Experimental Investigation of Nano Alumina and Nano Silica on Strength and Consistency of Oil Well Cement

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ABSTRACT

In oil and gas well cementing, a strong cement sheath is wanted to insure long-term safety of the wells. Successful completion of cementing job has become more complex, as drilling is being done in highly deviated and high pressure-high temperature wells. Use of nano materials in enhanced oil recovery, drilling fluid, oil well cementing and other applications is being investigated. This study is an attempt to investigate the effect of nano materials on oil well cement properties. Two types of nano materials were investigated, which are Nano silica (>40 nm) and Nano Alumina (80 nm) and high sulfate-resistant glass G cement is used. The investigated properties of oil well cement included compressive strength, thickening time, density, free water and rheological properties. All tests are conducted according to API specification and proceed in Laboratory of Drilling in Petroleum Technology Department in University of Technology and in Missan Oil Company. The experimental results show that NS and NAL behave like accelerators when added to cement and work to increase the compressive strength at 38°C but these increasing in compressive strength changes when the temperature is increased to 60°C. Also, adding NS and NAL lead to increasing in rheological parameter and reduce free water but the change in density is very small. The results show that the effect of NAL on compressive strength and thickening time is greater than the effect of NS but the effect of NS on free water and rheology is greater than the effect of NAL.

Key words: nano silica, nano alumina, oil well cement.

التحري المختبري للنانواولومينات و النانوسيليكا على مقاومة و تماسكية سمنت الابار النفطية

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الخلاصة

في عمليات تسميت أبار النفط والغاز فان وجود غلاف أسمنتي صلب يحيط بالبطانة هو من الامور المهمة لضمان سلامة طويلة الأجل للأبار. ان أكمال عمليات التسميت بنجاح أصبح أكثر تعقيدا بسبب زيادة الانحراف بعمليات الحفر وكذلك بسبب الضغوط و درجات الحرارة العالية للابار لذلك بدأت الدراسات وعمليات التحري عن أستخدام المواد النانوية في تطبيقات الاستخلاص المعزز للنفط و سوائل الحفر و عمليات التسميت وبقية التطبيقات الاخرى. هذه الدراسة هي محاولة للتحري عن تأثير مواد النانو على خصائص أسمنت آبار النفط. تم أستعمال نوعين من مواد النانو والتي هي نانو السيليكا (أصغر من 40 نانومتر) ونانو الألومينا (80 نانومتر) و كذلك تم أستعمال أسمنت عالي المقاومة من الصنف (G) حيث تمت دراسة تأثير هذه المواد على مقاومة الانضغاط، زمن التثخن، نسبة الماء الحر، الكثافة و على صفات الانسيابية لعجينة الأسمنت. أظهرت النتائج المختبرية ان المواد النانوية المضافة تتصرف كمعجلات لزمن التصلب و تعمل على زيادة مقاومة الانضغاط لصخرة الأسمنت عند درجة حرارة 38 درجة مئوية و لكن هذه الزيادة تتغير عند زيادة الحرارة الى 60 درجة مئوية. كذلك فأن أضافة المواد النانوية أدى الى زيادة معاملات

الخواص الأنسيابية والى أنخفاض نسبة الماء الحر لعجينة الأسمنت بينما كان التغير في الكثافة قليل جدا. وفقا لنتائج ال R^2 فأن موديل Power Law يعطي أفضل تطابق لتمثيل الخواص الانسيابية بالمقارنة مع الموديلات الاخرى. أظهرت النتائج أن تأثير نانو الألومينا على مقاومة الانضغاط و زمن التثخن هو أكبر من تأثير نانو السيليكا ولكن تأثير نانو السيليكا على نسبة الماء الحر والصفات الانسيابية أكبر من تأثير نانو الألومينا.

1. INTRODUCTION

Cementing of casing is one of the most basic steps in the drilling and completion of oil and gas wells process. Technology of well cementing is the application of many scientific and engineering disciplines. For successful cementing jobs, basic factors must consider such as: drilling fluid condition, spacers and flushes using, pipe movement, casing centralizing, displacement rate maximizing, slurry design for down hole condition, cement compositions selecting and testing, and a proper system selecting of cementing process, **Crook, et al., 2001**. Slurry that is used in cementing process must have suitable performance requirements such as low viscosity to make pumping process easy, low permeability, early strength development, and superior resistance to attack from aggressive fluids, **Griffin, 2013**. Using nanotechnology in the field of materials increases its durability and provides materials with super performance. It also enables best usage of resources of the nature and getting the required materials properties with minimal usage. In recent years, great efforts have been done to improve drilling and construction cement properties by using nano particles. The addition of nano fine particles can improve the properties of cement due to the effect of increasing surface area and reactivity through filling the nano pores of the cement. Nano silica, nano calcium carbonate, carbon nanotubes, nano titanium, nano alumina and polymer/clay Nano composites are the most generally used nanomaterial in construction industry. They are mostly used to reinforce different mechanical properties of the cementing materials such as crevice resistance, corrosion resistance, tensile strength and compressive strength, **Iqbal, and Mahajan, 2012**. In this study, the effects of Nano Alumina and Nano Silica on oil well cement properties are investigated. Free fluid, compressive strength, thickening time, density and rheological properties are measured. According to **Shahriar, 2011**, the fundamental knowledge of oil well cement slurry rheology is necessary to evaluate the ability to remove mud and optimize slurry placement. The rheology of fluids also has a major effect on solids setting and free fluid properties and also on the friction pressures, **Joel Of, 2009**. The intention of a free fluid test is to help determine the quantity of free fluid that will gather on the top of cement slurry between the time it is placed and the time it gels and sets up, **Joel OF, 2009**.

2. EXPERIMENTAL WORK

2.1 Materials

The cement powder used in this study, class G (HSR) cement according to API standards, was provided by Missan Oil Company, as shown in **Table1**. Nano Alumina and Nano Silica are used as additives to cement slurry. Nano material properties are shown in **Table 2** and **Table 3**.

2.2 Mixing Procedure

The sample preparations were done in accordance with API, **API Spec. 10A, 2002** and **API. 10B2, 2013**. The motor of mixer is opened and kept at 4 000 r/min ± 200 r/min while the cement with additive is added at a regular rate during no more than 15 sec. After that, putting the cover on the blending container and stay mixing at 12 000 r/min ± 500 r/min for 35 sec ±1 sec. the amount of water to cement is 44%bwoc.

2.3 Determination of Compressive Strength

Compressive strength of set cement is the force of compression required to crash the cubic of cement divided by the cross-sectional area of the cubic. Static and dynamic stresses effect on the column of cement inside the well. Static stress is due to the weight of casing and due to compressive stress that formed by fluid and formation activity. Dynamic stress is due to the process of drilling. To resist these stresses, C.S. of set cement is needed to test according to API procedure. The cement slurry to be tested was placed in the prepared molds and placed the specimens in the curing bath at atmospheric pressure and heating to test temperature 38 °C for 8 hr and to the second test temperature 60 °C for 8 hr. Compressive strengths were calculated using Eq. (1) based on the force that applied on surface area of cubes.

Compressive Strength = Force of Compression / Cross-Sectional Area (1)

Compressive strength of all cubes of sample should be recorded and found the middle value to the closest 50 kPa (10 psi). Compressive strength for 66% of cubes and the average of all results should be exceed the limit indicated in API (300psi at 38^{o}C and 1500psi at 60^{o}C).

2.4 Determination of Thickening Time

Thickening time is the timeframe of cement slurry to stay having the properties of pumping. It is the time needed for the consistency of slurry to reach 100 Bc under bottom hole condition. It is measured by utilizing cement consistometer. Thickening time is influenced by pumping rate because vortexes and streams resulting from turbulent flow increase it. And it is affected by softness of cement. The additives of thickening time are accelerators to reduce it and retarders to increase it. Accelerators are utilized for cementing shallow wells and surface casings. Retarders are used for cementing deep and hot wells. Practically, thickening time must be at least 25% greater than the necessary time for the cementing processes. Cement slurry to be tested was placed in the slurry container and then it is placed into the Consistometer. Rotating slurry container is by motor at (150r/min ± 15r/min) and controlling testing pressure and temperature to simulate the conditions inside the well by following the steps in schedules 5 in API. At 100 Bearden Consistency, the slurry considered un-pumpable and the test is finished. The acceptance requirement for thickening time must be between 90 min and 120 min.

2.5 Determination of Rheological Properties

Cement slurry is placed in atmospheric pressure consistometer for 20 minutes at 100 ° F. Then, the slurry is transferred to the viscometer device. The viscometer will automatically measure, compute and show shear stress readings according to API recommended practice 10 B2. Three models (Bingham plastic, Power law and Herschel-Bulkley) are used to represent the relationship between shear rate and shear stress.

2.6 Determination of Free Fluid

Cement slurry is placed in atmospheric pressure consistometer for 20 minutes at 100 ° F. After that, 760 g (according to API 10B2) of cement slurry was poured into a 500 cc (500 ml) graduated flask. Closing the mouth of the flask and putting it on a vibration free surface for 2 hours. The slurry was then examined for any free fluid on the top of the cement column. This free fluid was decanted and measured with a syringe to determine the percent of free water (ϕ) based on the weight and the specific gravity of the cement using Eq. (2).

$$\Phi = ((VFF * \rho) / ms) * 100 \tag{2}$$

Where VFF is the volume of free fluid (milliliter); ρ is the specific gravity of the slurry, and ms is the mass of the slurry (gram).

3. RESULTS AND DISSCUSION

3.1 Thickening Time Results

This section focuses on the thickening time behavior of oil well cement with NS and NAL. **Fig.1** and **Fig.2** present the results of seven concentrations by weight of cement of NAL and NS on thickening time of cement slurry. The biggest decline for thickening time value is about 30.2% when the addition of NS is 3%bwoc and the biggest decline for the thickening time value is about 31.25% when the addition of NAL is 3%bwoc. According to API, the time required to reach the consistency of oil well cement to 30 Bc must exceed 30 minutes. The results showed that all the additions of NS&NAL led to a very significant drop in thickening time at 30 Bc; therefor, all consistency after 30 minutes is greater than 30 Bc and this does not conform with the requirements of the API. Thickening time of oil well cement slurry with NS additives is shorter than thickening time of cement without NS additive because NS reacts rapidly with sulphate and make high amount of calcium sulphoaluminate (attringite, AFt) in the early hydration period and hence reduces the thickening time. These results (decreasing in thickening time) are similar to the results obtained by **Gopalakrishnan, 2015**. Thickening time of oil well cement with NAL additives is shorter than thickening time of cement without NAL additive. This may be due to the pozzolanic effect of NAL. That means it reacts chemically with $Ca(OH)_2$ at ordinary temperature in the presence of water so it leads to increase the speed of hydrogenation. Nano materials (NS and NAL) accelerated the thickening time of cement slurry class G. It is well known in oil well cementing practices, salts such as NaCl, **Rike, 1973**, and $CaCl_2$, **Michaux, 1990**, are used for the same purpose. This section will introduce a comparison between these accelerators in terms of thickening time property. **Fig. 3** presents this comparison.

3.2 Compressive Strength Results

This section focuses on the compressive strength behavior of oil well cement with NS and NAL. **Fig. 4** through **Fig. 7** presents the results of seven concentrations by weight of cement of NAL and NS on compressive strength of cement slurry. Compressive strength of oil well cement at 38°C is increased when amount of NS&NAL is increased. The reason of this strength improvement is due to the small size of nano particles in pores between cement particles; therefore, it increased the efficiency of the packing. The second reason is due to the effect of (NS&NAL) on promoting the pozzolanic interaction. That means it reacts chemically with $Ca(OH)_2$ (formed from dehydration interaction) at ordinary temperature in the presence of water and produce compounds having cementitious properties. At 60^oC, compressive strength is decreased when the amount of NS exceeds 2% bwoc. This means that raising the quantity of NS is good in increasing compressive strength to a specific cutoff after which further increase in the NS quantity leads to a reducing in the compressive strength. The reason for that truth is the additional rate of NS could prompt agglomeration of its particles under the present dispersion circumstance. This agglomeration happens when adding too much amount of NS because of the high surface area of nano-particles that leads to raise the tendency of these particles to pull each other's forming weakly clogs, therefore, decreases the compressive strength. Likewise, these clogs fill the voids of the cement and prevent the filling effect of nano particles for these voids and thus diminishing the cement strength. This agglomeration causes the active surface area that interacts to form the C-S-H gel to reduce furthermore prompts to form weak clusters that cannot support large strength causing a decline in the compressive strength of the cement. Another conceivable clarification is that the mixing water is insufficient to coat this large quantity of the tiny NS particles causing defects in the hydration process and formation of C-S-H gel. Therefore, they decrease compressive strength. At 60^oC, change in compressive strength happens because temperature is a factor that effect on pozzolanic interaction. Compressive strength of slurry is decreased when NAL is added in 1% and 2%bwoc and then it is begun to rise at 3%bwoc. This big change in the behavior of compressive strength is the result of change in temperature that it is increased to 60°C because Pozzolanic interaction occurred at ordinary temperature. Cement is considered unsuccessful when adding 1%bwoc of NAL because compressive strength has become under the allowable limit that is specified by API.

The effect of size of NS particles on the physical properties of cement will be study by making a comparison between the compressive strength test conducted by **Yausif, 2014**, and the results obtained in this study. From **Fig. 8** and **Fig. 9**, it can be observed that the improvement in the compressive strength of the cement slurry is increased when the size of NS became small but this improvement in the compressive strength arrives to a specific cutoff at low quantity of NS when the size of NS is larger than 50nm. The reason for this behavior is due to decrease of packing activity when the particle size of NS is increased and that leads to a decline in the compressive strength. Also, surface area of NS is increased when particle size is decreased and this leads to prompting pozzolanic reaction. At 60°C, the change in behavior of Compressive Strength between NS > 40nm and NS < 50nm is small when the amount of NS is 2%bwoc but Compressive Strength of NS > 40nm is greater than Compressive Strength of NS < 50nm when the amount of NS is less than 2%bwoc. In **Fig.10**, comparison is made between the impacts of adding nanomaterials to oil well cement. These nanomaterials are multi-wall CNT, **Nasiri, et al., 2014**, Ni NP, **Liu et al.2013**, nanoclay, **Rahman and Murtaza, 2015**, nano bentonite, **Roddy et al., 2010**, NS and NAL.

3.3 Result of Density Tests

From **Fig. 11** and **Table 4**, the addition of NS leads to a very small decrease in the density that it can be neglected while adding NAL leads to a small increase in the density, therefor; the effect of nano materials (NS & NAL) on slurry density is very small compared with the impact of materials (see **Table 5**) which add to improve density.

3.4 Free Water

This section focuses on free water behavior of bowc slurry with NS and NAL as additives. The results of the six cement mixtures are listed in **Fig. 12** and **Table 6**. The results are also compared with net cement in order to illustrate the change due to adding NAL and NS. The results are also compared with the effect of HPMC polymer. (HPMC) is self-active viscosifier polymer. It is stay stable when temperature is increased and promote viscosity at gelation temperature without any other chemical additives, **Abbas, et al., 2013**. It has feature to work as thickener, film foamer, suspension aid, water retention agent and surfactant at high temperature, **Sarkar, et al., 1995**. From **Fig. 13**, it can be seen that HPMC is more effective than NS and NAL on free water.

3.5 Rheological Results

This section focuses on the Rheological behavior of bowc slurry with NS and NAL as additives. The relationship between shear stress and shear rate and the models that represent these relationships are appeared in **Fig. 14** through **22**. These models are power-law, Bingham and Herschel-Bulkley model. According to R^2 results, power law model is the best one that represents the relationship between shear stress and shear rate. The addition of NS leads to increase plastic viscosity in Bingham model and power law exponent in power-law model. Plastic viscosity in Bingham model and power law exponent in power-law model is inversely proportional with Reynolds number. Therefore, the increasing of them leads the flow staying away from the turbulent region and that is not good in cementing process. For rheological parameters (k, final gel strength, μp and Yield-point), NS additives improves these parameters more than NAL. While for flow behavior index, each additives has approximately the same effect.

3.6 The Economic Comparison

Economic comparison is made between nano materials and additives of oil well cement based on the prices of them. The price of gamma Aluminum Oxide Nano powder (Purity: 99.5%, APS: 80 nm) is $392/1kg if it is bought directly from the company and the Price of Silicon Dioxide (SiO2) Nano powder (Purity: 99+%, 20-30 nm) is $156/1kg if it is bought directly from the company. The price of nano materials and additives of oil well cement that used In Iraqi oil fields are listed in the **Fig. 23**. Nano-materials price is very high compared with other materials. This can be seen through the following example:

The price of one ton of Omani cement class G is 400$ and with the chemical additives becomes 1000$, therefor, the cost of cementing operations of production casing with the company's profits and the cost of pumping (if consumed 50 tons) becomes 150 million Iraqi dinars (117187.5$) and the cost of cementing plug (if consumed 10 tons) is 45 million Iraqi dinars (35156.25$).

4. CONCLUSIONS

Based on this experimental study, several conclusions can be derived:
1- The experimental results show that NS and NAL behave like accelerators when added to cement.

2- The effect of nanoparticles (NAL and NS) on thickening time is less than the effect of conventional accelerators (NaCl, $CaCl^2$).

3- At 38°C, compressive strength is directly proportional to the quantity of NS and NAL and the effect of NAL on compressive strength is higher than the effect of NS.

4- When a comparison is made between the results of this study and the results conducted by Zakia A. Yausif. It can be said that compressive strength is affected by the size of NS. When the size of NS is increased, the improvement of the compressive strength is decreased.

5- NS is more effective in increasing compressive strength at 60°C but NAL is more effective in increasing compressive strength at 38°C.

6- The increase in compressive strength of oil well cement is due to the pozzolanic effect of NS and NAL which increases when the surface area of particle is increased and due to the packing effect of the small particle.

7- An increase concentration in NAL and NS resulted in higher plastic viscosity, yield point and gel strength for oil well cement and the effect of nanoparticles (NAL and NS) on density is very small.

8- According to R^2, the best model that reflects the relationship between shear stress and shear rate is power law model.

9- An addition of nanoparticles (NAL and NS) into oil well cement gives better results by reducing free water of slurry.

10- Results show that the effect of NAL on compressive strength and thickening time is greater than the effect of NS but the effect of NS on free water and rheology is greater than the effect of NAL.

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NOMENCLATURE

Aft = alumino-ferrite trisulfate hydrate

API = american Petroleum Institute

Bc = bearden Consistency

BWOC = by Weight Of Cement

C.S. = compressive strength

CMC = carboxymethylcellulose

CMHEC = carboxymethylhydroxyethylcellulose

CNT = carbon Nano-Tubes

C-S-H = calcium silicate hydrate

HPG = hydroxypropylguar

HPMC = hydroxypropylmethylcellulose

HSR = high sulfate-resistant

mw-CNT = multi-wall Carbon Nano-Tubes

n = power law exponent

NAL = nano-Al2O3

Ni NP = nickel nanoparticles

NS = nano silica

OPC = ordinary Portland cement

OWC = oil well cements

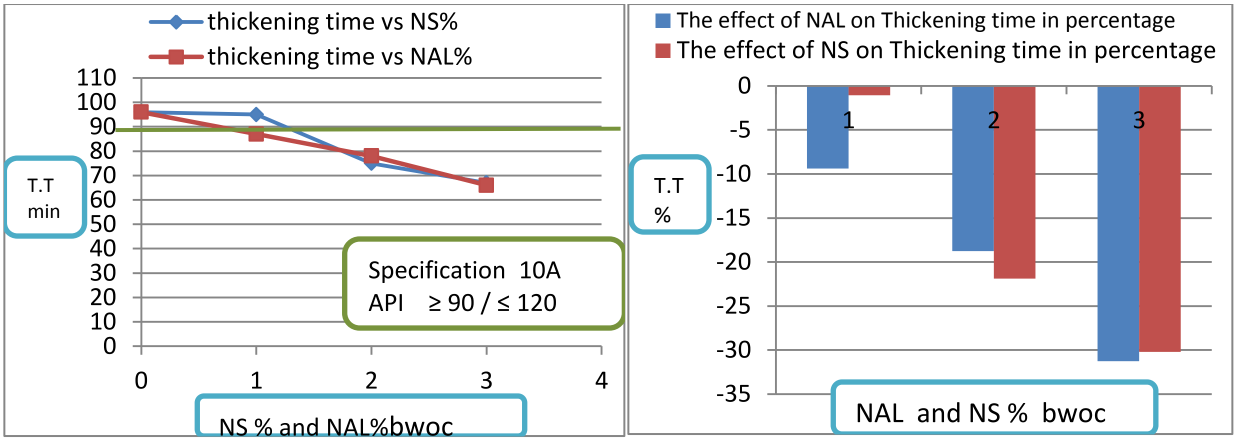


Figure 1. Thickening time (BC=100) vs NS&NAL% bwoc at 52°C and 5160 psi and in percentage.

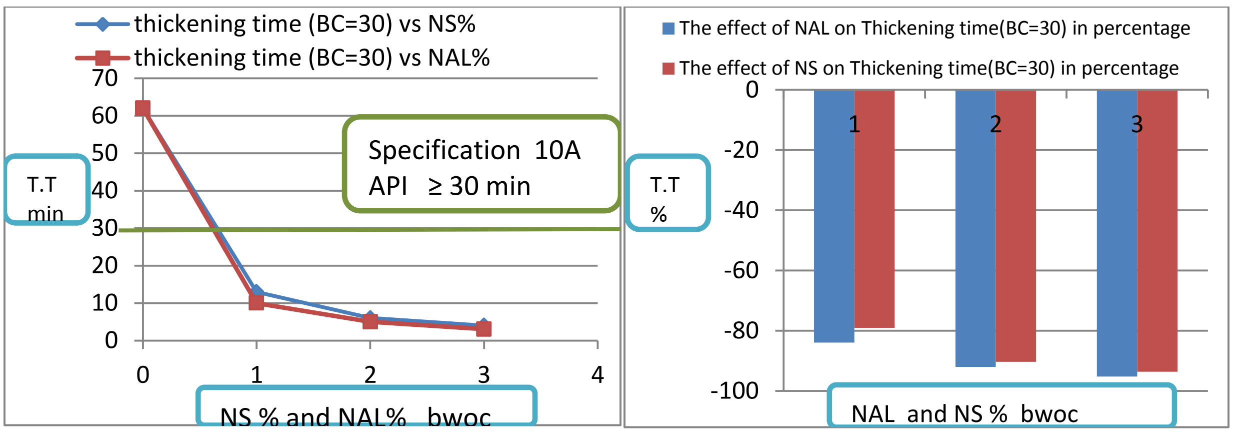


Figure 2. Thickening time (BC=30) vs NS&NAL% bwoc at 52°C and 5160 psi and in percentage.

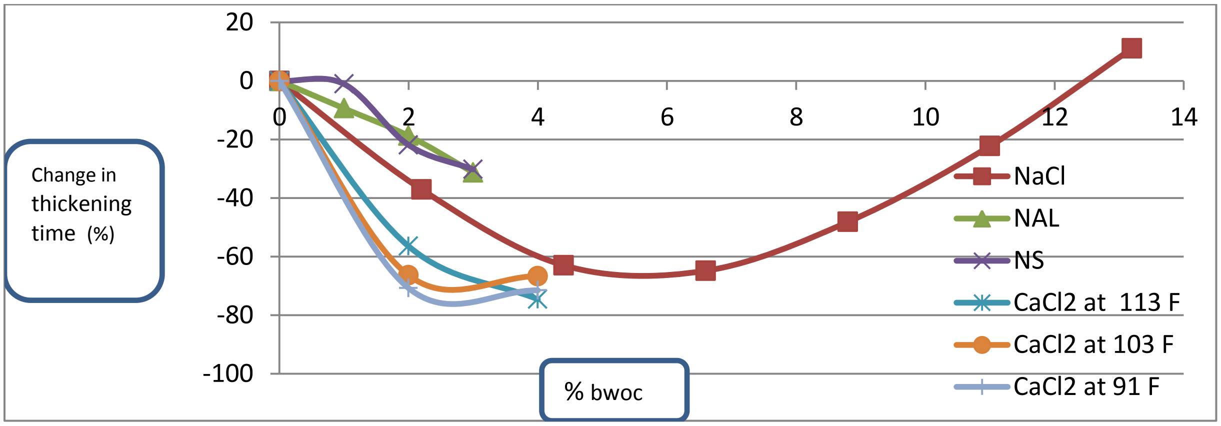
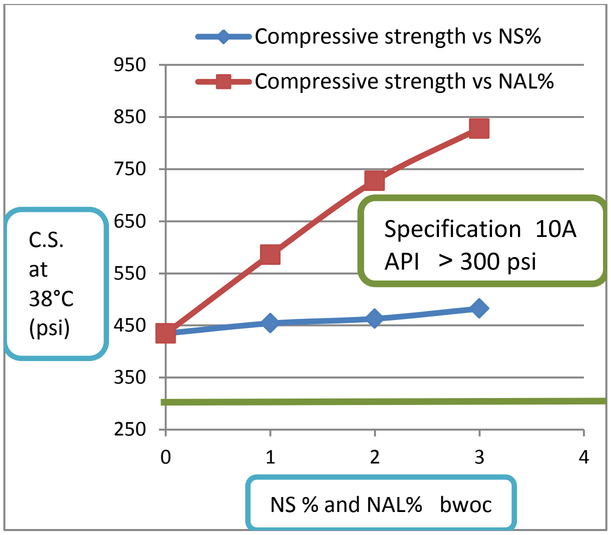


Figure 3. Comparison between the effects of nanomaterials and accelerator.



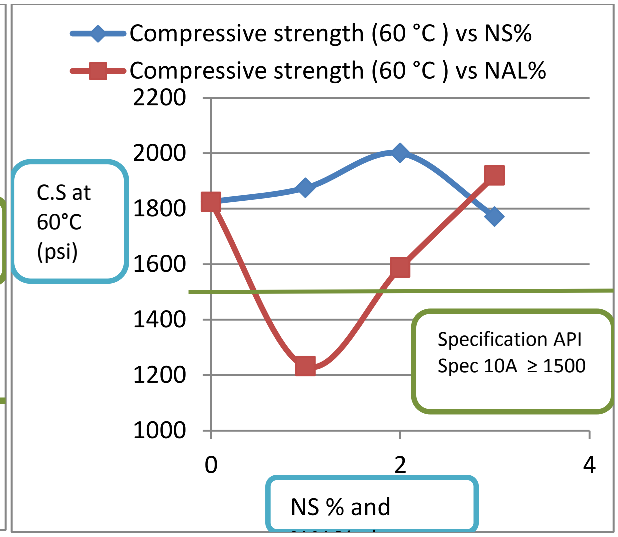
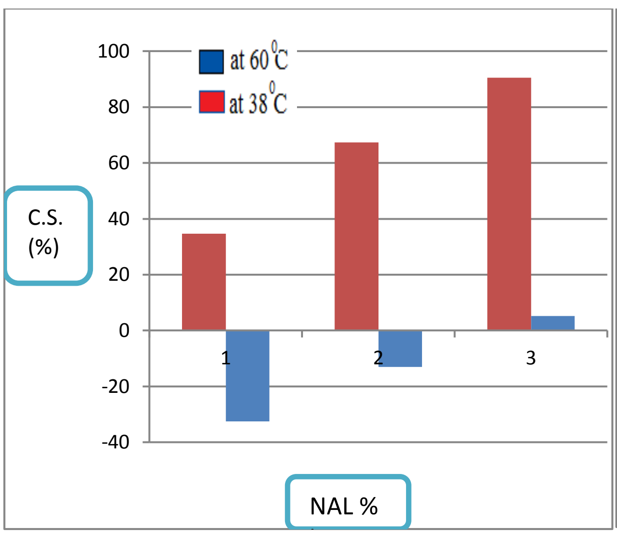


Figure 4. C.S. vs. NS and NAL% bwoc at 38^{o}C. **Figure 5.** C.S. vs. NS and NAL% bwoc at 60^{o}C.



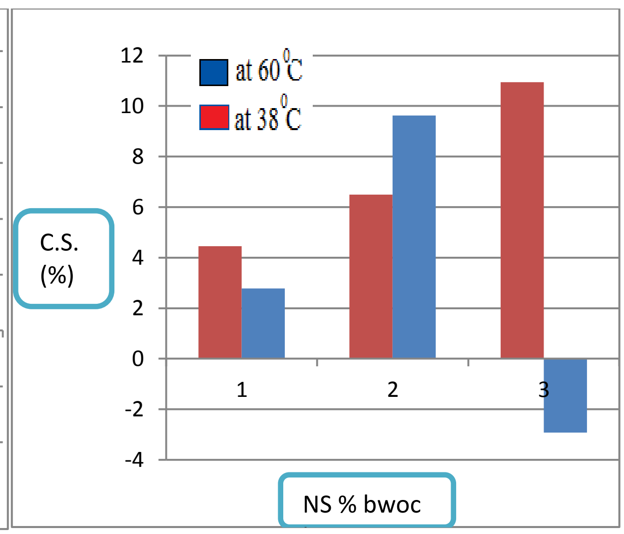


Figure 6. The effect of temperature on C.S. of cement slurry with NAL.

Figure 7. The effect of temperature on C.S. of cement slurry with NS.

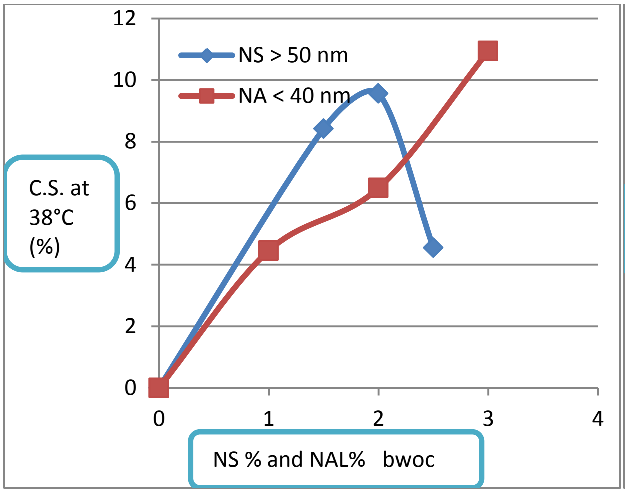


Figure 8. Comparison between the C.S. 38°C conducted by **Yausif, 2014**, and the results obtained in this study.

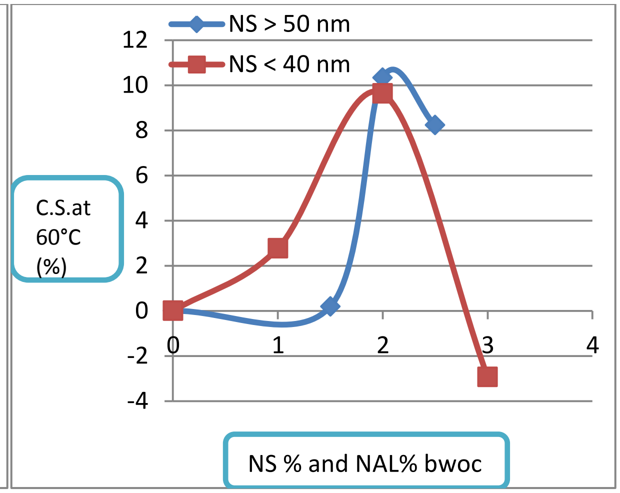


Figure 9. Comparison between the C.S. at 60°C conducted by **Yausif, 2014**, and the results obtained in this study.

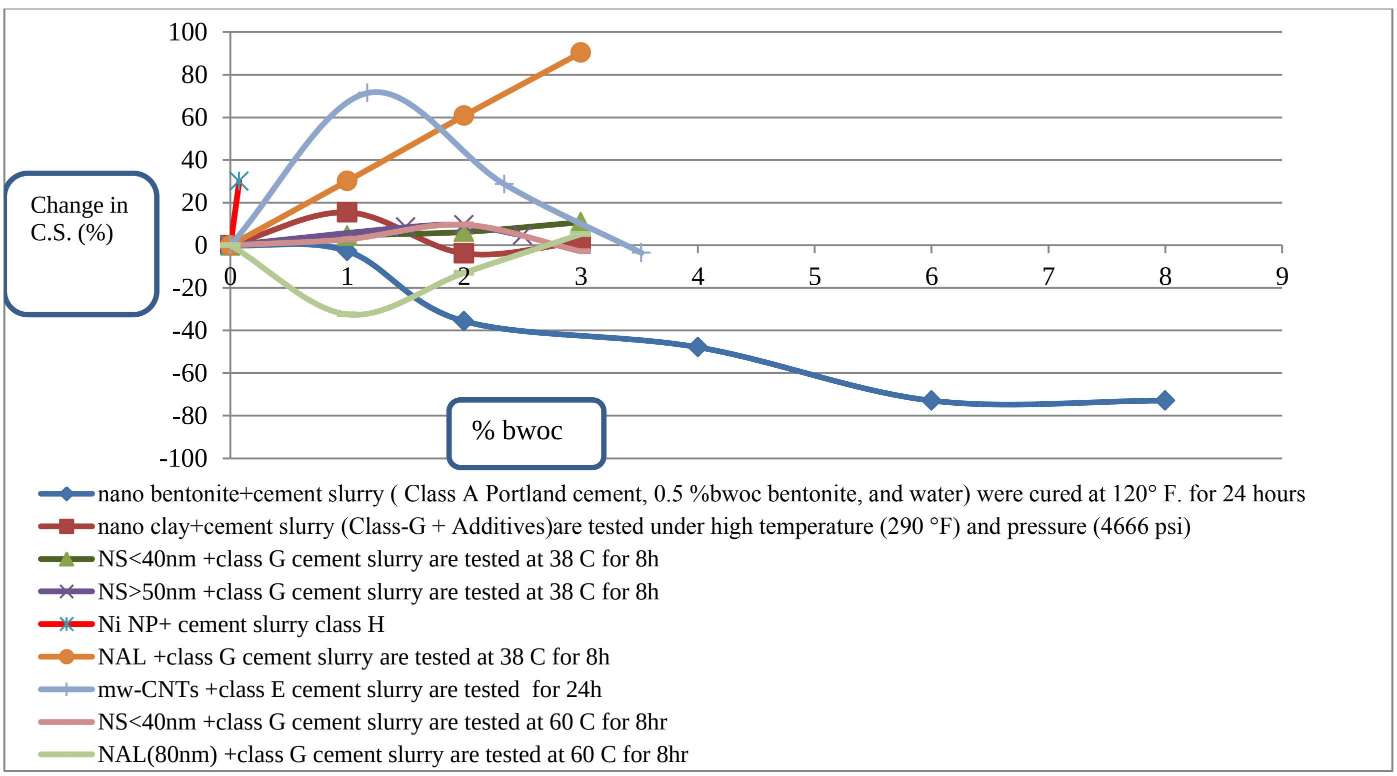


Figure 10. Comparison between the Effects of Nanomaterials on Compressive Strength.

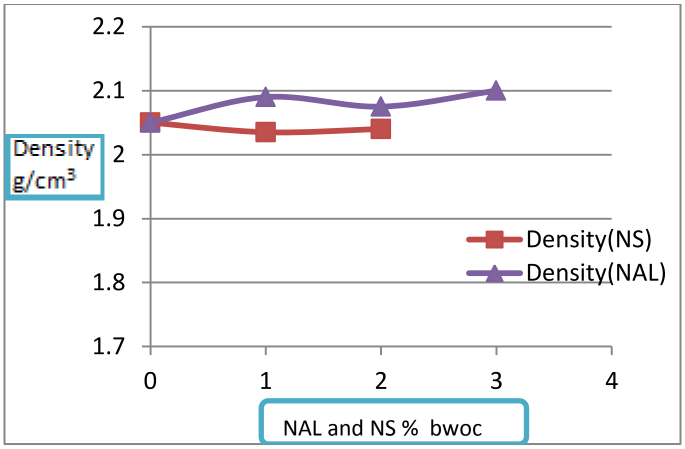


Figure 11. The effect of NS and NAL on density.

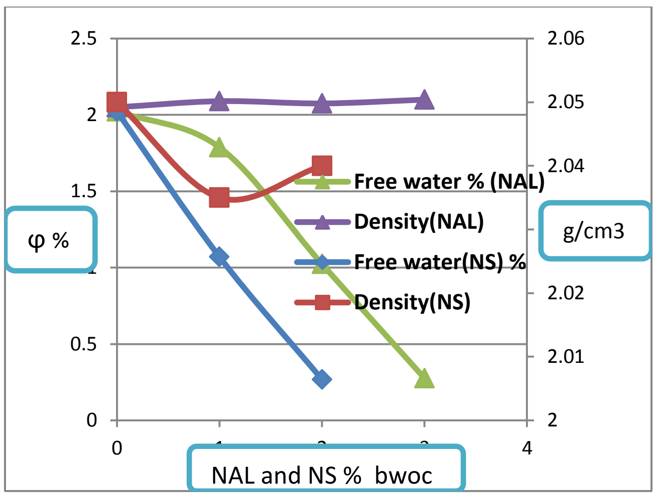


Figure 12. Free water % and density vs. NS and NAL %bwoc.

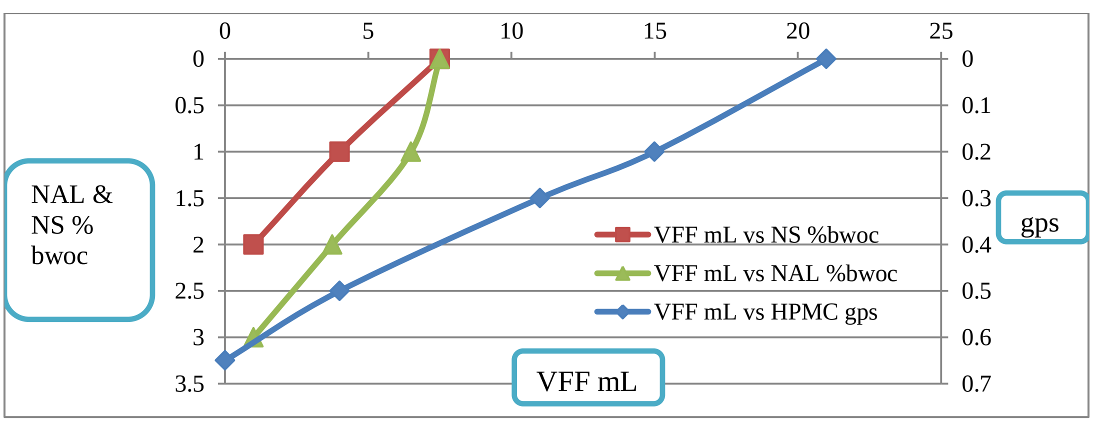


Figure 13. A comparison between the effect of nanomaterials and HPMC, **Abbas et al., 2014**. on the free water of slurry.

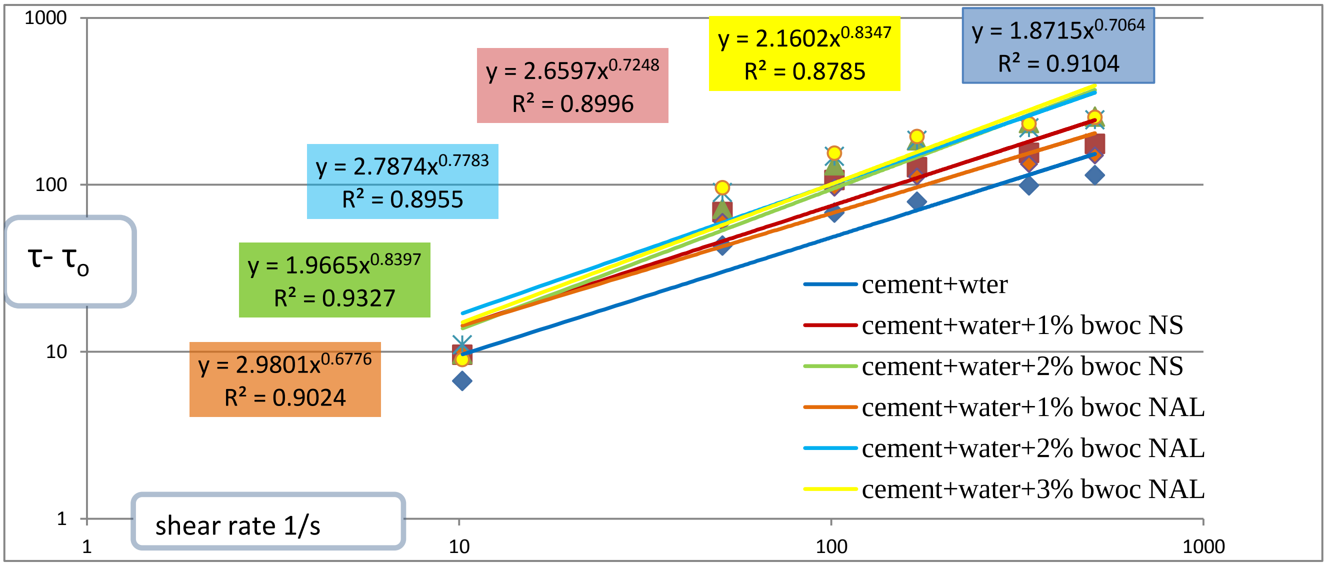


Figure 14. Herschel-Bulkley model.

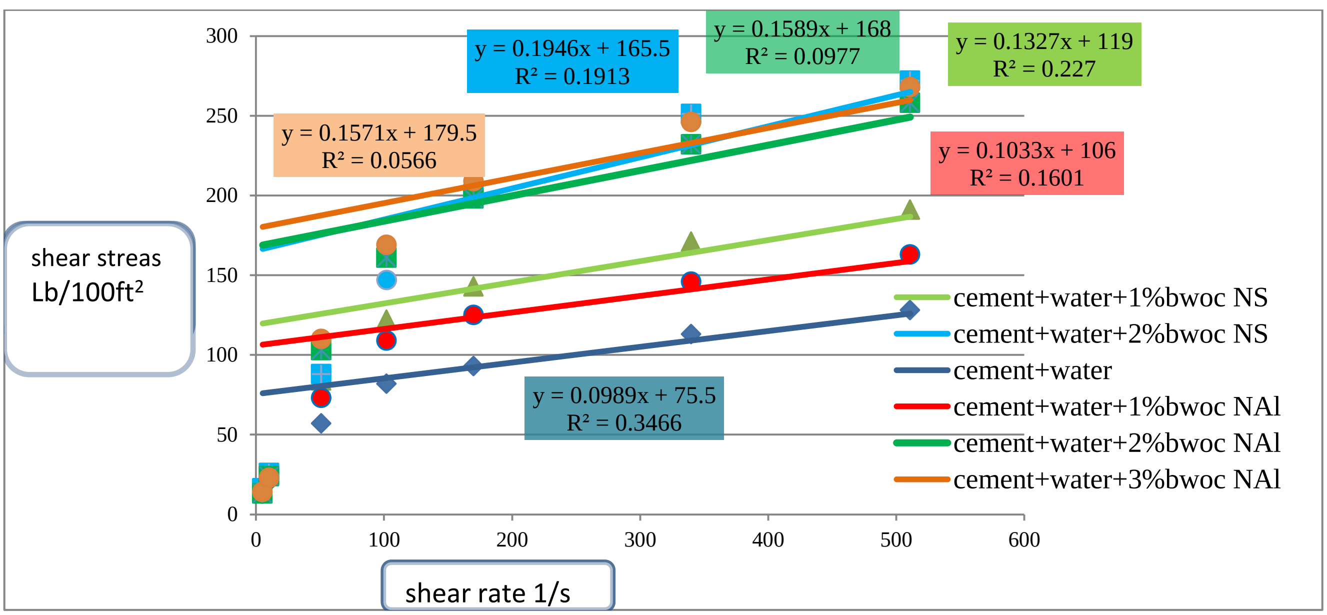


Figure 15. Bingham model.

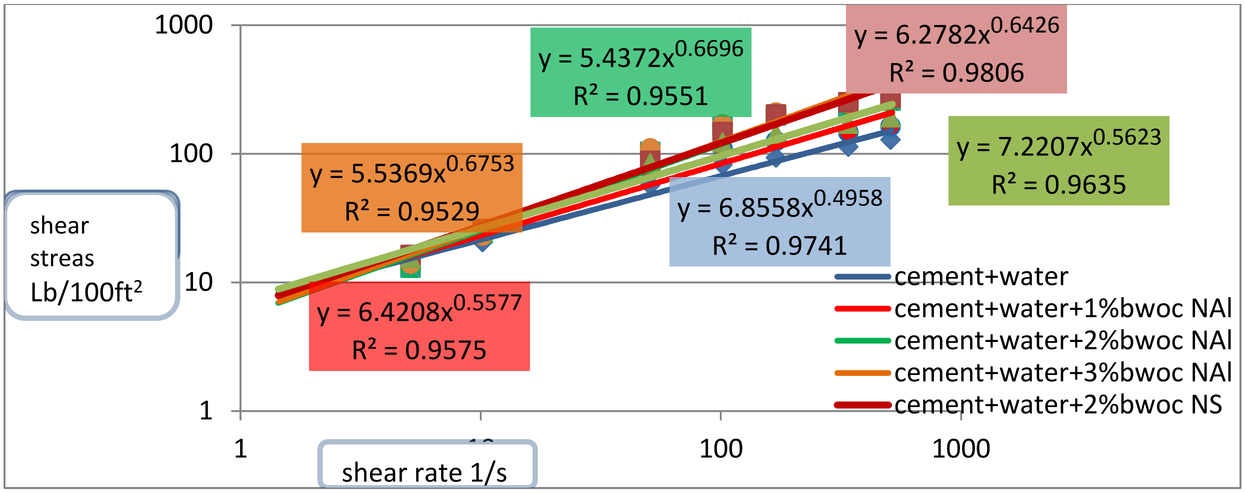
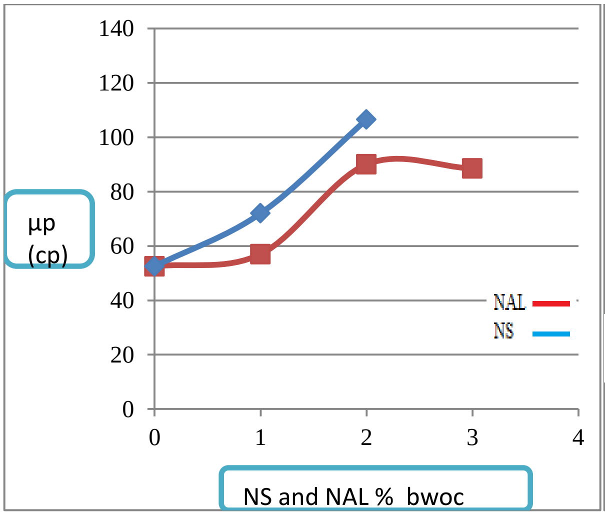


Figure 16. Power-law model.



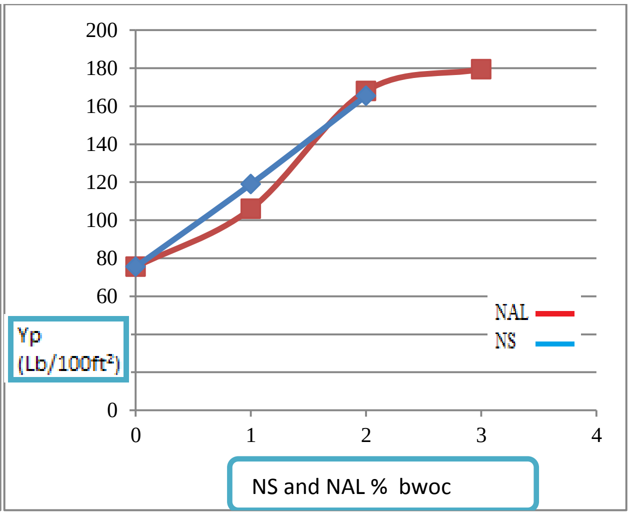
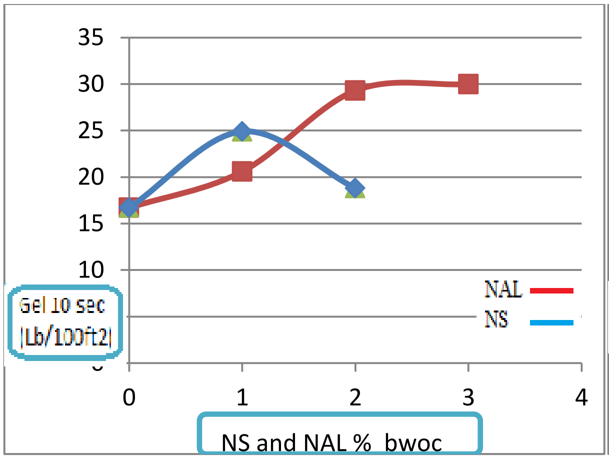


Figure 17. µp vs. NS and NAL % bwoc.

Figure 18. yp vs. NS and NAL % bwoc.



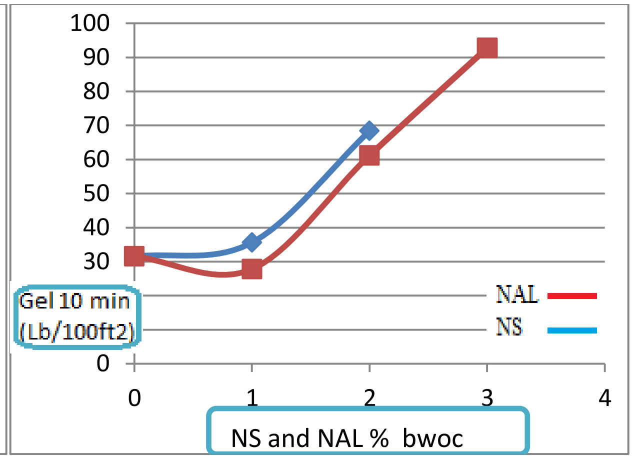
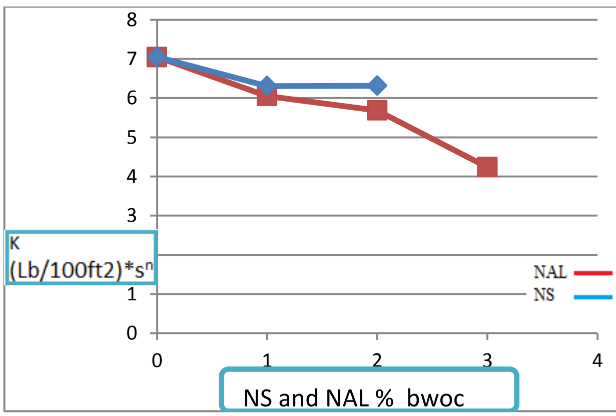


Figure 19. 10 sec Gel vs. NS&NAL % bwoc. **Figure 20.** 10 min Gel vs. NS&NAL % bwoc.



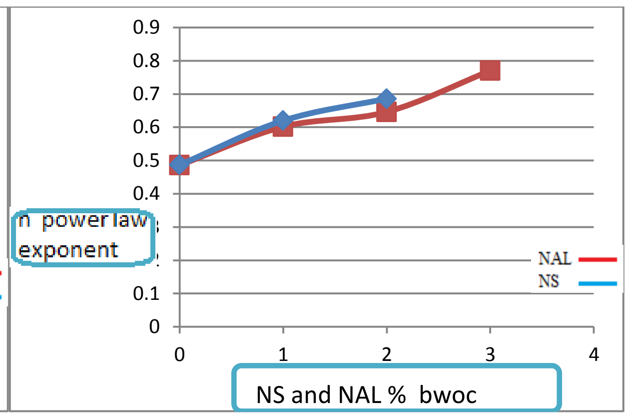


Figure 21. Consistency index vs. NS&NAL % **Figure 22.** Power-law exponent vs. NS&NAL %.

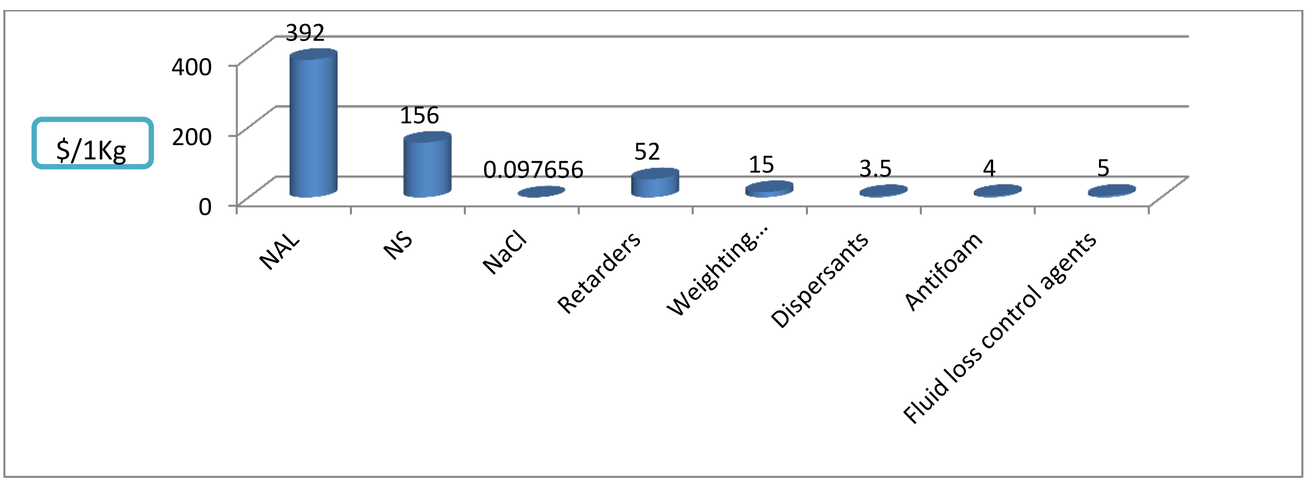


Figure 23. A comparison between the prices of additives with nano-materials.

Table 1. Chemical composition of cement that used in this study.

Chemical composition		
	Mean value	Specification API Spec 10A
MgO. %	0.6	≤ 6.0
$S0_3$. %	2.7	≤ 3.0
Loss on ignition, %	1.3	≤ 3.0
Insoluble residue, %	0.09	≤ 0.75
C3S. . %	56	≥ 48 / ≤ 65
C3A. %	1.7	≤ 3.0
Since TM is 0.75 (> 0.64)		
(C4AF + 2 x C3A), %	19.7	≤ 24,0
Na_2O-equivalent. %	0.57	≤ 0,75

Table 2. Specifications of NAL.

Product	Nano Aluminum Oxide
Size	80 nm
Purity	99%
Crystal	Gamma
Morphology	Needles
Form	Powder
Price	1g = 1.43$
Aluminum Oxide Nanopowder Dosage:	Recommended dosage is usually 1 to 5%

Table 3. Specifications of nano silica.

Product	SIO_2
Size	<40 nm
Purity	99.5%
Characteristics	Large specific surface area, more alkyl, hydrophilicity. It is a non-toxic, odourless and non-polluting inorganic non-metallic material with stable chemical property.
Crystal /surface treatment by	No
Form	Powder
Color	White
Price	1g = 1.43$
Recommended dosage	Ranging from 0.5% to 5%

Table 4. The effect of NS and NAL on density.

		Lb/gal	kg/M^3	g/cm^3
Cement + water		17.1	2050	2.05
Cement + water +NS % bowt	1	16.95	2035	2.035
	2	17	2040	2.04
Cement + water +NAL% bowt	1	17.45	2090	2.09
	2	17.25	2075	2.075
	3	17.5	2100	2.1

Table 5. Common weighting agents and extenders used in well cementing**, Saasen et al., 1996** and **Nelson et al., 2006.**

Extender	Slurry densities obtainable (lb/gal)	Weighting agents	Typical maximum slurry density (S.G) g/cm^3
Bentonite (clay based extenders)	11.5-15	Barite	2.28
Fly ash	13.1-14.1	Hematite	2.64
Sodium Silicates	11.1-14.5	Ilmenite	>2.40
Microspheres	8.5-15.0	Manganese tetraoxide	2.64
Silica Fume	≥11		
Foamed Cement	6.0-15.0		

Table 6. The effect of NAL and NS on free water parameter.

		g/cm^3	VFF ml	ϕ =
Cement + water		2.05	7.5	2.023026
NS % bowt	1	2.035	4	1.071053
	2	2.04	1	0.268421
NAL% bowt	1	2.09	6.5	1.7875
	2	2.075	3.75	1.023849
	3	2.1	1	0.276316