure 1.2: Overview of Thesis Outline

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Review of numerous studies carried out by past researchers regarding the characteristics of CKD and its early-age behaviour are reported in this chapter. The sources of CKD and its physiochemical characteristics are firstly described. Physiochemical characteristics of CKD which have been reported in this chapter include the physical properties of CKD, particle size distribution of CKD, morphological characteristics of CKD and followed by chemical properties of CKD. Next, the concept of CKD to be utilized as a SCM has been explained. In addition, the early-age behaviours of CKD as a SCM such as standard consistency percentages and setting times of CKD are being discussed. Throughout the extensive review, the selection of CKD samples to be utilized as a SCM in current research have been determined.

2.2 Cement Kiln Dust (CKD)

CKD can be defined as a by-product material that arises from industry of cement production and it appears in a fine powdery form. The bulk of this fine powdery dust, typically with high alkaline contents is being discharged from cement kilns with the purpose of avoiding the accumulation of excessive salts in the cement product. This is due to the high alkalinity content in the CKD that may cause neutralization reaction between the alkali and silica in some reactive aggregates in concrete which results in the releasing of excessive salts (Daous, 2004).

Moreover, CKD mainly consists of raw materials that behaved thermally unchanged, dried out clay, oxidized limestone, ash produced from combustion fuel, iron and chemical compounds that are newly produced such as lime, silica, alumina and etc. (Naik et al., 2003; United States Environmental Protection Agency (US EPA), 1993). However, the physical, mineralogical and chemical characteristics and compositions of CKD vary from plant to plant. These subject to the raw feeds that are being introduced, type of kiln operation, method of collection used, type of fuel consumed and the CKD collection points at different sections of a particular cement manufacturing plant(Gupta et al., 2015; Naik et al., 2003). It is shown in the study of Naik et al. (2003), where the concentration of free lime (CaO_f) that exists in CKD is usually highest at the dust collection point which is near to the rotatory kiln and it seems to be in a coarser texture. Furthermore, CKD with finer particles collected at points which are far away from the kiln is having higher concentration of sulphates and alkalis.

2.2.1 Source of CKD

CKD is a fine-grained solid material produced from cement production and is considered as the primary by-product from the cement making process. Typically, cement is being produced at high temperatures within a huge rotary kiln. It is where the finely ground raw materials enter and move downward to the cold end of the kiln, whereas the fuels and combustion air are being introduced and drawn upward from the hot end of the kiln. As the combustible air passes through the kiln, it will carry some of the finely ground raw materials, condensed fuel components and partially reacted feed to move toward the next section of the plant (United States Environmental Protection Agency (US EPA), 1993).

There are two types of cement kiln processes which are wet-process kilns, in which the raw materials flow into the kiln in a slurry form; and dry-process kilns, which allows the raw materials to enter into the kiln in a dry, ground form (Rahman, Rehman, & Al-Amoudi, 2011). Figure 2.1 and 2.2 illustrate the cement production in the rotatory kiln system through dry kiln and wet kiln process respectively.

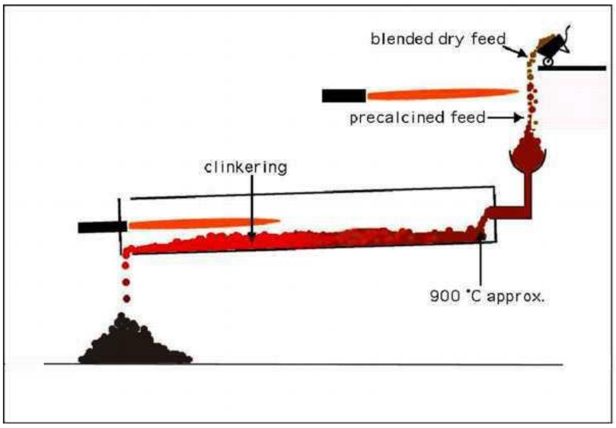

Figure 2.1: Dry-process Kiln ("Manufacturing - the cement kiln," n.d.)

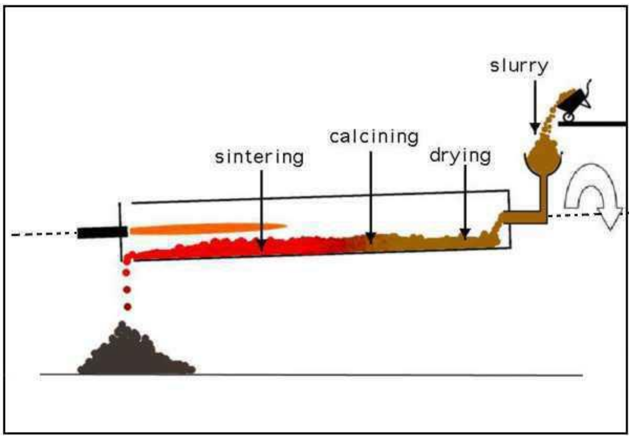

Figure 2.2: Wet-process Kiln ("Manufacturing - the cement kiln," n.d.)

Before the combusted air is being vented to the surrounding through the exhaust stacks, entrained solid substance is collected. These collected gases which constitute of CKD are required to be controlled by air pollution control devices (APCDs), otherwise they will be discharged to the atmosphere (United States Environmental Protection Agency (US EPA), 1993). Other than the rotatory kiln, there are also several numbers of dust collectors and APCDs located at other sections of a cement production plant. According to Lanzerstorfer and Feichtinger (2016), in order to ensure the low concentration of dust is being emitted and fall under the emission limit, three to four APCDs are commonly use in different sections of a cement production plant.

The APCDs control the amount of dust emissions from the cement plant to the surrounding. In the cement kiln system, the combusted air that moves out from the kiln primarily comprises of carbon dioxide, water, fly ash, sulphurs and nitrogen oxides which are derived from the combustion of fuels, impurity solids in the kiln, tiny feed and clinker materials. It may also consist of slurry water if wet kiln process is being adopted. Hence, these substances will pass through the APCDs and the non-toxic combusted air such as carbon dioxide and water vapour will be exhausted from a stack. However, unwanted clinker contaminants may volatilize in the combustion area of the kiln and precipitate as alkalis, sulphates and chlorine compounds which form part of the CKD (United States Environmental Protection Agency (US EPA), 1993).

2.3 Physiochemical Properties of CKD

Understanding the physiochemical properties of CKD is an important aspect before conducting current research. Thus, a plenty of literatures are reviewed to study the physiochemical properties of CKD which includes the physical, morphological and chemical properties.

2.3.1 Physical Properties of CKD

CKD is a grey or white colour solid powdery material in room temperature. Its appearance is similar to Portland cement. It is odourless and it may possess a high average pH value which ranges from pH 12 to pH 13 when it is soluble in water. However, it is only slightly soluble in water with a solubility of 0.1% to 1%. Moreover, CKD has a high boiling point which exceeds 1000°C (>1832°F). Its relative density is approximately 2.3 to 3.15 g/cm^3 (Cemex, n.d.; Leigh Hanson Heidelberg Cement Group, 2012). According to Gupta et al. (2015), the average specific surface area of CKD ranges from 4600 to 14000 cm^2/g, and its specific gravity is about 2.6 to 2.8.

2.3.2 Particle Size Distribution of CKD

From the aspect of particle size distribution of CKD, the study of El-Attar, Sadek and Salah (2017) had determined their CKD sample grading by using laser diffraction. Their result shows that the particle size distribution of their CKD sample ranged from 0.6µm to 6µm with an average size of 2.45µm.

However, in the study of Lanzerstorfer and Feichtinger (2016), result obtained for the particle size distribution of their CKD samples by using the similar laser diffraction method are deviated from El-Attar, Sadek, and Salah study (2017). This is shown when their CKD samples possess an acceptable range of particle distribution size from 24.82µm to 26.36µm, with a measured median of 25.62µm. Figure 2.3 shows the particle size distribution of CKD sample in the study of El-Attar, Sadek and Salah (2017) . Figure 2.4 shows the particle size distribution of CKD samples collected from three different field of APCDs (ESPs) in the study of Lanzerstorfer and Feichtinger (2016) respectively.

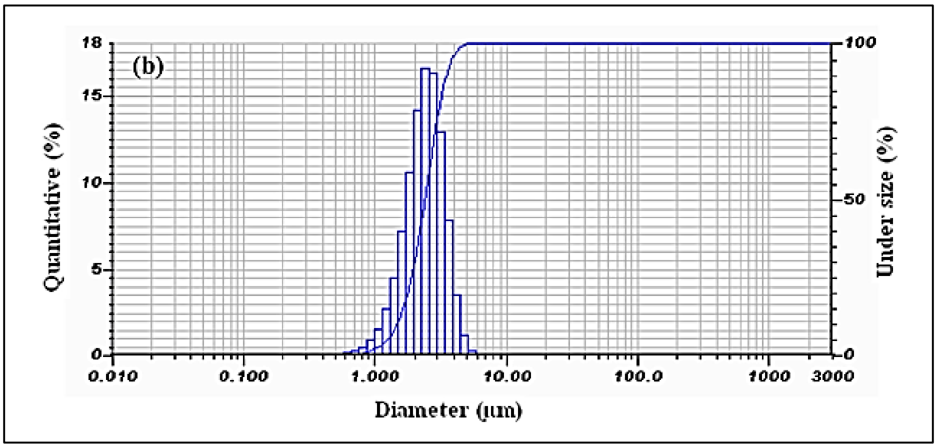

Figure 2.3: Particle Size Distribution of CKD (El-Attar et al., 2016)

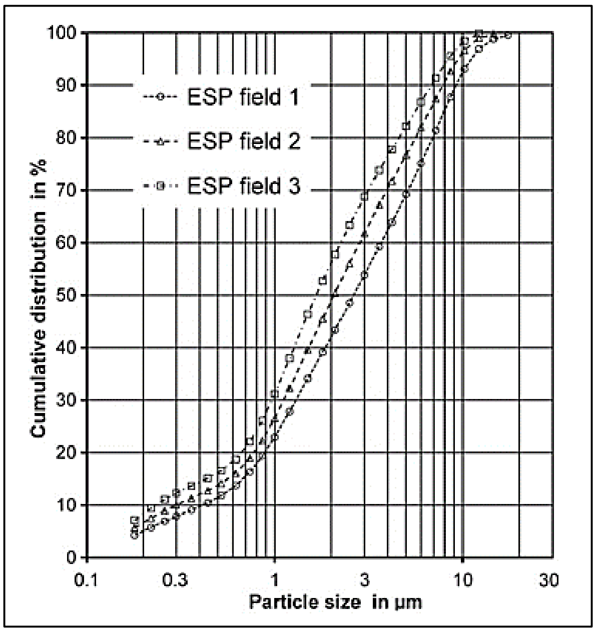

Figure 2.4: Particle Size Distribution of CKDs from Various Fields of ESP (Lanzerstorfer et al., 2016)

The deviation of results obtained between the above studies can be explained whereby the APCDs that were being used to collect their CKD samples may be different. This statement is being deduced because the second study has stated that their CKD samples were collected from a type of APCDs called Electrostatic Precipitator (ESP) whereas the first study did not state the APCDs source of CKD sample. Other than that, the deviation may be also due to the differences between their alkalinity and other factors.

According to Sreekrishnavilasam and Santagata (2005), the particle size distribution of CKD is influenced by the cement manufacturing process technology, method of dust collection (APCDs), chemical composition of CKD, alkali content and etc. Furthermore, the higher the alkalinity, the finer the particle size (Peethamparan, Olek, & Lovell, 2008). One of the factors that affects the particle size distribution of CKD had been proven by Colangelo and Cioffi (2013). In their studies, they had obtained two different types of CKD samples, named CKD_1 and CKD_2. Both CKDs were used to represent plants working in distinct situations and they were collected using different APCDs, which were ESP and baghouses respectively. As a result, particles of CKD_2 are the finest and most uniform compared to CKD_1 and other material samples. CKD_2 possesses a mean particle size of 9 µm and 57% of it ranges from 1µm to 10 µm whereas 93% are finer than 45 µm. However, 75% of the particles in CKD_1 are in the range of 1µm to 45 µm with a mean particle size of approximately 18 µm. Figure 2.5 illustrates the particle size distribution of CKD_1, CKD_2, granulated blast furnace slag (GBFS), marble sludge (MS) and cement (CEM).

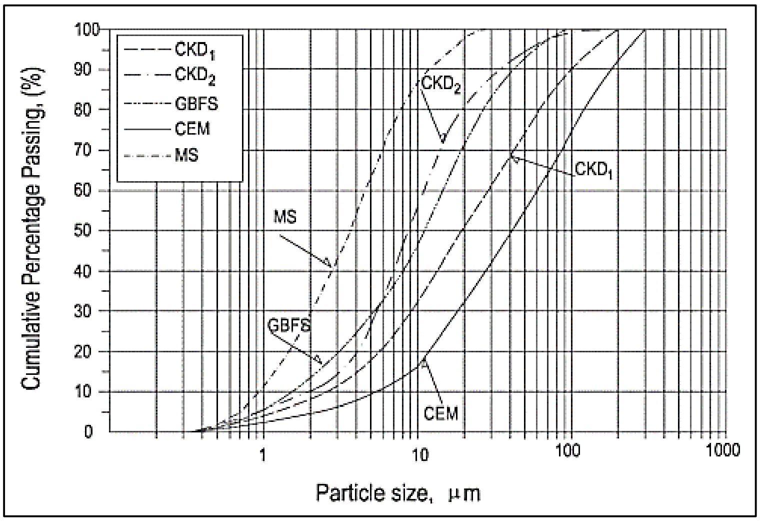

Figure 2.5: Particle Size Distribution of CKD_1, CKD_2, GBFS, MS and CEM (Colangelo & Cioffi, 2013)

2.3.3 Morphological Characteristics of CKD

Under a scanning electron microscope, CKD primarily consists of undispersed clusters or cemented agglomerates of particles with hardly defined shapes. Nevertheless, each CKD composed of some spherical particles which are known as fly ash particles. Figure 2.6 shows the morphologies of four CKD samples (CKD-1, CKD-2, CKD-3 and CKD-4) that are collected from different cement plants which possess different processing techniques, type of kilns and raw materials being used in the manufacturing process. In Figure 2.7, the cluster in CKD-1 has been zoomed in and it shows some of the spherical fly ash particles are covered with extremely fine deposits while the rest particles are having a smooth surface texture (Peethamparan et al., 2008).

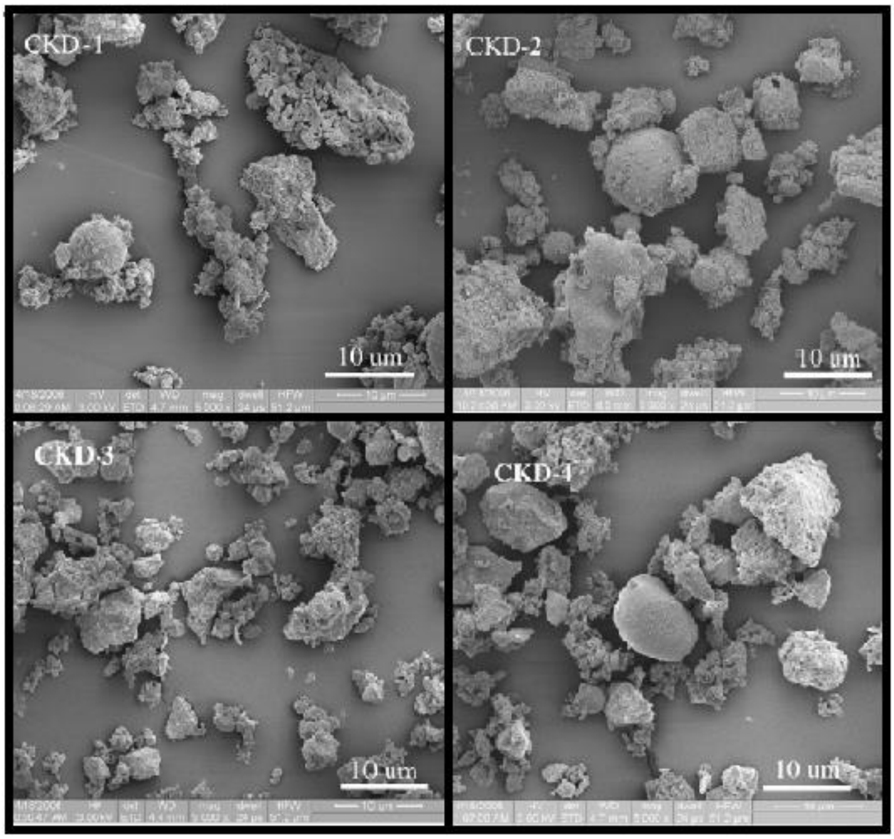

Figure 2.6: SEM results of CKD-1, CKD-2, CKD-3 and CKD-4 to 5 000× magnification (Peethamparan et al., 2008)

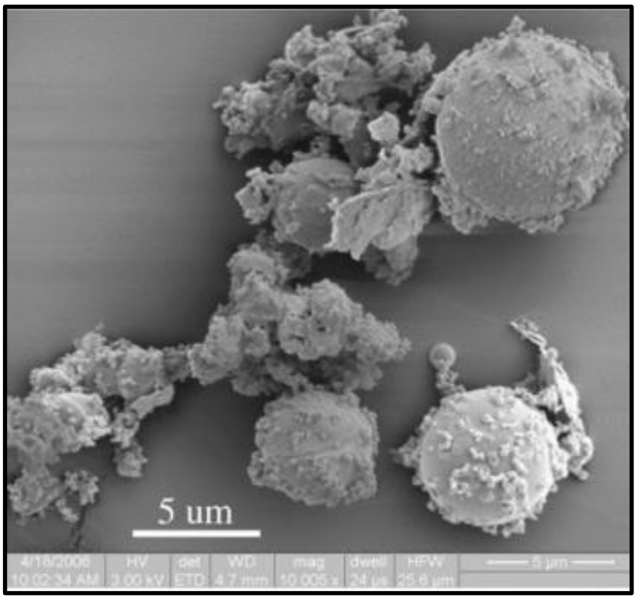

Figure 2.7: SEM result of CKD-1 to 10 005× magnification (Peethamparan et al., 2008)

2.3.4 Chemical Properties of CKD

Although the chemical composition of OPC from the market is found to be extremely consistent, the chemical composition of CKD that derived from their production may be varied significantly. This variability is due to several factors such as the type of kiln operations (wet or dry process), the dust collection techniques (APCDs), dust collection points within the system, fuel being used for combustion in rotatory kiln, etc.(Sreekrishnavilasam & Santagata, 2005).

Despite the fact that chemical composition of CKD varies from plant to plant, the primary constituents of CKD are most likely similar. There are many researchers have conducted their studies on the overall chemical composition of CKD in order to analyse the basic elements and chemical compounds exist in it. According to Anderson (2006), several points from his CKD's SEM results were being analysed through the use of EDX. It shows that there are two basic elements which having highest possible weightage in his CKD sample which are Ca and Si. Table 2.1 tabulates average range of basic chemical elements of CKD from different points in SEM results.

Table 2.1: Basic Elements of CKD (Anderson, 2006)

Elements	Average Weightage (%)
Na	0 – 1.3
Mg	0 – 0.8
Al	0 – 13.4
Si	0.5 – 43.9
S	0 – 6.2
Cl	0 – 0.8
K	0 – 19.4
Ca	0.1 – 43.9
Mn	0 – 0.1
Fe	0 – 3.3
H + O + C	35.9 – 55.9

According to several researchers, the most abundant chemical compounds are calcium oxide (CaO), silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), iron oxide (Fe_2O_3), sulphur trioxide (SO_3), sodium oxide (Na_2O), potassium oxide (K_2O) and magnesium oxide (MgO). Moreover, CKD may contain some other minor constituents such as chromium (III) oxide (Cr_2O_3), titanium dioxide (TiO_2), etc. (Maslehuddin et al., 2009). Table 2.2 tabulates the data of chemical constituents in CKD by various researchers.

Table 2.2: Chemical Constituents of CKD

Constituents	Weightage (%)				
	Chaunsali &Peethamparan, (2013)		Marku et al., (2012)	El-Attar et al. (2016)	Yoobanpot et al.,(2017)
	Sample 1	Sample 2			
CaO	61.15	41.90	43.50	60.56	53.89
SiO_2	14.55	13.16	15.90	6.1	14.94
Al_2O_3	4.46	3.51	3.43	1.37	4.07
Fe_2O_3	2.11	2.59	1.90	3.09	2.27
SO_3	10.62	1.13	1.64	5.41	10.96
MgO	3.84	1.08	-	0.44	1.84
Na_2O	0.80	2.10	4.66	0.64	2.78 (contains other minor constituents)
K_2O	3.45	0.70	0.30	2.56	3.62
CaO_f	-	-	29.40	-	-
Cl^-	-	-	2.56	2.75	-
LOI	-	-	35.50	15.04	5.63

From Table 2.2, all of the studies show that CaO is having the highest percentage content in CKD and it is followed by SiO_2. High percentage of these two compounds in CKD has made it to become valuable and potentially to become SCM. However, CaO_f exists in the CaO compound is another concern as it is the CaO that has not combined with SiO_2, Al_2O_3, Fe_2O_3, etc. during the burning process due to under-burning, insufficient grinding of raw mix or presence of traces of inhibitors. Thus, CaO_f is more valuable compared to CaO as CaO_f is more readily to be reacted with water. According to Marku et al. (2012), the concentration of CaO_f and other cementing materials such as SiO_2, Fe_2O_3 and etc. will determine its cementitious value.

In order to be used as an effective SCM, the mineralogical characteristic of CKD is another important aspect to be studied especially on the phases of SiO_2. SiO_2 is able to behave in different forms, such as crystalline state or amorphous state. According to the study of Sldozian (2014), amorphous SiO_2 (cristobalite) is more reactive in concrete mix compared to crystalline SiO_2 (Quartz) by showing better performance in term of mechanical properties. Several studies from varies researchers show that the primary crystal phases that obtained in CKD are CaO_f, calcite ($CaCO_3$), $CaSO_4$ and SiO_2. Figure 2.8 illustrates the XRD results whereby all the sharp peaks shown in the graph indicate that the compounds are in crystalline structure.

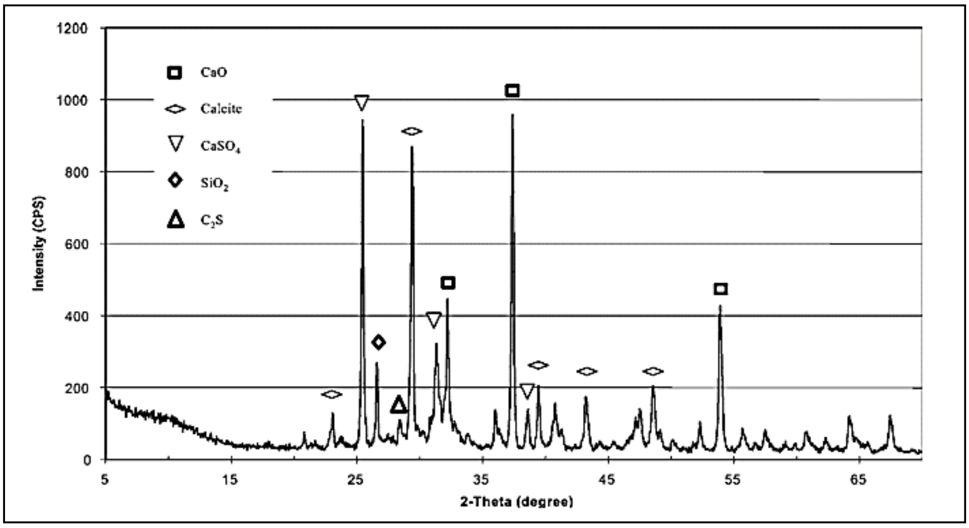

Figure 2.8: XRD Result of CKD (Wang, Mishulovich, & Shah, 2007)

On the other hand, from Table 2.2, some of the researchers studied on the loss on ignition (LOI) of CKD. The percentage obtained from LOI in the case of CKD primarily indicates the rough amount of carbon content and followed by other volatile substances such as alkalis, chlorides, sulphates, etc. The inconsistency of results obtained from LOI indicates the different amount of decarburization of $CaCO_3$ and $Ca(OH)_2$ from the raw feed in rotatory kiln (Maslehuddin et al., 2008; Sreekrishnavilasam & Santagata, 2005).

In order to justify the content of CaO that being produced when CKD undergoes a series of change in temperature, thermogravimetric analysis (TGA) is normally carried out by past researchers. When there is a significant loss of weight in CKD at certain temperatures, it means that carbon dioxide (CO_2) is being eliminated through the transformation of $CaCO_3$ or/and $Ca(OH)_2$ to CaO. These chemical processes are known as thermal decomposition or calcination of $CaCO_3$ and $Ca(OH)_2$ which shown in Chemical Equation 2.1 and Chemical Equation 2.2 respectively (Halikia, Zoumpoulakis, Christodoulou, & Prattis, 2001; Khanna, 2009).

Chemical Equation 2.1: Thermal Decomposition of $CaCO_3$

$$CaCO_3 \rightarrow CaO + CO_2$$

Chemical Equation 2.2: Thermal Decomposition of $Ca(OH)_2$

$$Ca(OH)_2 \rightarrow CaO + H_2O$$

In the studies of Peethamparan et al.(2008), TGA was being implemented on their four CKD samples that collected from different sources. The result shows that none of the samples is having remarkable change when the temperature is heated from 0 to 550℃. When the samples are being heated to 800℃, a substantial loss of weight is being detected in all the samples. Thus, thermal composition of $CaCO_3$ has occurred from 600 to 800 °C. Due to the highest chloride content in one of the CKD samples (CKD-4), thermal decomposition of chloride silicate of calcium ($Ca_3(SiO_4)Cl_2$) has occurred and caused a secondary drop of weight in CKD-4 at an approximate temperature of 915℃. In conclusion, $CaCO_3$ content in CDK-1, 2, 3 and 4 are roughly 30%, 7%, 64% and 58% respectively. Figure 2.9 shows the TGA results obtained from four different source of CKD samples.

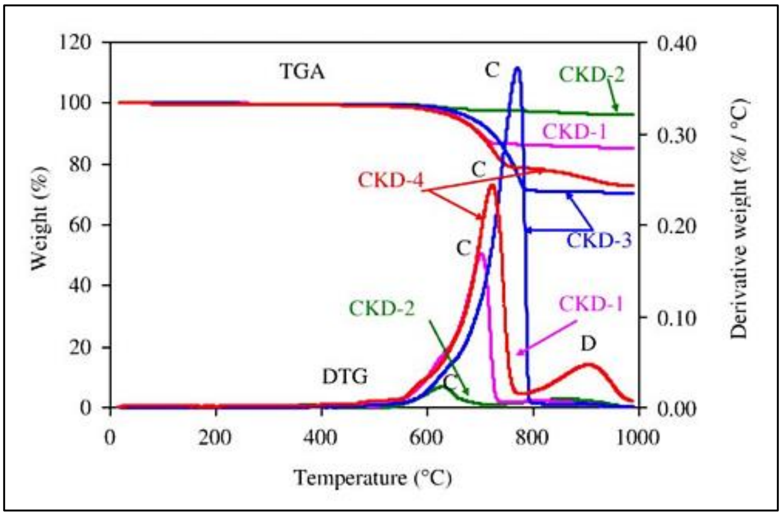

Figure 2.9: TGA results of CKD-1, CKD-2, CKD-3 and CKD-4 (Peethamparan et al., 2008)

where

C = Calcium carbonate ($CaCO_3$)

D = Chloride silicate of calcium ($Ca_3(SiO_4)Cl_2$)

Furthermore, functional groups that present in CKD is another essential aspect in characterizing its properties. A functional group can be defined as a specific group of atoms in a molecule that determine the physical and chemical properties of the molecules. It defines the nature of chemical reactions that may happen within the molecule. In addition, a molecule that contains functional group is said to be more reactive (Daley & Daley, 2005). In the research of Elrefaey (2017), FTIR was carried out on CKD with the purpose of determining the functional groups exists in it by observing the trough positions from the data obtained. According to his results, the three troughs obtained from the result indicate that CKD is having three types of functional groups which are hydroxyl (-OH), carbonate and silicate respectively. Figure 2.10 shows the FTIR result of CKD obtained by Elrefaey (2017).

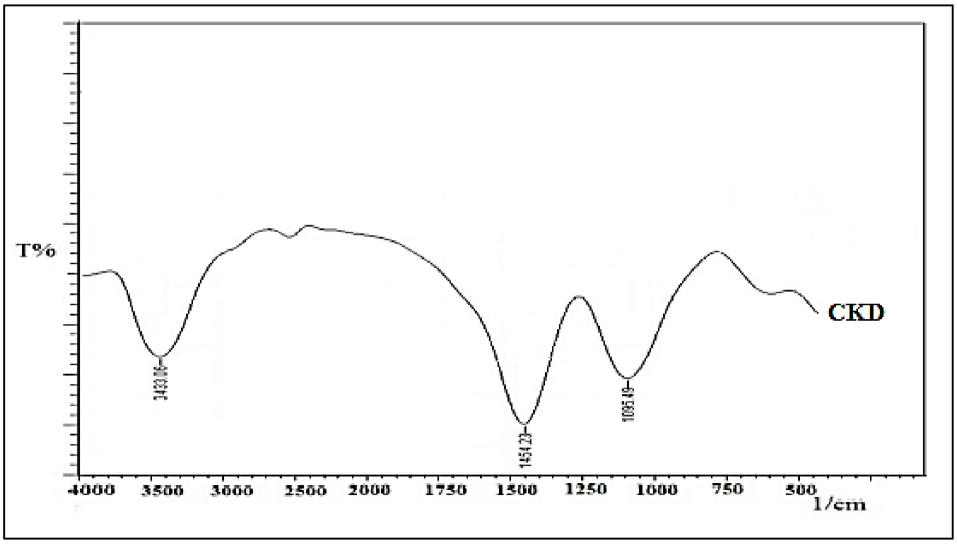

Figure 2.10: FTIR result of CKD (Thomas, 2015)

2.4 CKD as a SCM

SCMs can be separated into two groups based on the nature of their chemical reaction that will occur in cement mortar and concrete, namely hydraulic and pozzolanic. Hydraulic material is a substance that able to react directly with water to form cementitious compound (Neuwald, 2004). On the other hand, pozzolanic material is a substance that able to react chemically with calcium hydroxide ($Ca(OH)_2$) to form cementitious compound. However, CKD has catered both pozzolanic and hydraulic characteristics. It is able to react directly with calcium hydroxide ($Ca(OH)_2$) to form extra hydrates and even react directly with water to produce cement paste which is similar to cement hydration process.

When a pozzolanic SCM with high content of SiO_2or $Al_2O_3$is being introduced by incorporating it into mortar or concrete mix, the volume and types of hydrates formed will be affected. Hence, the strength and durability of such cement mortar or concrete will be influenced too. When SiO_2 and Al_2O_3 in CKD react with Ca^{2+} and OH^- that produced from cement hydration process, types of hydrates that will be produced are Calcium Silicate Hydrate (C-S-H) and Calcium Aluminate Hydrate (C-A-H).

Chemical Equation 2.3 and 2.4 show the chemical formulae of the formation of C-S-H from Alite ($3CaO \cdot SiO_2$ or C_3S) and Belite ($2CaO \cdot SiO_2$ or C_2S) respectively. Alite and Belite are the major sources of C-S-H formation in Ordinary Portland Cement (OPC) hydration process (Neuwald, 2004; Ylmén, 2013). On the other hand, formation of C-S-H and C-A-H formed from pozzolanic SCM are shown in Chemical Equation 2.5 and 2.6 respectively (Edwin & Dunstan, 2011).

<u>Chemical Equation 2.3: Formation of C-S-H from Alite in OPC Hydration</u>

$$2[3CaO \cdot SiO_2] + 11H_2O \rightarrow 3CaO \cdot 2SiO_2 \cdot 8H_2O + 3Ca(OH)_2$$

$$C_3S + \text{water} \rightarrow \text{C-S-H} + \text{CH}$$

<u>Chemical Equation 2.4: Formation of C-S-H from Belite in OPC Hydration</u>

$$2[2CaO \cdot SiO_2] + 9H_2O \rightarrow 3CaO \cdot 2SiO_2 \cdot 8H_2O + Ca(OH)_2$$

$$C_2S + \text{water} \rightarrow \text{C-S-H} + \text{CH}$$

<u>Chemical Equation 2.5: Formation of C-A-H from Pozzolanic CKD</u>

$$3Ca(OH)_2 + 2SiO_2 \rightarrow 3CaO \cdot 2SiO_2 \cdot 3H_2O$$

$$\text{CH} + SiO_2 \rightarrow \text{C-S-H}$$

<u>Chemical Equation 2.6: Formation of C-A-H from Pozzolanic CKD</u>

$$3Ca(OH)_2 + Al_2O_3 + 3H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 6H_2O$$

$$\text{CH} + Al_2O_3 + \text{water} \rightarrow \text{C-A-H}$$

2.4.1 Previous Researches on CKD as a SCM

Since it has been proven that CKD is potentially to be a SCM through its pozzolanic and hydraulic characteristics, there are a plenty of researchers have conducted their studies on the performance of mortar or concrete when CKD is being introduced. Therefore, several aspects on CKD performance as a SCM from pass researchers' studies will be reviewed.

2.4.1.1 Setting Time of CKD as a SCM

In order to evaluate the performance of a mortar or concrete mix, its early-age behaviour such as setting time is an important aspect. In the research of Chaunsali and Peethamparan (2013), the initial and final setting time of two different CKD samples have been studied, which are CKD (I) and CKD (II) that produced from dry-process and wet-process kiln respectively. In their study, both CKD samples have been used as 100% of binder component by weight of 70% in mortar mixture. According to their result obtained, CKD (I) has shorter setting times than CKD (II). This is due to the higher free lime and sulphate content in CKD (I). Moreover, smaller particle size distribution of CKD (I) has provided a greater total surface area for the free lime (CaO_f) to be reacted with water and converted to $Ca(OH)_2$, and hence it increases the creation of extra hydrates. The initial and final setting time of CKD in the study of Chaunsali and Peethamparan (2013) has been tabulated in Table 2.3.

Table 2.3: Setting Times of CKD (Chaunsali & Peethamparan, 2013)

CKD Sample	Initial Setting Time (min)	Final Setting Time (min)
CKD (I)	90	280
CKD (II)	>1440 (24hours)	-

On the other hand, the relationship between the percentage of CKD being used as a SCM and the setting time of the pastes with its water content required for normal consistency are shown in Figure 2.11. In this case, normal consistency can be defined as the consistency of a mortar or concrete mix which will allow the vicat plunger to penetrate to a point of 5 to 7mm from bottom of the vicat mould (Anuar, n.d.).

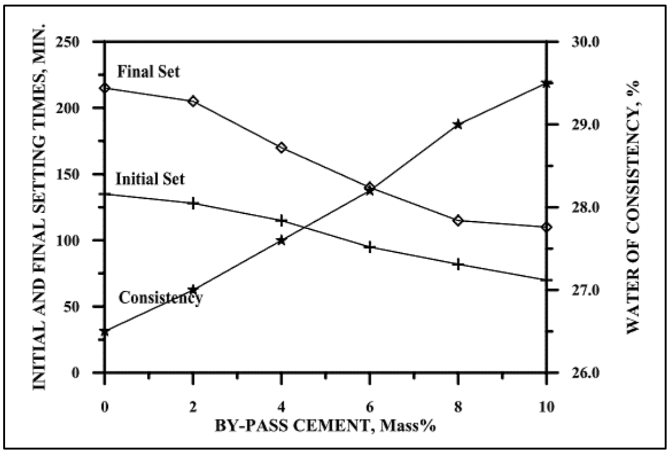

Figure 2.11: Relationship between the Percentage of CKD being used as SCM and the Setting Time of Pastes with its Water Content Required for Normal Consistency(El-Aleem, Abd-El-Aziz, Heikal, & Didamony, 2005)

According to the research of El-Aleem et al. (2005), it has been concluded that the water required for normal consistency is higher when CKD is being introduced. Moreover, the higher percentage of CKD is being used, the higher water demand to achieve normal consistency. In addition, the initial and final setting time are shorter when CKD is being used. When more CKD is being incorporated, the setting times get shorter. Since CKD is rich in alkalis, sulphates, volatile salts and has a great total surface area, water required is increased. However, the lime and alkalis present in CKD will lead to short setting times because these contents will accelerate the hydration process (El-Aleem et al., 2005).

2.4.1.2 Temperature of Hydration of CKD as a SCM

Hydration can be defined as a series of chemical reactions occurs between a cementitious material and water to generate products called hydrates which contribute to a firm mass (Dawood, 2012). During the hydration process, heat will be released to the surrounding as a result of exothermic chemical reaction. Therefore, there will be a change in surrounding temperature. It is a significant characteristic of CKD because the heat released from hydration process may lead to thermal cracking of mortar or concrete. Consequently, the durability of the cement products will be deteriorated (Samor, 2013).

According to the research conducted by Peethamparan et al.(2008), temperature change along the hydration process of CKD pastes are being studied. In their research, temperature of hydration for all four CKD samples (CKD-1, CKD-2, CKD-3 and CKD-4) with similar water content of 31% is being studied. It is being deduced that the free-lime (CaO_f) content in CKD will increase the ambient temperature because conversion of CaO_f to $Ca(OH)_2$ is considered as an exothermic chemical reaction. It is shown in Figure 2.12 where CKD-1 and CKD-2 that contained high amount of CaO_f have experienced a shoot in temperature of about 50 °C and 100 °C respectively. On the other hand, the low content of CaO_f in CKD-3 and CKD-4 did not create any remarkable change in temperature. Figure 2.12 shows the temperature of hydration in CKD-1, CKD-2, CKD-3 and CKD-4.

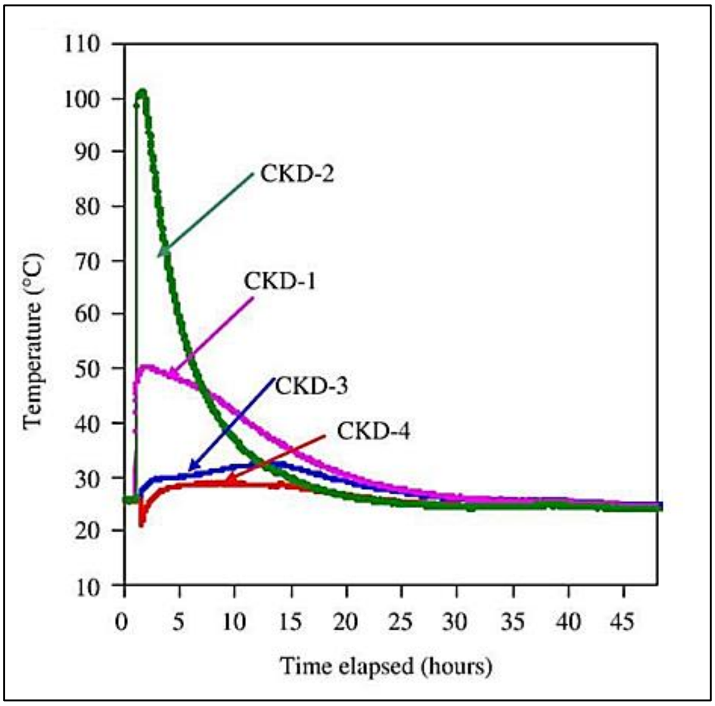

Figure 2.12: Temperature of Hydration in CKD-1, CKD-2, CKD-3 and CKD-4 (Peethamparan et al., 2008)

2.5 Concluding Remark

CKD is a solid waste generated from the cement production plant. However, it is potentially to be reused in various applications. For instance, CKD can act as a stabilizing agent for wastes, help in adjusting the pH value of agricultural soil, act as an anti-shredding agent in hot asphalt, be a SCM in concrete industry, etc. (El-Attar et al., 2016). In current research, the characteristics of CKD are being studied and the early-age performance of CKD as a SCM is examined. Throughout the review of various studies done by pass researchers, selection of CKD samples to be examined in current study have been concluded.

Firstly, the optimum average particle size of CKD to be used as SCM in current study should be as fine as possible. This is because the fine particle size of CKD possesses a greater total surface area for chemical reaction of CaO_f to take place. In the study of Chaunsali and Peethamparan (2013), CKD with average particle size of

4μm with 5% of CaO_f is optimal. Secondly, the CKD to be used as a SCM must be rich in SiO_2 and CaO (or CaO_f). In addition, CKD samples used must be obtained from cement plant with dry-kiln process to ensure its fine particle size and high content of CaO_f. Therefore, in current research, dry-kiln process CKD from collection point, which is near to the rotatory kiln is selected. Thus, comparison of properties are made between the CKD sample with Ordinary Portland Cement with the purpose of examining its potential to be used as SCM.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

Current research is to characterize various properties of CKD and to examine the early-age performance of CKD as a SCM. Thus, CKD sample obtained has gone through a series of laboratory tests that aims to justify physical, morphological and chemical properties of CKD include PSD, SEM, EDX, XRF, XRD, LOI, TGA and FTIR. On the other hand, early-age behaviours of CKD as a SCM were being examined through several laboratory tests, which are SCT and STT. Furthermore, this chapter will discuss in details regarding the materials used and the procedure to carry out such series of laboratory tests.

3.2 Materials

The main component used in this research is cement kiln dust (CKD). In addition, PCC was being mixed with CKD to make blended-cement paste.

3.2.1 Cement Kiln Dust (CKD)

CKD is a solid waste generated from the production plant of cement industry. It is in fine powdery form which contains high percentage of SiO_2 and CaO. Some may consists of small percentage of CaO_f. In current research, it is characterized and used as a SCM by blending it into PCC to produce blended-cement paste.

The CKD sample used in current study is obtained from Hume Cement Sdn Bhd. It is collected from the OPC manufacturing plant which operates by dry-kiln process. The collection point selected for the collection of CKD is a location near to the Gas Conditioning Tower (GCT) which is used to cool hot process gases from the preheating tower to a filter acceptable temperature. Figure 3.1 shows the CKD sample collected from Hume Cement Sdn Bhd.

Figure 3.1: CKD from Hume Cement Sdn Bhd

3.2.2 Portland Composite Cement (PCC)

The PCC comply with MS EN 197-1:2007 CEM II/B-M(S-L) 32.5R is obtained from Hume Cement Sdn Bhd as shown in Figure 3.2 and the properties of PCC provided by the company are tabulated in Table 3.1.

Figure 3.2: PCC from Hume Cement Sdn Bhd

Table 3.1: Properties of OPC

Item		MS EN 197-1:2007 CEM | 32.5N
Initial Setting Time (min)		Not less than 75
Mortar Prism Compressive Strength (N/mm^2)	2 Days	Not less than 10
	28 Days	Not less than 32.5
Soundness (mm)		Not more than 10
Sulphuric Anhydride SO_3 (%)		Not more than 3.5
Chloride (%)		Not more than 0.1

3.3 Experimental Program

An experimental program of current research is designed to illustrate the overall procedures and to avoid making mistakes. Firstly, CKD samples were collected from Hume Cement Sdn Bhd. Next, the samples gone through a series of laboratory test for the purpose of characterization. At the same time, various percentages of CKD were used to incorporate into PCC to make blended-cement pastes. These blended-cement pastes then undergone several tests, which aims to identify its early-age behaviour. All the results obtained are described and analysed in Chapter 4. The flow chart of experimental program is shown in Figure 3.3.

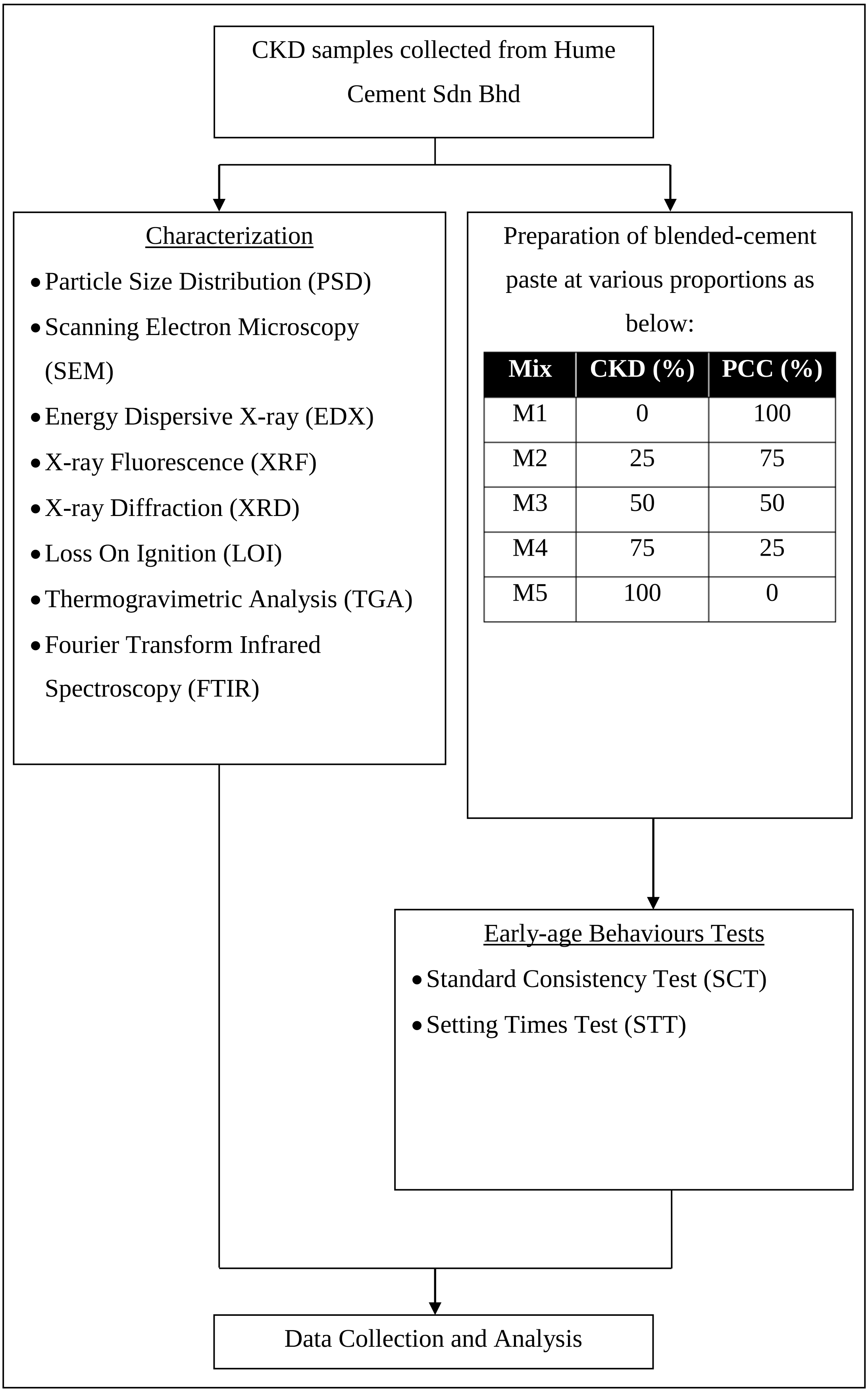

Figure 3.3: Flowchart of Experimental Program

3.4 Characterization of CKD

Once CKD had been collected from Hume Cement Sdn Bhd, they were characterized through a series of laboratory tests, which were PSD, SEM, EDX, XRF, XRD, LOI, TGA and FTIR. In addition, the specimens used in these tests were fresh and pure CKD powders. The objective of conducting these tests is to identify physical, morphological and chemical characteristics of CKD. Table 3.2 summarized the detailed information regarding all the tests that will be carried out for characterization purpose.

Table 3.2: Detailed Information of Characterization Tests on CKD

No.	Test	Testing Scope	Standard
1	PSD	Particle Size Distribution	BS ISO 13320:2009
2	SEM	Surface Morphology	BS ISO 16700:2004
3	EDX	Basic Chemical Elements	BS ISO 15632:2002
4	XRF	Chemical Compounds	BS ISO 29581-2:2010
5	XRD	Mineral Phases (Crystalline or Amorphous)	BS EN 13925-3:2005
6	LOI	Carbon Content and Volatile Substances	BS EN 14346:2006
7	TGA	$CaCO_3$ and $Ca(OH)_2$ Content	BS EN ISO 11358-1:2014
8	FTIR	Bonding Characteristics of Chemical Compounds (Functional Groups)	ASTM E3085 - 17

3.4.1 Particle Size Distribution (PSD)

Particle Size Distribution (PSD) is an analytical analysis to define the particle size distribution of a solid or liquid sample. There are various methods that can be used to obtain particle size of a sample. Some of the methods can be used for a wide range of samples, but some can only be used for specific applications. Thus, it is vital to pick the most appropriate technique for diverse samples as dissimilar approaches may produce different results for the same samples.

In current study, the laser diffraction method was adopted by using Particle Size Analyser with the model of Malvern ZEN 3600 Zetasizer which able to measure a particle size range from 0.6nm to 8.9 µm. It works on the principle that when a beam of light (laser) is scattered by a group of particles, the angle of light scattering is inversely proportional to particle size. Thus, the particle size distribution curve, range and mean of the particle size were determined. Figure 3.4 shows the Particle Size Analyser used in current study.

Figure 3.4: Particle Size Analyser (Malvern ZEN 3600 Zetasizer)

3.4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is an approach to observe a specimen's surface by magnifying such specimen from magnification of approximately 10× up to 300,000×. In addition, it is able to produce high spatial resolution images which ranges from 50nm to 100 nm. When the specimen is being irradiated with a fine electron beam, secondary electrons are radiated away from the surface of such specimen. Therefore, the topography on the surface of the specimen can be obtained by two-dimensional scanning of the electron beam over the surface and acquisition on an image from the detected secondary electron beam (El-Attar et al., 2016; JEOL, 2006).

In such, SEM approach was used to identify the morphological characteristics of CKD sample by collecting data over a selected area of the surface specimen under different magnification, which includes 500 ×, 2 000 ×, 5 000× and 20 000× magnification. Moreover, the scanning electron microscope model, JEOL JSM-6701F was being used for this test as shown in Figure 3.5.

Figure 3.5: Scanning Electron Microscope

3.4.3 Energy Dispersive X-ray (EDX)

Energy Dispersive X-ray (EDX) Spectroscopy is a type of chemical microanalysis practise and it is normally being used in conjunction with SEM. The technology that is being applied in this test is detecting the x-ray emitted from the specimen during bombardment by an electron beam. Thus, the elemental composition of such specimen can be characterized (Materials Evaluation and Engineering Inc., 2014).

In current study, the basic chemical elements present in the CKD specimen were identified through EDX analysis. This is where the energy values of CKD generated by EDX analysis consist of spectra that shown peaks corresponding to the elements which make up the true composition of CKD sample.

As EDX was carried out in conjunction with SEM, the equipment used was similar to SEM as shown in Figure 3.5. In addition, the parameters set for this test were accelerating voltage of 15keV, working distance of 10mm and live time of 100 seconds.

3.4.4 X-ray Fluorescence (XRF)

X-ray Fluorescence is a non-destructive analytical test to determine the chemical composition of a sample. The analyser of XRF measures the secondary x-ray emitted from the sample when it is excited by a primary x-ray source to determine the chemical constituents present in such sample (Thermo Fisher Scientific, n.d.).

In this study, chemical compositions of CKD were studied as each of the constituent produces a set of distinctive fluorescent x-ray which called as “fingerprint”. As a result, both qualitative and quantitative analysis were produced. Hence, in current research, quantitative results whereby the percentage value of each chemical composition exists in the specimen was analysed.

3.4.5 X-ray Diffraction (XRD)

X-ray Diffraction (XRD) can be defined as a rapid analytical technique mainly used to identify the crystalline phase of a sample and provide information on unit cell dimensions. XRD analysis is based on constructive interference of monochromatic x-rays and a crystalline sample. When the monochromatic incident x-rays are directed toward the sample, the interaction between such incident rays and the sample will generate constructive interference and a diffracted ray when it fulfils Bragg's law. The principle of Bragg's law is summarized in Equation (3.1) (Dutrow & Clark, 2017).

$$\boldsymbol{n\lambda = 2d\,sin\,\theta} \tag{3.1}$$

where
λ = wavelength of the X-ray, m
d = spacing between layers of atoms, m
θ = angle between incident rays and surface of the crystal, $^\circ$
n = an integer

In such, the x-ray diffractometer used in this test was SHIMADZU XRD-6100 as shown in Figure 3.6. It works on the principal whereby the x-rays directed on the CKD sample and its diffracted rays were collected. After the unique "fingerprint" of the crystals present in CKD sample were interpreted appropriately, the crystalline form of CKD were identified by comparing its "fingerprint" with standard reference patterns and measurements.

Figure 3.6: X-Ray Diffractometer

3.4.6 Loss On Ignition (LOI)

Loss on ignition (LOI) is an approach of determining the weight loss of a sample after it has been heated to a specific high temperature. The weight loss from the combustion process indicates the volatile compounds contained in the sample.

When characterizing CKD, LOI test was performed to determine the approximate amount of carbon content and other volatile substances such as alkalis, chlorides and sulphates within the CKD sample. Firstly, the dried CKD sample in crucible was placed in the furnace and heated to 950°C for 2 hours. After that, the CKD sample was cooled down completely and the weight of residual was weighted. Lastly, the LOI percentage of CKD sample was then calculated by comparing the weight of the sample before and after it has been ignited by using the formula shown in Equation (3.2) (Carbolite Gero Ltd., n.d.; UCL Department of Geography, n.d.).

$$LOI = \frac{W_0 - W_1}{W_0} \times 100 \quad (3.2)$$

where

LOI = percentage of loss on ignition, %

W_0 = initial weight of CKD sample, g

W_1 = final weight of CKD sample, g

3.4.7 Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) is an analysis to measure the weight change of a sample as a function temperature, time or vacuum. Generally, the function of temperature ranges from 25°C to 1500°C. This analysis is used to determine the composition of sample and to estimate its thermal stability. It is able to characterize sample that exhibits weight loss or gain due to attraction or emission of volatiles, decomposition, oxidation and reduction (Mohomed, 2016).

In this research, TGA was conducted by using Mettler Toledo 851e Thermo Gravimetric Analyser as shown in Figure 3.7. It was used to examine the weight change experienced by CKD from room temperature to 1000°C with heating rate of 10°C/min. The weight loss of CKD when undergone the heating process indicated the amount of CaO that was produced from calcination of $CaCO_3$ and $Ca(OH)_2$. The chemical formulae of such processes are stated in Chemical Equation 2.1 and 2.2.

Figure 3.7: Thermo Gravimetric Analyser

3.4.8 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a test that is able to provide quantitative and qualitative analysis for organic and inorganic samples. It examines the chemical bonds exist in a molecule by measuring the absorption of infrared radiation by the sample material versus wavelength. The infrared absorption bands help in identifying molecular components and structures because every different molecular fingerprint is distinctive (Intertek Group plc, n.d.).

Thus, FTIR was conducted on CKD sample by using Perkin Elmer System 2000 FT-IR Spectrometer RX 1 as shown in Figure 3.8. It was used to detect functional groups present in the CKD particles. As a result, plots of intensity versus wavenumber were shown clearly by observing the peak positions in the plots. The intensity in the graph defines the light transmission and absorption at each wavenumber whereas the wavenumber indicates the reciprocal of the wavelength.

Figure 3.8: Fourier Transform Infrared Spectrometer

3.5 Early-age Behaviours of Cement Kiln Dust (CKD)

Other than characterization, performance of CKD sample collected from Hume Cement Sdn Bhd as a SCM were examined. Several tests were carried out to examine the early-age behaviours such as SCT and STT. Before carrying out the tests, blended-cement pastes were prepared by mixing CKD powder and PCC at various proportion. Table 3.3 shows the 5 mixes (M1 to M5) with different percentage of CKD to be prepared. M1 is a controlled mix of 100% PCC. M2 to M5 show that CKD were used from 25% to 100% in the preparation of blended-cement paste.

Table 3.3: Percentage of CKD and PCC in Preparation of Blended-cement Pastes

Mix	CKD (%)	PCC (%)
M1	0	100
M2	25	75
M3	50	50
M4	75	25
M5	100	0

Hence, the specimens used in these tests were the above mixes. Table 3.4 summarized the detailed information regarding all the tests that were carried out for the determination of CKD's early-age behaviours.

Table 3.4: Detailed Information of Early-age Behaviour Tests on CKD

No.	Test	Testing Scope	Standard
1	SCT	Water Required for Normal Consistency	BS 4550-3-3.5:1978
2	STT	Initial and Final Setting time	BS EN 196-3:1995

3.5.1 Standard Consistency Test (SCT) and Setting Time Tests (STT)

In order to identify the standard consistency and setting times of the cement paste when CKD was being incorporated into the mix, SCT and STT were conducted. Standard consistency test (SCT) is also known as normal consistency test which is carried out using an experimental equipment, namely vicat apparatus. The vicat apparatus consists of a frame having a movable rod with a cap at one end and at the other end is connected to interchangeable attachment which may be a needle or a plunger. The use of needle in vicat apparatus is to identify the initial and final setting time whereas plunger is used to determine the normal consistency of the mortar of concrete mix. In addition, normal consistency can be defined as the consistency of a mortar or concrete mix which enables the plunger of a vicat apparatus to penetrate to a depth of 5 to 7mm from bottom of the vicat mould (Anuar, n.d.). Figure 3.9 shows the vicat apparatus used in current test.

Figure 3.9: Vicat Apparatus

In order to determine the normal consistency and setting times of CKD blended-cement paste, both needle and plunger were used in the vicat apparatus. When vicat plunger was used, the percentage of water required by the blended-cement to achieve standard consistency can be calculated through the use of formula shown in Equation (3.3).

$$\boldsymbol{P = \frac{W}{C} \times 100} \tag{3.3}$$

where

P = percentage of water in weight, %

W = weight of water added, g

C = weight of blended-cement added, g

When vicat needle was being used in vicat apparatus, initial and final setting time of CKD blended-cement pastes can be obtained. Firstly, the blended cement pastes were moulded and placed in a moist cabinet to allow for initial setting. Penetration tests were conducted periodically on the pastes by allowing 1mm Vicat needle to settle into these pastes. Next, the initial setting time of such pastes were calculated as the time elapsed between the initial contact of cement and water and the time when the penetration is at 25 mm. On the other hand, the final setting time were also calculated as the time when the needle does not sink visibly into the paste (American Association of State Highway and Transportation Officials, 2008).

CHAPTER 4

DATA AND ANALYSIS

4.1 Introduction

Results obtained from all the characterization tests and early-age behaviors analyses of CKD were discussed in current chapter. Characterization tests consist physical, morphological and chemical characteristics of CKD, whereas analyses on early-age behaviors consist of the investigation on standard consistency and setting times of CKD. Comprehensive analyses for all the tests described in this chapter were based on every laboratory test which have been carried out.

4.2 Characterization of CKD

To achieve the first aim of the study, CKD sample was characterised through a list of analyses, which were PSD, SEM, EDX, XRF, XRD, LOI, TGA and FTIR. Subsequently, the physical, morphological and chemical characteristics of CKD were analysed and reported in following subsections.

4.2.1 Particle Size Distribution (PSD) Analysis

In current study, the particle size of CKD is an essential aspect to be investigated in order to understand its behaviour when it is being cooperated into concrete mix. Thus, Particle Size Distribution Analysis for CKD sample was conductd and the result is shown in Figure 4.1.

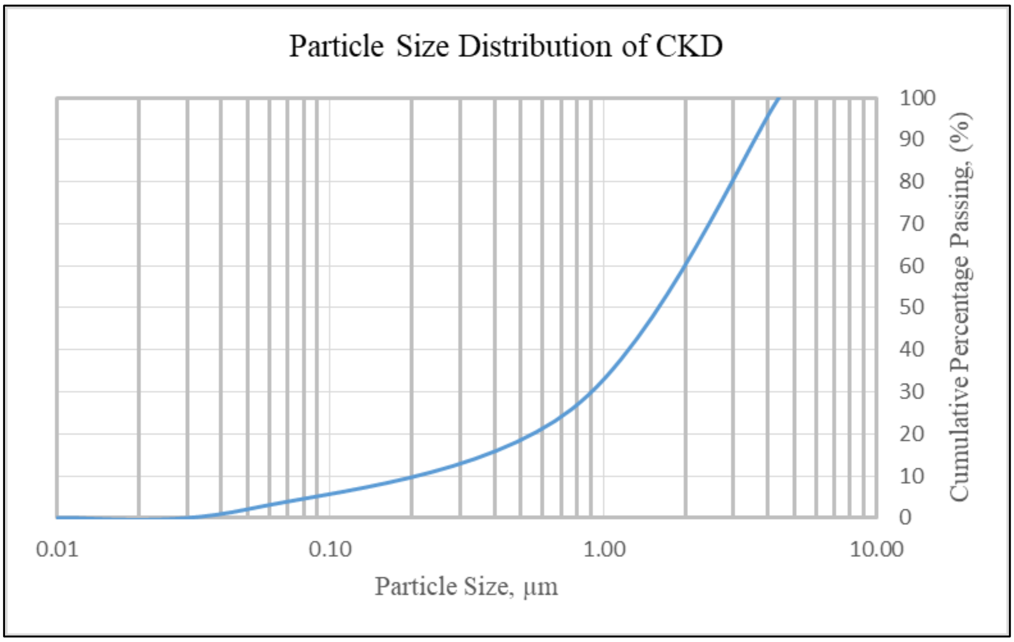

Figure 4.1: Particle Size Distribution of CKD Sample

According to Figure 4.1, it illustrates that the particle size of CKD is very fine, which ranges from 0.03 µm to 7.60 µm, while the mean of the particle size is approximately 0.90 µm. Moreover, 23.9% of the CKD particles are smaller than the size of 0.77 µm and 73.9% are smaller than 4.39 µm. As compared to previous reserachers studies, the result obtained in current analysis shows that the particle size of CKD sample is relatively fine. However, this result is significantly comparable with the study of El-Attar et al. (2016), in which their CKD sample has an average size of 2.45 µm.

On the hand, such analysis has proven that the CKD sample possess a finer mean particle size compared to typical OPC. Rougly 95% of OPC particles are finer than 45 µm whereas the mean particle size is about 15 µm (Khanna, 2009). The deviation between the average particle size of CKD sample and OPC may give rise to the differences in rate of hydration and rate of strength gain in a concrete. According to Zayed, Sedaghat, Bien-Aime, and Shanahan (2013), they have concluded that the smaller the mean particle size of cement particles, the higher the heat of hydration. This statement is established as finer cementitous particles are able to provide a greater total surface area for the hydration reaction to take place, provided that the mineralogical characteristics of the cementitous material remained constant. Subsequently, it contributes to more hydrates formation and the increase in mechanical strength of the concrete.

4.2.2 Scanning Electron Microscopy (SEM) Analysis

The morphological characteristic such as the surface texture and shape of CKD was studied through conducting SEM analysis. Figure 4.2 shows the SEM result under ×500 maginification, whereas Figure 4.3 shows the SEM result under ×2 000 magnification followed by Figure 4.4 and 4.5 which show the results under ×5 000 and ×20 000 magnification respectively.

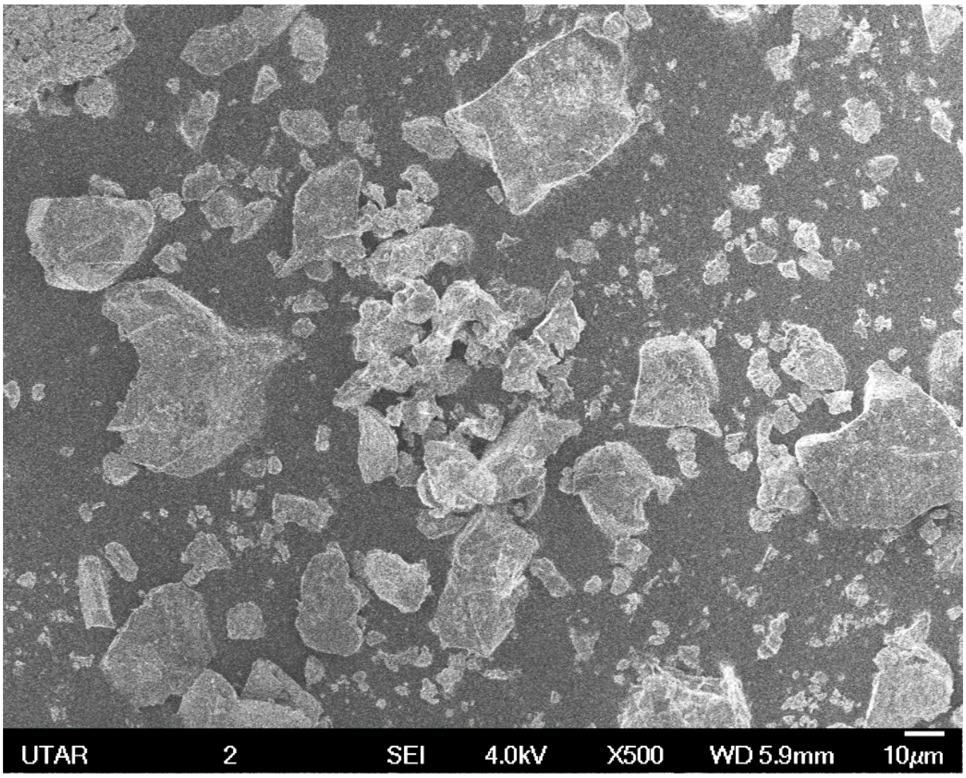

Figure 4.2: SEM Result of CKD under ×500 magnification

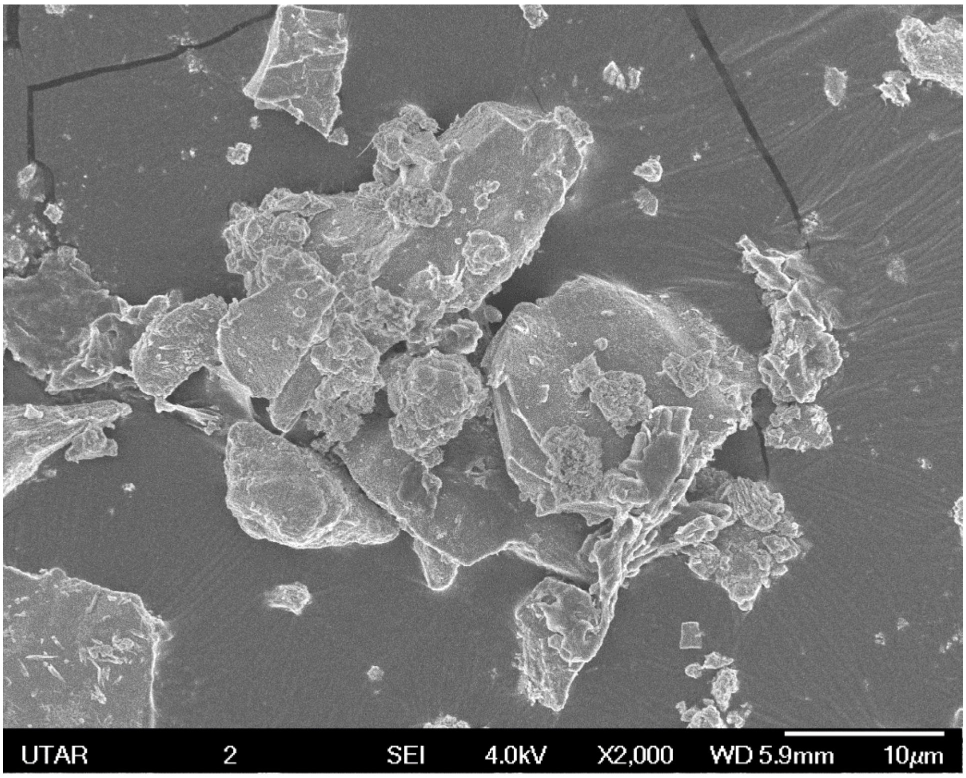

Figure 4.3: SEM Result of CKD under ×2 000 magnification

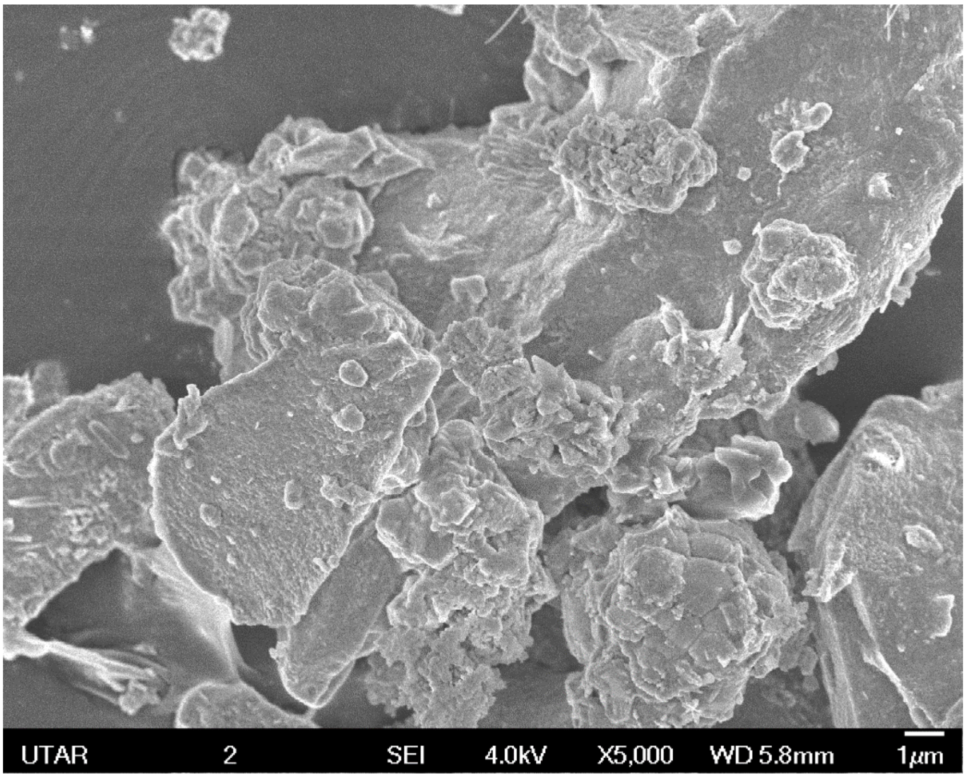

Figure 4.4: SEM Result of CKD under ×5 000 magnification

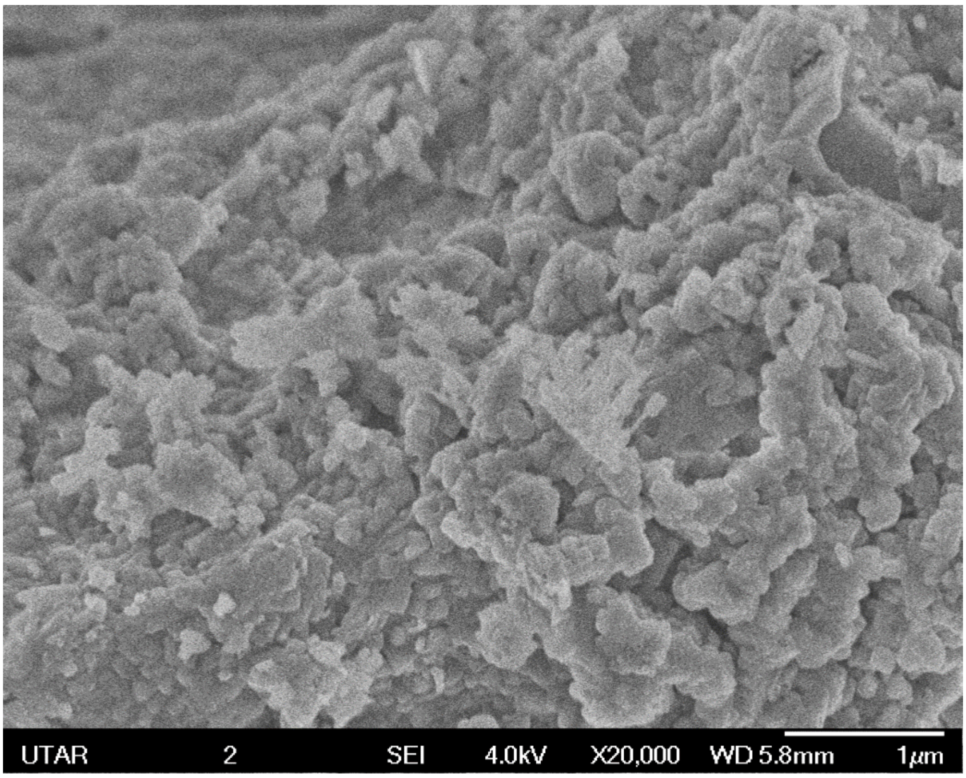

Figure 4.5: SEM Result of CKD under ×20 000 magnification

According to the results obtained, it shows that CKD sample in current research consists of undispersed cemented congregate with sharp corners, rough surface and irregular shapes. These results are significantly similar with previous researchers' studies which includes the study carried out by Yoobanpot et al. (2017). According to Lanzerstorfer et al. (2016), the nonspherical and sharp shape of most of the particles in CKD is due to their origin from the comminution of the raw material.

Morover, the roughness and sharpness of the CKD structure is similar to the OPC which increases its potential to become a SCM. This statement is being supported by the research of Yoobanpot et al. (2017), whereby the SEM results of OPC is shown in Figure 4.6. Figure 4.6 shows that the texture of OPC is very comparable with CKD as it consists of irregular shape of particles and having rough surfaces.

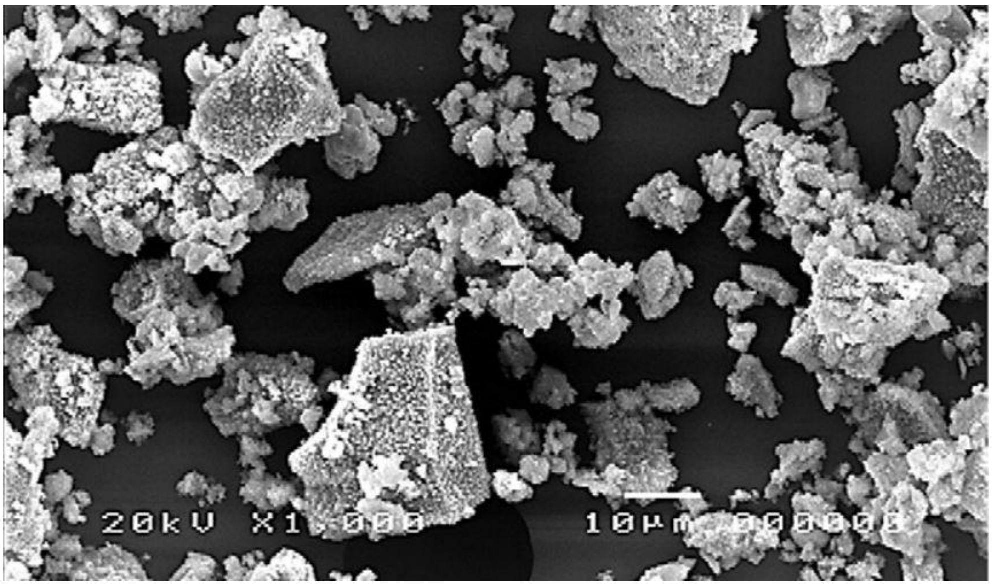

Figure 4.6: SEM Result of OPC (Yoobanpot et al., 2017)

On the other hand, some of the SEM results of CKD obtained by previous researchers such as Peethamparan, Olek, & Diamond (2009) consists of smooth and spherical particles which does not exist in the SEM results of current research. Such spherical particles are identified as fly ash particles. Figure 4.7 shows the SEM results of CKD obtained by Peethamparan et al. (2009).

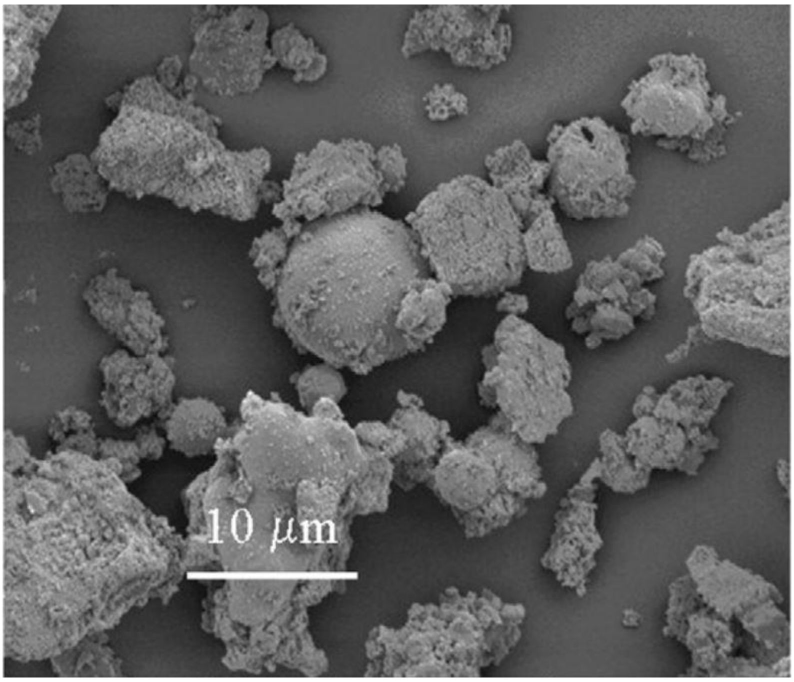

Figure 4.7: SEM Result of CKD (Peethamparan et al., 2009)

In the study of Colangelo and Cioffi (2013), they have concluded that rounded shape and surface smoothness of particles in a concrete mix will increase the workability, whereas irregular shape and coarse surfaces of particles will reduce the workability and thus increase the water demand for such concrete mix. Therefore, it can be concluded that CKD sample in current research having sharp edges and rough texture will contribute to high water demand when it is used as a SCM. Meanwhile, the CKD sample which contains spherical fly ash particles from previous researchers are more likely to have lower water demand and higher workablity when it is being incorporated into a concrete mix.

4.2.3 Energy Dispersive X-ray (EDX) Analysis

As EDX analysis was conducted in conjuction with SEM analysis, the basic elements exist in CKD sample were identified. In current research, EDX spectra were secured at three different locations in order to identify the average weightage of chemical elements contained in the CKD sample. Table 4.1 shows the weightage (%) of various elements present in three spectra locations on CKD and the average weightage (%) .

Table 4.1: Basic Elements in CKD

Element	Weightage (%)			
	Spectrum 1	Spectrum 2	Spectrum 3	Average
C	33.39	27.29	21.27	27.23
O	49.07	52.44	51.04	50.85
Mg	0.39	0.23	0.59	0.40
Al	1.09	2.78	1.85	1.91
Si	3.06	2.01	3.94	3.00
K	0.12	0.52	0.33	0.32
Ca	12.00	14.27	19.30	15.19
Fe	0.69	0.45	1.34	0.83
Cu	0.19	-	0.35	0.27
Total	100	100	100	100

According to the result obtained in Table 4.1, it shows that other than carbon and oxygen, most abunden elements in CKD are Ca, Si and followed by Al. These three elements are significant to make CKD as a potential SCM as the high weightage of Ca, Si and Al have been contribute to high concentration of CaO, SiO_2 and Al_2O_3 respectively. According to Jadhav and Debnath (2011), CaO, SiO_2 and Al_2O_3 are the chemical compositions that made up the main components of OPC, namely Alite ($3CaO{\cdot}SiO_2$ or C_3S), Belite ($2CaO{\cdot}SiO_2$ or C_2S) and Calcium Aluminate ($Al_2O_3{\cdot}CaO$ or C_3A). Thus, CKD sample in current research possesses a high potential to be used as a SCM due to its similarity of chemical compositions with OPC.

4.2.4 X-ray Fluorescence (XRF) Analysis

After the basic elements of CKD were determined, the chemical composition in CKD such as mineral oxides were identified through XRF analysis. Table 4.2 shows the constituents exist in CKD sample and its weightage (%).

Table 4.2: Chemical Compositions in CKD

Constituents	Weightage (%)
SiO_2	8.09
Al_2O_3	4.97
Fe_2O_3	2.26
CaO	45.01
MgO	0.72
SO_3	0.35
K_2O	0.52
Na_2O	0.02
LOI	38.03
Total	100
CaO_f	0.80

According to the XRF results shown in Table 4.2, it indicates that the major constituents present in CKD sample are CaO (45.01%), followed by SiO_2 (8.09%) and Al_2O_3 (4.97%) etc. Such results correspond to the EDX analysis which carried out prior to this current analysis as the most abundent minerals exist in CKD is Ca followed by Si and Al. Moreover, these results, in which CaO is the major composition present in CKD followed by SiO_2 and Al_2O_3 are also similar to the studies of previous researchers, which includes the study of Chaunsali and Peethamparan (2013), El-Attar et al. (2016), Marku et al. (2012) and Yoobanpot et al. (2017).

In order to justify the potential of CKD to be used as a SCM, its chemical constituents are being compared to the constituents in OPC. Table 4.3 shows the chemical composition of OPC from the research done by Sharma and Pandey (1999).

Table 4.3 : Chemical Compositions in OPC (Sharma & Pandey, 1999)

Constituents	Weightage (%)
SiO_2	21.14
Al_2O_3	4.80
Fe_2O_3	3.92
CaO	61.20
MgO	2.67
SO_3	2.08
K_2O	0.80
Na_2O	0.20
P_2O_5	0.32
TiO_2	0.10
Mn_2O_3	0.07
LOI	2.67
Total	99.97
CaO_f	0.50

From Table 4.3, it can be explained whereby the compositions of CKD mainly comprise of CaO, SiO_2, Al_2O_3 and Fe_2O_3 are similar to those found in OPC. However, the CaO and SiO_2 contents in CKD tend to be lower than in the OPC. In this point, it can be concluded that CKD is potentially to be a pozzolanic SCM as it has high contents of SiO_2 and Al_2O_3 despite the fact that SiO_2 content is slightly lower than in the OPC. Moreover, the highest percentage content of CaO present in CKD increases its ability to produce sufficient hydrates and $Ca(OH)_2$, which contributes to its usage as a SCM.

Nevertheless, the reactive stage of the CaO is a greater concern as the reactive CaO can be readily reacted with water to form $Ca(OH)_2$ and hence the pozzolanic reaction can be carried out. Thus, the presence of free lime content (CaO_f) in CKD is another concern. According to Table 4.2 and 4.3, it shows that the free lime content in

CKD is relatively low compared to OPC. According to Rahman et al. (2011), CKDs with high LOI and low alkali give rise to relatively lower strength and greater plasticity indices as high LOI value shows that the CKD is high in slow-reacting calcium carbonate and low in reactive free lime (CaO_f).

4.2.5 X-ray Diffraction (XRD) Analysis

Other than the chemical compositions exist in a CKD sample, the mineral phases present in the sample are also essential to be studied. In order to study the crystalline structures exist in CKD, EDX analysis was performed. Figure 4.8 shows the EDX result obtained from CKD in current research.

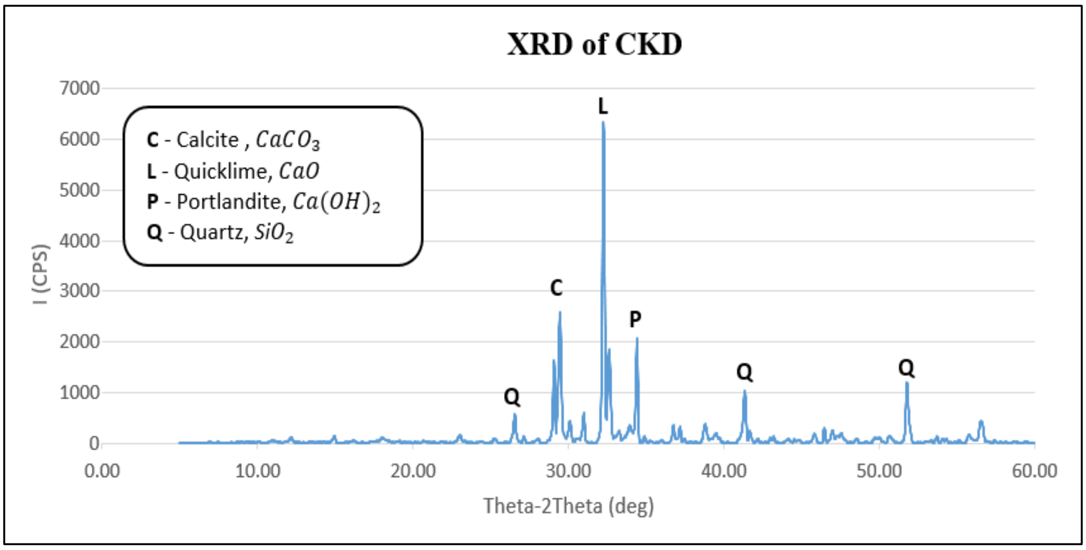

Figure 4.8: XRD Result of CKD

Figure 4.8 shows that quicklime (CaO) and calcite ($CaCO_3$) are the major crystalline phases occurr in CKD, whereas portlandite/calcium hydroxide ($Ca(OH)_2$) and quartz (SiO_2) etc. are the minor phases. This result is similar to many other studies carried out by previous researchers which includes Gupta et al. (2015). On the other hand, there are researchers such as El-Attar et al. (2016), Wang, Mishulovich, & Shah

(2007) proved that there are some other minor phases exist in their CKD samples such as sylvite (KCl), halite (NaCl) and anhydrite ($CaSO_4$).

Furthermore, comparison has been made between the XRD result of CKD obtained in current study and the XRD result of OPC. XRD of OPC to be referred for such comparison is the research conducted by Dhapekar, Majumdar and Gupta (2015). In their research, there are four major crystalline compounds present in OPC, which includes Belite (C_2S), Alite (C_3S), Brownmillerite (C_3A and C_4AF) and Gypsum, with the weightage of 42.5%, 39.30%, 12.50% and 5.70% respectively. Figure 4.9 shows the XRD of OPC obtained by Dhapekar et al. (2015).

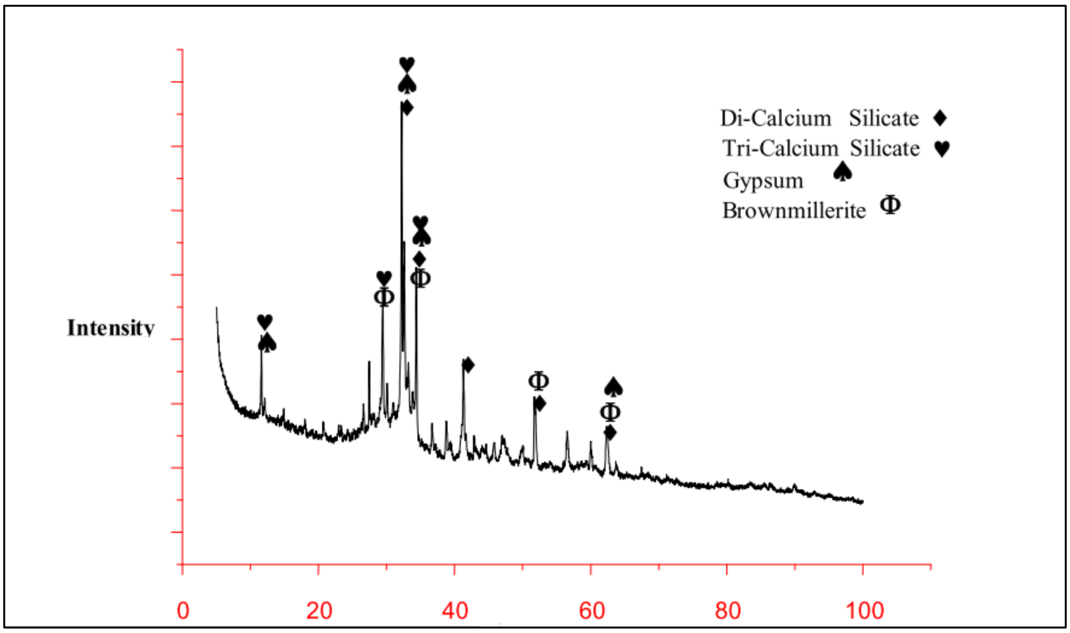

Figure 4.9 : XRD Result of OPC (Dhapekar et al., 2015)

As there are several peaks exist in CKD appeared to be similar with OPC, it is believed that there are existance of cementitous compounds in CKD such as C_2S, C_3S and Gypsum. This can be explained as there are significant peaks at the range of $25° \leq 2\theta \leq 35°$ for both CKD and OPC. Thus, it is feasible to be incorporated into concrete mix as a SCM.

4.2.6 Loss On Ignition (LOI) Analysis

In current research, LOI analysis was conducted with the purpose of determining the loss of mass in CKD sample when it is being heated to approximately 950 °C. The result obtained in this analysis, which is the percentage of loss on ignition is 38.03% as stated in Table 4.2. Such value is considered as a great value as it is much more higher than in OPC, which is approximately 1.00 to 3.00% (Peethamparan et al., 2009; Sharma & Pandey, 1999).

According to the research done by Sreekrishnavilasam and Santagata (2005), the loss of weight in LOI of CKD is the weight of chemically bound water, carbon dioxide (CO_2), non-carbonated carbon and some other minor volatile substances. Nevertheless, their research states that the main loss of weight in LOI test is contributed by the non-carbonated carbon which is the unburned clinker, namely calcium carbonate ($CaCO_3$) that experiences de-carbonation when ignited. When the calcium carbonate ($CaCO_3$) is ignited, it undergoes a process called thermal decomposition, in which CaO and CO_2 will be released.

According to Sreekrishnavilasam and Santagata (2005), the LOI value is the indication of the calcium carbonate ($CaCO_3$) and free lime content (CaO_f). When the LOI value is high, it means that there are high amount of calcium carbonate ($CaCO_3$) and low amount of free lime content (CaO_f) exists in the CKD. In fact, low LOI value with high CaO_f content contributes to its strength properties.

In such, CKD appears to be having low strength properties as it has a high value of LOI percentage (38.03%) with low value of CaO_f content (0.08%). In order to determine the exact amount of calcium carbonate ($CaCO_3$) compounds exist in CKD, TGA analysis was carreid out and will be dicussed in the following.

4.2.7 Thermogravemetric Analysis (TGA)

In current study, TGA is conducted to define the degree of calcination of calcium hydroxide ($Ca(OH)_2$) and calcium carbonate ($CaCO_3$). Figure 4.10 shows the thermogram of CKD sample which has undergone a temperature range from room temperature to 1000 °C, with the heating rate of 10 °C/min.

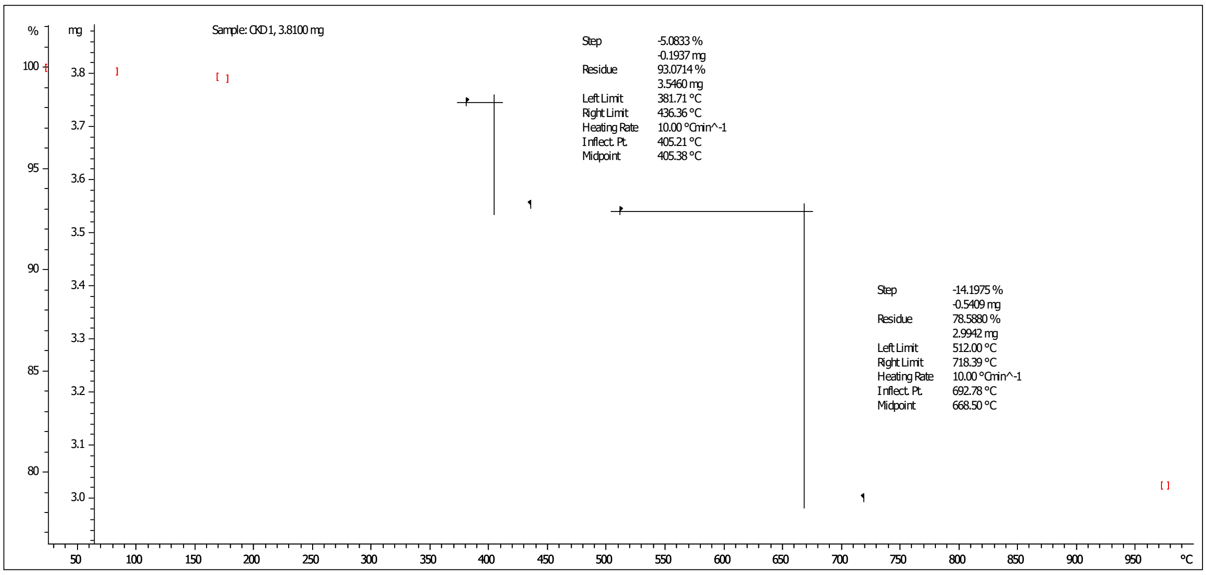

Figure 4.10: TGA Result of CKD

According to Figure 4.10, it shows that there are two remarkable change in the weight of CKD sample. The first significant drop of weight of the sample is between the temperature of approximately 380 °C to 450 °C and the second reduction of weight is between the temperature of 500 °C to 750 °C. It can be explained whereby the first and second reductions of weight in the CKD sample is the thermal decomposition of calcium hydroxide ($Ca(OH)_2$) and calcium carbonate ($CaCO_3$) respectively. This statement is supported by the study carried out by Khanna (2009) as one of their CKD samples possess a thermogram which is highly similar with the result obtained in current study, which is shown in Figure 4.11 .

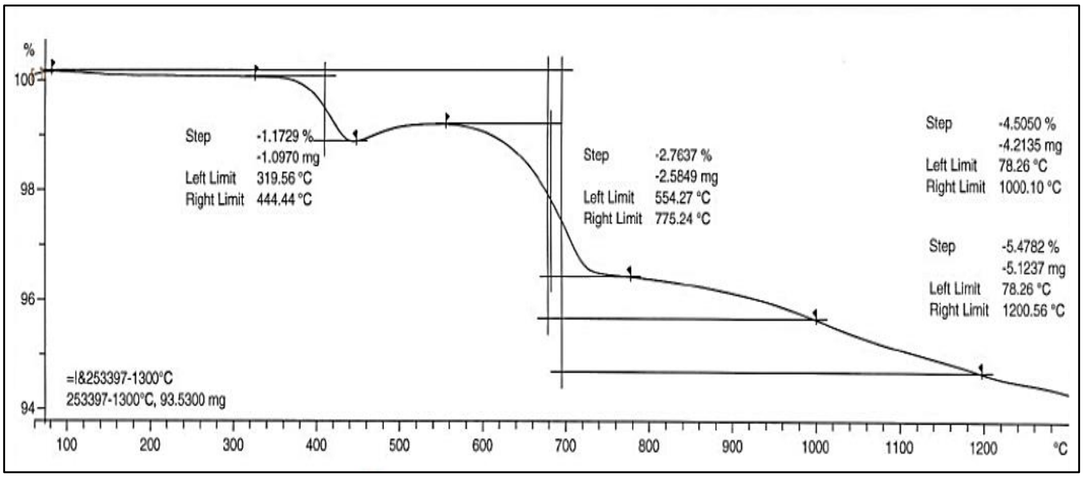

Figure 4.11: TGA Result of CKD (Khanna, 2009)

From the thermogram illustrated in Figure 4.10, the amount of calcium hydroxide ($Ca(OH)_2$) and calcium carbonate ($CaCO_3$) were approximately determined and tabulated in Table 4.5.

Table 4.4: Chemical Composition in LOI of CKD

Components	Weightage (%)
$Ca(OH)_2$	5.08
$CaCO_3$	14.20
Other volatile substances	18.75
Total LOI	38.03

According to Table 4.5, the low content of calcium hydroxide ($Ca(OH)_2$) present in CKD sample is due to the exosure to mositure. This is because the collection point of the CKD is located near to the water spray which is used to control temperature in the Gass Conditioning Tower (GCT) of cement manufacturing plant, which lead to small amount of CaO_f being hydrated.

Although the CKD is feasible to be used as SCM, it may possess low compressive strength as it contains high value of inert $CaCO_3$, which behaves unreactively in hydration process.

4.2.8 Fourier Transform Infrared Spectroscopy (FTIR)

In current research, bonding characteristics of chemical compounds in CKD was investigated through FTIR analysis. Figure 4.12 shows the spectra obtained by CKD in FTIR analysis.

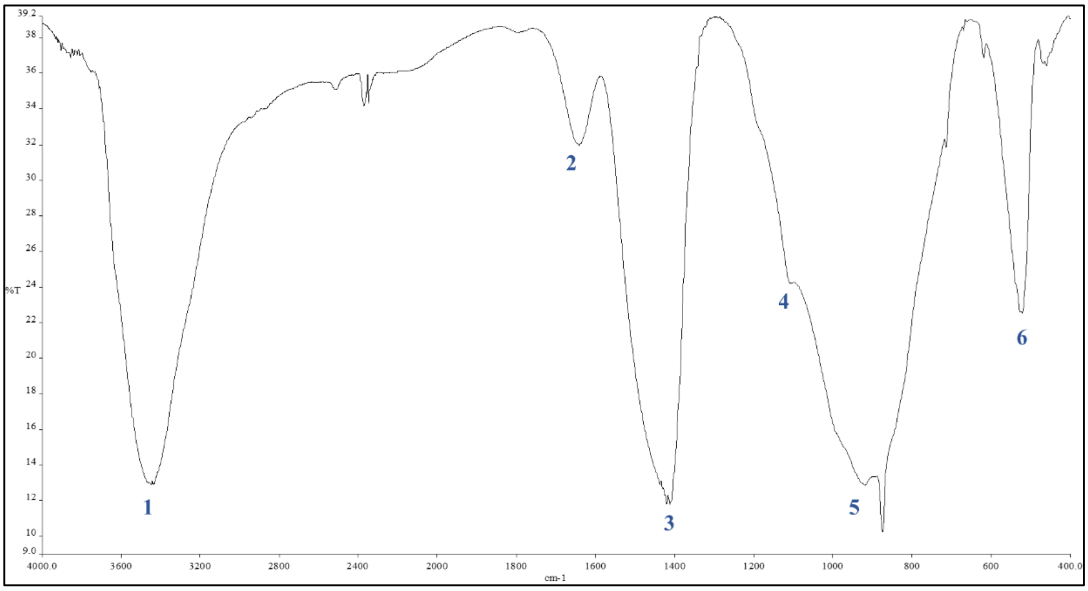

Figure 4.12: FTIR Result of CKD

According to Figure 4.12, six regions of significant IR absorption occurred in CKD. The functional groups and their characteristics of bonding vibrations in these regions are listed in Table 4.6.

Table 4.5: Functional Groups in CKD

Region	Functional Group
1	Stretching vibration of O-H bond
2	Bending vibrations of H-O-H
3	Stretching vibrations of CO_3^{2-}
4	Stretching vibrations of Si-O-Si
5	Out of plan vibrations of of CO_3^{2-}
6	Stretching vibration Si-O-Si and Al-O-Si

According to Figure 4.12 and Table 4.6, the result obained in FTIR analysis is relatively comparable to the study conducted by Elrefaey (2017) as shown in Figure 2.10. The three significant functional groups and compounds exist in the CKD sample of Elrefaey (2017) such as hydroxyl (-OH), carbonate (CO_3^{2-}) and silicate compound (Si-O-Si) are also exist in current research CKD sample. However, there is a presence of a compound was not mentioned in the research of Elrefaey (2017), which is the IR absorption occurred in region 2- Bending vibrations of H-O-H. The presence of these vibrations are attributed by the presence of bound water in the sample. This can be explained where the bending vibrations of water molecule exist at approximately 1596 cm^{-1}, which is located at region 2 of the IR spectra in current research (Thomas, 2015).

In addition, the existance of the three functional groups and chemical ions, such as hydroxyl (-OH), carbonate (CO_3^{2-}) and silicate compound (Si-O-Si) are correspond with the results obtained in XRF and TGA. This can be explained where these functional groups contribute to $Ca(OH)_2$, $CaCO_3$ and SiO_2 compounds.

4.3 Early-age Behaviours of CKD

To fulfill the second aim of current research, CKD was incorporated into cement paste at various proportions (as in Table 3.3) for the purpose of investigating its early-age behaviours. Such behaviours include standard consistency and setting times of the blended-cement pastes were analysed and reported in following subsections.

4.3.1 Standard Consistency Test (SCT) and Setting Time (STT) Analyses

In the early-age behaviours of CKD, SCT and STT analyses are inevitable to be carried out as it helps in determining the percentage of water required by the blended cement mix and the setting times of such mix. In current research, five porportions of cement mixes were prepared as stated in Table 3.3. Figure 4.13 indicates the standard consistency (%), initial and final setting times of the blended cement pastes with different percentage of CKD replacement in the pastes.

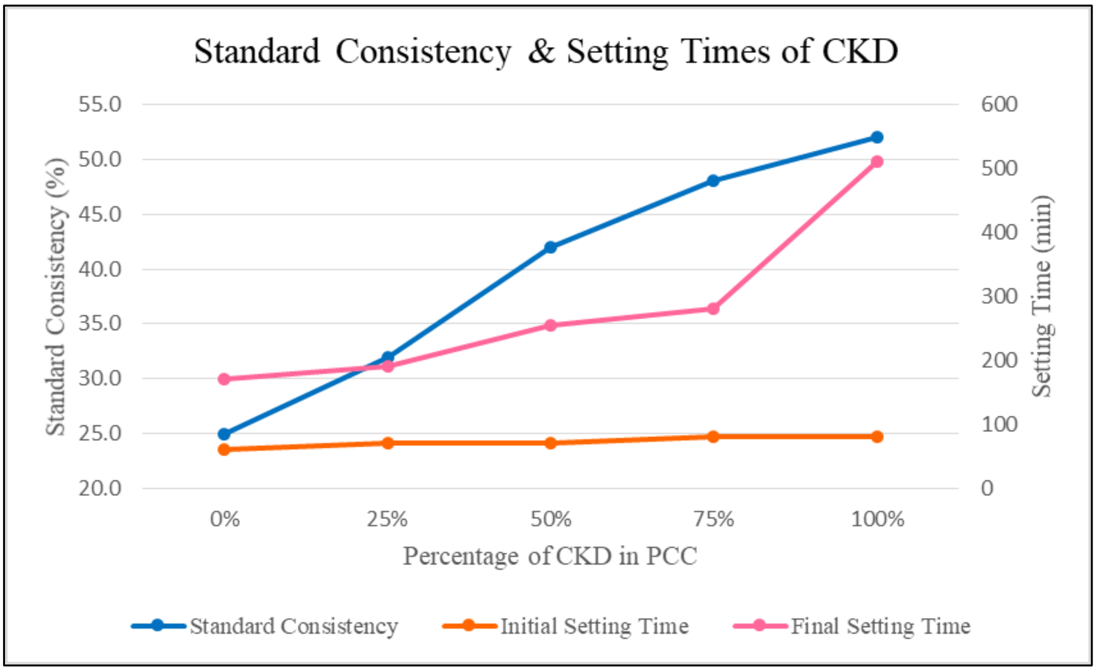

Figure 4.13: SCT and STT Results of CKD Blended-Cement Pastes

In order to have a clear comparison between the standard consistency of PCC and CKD, results obtained in 0% and 100% of CKD replacement in Figure 4.13 are compared. In M1 (0% of CKD replacement), the percentage of standard consistency obtained is approximately 25.0%, whereas in M5 (100% of CKD replacement), such percentage is about 52.0%. According to Figure 4.13, it shows that the standard consistency of the CKD-blended-cement pastes increases when the percentage of CKD used in the pastes increases, which is supported by the research of El-Aleem, Abd-El-Aziz, Heikal, & Didamony (2005). This statement can be explained in terms of average particle size in the cement mix. As discussed in current chapter, the average particle size of CKD is relatively smaller than the cement particles. Thus, when the replacement percentage of CKD increases, the mean particle size of the mix reduces, which contributes to greater total surface areas of particles. Therefore, more quantity of water is required to wet the surface areas of particles to complete the hydration process.

On the other hand, setting times of the CKD-blended-cement pastes are increasing with the increase of CKD replacement percentage. Figure 4.13 shows no significant increase in the initial setting time of the pastes, which ranges from 60 to 80 minutes. However, there is a great surge in the final setting time when the cement pastes increase the percentage of CKD proportions, which increases from 170 to 510 minutes. According to BS 12:1996, the initial setting time of OPC and PCC shall not less than 60 minutes and final setting time shall not more than 300 minutes. Thus, M1 to M4 (0% to 75% CKD replacement) have fulfilled such requirement.

However, the trend of the setting times of CKD mixes in current research opposes the study of El-Aleem et al. (2005). In their study, addition of CKD increases both the standard consistency and setting times (as shown in Figure 2.11). In addition, they have justified this results by stating that the high amounts of alkali, sulphates, volatile salts and CaO_f content accelerate the setting process. In fact, CKD sample used in current research possesses extremely low alkali contents, low content of CaO_f (lower than cement) and high content of inert $CaCO_3$. Thus, it is reasonable to deduce that the setting times of a cement pastes are relatively longer when percentage of CKD replacement increases.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 General Conclusion

Characterization tests and early-hydration behaviour investigations on CKD has been successfully completed. Throughout the extensive analyses, it can be concluded that CKD is generally feasible to be used as a SCM by partially incorporated into a concrete mix for the purpose of reducing cement usage. However, it is not advisable to be used in structural concrete as the strength development is relatively slower than OPC and PCC.

5.2 Physical, Morphological and Chemical Properties of CKD

According to the physical property, it shows that the average particle size of the CKD is relatively small compared to CKD, which contributes to its high water demand. CKD possesses an average particle size of 0.90 µm whereby the average particle size for OPC is approximately 15 µm. Thus, in this aspect, the feasibility of CKD to be used as an SCM is relatively low.

In the aspect of morphological property, CKD is extremely feasible to be utilized as SCM as it possesses textures and shapes which are highly similar with OPC, which are irregular in shapes and having rough surfaces.

On the other hand, CKD is potentially to be used as an SCM in terms of chemical characteristics. CKD possesses high content of CaO, SiO_2, Al_2O_3, which are 45.01%, 8.09% and 4.97% respectively. These are the compositions suitable to be used as SCM as it is able to carry out pozzolanic and hydraulic reaction by reacting with water and calcium hydroxide to form cementitious compounds such as C-S-H and C-A-H. However, this CKD sample possesses low strength properties as it has low content of CaO_f , which is 0.80% (functions to accelerate the hydration process) and high content of $CaCO_3$, which is 14.20% (retarding the hydration process).

5.3 Early-age Behaviour of CKD

In this aspect, it shows that CKD is feasible to be used as a SCM by replacing cement to a certain percentage. For the standard consistency test, it shows that the higher the replacement percentage of cement in a paste, the greater the water demand. It is shown whereby the normal consistency for CKD is approximately 52.0% where as normal consistency for PCC is only 25.0%.

In the aspect of setting times, CKD also requires longer initial and final setting time, which are 80 and 510 minutes respectively. However, PCC only takes up to 60 and 170 minutes respectively for initial and final setting time. In order to fulfil the BS requirement of setting times of cement (Initial setting time $\geq$ 60 minutes; Final setting time $\leq$ 300 minutes), maximum 75% of CKD can be used as SCM.

5.4 Recommendation

The present research aims to study the feasibility of CKD as a SCM to reduce cement usage by providing a deeper understanding of CKD's properties. Several recommendations regarding improvements made to current study are listed as follows:

1) Quantitative assessment on amorphous SiO_2 content present in CKD shall be carried out as amorphous state of SiO_2 is more reactive in pozzolanic reaction.

2) Temperature and heat of early-hydration of CKD should be investigated thoroughly to get a better understanding of its early-age behaviour.

3) A comprehensive study on engineering and durability properties of concrete and mortar incorporated with CKD can be conducted.

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