POZZOLANIC REACTIVITY OF CHEMICALLY PRE-TREATED PALM OIL CLINKER POWDER (POCP) WITH HYDROCHLORIC ACID

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Pengurusan sisa kelapa sawit yang tidak mantap dan efektif menjadi punca utama sisa kelapa sawit dibuang dan ditinggalkan di kawasan berhampiran ladang, secara tidak langsung menjadi ancaman serius kepada alam sekitar melalui potensi risiko pencemaran. Salah satu sisa kelapa sawit iaitu klinker minyak sawit (POC) tidak menjadi pilihan utama sebagai pengisi dan pengganti bahan untuk simen dalam pengeluaran konkrit akibat daripada rupa bentuknya yang berliang dan kasar, kawasan permukaan yang rendah, dan kurang kereaktifan bahan. Berikutan itu, kajian ini dilakukan untuk menyiasat kaedah meningkatkan kereaktifan pozolanik daripada POCP melalui kaedah mekanikal dan kimia pra-rawatan. Bagi rawatan mekanikal, POC telah dikisar menjadi bentuk yang lebih halus supaya boleh melepasi 300 mikron saiz ayakan dan dipanggil sebagai serbuk klinker kelapa sawit (POCP) dalam teks utama. Impregnasi POCP dalam asid hidroklorik (HCl) bertujuan untuk mengeluarkan sisa karbon yang tidak terbakar dan elemen-elemen kimia yang berlebihan, di samping meningkatkan kereaktifan pozolanik bahan. Walau bagaimanapun, parameter optimum untuk kimia pra-rawatan bagi POCP belum pernah dikaji lagi. Oleh itu, kajian ini bercadang untuk mengkaji kesan kepekatan HCl dan tempoh pemanasan kepada penyingkiran sisa karbon yang tidak terbakar dan kesan logam alkali oksida dalam proses pra-rawatan. Selain itu, kajian ini dijalankan untuk menganalisis kereaktifan pozolanik secara kimia pra-dirawat POCP dan perkembangannya dalam medium bersimen. Kombinasi parameter yang berbeza telah disiasat bagi mendapatkan parameter yang paling optimum berdasarkan penyingkiran yang lebih tinggi daripada kekotoran dan kenaikan SiO_2 dalam POCP. Analisis ini dilakukan melalui X-Ray pendarfluor analisis (XRF). Berdasarkan dapatan kajian, 0.1m - 1h dipilih sebagai parameter optimum dan digunakan seterusnya untuk menganalisis sifat-sifat POCP yang lain. Ujian penyerapan nitrogen, taburan saiz zarah, analisis mineralography, dan pengimejan mikrostruktur POCP telah dijalankan untuk mengkaji ciri-ciri fizikal bahan sumber. Sementara itu, kereaktifan pozolanik daripada POCP dikaji melalui ujian Chapelle, haba penghidratan, dan analisis termogravimetri. Kesan POCP sebagai pozolan juga diukur dengan menggabungkannya dalam campuran mortar. Berdasarkan dapatan kajian, POCP pra-rawatan 7.5 telah dipilih sebagai pengganti POCP yang paling sesuai untuk pengeluaran mortar mampan kerana kekuatan mampannya yang standing dengan mortar kawalan . Analisis statistik telah dijalankan untuk membuktikan keputusan kajian yang diperolehi dengan menggunakan kesimpulan matematik bagi memperolehi data yang lebih tepat. Mengikut data statistic menggunakan kaedah Chapelle, perbandingan menggunakan 0.1 M-1h dan 0.5 M-1h tidak mempunyai perbezaan yang ketara dari data min dan kesalahan piawaian. Justeru, pemilihan 0.1 M-1h sebagai parameter optimum dapat disokong melalui keputusan ini. Secara ringkas, pra-rawatan POCP dengan menggunakan parameter optimum (kepekatan HCl dan tempoh pemanasan) telah terbukti untuk meningkatkan kereaktifan pozolanik bahan dengan menaiktaraf sifat kimia dan fizikalnya.

ABSTRACT

Revolutionary act on modern cement production is marking a positive finding on the utilization of industrial solid waste and agro-based waste as cement replacement material (CRM) which is rich in silica (SiO_2) and alumina (Al_2O_3). Palm oil wastes are commonly dumped and abandoned in the vicinity due to an improper waste management and could become a serious threat to the environment through the potential risk of pollution. One of its by-product i.e. palm oil clinker (POC) is not a favourable choice as a filler and replacement material for cement in concrete production due to its coarse form, low surface area, and less reactivity. Besides, POC contains impurities of organic carbon and traces of alkali metal oxides and several transition elements, which could inhibit the reaction of Si precursors with Portlandite during hydration process. This study investigated the method of improving the pozzolanic reactivity of POCP through mechanical and chemical pre-treatment. In mechanical pre-treatment, POC was ground to a finer particle passing 300 µm and would be referred as palm oil clinker (POCP) in the main text. Impregnation of POCP particles in hydrochloric acid (HCl) solution was supposed to remove the unburned carbon and unnecessarily elements, besides enhancing the pozzolanic reactivity of the material. However, the optimum parameter for the chemical pre-treatment of POCP has not been fully investigated. Therefore, this research is proposed to investigate the effect of HCl concentration and heating period on the removal of unburned carbon and traces of alkali metals oxides for pre-treatment process. Besides, this study was conducted to analyse the pozzolanic reactivity of chemically pre-treated POCP and the performances in cementitious environment. Different combinations of parameters were investigated to obtain the most optimum parameter based on the higher removal of impurities and increment in SiO_2 proportion in POCP. The analysis was performed via X-Ray Fluorescence (XRF) analysis. Based on the results, 0.1M – 1h was chosen as the optimum parameter and would be used for the subsequence analysis. Nitrogen adsorption test, particle size distribution, mineralography analysis, and microstructure imaging of POCP were conducted to study the physical characteristic of the source material. Meanwhile, pozzolanic reactivity of POCP was studied via Chapelle test, heat of hydration, and Thermogravimetry analysis. The effect of POCP as a pozzolan was also measured by incorporating it in mortar mixtures. Different percentage of POCP inclusion were adopted and control specimen was used as the benchmark for the entire test. Inclusion of chemically pre-treated POCP in cementitious medium has marked a positive outcome in compressive strength and porosity results. Based on the results, treated 7.5 was chosen as the most applicable POCP replacement for the production of sustainable mortar due to the high strength (27.69 MPa), on par with control mortar. Statistical analysis was conducted to justify the obtained results using mathematical inferences for more solid conclusion and the accuracy of the data sets. According to the statistical analysis for Chapelle method, 0.1 M-1h was compared with 0.5 M-2h on the mean and standard error of the data. 0.1 M-1h has more reliable and consistent data, thus strengthen the decision of using 0.1 M-1h as optimum combination parameter for pre-treatment basis. In a nutshell, chemically pre-treat POCP by using optimum parameters (concentration of HCl and heating period) has proven to increase and enhance the pozzolanic reactivity of the material by improving its chemical and physical properties.

Keywords: Palm oil clinker powder, chemical pre-treatment, cement replacement material, strength, pozzolanic reactivity

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

Al_2O_3	Aluminum Oxide
C_2S	Dicalcium Silicate
C_3A	Tricalcium Aluminate
C_3S	Tricalcium Silicate
C_4AF	Tetracalcium AluminoFerrite
$Ca(OH)_2$	Calcium Hydroxide/ limewater
CaO	Calcium Oxide
C-A-S-H	Calcium Aluminate Sulfate Hydrates
CH	Calcium Hydrates
CO_2	Carbon Dioxide
C-S-H	Calcium Silicate Hydrates
FA	Fly Ash
Fe_2O_3	Iron (III) Oxide
H_2O	Water
HCl	Hydrochloric Acid
RHA	Rice Husk Ash
SiO_2	Silicon Dioxide
SO_3	Sulphur Oxide

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BET	Brunauer, Emmett and Tellerare
CRM	Cement Replacement Material
EFB	Empty Fruit Bunch
FESEM	Field-Emission Scanning Electron Microscope
MIP	Mercury Intrusion Porosimetry
MPOB	Malaysian Palm Oil Board
OPF	Oil Palm Fond
OPS	Oil Palm Shell
OPT	Oil Palm Trunk
POC	Palm Oil Clinker
POCC	Palm Oil Clinker Concrete
POCP	Palm Oil Clinker Powder
POFA	Palm Oil Fuel Ash
POME	Palm Oil Mill Effluent
PPF	Palm Pressed Fibre
R&D	Research & Development
SAI	Strength Activity Index
SCLWC	Self-compacting Lightweight Concrete
TGA	Thermogravimetric analysis
TPOFA	Treated Palm Oil Fuel Ash
UPV	Ultrasonic Pulse Velocity
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

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