

## **PREPARATION AND CHARACTERIZATION OF NANO- SILICA FROM IRAQI RICE HUSK WASTE WITH APPLICATIONS TO CEMENT USED IN OIL WELLS**

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### **Abstract**

Nano-silica was produced from Iraqi Rice husk (IRH) waste by precipitation method. The prepared silica nanoparticle samples were characterized by different analytical techniques, these included FTIR, XRD, SEM, and AFM. Moreover, nano-silica pozzolanic material was used for the first time in Iraq as cement admixture in oil field cement to improve compressive strength. The results show nanosilica cement admixture compressive strength was increased by 8%, 9% and 4% at 1.5%, 2%, and 2.5% nano-silica percentage addition respectively by weight of cement at 38 °C and 0.1%, 9%, and 7% compressive strength percent increase at 60 °C.

**Keywords:** Rice husk waste; Nano-Silica; Oil well; cement -admixture.

### **1. Introduction:**

Nano-silica  $\text{SiO}_2$  has found different applications as a filler in rubber and plastics, nano-composites, absorbent, drying powder, substrate for catalysts and anticorrosion agent [1-3]. There are different methods in the literatures which have been reported for preparing silica nano-particles, such as chemical vapor deposition [4] sol-gel processing [5-7], micro emulsion technique [8]. A variety of methods have been inspected for recovering amorphous silica from rice husk [9] and rice husk ash [10].

The production of reactive nano-scale silica from rice husk (RH) waste is a simple process compared to other conventional production techniques such as vapor phase reaction, with such a large availability of an inexpensive waste raw material, it becomes an economical method with high purification percent, high surface area, high reactivity, and amorphous in form. This economic method, as applied to waste material, also provides many advantages to the local agro-industry [3-11].

Several authors have reported preparing nanosilica from rice husk. Pukird et al. used rice husk ash and coconut shell charcoal to prepare silica nano-wires by thermal evaporation at a temperature of 1350 °C with the ratio of 1:2 by weight in atmosphere of nitrogen with diameters varying from 40 nm to 200 nm and a few micrometers length.[12]. Adam et al. synthesized mesoporous spherical silica nanoparticles from rice husk by a simple, template-free synthetic solvent extraction and sol-gel technique at room temperature (30 °C) with high surface area of (245 m<sup>2</sup>/g) and narrow pore size distribution (5.6–9.6 nm) with an average diameter of 50.9 nm.[13] Noushad et al. synthesized nano silica from rice husk by precipitation method using different acids, namely, orthophosphoric acid and nitric acid to extract silica, it is possible to directly obtain superfine silica particles from rice husk in spherical form ranging from approximately 70nm to 400nm when either acid is used in conjunction with polar solvents during the precipitation process. [14] Rafiee et al., produced a nanosized amorphous highly purified silica with a high surface area, high reactivity, by various chemical treatments under varied conditions of RH followed by thermal combustion under controlled conditions which gave 22.50% ash of which 90.469% was silica, this nano-silica showed strong and a large number of acidic sites in comparison with commercial silica, making it as a good support for catalysts. [3] Hariharan *et al.* synthesized nano silica by burning rice husk at 650° C for 2 h to yield amorphous almost spherical, homogeneous and agglomerated form of 90 nm in size and 99% purity[15].

Oil well cement (OWC) slurry is generally more complicated than that of conventional cement paste, In order to contend with bottom hole conditions (wide range of pressure and temperature), a number of additives are usually used in the OWC slurries, which exhibit different characteristics depending on the combination of admixture used incorporating various chemical and mineral admixtures used [16]. Several researchers have proposed that nano-particles can be incorporated into conventional building materials in order to improve some physicochemical properties for instance, silica nano-particles are used as an additive to high performance and self-compacting concrete [17]. Nano-silica is typically a highly effective pozzolanic material. It normally consists of very fine vitreous particles approximately 1000 times smaller than the average cement particles. It has proven to be an excellent admixture for cement to improve strength and durability and decrease permeability [18-19].

In this work nano-silica was prepared from Iraqi rice husk from Al-Najaf Province in Iraq by precipitation method. Various techniques were used to characterize the prepared nano-silica. The application of nano-silica as additives to Iraqi oil well cement type G slurry was carried out for the first time and the compressive strength of oil cement admixture was studied.

## 2. Experimental Work

### 2.1. Materials

The samples of rice husk used in the study were obtained from Al-Najaf Province in Iraq. Sodium hydroxide was obtained from (LoBAChemie) India, Hydrochloride acid (37%, analysis grade) from (Thomas Baker) India, Nitric acid HNO<sub>3</sub> from (BDH) England.



## 2.2. Pretreatment of the Rice Husk Ash (RHA)

RHA was obtained after washing rice husk with distilled water and dried, and then calcinated in a muffle furnace at 700 °C and 1000°C, and then 700 °C for 6 h. which was selected as ashes temperature.

## 2.3. Preparation of Nano-silica

After soaking the rice husk in distilled water overnight, it was washed thoroughly with distilled water, to remove impurities. Then, it was dried at 90°C. The dried rice husk was treated with (1N) HCl solution for 1 h at 75°C. The solid residue was washed thoroughly with distilled water, dried at 90°C. 40 g of acid treated rice husk was immersed in 600 ml of sodium hydroxide solution and heated for 1 h at 90 °C. The resultant solution obtained after filtration of rice husk was sodium silicate solution. Nitric acid was added to sodium silicate solution until its pH dropped to 8 and further stirred for another 45 minutes. The solution was centrifuged for 5 minutes at 4000 rpm and this process was repeated until whitish pellets were obtained. The pellets were calcinated at 600 for 30 min [14].

## 2.4. Acid Leaching Treatment

The metallic constituent has a considerable effect on the quality of silica produced from RH; also an efficient interaction takes place between the metallic ions and silica which leads to a considerable decrease in the surface area. Therefore it is preferred to treat RH with a prime acidic solution such as HCl.

## 2.5. Cement and Nano-silica Admixture

Cement slurry was prepared in this way: the required mass of water was placed in the mixing container, then different percentages of nano- silica (1.5,2 and 2.5%) were added to it then the lot was mixed at 4000 rpm while the cement sample was added at uniform rate in not more than 15 sec. When the mass of cement was added the mixing continued at 12000 rpm for 35 sec the slurry was transferred then to the mould .The curing of the specimens was carried out in curing bath preheated to the test temperature of 38o and 600 C respectively. Specimens cured at previous temperatures were removed from the curing bath 45 min.before testing began. They were removed from their moulds and transferred to a water bath and maintained at 2700C for 40 minute. Specimens were placed in the testing machine to measure compressive strength [19]. The silica samples for compressive strength measurement were prepared according to API spec 10A.ISO10426-1 [17, 20].

## 3. Result and Discussion

### 3-1 Chemical Analysis of Rice Husk Ash

Ashing in general is carried out to increase silicon dioxide percent, remove undesirable component (metallic ingredients) and reduce carbonaceous materials. (RHA) when burned under controlled atmosphere, amorphous silica is obtained which is highly reactive in nature, thus quality of (RHA) is dependent on diverse parameters such as ashing temperature and time, rate of heating. Quantitative chemical analysis results of rice husk ash (RHA) by XRF are shown in Table (1).

Table (1) it's clearly seen that although high percent of SiO<sub>2</sub> produced at (1000 oC) is (95.16%) this temperature was not selected as ashing temperature. This is due to crystalline structure of nano-silica produced; using of high temperature causes surface melting and accelerates the crystallization of amorphous silica and carbon fixation. Also strong interaction occurs between metallic ingredients and silica which leads to a considerable decrease in surface area [3]. Consequently ashes temperature at (700oC) was selected, due to amorphous structure of nano-silica which is obtained .This nano-silica has a pozzalonic activity which is necessary when used in cement and concrete [21]. The results are in good agreement with results obtained by [3, 22]

### 3-2 Acid Leaching Treatment

Quantitative chemical analyses of nano-silica after the acid leaching treatment are shown in Table 2. It is clearly seen from this table that the thermal and acid treatments are efficient resulting in high reduction in Al<sub>2</sub>O<sub>3</sub>, CaO, MgO, the silica (SiO<sub>2</sub>) percent in the RHA increases from 94.2% to 98.06%. This acid reacts chemically with metallic impurities, the reacted metals leached from acidic solution trough filtration. Thus effective removal of impurities occurs. While acid leaching affects the chemical composition of the husk, it does not affect the structure of the nano-silica obtained, whether crystalline or amorphous. Therefore, there is no change of phase from amorphous to crystalline; the pretreatment method employed has not affected nano-silica produced. [22, 23].

### 3-3 Characterization of Nano-silica

#### 3-3-1 FT-IR Spectra for Nano-silica

A-FT-IR spectra of the powders prepared from pure silica nano-size particles are shown in figure 1; the predominant peaks at 801 and 1076 cm<sup>-1</sup> are attributed to the symmetric and asymmetric stretching modes of (Si-O-Si) respectively. No peak was found between 1800 and 2300 cm<sup>-1</sup>It means that there were no original organic compounds in the silica after controlled combustion and extraction. Our results are in good agreement with results obtained in work by [24].

#### 3-3-2 X-Ray Diffractograms

X-Ray diffract diagrams for nano-silica are displayed in figure 2; a strong broad characteristic peak of nano pure silica at  $\approx 22^\circ$  (2 $\theta$ ), which shows the amorphous nature of the prepared nano-silica particles. The lack of sharpened peaks confined the deficiency of orderly crystallize configuration in the primed RH-nano-silica, the result obtained here are in agreement with the result obtained by [24, 25].

#### 3-3-3 Surface Area and Pore Volume

It is concluded that the amorphous nano-silica particles are of high purity, relatively high surface area of 286.4 m<sup>2</sup>/g and pore volume of 0.233 cm<sup>3</sup>/g .Silica with this specification (high surface area) is suitable compound to be used as support for catalyst or as adsorbent. On the other hand the reactivity of silica is directly related to its surface



area. The high surface area and pore volume are due to fact that the organic matter has been broken up during the thermal decomposition of RH therefore leaving a highly porous structure. The increasing pore volume in nano-silica particles confirms this phenomenon.

### 3-3-4 Scanning Electron Microscope for Nano -silica (SEM)

SEM images were utilized to identify the size, shape and dispensation of the nano-silica. Figure 3 shows the SEM image of synthesized nanostructures products prepared by precipitation method from rice husk. Nanosized silica with a mean diameter of ~50 nm is identified in highly agglomerated form, due to Van Der Waal's forces into larger aggregated nano-particles. This distinct capability for aggregation which is extant in the particles is due to the Van-der-Waal forces acting between the specific particles. Agglomeration is due to the hydrogen bonding between silanol groups on the surface. Thereafter rice husk SiO<sub>2</sub> nano-particles have to a large extent the shape from random aggregates to spherical nano-particles. As can be seen in the results obtained the particle size of nano-silica is not uniform (similar) and various primary particles appear to gather or coalesce at their faces. These results agree well with the result obtained by [6].

### 3-3-5 Atomic Force Microscopy (AFM)

Figure 4 shows the AFM topography which was studied with (496 - 499) pixel density for nanosilica by precipitation method indicates that particles in spherical form. Figure 5 shows a three-dimensional surface profile atomic force microscope observation identifying the diameters of prepared nano-silica. Particle size distribution shown in Table (3) indicates that nano-silica is in range of (60-120 nm) and with average diameter of 88.87 nm.

Results obtained from particles size distribution are shown in Table (3) and Figure 6, the most volume percentage is 13.39 % of particle size 80 nm.

### 3-4 Composition of Cement

Tables (4) and (5) show the physical properties and chemical composition of oil well cement class G used in the experimental work based on API standard. These properties indicate that this cement is suitable to be used since it is in the successful range of API specification standard [20].

### 3-5 Characterization of Nano-silica Admixture

#### 3-5-1 Scanning Electron Microscope for Nano-silica Admixture

Figure 7 and 8 show the SEM micrographs of the cement mixture; it demonstrates porous structure that is full of large size pores with presence of Ca (OH)<sub>2</sub> that is over-shadowed. Also it can be seen from the same figures the existence of many CH crystals is connected to the C-S-H gel which indicates that the hydration process is not completed and also explains the low results of compressive strengths for the blank mixture. Also, the same photos show that the concentration of the CH is higher than the

C-S-H gel concentration and that the CH hydrate needles cover a large area. These results are in good agreement with the results obtained by [24].

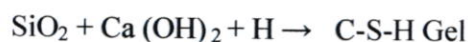
The SEM images shows admixture with added nano-silica as shown in figures 9 and 10 that is prepared with 2% nano-silica of nominal particle size (50) nm. These figures show that the microstructure of the cement mixture after incorporating (NS) is dense and more organized with a small number of  $\text{Ca}(\text{OH})_2$  crystals and small sized pores as compared with the blank mixture. The C-S-H gel exists in the form of clusters lapped and jointed together by many CH needles hydrates, it can also be noticed from the same photo that the CH needles are visible and there is a compact structure with the absence of the un-hydrated crystals and voids and more uniform and the structure is homogeneous than that of the blank sample, which explains the superior compressive strength results. This could be due to the high activity of many particles that promote the pozzolanic reaction to produce more C-S-H gel in order to record high compressive strength which is confirmed by the strength results, that is in good agreement with results of [24].

### 3-5-2 XRD for Cement and Nano-silica Admixture

Figures 11 and 12 show XRD oil well cement (OWC) before and after adding nano-silica particles. Calcium hydroxide (CH) was found out, at 17.90, 47.10 and  $\text{C}_3\text{S}$  was found out at 28.80, 32.40, and 33.70. It is clear from Figure 12, calcium hydroxide (CH) is almost decreased after the addition of nano-silica particles and can be concluded from that these nano-particles react with (CH) during the hydration process. However more alite ( $\text{C}_3\text{S}$ ) and belite ( $\text{C}_2\text{S}$ ) known as contributors to late strength of cement pastes appear after incorporating of 2% nano-silica under ambient conditions as it is inferred from figure 12 which indicates extra quantity of C-S-H is produced. The pozzolanic reactivity of nano-silica particles at early stage of hydration is obviously high and improves the microstructure of OWC system, thereby enhancing the compressive strength of OWC.

### 3-6 Compressive Strength Test of Cement

Table (6) shows the affect of adding nano-silica particles on compressive strength of oil well cement class G. The test was carried out for 8h at temperature 38°C and 60 °C as specified in API specification (10A) for cements and materials for well cementing [20]. Table 6 shows that the maximum compressive strength of oil well cement class G is obtained when 2% nanosilica particles are added to the cement. Nanosilica particles can block the pores in the (C-S-H) gels, behaving as a nano-filler. In addition, the amount of (C-S-H) increases as a result of the pozzolanic reaction of nano-silica with calcium hydroxide, which improves the strength and durability of the material [26].



A cement paste is comprised of C-S-H gels, pores. These pores represent spaces that accommodate nano-silica particles, consequently increasing the compressive strength of



pure cement paste; however, as nano-silica particles are too facile to accumulate because of their great surface energy, thus a large quantity of these particles cannot be uniformly displaced.

It is obviously noted that compressive strength increases with increasing the percentage of nano-phase additives in this case, nano-sized silica, but increase in amount of nanosilica decreases the compressive strength as it occurs when 2.5% nanosilica is added. This is attributed to nanosilica which is considered as a lighten agent, therefore it is shown this condition occurs as a result of fitness of the particles and accumulation attributed to the problems of dispersing the particles during mixing, thus intensifying the quantity of nano-silica percentage lowering slurry density which finally leads to lower compressive strength of cement. [17]

#### 4. Conclusions

Nano-silica was successfully prepared from abundant Iraqi rice husk by precipitation method, such a method is a simple and effective route for preparing particle in nano-scale. The characterization of Nano-silica by SEM, AFM and FT-IR shows the ultrafine products of nanosilica take the form of spherical particles and agglomerates with amorphous structure and high specific surface area of  $286.4 \text{ m}^2/\text{g}$  and pore volume of  $0.275 \text{ cm}^3/\text{g}$ , this leads to incorporation of cheap nano-silica produced at low cost and used in various applications such as oil well cement (OWC). Moreover, reaction of nano-silica particles with CH crystals of cement produces higher amount of C-S-H which in turn increases the strength and durability of the cement. The nanosilica cement admixture compressive strength is increased by 8%, 9% and 4% at 1.5%, 2% , 2.5% nano-silica percentage addition respectively by weight of cement at  $38^\circ\text{C}$  and 0.1% , 9% , 7% compressive strength percent increase at  $60^\circ\text{C}$ .

#### Acknowledgements

We would like to express our thanks to the Department of Chemical Engineering, University of Technology/ Baghdad, Iraq for their financial support.

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**Table 1.** Quantitative chemical analyses of rice husk ash (RHA)

	T (°C)	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	CaO (%)	SO <sub>3</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	MgO (%)	K <sub>2</sub> O (%)	Loi (%)
	700	94.2	0.15	2.47	0.15	0.5	0.57	0.67	0.28
From	1000	95.16	0.37	2.33	< 0.07	0.3	0.2	0.5	0.01

**Table 2.** Nano-silica quantitative chemical analyses

SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	CaO (%)	SO <sub>3</sub> (%)	MgO (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	Loi (%)
98.06	0.05	0.19	0.08	0.04	0.02	0.22

**Table 3.** Particle size distribution for nanosilica by precipitation method

Di (nm)<	V (%)	Acc. (%)	Di (nm)<	V (%)	Acc. (%)	Di (nm)<	V (%)	Acc. (%)
60.00	2.36	2.36	85.00	7.09	44.09	110.00	3.94	81.89
65.00	6.30	8.66	90.00	9.45	53.54	115.00	11.02	92.91
70.00	5.51	14.17	95.00	9.45	62.99	120.00	7.09	100.00
75.00	9.45	23.62	100.0	8.66	71.65			
80.00	13.39	37.01	105.0	6.30	77.95			

**Table 4.** Physical properties of oil well cement

Property	API	Standard	API Result
Density		1.9	1.82
Free water		5.9	3.75
Thickening time		(90-120)	96

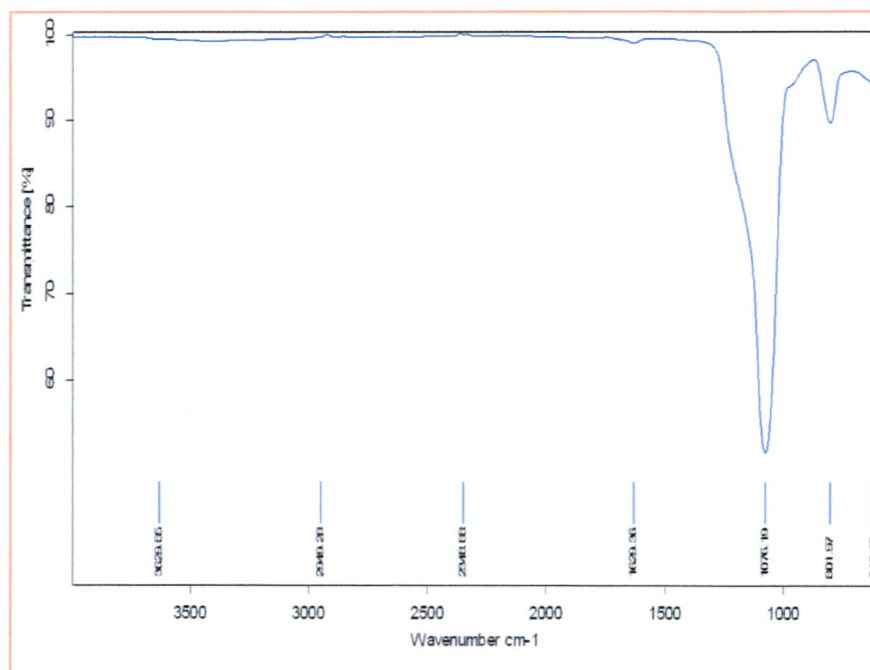


**Table 5.** Chemical composition of oil well cement

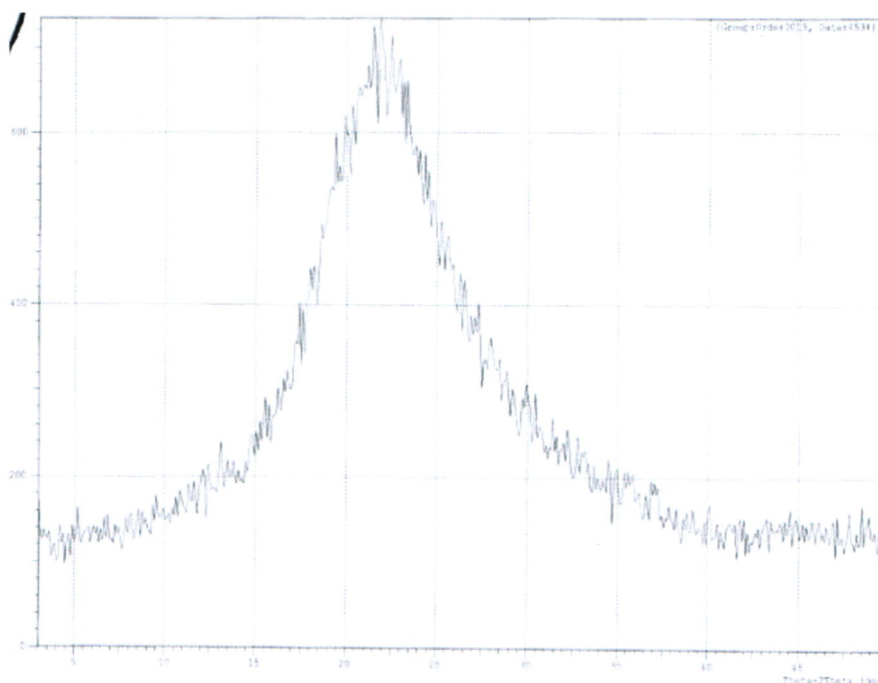
Component	API standard	API result
MgO	6	0.2
SO <sub>3</sub>	8	1.9
Na <sub>2</sub> O	0.75	0.52
C <sub>3</sub> S	65	61.33
C <sub>3</sub> A	3	1.72
Tetra- C <sub>3</sub> AC <sub>4</sub> AF +2 C <sub>3</sub> A	24	21.8
Insoluble residue	0.75	0.25
Loss on ignition	3	2.1

**Table 6.** Compressive strength tests

Compressive strength	Without nano-silica	With nano-silica		
		1.5%	2%	2.5%
(8 h psi and 38 °C)	879	953	963	919
(8 h psi and 60 °C)	2090	2094	2306	2262

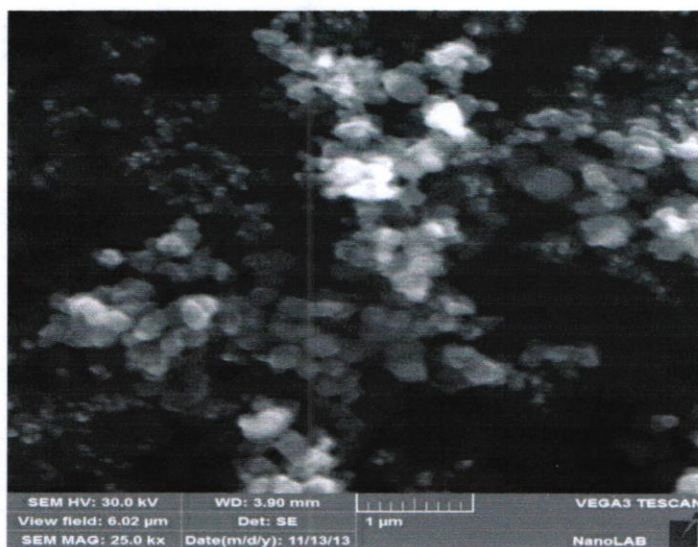


**Fig.1.** FTIR spectra of the nano-silica particles from (RH)

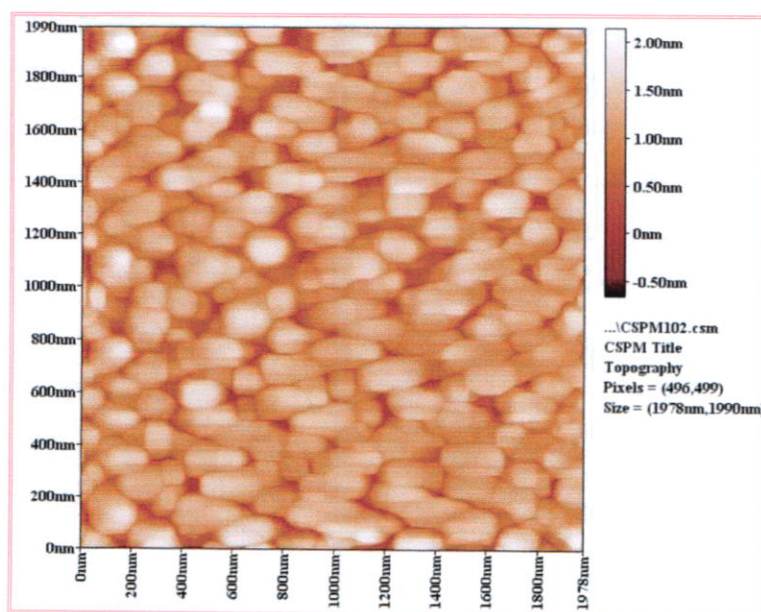


**Fig. 2.** XRD diagram of the nano- silica particles from (RH)

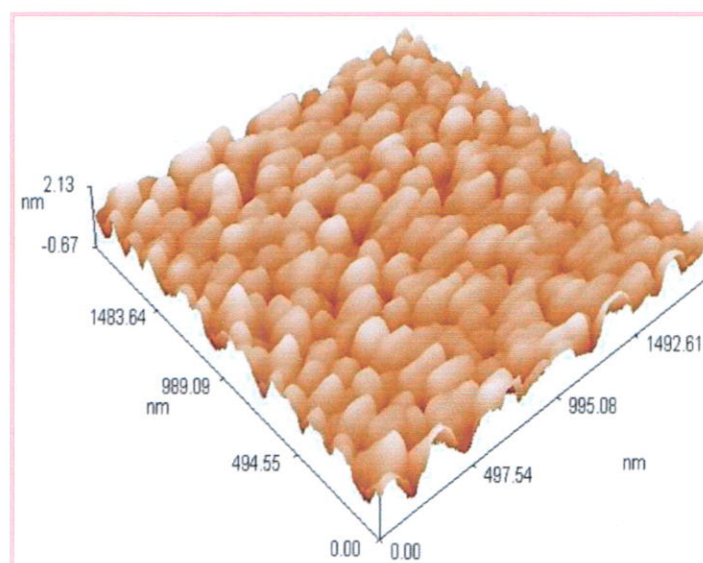




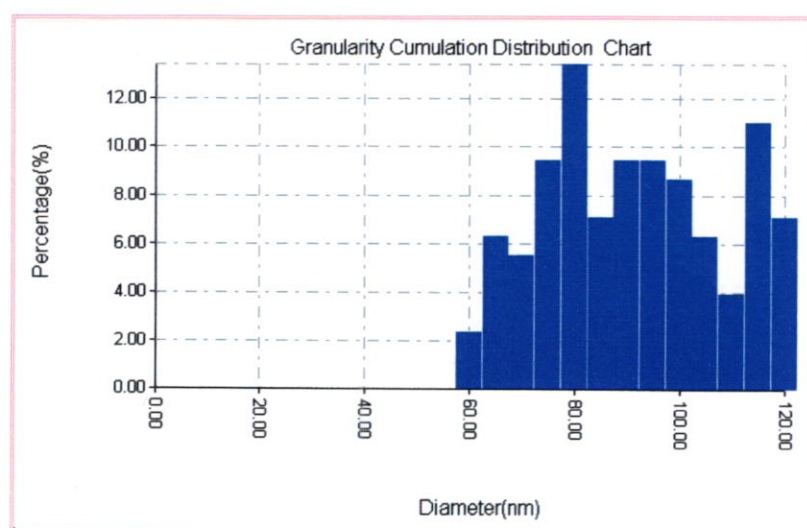
**Fig.3.** SEM of nano -silica by precipitation method.



**Fig.4.** AFM for Nanosilica

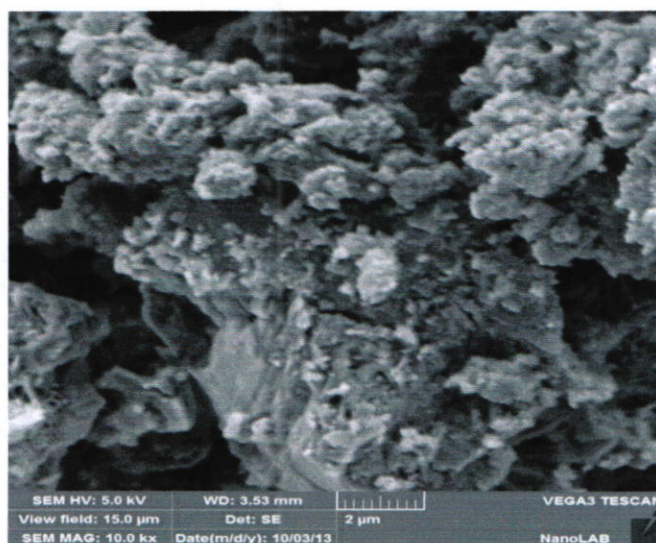


**Fig.5.** AFM three-dimensional surface profile for nano-silica particles.

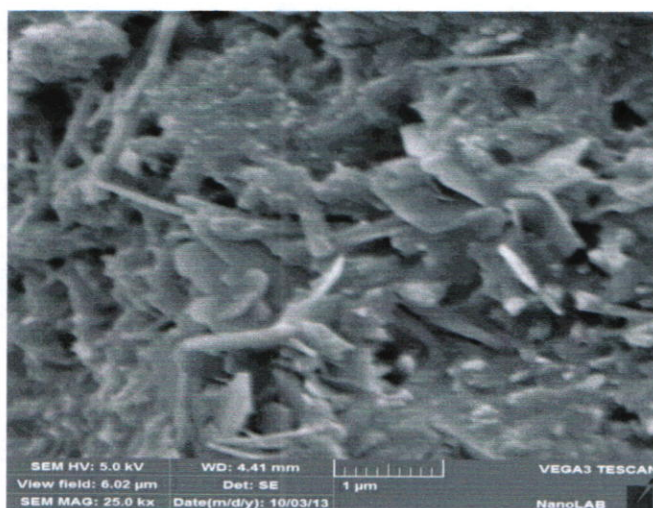


**Fig.6.** Particle size distribution for nanosilica particles.

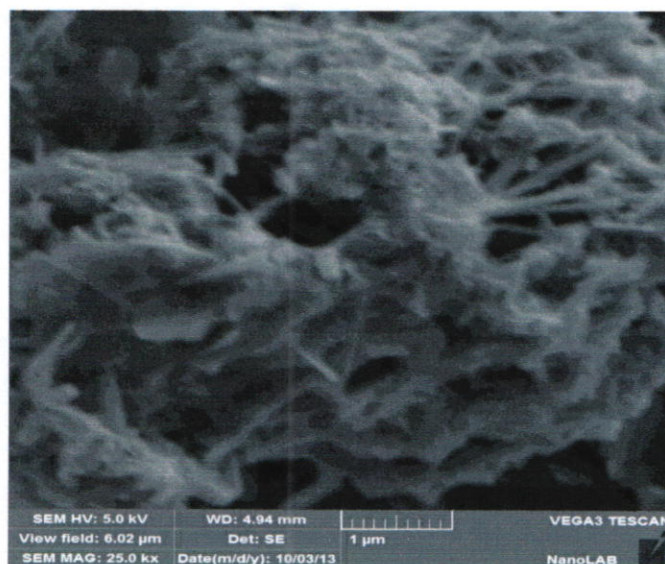




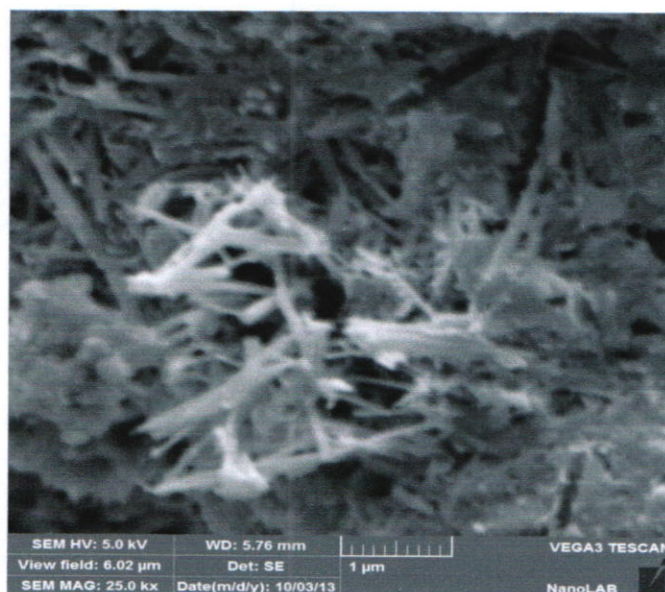
**Fig.7.** Cement without nano-silica at 38°C



**Fig.8.** Cement without nanosilica at 60°C.

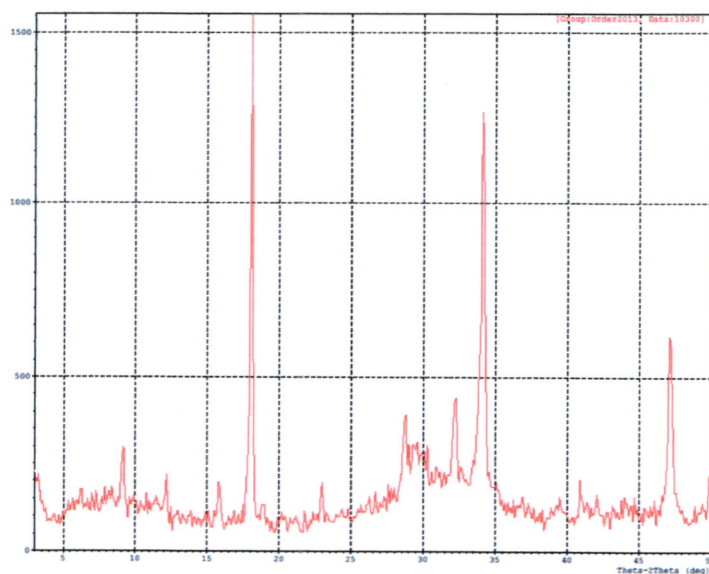


**Fig.9.** Cement and 2%nanosilica at 38 °C

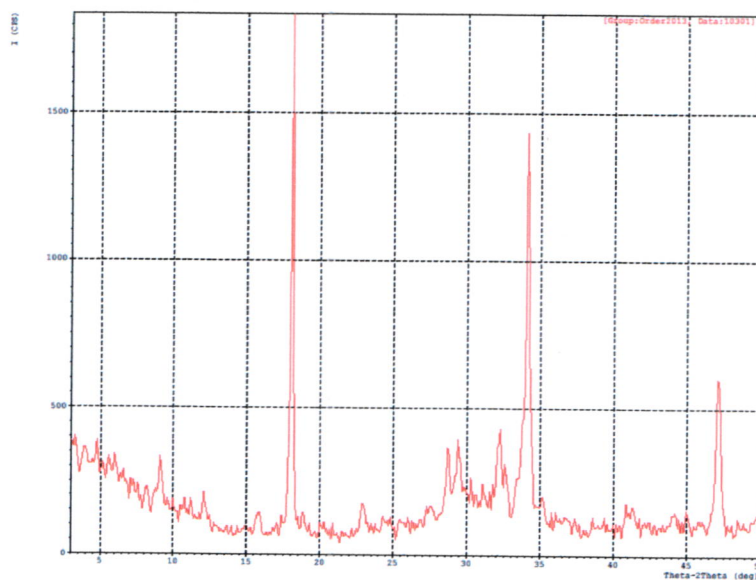


**Fig.10.** Cement and 2%nan-osilica at 60 °C





**Fig.11. XRD of oil well cement class G**



**Fig.12. XRD of cement with 2%nanosilica**