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Influence of nano metakaolin on thermo-physical, mechanical and microstructural properties of high-volume ferrochrome slag mortar

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HIGHLIGHTS

- The combined effects of NMK and waste FeCr-slag on properties of mortar were studied.
- Replacing 50% of sand by FeCr slag led to improved compressive and flexural strengths.
- NMK is effective in providing additional enhancement in mechanical performance.
- Significant increase in thermal conductivity of NMK-modified mortars has been achieved.
- Physical packing and pore refinement effects of NMK result in less water absorption.

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ABSTRACT

This study investigates the effect of nano metakaolin (NMK) on the thermo-physical, mechanical and microstructural properties of hardened cement mortar containing high amount of ferrochrome (FeCr) slag. Sand was replaced with FeCr-slag aggregate at the ratio of 50 mass, %. Cement was partially replaced with different amounts of NMK at the ratios of 2, 4, 6, 8, 10, 12 and 14 mass, %. Compressive strength, flexural strength, drying shrinkage, capillary water absorption and thermal conductivity of the hardened nano-modified mortars were determined in accordance with ASTM standards at 28 days of water curing. The microstructure characteristics of the hardened samples were investigated by scanning electron microscope (SEM) equipped with energy dispersive analytical X-ray unit (EDAX). The results showed considerable improvements in the mechanical and thermal performance of cement mortar, due to the incorporation of FeCr-slag; in addition, the replacement of cement by NMK was found to be effective in providing an additional enhancement in the mechanical strength and thermal conductivity. Furthermore, NMK containing mortar has considerably reduced drying shrinkage and capillary water absorption. The reduction in drying shrinkage for the mortar containing 50 mass, % FeCr-slag and modified with 10 mass, % NMK reached 87% relative to the reference mortar. The microstructure of the hardened mortar containing NMK appeared quite dense and compact with finer pore structure.

1. Introduction

The use of industrial solid wastes as a partial replacement of raw materials in construction activities is a promising method for reducing the environmental impact from the industry, compensating the lack of natural resources and reducing the demand for extraction of natural raw materials [1,2]. Concrete is the most widely used construction material [3]. According to the data collected by the U.S. Geological Survey (USGS) in 2011 [4], the annual global production of OPC was about 3.3 billion metric tons (mt). Based on the standard concrete mixture proportions for ordinary concrete [5], this quantity of OPC is utilized in approximately 27 billion mt of concrete, which requires 22 billion mt of aggregates and 2.2 billion mt of water, resulting in a yearly global average consumption rate of about 4 mt of concrete per capita [6]. Aggregate is a significant material used for the composition of mortar and concrete, which occupies major portion (i.e. about 70%–80%). Properties of a specific concrete mix will be determined by the proportion and type of sand used in concrete production since it has significant impact on the workability, density, strength, durability, and shrinkage of concrete. Several industrial by-products have

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been utilized successfully as sand or aggregates replacement in cement mortars and concrete including steel slag [3–8], copper slag [9,10], blast furnace slag [11] and recently ferrochrome slag (FeCr-slag) [12]. Previous study on replacing fine aggregate with iron slag, a by-product of iron and steel making industry, in concrete affirmed that, the compressive strength increases with increase in the substitution up to 30 mass, % of iron slag [8]. The mechanical properties of concrete containing various proportions of copper slag ranging from 0 to 100 mass, % as a replacement of sand have been investigated [16]. An increase in the compressive strength by about 55% has been attained at 40 mass, % replacement of fine aggregate by copper slag. Also this study revealed that, flexural strength at 28 days is higher than control mix at 20 mass, % replacement of fine aggregates by copper slag; an enhancement by 14% was obtained. The high toughness of copper slag was found to be responsible for the compressive and flexural strengths enhancements. Although; the use of waste metal sands such as copper slag, steel slag and iron ore sand/slag as a replacement of fine aggregate for improving the mechanical strength of cement and concrete has been examined, research studies investigating the potential use of FeCr-slag as fine aggregate on the mechanical and thermal properties of mortars and concrete are scarce in the literature [12]. Nanomaterials with different chemical properties have been used in the production of cement paste and mortar which led to significant improvement in the mechanical, physical and structural properties, and durability of cement composites [13]. The incorporation of nanoparticles into the cementitious materials may takes different forms as spherical materials (e.g., nano-SiO_2, nano-TiO_2, nano-Al_2O_3, nano-$CaCO_3$, nano-Fe_2O_3, nano-Fe_3O_4, etc.) [14] or as nanotubes or fibers (e.g., carbon nanotubes and carbon nanofibers, respectively) or as nano sheets (e.g., nano-clay) [15] or as nano platelets (e.g., nano graphene oxide) [17]. Many researchers have affirmed that even at small percentages, nano-SiO_2 can enhance the mechanical strength of cement composites [18–23] and the microstructure becomes more compact [20]. The changes in mechanical and micro-structural properties of cement mortar due to the incorporation of nano-metakaolin (NMK) have been considered by many researchers [25–27]. The inclusion of nano-clay materials such as NMK to cement paste and mortar enhances the compressive and flexural strengths and results in quite dense, compact and uniform microstructure. A previous study [28] reported that, incorporation of nano-clay in mortars improved the compressive strength by up to 24% in samples with 3% nano-clay. Nano-clay particles modify the properties of cement composites and exhibit a pozzolanic reaction with cement causing micro-filling effect [24]. The effect of doping of nano-metakaolin (NMK) into ordinary Portland cement (OPC) on the hydration and microstructural characteristics of hardened OPC–NMK pastes was recently studied, The OPC was partially replaced by NMK at 4, 6, 10 and 15% (by weight). The incorporation of NMK into cement has improved the compressive strength of the hardened pastes during all ages of hydration; an enhancement by about 28% was attained for the paste containing 10 wt% NMK at 28 days of hydration [25]. Although, the influence of micro- and nano-mineral additives on the properties of cement mortars and conventional concrete has been widely investigated [26], few efforts have been focused on the effect of nano materials on the behavior of cement mortars containing high amounts of FeCr-slag as sand replacement. The effect of fly ash as micro pozzolana on properties of concrete containing high amounts (25, 50 and 75 mass, %) of coarse FeCr-slag aggregate has been recently investigated. The results from the study revealed that, the effect of FeCr-slag aggregates on porosity and water absorption of concrete was insignificant while, fly ash (FA) enhanced these properties. The aim of this study is to investigate the influence of NMK as partial replacement for cement on the properties of high volume FeCr-slag mortars, replacing 50 mass, % of sand. The investigation includes thermo-physical, mechanical and microstructural characteristics. The motivations for choosing the NMK can be summarized as follows: (i) economic – this material is very cheap (cost effective), (ii) availability – this material is locally available and (iii) characterized by unique physical properties (e.g., superior surface area and platelet-like morphology, which makes it favourable to use in cementitious matrix reinforcement i.e. it acts as a fiber (2D reinforcement) among cement hydrates. The current research hypothesis is that, due to its ultra-fine size (superior surface area), the NMK particles will distribute and react more efficiently and hence, significantly contribute to strength development than micro pozzolan like (FA).

2. Experimental program

2.1. Materials

The ferrochrome slag was collected from the operating plants of ferrochrome industry with submerged arc electric furnaces, situated in Al Tamman Indsil Company (ATIC) in Oman. The slag is available as granulated having the suitable grain size which can be used as fine aggregate to partially substitute the fine sand in this study. The sand used in our study is natural siliceous sand passing 2.36-mm sieve and having specific gravity of 2.60. The grain size of FeCr-slag was also adjusted to ranging from 0.06 to 2.36 mm by using US standard sieve No. 8. The amount of material passing this sieve was about 85% of the as received batch. The particle size distribution of both the sand and granulated FeCr-slag is presented in Fig. 1.

The cement used in this study was ordinary Portland cement, OPC (CEM I: 42.5 N). The nano material used in this work is nano-metakaolin, NMK (thermally activated nano-kaolin). The Nano kaolin (NK) was supplied by Middle East Mining Investments Company (MEMCO). The oxide compositions of OPC, FeCr-slag and NMK were determined by X-ray fluorescence (XRF) as shown in Table 1. FeCr-slag contains as three major components alumina, silica and magnesia and includes appreciable amount of chromium. The mineralogical characterization of fine sand and FeCr-Slag in addition to NMK were performed by X-ray diffractometer and shown in Figs. 2 and 3, respectively. The XRD pattern of the sand confirms its siliceous nature (i.e. quartz is predominantly present)- whereas the pattern of FeCr-slag indicates the presence of dominant mineral phases like metallic phase of magnesiochromite ($MgCr_2O_4$), chromoferide, silicate phases like forsterite (Mg_2SiO_4), and Fayalite (Fe_2SiO_4. The NMK exhibited amorphous structure; the pattern interpretation led to identification of the kaolinite phase, illite and quartz.

The micro morphology of the NMK was investigated by the scanning electron microscope (SEM) and presented in Fig. 4. Fig. 4 shows that, NMK has plate like structure and characterized by large length to thickness aspect ratio and the platelet thickness is only 1–20 nm, although its dimensions in length and width can be measured in hundreds of nano-meters, with a majority of platelets in 200–500 nm range after purification. SEM integrated with Mountains map image analysis software was used to investigate surface micro roughness of the aggregates and the results are shown in Fig. 5. It can be seen that, the particle shape of sand is partly rounded, while FeCr-slag particles have irregular angular shape. Furthermore, the slag aggregates exhibited relatively rough surfaces as compared with sand grains, which are of smooth surfaces.

2.2. Samples preparation

The mortar was prepared using cement-sand ratio of 1:2.75. Fine sand was substituted by 50 mass, % of FeCr-slag and the OPC was partially replaced by different proportions of NMK at a rate of 0, 2, 4, 6, 8, 10, 12 and 14 mass, % as given in Table 2. The blended cement mortars were prepared using various amounts of water in order to maintain a constant workability degree among different samples. The dry OPC and FeCr-slag were mixed at a speed of 50 rpm using an electric mixer for 2 min to obtain a homogenous mix. The NMK particles were first dispersed in the mixing water (without surfactant), using high intensity ultrasonic bath (frequency: 20 kHz) for 15 min. to assure a good dispersion and to avoid agglomeration and, then they were added to mix to form the freshly blended cement mortars.

Three groups of samples were cast for testing, the first group was cast as cubes $50 \times 50 \times 50$ mm for compressive strength and capillary water absorption tests; the second group was cast as prisms $40 \times 40 \times 160$ mm for flexural strength and thermal conductivity tests and the third group consisted of samples of size $280 \times 25.4 \times 25.4$ mm for drying shrinkage measurement. The fresh mortars were kept in moulds for 24 h, and then de-moulded and allowed to cure in water for 3, 7, and 28 days. For compressive and flexural strengths, drying shrinkage and capillary water absorption, three samples per batch were tested, and the average results were calculated.