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Inclusion of nano metakaolin as additive in ultra high performance concrete (UHPC)

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HIGHLIGHTS

- To characterise the cementitious properties of nano metakaolin as additive in UHPC.
- To optimise the mix formulation of UHPC embedded with nano metakaolin.
- To investigate the workability of nano metakaolined UHPC as additive.
- To investigate the strength development for nano metakaolined UHPC.
- To investigate the morphology of nano metakaolined UHPC and compare with plain OPC and metakaolined UHPC.

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ABSTRACT

The utilisation of nano particles to enhance concrete can be seen in its densification and refining. In this study, nano metakaolined UHPC mixes were used as an additive range of 1%, 3%, 5%, 7%, 9% and 10% from cement weight. All UHPC specimens were compared to the plain UHPC and metakaolined UHPC mixes. The effect of nano metakaolined UHPC was evaluated in the forms of workability in fresh state, strength property and the morphology of the microstructure in the UHPC. The determination of the fresh property was conducted by a workability test. Meanwhile, the strength property was conducted by compressive strength in 3, 7 and 28 days. Next, the morphology in the UHPC microstructure was analysed by Scanning Electron Microscope (SEM). The uniqueness and difference morphology formation was analysed and compared to the plain UHPC. It can then be concluded that the nano metakaolined UHPC mix contributed to a low workability effect due to its clay properties and ultrafine size as compared to the OPC and metakaolin. The inclusion of nano metakaolin in UHPC shows similar compressive strength at early age but gradually increased at later ages as compared to the plain UHPC and metakaolined UHPC.

1. Introduction

Ultra High Performance Concrete (UHPC) is one of the concrete types that are being used in construction today. Before UHPC was invented, High Strength Concrete (HSC) whose the compressive strength is between 50 and 90 MPa was popular to be used in concrete design [1,2]. The HSC started to develop in construction in the 80's and the mix formulation was designed based on a mathematical approach and then finalised to HSC formulation and obtained by BS-EN and ASTM standard [3,4]. The HSC strength enhancement is gained with the help of additive such as silica fume, metakaolin and ground granulated blast furnace (GGBS). In addition, the selection on raw materials such as quartz and basalt also influences the HSC strength properties by increasing the strength due to high siliceous properties. Moreover, the hardness of quartz and basalt which is higher than granite also improves the strength properties of UHPC [2,5,6].

Since the new millennium, researchers' interest has grown to produce more durable and high strength concrete. The UHPC must achieve compressive strength higher than 150 MPa [7–9]. In achieving these properties, the selection of fine aggregates should be less than 10 mm [10–12]. This is to ensure a pack component and fewer voids created during the mixing can be achieved. Also, it is to note that there is a high content of cement used in preparing the UHPC [10,13,14]. In addition, pozzolan is required material to achieve the target strength. Silica fume is the popular pozzolanic material used in UHPC mix due to its fine particle. 98% of silica

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component provides a better strength compared to the OPC and other types of pozzolanic material [15,16].

To note, there are numerous addition of metakaolin in the UHPC and the performance is slightly lower than the silica fume [17,18]. However, the metakaolin effect on the durability properties is claimed to have been better than the OPC and silica fume [19,20]. Hence, in this study, the effect on strength properties of the metakaolin in UHPC was determined. The inclusion of nano particles as additive in UHPC was also introduced.

Nano comes from a Greek word which means dwarf or small [21,22]. Since the 80's the ideas to convert micro based materials into nano particles grew and it has helped to improve the quality of human life. The utilisation of nano technology in human daily routine has become more prominent in biotechnology, electronic, medical appliances and military equipment. In concrete, the addition of nano materials started with the blooming utilisation of UHPC in the construction industry. Theoretically, the addition of nano materials in concrete is to improve the microstructure of UHPC and create a neat and uniform component of concrete with less pores and voids. Studies on the pore volume in concrete is not intensively explored but it is well known that the microstructure of concrete is porous [23,24]. In order to overcome the problem, micro-based material such as pozzolanic material is incorporated in the concrete. Pozzolanic material shows a huge impact in reducing the porosity problem for normal and high strength concrete. However, for packed component in the microstructure of UHPC, the addition of micro-based material shows a slow effect in improving its performance. In normal concrete and the HSC, void and pore problems occur between the cement pastes, but in the UHPC, the problem is extended to the calcium silicate hydrate (C-S-H) structure [25–27]. The C-S-H gel in concrete is known as nano structures [28–30]. The uniformity of the C-S-H gel will ensure the performance of the concrete. The performance of the UHPC would be enhanced and durability properties can be gained by modifying the C-S-H structures. Micro base material, especially silica fume, shows a good UHPC enhancement.

The inclusion of silica fume in modifying the C-S-H structure was explored and reported [31,32]. However, other pozzolanic material such as metakaolin has not been revealed extensively. This lack of exploration generated the idea of introducing nano particles in UHPC. It is hypothesized that the nano particle perhaps can improve the UHPC structure by producing a better and more uniform C-S-H gel.

Hence in this study, the utilisation of nano metakaolin and metakaolin in UHPC was then experimented. At present, extensive research on nano metakaolin and metakaolin in UHPC, especially the crystal formation in the microstructure, has not been disclosed. Having smaller particle effect as a nano filler is hypothesized to be able to produce a more densified UHPC mix, which in turn enhances its performance.

2. Methodology

2.1. Preparation of raw material

The preparation of raw materials in this study is discussed briefly in this section. Ordinary Portland Cement (OPC) for this research was procured by YTL (M) Sdn Bhd. Fine aggregates was procured from river sand and sieved using a 4.75 mm sieve to remove the silt and clogging particles. Meanwhile, the 10 mm coarse aggregate, which was granite, was selected. To maintain the freshness of the UHPC mix, a Glenium Ace Suretec 389 hyper plasticizer was applied. It was supplied by BASF (M) Sdn. Bhd. The kaolin was supplied by AKI (M). To transform kaolin into nano kaolin, Fritsch High Energy Milling was used to perform milling process. For milling process, zirconia jar and ball was used in order to avoid heat during milling. Fifteen 5 mm balls were selected. The duration of milling was 24 h at a speed of 400 revolution per minute (rpm). The final size for nano metakaolin was confirmed using a Fritsch Laser Particle Analyser and the final size was 176 nano meter (nm). Using the same instrument, the size of kaolin was detected as 5.5 μm meter (μm). Finally, the nano kaolin and metakaolin underwent a calcination process to change into nano metakaolin and metakaolin. The duration and temperature of the calcination process was 3 h at 700 °C for both samples. Table 1 shows the chemical composition and Table 2 shows the particles size of cementitious materials used in this study with the surface area of cementitious material by using Fritsch laser particle analyser. From Table 1 proves that the silica and alumina content in the nano metakaolin have increased as compared to the OPC, metakaolin and nano kaolin. Apart from that, the effective size for nano metakaolin was detected and smaller than other cementitious materials. Based on this analysis, calcination of nano metakaolin improved to reduce the size and increased the pozzolanic reactivity of nano metakaolin by increasing the silica and alumina component. Fig. 1.1 shows the XRD analysis for kaolin, nano kaolin and nano metakaolin. The kaolinite content of nano kaolin and nano metakaolin is increased as compared to the kaolin and metakaolin. This is confirmed by the peak shows in Fig. 1.1. In other words, the transformation of crystal into amorphous phase for nano kaolin into nano metakaolin was confirmed.

3. Mixing and handling uhpc mix

3.1. Finalisation of the UHPC mix proportion

Since there is no standard design mix for a UHPC, the final mix designed in this study was finalised based on the Matte and Morranville (1999) series of trial mixes. The decision to select this mix was conducted on the cement content by Matte which were the lowest among other researchers [10,11]. However, the final design mix in this study was modified in order to create a new design mix for the UHPC that captured with the moderate cement, using normal type of aggregates and achieved compressive strength at day 28 more than 150 MPa.

Table 3 shows the difference between the Matte mix and the present author's mix after undergoing modification. In addition, the basis of this new design mix used in this study has undergone a series of trial mixes to achieve the targeted compressive strength mentioned before [8,13,33].

3.2. Preparation, mixing and handling of raw materials and UHPC specimens

The final mix proportion for this study was tabulated in Table 4. The mixing was commenced by preparing the paste of the UHPC. A

Table 1
Chemical composition of cementitious materials.

Chemical	Mass Percentage (%)				
	OPC	Kaolin	Metakaolin	Nano Kaolin	Nano Metakaolin
SiO_2	11.6	70.3	74.3	80.6	89.6
Al_2O_3	2.2	14.2	10.2	13.9	0.9
CaO	75.17	0.8	2.4	0.13	0.43
TiO_2	0.4	0.65	1.45	0.62	1.82
Fe_2O_3	5.38	0.76	1.82	0.5	2.0
K_2O	0.43	1.62	4.64	1.35	4.55
Remaining constituent	4.82	11.67	4.28	0.9	0.7