PERFORMANCE OF TERNARY BLENDED CEMENT MORTAR CONTAINING PALM OIL FUEL ASH AND METAKAOLIN

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Dedicated to

my family

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ABSTRACT

The partial substitution of Portland cement with pozzolans in concrete greatly reduces the environmental pollution due to CO_2 emission during cement production. Pozzolans equally enhance mechanical properties and guarantee the production of concrete with minimum costs. These added benefits, result in the increasing use of pozzolans as a significant innovation in the construction industry. Although palm oil fuel ash (POFA) as pozzolan improves strength and durability of concrete, it however delays early strength development due to its low pozzolanicity. Conversely, metakaolin (MK) improves early strength development but equally reduces workability and increases heat of hydration which can be detrimental to the durability of concrete. MK is also deficient in magnesium sulfate environment and at high temperatures. Thus, the scope of application of the binary blends of POFA and MK in the construction industry may be limited. However, the simultaneous use of these materials in the form of ternary blend has the potential to compensate for the deficiencies due to their synergistic interactions. Hence, this study was set out to investigate the effects of the combination of POFA and MK on the properties of cement mortar. Accordingly, a total of 17 different mortar mixtures of binary and ternary blends of POFA and MK at up to 30% replacement levels by weight, and water to binder ratio of 0.55 were used. An optimal ternary blend in terms of strength development and porosity reduction was selected for further detailed investigation. The properties of the optimal ternary assessed at its fresh state include; consistency, setting times, workability and temperature rise. While at its hardened state, compressive strength, sorptivity and microstructures were evaluated. The durability was studied in terms of resistance to sulfuric acid attack, sulfates attack and at high temperatures. The properties of the binders were also examined and their conformity to the relevant standards was confirmed. The results showed that the optimal ternary blend was 10% POFA and 10% MK. The ternary blend significantly improved the workability of mortar with minimal use of superplasticizer compared to MK binary blend. It was also discovered that while the MK binary blend increased the semi-adiabatic temperature by 7% compared to plain OPC, the ternary blend showed a reduction by 4%. Besides, the ternary blend was not only effective in offsetting the low compressive strength of POFA binary at early ages but also enhanced the long-term strength compared to MK, and POFA binary. The TGA and XRD data proved that the early strength improvement of the ternary blend was due to the high pozzolanicity of MK. Furthermore, the ternary blend exhibited superior performance over the MK binary blend and plain OPC in terms of resistance to magnesium sulphate attack and at high temperatures. Generally, the optimal ternary blend of OPC, MK and POFA showed better performance and can be used in construction particularly where the binary blends of either POFA or MK proved deficient. The combined use of POFA and MK would contribute not only to the development of environmental friendly material but also the reduction of CO_2 emission.

ABSTRAK

Penggantian sebahagian simen Portland dengan bahan pozolana dalam konkrit dapat mengurangkan masalah pencemaran alam sekitar disebabkan oleh pembebasan CO_2 semasa pengeluaran simen. Pozolana juga meningkatkan sifat mekanikal dan menjamin pengeluaran konkrit dengan kos yang minima. Kelebihan ini meningkatkan penggunaan pozolana sebagai satu inovasi dalam industri pembinaan. Walaupun abu kelapa sawit (POFA) sebagai bahan pozolana meningkatkan kekuatan dan ketahanlasakan konkrit bagaimanapun perkembangan kekuatan awal adalah kurang disebabkan rendah sifat pozolananya. Sebaliknya Metakolin (MK) meningkatkan perkembangan kekuatan awal tetapi mengurangkan kebolehkerjaan dan meningkatkan haba penghidratan yang boleh menimbulkan masalah ketahanlasakan konkrit. MK juga tidak tahan kepada persekitaran bermagnesium sulfat dan pada suhu yang tinggi. Oleh itu skop penggunaan adunan penduaan POFA dan MK di dalam industri pembinaan adalah terhad. Walau bagaimana pun, penggunaan bersama bahan ini secara sinergi dalam adunan pertigaan mempunyai potensi mengatasi kelemahan-kelemahan tersebut. Oleh yang demikian kajian ini dijalankan untuk mengkaji kesan gabungan POFA dan MK terhadap sifat simen motar. Sebanyak 17 jenis campuran adunan penduaan dan pertigaan POFA dan MK yang berbeza dengan penggantian sehingga 30% mengikut berat dan nisbah air-simen 0.55 telah dibuat. Campuran yang optima adunan pertigaan berpandukan peningkatan kekuatan dan pengurangan keporosan telah dipilih untuk kajian selanjutnya. Sifat campuran optima simen motar semasa basah dikaji dari aspek konsistensi, masa set, kebolehkerjaan dan peningkatan suhu. Sementara dalam keadaan keras, kekuatan mampatan, tahap serapan dan mikrostruktur adunan diuji. Ketahanlasakan diuji terhadap rintangan asid sulfurik, serangan sulfat dan pada suhu tinggi. Ciri-ciri pelekat juga dikaji dan pematuhannya kepada piawaian yang berkaitan dibuktikan. Keputusan menunjukkan campuran pertigaan optima adalah 10% POFA dan 10% MK. Aduan pertigaan didapati meningkatkan kebolehkerjaan mortar dengan penggunaan superpemplastik yang sedikit berbanding adunan penduaan MK. Kajian menunjukkan adunan penduaan MK meningkatkan suhu separuh adiabatik sebanyak 7% berbanding campuran simen (OPC) manakala adunan pertigaan menunjukkan pengurangan sebanyak 4%. Selain daripada itu, adunan pertigaan bukan sahaja mengatasi masalah kekuatan awal yang rendah bagi adunan penduaan POFA tetapi meningkatkan kekuatan jangka panjang berbanding adunan penduaan MK dan POFA. Data TGA dan XRD membuktikan peningkatan kekuatan awal adunan pertigaan disebabkan oleh sifat pozolana MK. Tambahan pula adunan pertigaan memperlihatkan prestasi yang lebih baik berbanding adunan penduaan dan campuran OPC terhadap serangan sulfat dan suhu yang tinggi. Secara keseluruhan adunan pertigaan yang optima OPC, MK dan POFA menunjukkan prestasi yang lebih baik dan boleh digunakan dalam pembinaan terutamanya bagi mengatasi kelemahan adunan penduaan MK dan POFA. Kombinasi POFA dan MK bukan sahaja dapat membangunkan bahan yang mesra alam sekitar tetapi juga dapat mengurangkan kadar pembebasan CO_2.

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LIST OF SYMBOLS

A	-	Cross-sectional area of specimen
Å	-	Angstrom
^oC	-	Degree Celsius
d	-	Density of water
D_{50}	-	Median particle size
f_m	-	Compressive strength
Hr	-	Hour(s)
H^+	-	Hydrogen Ion
I	-	Water absorption
Kv	-	Kilovolt
KBr	-	Potassium bromide
MPa	-	Mega Pascal
M_i	-	Initial mass of specimen
M_n	-	Mass of specimen at n days
m_t	-	Change in mass due to water absorption of specimen
n	-	Age in days
P	-	Total maximum load
P_r	-	Porosity
s	-	Second

S	-	Sorptivity
S_f	-	Flexural strength
T	-	Time
v_1, v_2	-	symmetric stretches vibration mode
v_3	-	antisymmetric stretches vibration mode
vp	-	variable-pressure
W_A	-	Weight of saturated specimen in air
W_D	-	Weight of oven-dried specimen
W_w	-	Weight of saturated specimen in water
μm	-	Micrometer
$\geq$	-	Greater than or equal to
$\leq$	-	less than or equal to
θ	-	Theta
λ	-	Lambda
Ø	-	Diameter

LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing and Materials
BA	-	Bagasse Ash
BET	-	Brunauer Emmett Teller
BS	-	British Standard
$CaCO_3$	-	Calcium Carbonate
$Ca(OH)_2$	-	Calcium Hydroxide
CSH	-	Calcium Silicate Hydrate
DSC	-	Differential Scanning
DTA	-	Differential Thermal Analysis
FESEM	-	Field Emission Scanning Electron Microscopy
FA	-	Fly Ash
FTIR	-	Fourier Transformed Infrared
GGBS	-	Ground Granulated Blast Slag
GLM	-	General Linear Model
GPOFA	-	Ground Palm Oil Fuel Ash
HCFA	-	High Calcium Fly Ash
HCl	-	Hydrochloric Acid
HSGC	-	High Strength Green Concrete
LOI	-	Loss on Ignition

MK	-	Metakaolin
N_2	-	Nitrogen
OPC	-	Ordinary Portland Cement
PC	-	Portland Cement
PFA	-	Pulverized Fly Ash
POFA	-	Palm Oil Fuel Ash
RHA	-	Rice Husk Ash
RILEM	-	International Union of Laboratories and Experts in Construction Materials, Systems and Structures
SCC	-	Self-Compacting Concrete
SF	-	Silica Fume
sp	-	Superplasticizer
TGA	-	Thermogravimetric Analysis
UPV	-	Ultrasonic Pulse Velocity
w/b	-	Water to Binder Ratio
XRD	-	X-Ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background to the Study

Concrete is second to water as the most widely used material in the globe (Mehta and Monteiro, 2006). The superior niche of concrete in the construction industry over other materials, such as steel and timber is undoubtedly attributed to its strength, durability, versatility nature and relative cheapness. However, concrete constitutes Portland cement as its most important component because of its binding characteristic but the key contributor to the embodied CO_2.

Cement production contributes to global warming due to CO_2 emission. For every one ton of Portland cement produced, an average of 0.87 ton of CO_2 is emitted to the atmosphere (Saca and Georgescu, 2014). In fact, due to the bulky annual global cement production output; which had reached 3737 metric ton (Mt) in the year 2012 and projected to increase to 4368 Mt by the year 2016 (Armstrong, 2013), cement production accounts for 5 to 8% of the total anthropogenic CO_2 emitted in the planet (Fernández-Carrasco *et al.*, 2012). However, the use of pozzolanic materials to partially substitute Portland cement in concrete has been recognized as one of the sustainable approaches for reducing CO_2 emission that arise from cement production (Saca and Georgescu, 2014).

Pozzolanic materials are siliceous, and aluminous materials that react with calcium hydroxide liberated during cement hydration to produce secondary cementitious compounds that enhance strength and durability of concrete (Grist *et al.*, 2013). The commonly used pozzolanic materials include; fly ash, silica fume, slag, metakaolin (MK), rice husk ash, palm oil fuel ash (POFA) and natural pozzolans. Apart from the benefits of reducing CO_2 emission as well as performance improvement of concrete, the utilization of these pozzolanic materials; which are mostly industrial wastes, helps in reducing not only the environmental burden related to their disposal but also the cost of concrete production. In palm oil producing countries such as Malaysia, Indonesia and Thailand, POFA is generated in huge quantity from palm oil mills as waste with a significant amount of the ash disposed off to landfills. For instance, according to Chandara *et al.* (2012), in 2009 alone, up to 3 million tons of POFA was generated in Malaysia and with the growth of palm oil industry, more waste generation should be anticipated and hence increase in volume of waste to landfills.

In an attempt to reduce the environmental impact of disposing POFA to landfills and cement production, as well as to produce affordable but high-performance cement based materials, considerable effort of research into the use of POFA as pozzolanic material is currently underway. POFA has been found to be a useful pozzolanic material that improves the performance of concrete (Awal and Hussin, 1997). On the other hand, due to the global abundance of kaolin reserve coupled with the prospective shortage of traditional pozzolanic materials (fly ash, slag and silica fume), the use of MK as pozzolan has also been investigated (Vejmelkova *et al.*, 2010). Metakaolin is produced from the thermal treatment of kaolinite clay or paper sludge at a controlled temperature of 500 to 800^oC (Frías *et al.*, 2008a and Kadri *et al.*, 2011).The incorporation of MK in the production of concrete enhances its mechanical properties and durability performance (Siddique and Klaus, 2009; Moser *et al.*, 2010; Shekarchi *et al.*, 2010). Moreover, the use of MK is also environmentally friendly with respect to reduction in CO_2 emission to the atmosphere by reducing the Portland cement consumption (Güneyisi *et al.*, 2008; Mermerdaş *et al.*, 2012).

However, due to the variability in the properties of pozzolanic materials and their different reaction patterns with the cement hydration products, the influence of each of these materials on the properties of concrete varies. While some materials are deficient, others exhibit contrasting influences on the properties of concrete. These limit the extent to which each pozzolanic material can substitute cement to achieve the desired concrete property. In view of the need to increase the level of alternatives to cement and also produce concrete of high performance, the use of ternary blend (combining two pozzolanic materials to partially replace Portland cement) utilizing their synergistic interactions has currently become a common practice. To date, intensive researches on the use of ternary blends such as MK and fly ash by Moser *et al.* (2010), MK and slag by Khatib and Hibbert (2005), and POFA and fly ash by Rukzon and Chindaprasirt (2009) have been conducted. Yet there has not been detailed study on the ternary blend of MK and POFA. It is, therefore, the intent of this study to investigate the effect of a ternary blend of MK and POFA on the properties of cement mortar.

1.2 Research Problem

Intensive researches on the use of pozzolanic materials have been undertaken over an extended period, and the benefits of using binary blends of POFA and metakaolin (MK) are widely established (Awal and Hussin, 1997; Bai *et al.*, 1999; Kroehong *et al.*, 2011; Kadri *et al.*, 2011; Megat Johari *et al.*, 2012; Cassagnabère *et al.*, 2013). It is known that MK improves early strength development, microstructures and some durability properties. But it was shown to reduce workability (Bai *et al.*, 1999; Cassagnabère *et al.*, 2013) as well as to increase heat of hydration (Bai and Wild, 2002; Kadri *et al.*, 2011) which could be detrimental to the durability of mainly mass concrete. Moreover, metakaolin was found to be deficient in resisting magnesium sulphate attack (Lee *et al.*, 2005) and elevated temperatures (above 400 ^{o}C) (Poon *et al.*, 2003; Nadeem *et al.*, 2014). On one hand, the use of POFA showed improvement in reducing heat of hydration, in resistance to sulphate attack and elevated temperatures but on the other hand, it is deficient in early strength development (Tangchirapat *et al.*, 2007). The deficiencies of these materials when singly used may restrict the scope of their use in construction industry. For instance, due to the slow strength development characteristic of POFA in concrete, POFA may not be a suitable material in precast industry or where early strength development is paramount. Also, due to the increase in heat of hydration, the use of metakaolin in mass concrete may be a disadvantage. While there are abundant information on the effects of POFA and MK as binary, limited information exists on their combining influence in ternary blends. Therefore, the potential improvement in the properties of the mortar due to the combining effect of POFA and MK through their synergistic interaction needs to be studied. The study may consequently lead to the development of environmental friendly cement-based materials with a wider scope of applications in construction industry.

1.3 Aim and Objectives

The aim of the study is to evaluate the combined effects of metakaolin (MK) and palm oil fuel ash (POFA) as pozzolanic materials on the performance of cement mortar. The aim is to be achieved through the following objectives:

(i) To characterize the physical and chemical properties of POFA and MK used in the study.

(ii) To determine the optimum replacement levels of POFA and MK ternary blended cement mortar.

(iii) To evaluate the fresh and hardened properties as well as microstructures of the optimized POFA and MK ternary blended cement mortar.

(iv) To investigate the durability performance of the optimized POFA and MK ternary blended cement mortar exposed to hostile environments.

1.4 Scope of the Study

This study was purely experimental in nature and it focused on examining the effects of combining MK and POFA on the properties of cement mortar. In the beginning, material characterizations such as physical properties, chemical and mineralogical compositions that are essential for explaining how these may influence the properties tested in mortar were carried out. In order to obtain the optimum ternary blend, tests on the properties of various binary and ternary blended mixes made with varying replacement levels limited up to 30% by weight and a constant water binder ratio of 0.55 were also performed. The properties considered were mortar flow as well as compressive strength, flexural strength and porosity after water-cured for up to 90 days. The established optimum ternary blend was used for the detail investigations on the fresh and hardened properties, microstructures and durability. However, for the detailed study, different water to binder ratios of 0.55 and 0.35 were used for the production of mortar.

At fresh state, the setting times, standard consistency, flowability and temperature of hydration of the optimized ternary blend were investigated while at the hardened state only the compressive strength, sorptivity, microstructures and durability properties for up to 6 months were considered. The durability was assessed in terms of resistance to magnesium and sodium sulphate attack, sulphuric acid attack and elevated temperature. The microstructure was evaluated using the Thermogravimetry Analysis (TGA), X-Ray Diffraction (XRD), Fourier Transformed Infrared (FTIR) and Field Emission Scanning Electron Microscope (FESEM) techniques.

The series of tests conducted in this study are based on the procedures of British Standards (BS), American Society for Testing and Materials (ASTM), International Union of Laboratories and Experts in Construction Materials, Systems and Structures (RILEM) and adopted methods in the literature reviewed. As these are well established, this enabled comparison with related studies, with information on their precision known.

In addition to compressive strength and sorptivity, the amount of $Ca(OH)_2$ depleted was used as a variable for assessing the microstructure of the optimized specimens at its hardened state. Meanwhile, residual compressive strength, residual mass and expansion were used as parameters for measuring the resistance of specimens to sulphuric acid, magnesium and sulphate attacks. The resistance to elevated temperature was however, measured in terms of residual compressive strength and ultrasonic pulse velocity (UPV). All the results were analyzed and presented in the forms of graph and output from the XRD, TG/DTG and FTIR tests. The findings were referred and compared with similar previous studies.

1.5 Significance of the Study

As this research study was aimed at gathering information on the use of ternary blend from a systematic investigation, it can be useful for the development of standard specifications for ternary blended system which are essential for their practical application. It can also contribute to the development of environmental friendly material that has a wide range of applications in construction industry. This will be more beneficial for the palm oil producing countries like Malaysia, as waste (POFA) from palm oil mills can be put to good use in addition to the environmental benefit of solving disposal problems of POFA to landfills. The outcome of the study can also provide the basis for further researches for better understanding of the behaviour of a ternary blend of POFA and MK, which will ultimately increase substance to the pool of existing knowledge.

1.6 Thesis Organisation

Chapter 1 provides a general appraisal and the rationale for conducting this research. Also, concise description of background problem, aim and objectives, scope and limitations, and significance of study are presented in this chapter.

Chapter 2 describes the properties of Portland cement and pozzolanic materials. The chapter also presents the review of previous studies on the effect of binary and ternary blends on the properties of paste, mortar and concrete. Although, there few or no literature available on the ternary blend of palm oil fuel ash and metakaolin, the benefits of ternary blends of other pozzolanic materials such as fly ash and silica fume over their binary counterparts are also reviewed.

Chapter 3 provides a detailed account of the materials and sample preparation as well as the test methods used during the experimental work. Subsequent chapters then present the results of these tests.

Chapter 4 examines the physical and chemical properties of Portland cement, palm oil fuel ash and metakaolin. The results of the optimization of calcination temperature of kaolin to produce metakaolin are also presented. In addition, the results and discussions on the optimization of the ternary blend used for the detailed study are presented in this chapter.

Chapter 5 covers the results and discussions on the fresh and hardened properties, and microstructures of the optimized ternary blended mortar. The properties of mortar studied in its fresh state include consistency, setting times, workability/flow, and adiabatic temperature rise. At the hardened state, the characteristics of mortar considered were compressive strength and sorptivity. The relationship between sorptivity and compressive strength of mortar was also highlighted in order to establish a correlation. Furthermore, the results of the microstructure of blended mortars using the thermogravimetry Analysis (TGA) and X-ray diffraction (XRD) are discussed.

Chapter 6 describes the results and discussions of the chemical attack and elevated temperature tests on the blended mortars. Fourier transform infrared (FTIR), X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) results of specimens after the attack are likewise presented in this chapter.

Chapter 7 shows the overall conclusions from this study and recommendations for further researches.

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