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# Hydration, microstructure and phase composition of composite cements containing nano-clay



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#### HIGHLIGHTS

• The initial and final setting times are accelerated by the partial replacement of NC.

- The results of the bulk density increase with GBFS contents up to 40% then decreases with 60%.
- The chemically combined water content increase with the NC contents up to 6 mass.
- The gel/space ratio shows a sharp increase with NC content up to 2 mass.
- SEM micrograph displayed a more dense interlocking close linkage structure composed of rod-like crystals.

#### ARTICLE INFO

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#### ABSTRACT

The effect of partial replacement of ordinary Portland cement (OPC) with nano-clay (NC) on the performance of physico-chemical, mechanical, hydration characteristics and microstructure of composite cement pastes containing ordinary Portland cement (OPC) and ground blast-furnace slag (GBFS) was studied. Initial and final setting times are accelerated by the partial replacement of OPC with NC. The values of compressive strength, bulk density, chemically combined water content and gel/space ratio of composite cement pastes increase by using NC up to 6 mass%, then decrease by further replacement up to 8 mass%. The free lime contents of composite cement pastes increase from 1 day up to 3 days, then decrease from 7 days up to 90 days. NC activates the hydration reaction of OPC and GBFS. SEM micrograph of mix M4S40 (54% OPC + 6% NC + 40% GBFS + 1%SP) deposits the presence of micro- and nanocrystalline CSH gel with dense interlocking close linkage structure composed mainly of larger crystalline hydrates. It can be concluded that 6 mass% NC is the optimum replacement level.

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#### 1. Introduction

Nano-technology is a new area of interest in building products and civil engineering. Several types of nano-materials have been incorporated into cement, concretes, ceramic and polymer to produce nano-composites [1–4]. The developments of nano-materials enhance the properties of existing materials [3,5–7]. Nanomaterials were used to improve the characteristics of cementitious materials [8–12]. These nano-materials can efficiently fill available micro- and nano-pores in the open pores system of hydrated cement improving the durability and structure properties of concrete [8,13–17].

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http://dx.doi.org/10.1016/j.conbuildmat.2016.02.177 0950-0618/© 2016 Elsevier Ltd. All rights reserved. Many studies have been performed on the application of clay minerals in composites to enhance the properties of concrete. However there is little knowledge on the application of NC on the durability of cement composites [18]. There is still need for dedicated studies on different types of NC's and the mechanisms by which they affect the hydration, mechanical and physical characteristics and microstructure of composite materials [19]. Nanomaterial acts not only as nucleating agent and filler reduces the pore size and porosity of the cement matrix, but also as a nanoreinforcement that strengthen the interfacial transition zone between the cement paste and the aggregate to promote the hydration of cement phases and improves the strength of cement matrix [3,20–26].

From economic point of view nano-particles are expensive; however NC does not add any significant cost, but improve the mechanical properties significantly [27,28]. The pozzolanic activity and the effect of partial replacement of OPC with nano-metakaolin



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#### Table 1

Chemical oxide composition of OPC, NC and GBFS, mass%.

	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	L.O.I	Total
OPC	20.41 61.24	4.94 20.89	3.40 5.38	63.00 0.16	1.80	2.62	0.40	0.22	0.57 0.70	2.65 10.02	100.01 100.06
NC GBFS	43.22	20.89 9.97	0.59	35.96	0.18 5.43	0.17 1.37	0.61 0.79	0.71 0.67	-	1.98	99.98

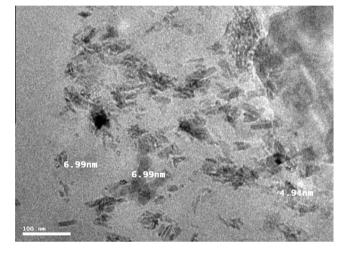


Fig. 1. TEM of NC.

(NMK) and silica fume (SF) on the compressive strength of the hardened composite cement pastes were discussed [29–31]. The optimum replacement of OPC by NMK was found to be 8–10%. The results of compressive strength revealed that replacement of NMK improved the mechanical properties of composite cement pastes. The values of compressive strength of the paste made of OPC-6% NMK-4% SF are comparable to those of OPC paste.

Nano clay (NC) is a new generation of processed clay for a wide range of high-performance nano-composite cement [5,32]. The effect of halloysite nano-clay particles on mechanical properties, thermal behavior and microstructure of cement mortars were investigated [5,18]. Non-modified NC and nano-smectite enhance the nucleation surfaces for the formation of CSH, CAH and CASH hydrated products and improve the microstructure of cement paste [33]. The influence of NC and calcined NC on the mechanical and thermal properties of cement nano-composites was presented. Calcined NC is prepared by heating NC at 750–825 °C [29,30] and at

Table 2

Mix compositions, consistency, initial and final setting times of composite cement pastes of the prepared composite cements, mass%.

900 °C for 2 h [34]. Substitution of OPC with 1 mass% calcined NC improves the mechanical and thermal properties of the nanocomposites cement [34]. QXRA and SEM analyses indicate that the calcined NC acts not only as a filler to improve the microstructure, but also as the activator to enhance the pozzolanic reaction. The effect of hybrid nano-clay and carbon nano-tubes in the enhancement of compressive strength of mortars was investigated [9,35]. The partial replacement of OPC with 2–8% calcined NC enhances the compressive and flexure strengths. 10% nano-clay increases the compressive and tensile strengths with 7 and 49%, respectively [36].

The economic, environmental and engineering benefits of reusing GBFS solid wastes by-products as a partial cement replacement have been established to get more durable concrete with less cement content and within a reasonable cost. The aim of this work is the inclusion of NC to improve the physico-chemical, mechanical and microstructural characteristics of composite cement containing GBFS.

#### 2. Materials and techniques

#### 2.1. Materials

The starting materials used were Type (I) ordinary Portland cement (OPC), nano clay (NC) and polycarboxylate based superplasticizer (SP). The ordinary Portland cement (OPC) provided from Lafarge Cement Company, Egypt. Its chemical analysis is given in Table 1. The Blaine surface area of OPC is 3050 cm<sup>2</sup>/g. NC used in this investigation was supplied by Middle East Company, Cairo, Egypt. NC was fired for 2 h at 750 °C to give active amorphous NC. The chemical composition of NC is given in Table 1. TEM of NC is presented in Fig. 1. Superplasticizer (SP) based on polycarboxylate polyester obtained from Sika Company, El-Abor City, Egypt. The physical and chemical properties of SP were given in the earlier publication [37].

#### 2.2. Experimental techniques

The ingredients were homogenized on a roller in a porcelain ball mill with four balls for 2 h to assure complete homogeneity. The cement blends were mixed in a rotary mixer. The mixing was performed as follows; NC stirred with the required water of standard consistency at high speed of 120 rpm for 1 min, SP was added, then stirred at high speed for additional 30 s. The cement was added to the mixer at medium speed (80 rpm) for 30 s. The mixture was allowed to rest for 90 s, and then mixed for 1 min at 120 rpm. The mix composition of the prepared composite

Mix.	OPC, %	NC, %	GBFS, %	SP, %	Consistency, %	Setting time, min.	
						Initial set	Final set
M0	100	0	0	0	26.5	190	345
M1	99	1	0	0	25.8	180	315
M2	98	2	0	0	26.5	174	300
M3	96	4	0	0	27.1	170	290
M4	94	6	0	0	27.6	165	275
M5	92	8	0	0	29.4	145	270
MOS	100	0	0	1	20.0	65	220
M1S	99	1	0	1	20.1	60	230
M2S	98	2	0	1	21.0	65	240
M3S	96	4	0	1	21.3	75	248
M4S	94	6	0	1	21.5	95	260
M4S30	64	6	30	1	20.6	65	240
M4S40	54	6	40	1	20.4	70	245
M4S60	34	6	60	1	20.0	78	250

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