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Research Article

Sustainable Nanopozzolan Modified Cement: Characterizations and Morphology of Calcium Silicate Hydrate during Hydration

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There are environmental and sustainable benefits of partially replacing cement with industrial by-products or synthetic materials in cement based products. Since microstructural behaviours of cement based products are the crucial parameters that govern their sustainability and durability, this study investigates the microstructural comparison between two different types of cement replacements as nanopozzolan modified cement (NPMC) in cement based product by focusing on the evidence of pozzolanic reactivity in corroboration with physical and mechanical properties. Characterization and morphology techniques using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDS), and scanning electron microscopy (SEM) were carried out to assess the pozzolanic reactivity of cement paste modified with the combination of nano- and micro silica as NPMC in comparison to unmodified cement paste (UCP) of 0.5 water to cement ratio (w/c). Results were then substantiated with compressive strength (CS) results as mechanical property. Results of this study showed clear evidence of pozzolanicity for all samples with varying reactivity with NPMC being the most reactive.

1. Introduction

As the fourth largest consumer amongst Southeast Asia's countries, Malaysia consumed 17 million tons of cement by the end of 2011 [1]. With the high demand, large scale production is needed for its satisfaction that consequently releases more carbon dioxide (CO_2) that causes greenhouse effects. Cement replacement for cement based materials is one of the options to meet the increasing sustainability, green technology demands, and requirement in construction industry since it offers two distinct benefits to the environment: it significantly reduces the amount of CO_2 released into the atmosphere and minimizes massive landfill disposal. Therefore interest in cement research is moving towards finding viable whole or partial cement replacement like using industrial wastes such as microsilica (MS) and nanoscale materials such as nanosilica (NS). In addition, recently, research and development (R&D) on cement based material is of interest to find new cement supplementary materials in nanoscale. For Malaysia, nanotechnology R&D was started by the government in 2001 and categorized as a strategic research (SR) program under IRPA in the Eighth Malaysia Plan (8MP) which spans from 2001 to 2005 and funded by the MOSTI [2].

MS and NS have one thing in common which is that they possess a pozzolanic characteristic. Pozzolanic materials have the high potential to be a substance to replace cement due to beneficial pozzolanic reaction that can improve cement based product properties. As generally known, calcium hydroxide (CH) or portlandite produced from the hydration process of cement will react with siliceous pozzolanic material to produce more C-S-H which is the substance that mainly contributes to strength for cement based material that can improve physical and mechanical properties such as reducing porosity and permeability. Hence, cement blended with pozzolanic material will have better strength and durability [3].

Table 1: Chemical and mineralogical composition of OPC.

Component, %	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O	Others	LOI
OPC	63.0	21.69	5.75	3.25	2.35	1.97	0.50	0.28	0.11	1.00

Previous studies have shown that MS modified concrete was stronger and more durable compared to conventional concrete. Due to its high pozzolanicity, MS has a very high content of amorphous silicon dioxide (SiO_2) and very fine spherical particles sizes which are the main reasons for its high pozzolanic activity [4]. The rate of the pozzolanic activity is related to the surface area of pozzolan particles whereby the higher the surface area of pozzolan particle is or the finer the particle is, the more reactive it would be [5]. Hence, the research and development for cement based material is of interest to find new cement supplementary material which has a particle size of nanoscale (10^{-9} m). Incorporation of nanomaterials in cement and concrete production can lead to major improvement in the civil infrastructure because durability and sustainability of cement based product are determined by its microstructural mass transfer in nanoscale [6].

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However there are no comparative studies on the potential performance of combination of MS and NS pozzolanic materials used as nanopozzolan modified cement (NPMC) in cement based products. Hypothetically, NPMC will produce a higher performance cement based product in terms of strength and durability due to their fineness and highly reactive pozzolanic properties. This study is an attempt to explore in that direction. Pozzolanic reactivity of MS, NS, and NPMC pastes was investigated and compared to unmodified cement paste (UCP). The efforts to investigate the potential usage of such nonconventional material which is typically of local or regional origin in cement based products will get a boost if there are systematic and comprehensive studies to quantify and characterize the performance of cement based products containing such materials and will contribute positively to waste management challenge.

2. Materials and Methods

2.1. Materials. Waste material and nanomaterial used as cement replacements in nanopozzolan modified cement (NPMC) were microsilica from Grace Construction Sdn. Bhd. with 94.5% SiO_2 and Sigma-Aldrich silicon dioxide nanopowder (CAS 7631-86-9) of 10–20 nm particle size (BET) with 99.5% SiO_2. Cement used was ordinary Portland cement (OPC) (ASTM Type 1 recognized by ASTM C150) manufactured by Cahaya Mata Sarawak Cement Sdn. Bhd. (CMS) and it exceeds the quality requirements specified in the Malaysian Standard MS 522: Part 1: 1989 Specifications for OPC. The chemical composition of the OPC is given in Table 1.

2.2. Sample Preparation, Characterization by Using XRD, FTIR, and EDS, and Morphology Using SEM Techniques. The mix proportion for cement paste was set at 0.5 water to cement ratio (w/c) for all specimens that were casted into Universal Container 30 mL, diameter 28 × 85 mm for XRD, EDS, and SEM. All specimens were cured in the concrete laboratory at Universiti Malaysia Sarawak at daily room temperature (T) and relative humidity (RH) in the range of 18–28°C and 65–90%, respectively. The fine powder (passing 75 μm) and polished small samples were prepared and analysed using XRD, FTIR, EDS, and SEM on day 28. Ethanol was used to discontinue the hydration process of these samples. XRD analysis for all prepared samples was performed using PANalytical equipment with CuKα radiation and λ of 0.1546 nm. The specifications were count step: 4 sec/step, step size: 0.02 degrees, and range: 5–65 2θ. Shimadzu Fourier Transform Infrared Spectroscopy (FTIR) 81001 Spectrophotometer was used to perform the FTIR analysis. The spectrum measurement method applied was attenuated total reflection (ATR) method. Transmission infrared spectrum of each sample was recorded using a Fourier transform infrared spectrophotometer (IR Affinity-1) in the region of 400 to 4000 cm^{-1} with 2.0 cm^{-1} resolution. The samples were scanned 20 times. SEM images and EDS analysis for all prepared small polished samples were captured by scanning electron microscope and energy-dispersive X-ray spectroscopy (JSM-6701F) supplied by JEOL Company Limited, Japan, which followed the ASTM C 1723-10 (2010) code of practice.

2.3. Sample Preparation and Mechanical Test. The mix proportion mortar was set at (cement : sand : water) 1 : 1.67 : 0.5 (w/c) for all specimens that were casted into 150 mm cubes for compressive strength (CS) tests. All specimens were cured in the Concrete Laboratory at Universiti Malaysia Sarawak for 7, 14, and 28 days. CS test was performed according to BS 1881-116 (1983) on 150 mm cubes samples. It was used to determine the maximum compressive load that a sample can carry per unit area. Compressive strength test was performed to determine the maximum compressive load that the sample can carry