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Mechanical performance of modified cement paste made with micro-fine POFA in ammonium nitrate environment

Sadia Tasnim [a], Muhammad Ekhlasur Rahman [a,*], Raudhah Binti Ahmadi [b]

[a] Faculty of Engineering and Science, Curtin University, Malaysia
[b] Department of Civil Engineering, University Malaysia Sarawak, 94300 Samarahan, Sarawak, Malaysia

HIGHLIGHTS

- Modified cement paste have been made using micro-fine palm oil fuel ash (POFA) in this study.
- Compressive strength, Energy Dispersive X-ray (EDX) and a Scanning Electron Microscopy (SEM) test were carried out.
- It was observed that, samples replaced with 20% POFA had the best results.

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ABSTRACT

An experimental study was conducted on modified cement paste cubes in ammonium nitrate solution. The modified cement paste was made using micro-fine palm oil fuel ash (POFA) as partial cement replacement. Compressive strength, Energy Dispersive X-ray (EDX) and a Scanning Electron Microscopy (SEM) test were carried out. The 50 mm cubes were cast containing 0%, 10%, 20% and 30% of POFA as cement replacement using constant water to cement ratio of 0.4. After 28 days of water curing, 50% of samples were immersed in 20% ammonium nitrate solution and the rest of samples were kept in room temperature. It was observed that, samples replaced with 20% POFA had the highest compressive strength in ammonium nitrate solution. In addition to that, it was also observed through EDX and SEM analysis that cement replaced with 20% POFA had lower Ca/Si ratio and there were more C-S-H structures in the modified cement paste in ammonium nitrate solution. Lower Ca/Si ratio and more C-S-H structures is an indication of higher pozzolanic reactions in the paste.

1. Introduction

Agro-industries generally generates large amount of solid wastes which are usually disposed in the environment. These solid wastes are capable of causing severe pollution problem such as land, water, air pollution if it is not managed or treated properly. Palm oil industry is an agro-industry which causes the production of large amount of solid waste. Palm oil fuel ash (POFA) is a solid waste produced from the palm oil industry through the combustion of fibers, shells and bunches Sata et al. [32]. According to Din [11], there are about 429 palm oil industries in Malaysia which has an annual capacity of 102 million tonnes. POFA produced from the industry is generally black in color. Through laboratory investigation, black color POFA can be converted into grayish color through heat treatment. POFA is a pozzolanic material, which has a high percentage of alumino-silicate content [17]. Recently, the use of POFA as a cement replacement has however caught the interest of many researchers because it improves the concrete characteristics. According to Tangchirapat et al. [25], finer size POFA possess better reaction with cement paste, thus produces concrete having strength higher than coarser size POFA. Kroehong et al. [15] studied the effect of POFA on cement paste using POFA size of 15.6 and 2.1 μm. The authors concluded the paste containing POFA with a greater fineness were denser and more homogeneous which resulted in improved compressive strength of the paste. According to Tangchirapat and Jaturapitakkul, [27], the degree of fineness of POFA is very significant. When the particles are smaller, they fill the voids in the cement paste, and thus contribute to an increase in the compressive strength. Generally about 20% replacement of OPC with POFA gives better mechanical properties results such as the compressive strength [21,4,26,15].

Besides reducing the amount of solid waste in the environment, the incorporation of POFA as a cement replacement would also reduce the emission of the green houses gas. When POFA is used

* Corresponding author.
E-mail address: merahman@curtin.edu.my (M.E. Rahman).

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as a cement replacement, the total cement usage is reduced. Hence the production of carbon dioxide during cement manufacturing is also reduced. Carbon dioxide is a major green house gas responsible for the global warming. Therefore, utilization of POFA as a cement replacement would result in a cleaner environment.

Ammonium nitrate is an artificial fertilizer manufactured for the purpose of providing nutrients to plant. According to Hallaji et al. [12], ammonium nitrate is highly corrosive material which causes deterioration to concrete structures. The calcium hydroxide in the cement paste is leached out when exposed to ammonium nitrate environment causing, an increase of the permeability and porosity of the paste. According to Arafa et al. [6], as the concentration of ammonium nitrate increased, the compressive strength decreased. This decrease is due to progressive decalcification reaction which causes more calcium hydroxide to be leached out. When, concrete is exposed to ammonium nitrate environment, the degradation is occurred in two stages. The first stage is known as the decalcification phenomena where the calcium hydroxide is leached out. In the second stage, there is an increase in the volume of the cement paste due to the formation of a new compound and is known as the expansion phenomena Pepenar [18].

Therefore, based on the authors' knowledge, there has been no investigation done using micro-fine POFA as a cement replacement against ammonium nitrate environment. Considering the pozzolanic characteristic of POFA especially when it is fine, it is therefore expected that, the use of micro-fine POFA would give improvement on the mechanical properties of concrete in ammonium nitrate environment. The purpose of this research is to investigate the mechanical and micro structural characteristic of modified cement paste made with micro-fine POFA in ammonium nitrate environment. The use of micro-fine POFA in the cement paste would result in reduction of calcium hydroxide content hence it will be less prone to ammonium nitrate attack.

2. Experimental program

2.1. Materials

2.1.1. Cement

Ordinary Portland cement was used in this research. It was obtained from a local manufacturer, Cahaya Mata Sarawak (CMS) Sdn. Bhd. The cement fulfilled the requirements set under ASTM C-150 [7] Type 1 Ordinary Portland Cement (OPC).

2.1.2. Palm oil fuel ash (POFA)

Palm oil fuel ash (POFA) is a solid waste of the palm oil mill manufacturing process. The POFA used in this study was collected from a local palm oil mill in Kuching, Malaysia. In order to improve its reactivity, the POFA was further treated. The raw, black POFA obtained from the palm oil mill had a median particle size (d_{50}) of 161.92 μm. The POFA was burnt using a furnace at 600 °C for one hour to remove any remaining carbon. The POFA was then ground using a grinder operating at 25,000 rpm for ten minutes to produce POFA particles smaller than 10 μm as known as treated POFA as well as micro-fine POFA.

2.1.3. Ammonium nitrate

Ammonium nitrate was obtained in pellet form the local market. A 20% Ammonium nitrate solution was prepared by mixing the pellets with water. After 28 days of water curing, the 50 mm cube samples were immersed in the 20% ammonium nitrate solution for 90 days. The container holding the samples and solution was closed in order to keep the concentration constant. The solution was changed at a regular interval of 30 days to maintain the same effect.

2.2. Mix propotion

Table 1 describes the mix proportion used in this study. Cement was replaced at rate of 0%, 10%, 20%, and 30% of ground POFA having a median particle (d_{50}) size of below 10 μm. A constant water to cement ratio of 0.4 was used for all the mix. Using this mix propotion, 50 mm cement cubes were casted. Table 2 shows the curing conditions. The workability of the each of the samples was omitted due to the fact that workability is a test usually carried out for concrete. For this research, only cement paste was used, therefore workability of the samples were not carried out. Consequently, it was found out according to Al-mulali et al. [7], POFA replacement and percentage level have influence on the concrete workability.

3. Test procedures

3.1. Physical properties, chemical compositions and micro-structure properties of POFA

The physical properties consisted of median particles (d_{50}) examined through a particle-size analyzer (CILAS 1090 Laser Particle Size Analyzer), a specific gravity test in accordance to ASTM D-854 [2] standards and a specific surface area test through the particle size analyzer (CILAS 1090 Laser Particle Size Analyzer). The chemical composition was determined using X-ray Fluorescences (XRF) technique. Consequently, Scanning Electron Microscopy in combination with Energy Dispersive X-ray Spectroscopy (SEM/EDX) was utilized to determine the microscopic image and element composition in POFA and OPC.

3.2. Compressive strength test

The compressive strength test of 50 mm cubes were carried out. Compressive strength test was carried out in accordance with BS1881: Part 116: 1983 Standards [8]. Five samples were tested and an average result was calculated. The calibrated compression machine was used for a 50 mm cube test at a loading rate of 700 N/s., which was within a loading range between 0.2 $N/mm^2/s$ and 0.4 $N/mm^2/s$.

3.3. Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) test

A scanning electron microscope (SEM) is a special type of microscope, which produces images of samples by scanning the samples. The electrons connects with the atoms in the sample and produce signals regarding the sample's composition and surface topography. The element analysis and chemical characterization of a sample is determined through EDX technique. The analysis is accomplished by the interaction of some source of X-ray excitation and sample.

In this research, SEM was carried out on modified cement pastes with POFA using a SEM machine. 10 mm samples were placed on a double-sided adhesive conductive carbon tape to prevent the scattering of loose particles. The samples were then coated with gold to increase the electrical conductivity in the samples. Several SEM images were made of different aspects of the sample. The SEM machine was set to produce images at 10 μm and 1 μm.

EDX analysis was carried out in the same machine as the SEM. After producing the SEM images, the samples were further analysed in the EDX test. Each sample was analysed for 10 min to determine all elements present in the samples.