AN IMPROVEMENT TO PHYSICAL PROPERTIES OF HEAVY-WEIGHT OIL WELL CEMENTS USING CARBON NANOTUBES

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ABSTRACT

This study experimentally investigates the effect of multi-walled carbon nanotubes (MWNT's), as a reinforcing material, on the physical properties of heavy-weight oil well cements. A candidate well is selected and the properties of the cement slurry used in a problematic section of the well are tested in the laboratory. Carbon nanotubes (CNT's) are added as fibers to the cement slurry and the improvements in the cement slurry and stone properties are studied. This work discusses the problems associated with conventional heavy-weight oil well cement used in the candidate well and reports the detail of the improvements on cement properties obtained by adding CNT's to cement slurry formulation. These properties include cement slurry rheological properties, free water, fluid loss, thickening time, cement stone elasticity, and compressive strength. When only 1 wt.% of CNT is added to the cement slurry, the yield point and plastic viscosity increase by eight and five times respectively, while the free water and fluid loss of cement slurry are reduced by 85% and 70% respectively. In addition, cement stone compressive strength increases by 73.8%. Moreover, the elastic properties of the cement stone are improved and higher values for the Young's modulus and Poisson's ratio are achieved; however, there is an optimum concentration of nano-additive at which the maximum yield point, plastic viscosity, compressive strength, Young's modulus, and Poisson's ratio are reached. The results of this study can be used to optimize the cement slurry design in any given set of conditions.

Keywords: Carbon Nanotubes, Oil Well Cement, Thickening Time, Compressive Strength, Rheology, Young's Modulus, Poisson's Ratio

INTRODUCTION

Recently, many casing collapsing problems have occurred in Marun oilfield in 9-5/8" casing string which covers Gachsaran formation. This formation consists of gypsum-anhydrite rocks and exhibits plastic behavior [1], which leads to horizontal stress in the casing string. In Marun oilfield, Gachsaran formation with plastic properties transmits the overburden pressure to the casing and leads to casing collapse [2]. The conventional cement slurry used in cementing this string has very poor properties and cannot resist against the stresses. Improving the elastic properties of the cement sheath covering this string can help to solve the problem. Casing collapse resistance is improved by higher cement Poisson's ratio and Young's modulus [3].

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More elastic cement sheath can resist higher stresses and enhance the protection of casing [2]. In addition, deformable cements can distribute the stress evenly [4].

Nanotechnology is considered as a key technology for the future and governments have invested billions of dollars to employ it [5]. Use of nanoparticles could be a way to enhance the performance of the oil well cement. Oil well cement can be modified by adding nanosized materials like nanoparticles or nanotubes. Primary cementing in oilfield applications includes the placement of cement slurry between the drilled formations and the well casing. The main goals of well cementing are casing protection and zonal isolation [1]. Zonal isolation must be maintained throughout the lifetime of the well. Correct cement design and cement placement are the key factors in achieving the primary cementing goals. The required properties for the cement stone and slurry are determined by the well conditions. These properties include cement slurry weight, cement slurry rheology, cement slurry thickening time, cement stone compressive and tensile strength, cement stone elasticity, and cement stone durability. The set cement sheath should withstand the stresses induced by the well events and maintain integrity during the life of the well [6]. Mechanical forces from the adjacent formations may damage the cement sheath and casing string. It has been found that in order to prevent mechanical damage to the cement, the best type of cement in terms of mechanical durability is the one with higher elastic properties than the adjacent formation. The uniform distribution of the in situ stress acting on the outside of the cement sheath can be achieved by a softer and more ductile cement stone, which means higher values of Young's modulus and Poisson's ratio. This will also enhance the collapse resistance of the casing string [7]. The successful application of high-density elastic cements to solve HP/HT challenges in south Texas is reported in the literature [5]. The elastic properties of the cement stone can be improved by adding nanosized particles in low volumetric fractions to the cement formulation. Carbon nanotubes, tubes of carbon with diameters in the range of 1-100 nanometers, have different structures in terms of length, thickness, and number of layers and thereby different characteristics. Due to their distinctive features such as module of elasticity being in the order of terapascals (TPa) and tensile strength being in the range of gigapascals (GPa), wide distribution, and reduced fiber spacing CNT's are potential candidates for such applications as nanoreinforcements in cement-based materials in order to enhance their mechanical properties and resistance to crack propagation [8].

MATERIALS AND METHODS

The materials used in this study are API well cement class E, tap water, Hidense, Micromax and salt as weighting agents, boric acid as retarder, fluid loss controller, cement friction reducer, antifoam, and multi-walled carbon nanotubes.

This study is based on the laboratory testing of the improvements in the cement slurry and stone by using CNT's at three concentrations. The MWNT's were synthesized by the catalytic chemical vapor deposition of natural gas over Co-Mo/MgO nanocatalyst. This nanocatalyst powder was prepared by a special in-house sol-gel method. This catalyst was exposed to the reactant carbon source gas under appropriate conditions of flow rate, pressure, and temperature to produce MWNT's. In the present work, natural gas was used as the gaseous carbon source and it was diluted with carrier gases at atmospheric pressure and ambient temperature. Transmissions electron microscopy (TEM) images of CNT's were obtained using a Philips model TM 200 FGE electron microscope. Raman spectroscopy was used to measure the purity of carbon nanotubes. CNT's diameter was between 10-20 nanometers and CNT's length was 10 micrometer. The purity of the CNT's was 95%. Figure 1 shows the TEM images and Raman spectrum of the synthesized CNT's.

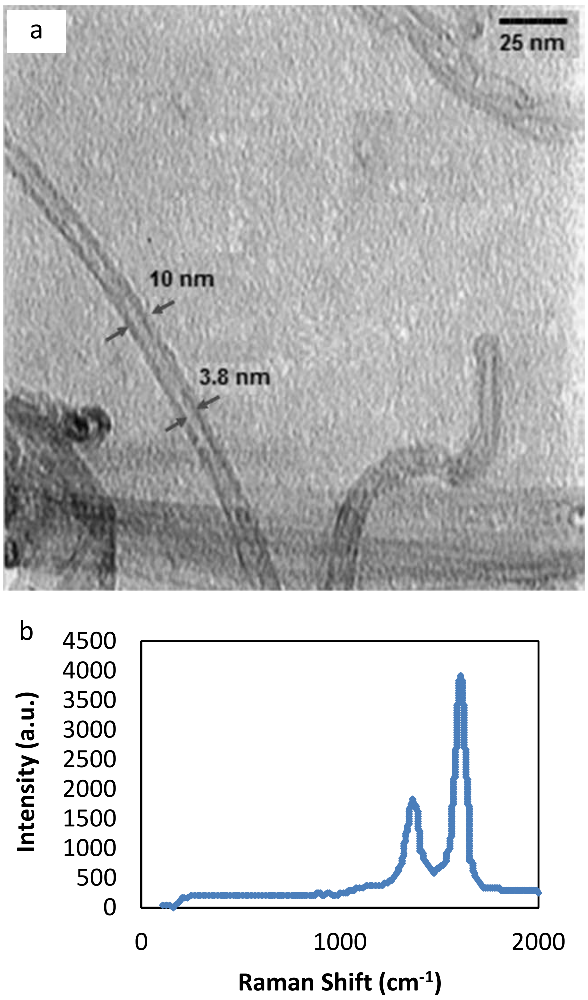

Figure 1: a) TEM image and b) Raman spectrum of the synthesized MWNT

Well#147 is selected as a candidate well in Marun oilfield in the south west of Iran. The conventional cement used in this well as well as the modified cement slurries are mixed in the laboratory and their properties including weight, rheology, free water, fluid loss, and thickening time are measured according to API RP 10B-2. The compressive strength of cement stones is measured by UCA. The elastic properties are measured on cylindrical samples by a uniaxial compressive strength tester equipped with axial and radial strain gauges (Figure 2).

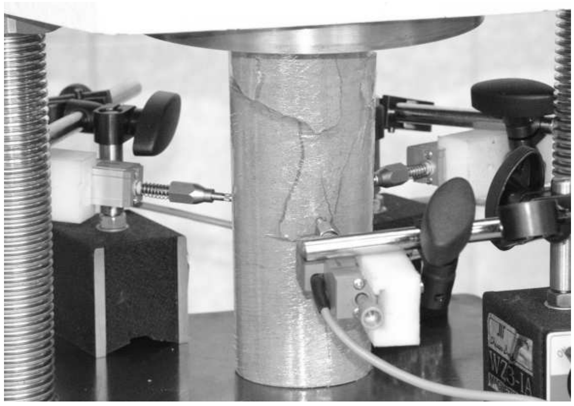

Figure 2: Uniaxial compressive strength tester equipped with axial and radial strain gauges

The elastic properties of the cement stone is determined based on the ISRM and ASTM rock mechanic standards. The cylindrical cement stone samples are prepared by curing the cement slurry for 48 hours in at 210 °F and 3000 psi in a rubber mold with a diameter of 67 mm and a length of 200 mm in a curing chamber. After the ends of the sample are cut flat, they are cooled down and kept saturated by merging in the tap water. The loading rate is 4000 psi/min which is the maximum loading rate of the available equipment. The compositions of the cement slurry samples are presented in Table 1.

RESULTS AND DISCUSSION

Table 1 shows the conventional cement recipe in Well#147 and 3 other recipes modified by different amounts of CNT's. The CNT's are added to the cement slurries at three different concentrations (i.e. 1, 2, 3% by weight of cement (BWOC)) in such a way that the slurry weight remains constant for better comparison. Cement friction reducer is added to the slurry as needed. Table 2 shows the measured properties of the cement slurries and stones according to API RP 10B-2. The elastic properties measured for 4 samples are also presented in Table 3.

Table 1: Cement slurry recipes

Component	Amount			
	Slurry Number 1	Slurry Number 2	Slurry Number 3	Slurry Number 4
API well cement class E	110 lb	110 lb	110 lb	110 lb
Carbon nanotube	-	1.1 lb/sx	2.2 lb/sx	3.3 lb/sx
Salt	20.5 lb/sx	35.2 lb/sx	11.88 lb/sx	11.88 lb/sx
Hidense	90 lb/sx	65.2 lb/sx	35.2 lb/sx	35.2 lb/sx
Boric acid	0.3 lb/sx	0.77 lb/sx	0.77 lb/sx	0.77 lb/sx
Micromax	-	36.74 lb/sx	36.74 lb/sx	36.74
Fluid loss controler	-	1.1 lb/sx	1.1 lb/sx	1.1 lb/sx
Cement friction reducer	-	1.98 lb/sx	1.98 lb/sx	1.98 lb/sx
Water	0.9581 ft^3	5.67 gal/sx	5.67 gal/sx	5.67 gal/sx
Antifoam	As Needed	As Needed	As Needed	As Needed

lb/sx: pounds per sack; gal/sx: gallon per sack

Table 2: Properties of the cement slurries

Sample	Slurry weight (PCF)	Down hole static temperature (°F)	Pressure (psi)	Free water (cc)	Fluid loss (cc)	Thickening time (min)	Compressive strength (psi)	Rheological Properties							
								(θ_{600})	(θ_{300})	(θ_{200})	(θ_{100})	(θ_6)	(θ_3)	Plastic Viscosity (cp)	Yield Point (lb/100ft^2)
1	146	210	10700	20	100	326	2100	129	54	35	20	4	4	51	3
2	146	210	10700	3	30	245	3650	>300	273	190	107	15	13	249	24
3	146	210	10700	2	20	220	2770	>300	266	181	100	19	15	249	17
4	146	210	10700	0.5	15	200	2150	>300	276	195	107	17	13	253.5	22.5

Rheological Properties

According to the measured rheological properties of four slurry samples, the slurries tend to thicken with increasing the CNT concentration (Table 1). Sample number 1 has the settling problem of the Hidense and the other samples need the cement friction reducer. Thicker slurry has more suspending capability and leads to a more homogenized cement sheath and constant cement stone properties along the casing string. The settling of the Hidense on the real well will result in a cement sheath with non-uniform properties along the well. At the bottom, the high concentration of the Hidense will lead to heavy cement with low compressive strength and at the top there will be light, thin, and two-phase slurry.

Table 3: Elastic properties of cement stones

Marun-147 modified cement elastic properties		
Sample	Properties	
	Young's Modulus (GPa)	Poisson's Ratio
1	2.43	0.03
2	2.76	0.06
3	2.48	0.16
4	3.60	0.20

CNT's increase the viscosity of cement slurry (Table 2). The increase is five times as high as the viscosity of the base sample, where it reaches from 51 cp for the base sample to 249 cp for the sample with 1 wt.% of nano-additive. The high surface area of the CNT nanoparticles added to the cement slurry, when the water content is kept constant, will lead to more water adsorption by the solid content, and thereby less free water [9]. CNT's fill the voids between cement grains decreasing the volume of pores between them, which in turn results in less free water. Therefore, there is a higher internal friction between solid particles and higher viscosity [10-11]. In addition, well-dispersed nanoparticles increase the viscosity of the liquid phase, which helps to suspend the solid particles [9]. Nanoparticles resist sedimentation, compared with larger particles, due to Brownian motion and interparticle forces. For the above reasons, it can be said that nanoparticles increase the stability of the cement slurry system.

Free Water and Fluid Loss

High free water of the cement slurry causes problems in zonal isolation especially in horizontal wells and creates heterogenic cement stone properties along the cement sheath. High fluid loss means a thicker cake and weaker bonding between cement sheath and the formation. On the other hand, loss of the water content of the cement slurry due to water absorption into the formation causes the cement slurry dehydration and increases the formation damage. It will also reduce the possibility of annular bridging by dehydrated cement [12]. Fluid loss control is also important in controlling the water-sensitive formation and gas migration problem. Free water and fluid loss of cement slurries are determined according to the recommended method of API-RP-10B (Figures 3 and 4).

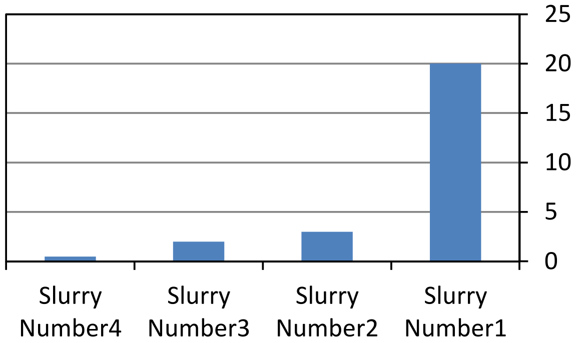

Figure 3: Free water of the slurries

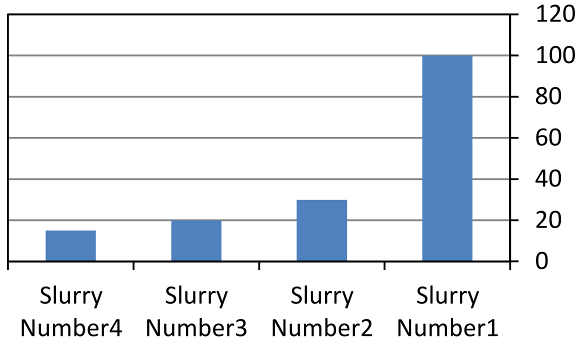

Figure 4: Fluid loss of the slurries

Free water and fluid loss decrease by increasing the CNT content (Table 2). The free water content is reduced from 20 cc in the base sample to 3 cc in the sample with 1 wt.% of nanotubes. Adding higher amounts of CNT's results in even lower values for free water content. It reaches 2 and 0.5 cc when the CNT concentration becomes 2 and 3 wt.% respectively. It could also be deduced from Table 2 that the effectiveness of the nano-additive in decreasing the free water and fluid loss decreases with increasing the concentration.

Similar to the reasoning mentioned above, this phenomenon could be explained as follows. The very high specific surface area of nanoparticles needs more water to be covered and thereby decreasing the amount of free water in the slurry [13]. Nanoparticles fill the voids between the cement grains, which results in the immobilization of free water (filler effect). CNT's act as nanofillers and improve the resistance to water permeability of cement slurry cake on the wellbore wall. Well-dispersed nanoparticles increase the viscosity of the liquid phase of the slurry leading to a less fluid loss [9]. In addition, the rapid formation of hydrated products consumes the water content of the slurry [13]. Similar results are also found in other studies [9-10, 13-14].

Thickening Time

The presence of the CNT's in the cement slurry decreases the thickening time as shown in Figure 5.

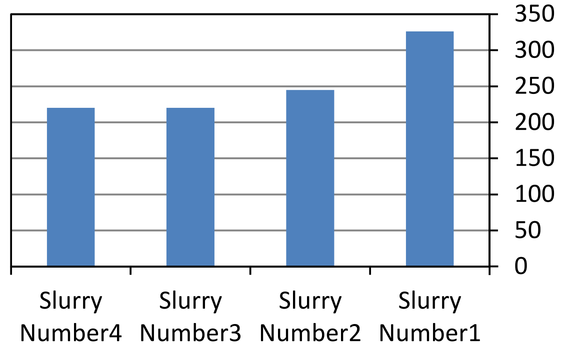

Figure 5: Thickening time of the slurries

It can be seen from Figure 5 and Table 2 that although the thickening time is 326 minutes for the sample without CNT, it decreases to 245, 220, and 200 minutes for the samples with 1, 2, and 3 wt.% of nano-additive respectively. Many similar results have also been reported elsewhere [8-9, 13-19]. Well-dispersed nanoparticles act as centers for the crystallization of cement hydrates, which accelerates the hydration [9]. Furthermore, the nanoparticles dispersed uniformly in a cement paste will accelerate cement hydration due to their higher activity [20]. The rate of hydration is affected by the dosage and size of the nanosized particles [21]. That is, an increase in the rate of reaction is proportional to the concentration of the CNT's. Moreover, smaller particles were found to accelerate the reaction more than larger particles. A decrease in the thickening time with increasing the content of nanoparticles is also reported in some other works [13, 15-18].

Compressive Strengths and Elastic Properties

The compressive strengths of 4 cement samples are measured by ultrasonic cement analyzer (UCA). Figure 6 shows the 24-hour compressive strength of 4 samples.

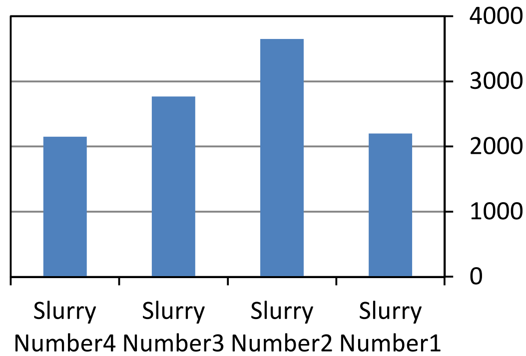

Figure 6: Compressive strength of the slurries

It can be seen from this figure that the compressive strengths of the samples with CNT's are higher than the one without CNT's. The increase in the quantity of this property is 73.8% when only 1 wt.% of CNT is added to the cement slurry. A higher compressive strength means a higher load-bearing capacity of the cement sheath and higher collapse resistance of the casing string [22]. The compressive strength is primarily controlled by the total porosity [20]. The nanoparticles will fill pores, which leads to increasing strength and improving the microstructure of the cement if the nanoparticles are uniformly dispersed [23-24]. In addition, nanoparticles behave as a promoter of pozzolanic reaction [23]. The increase is due to the formation of more hydrated products in the presence of nanoparticles [13]. Crack arrest and interlocking effects between the slip planes provided by CNT's improve the toughness, shear, tensile strength, and flexural strength of cement-based materials [9]. According to Figure 6, slurry sample number 2 with 1.1 lb/sx CNT's exhibits the maximum increase in compressive strength compared with the base slurry. Accordingly, by increasing the concentration of nanoparticles to 2.2 and 3.3 lb/sx in slurry samples number 3 and 4, the samples show a less significant increase in compressive strength being 32% and 2.5% respectively. Therefore, the effectiveness of CNT's in increasing the compressive strength of the samples decreases by increasing the volume fraction. In other words, an increase in the additive concentration causes reduced compressive strength, which is because of the unsuitable dispersion of nanoparticles and their aggregation in the concrete matrix [24]. This means that there is an optimum level of use of CNT's [15]. Similar results are also reported for CNT nanoparticles in the literature [25-28]. Furthermore, the increase in the compressive strength of the cement stones by nano-additives is reported in many studies [8-9, 13, 15-17, 24].

The elastic properties of the cement stone for the modified cement compositions with 3 concentrations of CNT's are also measured in this study (Table 3). Figures 7 and 8 show the axial and radial stress-strain curve of two cement stone samples.

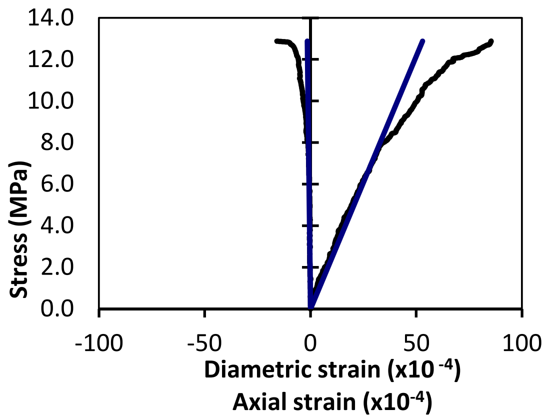

Figure 7: Axial and radial stress strain curve for slurry sample number 1

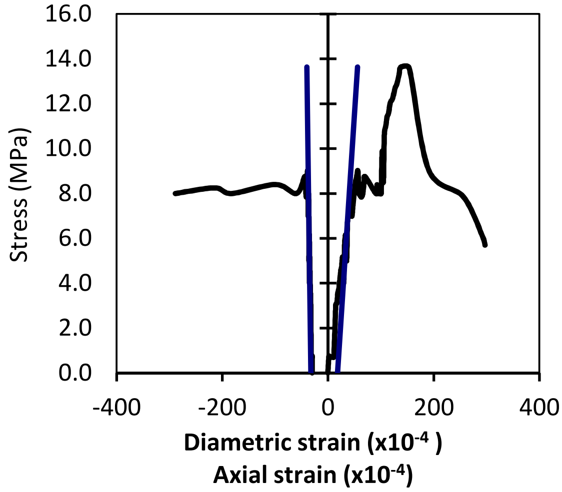

Figure 8: Axial and radial stress strain curve for slurry sample number 4

The Young's modulus and Poisson's ratio are determined from the curve. Similar to the previously mentioned properties, an increase in the Poisson's ratio and the Young's modulus can be observed (Figures 9 and 10).

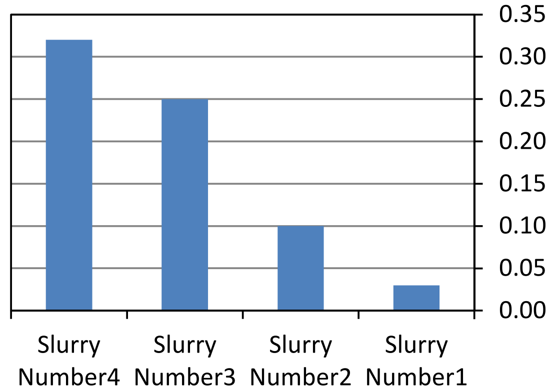

Figure 9: Poisson's ratio of the samples

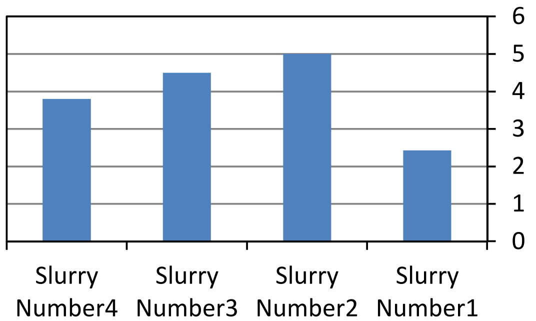

Figure 10: Young's modulus of the samples

The maximum value for these properties obtained for sample number 4 are 0.2 for Poisson's ratio and 3.6 for Young's modulus, whereas the measurements for the base sample show lower values of 0.03 and 2.43 for Poisson's ratio and Young's modulus respectively. Elastic cement systems have shown to improve the collapse resistance of the casings. A cement stone matrix consists of two elastic and rigid phases. Under the exposed tensions, the rigid phase resists and the elastic phase deforms. By increasing the elastic phase, the whole system tends to increase the overall elasticity. This results in the elastic phase playing the major role. The improvement in elastic properties is also due to the formation of more hydrated products in the presence of nanoparticles [13]. Casing collapse resistance is very sensitive to cement Poisson's ratio and cement Young's modulus [3]. A higher Young's modulus means that cement stone has temporary and reversible deformations under a certain stress and withstands higher stresses without breaking. A higher Poisson's ratio can distribute stresses and prevent the creation of high-stress points. The forces will be distributed along the cement sheath and the whole cement sheath will show an elastic behavior avoiding breakage by elastic deformation. The effectiveness of CNT in increasing the Young's modulus of the samples decreases by increasing its volume fraction. In other studies, it has also been shown that the Young's modulus of cement mortars is enhanced with adding CNT's [29].

CONCLUSIONS

This study confirms the results obtained in the previous studies and provides new experimental results for improving the elastic properties of cement stone by adding nanoparticles. Moreover, it has been demonstrated herein that:

1. CNT's improve the properties of the cement slurry and stone;
2. Adding CNT's to cement slurry increases the viscosity, thereby improving the suspending ability of the slurry;
3. Free water and fluid loss are also decreased by increasing the concentration of CNT's. This reduction could be as high as 85% for free water and 70% for fluid loss at 1 wt.% of CNTs;
4. The effectiveness of the nano-additive in decreasing the free water and fluid loss is higher at lower concentrations;
5. The thickening time decreases by increaseing the concentration of CNT's. While it is 326 minutes for the sample without CNT, it decreases to 245, 220, and 200 minutes for the samples with 1, 2, and 3 wt.% of nano-additive respectively;
6. There is an increase in compressive strength, Young's modulus, and Poisson's ratio of the cement stone samples;
7. The increase in the additive concentration causes reduced compressive strength and Young's modulus because of unsuitable dispersion of nanoparticles in the cement stone matrix; thus an optimum level of CNT's should be used.

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