

STUDY ON THE SOLUBLE SILICA FROM
PALM OIL CLINKER AS PARTIAL CEMENT
REPLACEMENT MATERIAL

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I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. Thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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ABSTRAK

Perkembangan industri minyak kelapa sawit dan industri pembinaan masing-masing membawa kepada isu pembuangan klinker kelapa sawit (POC) dan pelepasan karbon dioksida. Disebabkan POC mempunyai kandungan silika yang tinggi, telah timbul satu idea untuk mengekstrak silika larut dari POC dan menggantikan sebahagian simen dengan silika larut dalam mortar. Oleh itu, kajian ini bertujuan untuk mengkaji kesesuaian untuk menggantikan sebahagian simen dalam mortar dengan silika larut yang diekstrak dari POC, di mana keputusan disokong dengan mengenalpasti parameter yang lebih sesuai untuk pengekstrakan silika larut dan penilaian mortar dari segi kekuatan mampatan dan porositi. POC telah dikisar untuk dijadikan serbuk yang dipanggil serbuk klinker kelapa sawit (POCP) dan kemudian dirawat dengan asid hidroklorik 0.1M. POCP yang telah dilarut dengan asid hidroklorik kemudian digunakan untuk proses pengekstrakan silika larut. Kaedah Laine telah digunakan untuk mengekstrak silika larut dari serbuk klinker kelapa sawit, POCP yang telah melalui proses pengurusan asid. Tempoh masa yang sesuai untuk proses penyulingan dan jumlah POCP diperlukan untuk eksperimen pengekstrakan silika larut telah dikenalpasti untuk menyediakan silika larut yang berkandungan tinggi. Berdasarkan analisis X-Ray Fluorescence (XRF), didapati bahawa dengan menggunakan masa penyulingan yang lebih lama dan jumlah POCP yang lebih tinggi dalam proses pengekstrakan silika, silika larut yang lebih pekat dengan kandungan silika dapat dihasilkan. Didapati bahawa penggunaan 480g POCP dan tempoh masa 12 jam penyulingan dalam eksperimen pengekstrakan berjaya menghasilkan 53.50% silika terlarut, iaitu kandungan tertinggi antara semua eksperimen percubaan yang telah dilakukan. Silika larut yang dikumpul itu telah digunakan untuk menggantikan simen pada 0%, 2.5%, 5% dan 7.5%. Untuk menilai kualiti mortar yang mengandungi silika larut, ujian-ujian kekuatan mampatan dan porositi telah dijalankan. Didapati bahawa semua mortar berasaskan silika mempunyai sifat pengerasan yang cepat, di mana kekuatan mampatan yang tinggi telah direkodkan pada usia awal, disebabkan reaksi pozzolanic yang lebih awal. Semasa usia awal, kekuatan mampatan yang tertinggi, 16.90MPa telah direkodkan untuk mortar yang 5% simen telah digantikan oleh silika larut, di mana kekuatan mampatannya adalah 5.24% dan 6.20% lebih tinggi daripada mortar rujukan pada hari ke-3 dan hari ke-28. Walau bagaimanapun, disebabkan perkembangan kekuatan yang perlahan ditunjukkan oleh semua mortar berasaskan silika selepas usia awal, kekuatan yang disasarkan gagal dicapai pada hari ke-28 dan ke-56. Semua mortar berasaskan silika memperolehi porositi yang tinggi disebabkan oleh kekurangan jumlah simen dalam mortar. Pengurangan porositi selepas usia awal juga perlahan, di mana porositi S2.5, S5.0 dan S7.5 hanya menurun sebanyak 1.7%, 0.8% dan 1.7%, masing-masing daripada hari ke-7 hingga hari ke-28. Hubungan antara mampatan kekuatan dan porositi mortar telahpun dikaji. Telah didapati bahawa semasa usia awal, kekuatan mampatan tidak bergantung kepada porositi mortar; Walau bagaimanapun, porositi mortar menjadi faktor separa yang mempengaruhi kekuatan mampatan semasa umur kemudian. Oleh itu, porositi yang tinggi telah menjadi satu faktor utama yang menyebabkan perkembangan kekuatan mampatan perlahan di hari ke-28 dan ke-56. Disebabkan pengurangan jumlah simen dalam mortar berasaskan silika akan membawa kepada porositi yang tinggi dan kekuatan kemudian yang rendah, penggantian simen dengan silika larut dalam mortar tidak disyorkan. Walaubagaimanapun, disebabkan sifat-sifat kekuatan awal yang dipamerkan oleh mortar berasaskan silika larut, potensi silika yang larut untuk digunakan sebagai bahan tambahan untuk pengerasan pantas telah dibuktikan melalui kajian ini.

ABSTRACT

The growth of palm oil industry and construction industry have brought the issues of abundant dumping of palm oil clinker (POC) and carbon dioxide emission, respectively. Taking advantage of the high silica content in POC, the idea of extracting soluble silica from POC and using it to partially replace cement in mortar has been initiated. Therefore, this study aimed to investigate the feasibility of partially replacing cement in mortar with soluble silica extracted from POC, where the decision was supported by finding the suitable parameters for soluble silica extraction and evaluation of mortar in terms of compressive strength and porosity. The POC was ground into powder form called palm oil clinker powder (POCP) and then pre-treated with 0.1M hydrochloric acid. The acid-leached POCP was then used for soluble silica extraction. The Laine's Method that consists of the water reflux and distillation steps was adopted to extract the soluble silica from the acid leached palm oil clinker powder (POCP). Based on the X-Ray Fluorescence (XRF) analyses, it was found that using longer distillation time and higher amount of POCP for soluble silica extraction resulted in the extracted soluble silica solution of higher silica concentration. The use of 480g of POCP and 12 hours of distillation in the extraction experiment resulted in 53.50% of dissolved silica, which was the highest gain among the trial experiments. The extracted soluble silica was used to replace cement at 0%, 2.5%, 5% and 7.5%. To evaluate the influences of replacement with soluble silica, compressive strength and porosity tests were conducted. It was found that all soluble silica-based mortars possessed rapid hardening properties, where high compressive strength was recorded during early age, due to the earlier taken place pozzolanic reaction. During early age, the highest compressive strength of 16.90MPa was recorded for the mortar batch with cement replaced by 5% of soluble silica (S5.0), where its compressive strength was 5.24% and 6.20% higher than that of reference mortar at day 3 and day 7, respectively. However, due to the slow strength development of all soluble silica-based mortars after early age, the targeted reference strength was failed to be achieved at day 28 and 56. Result of high porosity was obtained for all soluble silica-based mortars due to the reduction of cement amount. The reduction of porosity after early age was also slow, where the porosity of mortar batches with cement replaced by 2.5%, 5.0% and 7.5% only decreased by 1.7%, 0.8% and 1.7%, respectively from day 7 to day 28. The correlation between compressive strength and porosity of mortar was studied. It was found that during early age, the mortar compressive strength was independent to the porosity; however, mortar porosity became partial factor affecting compressive strength during later age. Since the reduction of cement amount in soluble silica-based mortar leads to high porosity and low later strength, reducing cement amount in mortar and replacing it with soluble silica is not recommended; however, due to the early strength properties displayed by soluble silica-based mortar, the potential of soluble silica to be used as additive for rapid hardening could further be explored.

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LIST OF ABBREVIATIONS

ASR	Alkali Silica Reaction
CRM	Cement Replacement Material
C-S-H	Calcium Silicate Hydrate
C ₂ S	Dicalcium Silicate
C ₃ S	Tricalcium Silicate
EGH ₂	Ethylene Glycol
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier-Transform Infrared Spectroscopy
GGBS	Ground Granulated Blastfurnace Slag
OPC	Ordinary Portland Cement
POC	Palm Oil Clinker
POCP	Palm Oil Clinker Powder
POFA	Palm Oil Fuel Ash
RHA	Rice Husk Ash
SAI	Strength Activity Index
SCBA	Sugarcane Bagasse Ash
TGA	Thermogravimetry Analysis
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
EGH ₂	Ethylene Glycol

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Nowadays, the researchers have put a lot of efforts in finding different waste products as cement substitutes because cement has high carbon footprint. According to Rubenstein (2012), it was found that a single cement production industry accounts for around 5% of global carbon dioxide (CO₂) emissions. The current popular cement replacement materials (CRMs) such as fly ash, silica fume and ground granulated blastfurnace slag (ggbs) are all having one similar characteristic, which is containing high amount of amorphous silica (Newman & Choo, 2003). The amorphous silica in the waste material reacts with portlandite (Ca(OH)₂) in the concrete and forms a framework of calcium silicate hydrate gel (C-S-H) that can strengthen the concrete. Inspired by studies from Karim et al. (2017) and Nayakaa et al. (2018) which proved that palm oil clinker (POC), a type of waste from palm oil industry contains amorphous silica and is a potential CRM, this study proposes to use POC as a raw material for soluble silica extraction.

The process of producing C-S-H gel by reaction between reactive silica from CRM and Ca(OH)₂ in the cementitious system is called pozzolanic reaction, while the involved CRM is called pozzolan (Newman & Choo, 2003). In this study, the use of soluble silica extracted from POC powder (POCP) as soluble pozzolan is expected to possess the pozzolanic characteristic and involve actively in the pozzolanic reaction. The solubility property of silica enables reactive silica to be mixed evenly with water and dispersed uniformly in the fresh mortar, which is believed that it results in higher pozzolanic reactivity and maintained or improved fresh mix workability (Kusbiantoro et al., 2017).

In the past researches, some methods were found effective to enhance silica level and reduce the impurities content of the silica-containing waste: thermal treatment (Yuvakkumar et al., 2014) (Usman et al., 2014), and acid leaching treatment (Faizul et al., 2016) (Worathanakul et al., 2009). Prior to silica extraction from the waste, promotion of silica level and reduction of impurities level of the waste is beneficial because these determine the purity of silica extracted. Since acid leaching treatment has been popular to be applied to enhance silica content of some other wastes, this study proposes to apply acid leaching by hydrochloric acid at low concentration for POCP.

The common method that has been utilised extensively to extract silica from silica-containing materials is alkaline extraction method (Yuvakkumar et al., 2014) (Kalapathy et al., 2000) (Shelke et al., 2010). Alkali is applied as a solvent for dissolution of silica to occur because silica is highly soluble at high pH value. Hence, the liquid silica is extracted in the form of alkaline silicate solution. Besides the common alkaline extraction method, recently, a newly found silica extraction method was introduced by Richard Laine and his team which is claimed as Laine's method in this study. In Laine's method, a catalytic quantity of base is added to catalyse the reaction between silica in waste and liquid polyol to extract soluble silica in the form of alkoxy silane solution (Laine et al., 2014). In this study, Laine's extraction method is applied and the suitable parameters is determined to obtain a higher amount of dissolved silica. Laine's method instead of alkaline extraction method is applied in this study to extract silica from POC because Laine's method required a lower quantity of base to extract silica.

In order to study the feasibility of partially replacing cement with soluble silica extracted from POC, compressive strength test and porosity test are proposed because it is believed that the strength of concrete or mortar gives an overall picture of concrete or mortar quality (Neville, 2011). Meanwhile, concrete strength is highly dependent on the concrete porosity (Chen et al., 2013). The larger total volume of air voids in mortar or concrete leads to weaker mortar or concrete strength. Therefore, the changes of void volume or porosity and compressive strength of the mortar influenced by the inclusion of soluble silica in mortar are evaluated.

1.2 Problem Statement

Malaysia is the largest producer and exporter of palm oil in the world, and as a result, tonnes of waste products known as palm oil clinker or POC is produced from the palm oil refineries. The current practice to get rid of POC is by dumping it in landfill sites or incineration, which leads to environmental pollution. In fact, the POC contains a large amount of reactive silica which is very useful in many industries. Hence, the dumping of POC corresponds to the dumping of silica source. On the other hand, in construction industry, cement which is an important ingredient in concrete has always been studied to be replaced by alternative materials because it has high carbon footprint. Cement production is a significant source of global carbon dioxide (CO₂) emission, in which CO₂ is emitted in tremendous quantity from the heating of limestones and burning of fossil fuels throughout the production process. Subsequently, it leads to air pollution and global warming. Therefore, in an attempt to ameliorate these situations, this study is conducted to verify the suitability of partially replacing cement with soluble silica extracted from POC into mortar. The higher reactivity of silica in soluble form is expected to enhance the performance of cement-based mortar.

1.3 Objectives

The main objective of this study is to investigate the suitability of using soluble silica extracted from palm oil clinker (POC) as partial cement replacement material in mortar. Meanwhile, the sub-objectives of this study are as follows:

1. To determine the suitable distillation duration and amount of palm oil clinker powder used for extraction of soluble silica.
2. To attain the compressive strength and porosity of mortar containing soluble silica from palm oil clinker as a partial cement replacement material.

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