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Preliminary Investigation on G Cement Modified by Nano-Powder

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K E Y W O R D S

Cement dust, OWC glass G, smart cement, Water to cement ratio.

A B S T R A C T

A proper slurry design is critical to cementing work success. In the present investigation, a ball mill method was utilized for preparing a nano powder from a cement dust material, supplied via Al-Kufa Cement Factory, to reinforce the oil well cement by utilizing it as a partial replacement of oil well cement class (G) using different weight percentages (0.25%, 0.5%, 0.75% and 1%). A mixture having water to cement ratio of (0.44) was produced. The produced samples characterizations were achieved via the Atomic Force Microscope (AFM), the X-Ray Diffraction (XRD) as well as the density and compressive strength. Results showed that the structural characteristics were enhanced with the phase formation of the calcium silicate hydration (C-S-H), and both density and compressive strength were improved. Accordingly, obtained results suggest that the modified cement is suitable for the oil well uses.

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1. Introduction

Nanotechnology is neither recent science nor recent technology. Instead, it is the extension of science and technologies already present in the evolution of science for several years. In addition, it’s the logical work advancement achieved for examining the science phenomena at a smaller scale. Nearly, each feature of construction method will be contacted via the previous nanoscale technologies novelties [1]. Particles at nanoscale size represent parent materials having grain size and similar chemical structure [2]. Nanomaterials are typically used, in different applications, when their sizes range from (1) to (100) nm [3]. Nanoscale materials possess bigger surface area in comparison with the bulk of material manufactured in a bigger scale, which makes them chemically reactive as well as having electrical characteristics and strength [4]. The cement utilized in the wells of oil is classified as the class (G) cement, which is different from the Portland cement by a low amount of “celite” (C3A) and has larger grains than the Portland cement [5]. Generally, slurries of Oil Well Cement, OWC, are higher intricate compared to traditional cement. For OWC optimization (against broad range of temperature and pressure), some additives are used based on mixture combination employed such as different mineral mixtures and chemicals [5]. Cementing is the method of adding slurry of cement to the specified place in the annular between the wellbore and the casings for bonding the formation and casing, to preserve creating the formations, to avoid the migration of the formed fluid within the regions to control lost circulation. The characteristics of slurry are regarded to be significant, including; time of setting or time of pumping, strength, fluidity, density, suspension of stable particles, free fluid or free water; capability to withstand the dehydration in opposition to the permeability–loss of fluid; shrinkage, durability and permeability. The cement should last a longer time than the oil well, therefore avoiding the fluids from flowing between surface and subsurface [6].

Many investigations were carried out related to utilizing fiber and nano-powders in the well manufacturing, and their effects on the performance, properties, and the cementitious materials applications were studied as well. Ridha et al. [6] investigated that the (NG1), which is a geopolymer cement having (1%) nano-SiO_2, and highest value of strength at the curing temperatures and pressures (70°C/1500 psi and 120°C/4000 psi). The (NG3) including (3%) nano-SiO2 also reported as geopolymer cement strength, however it is not efficient as (NG1) because that the nano-SiO2 amounts in (NG3) surpass the nano-SiO_2 optimal content affecting mechanical properties of cement samples. Under high pressure and high temperature (HPHT), both (NG1) and (NG3) have high strength value compared to the class (G) and pure geopolymer cement. The mass loss outcomes were linear with the compressive strength, where (NG1) and (NG3) increased by the values of mass at the (HPHT) state. Choolaei et al. [7] studied the effect of nano-silica on the physical properties of oil well cement and showed that the nano-SiO2 acts as filler to enhance the microstructure of the mortar cement but also as a pozzolanic reaction promoter. In addition, nano-silica can be regarded as an agent for enhancing the cement paste microstructure. Therefore, it’s potential to add the particles of nano-SiO2 to the mixture of cement in order to achieve higher performance. Moreover, the addition of the nano-SiO2 reduces the lubricating features of the mixture. Further, fewer differences in plastic viscosity, as well as a decrease in the dormant period, were observed when nano-SiO2 is added.

In 2017, Ragehet et al. [8] performed a cement for a test at less than (25ºC). In this investigation, cement grade (G) was used alongside the retardants and accelerators of which these constituents were employed at specific levels. The viscosity, thickening time, water content, rheological characteristics, loss of fluid, and compressive strength were determined. After a couple of days, it was noted that the addition of the accelerator, retarder, weighting agent (1.83, 1.74, 1.67 g) makes the thickening time as 94, 109, 123 minute and the compressive strength (at 140 F and 24 hours) as (2530, 2187, 1600 psi).

In Iraq, there are two studies on the effect of the nano-material addition to the cement slab on the density, compressive strength, and time of thickening. In 2017, H. A. Hadi et al. [9] investigated the effect of Nano silica and Nano alumina on the uniformity and strength of the OWC. In this study, cement powder Glass”G” (HSR cement), supplied by the Company of Missan Oil, was used. The nano Silica and nano Alumina were used as additives to the alumina slurry at a percentage (1% and 2%) of nano-silica and at a percentage (1%, 2% and 3%) of nano alumina. The cement slurry was placed within laid out moulds, and samples were put inside a curing bath at the atmospheric pressure, heated at the test first temperature (38°C) for (8 hours) and then at the test second temperature (60°C) for (8 hours). Obtained results showed that the compressive strength of the cement of oil well at (38°C) increased when the quantity of (NAL) and (NS) is increased. Such enhancement in strength was due to the adhering effect of nanoparticle's with the particles of cement; therefore increases the efficiency of packing. The other cause is owing to the (NAL and NS) induced effect of the pozzolanic interaction. These reasons explain that it reacts chemically with $Ca(OH)_2$ (developed from the dehydration interaction) in water and creates complexes with the cementitious compounds. At (600ºC), the compressive strength reduced when the NS quantity of surpasses is 2% bwoc. Such a result explains that increased value of NS is associated with the compressive strength to certain cut off; however, a further increase in NS could result in a reduction in the compressive strength. This is due to the truth that the rate of adding NS can fasten its particles agglomeration under the current scattering condition. Some studied showed that nanomaterials (NAL and NS) speeded up the time of thickening of the cement class “G” slurry. For example in 2014, A. A. Abdulrahman et al. [10] investigated the nano-silica characterization obtained from the Iraqi rice husk and looked for its use in the OWC. This study aimed to produce nano-silica from Iraqi rice husk using two precipitation chemical approaches. In addition, these authors studied the OWC class "G" compressive strength through using various nano-silica percentages. The utilized cement class "G" in such investigation was obtained from the Company of South Oil, the nano-silica was utilized like additive. For the slurry of the cement, the effect of nano silica addition on OWC class "G" for (8 hours) at temperatures (380ºC) and (600ºC) is described in the (API) specification (10A) for the cement and the materials for the cementing of well. Obtained results showed that at 8 hours, the compressive strength (in psi) at 380^0C in the absence of nano silica was 879, while with the nano silica was 953, 963, and 919. Also at 8 hours, the compressive strength at 600ºC without the nano silica is (2090) and with the nano silica are 2094, 2306 and 2262. It's clear that the compressive strength increased by the increase of the nano silica percentage. However, increases in additive amount caused a reduction in the compressive strength (e.g. addition of (2.5%) nano silica). Such result could be explain that the silica may be regarded as lighter agent, so this specification was shown owing to the particles fineness and agglomeration resulted via the paticles dispersion difficulties through the mixing. Therefore, raising the nano silica amount decrased the density of the slurry, which led to reduce in compressive strength.

The objective of the present work is first to produce a nanopowder from a cement dust material supplied by Al-Kufa Cement Factory to inforce the oil well cement via using the manufactured nano powder as an (OWC) partial replacement. Secondly, characterize the produced samples using the SEM observations, XRD and AFM. Finally study the compressive strength for 8 hours and the test of density of the slurry of OWC class "G" at various percentages of nano cement dust to improve the cement and its mechanical properties for making it convenient for the characteristics of well.

2. Experimental Part

This part of study was developed for investigating the influence of the addition of the nanoparticles on the physical, mechanical, and structural properties of the cement class "G. The oil well cement was used in this research as class G, w/c ratio was made 0.44, and cement dust (CD) was used as additive (Figure 1 and 2).

Cement class mixtures were performed using nanomaterials, which was (nano cement dust) in four concentrations (0.25%, 0.5%, 0.75%, and 1%) per weight. To perform such steps, the samples developed for the OWC class "G" have to use cement with a weight of 792 g and water with a weight of 349 g, in accordance with the American Petroleum Institute (API, 1997) in nine classes (from A to J). The nanomaterials percentages were 0.25%, 0.5%, 0.75%, and 1% of weight of the material of cement. The cement of class "G" with the nanomaterials (cement dust) was compared with a reference mixture to evaluate the influence of the nanomaterials dosage. Table 2 Shows Mix Proportions.

Table 1: Physical properties of oil well cement

	Property	Standard API
1	Density, gm/cm^3	1.9 gm/cm^3
2	Free water, %	5.9%
3	Thickening time, min	(90-120 min)

Figure 1: Cement class G

Figure 2: Nano cement dust

Table 2: Mix Proportions of Cement Glass G specimens

Sample	Cement gm	Cement dust gm	Water
1	792	-	
2	790.02	1.98	
3	788.04	3.96	
4	786.06	5.94	0.44
5	784.08	7.92	

Where, the percentages of nano cement dust added to the cement by weight of cement are as follows:
- 0.25 % nano cement dust.
- 1% nano cement dust.

The required mass of mix water for class G should be 349 g put in the mixing container as shown in Figure 3. The nano cement dust was first added to it, and then mixing at 4000 rpm. While the sample of cement was added at a consistent rate but not higher than (15 sec).
After the whole cement was added to the mix water, the mixing must be done at 12000 rpm for 35 sec; the slurry was placed in prepared moulds to fill them to overflowing. The excess slurry was struke off using a straight edge, and a clean dry cover plate was placed on the top of the mould, as shown in Figure 4.

Fig. 3: Mixing cement slurry

Figure 4: Cement cubes with slurry

The test of chemical composition for the cement class “G” was conducted at the labs of Iraqi Geological Survey in the Ministry of Industry-Baghdad, Iraq, as given in Tables 1. The test of density was performed in accordance with (ASTM C-373) standard [11], and the true density (ρt) was found via establishing a model in water (Archimedes rule) using this equation [12-13]:
Where:
ρt: The true density (gm/cm3)
D: The distilled water density (1gm/cm3)
Wd: The sample dry weight of sample (gm)
Ws: The sample weight (gm) beyond the saturation in water
Wn: The sample weight (gm) when submerged in water
The (AFM) was used with digital devices; distinctive values of Roughness and Root Mean Square (RMS), model (AA300220V) and were recorded The test of compressive strength for samples in the form of cubes was conducted by utilizing a universal measuring machine (EVERY DENISON) with a capacity of (2000 KN). The mean result of (3) specimens was recorded for every sample, and this relation was employed:
$\sigma = P/A$
Where:
P: The maximum compressive load in N
A: The sample area in mm2
σ: The compressive strength in MPa
The XRD was obtained by diffract meter with the radiation (CuKa) having (λ = 1.54060 A°). Such test was performed in the (Center of Nano Technology and Advance Material Research) at (University of Technology). Also, the test of the cement dust powder particle size was also carried out in this Center, using the device (Model: Brookhaven Nano Brook 90 plus USA), and the size of the particle was found (100 nm). Table 3 Shows the Chemical composition of raw materials.

Table 3: Chemical composition of raw materials (Cement Class “G”)

Material	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	LOI
Weight%	30.38	2.61	5.95	0.33	42.64	3.80	1.840	7.97

3. Results and Discussion

I. Density result

Figure 5 shows the prepared samples density. Nanopowder takes the role of the filler to improve the density of the sample that can reduce the mixture porosity. On the other hand, the powder can speed up the hydration of cement via virtue of their free energy. The samples density is shown in Figure 5.

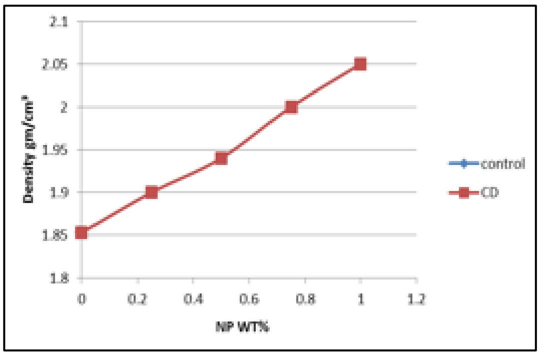

Figure 5: The Density of the prepared samples

II. Compressive strength result

Figure (6) shows the compressive strength. Figure (6a-6b) illustrates the compressive strength and the effect of adding nanopowder to OWC class G for 8 hr at the temperatures (38°C) and (60°C) as defined in the (API) Specification (10A) for the cement and materials for the well cementing. At temperature (38°C), the compressive strength of the control sample was (582 psi), while the addition of (1%) of nanopowder (cement dust) has improved the compressive strength by (15.2%). At temperature 60°C, the compressive strength of the control samples was (1578 psi) while the addition of 1% of nanopowder (cement dust) improved the compressive strength by (1.2) %. The discrepancy in the development of mortars' strength could be attributed to pozzolanic reactions between the cement and the nanopowder. Nanopowder becomes more active in the pozzolanic reactions as the pozzolanic reaction rate is proportional to the surface area for the reaction. Therefore, it's well recommended to use high purity of nanopowder as well as Blaine's fineness to enforce the cement mortars properties. Further, nanopowder could recover interspaces within cement resulting to increase of the strength of mortar. Therefore, it can be suggested that the addition of nanopowder to the cement mortars enhances their characteristics of strength. Accordingly, mortar strength was obtained as a result of an increase in the content of nanopowder rose. Figure (6a-6b) illustrates the compressive strength fluctuations.

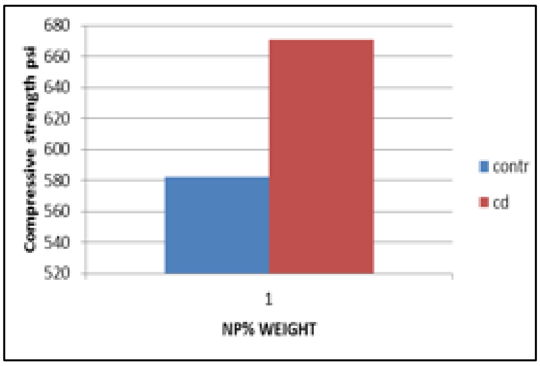

Figure 6a: The prepared samples compressive strength at 38^0C

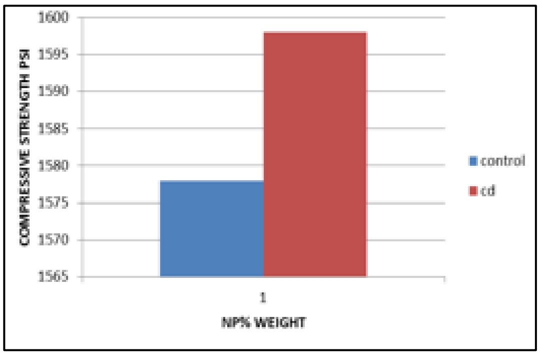

Figure 6b: The prepared samples compressive strength at 60^0C

III. Atomic force microscopy result

Atomic Force Microscopy is the technology for investigating the morphology of a surface at Nano to micro-scale. The mortar surface is shown in Figure (7) in (3D) surface, the mortar surface roughness without the cement dust addition was 18.79 nm, while by the adding of nano cement dust (0.25 wt%), the roughness was (15.38 nm), and with (1%) cement dust, it was (20.17 nm). Such differences depend upon the cement replacement rate with the nanopowder. From the (AFM) test, it can be noted that the mortar without the addition of cement dust has an average diameter of (103.01 nm), while with cement dust (0.25 wt%) the average diameter was (184.82 nm), and with (1%) cement dust it was (158.30 nm). Table 4 displays the (AFM) results of class "G" merely and with the additives.

IV. Result of X-ray diffraction

The cement mortar (XRD) pattern having no nanopowders is shown in the Figure (8a). In the cement mortar (after the time of curing) of the (reference and blended specimens), the phases' components shown in the Figure (8-b) were Portlandite: c-hexagonal crystallized, $Ca(OH)_2$, and JCPD (04-0733)/(CH); Ettringite: hexagonal crystallized, $Ca_6Al_2(SO_4)3(OH)$ $12.26H_2O$, and JCPDS (41-1451) (CASH); and Tobermorite: ort horhombic crystallized, Ca_5Si_6 (O, OH, F) $18.5H_2O$, and JCPDS (45-1480) (Tob). Moreover, the prevailed phases were Calcium Silicate Hydrate: Poor crystallized, crystallized, JCPDS (34-0002) (CSH), and $CaO.SiO_2.H_2O$; and Wollastonite: d-monoclinic crystallized, JCPDS (43-1460) (CS), and $CaSiO_3$. It was noticed that the portlandite and ettringite are included in the hydration whole stages. Additionally, the variations in the mineralogical components during the operations of hydration were observed, where the hydro-aluminates and the hydro-silicates existed; (tobermorite, ettringite, and portlandite), and the maximum peaks match to the calcium silicate hydrated (C-S-H) gels and the portlandite (CH) [14]. The characterization of (XRD) was conducted to investigate nanopowders activity with the specimens of cement mortar after the hydration. From the patterns of (XRD), it is obvious that the peaks of (CH) are almost reduced with blending via the (cement dust) nanopowders; so it has concluded that the nanopowders can react with the manufactured (CH) through the hydration. Thus, the nanopowders reactivity is important and has evolved the microstructure of the cementitious material, therefore it has improved the cementitious materials' mechanical properties, and the (CH) compound possesses potential in the cement mortar working as the pure product of hydration released from the cement. Clearly, the (CH) intensity is decreased when the cement is replaced by nanopowders Figure (8-b), which indicates the (CH) use up by pozzolanic reaction [15].

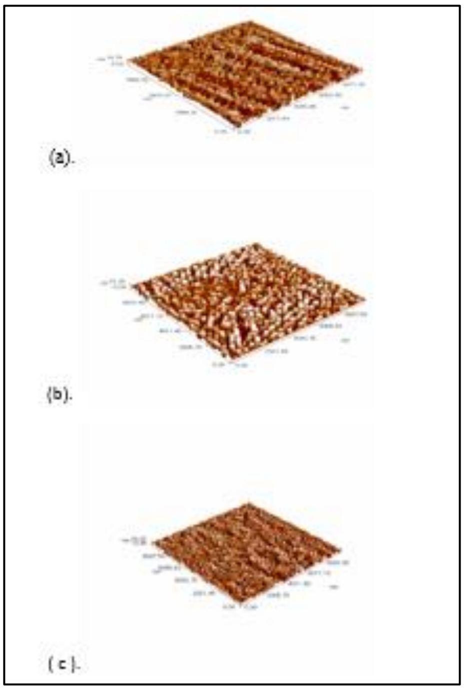

Figure 7: The AFM results of cement class "G": (a) Cement without addition, (b) Class "G" with 0.25% cement dust, and (c) Class "G" with 1% cement dust

Table 4: The (AFM) results of cement Class "G" without and with additives

Percentage, %	Roughness, nm	Diameter, nm
Zero sample	18.79	103.01
0.25% CD	15.38	103.85
1% CD	29.97	110.74

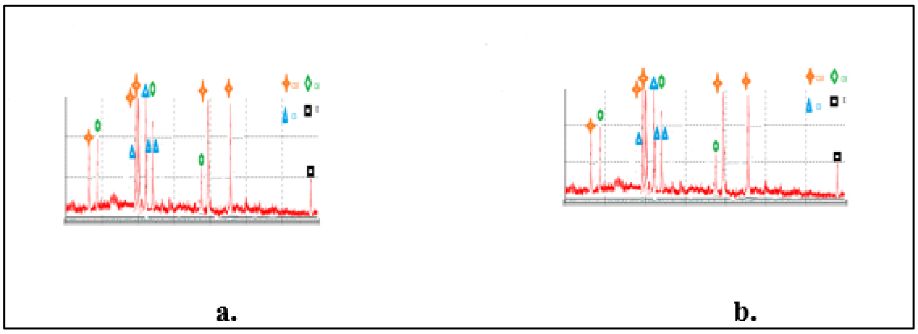

Figure 8: The pattern of the (XRD) of "G" cement mortar: (a) Without nanopowders, and (b) With 0.75% CD

4. Conclusion

The application of nanopowder as partial replacement of the Portland cement in mortar resulted in an increment in density and an increase in compressive resistance and increased the pozzolanic interaction, while decreased the surface roughness with increasing the cement dust, which contributes to solving a fundamental problem.

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