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# **NANOTECHNOLOGY EFFECT IN HEAVY OIL RHEOLOGY AND ITS PRODUCTIVITY IMPACT**

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## **Abstract**

The nanoparticles and nanofluids effect as viscosity reducers for heavy crude oil (HO) was evaluated. The effect of alumina, neutral silica and acidic silica nanoparticles was evaluated through n-C7 asphaltene adsorption and aggregation tests using UV-vis spectrophotometry and dynamic light scattering. The nanoparticles of acidic silica were used to prepare a water-based nanofluid at different concentrations in distilled water, and also with the addition of 1.0 wt% of a non-ionic surfactant. The shear rheological response was obtained as function of nanoparticle concentration, temperature (from 298 to 323 K and shear rate ranging from 0 to 50 s<sup>-1</sup>). Experimental results indicate that increasing the concentration of nanoparticles in the mixture, up to 10000 ppm, leads to a viscosity reduction of approximately 90% in comparison with the nanoparticle-free crude oil. At higher concentration of nanoparticles, the effectiveness of the heavy oil viscosity reduction diminishes. Rheological tests showed a non-Newtonian behavior for the mixtures tested at 298 K. However, as the temperature reaches 323 K the specimens behave in a Newtonian fashion. Coreflooding tests were conducted under typical reservoir conditions of pore and overburden pressures, i.e. 2600 and 3600 psi, respectively, and at 360 K. Results indicate that the addition of nanoparticles increases the heavy oil mobility and leads to an improvement in oil recovery of roughly 16 percent.

**Keywords:** rheology; heavy crude oil, nanoparticles

## ***Resumen***

*El efecto de nanopartículas y nanofluidos como reductores de la viscosidad del crudo pesado (HO), fue estudiado. El efecto de las nanopartículas de alúmina, sílice neutra y sílice ácida se evaluó a través de la*

*adsorción de asfaltenos n-C7 y pruebas de agregación utilizando espectrofotometría UV-VIS y dispersión de luz dinámica. Las nanopartículas de sílice ácida se utilizaron para preparar una nanofluido a base de agua a diferentes concentraciones, en agua destilada, y también con la adición de 1,0% en peso de un surfactante no iónico. Se obtuvo la caracterización reológica del crudo como función de la concentración de nanopartículas, la temperatura (298 a 323 K y las tasas de corte entre 0 a 50 s<sup>-1</sup>). Los resultados experimentales indican que el aumento de la concentración de las nanopartículas en la mezcla, hasta 10.000 ppm, inducen a una reducción de la viscosidad de aproximadamente 90% en comparación con el crudo pesado libre de nanopartículas. A mayor concentración de las nanopartículas, la eficacia de la reducción de la viscosidad del crudo pesado disminuye. Las pruebas reológicas mostraron un comportamiento no newtoniano para las mezclas evaluadas a 298 K. Sin embargo, cuando la temperatura alcanza los 323 K los especímenes se comportan de una manera newtoniana. Pruebas de desplazamiento se llevaron a cabo bajo condiciones típicas de yacimiento con valores de presión poro y presiones de sobrecarga, de 2600 y 3600 psi, respectivamente, y a 360 K. Los resultados indican que la adición de nanopartículas aumenta la movilidad del crudo pesado y conduce a una mejora en la recuperación de petróleo de aproximadamente 16 por ciento.*

**Palabras clave:** reología; crudo pesado, nanopartículas

## 1. Introduction

HO has an API gravity lower than 20° and the corresponding value for EHO is lower than 10° (Rana, Samano et al. 2007, Shah, Fishwick et al. 2010, Chew 2014). HO and EHO are also characterized by containing large concentrations of heavy metals and heteroatoms and heavy compounds such as asphaltenes, as well as low H/C ratio (Ghannam, Hasan et al. 2012), which directly influence the observed crude oil viscosity, typically higher than 100 cP at reservoir conditions (Ghannam, Hasan et al. 2012). Asphaltenes are refractory molecules with high molecular weight that can impact the different stages of the production system and refining streams (Groenzin and Mullins 1999). Asphaltenes contain heteroatoms such as O, N and S and metals such as Ni, Fe and V. The presence of heteroatoms and their location in the structure make asphaltenes the most polar molecules present in crude oil, leading to their self-association and further formation of large asphaltic flocs. At high concentration of asphaltene, namely > 40000 mg/L, the increase in viscosity is mostly due to the formation of a viscoelastic network of nanoaggregates (Mullins, Betancourt et al. 2007, Yudin and Anisimov 2007). In addition, sulfur can form strong C-S and C=S bonds, which can also contribute to an increase in the crude oil viscosity. (Chuan, Guang-Lun et al. 2010, Ghanavati, Shojaei et al. 2013). To improve production, transport and refining of HO and EHO, several techniques under reservoir and surface conditions have been used, namely: i) emulsification of oil-in-water, ii) use of annular flow as a method for reducing the drag forces (Joseph, Bai et al. 1997, Joseph, Bai et al. 1999, Saniere, Hénaut et al. 2004, Wylde, Leinweber et al. 2012), iii) deasphalting oil, iv) application of thermal processes and v) the dilution with solvents and light hydrocarbons such as naphtha (Al-Maamari and Buckley 2003, Gharfeh, Yen et al. 2004, Oskui, Reza et al. 2011), toluene (Urquhart 1986), xylene (Gharfeh, Yen et al. 2004), gasoline (Hart 2014), diesel (Heim, Wolf et al. 1984, McMillen 1985), light oil (Urquhart 1986), or mixtures. Naphtha has been so far the most used diluent (McCants

1992, Tipman and Sankey 1993, Argillier, Barre et al. 2001). In some instances, mainly in Venezuela and Colombia, the quantity of naphtha used in the upgrading of these crude oil types can reach 20-40 vol% (Martínez-Palou, de Lourdes Mosqueira et al. 2011). Adding naphtha can effectively reduce the viscosity and increase the API gravity of HO and EHO (Alvarez, Poteau et al. 2008, Hasan, Ghannam et al. 2010, Mortazavi-Manesh and Shaw 2014). The excessive use of naphtha for diluting crude oil implies an increase in the operational costs (Urquhart 1986), becoming an impractical technology (Urquhart 1986). Dilution with naphtha is an unfriendly technology for humans due to its lower boiling point, 100% volatiles content, and lower explosive limits (Urquhart 1986). To the best of our knowledge, there are no studies reporting on the inhibition of asphaltene self-association processes using nanoparticles, no findings on their impact on relative permeability curves and crude oil recovery (%) under flow conditions in porous media have been published at typical reservoir temperature and pressure; their use in field test based on “huff-puff” type stimulations is also absent. Nanoparticles have been used by the oil industry for formation damage inhibition (Amanullah and Al-Tahini 2009, Mohammadi, Akbari et al. 2011, Franco, Patiño et al. 2013, Franco, Nassar et al. 2013), HO and EHO upgrading (Franco, Montoya et al. 2013), enhanced (EOR) and improve oil recovery (IOR) processes (Franco, Nassar et al. 2013, Nassar, Betancur et al. 2015), and wastewater remediation (Franco, Martínez et al. 2014). Due to their particle sizes, between 1 and 100 nm, large available surface area, high dispersibility and tunable physicochemical characteristics, nanoparticles are prone to selectively adsorb asphaltene and inhibit their self-association (Nassar, Hassan et al. 2011). In a previous study (Franco, Nassar et al. 2013, Nassar, Betancur et al. 2015, Zabala. R 2016), our research group focused on using silica,  $\gamma$ -alumina and magnetite nanoparticles to inhibit the aggregation of asphaltene under varying temperature, solvent ratios and asphaltene concentration (Nassar, Betancur et al. 2015). Silica nanoparticles induce higher percentages of reduction of the asphaltene mean aggregate size, which could prevent the formation of large viscoelastic network and reduce the oil viscosity. In this order, the aim of this paper is to evaluate the effect of nanoparticles on the rheological properties of a Colombian HO at varying conditions of temperature, shear rate and dosage of nanoparticles. The selection of the nanoparticles optimized towards the adsorption and size reduction of asphaltene was achieved using adsorption isotherms and the growth kinetics asphaltene. In addition to rheological flow curves, coreflooding tests at reservoir conditions were also performed. The results in this paper are expected to open a wider landscape about the use of nanoparticles in IOR processes based mainly on “huff & puff” configurations.

## 2. Experimentation

### 2.1 Synthesis and characterizations of nanoparticles/nanofluids

SiO<sub>2</sub> nanoparticles were synthesized accordingly to the sol-gel method as described in previous works (Hench and West 1990, Márquez 2015). The procedure for acidification of the surface of SiO<sub>2</sub> nanoparticles consisted of mixing nanoparticles and sulfuric acid at 0.3 wt% to obtain a solution at pH = 4. The nanoparticles were dried at 393 K for 4 hours. The carrier fluid used was composed of a mixture of distilled water and 5 wt% surfactant. The procedure followed for the preparation of the nanofluid is described elsewhere (Haddad, Abid et al. 2014, Sidik, Mohammed et al. 2014). The mean particle size of the nanoparticles ( $d_p$ ) was measured using

dynamic light scattering (DLS) with the help of a nanoplus-3 from Micromeritics (Norcross,GA ) at 298 K and equipped with a 0.9 mL glass cell (Nassar, Betancur et al. 2015). The BET surface area (SBET) of the nanoparticles was measured through nitrogen physisorption at 77 K using an Autosorb-1 from Quantacrome BET, after outgassing samples overnight at 413 K under high vacuum ( $10^{-6}$  mbar). Total acidity of the nanoparticles surface was determined via a  $\text{NH}_3$ -Temperature programmed desorption. We used a ChemBet TPR/TPD (Quantachrome Instruments, USA). The desorbed amount is calculated using a calibration curve (Nassar, Hassan et al. 2011, Nassar, Franco et al. 2015). The  $d_{p50}$  for nanoparticles was 11, 11 nm and 35 nm for  $\text{SiO}_2$ ,  $\text{SiO}_2\text{A}$  and  $\text{Al}_2\text{O}_3$  respectively, SBET was 210, 187, 123  $\text{m}^2/\text{g}$  for  $\text{SiO}_2$ ,  $\text{SiO}_2\text{A}$  and  $\text{Al}_2\text{O}_3$  respectively and values of  $\text{NH}_3$  uptake obtained for the nanoparticles used in this study were 1.19, 1.42 and 0.34  $\text{mmol/g}$ .

## 2.2 n-C<sub>7</sub> asphaltenes adsorption

Adsorption isotherms for n-C<sub>7</sub> asphaltenes on the nanoparticles were performed at 303 K, in a concentration (C) range of 100-1500 mg/L and at a ratio of nanoparticle-to-solution of 100 mg to 10 mL (Franco, Montoya et al. 2013, Guzmán, Betancur et al. 2016). An UV-vis spectrophotometer Genesys 10S (Thermo Scientific, Waltham, MA) was used to determine the asphaltene concentration in solution before and after the adsorption process.

## 2.3 n-C<sub>7</sub> asphaltenes aggregation experiments

DLS measurements of heavy oil model solutions in presence and absence of nanoparticles were performed to evaluate the average aggregate size of n-C<sub>7</sub> asphaltenes as a function of nanoparticle addition. n-C<sub>7</sub> asphaltenes were dissolved in a mixture n-heptane/toluene (Heptol) 40% v/v at a concentration of 1000 mg/L. The mixture was magnetically stirred at 300 rpm and aliquots were taken to obtain the mean asphaltene aggregate size as a function of time. The dosage of nanoparticles was fixed at 10 g/L to guarantee total decantation of the nanoparticles and thus obtain an accurate measure of aggregate size of n-C<sub>7</sub> asphaltenes (Nassar, Betancur et al. 2015).

## 2.4 Evaluation of nanoparticles/nanofluids as viscosity reducers

Rheological measurements were performed using a Bohlin rotational rheometer C-VOR 200 (Malvern Instruments, Worcestershire - UK), equipped with a Peltier plate for temperature control, with a plate-plate geometry and a gap of 150 microns. To analyze the change in viscosity induced by nanoparticles addition, several concentrations were evaluated, namely 0, 1, 3 and 4 wt% at room temperature at shear rate values 0 – 100  $\text{s}^{-1}$ . Each measurement was repeated three times.

## 2.5 Coreflooding tests

The selected core has a length of 25 cm, with a diameter of 3.81 cm and a porosity of 11%. The injection fluid consisted of a synthetic brine of 2 wt% of KCl. The core absolute permeability was estimated as 1.1 Darcy, through the injection of 20 pore volumes (PV) of the prepared brine. The nanofluid used for performing the test is prepared according to the results of the rheological measurements according to the one that generates greater viscosity reduction. the “base system test” consisted of subjecting the porous media to a period of aging time needed for wettability restoration. This is achieved by continuous injection of heavy oil during 15

days. Once the system is oil-wet, oil ( $k_o$ ) and water ( $k_w$ ), corresponding to the end-point effective permeabilities of the oil and water phases, respectively, are measured. This is performed first by injecting 20 PV of water at conditions of oil residual saturation ( $S_{or}$ ) followed by 20 PV of oil at conditions of water residual saturation ( $S_{wr}$ ). Then, oil ( $k_{ro}$ ) and water relative permeability ( $k_{rw}$ ) and oil recovery curves are obtained. Posteriorly, the sample is saturated with oil again, in a secondary drainage process, in order to prepare the core for nanofluid injection. The “post nanoparticles injection” system is obtained by injecting 1 PV in production direction and then letting it soaking for a period of 12 h. Then values of  $k_o$ ,  $k_w$ ,  $k_{rw}$ ,  $k_{ro}$  and recovery curves are constructed following the aforementioned procedure.

### 3. Results

#### 3.1 Adsorption Isotherms and Aggregation Kinetics

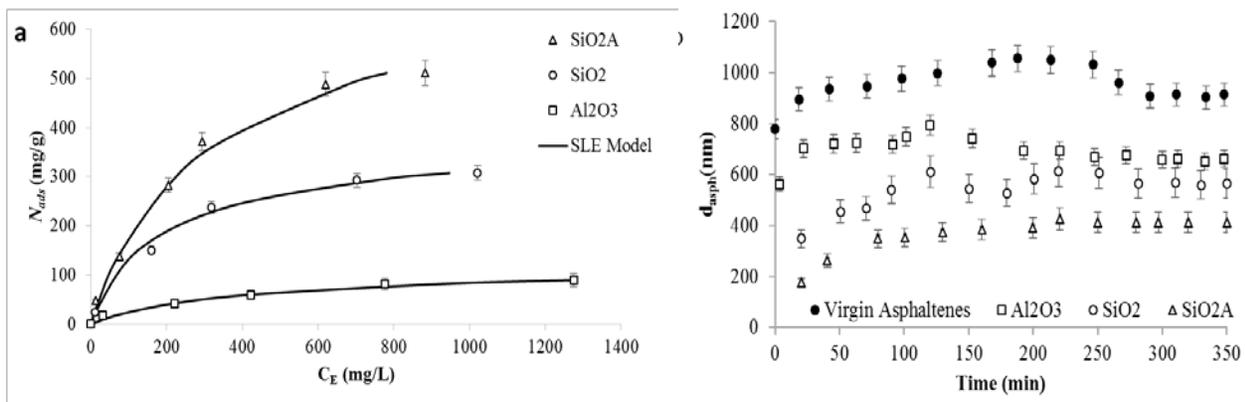


Figure 1. a) Adsorption Isotherms of asphaltenes onto nanoparticles b) Asphaltenes aggregation kinetic on nanoparticles presence and absence at 298 K

The results indicate that the adsorption ability to capture  $n$ -C<sub>7</sub> asphaltenes by nanoparticles follows the order SiO<sub>2</sub>A > SiO<sub>2</sub> > Al<sub>2</sub>O<sub>3</sub>, however, the nanoparticles evaluated also must have the ability to reduce the mean size of asphaltene aggregates ( $d_{asp}$ ) in the fluid with the objective of impacting the configuration and distribution of them in the oil matrix. In heavy crude oils with asphaltene content above 5 wt%, the configuration of asphaltenes is generally formed by a series of clusters of nanoaggregates, forming a viscoelastic network of large size (Mullins, Betancourt et al. 2007, Yudin and Anisimov 2007). For this reason, it is expected for the nanoparticles to have the ability to break the attachment points within the viscoelastic network and greatly reduce the size of the aggregates of asphaltenes, resulting in the reduction of the crude oil viscosity. In Figure 1 b), the curve of  $n$ -C<sub>7</sub> asphaltenes in absence of nanoparticles, initially reflects a growth in  $d_{asp}$  as a function of time, followed by a reduction on this value until stabilization after approximately 300 min. This likely occurs due to aggregation-fragmentation forces that exist under certain shear conditions, which in turn have a direct influence on the mean asphaltene aggregate size (growth/reduction) (Acevedo, Castillo et al. 1998, Spiecker, Gawrys et al. 2003, Nassar, Betancur et al. 2015). The behavior of  $n$ -C<sub>7</sub> asphaltenes in the presence of a nanoparticle system is similar, but with a smaller  $d_{asp}$  and a faster stabilization after 180 min. The evaluated

nanoparticles were able to successfully reduce the  $d_{asp}$  in decreasing order of effectiveness,  $SiO_2A < SiO_2 < Al_2O_3$ . Therefore, the  $SiO_2A$  nanoparticles were selected to conduct an evaluation of the HO rheological changes.

### 3.2 Rheologic Measurements.

#### 3.2.1 Effect of Temperature and Nanoparticles on oil viscosity.

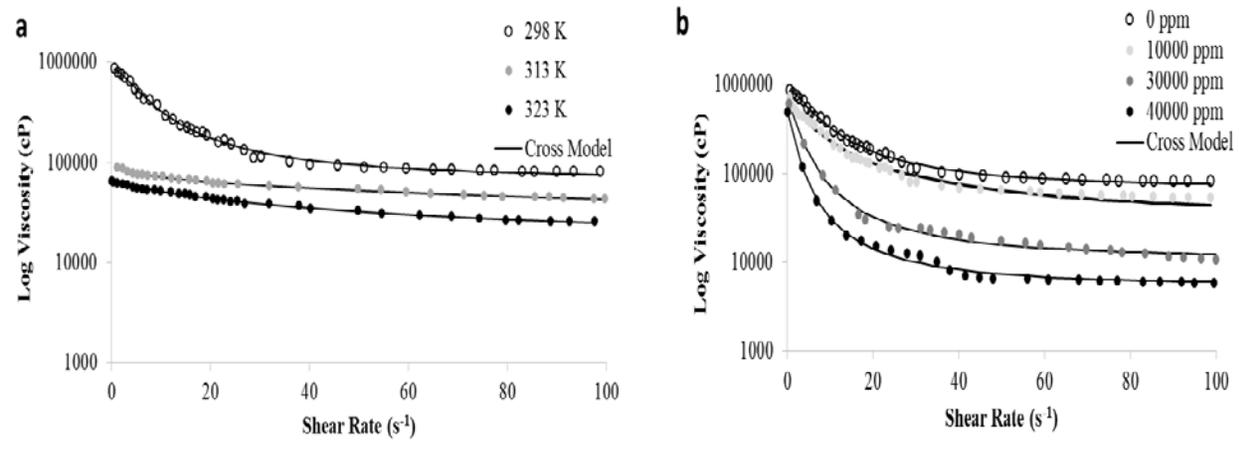


Figure 2. a) Viscosity as function of shear rate for temperatures of 298, 313 and 323 K for virgin crude oil. b) Viscosity of shear rate for crude oil in absence and presence of different dosages of  $SiO_2A$  nanoparticles at 298 K.

Besides the effect of shear rate and time of application thereof, viscosity is strongly temperature-dependent. The results revealed that for a fixed value of shear rate, the crude oil viscosity decreases as temperature increases. A temperature increase should generate a decrease in viscosity, a behavior is typical of liquids. As shown in Figure 2 b), as the concentration of nanoparticles in the fluid increases, the viscosity is reduced. For the highest concentration evaluated (4 wt%), viscosity reductions of more than one order of magnitude for all shear rate evaluated are observed. When an amount equivalent to 1 wt% of nanoparticles is added to the oil, the decrease in viscosity is minimal with similar values to those of heavy oil. The behavior described by curves shows the rheological non-Newtonian behavior, with decreasing viscosity, while the content of each of the evaluated nanoparticles mixtures increases.

#### 3.2.2 Effect of nanofluid on oil viscosity

It is noticed that the addition of solid nanoparticles to heavy crude oil generates a viscosity reduction, which offers the necessary conditions for optimizing oil mobility. However, for a possible industrial IOR application, nanoparticles have to be suspended in a carrying fluid in order to supply injectivity requirements.

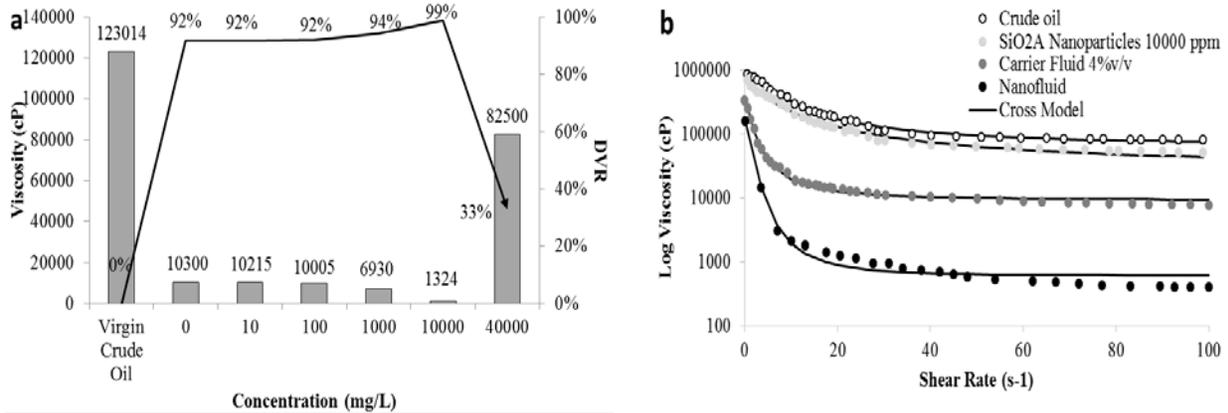


Figure 3. a) Viscosity of crude oil in absence and presence of nanofluid with different concentration of nanoparticles at 298 K and shear rate of 10 s<sup>-1</sup>. b) Viscosity as function of shear rate for crude in absence and presence of the prepared carrier fluid at 4% v/v, SiO<sub>2</sub> nanoparticles at 10000 mg/L the selected nanofluid.

Again, it is seen that as the concentration of nanoparticles increases in the nanofluid, the mixture viscosity decreases. The optimum point is at a concentration of 10000 mg/L, where a maximum degree of viscosity reduction (DVR) is obtained. However with increasing concentration of nanoparticles up to 40000 mg/L, the DVR is much lower (33%), which is possibly due to aggregation of the solid particles in the nanofluid, hence reducing interaction with asphaltenes present in crude oil. A synergistic effect is seen that uses of power nanofluids on the nanoparticles or the carrier fluid. In effect of the addition of a liquid medium to transport the nanoparticles is favorable for reducing viscosity, this could be due to two main reasons: 1) the liquid medium serves as a partial extender of oil, and 2) the nanoparticles are scattered in the medium and when mixed with oil they can occupy more space and foster greater contact with the asphaltenes present in crude in comparison to the case when they are mixed in solid form (Zabala. R 2016).

### 3.3 The Coreflooding Test

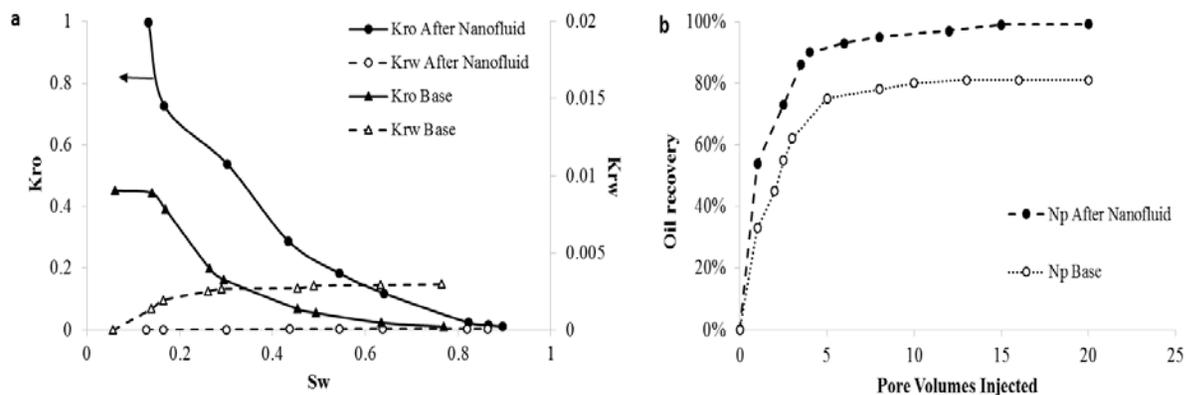


Figure 4. a) The relative permeability curves for the base, the core with crude oil-wet and the treated system after nanofluid injection in their respective formation plug.

Figure 4 a) shows that the Kro curve for the core treated is higher than that for the base system, indicating that the treatment is effective in increasing the relative permeability of oil. According to previous results of the static tests, including treatment dramatically reduces the oil viscosity and therefore increases mobility of oil in the porous medium. b) shows that for a 81% of oil recovery obtained for the base system and after injection of nanofluid, 97% of recovery is obtained, i.e. the system increases its oil production capacity by 16%.

#### 4. Conclusion

The importance and impact of the proposed technology as optimizer agent mobility heavy oil is demonstrated. This is especially important in IOR processes with huff-n-puff configurations where a soaking time is required.

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