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An Experimental Study to Demonstrate the Effect of Alumina Nanoparticles and Synthetic Fibers on Oil Well Cement Class G

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Abstract

In the drilling and production operations, the effectiveness of cementing jobs is crucial for efficient progress. The compressive strength of oil well cement is a key characteristic that reflects its ability to withstand forceful conditions over time. This study evaluates and improves the compressive strength and thickening time of Iraqi oil well cement class G from Babylon cement factory using two types of additives (Nano Alumina and Synthetic Fiber) to comply with the American Petroleum Institute (API) specifications. The additives were used in different proportions, and a set of samples was prepared under different conditions. Compressive strength and thickening time measurements were taken under different conditions. The amounts of Nano Alumina (0.5%, 1%, and 1.5% by weight of cement (BWOC)) were selected with synthetic fiber (0.5 g, 1 g, and 1.5 g, respectively). The results show a significant improvement in compressive strength, with all values meeting the API requirements, and a decrease in the thickening time of Iraqi oil well cement, depending on the proportions of additives. The most significant improvement in compressive strength was achieved in the sample containing 1.5% Nano Alumina by weight of cement (BWOC) and 1.5 g Synthetic Fiber (Barolift), where the compressive strength increased by 40.7% and 33.8% at a temperature of 38 °C and 60 °C, respectively, while the thickening time decreased by 26.53% at this ratio of additives. The results demonstrate the feasibility of using these additives to enhance the performance of Iraqi oil well cement, expanding its potential application in Iraqi oil fields.

Keywords: oil well cement, compressive strength, Nano Alumina, Synthetic Fiber.

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1- Introduction

The process of drilling and completing oil and gas wells includes several processes, one of the most fundamental of which is the cementing of the casing. The application of numerous scientific and engineering skills covers the technology of well cementing. Basic considerations, such as the condition of the drilling mud, the use of spacers and flushes, the pipe movement, the centralization of the casing, the optimization of the displacement rate, the design of the slurry for the down hole condition, the selection and testing of cement compositions, and the selection of the appropriate system for the cementing process, are required for successful cementing jobs [1]. Any new materials that are used must make it possible for the cement to be stronger, more durable, flexible, resistant to shocks, and easier to pump [2]. Recently, Iraq, which ranks highly among oil-producing nations, began making cement for oil wells. The cementing process is an important element of oil well drilling operations. His achievement is essential to the maintenance of the consequent process. Installation in oil wells requires the use of cement slurry suited for strong and permanent cement bonding. Portland cement is a fine powder produced through roasting a mixture of 80 % limestone and 20 % clay at 1450 degrees Celsius. Undoubtedly, this substance is authorized for use in 99 percent of oil well cementing operations globally [3]. In order for Portland cement to be designated petroleum cement and, therefore, to be suitable for use in oil well operations, its physical, chemical, and mechanical characteristics need to be in accordance with the standards set by the American Petroleum Institute (API) [4].

To enhance cement slurry's flow qualities, various additives, and Chemical additives and some examples of these including superplasticizers, retarders, accelerators, and viscosity modifiers are used. Admixture is a material that is neither cement, water, or aggregate that is used as an ingredient in concrete and is added to the mixture either proximately before or during the mixing process. Admixture is also known as an additive [5]. Cement admixtures come in a wide variety, and there are currently many of them accessible on the market. These admixtures are employed to improve the characteristics of oil well cement slurry and to accomplish a successful cementing job. It is important to optimize the setting time of the cement slurry in order to make optimal use of the local cement for oil well cementing. This will allow for the most efficient use of local cement [6]. Any increase in the

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cement slurry's viscosity can have an effect on the time it takes to thicken, which can ultimately result in the cement being unable to be pumped [7].

The compressive strength is an indication of the capability of the set cement to protect and maintain the casing string, as well as to provide zonal isolation. There are two different types of compressive strength that are defined for cement used in the oil and gas industry. The early-age compressive strength of cement refers to the compressive strength of cement at initial time following the creation and placing of cement slurry within a well bore. The long-term compressive strength of cement refers to the compressive strength of cement after the graduation of the hydration process and well exploitation for a long time of the production process of the well. In the process of designing oil well cement, one of the most essential tasks is the development of oil well cement with a high early-age compressive strength [8]. The standard minimum strength that is required for any well bore operation is 500 psi in 24 hours at the bottom-hole static temperature. This is the standard that is most commonly employed. A minimum of 500 psi is required for an 8-hour curing time, and a minimum of 1000 psi is required for a 24-hour curing time, according to the API specifications [9].

The application of nanotechnology in the field of materials results in an increase in the durability of those materials as well as an improvement in their performance. It also provides the most efficient use of natural resources and the acquisition of desired material attributes with minimum consumption. Recently, a significant amount of focus has been placed on enhancing the qualities of drilling and construction cement by employing nanomaterials. Because of the benefit of a larger surface area and reactivity by filling the nanoholes of the cement, the addition of nanoparticles can improve the characteristics of cement. Nano silica, carbon nanotubes, Nano titanium, Nano Al_2O_3 and polymer-clay Nanocomposites are the most commonly utilized nanoparticles in the construction industries. They are primarily employed to strengthen various mechanical characteristics of cementing material, such as resistance to decomposition, tensile strength, and compressive strength [10].

In most investigations of nanoparticles in nanomaterial concentrate, the addition of nanoparticles modified the microstructures by activating pozzolanic reactions. However, there is little research on the effect of these additives on characteristics such as hardness, fracture, and abrasion resistance [11].

The addition of fibers is one of the most prevalent and efficient techniques for enhancing the mechanical characteristics of cement-based materials. Decades of research have examined the effect of discrete macro- to microfibers on suppressing crack formation in cement-based materials [12].

Many studies focused on the use of nanomaterials as well as fibers, Ali et al. (2011) has been reported that cement-based materials can benefit from the incorporation of Nano Alumina. The effect of adding Nano Alumina on the mechanical characteristics as well as the percentage of water absorption of concrete that is cured in water and saturated limewater. Component Nano Alumina (Al_2O_3) reacts with the calcium hydroxide that is produced as a byproduct of the hydration of calcium silicates. The incorporation of Nano sized Al_2O_3 into concrete's constituent components results in increased hardness. The incorporation of Nano Alumina led to the achievement of a significant milestone in high compressive strength. The impact of nanoscale Al_2O_3 on the compressive strength of hardened cement in various curing settings has also been the subject of extensive research [13]. Ali Salman (2014) studied the preparation and analysis of Nano silica for the first period from the Ardma location in Al-Anbar province by a combination of top-down and bottom-up methods involving Ball milling and drying procedures. Three distinct ranges (30–100) nm, (60–120) nm, and (90–170) nm of NS were added with three percentages (2%, 6%, and 10% by weight of cement, (BWOC)), and the water-cement ratio was 0.48. After 28 days, the scanning electron microscope (SEM) images of the concrete mixtures indicate the presence of numerous Calcium Hydroxide crystal needles that are overshadowed and cover an important portion with a porous structure. The mechanical characteristics optimization results showed an increase in tensile strength of 22.8% and an increase in compressive strength of 29.889%. By milling silica sand with a steel ball, NS can be produced in vast amounts at a low cost [14]. Dhorgham and Almahdawi (2016) investigated the appropriate use of Sodium Polyacrylate (SP) in cement slurry preparation. Results showed that a 15% addition to the cement slurry raised compressive strength from 680 to 1700 pounds per square inch (psi). SP substantially affected the performance of the slurry and the water-cement ratio, as well as speeding up the initial and final setting times. However, the addition of SP must not exceed 15% in reducing an increase in the pressure of the pump due to the increased cement slurry viscosity, which can block circulation [3]. The effect of Nano Alumina (NA) and Nano Silica (NS) on the hardness and thickening time of High Sulfate Resistance (HSR) class G cement was investigated by Abdul Hadi and Abdul Ameer (2017). They found that NA had a greater impact on compressive strength than NS and that as Nanoparticles expand in size, the compressive strength of the cement decreases. When NP concentration is high, viscosity, gel strength, and yield point all increase [15].

In order to make Iraqi cement more suitable for use in oil wells, Hadi and Al-Haleem (2020) introduced synthetic fiber (Barolift). In order to learn more about the characteristics of the base cement and the cement slurry with 1g of Barolift, a series of experiments were carried out. In this case, the results demonstrated that Barolift acted as a retarder to extend the thickening time, increase the amount of free water by a negligible amount, improve the mechanical characteristics, reduce the porosity, and aid in the formulation of a new cement slurry [16].

This work aims to improve and evaluate the compressive strength and thickening time of Iraqi oil well cement (local cement) Class G under different conditions using mixtures of various additives, according to the American Petroleum Institute (API). In other words, this effort relates to using Iraqi oil well cement to reinforce functional processes in our field instead of commercial cement.

2- Experimental Work

2.1. Materials

Materials used to prepare cement samples and additives that were used in this study are:

a. Alumina Nanoparticles (Al_2O_3)

The Nano Alumina that was applied in this investigation was produced by Skyspring Nanomaterials, Inc., and it has the characteristics that are listed in Table 1.

Table 1. Alumina Nanoparticles Specifications

Product	Aluminum Oxide Nanopowder
CAS Number	1344-28-1
Purity	99.9+%
Particle size	50nm
Color	Milky white
specific surface area	58 m^2/g
Morphology	nearly spherical
Crystal	Alpha
Density	3.5-3.9 g/cm³
Empirical Formula	Al_2O_3

b. Synthetic Fiber

The synthetic fibers used in this study are synthetic cellulose fibers of 0.5-inch length also known commercially as "Barolift." It is produced by Halliburton Oil and Gas Company.

c. Distilled Water

d. Cement

The cement that was used in this investigation was oil well cement Class G and HSR grade, and it was produced by the Iraqi Babylon cement factory. The chemical composition of Iraqi owc class G is listed in Table 2.

Table 2. Iraqi Cement's Chemical Compositions [17]

Component	In Iraqi Cement%	API Standard%
MgO	1.77	0.9
Al2O3	2.996	5.6
SiO_2	18.63	24.66
SO3	3.072	1.55
CaO	69.49	61.87
MnO	0.05061	0.75
Fe2O3	5.587	0.38

Table 2 summarizes the most significant variations in the concentrations of elements between Iraqi oil well cement and the API standard. These variations have a significant impact on the quality of Iraqi cement, which leads to a reduction in the cement's physical and chemical properties.

One of the most significant observations is that the concentration of calcium oxide (CaO) is higher than the quantity that is considered normal according to API specifications. This can result in a reduction in the strength of oil well cement and an early start to the expansion process. In addition, the concentration of silica (SiO_2) and aluminum (Al_2O_3) is lower than their standard quantities, which results in a decrease in the strength of the cement.

2.2. Apparatus

The devices and equipment that were used in this study in order to prepare the cement slurry samples and conduct the tests are listed in Table 3.

Table 3. Devices and Equipment used in this Study

The apparatus	Model
Compressive Strength Instrument	OFITE model 250
HPHT Consistometer	OFITE model 120
Constant Speed Mixer	OFITE model 20
Electronic Balance	ADAM QBW-3000-gram balance
Water Bath	Stuart model SWBD

2.3. Procedure for Preparing and Measuring Samples

a. Cement slurry samples Preparation

The API specification requires that materials be measured precisely. After measuring, the water, cement, and additives were combined in a constant-speed mixer at a speed of 4,000 revolutions per minute. Several cement slurry samples were made with different additives concentrations of Nano Alumina and Synthetic Fiber. Table 4 provides an explanation of the materials that were used in the preparation of cement slurry samples as well as their concentrations.

Based on previous research, the addition of Nano Alumina in proportions greater than 1% BWOC leads to a significant decrease in the thickening time of cement [16]; therefore, the addition of Nano Alumina in small proportions ranging from (0.5-1.5%) BWOC was selected to reduce the effect of Nano Alumina on the thickening time, and the addition of synthetic fiber with it in proportions ranging from (0.5-1.5g) to enhance the compressive strength of cement.

Table 4. Compositions and Concentrations of Prepared Cement Slurry Samples

Samples NO.	1	2	3	4
Mix water (g)	349	349	349	349
Oil well Cement class G (g)	792	792	792	792
Nano-material %(BWOC)	0	0.5	1	1.5
Nano-Alumina (g)	0	3.96	7.92	11.88
Synthetic fiber (g)	0	0.5	1	1.5

b. Compressive Strength test

In accordance with API standards, compressive strength tests are conducted. The OFITE model 250 is a computer-controlled compressive strength examiner used to measure the compressive strength of owc by continuously applying a force of 4 inch^2 until breakdown [18].

The method used to measure the compressive strength of samples that had been prepared and treated at two different temperatures, 38 °C, and 60 °C. These included cleaning and drying the contact surface and molds of the plate, pouring cement slurry into the molds, covering the mold with a clean, dry cover plate, submerging the specimens in a water bath for eight hours at atmospheric pressure and (38°C and 60°C) curing temperatures, extracting specimens from the water bath and after 45 minutes placing them into a compressive strength apparatus. The specimen was then exposed to a load, and compressive strength was reported [5].

c. Thickening time test

The thickening time test was conducted in accordance with API standard specification 10A using the HPHT Consist meter, which is the best approach for determining how long the Iraqi oil well cement should stay pumpable under HPHT conditions (52 °C, 5160 psi).

3- Results and Discussions

According to the API specification 10A [5], the compressive strength and thickening time of samples of Iraqi class G cement prepared with different proportions of Nano-Alumina (NA) and Synthetic Fiber (Barolift) additives were evaluated after being subjected to different conditions.

3.1. The compressive strength of cement slurry at a temperature of 38 ° C

The results of the compressive strength value at 38 ° C for class G Iraqi owc with and without additions are shown in Fig. 1.

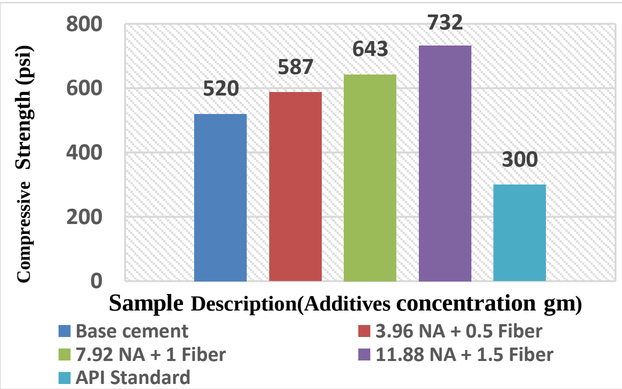

Fig. 1. Compressive Strength of Cement Slurry at 38 °C, 8 hr. Curing

The results obtained indicate that the value of compressive strength of Iraqi class G cement without any additives (base cement) at a temperature of 38 °C and during an eight-hour curing period is acceptable and compatible with the requirements of the API specification 10A [5] (above 300 psi) and that indicates the success of this type of cement at this temperature in terms of compressive strength.

Additions of Nano Alumina and Synthetic Fiber (Barolift) together to the three samples in different concentrations, as shown in Fig. 1, led to a significant increase in the compressive strength values, where the compressive strength of all samples was under a temperature of 38 °C and an eight-hour curing period under API specifications10A.

The reason for the improvement of compressive strength is due to the additives used, where the addition of Nano-Alumina led to the rapid consumption of CaOH, which was developed during cement hydration, especially in the early ages associated with the significant interaction of Nano-Alumina particles, which has a large surface area. This is identical to what has been proven by Abdul Hadi and Abdul Ameer when using this type of nanomaterials as additives to cement slurry at a temperature of 38 °C [15].

In addition, the synthetic fiber used in this study contains cellulose. It is known that the presence of voids inside the cement mixture reduces the compressive strength, so the added fiber fills these voids and is considered a binder element for the cement slurry composition and thus increases the compressive strength. As it is noted that the higher the percentage of additives, there was significant the increase in the compressive strength values of cement. According to the microstructural study, synthetic fiber can speed up the pozzolanic reaction and produce an increased number of C-S-H products [16].

3.2. The compressive strength of cement slurry at a temperature of 60 ° C

The compressive strength results at 60 ° C for class G Iraqi owc with and without additions are shown in Fig. 2.

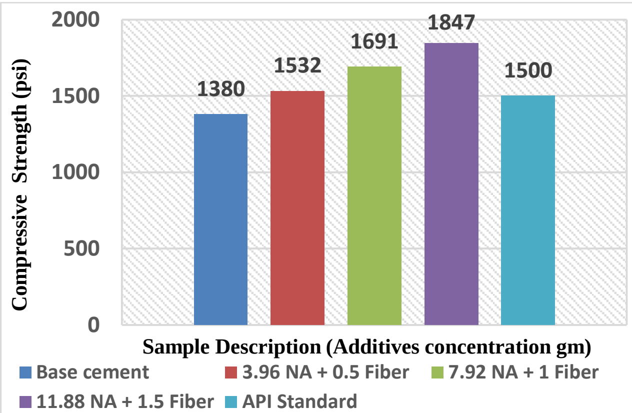

Fig. 2. Compressive Strength of Cement Slurry at 60 °C, 8 hr. Curing

The results obtained indicate that the compressive strength value of Iraqi Class G cement without any additives (base cement) at a temperature of 60 ° C and during an eight-hour curing period was 1380 psi, which is unacceptable and not compatible with the requirements of API specifications10A [5] (1500 psi) and that indicates the failure of the compressive strength of cement in these conditions. This may be because of the effect that high heat treatment (curing) has on early strength development. Also, as shown in Table 2, the chemical compositions of the Iraqi cement used in this study in some of its compounds were incompatible with API specifications10A.

On the other hand, the results showed a significant increase in the compressive strength at a condition of 60 °C and a curing period of eight hours when adding Nano Alumina and Synthetic Fiber (Barolift) together, where the compressive strength of all samples containing different percentages of additives was compatible with API specifications10A [5] (higher than 1500 psi).

It also appears in Table 2 that the cement used in this study had a low percentage of aluminum oxide compared to the API specifications, where the Nano-Alumina particles with a large surface area compensated for the lack of aluminum oxide in the cement. Cement has been modified with Nano Alumina to increase its compressive strength and initial electrical resistivity. These modifications were made possible through the use of Nano Alumina [19].

Also, the addition of synthetic fiber (Barolift) led to filling the voids formed in the cement mortar. This increase in compressive strength results from barolift's capability to limit the emergence of micro-cracks by forming strong cross-links between cement particles at high temperatures. The fibre's principal mechanism is forming a mesh network that may improve the cement's strength and provide further control of lost circulation [20]. It is compatible with all types of liquids without significantly changing their flow characteristics. This is similar to the results reached by Ali et al. when using this type of fiber [21].

From the results presented in Fig. 1 and Fig. 2, it can be concluded that adding Nano Alumina and synthetic fibers to a certain extent to the cement slurry increases the compressive strength, and the increase will become more evident in subsequent ages, similar to the effect of pozzolanic.

This proves the possibility of using nanomaterials in enhancing compressive strength for diverse cement applications, even outside of the petroleum industry, as demonstrated by Raouf et al. When utilizing nanoparticles in concrete [22] also Ali et al. showed mechanical properties enhancement of Cement when using nanomaterials [23].

3.3. The thickening time of cement slurry at 52 °C and 5160 psi

The thickening time results at 52 °C and 5160 psi for class G Iraqi owc with and without additions are shown in Fig. 3.

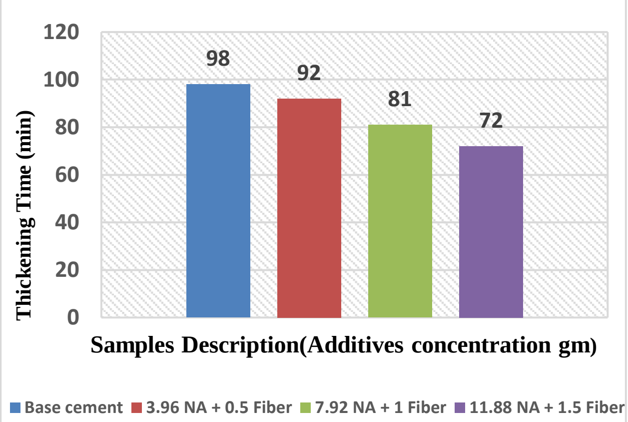

Fig. 3. Thickening Time of Cement Slurry at 52 °C, 5160 psi

The results obtained indicate that the thickening time value of Iraqi Class G cement without any additives (base cement) at a temperature of 52 ° C and pressure 5160 psi was 98 min, which is acceptable and compatible with the requirements of API standard specifications10A range (90-120 min) [5].

Fig. 3 demonstrates that adding combined 0.5% NA (BWOC) and 0.5g of Synthetic Fiber resulted in a slight decrease in thickening time, although it remained within the API standard range (90-120 min). The results also demonstrated that when the amount of combined NA and Synthetic Fiber increased, the thickening time decreased (Bc=100) below the API 10A allowable range (90-120 min). The results indicate that the addition of Nano Alumina in a concentration higher than 1% BWOC significantly reduces the thickening time of cement, making it non-compliant with API requirements. This may be due to the pozzolanic reactions generated by NA (Pozzolanic are Siliceous and Aluminous materials). This reveals that NA reacts with calcium hydroxide $Ca(OH)_2$ in the presence of water at ambient temperature to produce mixes with cementitious properties and a higher hydration rate. Due to their small size, Nano alumina particles can also fill the voids between cement grains and have the ability to absorb the lubricating water contained in the mixture.

3.4. The Economic Comparison

With regard to the economic cost, synthetic fibers are very cheap compared to the usual cement additives, as for nanomaterials, they are very expensive, so the following comparison shows the use of these materials economically.

An economic comparison is performed between Nanoparticles and additives of oil well cement based on their cost. The Nano Alumina (Purity: 99.5%) cost is $392/1kg if it is bought directly from the manufacturer [15]. The Prices of Nanomaterials and additives of oil well cement used in Iraqi oil fields are listed in Fig. 4.

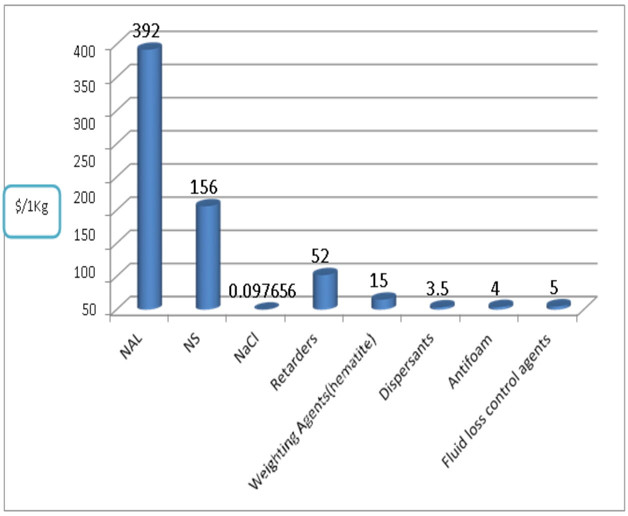

Fig. 4. A Comparison of the Costs of Nanomaterials with Additives

Compared to other materials, nanomaterials are extremely expensive. The following example demonstrates this:

The cost of one ton of cement class G is $400, which increases to $1,000 with the addition of chemical additives; consequently, the cost of cementing operations of production casing with the company's profits and the cost of pumping (if 50 tons are taken) is 150 million Iraqi dinars (100,000 dollars), and the cost of cementing plug (if 10 tons are consumed) is 45 million Iraqi dinars (30,000 dollars) [15].

4- Conclusions

In this study, the effects of the combined Synthetic Fiber and Nano Alumina on the compressive strength and thickening time of Iraqi oil well cement class G were evaluated. The two components worked together to create the composite material. Based on the experimental data and thorough analysis the following conclusions can be drawn:

1. The concentrations of additives of Nano Alumina and Synthetic Fiber significantly affected the properties of cement.
2. The addition of Nano Alumina in small proportions by the weight of the cement with the Synthetic Fiber led to an increase in the compressive strength of the cement under different conditions and slight decrease in thickening time.
3. The addition of 1.5% BWOC of NA with 1.5 grams of Synthetic Fiber (Barolift) increased the compressive strength during the eight-hour curing period by 40.7% and 33.8% at a condition of 38 °C and 60 °C, respectively and decreased the thickening time at 52 °C and 5160 psi by 26.53 %.
4. The addition of Nano Alumina in concentrations higher than 1% BWOC causes a significant decrease in the thickening time of cement; therefore, the addition of Nano Alumina in small proportions ranging from (0.5-1.5%) BWOC was chosen to minimize the impact of Nano Alumina on the thickening time, and the addition of Synthetic Fiber with it in proportions ranging from (0.5-1.5g) was chosen to improve the compressive strength of cement.
5. The ideal proportion of the additives was 3.96 g of NA and 0.5 g of synthetic fibers together, where the compressive strength and thickening time were compatible with API specifications when using these ratios of additives with Iraqi oil well cement class G.

Finally, it can be recommended to conduct other studies, such as rheological properties, on this type of cement using the same additives to see the extent to which it can be applied in Iraqi fields with these additions.

Nomenclatures

API	American Petroleum Institute
BWOC	By Weight Of Cement
HPHT	high pressure and high temperature
HSR	High sulfate-resistant
NA	Nano Alumina
nm	Nanometer
NP	Nanoparticles
NS	Nano Silica
OWC	Oil Well Cement
µm	Micrometer

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دراسة تجريبية لتوضيح تأثير جزيئات الألومينا النانوية والألياف الصناعية على أسمنت آبار النفط فئة G

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الخلاصة

من الضروري أن تكون عملية تدعيم ابار النفط والغاز فعالة من أجل إحراز تقدم في عمليات الحفر والإنتاج بكفاءة. يتركز نجاح عملية التسميت بشكل أساسي على تصميم ملاط الاسمنت. من الخصائص الأساسية لاسمنت الابار النفطية هي قوة الانضغاطية، والتي تشير إلى قدرة المادة على تحمل الظروف القوية بمرور الوقت. في هذه الدراسة تم تقييم وتحسين مقاومة الانضغاط ووقت التثخين لأسمنت آبار النفط العراقي فئة G باستخدام نوعين من الإضافات (نانو ألومينا والألياف الصناعية) لتتوافق مع مواصفات معهد البترول الأمريكي (API). تم استخدام المواد المضافة بنسب مختلفة ، وتم تحضير مجموعة من العينات تحت ظروف مختلفة. تم قياس قوة الانضغاط وزمن التثخين لملاط اسمنت الابار النفطية في ضل ضروف مختلفة. تم اختيار كميات النانو ألومينا (٠,٥ ، ١ ، ١,٥)% من وزن الاسمنت (BWOC) والألياف الاصطناعية (٠,٥ ، ١ ، ١,٥) جرام على التوالي معا. أظهرت النتائج زيادة ملحوظة وكبيرة في مقاومة الانضغاط، وكانت جميع قيم مقاومة الانضغاط متوافقة مع متطلبات معهد البترول الأمريكي (API) واضهرت ايضا انخفاضا في زمن التثخن لاسمنت الابار النفطية العراقي، اعتمادا على نسب المواد المضافة. كانت أكبر زيادة في مقاومة الانضغاط في العينة التي تحتوي على ١,٥٪ NA BWOC و ١,٥ جم من الألياف الاصطناعية (Barolift)، حيث زادت مقاومة الانضغاط بنسبة ٤٠,٧% و ٣٣,٨% عند درجة حرارة ٣٨ درجة مئوية و ٦٠ درجة مئوية على التوالي، لكن زمن التثخين انخفض بنسبة ٢٦,٥٣ % عند هذه النسبة من المواد المضافة. وأظهرت النتائج أن استخدام هذا النوع من المضافات هو وسيلة مجدية لتحسين كفاءة اسمنت آبار النفط العراقي فئة G لزيادة مدى امكانية تطبيقه في حقول النفط العراقية.

الكلمات الدالة: سمنت الابار النفطية، مقاومة الانضغاط، نانو الومينا، الفايبر الاصطناعي.