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Influence of Petroleum Coke on The Rheological and Mechanical Properties of Cement Grouts for Oil Wells

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Abstract

Petroleum coke is a by-product from the coking process of high molecular weight hydrocarbons, its accumulation in Venezuela has been due to the increasing exploitation of heavy and extra-heavy crudes from the Orinoco Oil Belt, which is why it is urgent to propose other uses for it. The purpose of this research was to demonstrate the influence of petroleum coke on the rheological and mechanical properties (compressive strength and density) of cement slurries for oil wells; For this, the laboratory tests established in American Petroleum Institute (API) standards 10 A and 10 B were implemented: rheology, thickening time, free fluid, filtering and resistance to compression required to determine the effect of different concentrations of the material on the properties of the grouts. Similarly, three (3) low-density systems were formulated to investigate whether coke works efficiently as an extender additive since it has a specific gravity lower than that of cement. Through the statistical analysis of the results obtained, it was determined that coke works as an agent capable of producing exponential increases in rheological properties and resistance to compression. Also, it was verified that the solid allows reducing the density of the systems having as an advantage that it improves the compressive stress. The incorporation of the material for the aforementioned purposes in the cementing of wells would mean a solution to the growing accumulations of it and would contribute to the economy of these operations as well as representing an innovation in research.

Keywords*: Petroleum coke; Cementing slurry; Additive; Rheology; Compressive strength; Density.*

1. Introduction

Cementing an oil well is a primary operation of the drilling phase in the oil industry and its success is a decisive factor in the continuation of the following actions [1]. In cementing activity, the space (ring) between the exterior of the casing and the adjacent rock formation is filled with a pumpable mixture of pure cement, water and various additives known as cementing grout, which is left to set for hours or days generally, while strongly solidifying to bond the liner to the formation [2-4] and producing a stronger, denser and / or less porous medium between [5] the pit walls and the liner.

In the oil industry, to obtain effective zonal isolation, the design of the cement grout must meet a series of short-term criteria, such as free water, thickening time, loss of filtering, development of resistance and contraction, in addition to various long-term requirements, including resistance to chemical attack, thermal stability and mechanical integrity of the cement structure [6]. Cementitious materials must have desirable rheological properties (i.e., flowability, stability, and initial viscosity), which directly determine both workability, pumpability, self-leveling and compaction, and the ability to penetrate material holes in the engineering practice [7-8].

The rheological properties are essential for cement grouts, they establish the quality of the final product, help to predict its performance and physical properties during and after the process. When performing the oil and gas well cementing operation, the rheological properties of the cement slurry are not only the main factor affecting the efficiency of ring displacement and the quality of cementing, but are also important in ensuring the cementing safety [9].

Another important mechanical property is compressive strength, which is used to test the reliability of cementing and is defined as the ability of a material to resist deformation when a load is applied [10]. Forged cement with good compressive strength should be able to withstand hard and corrosive formations, loss of circulation zone, carbon (IV) oxide, other toxic gas intrusions, and extremely high temperatures [11].

The presence of additives with different chemical compositions for different purposes mixed in the cement influence the rheological behavior and the control of the main properties of the cement slurries, such as thickening time, consistency, fluid loss, free water, setting time, the development of cement resistance, density and the possibility of presenting special requirements (control of gas migration, thixotropy, expansion, strong links with protection and formation pipes), among others [12].

However, the addition of materials did not occur with the start of well cementing, the first cemented well dates from 1903 in the Lompoc field in California; By the 1940s, there were only two types of cement and three additives in the oil industry, and 25 years later, there were 8 types of API cement and 38 additives. Currently there are 8 types of API cement and more than 100 types of solid or liquid additives available by service companies [13], all of them duly studied and corroborated in their functions for which they were formulated.

For this reason, there is sufficient literature on the addition of other materials to cement slurries from oil and / or gas wells, which have revealed the effect they have on their rheological and mechanical properties, such as: gypsum and anhydrite [14], silica [15], metakaolin [16], spent catalyst FCC [17], polymers [5,18-19], bentonite [20], clay [21], zeolite [22], nanoclay [23], nano- SiO_2 [24-25], nano- TiO_2 [25], activated carbon [7] and halloysite nanotube [26].

The search for alternative materials for cementing oil wells has gained momentum worldwide. Any new material must allow cement that is stronger, more durable, flexible and resistant to the stresses exerted on it [27]. In Venezuela coke is considered a waste, so it is not used in any way and accumulates in large quantities on the surface; that is why, the present work tried to know the influence of petroleum coke in different concentrations on the rheological and mechanical properties (compression stress and density) of type B cement slurries for oil wells, under the criteria established by the API standards.

2. Experimental section

The formulation of a type B cement grout (control grout) was started according to 10A API Standard [28] (Table 1). The control was modified by adding delayed petroleum coke from the PetroPiar Improvement Complex of Petróleos de Venezuela S.A (PDVSA), it was pulverized in a hammer mill and six (6) new grouts were formulated (Table 2). The laboratory tests were carried out at the Cementation Laboratory of PDVSA located in Maturín-Venezuela.

Table 1. Slurry requirements for quality tests with type A and B cements

Component	Class A and B
Mixing water	355 ± 0.5 g
Cement	772 ± 0.5 g

Table 2. Obtained slurry formulations with its respective coke concentration (*BWOC: By Weight of Cement)*

Cement grouts		Petroleum coke		Cement		Water	
		Percentage (%BWOC)	Mass (g)	Percentage (%BWOC)	Mass (g)	Percentage (%BWOC)	Mass (g)
Control		0	0	100	772	46.24	355
Petroleum coke	A	0,5	3.84	100	767.41	45.92	352.41
	B	1	7.66	100	766.47	45.60	349.51
	C	2	15.29	100	764.61	44.96	343.75
	D	3	22.88	100	762.75	44.32	338.02
	E	4	30.44	100	760.90	43.67	332.31
	F	5	37,95	100	759.06	43.03	326.63

2.1. Grout quality

The experimental protocol followed for the quality study of the different cement slurries (control and with the addition of petroleum coke) was framed in 10A API specifications [28]. They were studied and verified compliance with the parameters: density, Water/Cement (W/C) ratio, pumping time (consistency at 30 min and thickening time), free water, and compression stress at 8 hours. The premise of the study was to maintain the grout density at 1.9 g/cm^3 (15.6 ppg: pounds per gallon).

2.2. Study of rheological properties and compressive strength

The rheological properties of plastic viscosity, yield stress, gels were determined by applying the procedures established in 10B API Standard [29], as well the rheological model to which the grouts were adjusted was established; also the compressive strength was determined.

Statistical analysis ANOVA (Analysis of Variance) was applied with a significance level (α) of 0,05 to determine if there were statistical significant differences between the properties under study; a regression analysis was performed for the properties of plastic viscosity, yield stress and compressive strength, using the statistical software Infostat ®.

2.3. Specification of the use of petroleum coke as a spreader additive

Based on the knowledge of the lower specific gravity of petroleum coke (1.38) with respect to the specific gravity of cement (3.14), its possible use as a spreading agent (density reducer) was studied. Based on the equivalent sack concept, that is, the absolute solids volume (cement) is 94 pounds (42.64 kg) corresponding to the mass of a cement sack and keeping the water volume constant, a mass balance was performed based on the properties shown in Table 3, then the cement percentage of a base grout with a density of 1.9g/cm^3 (15.6 ppg) (Table 4) was reduced by adding petroleum coke to obtain grouts with densities of 1.7 g/cm^3 (14.5 ppg), 1.6 g/cm^3 (13.5 ppg) and 1,5 g/cm^3 (12.5 ppg); After obtaining the compressive strength, these values were compared, because normally if low-density grouts are required in the field, the compressive strength is affected. The experimental procedures applied are established in 10B API Standards [29].

Table 3. Data for mass balance

Data	Value	Unit
Solid (cement + Petroleum coke) 1 bag (sxs)	42.64	kg
Specific gravity of cement	3.14	Dimensionless
Petroleum coke specific gravity	1.38	Dimensionless
Cement density	3.13	g/cm^3
Petroleum coke density	1.20	g/cm^3
Water density	1.00	g/cm^3
Water volume	0.01973	m^3
Water mass	19.72	kg

Table 4. Starting data for obtaining the grouts of 1.77g/cm^3, 1.64g/cm^3 and 1.52g/cm^3

Cement %	Cement mass (kg/sxs)	Petroleum coke %	Petroleum coke mass (lb/sxs)	Solid mass (kg/sxs)	Required density (g/cm^3)
1.00	42.6	0.00	0.00	42.6	1.9

3. Results and discussion

3.1. Grout quality

The results of the properties under study (Table 5), show that it was possible to obtain the desired density of 1.9 g/cm^3 in all the grouts, but to the detriment of the W / C ratio, which was maintained in the first three grouts (control, A and B) and from them a decrease was shown to 0.45 in grout C to 0.43 in grout F, representing a range of reduction from 2.17% to

6.5% in property, this as a consequence of the increase of the solids with respect to the water present in the formulation. The W/C ratio plays an important role in influencing the grout to penetrate the ground or cracks in the rocks. Increasing the W / C ratio improves the fluidity of fresh grouts, however, the high W/C ratio negatively affects the mechanical properties of the grouts: the higher the W/C ratio, the greater the reduction in development of resistance [30].

Table 5. Performance test results and API 10A requirements

Cement grouts	Density (g/cm^3)	W/C ratio	Pumpability time		Free water (%)	Compressive strength (MPa) 8 h
			Consistency at 30 min	Thickening time (min/70Bc)		
Control	1.9	0.46	18	156	1	3.7
A	1.9	0.46	20	130	1	3.8
B	1.9	0.46	19	133	1	3.9
C	1.9	0.45	23	128	2	4.0
D	1.9	0.44	25	118	2	4.2
E	1.9	0.44	24	112	2	5.0
F	1.9	0.43	22	102	2	5.9
API requirements	1.9	0.46	<30	>90 min/100Bc	< 5.9	>1.4 MPa

Bc: (Bearden unit of consistency) according to 10A API specifications [28] it's the measure of the consistency of a cement slurry when determined on a pressurized consistometer.

Despite its decrease, the obtained W/C ratio values are not outside the requirements established in the 10A API Standards for the different types of cements: 0.46 (Cement A and B), 0.56 (Cement C), 0.38 (Cements D, E, F and H) and 0.44 (Cement G).

In previous works, reductions between 24% - 66.3% in the W/C ratio have been found by increasing the concentration of polymers (added material) keeping the density constant [5]. Or W/C ratio variations of 0, 38; 0, 44 and 0, 48 due to changes in the proportion of C-S-H-PCE nano foil [31]. Opposite results were found by increasing the proportions of activated carbon and maintaining a constant W / C ratio (0.5), reaching a variation in the density of 2.17 to 2.21 kg/m^3 in the slurry [7].

The W / C ratio decrease of the grouts did not affect the behavior of the other properties under study. Thus, the pumpability time of all grouts showed values within the norm; the consistency reflected at 30 min were in the range of 18-25 Bc (<30Bc) and the thickening time decreased from 156 Bc to 102 Bc with the addition of petroleum coke, values always greater than the minimum 90 Bc. The free water present was 1% or 2%, being below the minimum 5.9 %, so the tendency of the water to separate from the slurries before setting is low and acceptable. Finally, the compression stress at 8 h presented values from 3.7 MPa (control) to 5.9 MPa (grouts F), higher than the stipulated minimum (1.4 MPa). The high values of compressive stress may perhaps be due to the addition of petroleum coke to the chemical composition of the cement used in the study (presence of tricalcium aluminate (C3A) or the good hydration of the cement particles due to its Blaine fineness; both aspects not covered in the present study.

3.2. Effect on the physical properties of the grouts and on the compressive strength

Based on the obtained results, concerning the rheological properties and compressive strength (Table 6), an increase in the proportion of petroleum coke in the grouts was accompanied with an increase in them.

By applying ANOVA (Table 7), it was statistically demonstrated that the addition of petroleum coke caused statistical significant differences in rheological properties and compressive stress between the control slurry and the slurries with different percentages of coke (p-value <0.05); the obtained Pearson's correlation coefficients (R^2) showed that the data fitted strongly to the model, and low variation coefficients (C.V) (<20) reflect the non-existence of a problem with the applied model; therefore, it can be expressed that the addition of petroleum coke in the cement grouts has an effect on the properties under study, becoming a rheological modifier that also increases the compressive stress.

The rheological properties of cement grouts in the fresh state are extremely important in completing the oil wells cementing process, especially when another material is added to the composition of the cement grout [26].

Table 6.I Average values of rheological properties and compressive strength

Cement grouts	Plastic viscosity (mPa.s)	Yield Stress(Pa)	Gels (Pa)		Compressive strength (MPa)(24 h)
			10s	10min	
Control	88.2	17.95	7.66	10.05	21.80
A	91.7	18.19	6.70	8.14	22.25
B	116.5	21.26	6.70	8.14	23.33
C	118.5	20.06	7.66	11.97	23.57
D	127.9	23.89	9.10	13.89	24.65
E	158.0	22.17	9.10	13.41	28.88
F	141.4	25.71	10.05	13.41	34.49

Table 7. Analysis of variance (ANOVA)

Property		Analysis of variance				
		N	R^2	R^2 Aj	C.V	p-valor
Plastic viscosity		21	0.99	0.98	1.02	<0.0001
Yield Stress		21	0.98	0.97	2.33	<0.0001
Gels	10 s	21	0.81	0.79	9.37	0.0002
	10 min	21	0.93	0.91	7.04	<0.0001
Compressive strength		21	0.95	0.93	4.8	<0.0001

3.3. Plastic viscosity and yield stress

The growth of plastic viscosity was in the range of 3.9% to 60.3% and the yield point in the range of 1.3% to 43.2%, for 0.5% and 5% of added coke respectively. The increase in properties was mainly due to the fact that by adding more solid particles the interaction between them increase, besides there could be an increase in the surface area of petroleum coke, also the size and irregularity of its shape that tend to form a net structure inside the cement suspension. All of this led to greater opposition of fluids to tangential deformations and to obtaining thicker grouts with a low tendency to settle.

Some authors consider the increase in plastic viscosity and the yield point as a negative effect due to the increase in the content of the added material [30-32] . However, as long as the plastic viscosity is not so high as to impede the pumpability of the slurry, a higher viscosity can help cement to displace the fluid more efficiently, an important factor in successful cementing of oil wells [32].

However, it is important to note that in cementing work, it must be guaranteed that the plastic viscosity and yield point of each phase must be greater than that of the previous phase, that is, that the hierarchy of the fluids pumped into the well must be respected in order to ensure efficient removal of used drilling fluids and also avoid channeling, which is the phenomenon that occurs when a lower density fluid moves through another of higher density. This behavior was observed when adding petroleum coke in higher percentages (Fig. 1 and Fig. 2). An increase in the percentage of petroleum coke led to the plastic viscosity adjusting to the equation $y = 81.43e^{0.092x}$ and the yield stress to the equation $y = 16.85e^{0.056x}$, therefore petroleum coke is considered as a rheological modifying agent.

These results are in agreement with the conclusions of several investigators confirming increases in plastic viscosity and elastic limit when adding additional material to the grout. In this sense, increases between 35 mPa.s and 162 mPa.s in plastic viscosity have been evidenced and the yield stress increased between 34.81 and 63.68 Pa and with the addition of two different polymers at a temperature of 25ºC [5]. Likewise, the graphic behavior reflected by them in both properties was linear.

Likewise, the addition of activated carbon, conduced to an increase from 20.42 to 33.62 mPa.s and from 64.63 Pa to 108 Pa for the plastic viscosity and yield point respectively, presenting a non-linear behavior [7], with the addition of nano Silica the pour point ranged between 4.5 and 5.7lb/100pie^{2}and the plastic viscosity between 20.9 and 37.9 mPa.s [30] and when adding halloysite there was an increase from 54.38 to 65.23 mPa.s (plastic viscosity) and 0.53 to 0.67 Pa (yield stress).

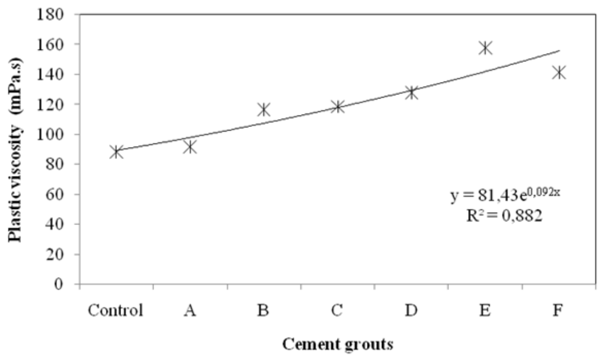

Figure 1. Exponential behavior of the plastic viscosity property

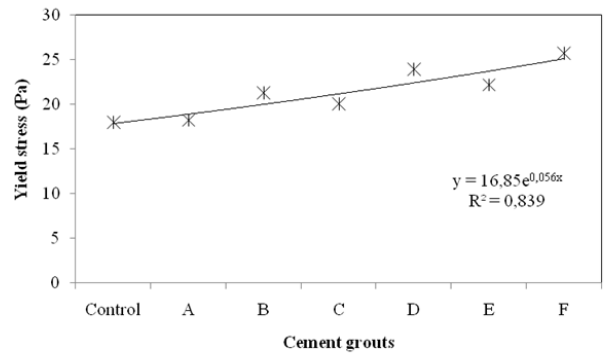

Figure 2. Exponential behavior of the yield stress property

3.4. Gels

Cement grouts form gels due to the hydration reaction of hydrated calcium silicate (C-S-H). According to the results, the gels of the control cement grout were higher than those presented by the coke grouts A and B, but lower than those shown by the grouts C, D, E and F. This behavior was due to the fact that the cement of the slurry formulated with the addition of 1% or lower of petroleum coke did not generate enough Portlandite [$Ca(OH)_2$] to form gels, so it may take longer for this to occur. While higher than 1% additions of coke accelerated it. The results obtained are consistent with those shown when adding to the slurry of cement spent catalysts FCC [17], zeolite [33] and nano clay [32], these reflected improvement in gel values at 10s and 10 min with higher concentrations of the added material.

3.5. Rheological model

10B API Standards [29] establish that the most widely used rheological models to predict the rheological properties of cement slurries are the Bingham plastic model and the Power Law model. The results showed that the Power Law model was the rheological model that best described the behavior of the slurries (Table 8), being an indication that the cutting effort is not proportional to the cutting rate.

Table 8. Rheological model

Cement grouts	Bingham Plastic (B.P)			Power Law (P.L)			Model
	Plastic viscosity (mPa.s)	Yield stress (Pa)	Correlation coefficient	N	K (Pa)	Correlation coefficient	
Control	88.2	17.95	0.8003	0.428	0.092	0.9805	P.L
A	116.5	18.19	0.7594	0.495	0.081	0.9612	P.L
B	91.7	21.26	0.8049	0.431	0.092	0.9821	P.L
C	118.5	20.06	0.7949	0.501	0.077	0.9758	P.L
D	127.9	23.89	0.8015	0.455	0.110	0.9787	P.L
E	158.0	22.17	0.8140	0,548	0.073	0.9700	P.L
F	141.4	25.71	0.8329	0,440	0.130	0.9799	P.L

Cement grouts normally exhibit pseudo-plastic behavior such as those included in the Power Law, which flow immediately when a pressure gradient is applied. However, unlike Newtonian fluids, the relationship between shear stress and cutting speed is not linear. Previous works showed that by adding other materials such as halloysite [26], nano-SiO2 [24] and nano silica [30], their grouts were adjusted to the Bingham plastic model.

3.6. Compressive strength

Concerning the compression resistance, this must be greater than 10,3 MPa at 24 hours [28], it was evident that all the grouts had a higher value than that (Table 6) and the addition of petroleum coke increased it in a range 2% (slurry A) to 58.2% (slurry F) with respect to the control slurry. The increase in this property was due to the dispersion of the cement particles and the improvement of the stress bond between the particles leading to a decrease in the porosity and the ratio of empty spaces of the cement-based grout, which indicates that the cement has a great capacity to resist tension.

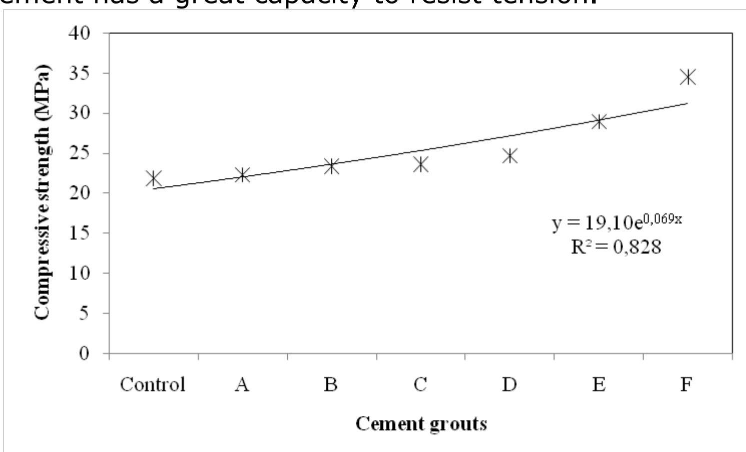

Figure 3. Exponential behavior of the compressive strength property

The increase in compressive stress caused by the addition of petroleum coke (Fig. 3), described a behavior given by the equation $y = 19.10\ e^{0.069x}$. This effect is considered favorable because the higher the resistance, the greater the pressure required to destroy the cement once it has solidified, and it also benefits the stability of the well.

Likewise, increases in compressive strength from 90 to 544% have been reflected when adding polymers [5] and nano clay(0.34 MPa to 3.45 MPa) [32], from: 19.3% - 40.5% with nano silica [30], from 5% with activated carbon [7], reflecting a non-linear behavior in the four studies, halloysite increased this property up to 36.2% with a linear behavior up to the first three percentages of material addition [26].

3.7. Extending agent

The compressive strength of light cements varies with density and temperature [34]. Table 9 shows the material balance obtained for the required densities, it is detailed that there was a coke addition of 11%, 23% and 37% to obtain densities of 1.7g/cm^3(14.5 ppg), 1.6 g/cm^3 (13.5 ppg) and 1.5g/cm^3 (12.5 ppg) respectively.

Table 9. Material balance

% Cement	Mass cement (kg/sxs)	% Petroleum coke	Mass petroleum coke (lb/sxs)	Mass Solid (kg/sxs)	Density Required (g/cm^3)
100	42.60	0.00	0.00	42.60	9.1
89	37.95	11	4.65	42.60	1.74
83	35.39	17	7.21	42.60	1.68
77	32.83	23	9.77	42.60	1.62
70	29.85	30	12.75	42.60	1.56
63	26.86	37	15.74	42.60	1.50

Table 10 shows that the addition of petroleum coke improved the compressive strength of cement grouts, 7.35 MPa, 13.91 MPa and 27.74 MPa values were obtained for the grout with petroleum coke densities 1.5; 1.62 and 1.74 g/cm3 respectively, compared to the base cement grout, which showed values of 6.16 MPa, 6.68 MPa and 23.05 MPa for the same densities; this reflects an improvement in the compressive strength of 16.2% (1.5g/cm^3), 52.0 %

(1.6 g/cm^3) and 20.4% (1.7g/cm^3) (Table 10). It is also observed that the compression resistance value (27.74MPa) of the petroleum coke grout for a density of 1.7g/cm^3 is greater than the reflected value for the base grout of 9.1g/cm^3 (24.24 MPa).

Table 10. Compressive strength of cement grouts without petroleum coke and with petroleum coke

Density (g/cm^3)	Compressive strength(MPa)		Difference (%)
	Cement slurries	Slurry with petroleum coke	
1.9	24.24	N.A	N.A
1.74	23.05	27.74	20.4
1.62	6.68	13.91	52.0
1.5	6.16	7.35	16.2

Reports of other light cement slurries with spreader materials stated compressive strength values using sodium metasilicate of 4.36 MPa (1.5g/cm^3), 9.15 MPa (1.6g/cm^3) and 13.07MPa (1.7g/cm^3) [35]; added Fly Ash was 3.83 MPa (1.5g/cm^3), 10.01MPa (1.6 g/cm^3) and 15.65 MPa (1.7 g/cm^3) [36] and with Dry-Blended 2.48 MPa (1.5g/cm^3), 4.69 MPa (1.6g/cm^3) and 7.58 MPa (1.7g/cm^3) [35]. Likewise, with ultra light grout glass spheres they managed to obtain a 7.6 MPa compression resistance for a density of 1.4 g/cm^3 [34] and between 8.0 and 11.0 MPa depending on the density of the glass spheres for a 1.5g/cm^3 cement grout [37].

It is observed that the resistance to compression values of the coke grouts can be considered as good regarding to the other extenders because they mainly reflected higher values than them.

The results obtained showed that petroleum coke has great potential to be added to the cement used in the cementing of oil wells, since it improves the compressive strength and the rheology of the cement can be adapted to meet the requirements of pumping. The tendency to increase and variations in the values of the properties under study are consistent with the previously cited studies that added another material to the cement grouts. However, it is important to emphasize that there was no control over the grain size of the petroleum coke or its shape as it is an exploratory study, but it would be important to know the effect of the said material at the nano particle level.

Likewise, it was observed the possible use of petroleum coke as a spreading material so a more in-depth analysis should be carried out on this topic, including an analysis of properties and cost. The study represents a possible alternative to the large amount of petroleum coke stored in the General of Division José Antonio Anzoátegui Petrochemical Complex due to the production of 20 ton/day of it [38], as a result of the improvement of the heavy and extra-heavy crudes of the Orinoco Oil Belt.

4. Conclusion

The addition of petroleum coke to the cement grout in different percentages (0.5%, 1%, 2%; 3%, 4% and 5%) did not affect its quality by meeting the minimum and / or maximums values stipulated in the 10A API standards, with only the W / C ratio being modified to maintain the density of 1.9g/cm^3. Petroleum coke can be considered as a rheological modifying agent and that increases the compressive stress of cement slurries, the change in properties is governed by an exponential type equation. Petroleum coke can be used as a density reducer for cement slurries (spreader) having as a great advantage that it improves the compressive strength.

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