Preparation of Nano Silica particles by laboratory from Iraqi sand and added it to concrete to improve hardness specifications

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Abstract:

Concrete is the most common material used for construction purposes as well as in the lining of oil wells. In this work, the concrete specifications are improved through the preparation of Nano silica particles (SiO₂) by laboratory from Iraqi sand and added to the concrete, which gives results with a large gap in the test of compressive strength after the addition of silica nanoparticles prepared to the concrete mixtures compared to the same mixtures without the addition of silica, to give high hardness and great capacity for bearing of compressive strength as well as increase the resistance to water penetration during the concrete structure and especially the padded cement for the oil well. In this research, Nano silica particles are prepared from the Iraqi sands available in large quantities in the cities of Iraq. The two types of sand are used: the first one is the sand of the Akheider area in Karbala province and the second is the sand of the western region in Anbar province. The Nano silica particles have been prepared in two ways: chemical and physical methods (milling method).

The specifications of the prepared materials are measured by several diagnostic devices, including the scanning electron microscope (SEM), the particle size analyzer apparatus, the X-ray diffraction (XRD) and the infrared spectrometer (FTIR). The results of this project proves the possibility of producing Nano silica particles with high purity and excellent specifications, which can be invested for the commercial purposes in the future.

In this research, two types of cement are used. The first is class G cement used for lining the oil wells, and the second is the Iraqi cement resistant (Portland cement) (Tasuloga

Company / Sulaymaniyah province) used in the building construction. The prepared Nano silica is added to the cement at rates ranging from 0.25, 0.5, 1 and 2% by weight, with fixed of cement amount, water, and mixed the mixture well by standard mixing machine and poured into mold cubes (5 x 5 x 5 cm) according to US specifications.

In the concrete that used of the buildings construction, it is conducted a mixture of cement and normal sand (to both of two types of sands) and added the prepared Nano silica by a percentage ratio, and poured into a cubic templates, then conducting the tests of compressive strength which is of great importance in determining the strength of prepared concrete.

The results indicates that the hardness of the concrete containing Nano materials is significantly increased and reached a level exceeding 4000 PSIG when adding 1% of the silica nanoparticles to the cement used.

On the other hand, the results shows that the Nano materials in the concrete because of their high surface area reduced the permeability of the water into the concrete structure because these substances are hydrophobic, which reduces the permeability of sulfate ions and salts, which prolong the life of the concrete for long periods of time especially in wells Oil, so this research is of great economic importance in the investment of Iraqi wealth for the production of new engineering materials.

Keywords: Nano silica, the concrete, compressive strength, particles, class G cement, the Akheider sand, western region sand.

تحضير دقائق السيليكا النانوية مختبريا من الرمال العراقية واضافتها الى الخرسانة لتحسين مواصفات التصلب

الخلاصة:

تعتبر الخرسانة هي المادة الأكثر شيوعا المستخدمة لأغراض البناء وكذلك في عمليات تبطين الآبار النفطية. لذلك في هذا البحث تم تحسين مواصفات صلابة الخرسانة من خلال اضافة السيليكا النانوية SiO₂ التي تم تحضيرها مختبريا من الرمال العراقية و التي اعطت نتائج بفارق كبير بفحص الانضغاطية compressive strength بعد اضافة السيليكا النانوية التي تم تحضيرها اضافة السيليكا النانوية التي تم تحضيرها اختافة السيليكا النانوية و التي اعطت نتائج بفارق كبير بفحص الانضغاطية المتنادين الزمان بعد من خلال اضافة السيليكا النانوية و التي التي تم تحضيرها مختبريا من الرمال العراقية و التي المات المال العراقية و التي المال العراقية من خلال النانوية بالمال العراقية و التي المال المال النانوية و التي المال المال النانوية و التي المال المال

لتعطي صلادة عالية وطاقة كبيرة للتحمل او الانضغاط فضلاً عن زيادة مقاومتها لنفاذ الماء خلال الهيكل الكونكريتي وبالخصوص السمنت المبطن للبئر النفطي.

في هذا البحث تم تحضير السيليكا النانوية SiO₂ من الرمال العراقية المتوفرة بكميات كبيرة في محافظات العراق. حيث تم استخدام نوعين من الرمال الأول رمال منطقة الاخيضر في محافظة كربلاء والثاني رمال المنطقة الغربية في محافظة الانبار. وقد تم تحضير SiO₂ النانوية بطريقتين هما الطريقة الكيميائية والطريقة الفيزياوية (طريقة الطحن).

وتم قياس مواصفات المواد المحضرة بعدة اجهزة تشخيصية منها المجهر الالكتروني الماسح SEM، وجهاز قياس الحجم الحبيبي للدقائق Particle Size Analyzer ، وجهاز حيود الاشعة السينية XRD ومطياف الاشعة تحت الحمراء FTIR . أثبتت نتائج هذا المشروع أمكانية أنتاج السيليكا النانوية بنقاوة ومواصفات عاليتين والتي من الممكن استثمارها لإغراض تجارية مستقبلا وبشكل اقتصادي.

في هذا البحث تم استخدام نوعين من الاسمنت، الأول هو الاسمنت نوع G المستخدم في تبطين الآبار النفطية، والثاني الاسمنت البورتلاندي العراقي المقاوم للأملاح (شركة طاسلوجة / محافظة السليمانية). تم إضافة SiO₂ النانوية المحضرة الى الاسمنت بنسب تراوحت بين (0.25, 0.20, 1, 2)% وزناً مع تثبيت كمية الأسمنت و الماء و أعداد الخلطة و صبها في قوالب مكعبة الشكل (5×5×5) سم وحسب المواصفات الأميركية اما في الخرسانة الخاصة بالمباني فتم عمل خلطة من الأسمنت و الرمل غير المعامل (رمل الاخيضر ورمل المنطقة الغربية) مع نسبة من السليكا المحضرة مع تثبيت كمية الأسمنت و الماء و صبها في القالب. تم التركيز على أجراء اختبارات الانصغاطية (compressive strength) لمالها من أهمية كبيرة في تحديد قوة الخرسانة المحضرة.

أظهرت النتائج زيادة صلابة الخرسانة الحاوية على المواد النانوية بشكل كبير وملفت للنظر تصل الى مستوى تجاوز Psig كا 4000 عند إضافة %1 من السيليكا النانوية الى الأسمنت المستخدم . ومن جانب اخر أوضحت النتائج ان المواد النانوية الموجودة في الخرسانة وبسبب مساحتها السطحية العالية أدت الى تقليل نفاذية الماء الى داخل الهيكل الكونكريتي كون هذه المواد كار هة للماء (Hydrophobic) مما يؤدي الى تقليل نفاذية ايونات الكبريتات والأملاح مما يطيل عمر الخرسانة لفترات زمنية طويلة وخاصة في الآبار النفطية، لذلك يعتبر هذا البحث ذو أهمية اقتصادية كبيرة في استثمار الثروات العراقية من اجل أنتاج مواد هندسية جديدة.

الكلمات الدالة: السيليكا النانوية، الخرسانة، قوة الانضغاطية، الدقائق، سمنت نوع G، رمل الاخيضر، رمل المنطقة الغربية.

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<u>1. Introduction:</u>

Concrete mixtures are important engineering materials involved in many constructions work as well as in the oil and gas industry in lining oil wells. Concrete preparation and design usually consume almost all of the world's cement production. The use of large quantities of cement produces an increase in CO_2 emissions. These emissions have a significant impact on the environment [1]. Therefore, many researchers worked to improve the specifications of these cement mixtures by adding some different materials [2].

Sand is the material that is most commonly chemically composed of silicon dioxide known as SiO2. The higher percentage of silica in sand means the sand was best. Sand is a raw material for many industries such as glass, concrete, glass water (sodium silicate) and other industries [3].

The Iraqi lands west of the Euphrates are characterized by containing large quantities of different types of sand. The most important sites where the sand is located in large quantities are Karbala (Al Akheider area) that a light red sand, Al Habbaniyah area in Anbar with white sand, The western region of Anbar with white sand, which is characterized by containing a high quantities of silica up to (96%) minimum, and therefore considered as raw materials for the manufacture of the finest glass [4].

Therefore, the sand is used with cement in the manufacture of concrete, and the sand is pure the concrete would be hardest, which is used as a basis in the construction of buildings after mixing in certain proportions with cement and gravel and the addition of water to work cement paste, and when the use of cement in the lining of oil wells. The lining must be strong, solid and non-permeable. In order to obtain good lining of the oil wells, a strong cement mixture must be obtained that is not permeable to water and salts, and to avoid permeable of the water and salts inside of the concrete.

It is worth mentioning that the salts in case of entry into the structure of concrete is very harmful as it makes to destroy the chemical structure of the concrete, so must be fill the interstitial spaces in the structure of concrete and reduce its effectiveness to the minimum [5, 6,7].

The design of concrete mixtures means that the relative values of their components are determined to suit the desired requirements of that application. This is done by using volume or weight ratios proven to be effective through experience. Sometimes the proportions of the materials used are calculated on a technical basis to include the properties of the materials used and the properties required in the hardened concrete (eg, the resistance of the loads or the resistance of the scratch), and the concrete must have some important requirements such as the easy casting and final leveling of the concrete surface. To use of existing materials for obtaining the concrete with properties required in soft and rigid cases and can be considered as a resistance to compressibility of concrete as evidence of the quality of hardened concrete [7, 8].

Nano silica:

We can define nanomaterials as the class of advanced materials that can be produced to measure their dimensions or the dimensions of their internal granules between 3 nm and 311 nm. The small sizes and measurements of these materials lead to behavior that is different from the large traditional materials that increase their dimensions at 311 nm and have highly discriminating qualities and qualities that cannot be found in the traditional materials. [9]

Therefore, the use of nanomaterials in engineering applications has taken a wide range in application due to the characteristics of these materials of high surface area (the ratio of space to size is very high), and the possession of these materials of high internal energy, as well as control of their hydrophobic of water. One of the most important nanomaterials is the use of silica nanoparticles in various engineering applications, including in concrete mixtures of various types [10, 11].

Silica nanoparticles can be prepared in a number of ways, the most important of which are chemical and physical methods. The chemical method is one of the most important ways in which silica is produced from its raw materials such as sand, Feldspar or quartz.

The process involves steps including initial material preparation and reaction by melting raw material with NaOH to produce sodium silicate or so-called glass water. Sodium silicate is treated with concentrated sulfuric acid or concentrated hydrochloric acid to precipitate silica gel in the acid medium. Subsequent processes of the resulting material can then be carried out to obtain nanoparticles. [12, 13] The equations below show the most important chemical reactions done in this way [12]:

Sand rich by
$$(SiO_2) + 2 NaOH \rightarrow Na_2SiO_3 + H_2O$$
 (1)
 $Na_2SiO_3 + 2HCl + H_2O \rightarrow 2NaCl(s) + Si (OH)_4$ (2)
 $Si(OH)_4 \rightarrow SiO_2 + 2H_2O$ (3)

There is also another method of physical way based on the principle from top to bottom adopted in the philosophy of preparing the grains of nanoparticles (up down) as shown in Figure (1), and includes this method to conduct the initial treatment of sand washing with high content of silica And then drying the sand and grinding it with nanomill. [14, 15, 16]

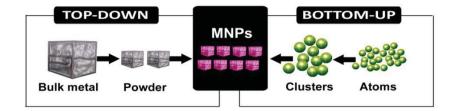


Fig. (1) The philosophy of preparation of nanoparticles (Top-Down or Bottom-Up)

2. Experimental work:

2.1 Materials used in research:

- Al-Akhaydar Sands
- Sands of the Western Region
- Cement type G
- Cement resistant (Portland) from Tasluga factory
- NaOH
- Hydrochloric acid

2.2 Devices and equipment used for examination and diagnosis:

In order to diagnose the properties and specifications of the nanoparticles produced in this research; many advanced devices were used in the Nanotechnology Center/ University of Technology and the Petroleum Research and Development Center,

- Scanning Electron Microscope (SEM-VEGA) (SEM).
- X-ray diffraction device used to detect the crystalline structure of nanomaterials.
- Fourier Transform Infrared Spectroscopy (FTIR)
- Measuring device of crystals.

• On the other hand, a compressive strength test was conducted for the concrete cubes prepared in the research.

• Special mixer for preparing concrete mixtures.

• A special mold was used to pour the prepared concrete mixtures with dimensions of (5x5x5) cm.

2.3 Methods of preparing Nano silica from Iraqi sand:

Two methods were used in the preparation of silica nanoparticles, the chemical method, and the method of the mill, and the use of two types of Iraqi sand, and the preparation methods as shown below:

2.3.1 Use of the chemical method for the preparation of silica nanoparticles:

This method involved several steps to conduct the chemical reaction followed by purification and grinding steps. In this process, the Al-Akhaydar sand was used as raw material for the production of silica nanoparticles. The chemical reaction between Sand and NaOH was carried out. The steps below summarize the procedure used to prepare silica nanoparticles: -

1. Weigh 25 g of pre-washed Al-Akhaydar sand with diluted hydrochloric acid (0.1N HCl) and then rinse thoroughly with distilled water several times.

2 - Drying the sample to a temperature of 100 $^{\circ}$ C and add 175g of sodium hydroxide (NaOH).

3. The mixture is then fusion at a temperature of 550 $^{\circ}$ C for one hour. The heat-reactive substances are then cooled and the silica deposition by concentrated hydrochloric acid is gradually added to the mixture until the medium is converted to pH (1).

4 - Filter the precipitate and wash several times with distilled water to get rid of sodium chloride NaCl, which is formed as a by-product during the reaction process. The final product is then dried at $105 \degree$ C.

5. Figure (2) illustrates an outline of the synthesis of the steps for the production of the nano silica in the chemical way. Figure (3) shows the particulate sand used prior to the reaction, as well as the silica nanoparticles resulting from the steps and phases of the reaction.

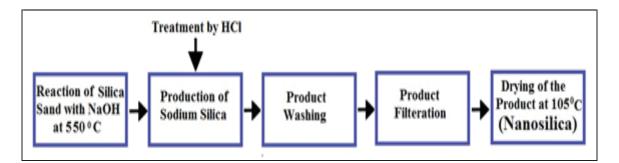


Fig. (2) The stages of the production of silica nanoparticles prepared by the chemical method



Fig. (3) Silica nanoparticles prepared by the chemical method of the pre-reaction and post-reaction Al-Akhaydar sand

2.3.2 Use of the physical method for the production of nanosilica particles:

In this method, the western region sands were used, which have a very high silica content of 96%. Therefore, some primary treatments were done without the need to treat them chemically. The sand was first washed with diluted acid (0.1N HCl) to get rid of impurities and then washed several times with distilled water. Then, the sample is dried by oven at 105 $^{\circ}$ C for 2 hours. After that grinding the sample with a conventional mill for two hours and then carried out the process of sieving with the sieve (50 micron size). Finally, the output was grinded with a Nano mill for four hours. Figure (4) shows the shape of the sand before and after the treatment in the physical way.





2.4 Preparation of concrete mixtures:

We conducted a number of concrete mixtures have been prepared by mixing fixed percentages of cement used in lining oil wells (Class G) and water only, according to the specification of oil wells approved in the petroleum industry (American specifications), so that the ratio of water to cement is 44%. And then add different percentages of silica (SiO₂) nanoparticles that prepared in the above two ways (chemical and physical). Using a special mixer and pour in a mold as follows:

- 1. Constant quantities of cement and water were weighed and placed in the special mixer and then mixed at 140 rpm for one minute. This is the mother sample where the percentage of prepared silica (SiO₂) is zero (without addition).
- The previously prepared silica nanoparticles were added at different rates (0.25, 0.5, 1, and 2%) for each mixture and continued mixing at the same speed for half a minute.
- 3. Then the prepared concrete mixture was poured in a mold of (5 x 5 x 5) cm and left for the second day with cover to avoid water evaporation.
- 4. The next day, the concrete cubes are removed from the molds and immersed in water for 3 days, after which they are released from water and left for 25 days before any tests are carried out. Figure (5) shows the shape of the resulting concrete cubes.



Fig. (5) Some samples of concrete cubes produced from mixtures prepared

3. Discussion of results and diagrams:

3.1 Production of silica nanoparticles:

3.1.1 X-ray diffraction results (XRD)

A number of tests have been conducted on the prepared material (Nano silica) in this work to diagnose its crystalline characteristics, including x-ray diffraction testing. This technique is based on monitoring the scattering of the intensity of a bands of x-rays falling on the sample. Figure (6) shows an X-ray diffraction test of Nano silica that prepared by the chemical method presenting the interaction of sample for the of Al-Akhaydar sand.

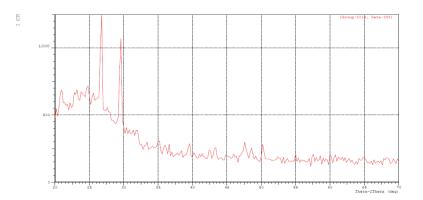


Fig. (6) XRD test of the prepared nanosilica form of chemical way for Al-Akhaydar sand

On the other hand, Figure (7) shows the X-ray diffraction of the prepared Nano silica by the physical method (sample 2) by grinding the sand of the western region by the nanomill, after conducting the preliminary operations of washing, drying and screening of the raw sand.

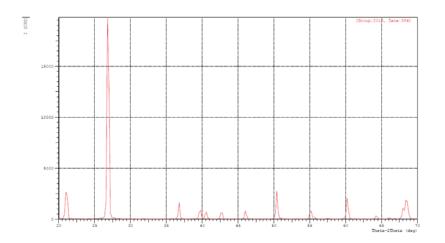


Fig. (7) XRD test of silica nanoparticles prepared by physical way from the western region sands

3.1.2 Scanning electron microscopy (SEM) results

The scanning electron microscopy of the prepared Nano silica particles was done in different ways in order to know the nature of the surface, shape and size of the prepared material in every way. Figure (8) shows the image of the microscope with magnification $(20\mu m)$ as in (a) and $(2\mu m)$ as in (b) for the sample of Nano silica particles (sample 1) prepared by chemical way.

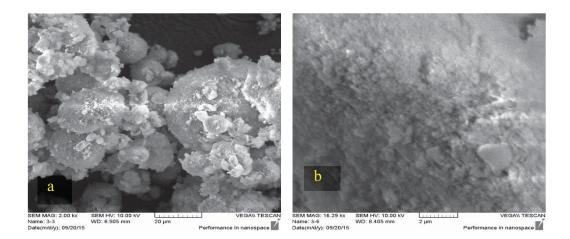


Fig. (8) The microscopic examination (SEM) of the material prepared by chemical method from the sand of Al-Akhaydar by two magnifications (20μm) in (a) and (2 μm) in (b)

By observing the magnifications, nanosilica SiO_2 was observed in the form of small silica nanoparticles with a diameter between 100-150 nm.

On the other hand, Figure (9) shows the images of the electron microscopy microscope $(20\mu m)$ as in (a) and $(2\mu m)$ as in (b) of the physically prepared silica nanoparticles, prepared by the nanomill grinding (sample 2). The silica prepared in this way were observed to be of silica nanoparticles and clearly in the form of particles ranging in diameter between 200-250 nm.

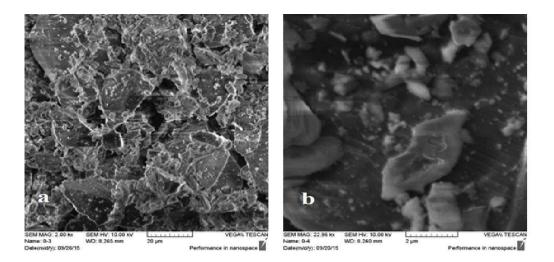


Fig. (9) The microscopic examination of the material prepared by the physical method of the western region sands (20μm) in (a) and (2 μm) in (b) 3.1.3 Results of (FTIR) tests:

The analysis of the FTIR of the prepared sample sand was done using the chemical method (25g NaOH + 175g sand) as shown in Figure (10). It was observed that several metal oxides were present in the chemical structure of the sand during the 800 cm⁻¹. The SiO₂ is clearly observed at the 1100 cm⁻¹ wave number, which represents a high proportion of silicon. On the other hand, it is possible to observe the 1150 curved beam and the 1190 cm⁻¹ package representing the appearance of siloxane (Si- O-Si) asymmetric, on the other side of the package for the range 465 to the area of 800 cm⁻¹ it returns to the corresponding (Si-O-Si). The results showed that the Si-OH at 935 cm⁻¹ beam was shown due to the high concentration of Silanol groups and the small size of the nanoparticles of the prepared material. The emergence of the O-H group was

observed in the 1630 cm⁻¹ range as well as the corresponding group at 3400 cm⁻¹ which represents the absorption of water molecules.

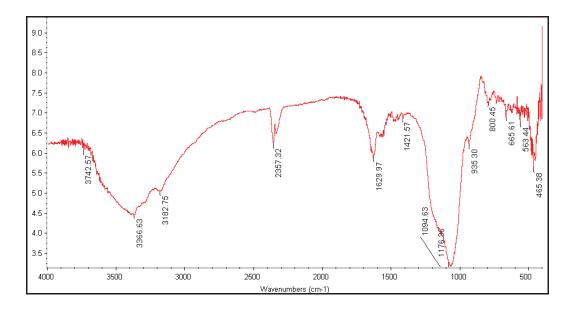


Fig. (10) Examination of the FTIR of the sample prepared from Al-Akhaydar sand in the chemical way and by reaction of (25g NaOH + 175g Sand)

The FTIR test of the sample from the Western Region Sand was also done in the physical way as shown in Figure (11)

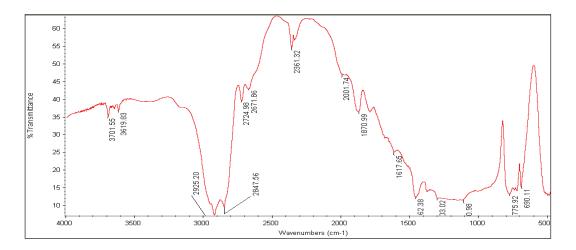


Fig. (11) Examination of the FTIR of the sample from the Western Region Sand in the physical way

On the other hand, the surface area of the resulting samples was examined as shown in Table (1). The results showed that silica nanoparticles prepared from the sand of the Al-Akhaydar had a higher surface area of 242.549 m² / g compared with the sands of the western region which reached 0.27 m² / g.

Surface area (m²/g)	Nano silica from Al- Akhaydar sand by chemical way	Nano silica from western region by physical way
	242.549	0.2717

Table (1) Surface area of nano silica prepared by chemical methods

3.2 Concrete tests:

3.2.1 Specification of sand used in research

In this research, two types of Iraqi sand were used as raw material for the preparation of silica nanoparticles, namely the sands of the Al-Akhaydar region in Karbala and the sand of the western region in Anbar as shown in Table (2).

Component	Al-Akhaydar sand	western region (Aradhma site)	
SiO ₂	Min 85 %	Min 96 %	
Al ₂ O ₃	0.45 %	Max 1.6 %	
Fe ₂ O ₃	0.35 %	Max 1%	
LOI	5%	2%	

Table (2) The chemical composition of both species

3.2.2 Specifications of cement used in the preparation of concrete mixtures:

Two types of cement were used in the preparation of concrete mixtures, namely cement for the lining of oil wells (Class G) and Portland cement used in construction work. Table (3) shows the structural specifications of both types of cement.

		Results				
No.	Test (%)	Sample Code			Test Method	
110.	1 cst (70)	API	Cement	Portland	i est Methou	
		Standard	type G	Cement		
1	Magnesium Oxide MgO (max.)	6.0	5.54	4.5	ASTM D-511	
2	SO_3 (max.)	3.0	1.78	0.534	ASTM D-516-80	
3	Loss On Ignition (max.)	3.0	0.92	0.95	ASTM C114	
4	Tri calcium silicate C ₃ S	65/48	60.40	49.863	API/10A/ISO10426-	
4	(max/min)	03/40			1:2000	
5	Tri calcium aluminate C ₃ A	2	3 0.82	5.562	API/10A/ISO10426-	
5	(max)	5			1:2000	
	Tetra calcium aluminoferrite		17.32	22.424	API/10A/ISO10426-	
6	plus twice the tricalcium	24			1:2000	
	aluminate C ₄ AF+2C ₃ A (max.)				1.2000	
7	Na ₂ O equivalent (max.)	0.75	0.551	0.326	API/10A/ISO10426-	
	wa20 equivalent (max.)				1:2000	

Table (3) Specifications of cement class (G) oil well and Portland cement resistant

4.2.3 Compressive strength tests results

Due to the importance of examining the compressive strength to the type of concrete in various engineering applications, the effect of adding silica nanoparticles in different percentages on the prepared concrete was tested. Therefore, in the concrete mixtures for the lining of the oil wells, the silica nanoparticles was added to it (Al-Akhaydar sand after chemical treatment, the first sample), the sample has been configured to examine of the compressive strength on the cubes of concrete after 25 days from the date of mixing. Table (4) shows the most important results obtained from the compressive tests on samples that were added the Nano silica to it for installation during the concrete preparation process.

No.	SiO ₂ (%)	Cement (Class G) (g)	Water (g)	Compressive Strength (psi)
1	0	600	264	2813
2	0.25	600	264	3414
3	0.5	600	264	3579
4	1	600	264	4808
5	2	600	264	5120

 Table (4) Results of the compressive strength test for mixtures the addition of the added silica of the nanosilica sand prepared by the chemical method

From the results shown in the table (4), we observe the obvious effect of silica nanoparticles on the amount of compressive strength. The highest compressive value reached 4808 psig and 5120 psig at the adding ratio of 1% and 2% respectively, compared with 2813 psi for the concrete sample of the concrete prepared without any addition of nanomaterials, the addition of silica nanoparticles is responsible for the significant increase in the value of compression, as these materials have penetrated the gaps in the installation of this on the one hand, on the other hand, the high surface area possessed by nanomaterials prepared from the sand of Al-Akhaydar. it had a great role in improving concrete specifications by increasing the spread of the material inside the concrete.

On the other hand, Table (5) presents the results of the concrete mixtures related to the lining of the oil wells and the addition of the SiO_2 silica nanoparticles that prepared by the physical method of the sand of the western region. The compressive inspection of the concrete (concrete cubes) was done at 25 days after the mixing date. The results shown in the table below showed the apparent effect of silica nanoparticles on compressive values, with the highest compressive value of 4850 psig and 5145 psig at 1% and 2% respectively, compared to 2813 psig of the sample without any addition to nanoparticles.

No.	SiO ₂ (%)	Cement (Class G) (g)	Water (g)	Compressive Strength (psi)
1	0	600	264	2813
2	0.25	600	264	3680
3	0.5	600	264	3767
4	1	600	264	4850
5	2	600	264	5145

Table (5) Results of the compressive strength test for mixtures when added
nanosilica prepared from the sand of the western region after 25 days

The reason for the large increase in the values of compression is due to the presence of silica nanoparticles added to concrete cement used in the lining of oil wells, where the results showed that the hardness of the concrete containing this nanoparticle significantly and strikingly the value reached the level exceeded 5000 (psi) 2%, so it is possible to say that the nanomaterials found in the concrete due to its high surface area have reduced the permeability of water into the concrete structure because these materials are hydrophobic, which reduces the permeability of sulphate ions and salts, which prolong the life of concrete Long periods of time.

Additional concrete mixtures were prepared in the same way as the previous one, using salt resistant cement (Iraqi origin from Tasuluja/ Sulaymaniyah) and mixing it with silica prepared by the physical method of sand in the western region. These mixtures were made in January and the temperatures were low. The period of hardening and acquiring the appropriate hardness requires more time than the summer mixtures, and the results as shown in Table (6).

Table (6) Results of the compressive strength test of concrete mixtures added to silica prepared by the physical method of the western region sands

No.	Resistant Cement	Sand of western region	Water	SiO ₂ nanoparticles prepared by physical way from the Sand of western region	Compressive after strength 25 days (psi)
1	300 g	400 g (without treatment)	250 g	0	1510
2	300 g	392 g (without treatment)	250 g	8 g (1 %)	2616
3	300 g	384 g (without treatment)	250 g	16 g (2 %)	3766

4. Conclusions:

1. In this research, silica nanoparticles were prepared successfully by exploiting the local raw materials namely sand and used it in the production of concrete with high specifications in oil and construction projects.

2. The preparation of silica nanoparticles from local materials (sand material} is a new technology and worthy of research and application because of the good economic feasibility of the production of this material at relatively low cost and raw materials available in Iraq with high specifications.

3. The results showed that the specification of concrete with low amounts of silica nanoparticles a very high quantum leap in the compressive values and bear of the concrete after the addition of the nanoparticles exceeding 4000 psig. These results confirm the effectiveness of the addition of silica nanoparticles.

4. Because the sand of the western region contains a high percentage of silica up to 96%, therefore, the obtain of silica nanoparticles made it easier to use chemical and physical

treatment. In terms of engineering and economic, these methods of preparation are simple and inexpensive methods compared to other methods of other researches. [20]

5. The results of the research showed that the use of sand from the area of Akheider in the city of Karbala for the preparation of silica nanoparticles is somewhat complicated in the way of preparation, and results confirmed that the silica nanoparticles obtained were excellent specifications and high purity and regular crystalline structure, and its effective to improve the specifications of concrete that used in the cementation of oil wells or used in structural construction.

6. The results showed that the concrete mixtures for the cementation of oil wells, which used cement class G, presented high values of compressive strength that can be bear, where the difference was significant when comparing the compressibility before and after the addition. These results are a positive indicator of the need to add Nano silica with this type of concrete mixtures to obtain high compressive concrete and thus maintain the structure of the oil well and maintain the production pipes under the lining of the cement as well as bearing the factors and ground effects and groundwater on the equipment under the surface of the earth.

7. The nanoparticles that prepared in their synthetic nature are hydrophobic. Concrete will thus be protected from water penetration and thus protect it from the attack of dissolved salts in the groundwater which present in the soil. Thus, the operational life of the working facilities in the oil wells will be much greater when add small amounts of silica nanoparticles to it.

8. The surface area of chemical-produced silica nanoparticles was very large compared to raw materials or other ways to produce nanoparticles. The nano crystalline silica produced by the nano crystalline method (the physical method), especially the sand of the western region, had a much smaller surface area than the raw materials manufactured.

9. The results of the XRD tests showed that silica nanoparticles were crystallized in the crystalline structure through the peak of the silica peak that appeared, as well as the area of peak, and the amount of silica nanoparticles in the prepared material was large.

10. The results of the electron microscopy SEM, as well as the tests of the size of the nanoparticle to the formation of silica and a clear nanoparticle and in the form of particles less than 100 nm, which confirms the validity of the orientation in the selection of methods of preparation of the nano silica from both types of sand.

11. The infrared spectra (FTIR) of the prepared silica also indicate that the SiO_2 spectrum is large in size and area at 1100 cm-1. When comparing and matching the silica spectrum with the scientific literature, it is found to be fully applicable with silica under research to assure the validity of prepared nanomaterials.

References:

- 1.Ismael FLORES-VIVIAN, Rani G.K. PRADOTO, Mohamadreza MOINI, Konstantin SOBOLEV, "THE USE OF NANOPARTICLES TO IMPROVE THE PERFORMANCE OF CONCRETE", University of Wisconsin, Milwaukee, 16. - 18. 10. 2013, Brno, Czech Republic, EU, NANOCON, 2013.
- 2.S. Tanveer Hussain1, K.V.S.Gopala Krishna Sastry2 1M.Tech student in civil engineering department, GPREC, Kurnool, AP, India 2Associate Professor of civil engineering department, GPREC, Kurnool, AP, "STUDY OF STRENGTH PROPERTIES OF CONCRETE BY USING MICRO SILICA AND NANO SILICA", India/ IJRET: International Journal of Research in Engineering and Technology eISSN: 2319-1163 | pISSN: 2321-7308.

4. حنان عبد الكريم عمران، "التربة في العراق، مفهومها، انواعها، مشاكلها"، كلية التربية الاساسية، جامعة بابل، 2012.

- 5.R. Yu, P. Spiesz, H.J.H. Brouwers, "Effect of nano-silica on the hydration and microstructure development of Ultra-High Performance Concrete (UHPC) with a low binder amount", Construction and Building Materials, 2014.
- 6.admixtures for concrete/ The concrete institute.
- 7.Munasir, A. Sulton, Triwikantoro, M. Zainuri, and Darminto, "Synthesis of silica nanopowder produced from Indonesian natural sand via alkalifussion route", Indonesia, 2013.

8. المهندس محمود كريم خالد، "الاضافات الخرسانية" ، الجامعة التكنولوجية، بغداد.

9.د. محمد شريف الاسكندراني، "تكنولوجيا النانو من أجل غد أفضل"، المجلس الوطني للثقافة والفنون والآداب - الكويت – سلسلة عالم المعرفة، أبريل، 2010.

- A. Jayaraman1, V. Senthil kumar, "Optimization of fully replacement of natural sand by m-sand In high performance concrete with nanosilica", International Journal of Emerging Technology and Advanced ngineering, Website: <u>www.ijetae.com</u>, (ISSN 2250-2459, ISO 9001:2008 Certified Journal, Volume 3, Issue 11, November 2013).
- 11. Ibtehag .Z . Solyman, Salim. M. Khalil / collage of education for women, "The effect of Sodium Silicate and Sulphuer (from Mishraq) in Strength of Concrete", Collage of Sciences/ University of Mosul/ 2007.
- Munasir, Sulton A., Triwikantoro, M.Zainuri and Darminto, "Syntheses of Silica Nanopowder Produced from Indonesian Natural Sand via Alkalifussion Route", International Conference on Theoretical and Applied Physics (ICTAP), Indonesia, 2013 AIP Publishing LLC 978-0-7354-1181-4.
- 13. A. Lazaro, "Nano-silica production by a sustainable process; application in building materials", 8th fib PhD Symposium in Kgs. Lyngby, Denmark (2010), pp 1-6.
- 14. "Monothematic Issue about Nanotechnologies", History of Science and Technology, EPMagazine N. 34, Issue 1, April 2014.
- 15. Andres La Rosa, Mingdi Yan, Rodolfo Fernandez, Xiaohua Wang and Elia Zegarra,"Top-down and Bottom-up approaches to nanotechnology",
- 16. Hai-Dong Yu, Michelle D. Regulacio, Enyi Ye and Ming-Yong Han, "Chemical routes to top-down nanofabrication", 2013,42, 6006-6018.