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Effect of Graphene Oxide Nanosheets dispersion in cement mortar composites incorporating Metakaolin and Silica Fume

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HIGHLIGHTS

- GOD addition improves the microstructure of cement composite mortars.
- The mechanical properties were enhanced by the addition of MK, SF and GOD.
- The addition of GOD with MK and SF improved the transport properties.
- SEM confirms the addition of MK and SF with GOD accelerated the hydration process and densified the microstructure.

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ABSTRACT

In this work, the reinforcing effects of Graphene Oxide Nanosheets dispersions (GOD) on high strength cement mortar are investigated, evaluating possible improvements in mechanical properties, fluidity, Sorptivity and water absorption resistance. Mortar with water/cement ratio of 0.5, GOD dosage varying from 0 to 0.20% by weight of cement and sand at a proportion of 3× weight of the cement were prepared, in addition to 10% and 20% replacement/no replacement with Silica Fume and Metakaolin respectively to improve the GO nanosheets dispersion. The GO used in the matrix forms aggregate due to presence of divalent Calcium ions. Hence to split the particles vigorous mixing was done. The results indicate that the addition of GOD accelerate the cement hydration reactions, increase the compressive strength and tensile strength of the mortar, reduce the pore volume and harden cement properties. However, the fluidity decreased with increasing GOD content. The optimal compressive strength was achieved with samples designed with GOD dosage of 0.05% with replacement, for which the corresponding increases were 72.41%, 84.61% and 90.90%, after 3,7 and 28 days respectively, compared with samples without GOD and replacement. The tensile strength of the cement mortar increased with GOD content to 132%, 178.6% and 181.2% for 3, 7 and 28 days respectively with replacement and GOD dosage of 0.10%. Furthermore, FE-SEM observation indicated that the GOD probably had an effect on the shape of the cement hydration products. It revealed samples containing 0.10 wt% GOD with replacement exhibited good bonding between the GOD and the surrounding cement matrix. The hydration mechanism of cement composites with GOD and replacement of cement with Silica Fume and Metakaolin needs further study.

1. Introduction

Concrete has been broadly utilized as a part of structural designing everywhere throughout the world for more than one hundred years. It has a generally high compressive quality, yet low flexural and rigid qualities. Perceptible steel support bars are normally used to enhance the quality and malleability of this sort of material, however in late decades broad research on the impacts of smaller scale and large scale fibres in controlling the development of cracks in cementitious materials has been completed [1,2]. In this manner, utilization of discrete fibres brings about more uniform distribution of stress inside cementitious materials. However, these microfibers cannot stop or prevent the initiation of micro cracks in a concrete matrix.

Advancements in nanotechnology in the course of the most recent years have started to exhibit the potential utilizations of nanoscale fillers in composites material [3,4]. These improvements have been driven by the possibilities of utilizing those nanoscale fillers for altering the properties of understood materials, for investigating their principal impact at microscopic and macroscopic levels [5,6] and for the advancement of new materials [7,8]. To

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be sure, nanoscale fillers are presently being fused into various materials and composites for particular applications [9–11]. Furthermore, those current advancements have additionally fortified new thoughts for handling procedures keeping in mind the end goal to enhance the properties of numerous traditional materials essentially. Recently, accomplishments in nanotechnology have created some nanomaterials with incredible execution, for example, carbon nanotubes (CNTs), nanosilica, nanotitanium oxide and Graphene Oxide Nanosheets (GO), which can be utilized as reinforcements to improve defects of cement-based materials at the nanoscopic level [12–15]. On account of the special mechanical, electrical, optical and catalytic properties of nanoparticles, the cooperation between the nanoscale filler and the ceramic matrix material could offer the likelihood of changing cement chemical and physical properties, making new components of collaboration [16] that could be helpful agents on self-cleaning procedures [17] and strategies related with abrasion resistance [18,19,53]. Specifically, reinforcing nanomaterials, for example, carbon nanofibers (CNF) and carbon nanotubes (CNT), has been appeared to enhance mechanical quality and solidness of cementitious nanocomposites [20], albeit, some opposing outcomes have been accounted for [21,22]. One test of studying these nanocomposites is the trouble in dispersing CNT's and CNF in the OPC lattice. The agglomeration of CNT's and CNF's, due to strong Van der Waals forces leads to difficulties associated with dispersion of these materials in the matrix [23].

GO has pulled in huge consideration due its high specific surface area, high strength, adaptability and hydrophilic character [24,25]. In this 2-dimensional material, the oxygen-containing functional groups appended to the GO enhance its dispersion in water [26–28], and in the cement framework [22,23,29] influencing the mortar hydration and mechanical properties [30]. GO bears functional groups like hydroxyl, epoxy groups on their basal planes and carboxyl, carbonyl groups on the edges. These functional groups give GO, a hydrophilic nature [31]. The high surface area and remarkable structure of GO might be advantageous for enhancing bonding strength between GO and the encompassing cement matrix [32–34]. In this way, GO has been acknowledged as great added substances to be joined in cement matrix for strengthening. It was accounted for that GO effortlessly formed composites with cement matrix, and adequately expanded the mechanical properties of cement pastes or mortars [33–43].

Few studies have focused on Graphene Oxide Nanosheets-based mortar composites. In earlier investigations little amounts of GO has been added to cement mortars. Chen et al. [44] reported that the additions of 0.4% by weight of cement (bwoc) GO in cement mortar increased the flexural and compressive strength by 6% and 5%, respectively when the GO was dispersed through sonication and electrophoretic deposition (in carbon fibre). Wang et al. [45] reported that the use of GOD (0.05%) also significantly increased the flexural and the compressive strength by 71% and 24% respectively. Lv et al. [46] reported that the addition of 0.03% by weight of cement (bwoc) GO into cement mortar improved the tensile strength by 78.6% by dispersing through ultrasonification. Babak et al. [47] reported that the addition of GO (2% bwoc) into cement mortar improved the tensile strength by 24.7%. Mohammed et al. [25] explored the impact of GO on other properties of cement. The researchers proved that the addition of GO to cement composite can improve the cement matrix transport properties by performing water Sorptivity and chloride penetration tests. Lu et al. [55] reported that using 0.01% GO nanosheets caused a 7.82% in compressive strength after 28 days of curing. The results indicate that the introduction of GO in small amounts greatly improve the mechanical and transport properties of cement mortar. However, the properties of High-strength cement mortar, has rarely been explored. The High-strength cement mortar is mainly composed of cement, puzzolans such as silica fume and Metakaolin, fine sand, water and GO. The presence of Metakaolin (MK) and silica Fume (SF) results in densification of microstructure of the cement composite. Research studies revealed that the optimum MK replacement level in order to obtain maximum mechanical strength was considered at 15–20% [48]. Aghabaglou et al. [49] reported that at 10% of cement replaced by SF improved the mechanical properties of cement mortar when compared with MK and fly- ash replacement at 10% for cement mortar investigated individually. These materials have only been explored individually in cement mortars in absence of Graphene Oxide Nanosheets at small percentage dosages.

In the present study, the effect of GO, incorporating MK and SF, on the mechanical and fluidity behaviour of the cement mortar was investigated. In addition, the transport properties (Water absorption and Sorptivity) were also discussed. Moreover, the morphology of hardened cement mortar consisting of GO was observed using field-emission scanning electron microscopy (FE-SEM).

2. Experimental

2.1. Materials

The Ordinary Portland cement type OPC-53 Grade (JK Lakshmi Cement Ltd.) conforming to IS: 12269-1987 was used in the mortar study. The pozzolanic additions of 10% and 20% by weight of cement were utilized as a replacement for Portland cement. Fig. 1 shows the Silica Fume (SF) obtained from Elkem Silicon Materials Ltd., Mumbai was used to replace Portland cement at 10 wt%. The particle size is approximately 50 nm to 1 μm exhibiting a spherical shape. Fig. 2 shows the Metakaolin (METACEM 85C-MK) used in the study were procured by 20 μm Limited, Vadodara which replaced the cement at 20 wt%. The physical properties of cement are shown in Table 1. The Table 2 shows the chemical composition of Cement, SF and MK. From Table 2, it can be observed that the MK contains 51–53% of Silica and 42–44% of Alumina, whereas SF is composed by mainly Silica (95%). The locally available river sand under zone II, specific gravity 2.7 passing through 4.75 mm IS sieve, conforming to IS 383 1970 was used as a fine aggregate. The Graphene Oxide Nanosheets dispersions (GOD) manufactured by the United Nanotech Innovations Pvt. Ltd, Bangalore was used as nanofillers without further processing.

2.2. Preparation of Graphene Oxide Nanosheets suspension

The Graphene Oxide Nanosheets ($C_{54}H_{17}$ + O + $(OH)_3$ + COOH) was synthesized by modified Hummers Method [50]. In the process, most importantly, graphite powder (1 g) and sodium nitrate (1 g) were added to 100 mL concentrated H_2SO_4 in an ice water-bath (beneath 5C). Progressively, $KMnO_4$ (3 g) was gradually included under stirring for 30 min. At that point the mixture was exchanged to water-bath for 2 h. At last, deionised water and 30% H_2O_2 solution (5 mL) was added to end the oxidation response. Furthermore, the shade of the arrangement abandoned dull dark colored into yellow. The sample obtained was cooled, filtered and washed with 10% HCL solution. The dried product was then ground with mortar and pestle and transferred to the solvent (water) and was sonicated in an Ultrasonifier (United Nanotech systems). The nominal concentration of the solution was adjusted to 0.89 mg ml^{-1}. The Table 3 gives the technical parameters of the GO utilized.

Fig. 1. Microsilica 920 D.