

Petcoke composites as HPHT fluid-loss control additive for oil-based drilling fluids

Compósitos de petcoke como aditivos de control de filtrado APAT en fluidos de perforación base aceite

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Abstract

In the present work, composites based on petroleum coke (shot petcoke) and unmodified lignites as high pressure and high temperature fluid loss control additives (HPHT) in oil-based drilling fluids were synthesized and evaluated. Several petcoke composites were synthesized with coke content among 48 wt% and 85 wt%. Petroleum coke composites with lignites controlled the fluid-loss better than organophilic lignite. Petcoke composite with leonardite (a type of lignite) (FPC-L) was that showed the smaller fluid-loss (6.8 mL) in organophilic lignite (FOL) comparison (5.7 mL), because of colloidal lignite (fouling) helps plug off the permeable parts of filter-cake. Applying the reverse osmosis filtration models (Hermia's models); the blocking mechanisms that occurred most probably were found. FOL fluid-loss control mechanism is by filter-cake formation, while FPC-L is by filter-cake fouling. Petcoke composites controlling fluid-loss by three mechanisms colloidal fouling of the cake filtration: (i) intermediate blocking, (ii) standard blocking and (iii) complete blocking. Colloidal lignite is a determinant factor in the fouling of pore volume and permeability the filter-cake. Cake filtration permeability was estimated by $^1\text{H-NMR}$. Use lignite-petcoke composites as fluid-loss control additive of lower environment impact for oil-based drilling fluids.

Keyword: petcoke composites, leonardite, organophilic lignite, HPHT fluid-loss control additive, membrane fouling, reverse osmosis.

Resumen

En el presente trabajo de investigación, se realizó la síntesis y la evaluación de compósitos de coque de petróleo (petcoque) como agentes controladores de filtrado en los fluidos de perforación petrolera base aceite mineral. Se obtuvieron compósitos de petcoque desde 48% p/p hasta 85% p/p de coque de petróleo, encontrándose mejores desempeños como agentes controladores de filtrados, cuando el compósito contiene partículas de coque alrededor de 200 mesh, solvente alifático, surfactante catiónico y coloides húmicos. En base a la aplicación de los modelos de filtración propuestos por Hermia, el compósito de petcoque (FCC) mantiene en promedio un volumen de filtrado ($6,8 \pm 0,1$ mL) en el mismo orden de magnitud al generado por el lignito organofílico (FLO) ($5,7 \pm 0,1$ mL); pero a través de mecanismos de filtración distintos. El lignito organofílico manifestó una clara tendencia a la formación de torta de filtración o revoque, en cambio el compósito de petcoque, lo hace por medio de bloqueos estándar e intermedio de los poros de la torta de filtración o revoque. Los compósitos de petcoque-lignitos naturales importados (FCC/LI), muestran los menores flujos de filtrado (cm/s) en función del tiempo, en comparación con los flujos dados por el lignito organofílico de referencia y el compósito de petcoque sin lignito. La contribución de las partículas coloidales húmicas, es un factor importante en la colmatación de los poros de la torta de filtración o revoque. Los mecanismos de filtración preferentemente en estos compósitos, fueron bloqueos estándar ($n = 1,5$) y completo ($n = 2$) de los poros de la torta de filtración. Las permeabilidades de las tortas de filtración fueron estimadas por $^1\text{H-NMR}$. Es factible el uso de compósitos de petcoque como aditivos controladores de filtrado de bajo impacto ambiental para fluidos de perforación base aceite.

Palabras clave: compósitos coque de petróleo, leonardita, lignito organofílico, aditivo de control de filtrado, ensuciamiento de membrana, osmosis inversa.

1 Introduction

Venezuela, has a high production of petroleum coke (~22,000 t/day), which come from the improvement of the heavy oil in the José Antonio Anzoátegui Cryogenic Complex, Anzoátegui State. The objective is diversifying the industrial applications of this petroleum coke (petcoke). It is proposed to prepare a product from petroleum coke that functions as a fluid-loss control additive in oil-based drilling fluids and replace organophilic lignites normally used.

Organophilic lignites rank third in the list of imported well drilling additives by Venezuela, with an estimated 9,100 t/year. Organophilic lignite is straight lignite that has been treated with quaternary amine compounds to make it oil dispersible in oil- and synthetic-base fluids. Therefore, coke-lignite composites were synthesized by lipophilic alteration of intra and supra petcoke surfaces with a cationic surfactant and synthetic co-polymer (Beime y col., 1954, Patel y col., 1987, Álvarez y col., 1998, API 13I 2002, Santiago y col., 2003, Boskovic y col., 2005, Mathew y col., 2007) and to achieve a material that works properly as a filtering medium. Filter-cake permeability is one of the fundamental parameters that control both dynamic and static filtration. The size, shape and capacity of the particles to deform under constant pressure are important factors for the control of permeability (Krumbein y col., 1943, Sutzkover-Gutman y col., 2010).

Humic substances are like natural particles, compositional variability, size, molecular weight and functional groups, which make them interesting in the formation of a less permeable filter-cake (Sutzkover-Gutman y col., 2010).

This work evaluated the fouling of petcoke composites with colloidal lignites as an HPHT filtered control mechanism in oil-based drilling fluids. The equations used are derived from the filtration laws by blocking, assuming a Poiseuille flow and Hermia's models applying.

1. Filter-cake: particles are larger than the pores of the medium, depositing on the surface of the membrane ($\varnothing_{\text{particle}} \gg \varnothing_{\text{pore}}$).
2. Intermediate blocking: particles block a pore or deposit on the surface of the membrane (overlap is possible) ($\varnothing_{\text{particle}} \sim \varnothing_{\text{pore}}$).
3. Standard blocking (pore constriction): particles are deposited on the inner wall of the pore, reducing its diameter ($\varnothing_{\text{particle}} \ll \varnothing_{\text{pore}}$).
4. Complete blocking: particles block the pores without overlapping. That is, each particle blocks a pore ($\varnothing_{\text{particle}} \sim \varnothing_{\text{pore}}$).

2 Parte Experimental

2.1 Materials

Petroleum coke type shot, cationic surfactant, styrene-butadiene rubber (SBR) co-polymer and ACS grade aliphatic solvents were used.

2.2 Preparation and sample treatment

Petcoke shot and petcoke shot-lignite samples are milled and sieved to obtain a particle size distribution adequate. Samples are placed in a stainless steel reactor and then a certain amount of aliphatic solvent, polymer solution and cationic surfactant were added. Reactor was pressurized with inert gas. Temperature was raised to 60 °C with constant agitation during 4 hours (Beime y col., 1954, Álvarez y col., 1998, Boskovic y col., 2005, Mathew y col., 2007). Table 1 shows petcoke composites prepared with different percentages of petcoke shot.

Table 1. Formulations of petcoke composites

Composites	Shot petcoke (wt%)
Petcoke (PC)	85.7
Petcoke with 10 wt% lignite (leonardita type) (PC-10L)	73.3
Petcoke with 20 wt% lignite (leonardita type) (PC-20L)	61.0
Petcoke with 10 wt% local lignite (PC-10LL)	73.3
Petcoke with 20 wt% local lignite (PC-20LL)	61.0

2.3 Shot petcoke composites evaluation

Prepared petcoke samples were evaluated by means of the following techniques:

2.3.1 HPHT fluid-loss and rheological performance tests

Eight (8) oil-based drilling fluids were prepared by quadruplicate with the organophilic lignite (reference), petcoke and petcoke-lignite composites (Tables 2 and 3). The filtration volumes obtained from each drilling fluid were evaluated using models proposed by Hermia to describe four (4) fouling mechanisms for flow decrease at constant filtration pressure and applying the equation $d^2t/dV^2 = k$ (dt/dV)ⁿ and assigning the exponent (n) the values (0, 1, 1.5, 2) and k to distinguish the filtration mechanisms by membrane blocking (Hermans 1936, Hermia 1982) (see Table 4).

2.4 Measured of filter-cake permeability by ¹H-NMR spectroscopy.

A resonance instruments Maran-Ultra 2 MHz bench-top spectrometer was used to carry out the filter-cake permeability measurement and RiNMR/WinDXP software utilization for data acquisition and processing. Permeability

tests were making on all of the filter-cakes obtained of the HPHT fluid-loss test.

Table 2. OBDF formulations with organophilic lignite reference (FOL), petcoke composite (FPC) and petcoke-leonardite (FPC-L).

Formulation (Density: 12 ppg)	UN	FOL	FPC	FPC- 10L	FPC- 20L
Mineral oil	mL	220	220	220	220
Organophilic clay	g	8	8	8	8
Polar active	g	4	4	4	4
Organophilic lignite	g	14	-----	-----	-----
Composite	g	-----	21	21	21
Humectant	g	8	8	8	8
Hydrated lime	g	1.5	1.5	1.5	1.5
Barite	g	283	283	283	283

Table 3. OBDF formulations with organophilic lignite reference (FOL), petcoke composite (FPC) and petcoke-local lignite (FPC-LL).

Formulation (Density: 12 ppg)	UN	FOL	FPC	FPC- 10LL	FPC- 20LL
Mineral oil	mL	220	220	220	220
Organophilic clay	g	8	8	8	8
Polar active	g	4	4	4	4
Organophilic lignite	g	14	-----	-----	-----
Composite	g	-----	21	21	21
Humectant	g	8	8	8	8
Hydrated lime	g	1.5	1.5	1.5	1.5
Barite	g	283	283	283	283

Table 4. Hermia's models solutions.

Blocking mechanism	Equations for flow decline
Filter-cake, $n = 0$	$1/J^2 = 1/J_0^2 + \Gamma_c t$
Intermediate blocking, $n = 1$	$1/J = 1/J_0 + \Gamma_i t$
Standard blocking, $n = 1.5$	$1/J^{1/2} = 1/J_0^{1/2} + \Gamma_d t$
Complete blocking, $n = 2$	$-\ln(1/J) = \ln(1/J_0) + \Gamma_b t$

Where J : filtrate flow (s/cm); J₀: initial filtrate flow (s/cm); t : filtrate time (s) and Γ : equation slope

2.5 Morphology of petcoke composites.

Morphology with scanning electron microscopy (SEM), using the JEOL JSM6490LV instrument for visual analysis of petcoke composites surface to observe the lipophilic coating with the SBR co-polymer.

3 Resultados y Discusión

3.1 Behavior of petcoke composites as fluid-loss control additive in OBDF at 300 °F/500 psi.

Tables 5 and 6 shows the volumes of cumulative filtrated at 5, 10, 15, 20, 25 and 30 minutes up to 300 °F/500psi. Table

5 shows that filtrate accumulated volume of petcoke composite formulation (FPC) is higher (6.8 mL) compared to volume (5.7 mL) of organophilic lignite reference formulation (FOL). In contrast, the filtrate volumes of petcoke-leonardite composite formulations (FPC-10L and FPC-20L) were minors to reference formulation volume (FOL). Different behavior was presented by petcoke-local lignite formulations, the high volume of filtrate was provided by the formulation (FPC-10LL and FPC-20LL) with 10 wt% and 20 wt% local lignite in petcoke composite. Due to the local lignite mineral nature compared to the leonardite (oxidized lignite). Humic acids supply was poor and therefore the amount of local lignite in the petcoke composite must be increased to increase fouling. Humic acids (HA) are soluble and dispersible colloidal substances, capable of filtration medium fouling (Hermans 1936, Sutzkover-Gutman y col., 2010). Consequently, the decrease in the filtrated volume was possible to three factors i) petcoke modified surface, ii) petcoke composite particle size distribution and iii) colloidal humic particles source.

Table 5. Average filtrated volumes of OBDF formulations with organophilic lignite, petcoke and petcoke-leonardite composites

Time (min)	FOL Filtrated volumen (mL) ± 0.1	FPC Filtrated volumen (mL) ± 0.1	FPC-10L Filtrated volumen (mL) ± 0.1	FPC-20L Filtrated volumen (mL) ± 0.1
5	1.9	2.0	1.5	1.3
10	2.9	3.4	2.7	2.2
15	3.7	4.7	3.6	2.8
20	4.4	5.5	4.4	3.5
25	5.2	6.0	5.0	4.0
30	5.7	6.8	5.6	4.4
total 30	5.7	6.8	5.6	4.4

Table 6. Average filtrated volumes of OBDF formulations with organophilic lignite, petcoke and petcoke-local lignite composites.

Time (min)	FOL Filtrated volumen (mL) ± 0.1	FPC Filtrated volumen (mL) ± 0.1	FPC-10LL Filtrated volumen (mL) ± 0.1	FPC-20LL Filtrated volumen (mL) ± 0.1
5	1.9	2.0	2.9	2.3
10	2.9	3.4	4.5	3.8
15	3.7	4.7	5.7	4.9
20	4.4	5.5	6.7	5.7
25	5.2	6.0	7.6	6.5
30	5.7	6.8	8.4	7.0
total 30	5.7	6.8	8.4	7.0

When applying the Hermia's model, the homogeneity-sphericity of the particles and the cylindrical geometry of the pores (Hagen-Poiseuille) are ideally assumed (Hermans 1936, Sutzkover-Gutman y col., 2010). Thus, is tacit the inherent difficulty for identifying the blocking mechanisms

that govern flow under real conditions. Therefore, the selection of the dominant blocking mechanisms is through the evaluation of the linear correlation coefficient (R^2) of the flow equation $d^2t/dV^2 = k (dt/dV)^n$ given for each Hermia's model and OBDf. Three factors evaluation that contribute to the fluid-loss reduction can be addressed by calculating the filtrated flows as a function of time ($F(t)$) and volume ($F(v)$).

Table 7 shows the linear correlation coefficients (R^2) for each Hermia's model as a function of time and volume filtrated. Model with R^2 closer or equal to 1.0000, will indicate the blocking mechanism of greater tendency that contributes to fluid-loss decrease. For case of FOL, the average filtrated volume was 5.7 mL and predominant blocking mechanism was the filter-cake ($n = 0$) which is consistent with that reported in the literature for this substance type used in OBDf (Patel y col., 1987) (Fig. 1).

Table 7. Flow equation linear correlation coefficient (R^2) for the FOL formulation.

FLO	F(t)			
	$n_t = 0$	$n_t = 1$	$n_t = 1.5$	$n_t = 2$
R^2	0.9927	0.9694	0.9507	0.9277
FLO	F(v)			
	$n_v = 0$	$n_v = 1$	$n_v = 1.5$	$n_v = 2$
R^2	0.9888	0.9622	0.9238	0.9189

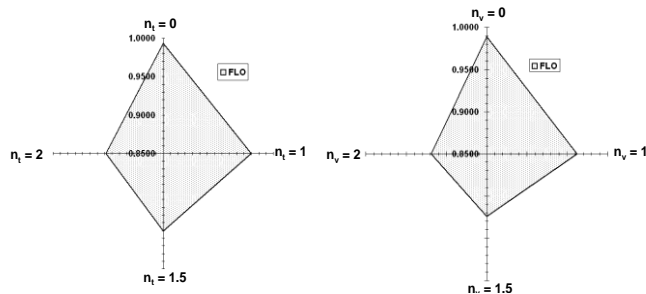


Fig. 1. Organophilic lignite (FOL) tendency to form filter-cake as preferential fouling mechanism.

For the FPC the average filtrated volume was 6.8 mL, with an increase of 1.1 mL compared to FOL, which represents an increase in fluid-loss of 20% (see Table 4). Linear correlation coefficients (R^2) for each Hermia's model using FPC (Table 8) shows a blocking mechanisms combination preferentially by standard ($n = 1.5$) and complete ($n = 2$) blocking.

Table 8. Flow equation linear correlation coefficient (R^2) for the FPC formulations in function of time and volume ($F(v)$ & $F(t)$).

FPC	F(t)			
	$n_t = 0$	$n_t = 1$	$n_t = 1.5$	$n_t = 2$
R^2	0.9773	0.9887	0.9905	0.9893
FPC	F(v)			
	$n_v = 0$	$n_v = 1$	$n_v = 1.5$	$n_v = 2$
R^2	0.9262	0.9550	0.9500	0.9728

Figure 2 clearly shows the filtrated-control blocking mechanism exercised by the FPC.

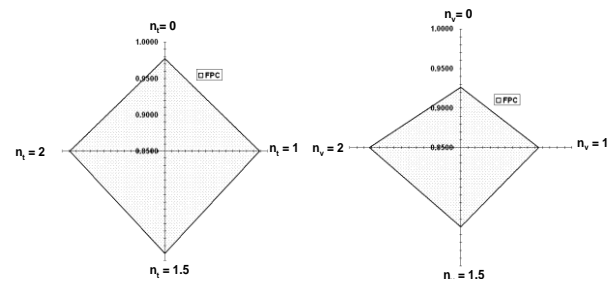


Fig. 2. Petcoke composite (FPC) tendency to form standard and complete blocking as preferential fouling mechanism.

Filtrated volumes of petcoke-leonardite composites (FPC-L) were similar to filtrated volume provided by FOL (see Table 5). FPC-20L formulation exhibited the lowest filtrate volume of the petcoke-leonardite formulations with 4.4 mL. Tables 9 y 10 shows the linear correlation coefficients (R^2) of flow equations from filtrated volume values provided by petcoke-leonardite composites. Predominant blocking mechanisms are standard ($n = 1.5$) and complete ($n = 2$) blocking. Figures 3, 4 and 5 shows the filtrated-control blocking mechanism exercised by the FPC-L with 10 wt% and 20 wt% of leonardite.

Table 9. Flow equation linear correlation coefficient (R^2) for the petcoke-leonardite (FPC-L) formulations in function of time ($F(t)$).

FPC-L	F(t)			
	$n_t = 0$	$n_t = 1$	$n_t = 1.5$	$n_t = 2$
10L	0.9953	0.9989	0.9975	0.9938
20L	0.9947	0.9881	0.9793	0.9671
30L	0.9857	0.9928	0.9942	0.9942

Table 10. Flow equation linear correlation coefficient (R^2) for the petcoke-leonardite (FPC-L) formulations in function of volume ($F(v)$).

FPC-L	F(v)			
	$n_v = 0$	$n_v = 1$	$n_v = 1.5$	$n_v = 2$
10L	0.9801	0.9933	0.9967	0.9979
20L	0.9874	0.9869	0.9813	0.9724
30L	0.9553	0.9722	0.9787	0.9838

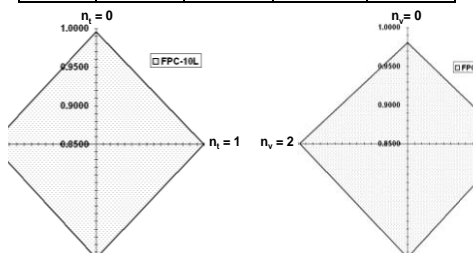


Fig. 3. Petcoke-leonardite composite (FPC-10L) tendency to form standard and complete blocking as preferential fouling mechanism.

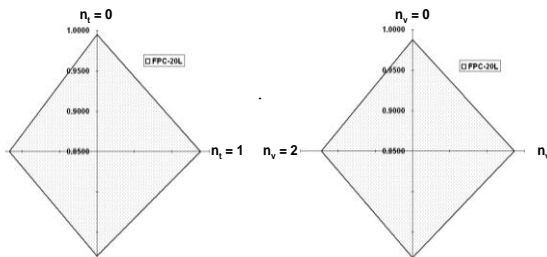


Fig. 4. Petcoke-leonardite composite (FPC-20L) tendency to form filter-cake and standard blocking as preferential fouling mechanism.

Petcoke-local lignite composites showed an average filter-loss of 7.1 mL, with a greater filter-loss difference with respect to the reference in 1.4 mL. In contrast, petcoke-local lignite composite with 20 wt% local lignite (FPC-20LL), achieved a filter-loss hight to organophilic lignite with 7.0 mL. Tables 11 and 12 shows the linear correlation coefficients (R^2) of flow equations from filtrated volume values provided by petcoke-local lignite composites. Blocking mechanisms combination preferentially by standard ($n = 1.5$) and complete ($n = 2$) blocking, either this, depending on time and volume. Figures 6, 7 and 8 show the filtrated-control blocking mechanism exercised by petcoke-lignite composites of three local-lignite concentrations 10 wt%, 20 wt% and 30 wt% respectively.

Table 11. Flow equation linear correlation coefficient (R^2) for the petcoke-local lignite (FPC-LL) formulations in function of time ($F(t)$).

FPC-LL	F(t)			
	$n_t = 0$	$n_t = 1$	$n_t = 1.5$	$n_t = 2$
10LL	0.9989	0.9884	0.9694	0.9452
20LL	0.9975	0.9966	0.9830	0.9719

Table 12. Flow equation linear correlation coefficient (R^2) for the petcoke-local lignite (FPC-LL) formulations in function of volume ($F(v)$).

FPC-LL	F(v)			
	$n_v = 0$	$n_v = 1$	$n_v = 1.5$	$n_v = 2$
10LL	0.9995	0.9927	0.9860	0.9641
20LL	0.9854	0.9958	0.9963	0.9936

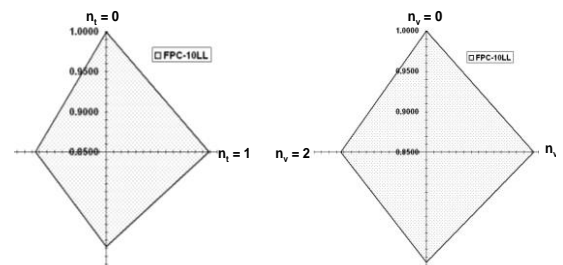


Fig. 5. Petcoke-local lignite composite (FPC-10LL) tendency to form filter-cake and standard blocking as preferential fouling mechanism.

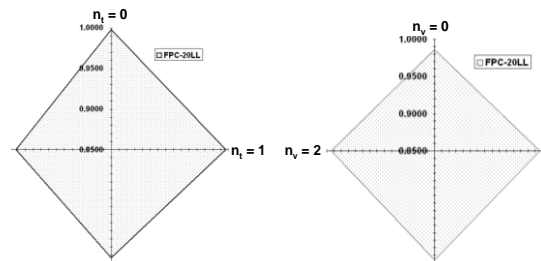


Fig. 6. Petcoke-local lignite composite (FPC-20LL) tendency to form filter-cake and standard blocking as preferential fouling mechanism.

3.2 Permeability measurement of filter-cake by $^1\text{H-NMR}$.

Absolute permeability was measured by means of Kenyon empirical equation (Equation 1), adjusting the δ parameter and longitudinal relaxation time (T1 value) (Kenyon 1988) to four filter-cakes of the FOL, FPC, FPC-20L and FPC-20LL formulations (Table 13).

Table 14 shows the estimated permeability for each filter-cake. Cake filtration with petcoke-local lignite and leonardite has the lowest permeability values. Mechanism fouling by humic substances of the interconnected pores in the filter-cake, functions to fluid-loss control. Fouling of humic substances is attributed to the accumulation of colloidal particles in the cake filtration (Krumbein y col., 1943, Sutzkovert-Gutman y col., 2010).

$$K_{\text{NMR}} = C(\Phi_{\text{NMR}})^a (T_{2,\log})^b \quad (1)$$

Table 13. Physical parameters for permeability calculation by $^1\text{H-NMR}$.

Formulations	RD	TAU (τ)	NECH	Ns	T1	T2
FOL	5.10^6	600	128	64	0.97	5.48
FPC	5.10^6	600	128	64	0.96	8.87
FPC-20L	5.10^6	600	128	64	0.91	4.32
FPC-20LL	5.10^6	600	128	64	0.74	3.29

Formulations	Area $\times 10^3$	Volume (mL)	Area/Ns	$^1\text{H-NMR}$	Delta (δ)
FOL	107	3.84	88.349	18.94	0.10
FPC	54	4.05	93.857	9.07	0.10
FPC-20L	55	3.74	85.339	10.14	0.10
FPC-20LL	56	3.75	86.089	10.22	0.10

Where:

TAU (τ): it is twice the inter-echo time in the pulse sequences CPMG, which was used during the run.

Ns: number of Scans in the $^1\text{H-NMR}$ experiments.

NECH: number of echoes in the pulse sequence CPMG, which were used in the measurement.

T1: longitudinal relaxation time obtained through the INVREC sequence, using the RINMR software.

T2: transverse relaxation time obtained through the CPMG sequence, using the RINMR software.

Area: it is the constant area under the curve that was obtained by the distribution of T2, with the WinDX software.

RD: represents the maximum measurement time that was used in the experiment.

Table 14. Estimated permeabilities (k) of filter cakes by $^1\text{H-NMR}$.

Formulation	Length (mm)	Diameter (mm)	Radius (mm)	Permeability (K) (mD)
FOL	1.55	55.19	27.595	4.64
FPC	1.74	54.41	27.205	3.91
FPC-20L	1.60	54.41	27.205	3.90
FPC-20LL	1.61	54.45	27.225	3.97

3.3 Evaluation of petcoke composite morphologies by SEM.

Lipophilic coating of the petcoke and petcoke-lignite composites with the SBR co-polymer to obtain a product with better characteristics to form a cake filtration can be seen in the micrographs made to the samples before and after the coating (Figures 9 and 10).

Fig. 7. Micrography (a) Shot petcoke particle and (b) petcoke composite particle.

Fig. 8. Micrography (c) petcoke-leonardite composite particle (d) petcoke-local lignite composite particle.

4 Conclusions

Preparation of petroleum coke-based composite as additive to fluid-loss control at 300 °F/500 psi of oil-based drilling fluids was feasible. Cake filtration evaluate of oil based drilling fluids applying Hermia's model as a function of time and volume filtrated was possible.

Organophilic lignite shows a clear tendency to fluid-loss control by cake filtration and petcoke composite preferentially through standard and intermediate blocking of cake filtration pores.

Drilling fluids containing petcoke-leonardite composite (FPC-L), shows lower filtrate flow (J) compared to organophilic lignite. The contribution of colloidal humic substances is decisive to reduce the permeability of the filter-cake. Filtration blocking mechanisms found were: standard and complete blocking. Petcoke-local lignite composite (FPC-LL) controls the loss of filtrate similarly to the petcoke-leonardite composite when the lignite content was 20 wt%. The filtration blocking mechanisms found were: filter-cake and intermediate blocking.

Conflicto de intereses:

El autor declara que no hay conflicto de intereses.

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