Improving The Reactivity Of Clay Nano-Partciles In High Strength Mortars Through Indirect Sonication Method

A.Maher El-Tair, M.S. El-Feky, Kamal G. Sharobim, Hassan Mohammedin, M. Kohail

Abstract: The major problem that faces the use of nano-clay in concrete or in cement mortars is its high-water demand. Many researchers had investigated the reason for the high water demand was due to the high agglomeration behavior of nano-clay when mixed with water. Sonication is one of the methods used to de-agglomerate nano-clay particles and to improve the dispersion of nano-clay in the cement matrix. This research aims to reach the optimum indirect sonication time and the solid to liquid ratio that enhances the dispersion of nano-clay and consequently increases its reactivity in high strength cement mortars. The particle size distribution of the sonicated nano-clay particles and the corresponding specific surface area were the main keys governing the optimization process of the studied parameters. Using the optimum solid to liquid ratio with the corresponding sonication time, cement mortars were prepared, and their compressive strength as compared to a control mix (without nano-clay addition) was measured as an indication of the reactivity of nano-clay. In addition the performed microstructural analysis, SEM and XRD helped in confirming the compressive strength results. The results revealed that the optimum solid to liquid ratio is 1:6. Moreover, for every concentration, there is an optimum indirect sonication time. The optimum sonication time found to be 10 minutes for a solid to liquid ratio 1:6. By using the optimum concentration, an improvement in compressive strength of 18.7% and 22.6% after 7 and 28 days reached as compared to the control mix.

Index Terms: Indirect Sonication – Particle Size Distribution - Compressive Strength – Microstructure Analysis – Solid to Liquid ratio

1. INTRODUCTION

The production of cement is one of the main sources for carbon dioxide emission [1-3]. Carbon dioxide produced from the cement industry ranges from 6 -7 % of the total carbon dioxide emission as reported by International Energy Agency (IEA) [4]. In 2050, the production of Portland cement is expected to increase by 200% [5-7]. Using supplementary cementitious materials as a partial replacement to Portland cement had been studied and investigated using materials in micro-scale; silica fume, fly ash, and ground blast furnace slag and materials in nano-scale; nano-silica, nano-clay, nano-cellolous, carbon nano-tube [8-11]. There are two main benefits for using supplementary materials; an environmental benefit and a technical one. For the environmental one, is the reduction of amount of Portland cement in the construction industry. While for the technical benefit; is improving the microstructure, the mechanical and the durability properties of concrete [12-20]. Generally, Nano-materials improve the reactivity of concrete through mineralogical composition and particle size distribution [20-27]. This improvement can be achieved through enhancing the homogeneity of the cement matrix through reaction occurred between the nano-materials with calcium hydroxide $Ca(OH)_2$ to produce addition calcium silicate hydrate C-S-H gel [12, 28-32]. Nano-clay can be added to the cement mortars and concrete to improve the microstructure through filling effect, pozzolanic effect in addition to the damming effect, which is characterized by this option than any other nano-materials, in which the structure of the nano-clay particle is platy, elongated, and thin shape [33-37].

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Nano-clay contains alumina that reacts with CH to produce alumina-containing phases, including C_4AH_{13}, C_2ASH_8, and C_3AH_6 [38]. Many litterateurs investigated the effect of partially replacing cement with nano-clay. All the researchers found that nano-clay improve the compressive and flexural strength of concrete [34-36]. Nano-clay also, improve the resistance to alkali-silica reaction and the sulfate resistance due to the damming and the filling effect of nano-clay particles which enclose the nano-voids of the cement matrix and block the penetration of any chlorides or sulfates [38-43]. However, it found that increasing nano-clay replacement ratio results in an increase in the water demand due to the agglomeration occurred to nano-clay particles because of the difficulty in the dispersion of its particles in the cement matrix [43-46]. Others had worked on improving the dispersion of different nano-materials in the cement matrix and in concrete to modify its granulometric distribution and to improve its densification, especially nano-silica, carbon nano-tubes, and nano-cellulose [47-50]. Sonication is one of the effective methods to solve the agglomeration occurred and to improve the dispersion of nano-particles in the cement matrix [49, 50, 53-57]. Indirect sonication or bath sonicator was found to be the optimum method for improving the dispersion of clay nano-particles through the cement matrix [58-61]. Norhasri et al. [45] reported that nano-clay had no effect on the compressive strength of ultra-high performance concrete at early age while the effect of nano-clay was observed on the strength at later ages. In addition to, the negative impact of nano-clay on the workability of concrete. On the other hand, Morsy et al. [32] had found that the optimum replacement ratio of nano-clay to Portland cement was 8%, which improve the compressive and flexure strength by 7% and 49%, respectively. Shakir et al. [51] had reported that the optimum nano-clay percentage is 10% of the weight of cement with an improvement in the compressive and splitting tensile strength is 63.1% and 25.6%, respectively as compared to the control mix. However, Patel [52] had found that the optimum percentage of nano-clay is 1% only of the cement weight. From the above literature, it noted that there is no specific replacement ratio obtained for nano-clay as a replacing material for Portland cement. This was due to the reduction in the workability of concrete and the poor dispersion of clay nano-particles in the cement matrix [36, 37]. In this paper, improving the dispersion of clay nano-particles will be investigated in terms of particle size distribution and specific surface area through indirect sonication method. In addition microstructure analysis (XRD, SEM) will be performed to study the effect of sonicated nano-clay on the microstructure of cement mortars.

2 EXPERIMENTAL PROGRAM

The experimental program carried out in this research divided into two phases; the first phase objective is to optimize the liquid to solid ratio of nano-clay using indirect sonication method on different durations. Where a group of 7 different concentrations of nano-clay; 1:30, 1:10, 1:7.5, 1:6, 1:4, 1:3, and 1:2, were sonicated for 5 different durations; 0, 5, 10, 15 and 20 minutes for each. The optimum concentration was chosen based on the optimum particle size distribution, and the specific surface area of the sonicated samples. In the second phase, the effect of nano-clay dispersion on the compressive strength was studied using the optimum concentration and with the corresponding indirect sonication time. 13 mixtures were used to study the effect of clay nano-particles after subjected to sonication on the compressive strength of the cement mortars. In addition, Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) was carried out to study the impact of sonication on improving the microstructure of cement mortars.

2.1 Materials

Ordinary Portland cement (Type I) complying with the requirements of ASTM C150 [62] standard was used. Silica fume used was produced by the Egyptian ferro-alloys company in Aswan, Egypt. Nano-clay was produced by the physics department in the Housing and Building National Research Center (HBRC) in Egypt, with average particles size 30 nm. Chemical and physical properties of cement, silica fume, and nano-clay listed in Table 1. Transmission Electron Micrographs (TEM) and powder X-Ray Diffraction (XRD) diagrams of nano-clay particles are shown in Figures 1 and 2, respectively. Natural Silicious sand free of alkali-reactive materials from Suez quarries, in Egypt was used. The properties of sand are listed in Table 2. Sika Viscorete 3425 was used as a polycarboxylic ether additive.

2.2 Equipment used

Bath sonicator (indirect sonication method), produced by FALC instruments, Italy. The ultrasonic waves are subjected to nano-clay through the water bath and then through the thickness of the container before reaching the solution, 40 kHz frequency and 100% power of sonication [49]. Temperature is set to 20 °C and tank temperature does not exceed 40 °C.

2.3 Experiments

2.3.1 Particle size distribution

This method is used to optimize the optimum dispersion of nano-clay in water. Mastersizer 3000, is used to measure the particle size distribution through the use of a laser light scattering technique. The data acquisition rate is 10 kHz. Its typical measurement time is less than 10 s. Particle size varies from 0.01 to 3500 μm.

2.3.2 Scanning Electron Microscope (SEM)

Scanning Electron Microscope is examined through QUANTA FEG250. Through SEM the nano-particle distribution and characterization the concrete mixtures can be determined. In addition to helping in interpret the compressive strength results of the mortar samples.

2.3.3 X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) is used to detect the changes in the hydration reaction and products. The tested samples were taken from the crushed product after applying the compressive strength.

2.3.4 Compressive Strength Test

The compressive strength test of the mortar samples is determined after 7 and 28 days of moisture curing as per ASTM C39 [64]. The test is carried out using a universal testing machine SHIMADZU 1000 KN.

2.4 Samples Preparation

33 samples were chosen to study the optimum solid to liquid ratio and to determine the optimum indirect sonication time for nano-clay. The preparation process for nano-clay samples performed using bath sonicator. 7 solid to liquid ratios were examined, which are 1:30, 1:10, 1:7.5, 1:6, 1:4, 1:3, and 1:2. In which 5 gm of nano-clay were added to 150 ml of water in a glass beaker and subjected to indirect sonication times 0, 5, 10, 15 and 20 minutes for solid to liquid ratio 1:30. The same procedures were done for the other concentrations. All the cement mortar mixes cast in 7x7x7 cm cubes.

2.4.1 Mixing Procedures

The mixing procedures were done as the following: (a) Weight all constituents' materials, as shown in Table 3. (b) For control and as receive samples, nano-clay added in a dry state to cement and silica fume and was mixed for 2 minutes, and then half of the water was added to the dry mix. (c) The remaining of the water was added to the superplasticizer and then added to the dry mix, and then mixed for 3 minutes. (d) While for the sonicated mixes, nano-clay was subjected to indirect sonication process with the corresponding solid to liquid ratio for each mix prior adding to the dry mix. (e) The dry mix of cement and silica fume was mixed together for 2 minutes, before the solution of the nano-clay added to them. (f) After adding the nano-material solution to cement and silica fume, the remaining of the water was added to the mix with the superplasticizer and then mixed for 3 minutes. (g) Finally, sand was added to the mix and mixed for 3 minutes.

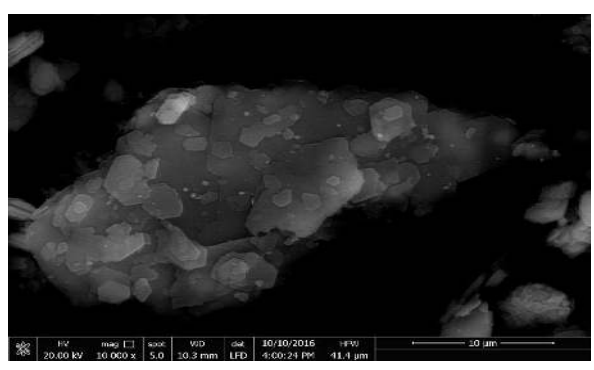


Figure 1: *TEM Microscopy of Nano-Clay*

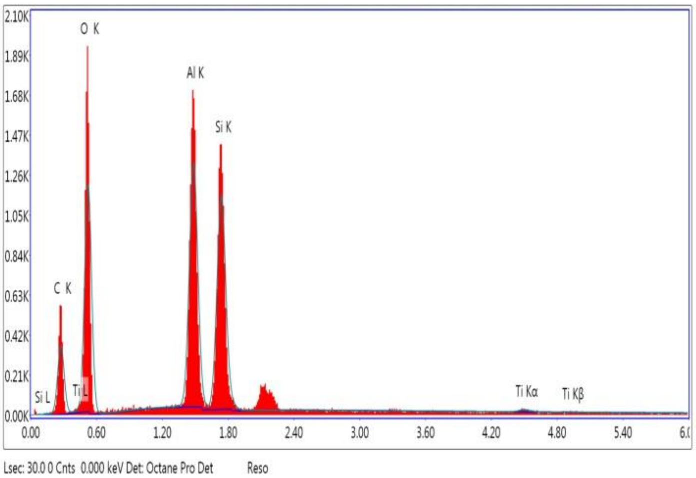


Figure 2: *XRD Analysis of Nano-Clay*

Table 1:*Chemical Composition of Portland Cement, Silica fume, and Nano-Clay*

Element	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	TiO_2	L.O.I
Cement	20.13	5.32	3.61	61.63	2.39	2.87	0.37	0.13	-	1.96
Silica Fume	92	1.1	1.45	1.2	1.55	0.25	0.45	1.45	-	-
Nano-Clay	48	36	0.3	0.28	0.02	-	0.07	-	1.5	12.7

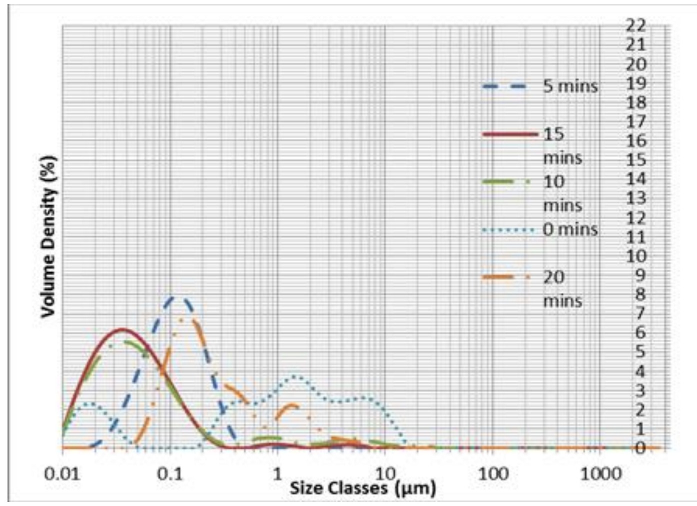
Table 2: *Properties of Sand*

Property	Sand	Specification according to ASTM C33 [52]
Specific Weight	2.89	-
Bulk Density	1.67	-
Fineness Modulus	2.75	2.3: 3.1
Clay and Fine Dust Content (%)	1.95	not more than 3%

3 RESULTS AND DISCUSSION

3.1 Optimum sonication time for each concentration

From Figure 3 and Table 4, generally increasing the sonication time the sub-nano-metric particles of nano-clay content and the specific surface area increased to reach an optimum value and then reduced again for every concentration. The optimum solid to liquid ratio obtained was 1:6, with an indirect sonication time 10 minutes. In which the specific surface area of nano-clay reached 213400 m^2/kg and the sub-nano-metric particle size reduced to be 0.0298 nm. The increase of specific surface area with the increase in the sonication time to a certain limit can be attributed to increasing the electrostatic force, which makes the particle collide with each other and breaks down the Van der Waals forces in the nano-clay agglomerates. In addition, breaking the primary particles and reduce its sub-nano-metric size, as shown in Table 4. While increasing the sonication time over the optimum sonication time, the specific surface area reduced again. This can explained that by increasing the electrostatic force than a certain limit, the collision frequency reduced which leads to a reduction in the particle breakage resulting in agglomeration formation between nano-clay particles can generated.
While at lower solid to liquid ratios, the results show low values of the specific surface areas, was due to the small quantity of the suspensions for clay nano-particles to be well dispersed in the solution. This leads to a reduction in the repulsive force resulting in a re-agglomeration of nano-clay particles.



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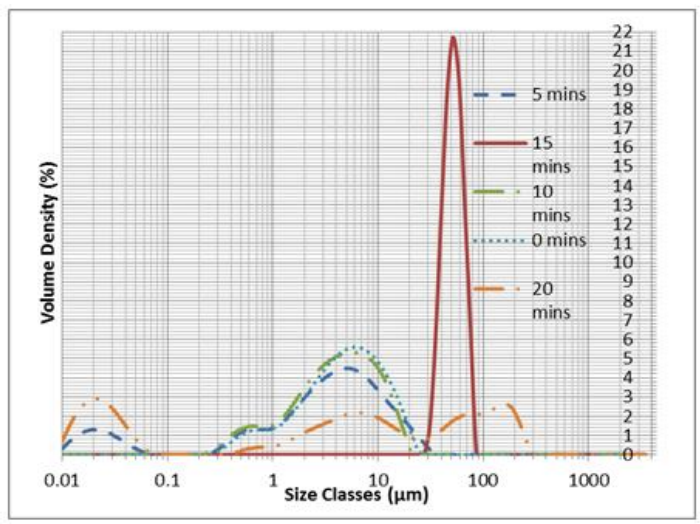

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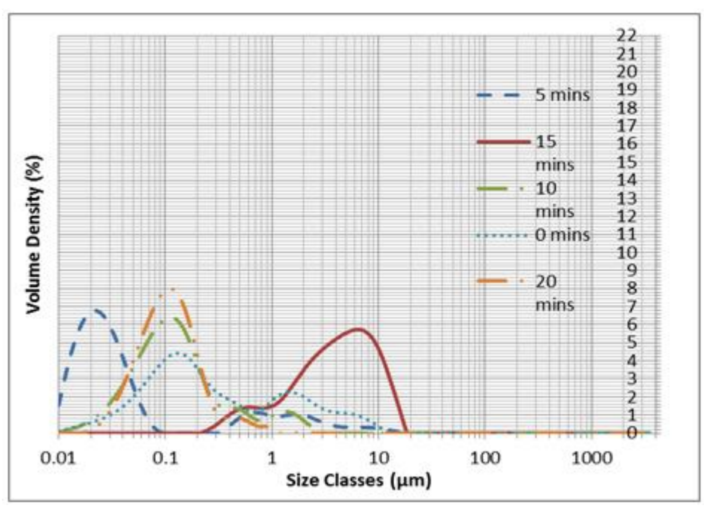

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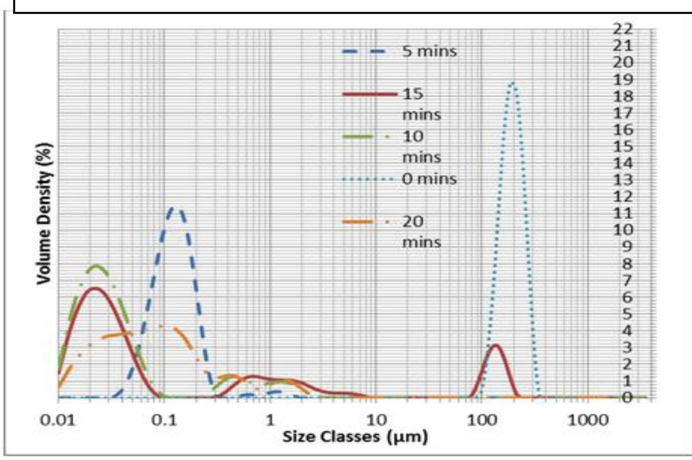
***Table 3:** Nano-Clay Cement Mortars Mixes Constituents (gm.*

Mix	Cement	Silica Fume	Sand	Water	S.P.	N.C%	Concentration	Sonication Time
C	800	200	2000	260	30	0	-	Control
OC1	800	200	2000	260	30	1	powder	0
OC3	800	200	2000	260	30	3	powder	0
OC5	800	200	2000	260	30	5	powder	0
NC1	800	200	2000	260	30	1	1:6	10 mins
NC3	800	200	2000	260	30	3	1:6	10 mins
NC5	800	200	2000	260	30	5	1:6	10 mins
MC1	800	200	2000	260	30	1	1:4	5 mins
MC3	800	200	2000	260	30	3	1:4	5 mins
MC5	800	200	2000	260	30	5	1:4	5 mins
XC1	800	200	2000	260	30	1	1:2	5 mins
XC3	800	200	2000	260	30	3	1:2	5 mins
XC5	800	200	2000	260	30	5	1:2	5 mins



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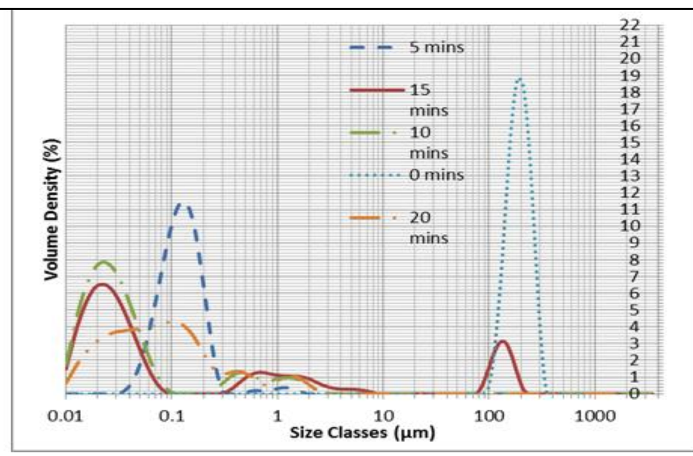

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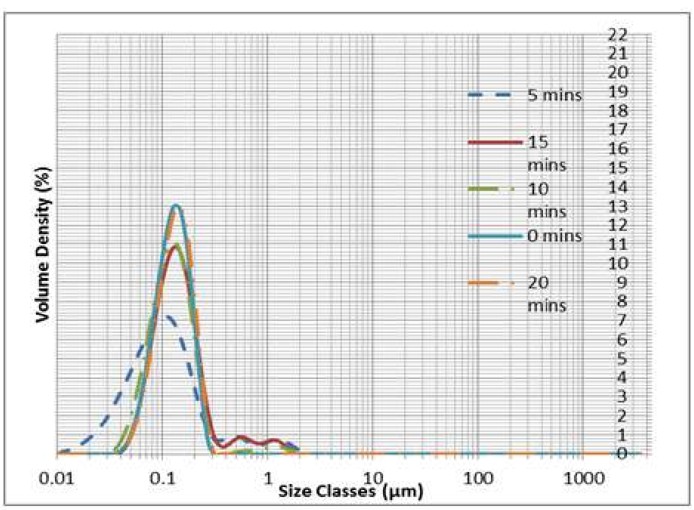

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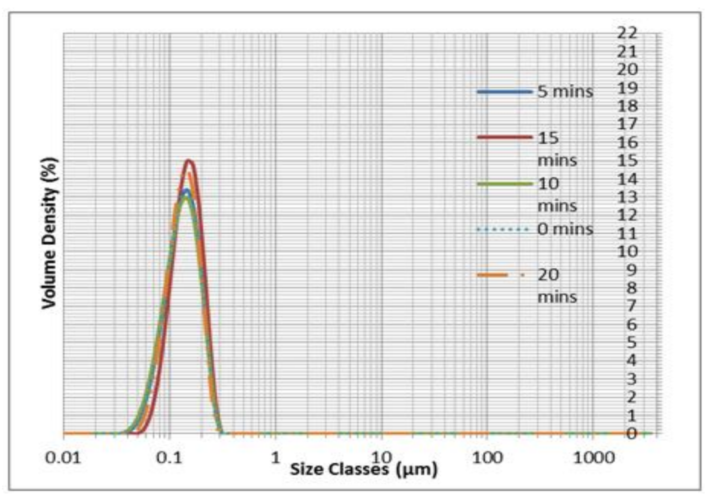


(G)

Figure 3: Particle size distribution of nano-clay samples (a) concentration at 1:30, (b) at concentration 1:10, (c) at concentration 1:7.5, (d) at concentration 1:6, (e) at concentration 1:4, (f) at concentration 1:3, and (g) at concentration 1:2

***Table 4**: Specific Surface Area and mean particle size distribution for each sonication time and concentration by the Mastersizer 3000*

Concentration		Time (minutes)				
		0	5	10	15	20
1:30	Surface area (m^2/kg)	62160	63870	133900	169400	31020
	D50 (nm)	1.3	0.116	0.0492	0.0435	0.217
1:10	Surface area (m^2/kg)	2179	36230	2615	111.6	77320
	D50 (nm)	5.13	3.79	4.39	55.2	6.88
1:7.5	Surface area (m^2/kg)	47680	182300	67230	2648	62690
	D50 (nm)	0.218	0.034	0.122	4.38	0.115
1:6	Surface area (m^2/kg)	30.55	51050	213400	176400	120700
	D50 (nm)	204	0.13	0.0298	0.0354	0.0787
1:4	Surface area (m^2/kg)	45120	94900	74820	-	-
	D50 (nm)	0.145	0.102	0.102	-	-
1:3	Surface area (m^2/kg)	49260	75480	52020	45210	47320
	D50 (nm)	0.134	0.104	0.128	0.143	0.14
1:2	Surface area (m^2/kg)	46120	46170	47390	41260	45120
	D50 (nm)	0.143	0.143	0.14	0.156	0.143

3.2 Effect of sonication on the compressive strength

In this section, the impact of sonication in enhancing the reactivity of clay nano-particles and improving the compressive strength was observed through making a comparison between the optimum solid to liquid ratio obtained in the previous section, unsonicated nano-clay particles and sonicated nano-clay was different solid to liquid ratio than the optimum obtained. Nano-clay added as an adding material to cement with a ratio of 1, 2, and 3% of the weight of cement. The solid to liquid ratios used was 1:6, 1:4, and the third one was using unsonicated nano-clay (added as a powder). The results showed that the mix using solid to liquid ratio 1:6 gives the optimum compressive strength, which is 75.56 MPa after 28 days with an improvement of 22.6% than the control mix. While for a solid to liquid ratio 1:4, the compressive strength was 70.80 MPa after 28 days with an improvement of 14.9% than the control mix. While for the unsonicated nano-clay mix the compressive strength was 63.4 MPa with an improvement 2.9% than the control mix. Using 3% nano-clay gives the highest compressive strength regardless using nano-clay a powder or in a solution, as shown in Figures 4, 5, and 6. The small impact of unsonicated nano-clay on compressive strength than the sonicated one was attributed to the presence of a relatively large number of small size clay nano-particles agglomerates in the mix. In which longer time was needed by the unsonicated nano-clay to react with the excess CH to form the additional C-S-H gel. As for the low reactivity at the early age, the agglomeration formed by the unsonicated nano-clay acted as a filler leading to a decrease in the porosity of the matrix resulting in relatively better compaction and higher compressive strength than the control mix. While for sonicated nano-clay, the improvement for 7-day compressive strength was attributed for two reasons; the first was due to the packing effect of nano-clay particles as it acts as a filler to fill into the transitional space of the microstructure of cement mortar. While the second reason, was due to the pozzolanic effect that results due to the reaction between silica and alumina elements in nano-clay with the lime element of calcium hydroxide $Ca(OH)_2$ and oxide CaO in cement to improve the bonding strength and solid volume, this conclusion complies with Morsy et al. [14]. In addition, sonication increases the specific surface area of clay nano-particles, which leads to improving its pozzolanic reactivity and leads to increase in the strength and improving the microstructure of the nano-clay mortars, and that's will be illustrated later in the next section.

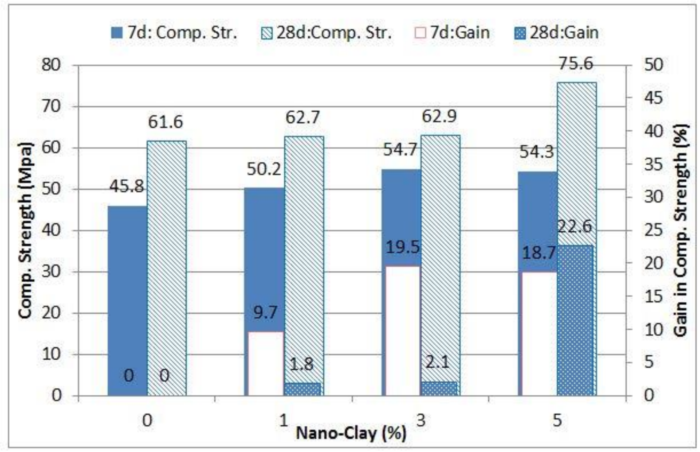


Figure 4: *7and 28-day compressive strength of unsonicated nano-clay mortars and the gain in compressive strength as compared to the control mix*

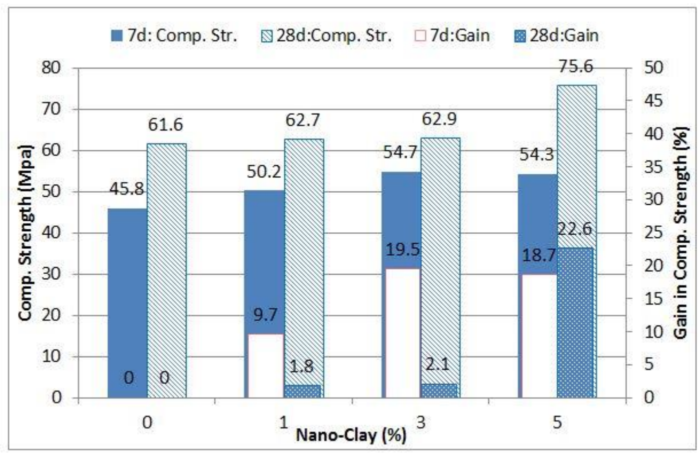


Figure 5: *7 and 28-day compressive strength of sonicated nano-clay mortars with a solid to liquid ratio 1:6 and the gain in compressive strength as compared to the control mi*

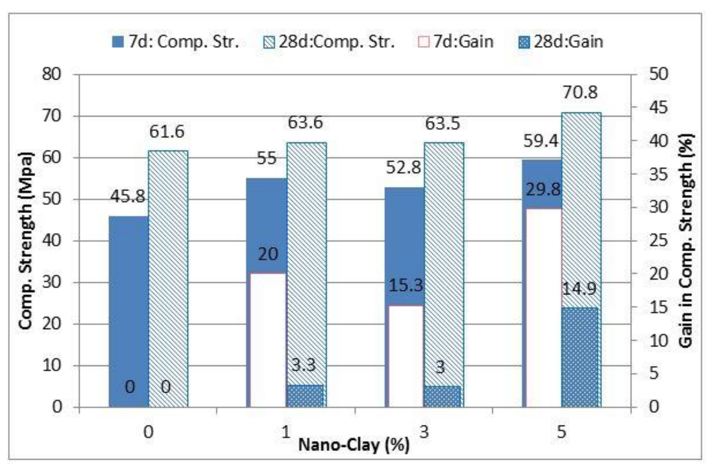
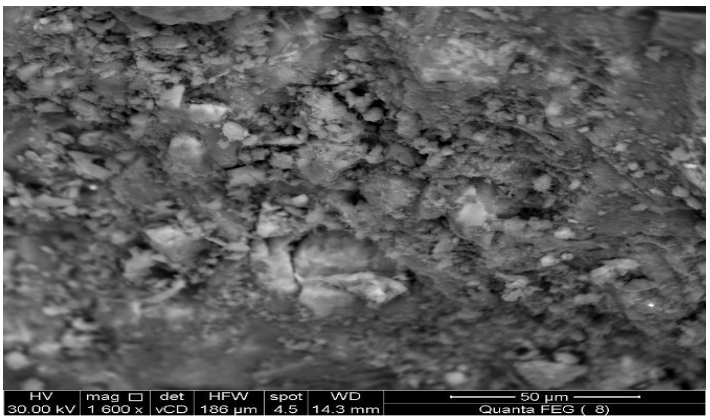


Figure 6: *7 and 28-day compressive strength of sonicated nano-clay mortars with a solid to liquid ratio 1:4 and the gain in 7-day compressive strength as compared to the control mix*

3.3 Microstructure Analysis

Microstructure test was carried out using both Scanning

Electron Microscopy (SEM) and X-Ray Diffraction (XRD) as shown in Figures 7 and 8 respectively. Figure 7 shows the SEM of mortar samples without nano-clay (a), with unsonicated nano-clay (b), with sonicated nano-clay at solid to liquid ratio 1:6 (c) with age 28 days. For the SEM analysis, when comparing the control samples with the sonicated and unsonicated nano-clay were complying with the results obtained in the compressive strength. This can be attributed to the relatively high reactivity of nano-clay particles after subjected to electrostatic force through sonication than using nano-clay as a powder or without even using nano-clay. In which the ettringite needles were clearly obvious in addition to calcium silicate hydrate plates (C-S-H) in the control mix and unsonicated nano-clay specimens. However, in the sonicated nano-clay specimens a dense structure without any pores or unreacted CH plates appears. As nano-clay can absorb the $Ca(OH)_2$ crystals and fill the voids of the C-S-H gel structure and act as a nucleus to tightly bond the C-S-H gel particles.



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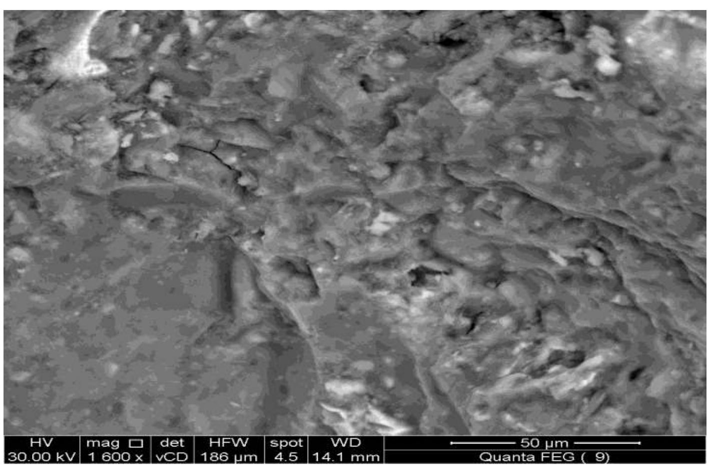

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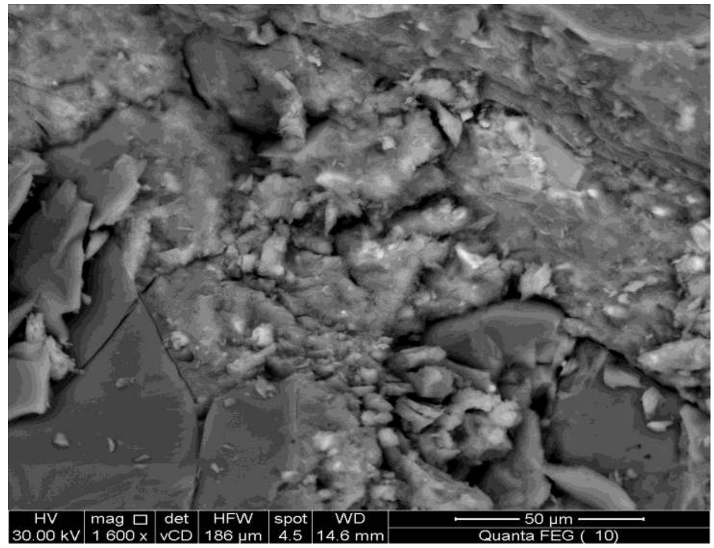
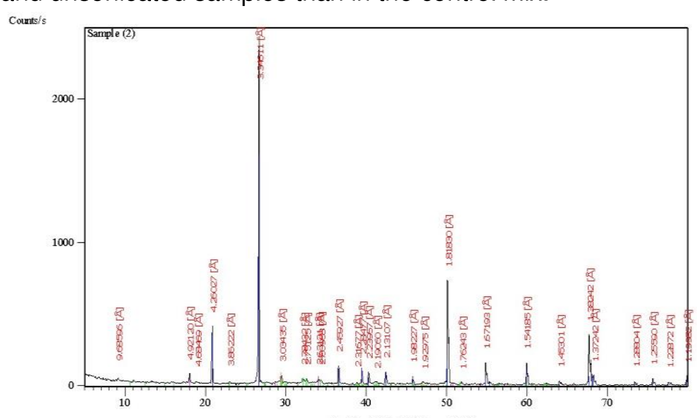


(c)

Figure 7: *SEM pictures of (a) cement mortar without nano-clay, (b) with unsonicated nano-clay, and (c) with sonicated nano-clay*

XRD analysis performed to detect changes in the hydration products due to the presence of nano-clay. From Figure 8, it can be clearly observe the impact of indirect sonication on the reactivity of nano-clay in the cement mortars. as the C-S-H content had increase, which leads to a reduction in the values of CH resulting in preventing CH crystals from growth. Also, the values of calcium carbonate had reduced in both sonicated and unsonicated samples than in the control mix.



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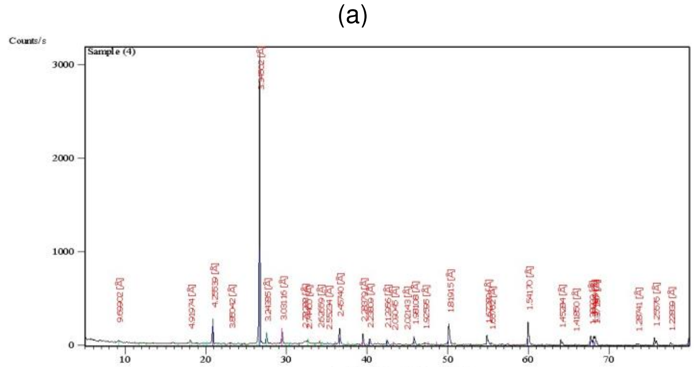

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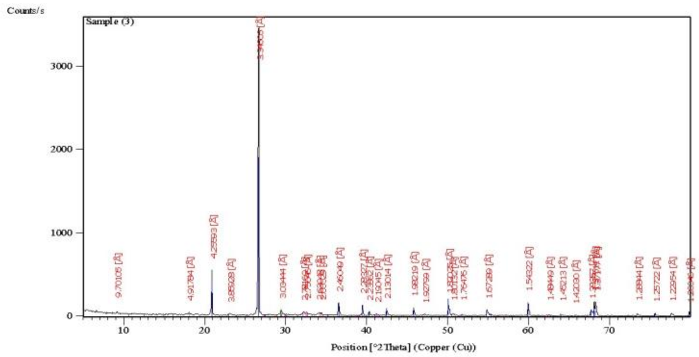

(c)

Figure 8*: XRD analysis of (a) cement mortar without nano-clay, (b) with unsonicated nano-clay, and (c) with sonicated nano-clay*

4 CONCLUSION

From the presented experimental works, the following conclusions can be drawn; the optimum solid to liquid ratio of nano-clay is 1:6 and with an indirect sonication time of 10 minutes, which results in the optimum specific surface area and particle size distribution. In addition, the re-agglomeration was minimal at this ratio. The optimum ratio with indirect sonication improves the compressive strength by 18.7% and 14.9% at 7 and 28 days, respectively. Microstructure analysis reveals that nano-clay improves the microstructure of the cement paste and produces a denser and more homogeneous matrix than without nano-clay. Sonicated nano-clay improves the microstructure of the cement matrix and consumes calcium hydroxide crystals to produce more and denser C-S-H crystals. The optimum sonicated nano-clay replacement percentage is 5% of the cement weight.

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