EFFECTS OF CHEMICAL REAGENTS ON THE PROPERTIES OF FLY ASH BASED GEOPOLYMER

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ABSTRACT

Geopolymer with high calcium fly ash has a wide prospect in the field of construction, hence due to this fact, it has been significantly considered in the initiation of new era of green materials. Nevertheless, introducing high calcium fly ash in geopolymer has also created a few problems. Addition of high proportion of Ca in geopolymer has diverse effects on the reactivity of fly ash in geopolymer. Moreover, it also could hinder the polymerization process and alter the microstructure of geopolymer. It will significantly result in the high viscosity and contribute to the rapid setting time, low workability and low compressive strength performance. Therefore, alternative chemical reagents such as sodium nitrate, calcium nitrate, citric acid, sucrose and di-potassium hydrogen phosphate with dosage of 0.5 %, 1.5 % and 2.5 % were studied to overwhelm these issues in high calcium geopolymer. The effect of these admixtures on fresh geopolymer was evaluated by considering the setting time and flow table workability characteristic. Another set of parameters like degree of hydration, compressive strength, porosity, scanning electron microscopy and energy-dispersive X-ray spectroscopy tests were conducted to provide the fundamental and elemental composition information on the hardened geopolymer binder. The result shows that the increment of sodium nitrate with proportion of 0.5 % to 2.5 % by weight of fly ash considerably exhibits a prolonged initial setting time but decreased the flowability of geopolymer binder. Meanwhile, calcium nitrate and sucrose has increased the flowability of geopolymer binder while reduced the setting time. Inclusion of di-potassium hydrogen phosphate and citric acid has accelerated the setting time and reduced the flowability of geopolymer binder with the increment of the dosage. The compressive strength of geopolymer binder with the inclusion of alternative chemical reagents are all higher than control except for inclusion of calcium nitrate. Inclusion of 0.5 % alternative chemical reagents were observed to be effective as a retarder and significantly causes the extension of initial setting time. Each type of alkali cation and anion have its own effects on every stage of geopolymerization process. They play the main role in ascertaining the final structure by controlling the crystal growth and contributes to the structural development by forming alumino-silicate structures. Therefore it is concluded that the 0.5 % incorporation of alternative chemical reagents has the potential use as the chemical admixtures for geopolymer.

ABSTRAK

Penggunaan abu terbang yang mengandungi jumlah kalsium yang tinggi telah mewujudkan satu era baru bahan hijau kerana ianya telah diaplikasikan secara meluas di dalam bidang pembinaan. Walau bagaimanapun, penggunaan abu terbang yang mengandungi kandungan kalsium yang tinggi dalam geopolimer telah mewujudkan beberapa permasalahan. Penambahan kadar Ca yang banyak telah memberi kesan ke atas kereaktifan abu terbang dalam geopolimer. Selain itu, ia juga boleh menghalang proses pempolimeran dan menyebabkan perubahan kepada mikrostruktur geopolimer. Ini akan meningkatkan kadar kelikatan dan menyumbang kepada kadar pengerasan yang tinggi, kebolehkerjaan yang rendah serta kekuatan mampatan yang rendah. Oleh itu, reagen kimia alternatif seperti natrium nitrat, kalsium nitrat, asid sitrik, sukrosa dan di-kalium hidrogen fosfat telah dikaji untuk penambahbaikan sifat-sifat geopolimer. Kesan bahan tambah ini pada ciri geopolimer basah dinilai dengan melakukan ujian penetapan masa dan ujian kebolehkerjaan. Selain daripada itu, ujian tahap hidrasi, kekuatan mampatan, keliangan, imbasan elektron mikroskop dan ujian sinar-X spektroskopi tenaga serakan telah dilakukan untuk mengetahui komposisi asas dan unsur pengerasan pengikat geopolimer. Hasil kajian menunjukkan bahawa kenaikan natrium nitrat daripada 0.5 % kepada 2.5 % mengikut berat abu terbang telah memperlambatkan masa pengerasan tetapi merendahkan kebolehaliran geopolimer pengikat. Sementara itu, kalsium nitrat dan sukrosa telah meningkatkan kadar kebolehaliran geopolimer pengikat tetapi mengurangkan masa pengerasan. Kemasukan di-hidrogen fosfat kalium dan asid sitrik ke dalam geopolimer pengikat telah mempercepatkan masa penetapan dan mengurangkan kebolehaliran apabila dos ditingkatkan. Kekuatan mampatan geopolimer pengikat dengan kemasukan reagen kimia alternatif adalah lebih tinggi daripada kawalan spesimen kecuali kalsium nitrat. Kemasukan 0.5% reagen kimia alternatif dapat dikelaskan sebagai bahan pelambat. Setiap jenis kation dan anion alkali mempunyai kesan yang tersendiri pada setiap peringkat dalam proses pengeopolimeran. Proses pengeopolimeran memainkan peranan utama dalam pembentukkan struktur akhir dengan mengawal pertumbuhan kristal dan membentuk struktur alumino-silikat. Kesimpulannya, penggunaan 0.5 % bahan tambah kimia alternatif mempunyai potensi sebagai bahan tambah kimia didalam geopolymer.

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A	Cross sectional area
C	Penetration reading at the time E
D	Penetration reading at time H
E	Time in minutes of last penetration greater than 25mm
F	Maximum loading at failure
F_m	Compressive strength
g	Gram
g/mol	Gram per mol
H	Time in minutes of first penetration less than 25mm
kg/m^3	Kilogram per metre cube
M	Molar
MPa	Mega pascal
m_{sample}	Weight of powdery sample
$m_{residue}$	Weight of dried residue
M_s	Modulus ratio
mm	Millimetre
mm^2	Milimetre square
m^2/g	Meter square per gram
N	Newton
N/s	Newton per second
P	Total load
s	Second
μm	micrometre

%	Percentage
ºC	Degree Celsius

LIST OF ABBREVIATIONS

ACI	American concrete institute
Al	Aluminium
Al_2O_3	Aluminium oxide
ASTM	American society of testing materials
BET	Brunauer emmett teller
C	Carbon
Ca	Calcium
CaO	Calcium oxide
$Ca(OH)_2$	Calcium hydroxide
$Ca(NO_3)_2$	Calcium nitrate
$C_6H_8O_7$	Citric acid
$C_{12}H_{22}O_{11}$	Sucrose
Cr	Chromium
CO_2	Carbon dioxide
C_3S	Tricalcium silicate
C_2S	Dicalcium silicate
C_3A	Tricalcium aluminate
C_4AF	Tetracalcium aluminoferrite
C-S-H	Calcium silicate hydrate
CASH	Calcium aluminum silicate hydrate
EDX	Energy dispersive x-ray analysis
Fe	Iron
Fe^{2+}	Iron (II)

Fe^{3+}	Iron (III)
FeO	Iron (II) oxide
Fe_2O_3	Ferric oxide
FESEM	Field emission scanning electron microscopy
Hg	Mercury
H_2O	Water
K	Potassium
K_2O	Potassium oxide
K_2HPO_4	Di-potassium hydrogen phosphate
$KSiO_3$	Potassium silicate
KOH	Potassium hydroxide
LOI	Loss on ignition
Mg	Magnesium
MgO	Magnesium oxide
Mn_2O_3	Manganese (III) oxide
Na	Sodium
NaOH	Sodium hydroxide
Na_2O	Sodium oxide
Na_2SiO_3	Sodium silicate
$NaNO_3$	Sodium nitrate
NASH	Sodium aluminate silicate hydrate
O_2	Oxygen
OH	Hydroxide
OPC	Ordinary Portland cement
Pb	Lean

P_2O_5	Phosphorus pentoxide
SEM	Scanning electron microscopy
Si	Silicon
SiO_2	Silica oxide
SO_3	Sulphur trioxides
SrO	Strontium oxide
Ti	Titanium
TiO_2	Titanium dioxide
Th	Thorium
U	Uranium
UK	United Kingdom
USA	United States of America
XRF	X-ray fluorescence
w/c	Water to cement ratio

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Deficient landfill space for the disposal of industrial by-products and environmental awareness are some of the leading challenges for industries. Concrete material industries are one of the industrial sector that impaired most of the environmental compliance. Therefore, utilization of industrial waste by-products such as fly ash, bottom ash, palm oil fuel ash, rice husk ash, foundry sand, silica fume and slag can reassure recommendable improvement to environmental excellences and the industry energy efficiencies. Generally, this method utilized the by-product as a replacement material for fine aggregate, coarse aggregate or Portland cement. Thereby, it can reduce the landfill space demand and also the environmental pollution associated with the Portland cement manufacturing.

Portland cement is a fundamental ingredient of concrete and mortar that has been in use since 1824. It is named after Portland limestone in Dorset because of its close similarity to the rock after hydration had taken place. The fine grounded grey powder is made from a mixture of elements manufactured from natural materials like limestone, clay, sand, and shale. Hydration process occurs during the mixing process of Portland cement; water produces a very stiff and strong binding medium suitable for combining the aggregate particles.

Nowadays, annual production of Portland cement has been flourishing. Every one tonne of OPC produced is equally associated with one tonne of carbon dioxide released during the manufacturing process of OPC, as a result of the combustion of fossil fuel and calcination of limestone (Kong and Sanjayan, 2008). Moreover, the production of Portland cement has also give a negative impact on the atmosphere through consequently enhancing global warming due to the greenhouse gases produced (Elimbi et al., 2011). In addition, cement is not only high energy intensive but also consumes significant amount of natural resources. Therefore, an alternative binder is needed to reduce the utilization of Portland cement. According to Somna et al. (2011), geopolymer is currently one of the best alternative binders to Portland cement. The term geopolymer was introduced by the French scientist, Prof. Joseph Davidovits in year 1970. Geopolymer is classified as inorganic polymers formed by the reaction between the alkaline solution produced by amalgamation of potassium or sodium hydroxide and potassium or sodium silicate with aluminosilicate source material which is rich in Silicon and Aluminium. In addition, geopolymer gives excellent fire resistance, low cost, manageable production, environmentally friendly and tremendous thermal properties as compared to conventional concrete produced with OPC (He et al., 2010).

Fly ash has been utilized in the construction industry for many years such as building materials applications, road base, concrete pavement, embankment, asphalt, structure fill and soil stabilization. Fly ash has recently become the foremost source material in geopolymer production as a cement replacement or as a performance enhancer for OPC. Typically, fly ash comes as a coal combustion product that is high in silica and alumina content. In addition, fly ash is a heterogeneous mixture of amorphous and crystalline phase and is generally considered to be a ferroaluminosilicate element (Mattigod et al., 1990). Fly ash is a pozzolanic material and because of its cementitious properties, it can be applied as a cement substitute for producing high performance concrete, mortar and grout. Fly ash is an industrial by product that reacts with the alkaline activating solution to provide a source silicon (Si) and aluminium (Al) and subsequently polymerizes into molecular chains and creates a hardened binder. Regardless of the geopolymer prospect as future cement, there has been lack of suitable commercial chemical admixtures to react with this alkali–activated cement. Therefore, this research is proposing the alternative chemical reagents as the prospective admixture for geopolymer binder, hence further improvement of the geopolymer properties is achievable.

The cement industry is currently facing pressure to find a new technological solution to the greenhouse gas emission resulting from the cement production. Cement generates a lot of carbon dioxide and has a negative influence on the environment. By employing the use of fly ash, it alleviates excessive reliance on cement. Fly ash is one of the most ample by-products of coal combustion that have been historically disposed of in landfills. This creates a serious solid waste problems due to the large amounts of fly ash produced worldwide. Figure 1.1 shows the production of fly ash in nine countries that include Netherland, Italy, Denmark, France, Canada, Australia, UK, Germany, USA, China, and India. India is the leading country in generation of fly ash, with an estimated production of 112 million tones per year. Meanwhile, Denmark, Italy and Netherland are the lowest fly ash producing countries with an estimated production of 2 million tones per year.

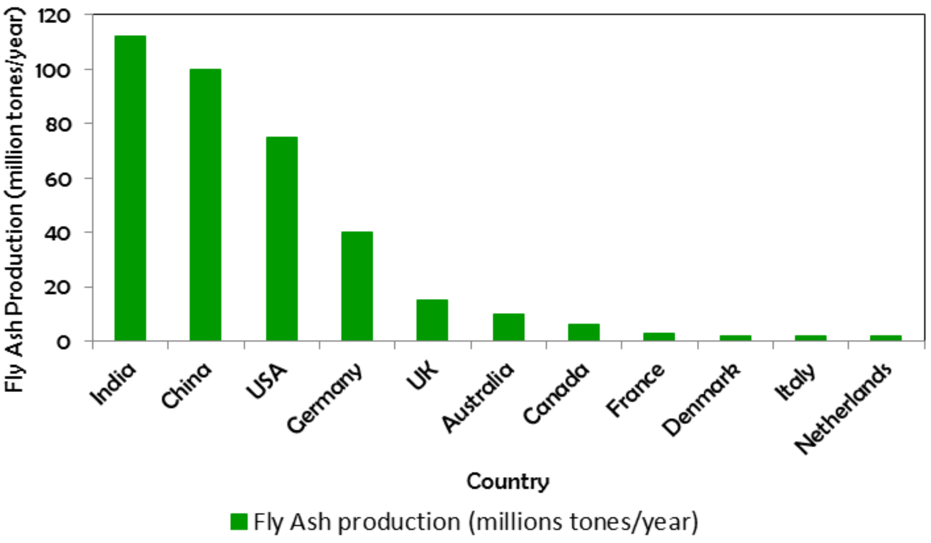

Figure 1.1: Fly ash production

Source: Alam and Akhtar (2011)

Generally, fly ash is disposed on the landfill. Therefore, it requires more storage area and high economic cost, which can inflict further problems on the availability of waste storage space and critical financial issues (Mavroulidao and Lawrence, 2009 and Dhadse et al., 2007). Meanwhile, landfill area is a place where all fly ash is disposed and significantly affects the environment state. Landfill and leachate have always been well related. Leachate is very dangerous as it contaminates the ground water. Since fly ash contains large amounts of toxic metals such as U, Th, Cr, Pb and Hg, a lot of toxic metal leachate accumulates. Toxic metal leachate contaminates the underground water resources and if located to the surface water sources, it may disrupt the aquatic life. Therefore, by enhancing the usage of fly ash as a full replacement of cement in geopolymer concrete, mortar and grout, it will significantly reduce the environmental pollution and financial loss impact.

1.2 Problem Statement

Introduction of geopolymer technology has produced excellent performance as a sustainable binder material than ordinary Portland cement in term of the properties of concrete. Nevertheless, the setting time and workability of geopolymer binder is largely influenced by the CaO content in the source materials. Fly ash consists of two types, that differ in percentages of calcium and are classified as Class C and F. Utilization of high calcium fly ash (Class C) as the source materials in geopolymer phase alters rapid stiffening and low workability. With a low workability, a mixture does not have enough paste to fill all the voids between the aggregates. Therefore, it will be difficult to place and cannot be fully compacted with adversely extruded to form honeycomb and porous concrete. Due to this, it turns out to be an issue during the in – situ casting application. Puligilla and Mondal (2013) states that the occurrence of calcium in fly ash based geopolymer initiates the early onset of hardening, although when all the parameters are constant, the hardening rate of fly ash geopolymer is still enhanced. However, the presence of calcium content in fly ash play a significant role in compressive strength development. The presence of calcium ions provides a faster reactivity and thus yields proper hardening of geopolymer in shorter curing time. Therefore, high calcium fly ash is an effective source material in geopolymer. However, it needs improvement in fresh properties which is setting time and workability. Currently there are no suitable commercial admixtures available for geopolymer, therefore an alternative chemical reagent is urgently needed, particularly for the high calcium source material, to enhance the performance of geopolymer binder during fresh and hardened state. Sodium nitrate, calcium nitrate and di-potassium hydrogen phosphate were chosen as the alternative chemical reagent in this research because there were no available literatures that have been done on Class C fly ash based geopolymer. Therefore, this research proposes alternative chemical reagents of sodium nitrate, sucrose, citric acid, calcium nitrate and di-potassium hydrogen phosphate to be studied as the alternative chemical reagent for fly ash based geopolymer and to observe their effects on the fresh and hardened geopolymer properties.

1.3 Objectives of Study

The objectives of this study are:

1. To determine the effects of alternative chemical reagents on the fresh and hardened geopolymer binder properties.
2. To determine the optimum proportion of alternative chemical reagents that contributes to the improvement of geopolymer binder properties.
3. To determine the classification of alternative chemical reagents in geopolymer admixture system.

1.4 Scope of Study

This research focuses on the effect of using alternative chemical reagents with different proportion of chemical reagents on fresh and hardened geopolymer binder properties. Fly ash was collected from Manjung Coal-Fired Power Plant. Based on the classification given in ASTM C 618-12 ($SiO_2 + Al_2O_3 + Fe_2O_3 \geq 50\%$ and $CaO \geq 10\%$), it was categorized as Class C fly ash. Therefore is used as the main source material in this research. A combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) was used as the activating alkaline solution. Na_2SiO_3 with $SiO_2/Na_2O=2.0$ was combined with 6 M NaOH solution to activate the source material. All the specimens were cured at room temperature (28±2 °C). Variations of alternative chemical reagents were used to observe the effects on the geopolymerization which are sodium nitrate, citric acid, sucrose, calcium nitrate and di-potassium hydrogen phosphate with various proportions were included at 0.5 %, 1.5 %, and 2.5 % of fly ash weight. Evaluation of the chemical reagents was conducted based on workability and setting time test on the fresh geopolymer paste. Meanwhile, performance of hardened geopolymer specimens was measured by degree of reaction, compressive strength and porosity test. Degree of reaction was tested at the ages of 1 and 7 days, while the compressive and porosity test at the 1, 7, 28, and 90 days. SEM and EDX test were conducted to investigate the material characterization on the day of 1 and 90.

1.5 Significance of Study

This research investigated the performance of numerous chemical reagents in fly ash based geopolymer binder. Chemical reagents such as citric acid, sodium nitrate, sucrose, calcium nitrate and di-potassium hydrogen phosphate effect on the geopolymerization process was observed. High content of CaO in the source materials significantly affected the setting time and workability of geopolymer binder. Utilization of high calcium (Class C) fly ash as the source material component would accelerate the stiffening rate of geopolymer binder. This rapid formation of a geopolymer binder framework was conducive to the early strength development. Nevertheless, it significantly decreases the setting time and workability performance. Therefore, the inclusion of these types of chemical reagents into the geopolymer binder assisted in recognizing the suitable admixtures and its optimum dosage that can help in prolonging the setting time, high workability and enhancing the mechanical properties of geopolymer concurrently.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The available published literature on geopolymer technology and ordinary Portland cement (OPC) are presented in this chapter. Geopolymer has recently arisen as a novel engineering binder material that fully replace of utilization OPC in concrete. Normally in concrete, OPC and water are the main ingredient. However, in geopolymer the main sources were aluminosilicate powder and alkaline solution. Alkaline solution plays the main role to activate the geopolymerization process and geopolymer cements are called as alkali activated materials.

2.2 Ordinary Portland Cement (OPC)

OPC is a fine gray ground powder that carries an important role in concrete. Amalgamation between water and OPC generate the hydration process to form a very hard and strong binding medium for the aggregate particles. The basic raw material that utilized during the production of OPC cements are a combination of calcareous and argillaceous. Calcareous rock (limestone or chalk) generates the calcium carbonate and for silica, alumina and iron oxide is found in argillaceous rocks such as clay or shale. There are four main chemical compounds of OPC cements which are tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite. In addition, Neville (2006) has introduced the shortened notation in cement compounds were used to illustrate the oxide composition such as CaO=C, SiO_2=S, Al_2O_3=A and Fe_2O_3=F. The oxide compositions of these compounds are tabulated in Table 2.1.

Table 2.1: Main compounds of Portland cement

Name of Compound	Chemical Composition	Abbreviation
Tricalcium silicate	$3CaO.SiO_2$	C_3S
Dicalcium silicate	$2CaO.SiO_2$	C_2S
Tricalcium aluminate	$3CaO.Al_2O_3$	C_3A
Tetracalcium aluminoferrite	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF

Source: Neville (2006)

Figure 2.1 shows the simplified illustration of cement paste hydration. According to Moir (2003), during the hydration process, silicate phase (C_3S and C_2S) and aluminate phases (C_3A and C_4AF) occur separately as the reaction is very complex. Smaller C_3A and C_4AF particles formed around the silicate particle during the fresh cement grain. The hydration of cement involve in several distinct periods. On the dissolution stage, an amorphous gel was formed round the cement grains when the reaction occurs between sulphate, aluminate and water and produce short rods of ettringite. After that, the cement hydration slows down and the induction stage begins. During this stage, about 30% of cement are counters to form C–S–H and calcium hydroxide and its take about 3 hours. Calcium ion was also released into the solution. After about 3 hours of hydration, the induction stage ends and the acceleratory stage begin.

After 10 hours hydration C_3S has produced the outer C–S–H, which grows out from the ettringite rods rather than directly out from the surface of the C_3S particles. Therefore, in the initial phase of the reaction, the silicate ions must migrate through the aluminum and iron rich phase to form the C–S–H and its contribute to the early strength. This network of ettringite and C–S–H appears to form a “hydrating shell” about 1 µm from the surface of anhydrous C_3S. A small amount of “inner C–S–H” forms inside this shell. After 1–3 days of hydration, reactions slow down and the deceleratory stage begins. In this stage, C_3A reacts with ettringite to form some monosulfate. Then the inner C–S–H continues to grow near the C_3S surface, narrowing the 1 µm gap between the “hydrating shell” and anhydrous C_3S. The rate of hydration is likely to depend on the diffusion rate of water or ions to the anhydrous surface. After 2 weeks hydration densification stage begins where the gap between the “hydrating shell” and the grain is completely filled with C–S–H. The original, “outer C–S–H” becomes more fibrous.

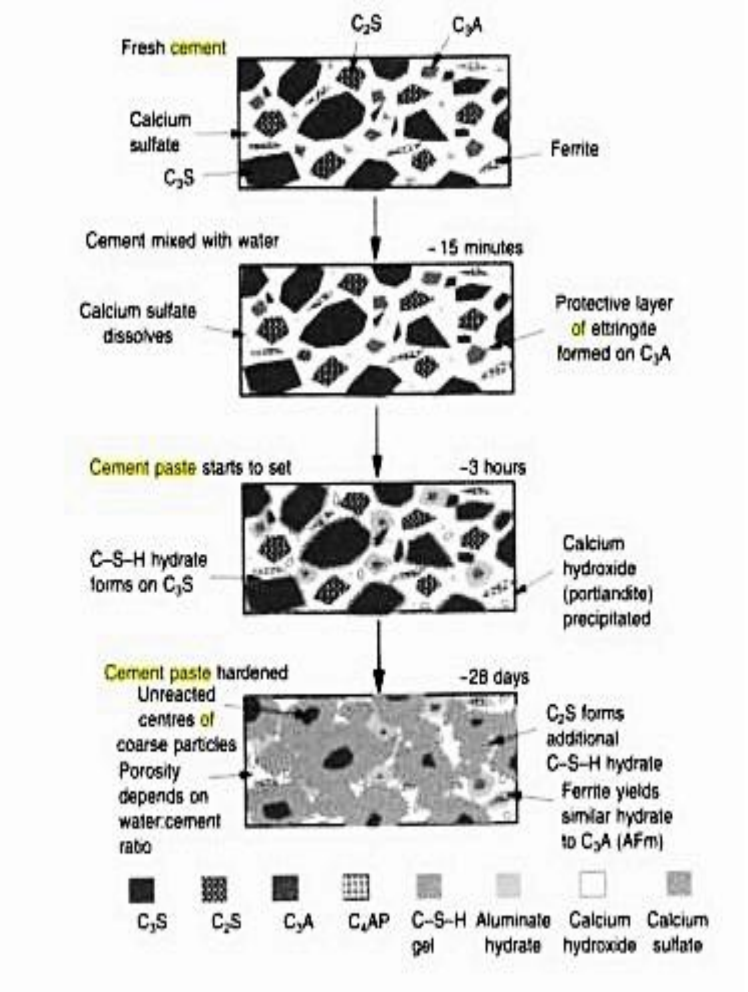

Figure 2.1: Simplified illustration of cement paste hydration

Source: Moir (2003)

2.3 Geopolymer

Geopolymer is a term that can be illustrated as an inorganic polymer based on aluminosilicates and can be generated by synthesizing pozzolanic compounds or aluminosilicates source materials with highly alkaline solutions such as sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium silicate or potassium silicate.

CHAPTER 1

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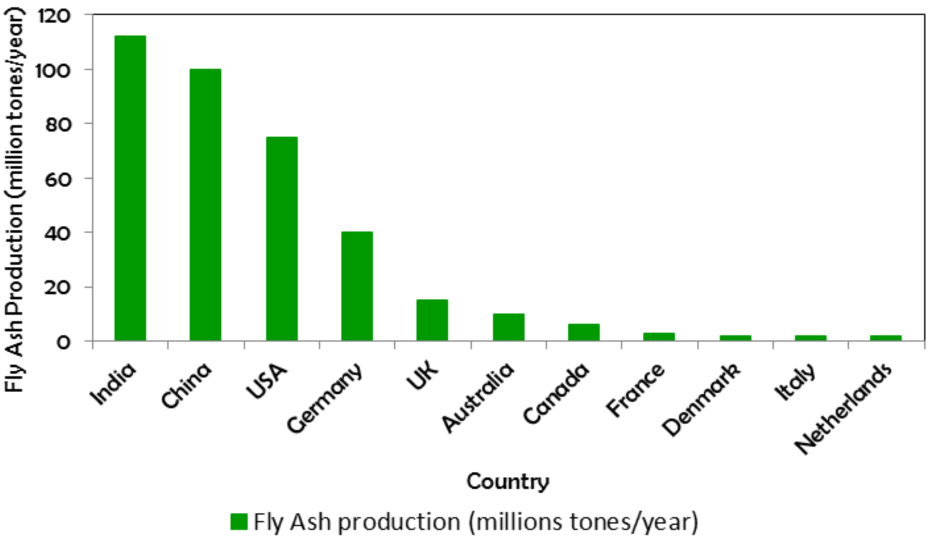

Figure 1.1: Fly ash production

Source: Alam and Akhtar (2011)

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CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter focuses on the production details of geopolymer binder with the addition of alternative chemical reagents. Geopolymer is one of the potential cement substitute material, nevertheless it lacks the suitable commercial admixtures to modify the early reaction of geopolymer binder. The purpose of this research is to evaluate the performance of alternative chemical reagents in fly ash based geopolymer binder. Fly ash based geopolymer binder was added with different chemical reagents such as citric acid, sodium nitrate, sucrose, calcium nitrate and di-potassium hydrogen phosphate in various percentages. The inclusion of chemical reagents was expected to improve the setting time, workability and mechanical performance of the geopolymer binder. In this research, the testing was conducted in two conditions, which are during fresh and hardened state. Setting time and workability test were conducted on the fresh paste, while the degree of reaction, compressive strength, porosity, EDX and SEM testing were conducted on the hardened specimens. *t*- test was selected as the statistical analysis method to determine the effectiveness of the data while SYSTAT was used to deliberate the interrelationships between pairs of variables that affect polymerization of geopolymer precursors to the geopolymer properties. Figure 3.1 shows the flow of the research methodology that was performed throughout the research period.

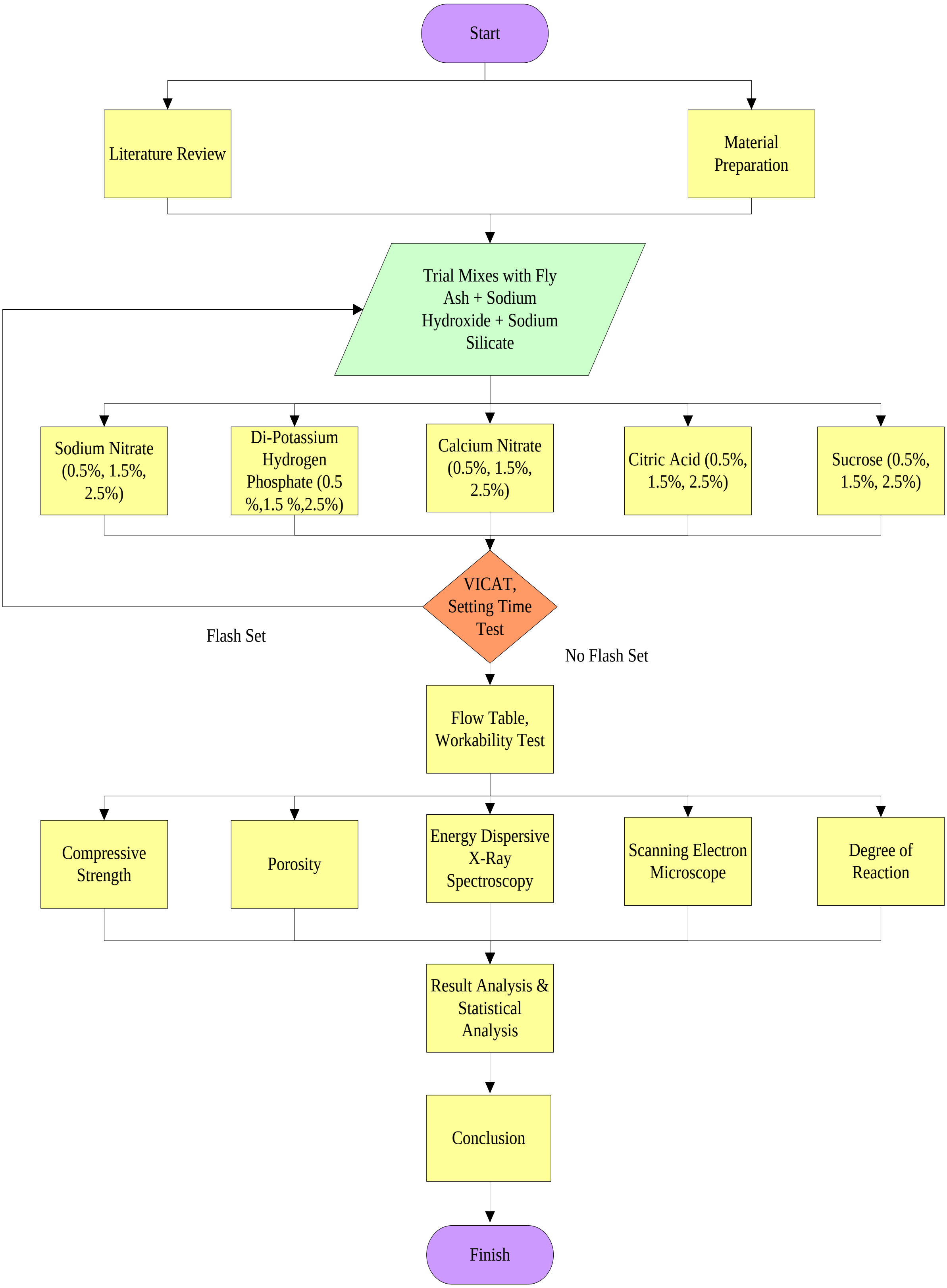

Figure 3.1: Flow chart of research methodology

3.2 Material Preparation

In this research, three types of raw materials were used, which are fly ash, alkaline activator (sodium hydroxide and sodium silicate) and chemical reagents (sodium nitrate, citric acid, sucrose, calcium nitrate and di-potassium hydrogen phosphate).

3.2.1 *Fly Ash*

Fly ash was used as the primary source material in this research. It was classified as Class C fly ash and was obtained from Manjung Coal – Fired Power Plant, Perak as shown in Figure 3.2. The chemical compositions of fly ash were determined using X-Ray Fluorescence (XRF) analysis and tabulated in Table 3.1. From Table 3.1, SiO_2 (36.37 %), Al_2O_3 (17.57 %), Fe_2O_3 (12.43 %) and CaO (10.58 %) were detected as the major oxides in fly ash with 1.19 % Loss on Ignition (LOI).

Table 3.1: Oxide composition of fly ash

Oxide	**% wt**
Al_2O_3	17.57
SiO_2	36.37
P_2O_5	0.28
SO_3	1.39
K_2O	1.77
CaO	10.58
TiO_2	0.88
Fe_2O_3	12.43
SrO	0.12
Mn_2O_3	0.11
MgO	3.05
LOI	1.19