THE POTENTIALS OF *PENNISETUM PURPUREUM* ASH AS CEMENT REPLACEMENT MATERIAL

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I dedicated this thesis to my beloved husband and entire family whom never stop believing in me to complete this thesis work, and to my dear Muhammad Ahnaf.

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ABSTRACT

Cement production requires non-renewable natural resources that will be depleted in coming years. An alternative needs to be found to promote sustainable development for the construction industry. Therefore, this study introduced *Pennisetum Purpureum* ash (PPA) as a natural pozzolan. Limited information was published on the influence of PPA particle size in cement products. Therefore, this study investigated the physical and chemical properties of PPA as a cement replacement material and the effects of particle sizes of PPA with different replacement percentages on compressive strength and water absorption of cement mortar. The morphology of mortar containing PPA was also analysed. The PPA was prepared through controlled burning and grinding process before going through particle size analysis, XRF and SEM. The PPA used were ground for 1 hour (1H-PPA), 3 hours (3H-PPA) and 6 hours (6H-PPA) while the percentage of replacement in mortar were 5%, 10%, 15%, and 20%. The mortar was cured for 3, 7 and 28 days for mechanical properties characterisation. It was found that the particle size of PPA decreased as the time of grinding increased, which also increased the surface area. The particle size for 1H-PPA, 3H-PPA and 6H-PPA were 9.30µm, 10.25µm and 10.58µm respectively. The total reactive oxides for 1H-PPA, 3H-PPA and 6H-PPA were 38%, 37% and 41.1% respectively while the loss on ignition were above 5%. As a conclusion, the grinding process degraded the honeycomb-like PPA structure into smaller particle size with highly irregular shape but in better dispersion. The 15% replacement of 6H-PPA mortar at 28 days showed the significant performance (30.07 N/mm^2 of strength) with increment strength by 43.74% compared to control mortar with the lowest water absorption rate of 8.2%.

ABSTRAK

Penghasilan simen memerlukan sumber semulajadi yang tidak boleh diperbaharui semula akan habis pada tahun mendatang. Satu alternatif perlu dicari untuk mempromosikan pembangunan lestari bagi industri pembinaan. Oleh itu, kajian ini memperkenalkan abu *Pennisetum Purpureum* (PPA) sebagai pozzolan semula jadi. Penerbitan yang agak terhad mengenai pengaruh saiz zarah PPA dalam produk berasaskan simen. Oleh itu, kajian ini menkaji sifat fizikal dan kimia PPA sebagai bahan pengganti simen, kesan saiz zarah PPA dengan peratusan penggantian yang berbeza terhadap kekuatan mampatan dan penyerapan air mortar simen. Morfologi mortar yang mengandungi PPA juga dianalisis. PPA dihasilkan melalui proses pembakaran dan pengisaran yang terkawal sebelum melalui analisis saiz zarah, XRF dan SEM. PPA yang digunakan adalah 1 jam pengisaran (1H-PPA), 3 jam pengisaran (3H-PPA) dan 6 jam pengisaran (6H-PPA) manakala peratus penggantian mortar adalah 5%, 10%, 15%, dan 20%. Mortar telah diawet selama 3, 7 dan 28 hari untuk ujian pencirian sifat mekanikal. Kajian ini telah mendapati bahawa saiz zarah PPA menurun apabila masa pengisaran bertambah, dimana ia juga meningkatkan luas permukaan partikel. Saiz zarah untuk 1H-PPA, 3H-PPA dan 6H-PPA masing-masing adalah 9.30µm, 10.25µm dan 10.58µm. Jumlah oksida reaktif untuk 1H-PPA, 3H-PPA dan 6H-PPA masing-masing adalah 38%, 37% dan 41.1%, manakala kehilangan pada pencucuhan berada di atas 5%. Telah didapati bahawa proses pengisaran menjadikan struktur PPA yang asalnya seolah-olah seperti sarang lebah kepada saiz zarah yang lebih kecil dengan bentuk yang sangat tidak teratur tetapi dalam penyebaran yang lebih baik. Penggantian mortar 6H-PPA sebanyak 15% pada 28 hari menunjukkan prestasi yang terbaik (30.07 N/mm^2 kekuatan) dengan peratus peningkatan kekuatan sebanyak 43.74% dengan kadar penyerapan air terendah sebanyak 8.2% jika dibandingkan dengan mortar kawalan.

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
Al_2O_3	Aluminium oxide
BA	Bagasse ash
BLA	Bamboo leaf ash
C	Carbon
CaO	Calcium oxide
$Ca(OH)_2$	Calcium hydroxide
CCA	Corn cob ash
CH	Calcium hydroxide
CO_2	Carbon dioxide
C_3A	Tricalcium aluminate
C_4AF	Tetracalcium aluminoferrite
Cl	Chloride
C_2S	Dicalcium silicate
C_3S	Tricalcium silicate
C-S-H	Calcium silicate hydrate
˚C	Degree Celsius
EGA	Elephant grass ash
Fe_2O_3	Iron oxide
FST	Final setting time
g	Gram
HCl	Hydrochloric acid
H_2O	Water
IST	Initial setting time
K_2O	Pottasium oxide

kg	Kilogram
LOI	Loss in ignition
MgO	magnesium oxide
ml	Milliliter
mm	Millimeter
µm	Micrometer
MPa	Megapascal
N	Newton
N/mm^2	Newton per millimeter square
POFA	Palm oil fuel ash
PPA	Pennisetum Purpureum ash
UG-PPA	Unground Pennisetum Purpureum ash
1H-PPA	1 hour ground Pennisetum Purpureum ash
3H-PPA	3 hours ground Pennisetum Purpureum ash
6H-PPA	6 hours ground Pennisetum Purpureum ash
RHA	Rice husk ash
SCM	Supplementary cementitious materials
SEM	Scanning Electron Microscopy
SF	Silica fume
%	Percentage
wt%	Weight percent
SiO_2	Silica oxide
SO_3	Sulphur trioxide
TiO_2	Titanium dioxide
XRD	X-Ray Diffraction
XRF	X-Ray Fluoresence

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The cement industry is one of the strategic industries of Malaysia, acting as the contributor to several other industries like real-estate, construction and infrastructure and also plays an important role in the world economy (Ghiasvand and Ramezanianpour, 2017). Cement is expected to increase in demand in the near future mainly because of the consistent government investments and key infrastructure projects along with the ongoing residential and commercial developments. While in United State, an average of 75 to 90 million tons per year of cement consumption has been estimated by the Portland Cement Association, and this number will exceed 100 million tons per year by 1997. According to the United State Bureau of Mines, a total of 1.25 billion tons cement was produced worldwide in 1991.

Meanwhile, in Malaysia's cement industry, continuous growth in demand has been faced by manufacturers since 2009. Due to overcapacity and contraction in local demand, the cement supply exceeded demand in 2016. The increase in cement demand also led to intense price pressure in 2016. In 2017, the estimated annual production of cement was 40.2 million metric ton produced by 8 cement manufacturers with 18 cement plants nationwide. In 2016, the capacity of utilisation was recorded as 59%, lower from the past few years which were 67% - 72% (Market Review of Building Materials in the Construction Industry Report, Malaysia Competition Commission by Ipsos Business Consulting 2017).

The concept of sustainable development defined by United Nation Sustainable Development (1987) is meeting the needs of the present without compromising the ability of future generations to meet their own needs. Meanwhile, according to Amrina and Vilsi (2015), the aim of a sustainable manufacturing approach was to reduce the intensity of material usage and energy consumption, improving the final products while decreasing the unwanted byproducts. Recently, the sustainability concept in engineering practice is not only focused on the environmentally friendly but also on the development of new materials for construction purposes. As there are many other raw materials considered as waste that can be reutilised as by-products to be used as cement replacement materials especially waste produced by industriV al activities. Previous studies have found that biomass waste ashes that contained high amount of silica contents such as sugar cane bagasse ashes, rice husk ashes, fly ash, bottom ash and forest residues can be used as supplementary cementitious materials (SCMs). The 5% of anthropogenic carbon dioxide (CO_2) emissions came from world cement industry (Wang, Zhu and Geng, 2013). The energy consumption and carbon dioxide (CO_2) emissions also can be reduced through using SCMs (Ghiasvand and Ramezanianpour, 2017). A portion of cement in a concrete system replaced by SCMs will participate in primary or secondary hydration reactions in which, increasing the volume fraction of binding phases and decreasing porosity in the microstructure of the concrete (Ghiasvand and Ramezanianpour, 2017).

At present, Brazil is using 100% of agricultural plant called elephant grass or Napier grass (*Pennisetum Purpureum*) as a renewable energy source in several thermoelectric power plants there. This approach has been taken as an alternative energy resource since elephant grass is an abundant and fast growing plant that has reasonable energy content and higher calorific value fuels also categorised as biomass materials (Nakanishi *et al*., 2014). Biomass is defined as a sustainable energy source with carbon dioxide (CO_2) neutralisation potential. From this activity, a large quantity of elephant grass ashes was generated during direct burning process that will finally be deposited in landfills with the consequent environmental, technical, economic and social problems (Cordeiro and Sales, 2015). Meanwhile, the reuse of biomass waste (elephant grass ashes) will decrease the impact associated with the environment, apart from reducing dependence on natural resources that cannot be renewed. The elephant grass contains a considerable amount of amorphous silica due to the extraction of ortho-silicic acid from ground water by the plant and its ashes can be economically attractive to the production of a pozzolan which is similar to some by-products used as biomass such as sugar cane bagasse and rice husk (Cordeiro and Sales, 2015). The ashes from elephant grass is suitable for use as an active cement addition as it presents high silica content which is more than 50% and mostly reactive silica for its low crystalline and amorphous nature (Nakanishi *et al*., 2015).

Supplementary cementitious material used in concretes, mortars and paste may come from the pozzolans extracted from agricultural by-products with over 60% of unit weight amorphous or poorly crystalline silica (Cordeiro and Sales, 2015). It seems that biomass resources have both advantages in serving alternatives energy and reutilisation of their waste in cement industry and country will be able to develop the sustainable approach.

The search for alternate mortar and concrete components, preferably made from recycled and waste materials, becomes crucial. Still, the basic condition for the implementation of such innovative approaches is the assurance that it will not result in any significant reduction in the quality of the structures and elements built with these products. Napier grass, scientifically known as *Pennisetum Purpureum*, is a newly-identified plant which forms robust bamboo-like clumps and is highly sustainable throughout Malaysia.

1.2 Problem Statement

In 2017, the estimated annual production of cement was 40.2 million metric ton produced by 8 cement manufacturers with 18 cement plants nationwide in order to meet the growth demand since 2009 (Market review of building materials in the construction industry report, Malaysia Competition Commission by Ipsos Business Consulting 2017). The cement production required a natural resource of calcium, usually limestone and fossil fuels are also source of silica such as clay or sand. This kind of resources is non-renewable and will be depleted in coming years, beside their byproducts remarked as emitters of pollutants (Ali, Saidur and Hossain, 2011; Pardo, Moya and Merceir, 2011). As reported in Malaysia Cement Industry (Report, 2015), the industry will also have to meet global pollution and emission standards which will take considerable amount of investment since cement industries were responsible for carbon dioxide emission. It is well known that carbon dioxide is one of the major constituents of the greenhouse gases causing global warming and environmental pollution. Hence, the global warming will keep arising if enormous amount of carbon dioxide is emitted from the massive production of cement.

Therefore, sustainable manufacturing approaches in cement industry become crucial (Uson *et al*., 2013).

An alternative needs to be found to help the cement industry not just to protect the environment, but also to promote sustainable development for the construction industry and the country. Therefore, the search for the alternatives binders, natural admixtures or cement replacement materials has been the subject of many publications. Concrete materials should not only possess good workability, excellent mechanical properties and durability but also offer environmental and economic benefits. An immediate alternative needs to be found to reduce the cement consumption and environmental pollution without compromising the demand for the cement materials. The alternatives should not be compromising the engineering properties and should even improve the performances of cement based products. Other than traditional cementitious materials such as rice husk ash, silica fume and fly ash, one of the alternatives is the ashes extracted from elephant grass or known as *Pennisetum Purpureum* ashes (PPA). The leaves of elephant grass are currently used as a food source of ruminants but its trunks are dumped to the landfill. The trunks from the grass could be recycled into silica base products. Apart from that, the elephant grass is a fast growing plant and is widely planted in Malaysia. Malaysia is one the Asia countries which dispose a large amount of agricultural waste that may result in social and environment problem if it is not properly disposed. Recycling the waste as construction materials is one of the methods of treating the agricultural waste where this approach also resulted in energy and cost saving (Sathawane *et al*.,2013). Due to the lack of similar research works on PPA usage in Malaysia, a further study needs to be done to report the potentials of elephant grass ashes as cement replacement materials.

Several studies have been published on the performance of PPA (Cordeiro and Sales, 2015; Cordeiro and Sales, 2016; Nakanishi *et al*., 2016). However, limited information is available on the influence of PPA particle size in cement based products. Therefore, this study investigated the effects of partial replacement of cement with different percentages and particle sizes of PPA on the physical properties, compressive strength and water absorption of cement mortar to develop a structure with most favourable properties.

1.3 Objectives of the study

The main focus in this study was to find the potential of elephant grass ashes as a cement replacement material. It should be noted that the *Pennisetum Purpurem* ashes (PPA) term was used in this study. The following objectives are:

i. To characterise the physical and chemical properties of PPA as a cement replacement material.
ii. To investigate the effect of PPA on compressive strength and water absorption of cement mortar.
iii. To analyse the morphology properties of cement mortar containing PPA.

1.4 Scopes of study

This study involved the usage of *Pennisetum Purpureum* ashes (PPA) as partial cement replacement material. The PPA used was processed from the whole part of the *Pennisetum Purpureum* grass through controlled burning and grinding process. The PPA were used in three different sizes based on the different grinding time (1 hour, 3 hour and 6 hour). Meanwhile, the percentage of replacement of PPA were 0%, 5%, 10%, 15%, and up to 20%.

This study was divided into three phases. In the first phase, the cement and PPA properties were tested. For the raw materials, the testing involved were particle size analysis using laser particle size analyser (Fristch Analysette 22), and testing for their loss on ignition according to standard ASTM C114. Meanwhile, X-Ray Fluoresence (XRF) and Scanning Electron Microscopy (SEM) were conducted onto these raw materials to obtain their chemical composition and morphology conditions. The fresh state of cement and PPA paste were through standard consistency (ASTM C187) and setting time behavior (ASTM C191). All testings were important to characterise the properties of cement also PPA as cement replacement material.

In the second phase, the mechanical properties were found through compressive strength test (ASTM C109) and water absorption (ASTM C1403) using cement mortar size of 50 mm × 50 mm × 50 mm at 3, 7, and 28 days of curing period. The curing method in this study was wet curing. The crushed specimen after mechanical testing was collected for the specimen preparation to conduct the

Scanning Electron Microscopy analysis (SEM) as a third phase of the study. SEM testing was conducted to evaluate the morphology of cement mortar containing PPA.

1.5 Significance of study

This study could provide detailed information on the usage of *Pennisetum Purpureum* grass in construction industries apart from being used as fiber and livestock feeding. Apart from that, this study could enrich the relationship between particle size and the strength of the building materials using PPA as alternative material other than rice husk ash, fly ash, bottom ash and other byproducts.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Mortar basically consists of cement, sand and water. Ordinary Portland Cement (OPC) plays an important role as a binder in mortar. The productions of OPC contribute to high carbon dioxide (CO_2) emission and high energy consumption; therefore, alternative binding materials are needed to partially replace the OPC. Furthermore, cement is considered as a vital material to be used in the construction industries and its production requires large amount of natural resources that cannot be renewed. Any action helping to improve cement sustainability is one of the great interests for environmental protection. Mortar with cement replacement material of mineral admixtures or pozzolan is called modified mortar (Gingos and Mohamed Sutan, 2011) in which this pozzolan helps to improve the durability of produced mortar. The *Pennisetum Purpureum* ash (PPA) was used as cement replacement to produce mortar blended with PPA at different particle sizes.

2.2 Ordinary Portland cement

Cement is a vital material in construction industry that acts as the binding agent that holds sand and other aggregates together in a hard and stone-like mass. There are two types of cement used in the construction industry; hydraulic and non-hydraulic cement. Hydraulic cement is any cement that can combine with water to turn into solid products while non-hydraulic cement does not require water to transform into solid products.

Cement is manufactured through calcination process by heating limestone (calcium carbonate) with other materials such as clay to 1450 °C in a kiln (Somayaji, 2001). Table 2.1 shows the common raw materials and compounds used to manufacture the portland cement. In this process, a molecule of carbon dioxide was released from the calcium carbonate to form calcium oxide, or quicklime, which was then blended with the other materials that have been included in the mix to form calcium silicates and other cementitious compounds. The resulting hard substance, called clinker, was then ground with a small amount of gypsum into a powder to make Ordinary Portland Cement (OPC), which is the most commonly used type of hydraulic cement and this type of cement is commonly in grey or white colour.

Ordinary Portland cement was comprised of the four main cement minerals, which are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetracalcium aluminoferrite (C_4AF), together with the remainder consisting of calcium sulfate, alkali sulfates, unreacted calcium oxide (CaO), magnesium oxide (MgO), and other minor constituents left over from the clinkering and grinding steps (Marchon and Flatt, 2016). The four cement minerals play different roles in the hydration process which converts the dry cement into hardened cement paste. The C_3S and C_2S or known as alite and belite are the major contributors to the main hydration products, calcium silicate hydrates (C-S-H) gel. However, the C_3S hydrates much more quickly than the C_2S which makes it responsible for the early strength development. The C_3A and C_4AF minerals also hydrate, but the products that are formed do not affect much on the properties of the cement paste.

Table 2.1: Common raw materials ad compounds used in the manufacturing of portland cement (Somayaji, 2001)

Raw material	**Compound**
Cement rock	Lime, silica, alumina
Limestone	Lime
Slag	Lime, silica, alumina
Marble	Lime
Clay	Silica, alumina
Shale	Alumina
Fly ash	Alumina, silica
Kaolin	Alumina

Table 2.1 (continued)

Bauxite	Alumina
Sand	Silica
Quartzite	Silica
Iron ore	Iron oxide
Gypsum	Sulfate

2.3 Mechanism of hydration process of Portland cement

The hydration reaction was promoted by various components at different dissolution rates, where the Ca^{2+} concentration increased by dissolution of C_3S and C_2S which then formed a large quantity of $Ca(OH)_2$ and amorphous C-S-H gels in the pastes (Yin *et al.*, 2018). Majority of reviews regarding the hydration process of Portland cement were focusing on the hydration characteristics of tricalcium silicate, C_3S. The C_3S (alite) constituted about 50% to 70% of Portland cement by mass. In addition, C_3S was found in dominating the early hydration period that is responsible for setting and early strength development. On the other hand, other silicate C_2S (belite) was responsible in late strength development due to its lower reactivity. The process mentioned is responsible for the formation of the calcium silicate hydrate gel (C-S-H) as hydrated products (Bullard *et al.*, 2011). Calcium silicate hydrate gel (C-S-H) formed in a mass of interlocking needles provided the strength of cement system. In addition, a crystalline phase, calcium hydroxide known as portlandite (CH) was observed to be as hydrated products (Marchon and Flatt, 2016). Equation 2.1 illustrated the reaction of amorphous silica with Ca^{2+} and OH^- ions with calcium hydroxide during the cement hydration process formed C-S-H gel that contribute to the strength improvement and durability of mortar and concrete. The C-S-H gel produced in previous hydration process in cement mixture reacted with remaining $Ca(OH)_2$ to produce more C-S-H gel when another pozzolan was added in which is called as pozzolanic reaction.

$$2C_3S + 6H_2O \rightarrow C\text{-}S\text{-}H + 3CH \tag{2.1}$$

As a matter of fact, the size of a cement particle also has an important effect on the rate at which it will hydrate when exposed to water. As it reacts, a layer of hydration product forms around the outside of the particle, separating the unreacted core of the particle from the surrounding water. As this layer grows thicker, the rate of hydration slows down. Therefore, a small particle will react much more quickly than a large particle. A particle that has a diameter of 1 μm will react completely in about 1 day, whereas a particle with a diameter of 10 μm will react completely in about 1 month. Particles larger than about 50 μm will probably never become fully reacted, even if there is a sufficient source of water. Clearly, the particle size distribution is critical for controlling the rate at which cement sets and gains strength.

The filler effect or particle size effect is defined as the proper arrangement of small particles that fill the voids and contribute to the increment of compressive strength without any chemical reaction. Smaller pozzolan particles tend to produce a higher compressive strength than coarser particles (Jaturapitakkul *et al*., 2011). Fine particles as refinement on pore structure acts as a nucleation point for hydration products, restricts the development of unfavourable crystals generated in hydration process. Filler effect gives increment in compressive strength and decreases the permeability of mortar and concrete related to physical effects that can be justified. If the particle size distribution of the minerals admixtures is refined, it can increase the packing density of the mixture as well as the chemical reactivity of the materials due to the increase in a specific surface area (Cordeiro *et al*., 2009a). Regarding their use as a cement replacement, mineral admixtures affect the performances of paste, mortar and concrete owing to both physical and chemical effects. The physical effects are primarily associated with their influence on the packing characteristics of the mixture, which depend on size, shape and texture of the particles.

Meanwhile, the chemical effects are associated with their capability of providing siliceous / aluminous compounds that will chemically react with calcium hydroxide in the presence of water (Cordeiro *et al*., 2009b). According to Goldman and Bentur (1994), the capability of pozzolanic materials of enhancing the strength of concrete is more closely associated with physical than chemical effects.

2.4 Basic microstructure of hydrated products

The structure and properties of hydration products, together with microstructure of hardened cement paste, plays important roles in determining the hardened paste properties. The hydrated cement paste microstructure is composed of just three phases

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