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NANOPARTICLES SELECTION FOR HEAVY OIL RECOVERY, STUDY CASE ON THE ECUADOR EAST

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ABSTRACT

Nanoparticles application for heavy oil recovery has shown positive results in laboratory experiments simulating enhancing oil recovery. This work consists in a comparative analysis of the nanoparticles to determinate that has better applicability for viscosity reduction and increase of oil recovery on the Ecuador east basin. The majority studies are based on the use of the nanoparticles such as: aluminum oxide, silicon oxide, titanium oxide, nickel oxide, among others that at certain concentrations were mixed with formation water or brine to obtain a respective nanofluid to be injected into samples of wet cores with heavy oil obtained from a reservoir. The analysis result indicates that aluminum oxide reduces the oil viscosity in a more effective way in addition of obtaining the best recovery percentage in comparison with the other nanofluids, considering that also helps to increase API grades turning them in a lighter hydrocarbon for its refining process.

Keywords: Nanoparticles, nanofluids, viscosity, API grades, oil recovery.

1. INTRODUCTION

Currently the quality of Ecuadorian oil that is found in the most important reservoir has an average density between 15 to 19° API, being considered therefore "heavy hydrocarbons". Which due to its high viscosity have a low mobility preventing the effective flow of the same, imperative characteristic in terms of production.

In this way, significant challenges are generated at the moment of extraction, since to make said production effective, the mobility of oil must be improved by altering the properties of the fluid and the reservoir.

Because oil displacement efficiency and recovery tend to decrease as soon as the mobility ratio increases (Equation. 1), techniques are used to minimize the viscosity of the oil by means of thermal processes such as vapor injection, increasing the viscosity of the water through of the addition of polymers, altering the relative permeabilities of the fluids by varying the reservoir wettability.

$$M_{w-o} = \frac{Kr_w * \mu_o}{Kr_o * \mu_w}$$

Equation. 1: Watter-oil mobility ratio.

The main disadvantage of vapor injection is the loss of heat during the injection process that limits its applicability to deep reservoir, such as chemical injection (polymers, surfactants, alkaline fluids) which are not applicable to high water salinity, in addition the investments for the purchase of chemicals.

Due to these limitations, the study of another alternative is made to alter the characteristics of fluid and reservoir, through the use of nanoparticles, which have physical and chemical properties that are useful for the processes of recovery and improvement of the reservoir.

For the injection of the nanoparticles to the porous medium where the change of properties will take place, in the nucleus (laboratory case) or in the reservoir, the preparations of nanofluids must be carried out by mixing nanoparticles such as aluminum oxide (Al_2O_3), nickel oxide (NiO), silicon oxide (SiO_2), titanium oxide (TiO_2) or mixtures of these nanoparticles (average size between 20nmy 50nm) with treated saline water (processed formation water) / petroleum or ethylene glycol (Figure-1).

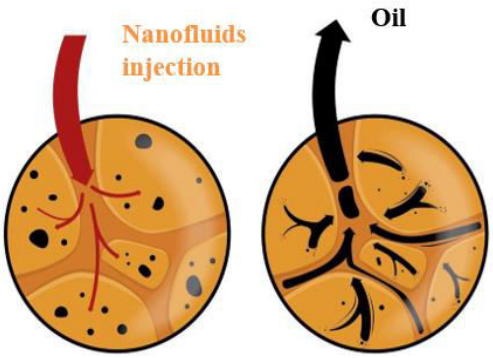

Figure-1. Nanofluids injection.

In addition, nanoparticles can be used to improve water disposal, break emulsions, even if they can be used as inhibitors of precipitation of elements and maintenance of the moment pressure above the bubble point.

The main mechanism for the diffusion of nanofluids in oil production is called the pressure gradient of rupture ($\Delta\gamma$), which is correlated with the ability of a fluid to propagate along a Surface, said mechanism is driven by the Brownian movement and the electrostatic repulsion between the nanoparticles, this repulsion force will be greater when the nanoparticles are smaller and will

increase as the number of nanoparticles increases, emphasizing that the size of nanotechnology is usually smaller than that of the throat of the application in an oil reservoir.

2. OPERATIONAL THEORY

Nanoparticles have physical and chemical properties very different from those of the same material son a conventional scale. The properties of nanoparticles depend on their form, size, surface characteristic and internal structure. The main parameters of the nanoparticles are their shape (including aspect ratios where appropriate), the size and the morphological sub-structure of the substance (Table-1). In the presence of chemical agents (surfactants), the Surface and interfacial properties can be modified. [1]

Table-1. Nanoparticles properties.

Name	Formula	Size (nm)	Appearance	SSA (m^2/g)	Molecular weight (g/mol)	Fusion point (°C)	Ebullition point (°C)
Aluminum Oxide	Al_2O_3	40	White	69	101,961	2.054	3.000
Nickel Oxide	NiO	50	Dark Gray	>6	74,96	1.984	=3.075
Silicon Oxide	SiO_2	15	White	650	60,08	>1.600	2.230
Titanium Oxide	TiO_2	50	Light yellow	50	79,866	1.830-1850	2.500-3.000

For the injection of the nanoparticles into the porous medium, the preparation of nanofluids must be carried out by mixing nanoparticles with treated formation water.

For this type of work the main mechanism for the propagation of nanofluids in the production of oil is called the pressure gradient of rupture, said mechanism is driven by the Brownian movement and the electrostatic repulsion between the nanoparticles, this repulsion force depends on the size and quantity of nanoparticles, said fore will be greater when the size of the nanoparticles is smaller, and will increase as the quantity thereof increases. [2]

The presence of these nanoparticles in three phase contact zone has a tendency to create a film structure (wedge shape). The pressure gradient rupture correlates with the ability of fluids to spread along the surface of a substrate due to the imbalance of interfacial forces between the oil phase and the aqueous phase. The interfacial forces will cause the contact angle (θ) of the aqueous phase (nanofluid) to decrease to 1° and the result is a film, (Figure-2), this film will act as a separator of forming fluid such as: gas, oil, paraffin and water.

Driven by the aqueous pressure of the liquid portion, the nanofluid is able to propagate along the surface in the form of a monolayer of particles and becomes completely disseminated when the contact angle is zero.

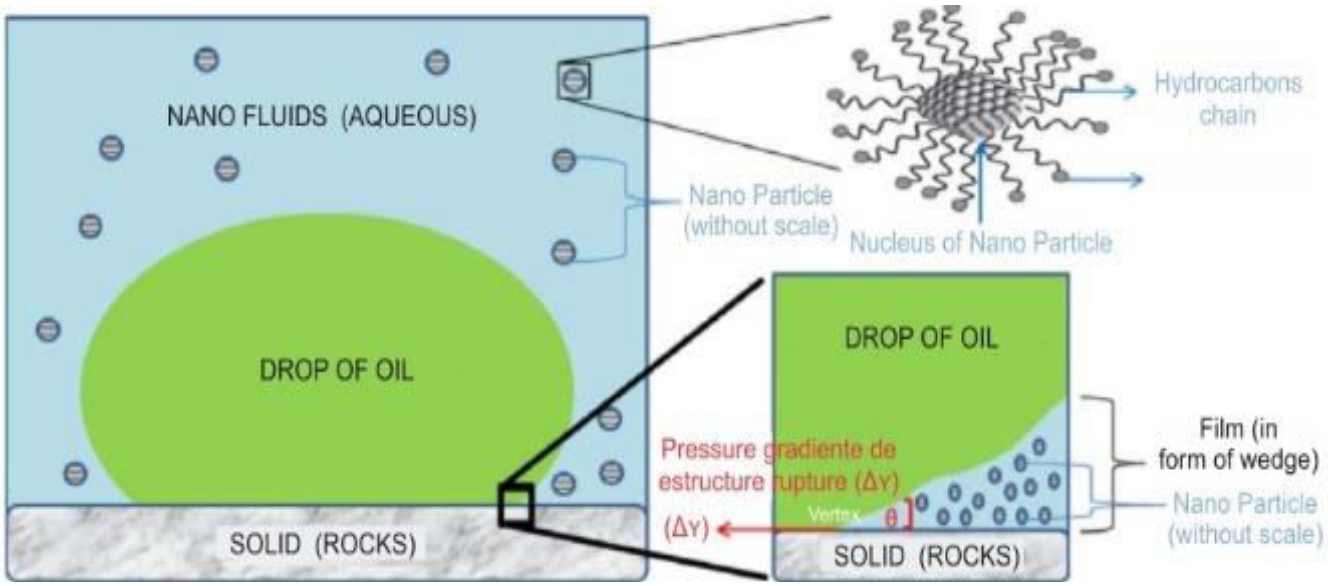

Figure-2. Scheme of the nanoparticles and the mechanism of the pressure gradient of rupture.
Source: SPE 164106.

The tension of the film is high near the vertex due to the structuring of nanoparticles in the wedge confinement; this drives the nanofluids to spreading at the tip of the wedge while the tension of the film increases towards the vertex of the wedge.

The coefficient of dispersion increases exponentially with a decrease in the thickness of the film or the decrease in the number of layers of particles within the film, as the thickness of the film decreases towards the vertex of wedge, the pressure gradient of rupture increases. [2]

The mobilization of the nanoparticles depends strongly on the viscosity of the dispersant fluid, which increases the stability of the dispersion, in some experiments that have been carried out, it was observed that the viscosity is very important to able to displace these particles through the porous medium, the transport potential of the nanoparticles has been improved by diffusion with polymers dispersing the nanoparticles (20-200 nm). [3]

Furthermore, the chemical functions of the surface of the particles can improve the stability of the dispersion and therefore the reduction of flocculation, another way of moving them is dispersing them in a higher concentration than normal.

The mobilization of nanoparticles constantly improves and not only the size of the particle allows an easy passage through the porous medium, it must take into account the repulsions between the particles and the walls of the pores, however, the methods and conditions used to prepare the nanoparticle suspension can still strongly influence the mobility of the particles and the size and charge characteristics of the particles. [3]

Within the porous medium there are chemical reactions that occur through catalytic hydrogenation. Catalysis is the process by which the rate of a chemical reaction is increased or decreased when a substance (catalyst) influences the affinity of two reagents. Catalysis is considered one of the greatest contributions to chemistry, since without it, processes such as oil refining in the oil industry could not be carried out. [4]

In this case study will work with heterogeneous catalysis which occurs when the catalyst and the substances react in different states.

This type of catalysis is deployed with the addition of the nanoparticles, so the surface area must be taken into account because of its influence on the activation energy (E_a) since the catalysts absorb molecules of the reactive weakening the bonds and decreasing the energy activation required.

Knowing that a catalyst accelerates a reaction must fulfill the following specifications:

- Activity: must guarantee the rate of reaction with the adequate property of surface area for decrease and reaction of the activation energy.
- Selectivity: in such a way that it minimizes the secondary products and facilitates the formation of a pragmatic group in front of others.

Taking into account that in a hydrogenation the reactions are exothermic, these do not happen spontaneously because the energies of activation are high, just heating does not provide the necessary energy for the molecules to reach the state of transition; however, the reaction can occur gradually when catalyst is added so that a catalytic hydrogenation can be carried out, in which the catalyst facilitates the development of the hydrogenation reaction since on the metal surface the hydrogen molecules are absorbed and then the π H_2 bonds are broken and a simple bond is formed with the metallic nanoparticles, the crude to react is also adsorbed on the metal surface, with its π bonds interacting with the empty orbitals of the metal. The crude molecule moves around the surface until it collides with a hydrogen atom bound to the metal, suffers the reaction and then detaches itself from the metal as a hydrogenated product. [4]

So, it is concluded that the catalytic hydrogenation is an efficient method for the addition of H_2 to carbon-carbon double, triple and / or an aromatic nucleus, it should be emphasized that the improvement that is going to be make for the crude oil through this technique is partial and it will not reach all the molecules in the reservoir. [3]

One of the characteristics of catalytic hydrogenation is the activation energy, which is the minimum energy necessary for a given chemical reaction to take a place in a given process (Eq. 2). From the Arrhenius equation, the constant of the velocity can be related to the temperature (Figure-3).

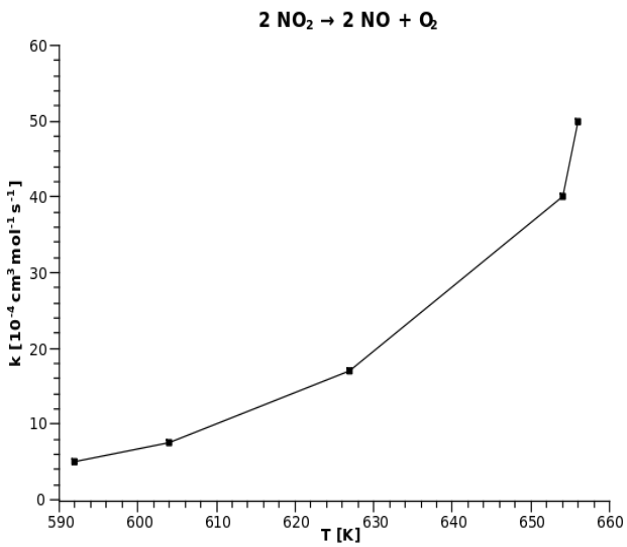

Figure-3. Arrhenius graph (Ratio of the rate constant to temperature). $k = Ae^{-Ea/RT}$

Equation. 2: Ratio of the rate to temperature.

This equation represents the study of elementary chemical reactions. Arrhenius discovered that during the process of a reaction the reactant molecules are triggered by mutual shock and there is a balance between the molecules in the basal state and the activities. [4]

3. COMPARATIVE ANALYSIS

The following analysis is carred out to studies developed for the determination of the nanofluid with better efficiency in the production of heavy hydrocarbons, in this case of study the objective oil was 17, 45 °API.

The core samples of Berea used were subjected to laboratory tests in a gas permeameter and a Helium porosimeter to obtain their permeability and viscosity values, the respective results and the properties of the cores are shown in Table-2.

Table-2. Measured dimensions and average petrophysical properties of cores samples at initial conditions. [5]

N°	Core	Length (cm)	Diameter (cm)	Apparent total volume (cm^3)	Porous volumen (cm^3)	Porosity (%)	Air permeability (md)	Liquid permeability (md)
1	W1	6,75	3,82	77,233	16	20,7	190,840	≈60
2	A1	7,6	3,8	86,193	17,5	20,3	185,526	≈60
3	A2	7,1	3,8	80,522	14	17,4	188,183	≈60
4	A3	6,6	3,8	74,852	14,5	19,4	181,199	≈60
5	B1	6,3	3,8	71,449	14,5	20,3	164,469	≈60
6	B2	6,5	3,8	73,717	14,5	19,7	157,835	≈60
7	B3	6,7	3,8	75,986	15,5	20,4	165,710	≈60
8	C1	6,4	3,8	72,583	13,8	19,0	165,267	≈60
9	C2	6,8	3,8	77,120	16	20,7	175,043	≈60
10	C3	6,4	3,8	72,583	15,8	21,8	182,712	≈60
11	D1	6,4	3,8	72,583	14,5	20,0	178,561	≈60
12	D2	6,6	3,8	74,852	18	24,0	178,408	≈60
13	D3	6,4	3,8	72,583	14,5	20,0	179,894	≈60
14	E1	6,93	3,82	79,235	16	20,2	195,909	≈60
15	E2	6,78	3,82	77,588	16	20,6	191,893	≈60
16	E3	6,90	3,82	78,914	16	20,3	207,644	≈60
17	F1	6,70	3,82	76,673	15,7	20,5	199,062	≈60
18	G1	6,65	3,82	76,089	15,5	20,4	191,795	≈60
19	G2	6,88	3,82	78,663	16,3	20,7	195,160	≈60
20	H1	6,87	3,82	78,571	14,5	18,5	200,410	≈60
21	H2	7,06	3,82	80,69	13	16,1	189,456	≈60

Another fundamental detail is the formation water (FW) used, which initially had a conductivity of 217, 4 ms/cm (160.000 ppm of salinity). Due to particles and / or sediments and the salinity of the same the formation water was submitted to filtration processes to remove suspended particles and it was also diluted with pure water in order to obtain treated formation water with conductivity of 46, 9 ms/cm (30.000 ppm of salinity).

In the preparation of the nanofluids the nanoparticles were dispersed in the treated formation water of 3% by weight of salinity and from that create each nanofluid with different concentrations by weight.

The laboratory experiments to analyze the behavior of the nanofluids were given by "coreflood" processes consisting of 5-stage flow. (Table-3). [5]

Stage 1: The percentage of oil recovery generated by the injection of each of the nanoparticles at different concentrations and by the injection of formation water in different cores is compared.

Stage 2: A mixture (ratio of 1:1) of the 2 nanoparticles that showed a better efficiency in the recovery percentage in Stage 1 is make and the percentage of oil recovery is determined due to the injection of the mixture of the 2 nanoparticles.

Stage 3: Work with the nanoparticle mixture to implement it in the oil recovery from other core samples.

Stage 4: The effect of water salinity on the selected nanoparticle was analyzed. The injection fluid was the formation water at 160.000 ppm of salinity and the oil production was determined, then the nanoparticles are dispersed in the formation water (same salinity) to determine their potential in the increase of oil recovery.

Stage 5: The performance of the selected nanofluid under reservoir conditions was analyzed.

In relation to the concentration of particles it is emphasized that a high concentration of them can cause inconvenience because at low rates of operation the particles can be retained and accumulate in the throat of the pores.

Table-3. Drainage processes and scenarios of enhanced oil recovery due to nanofluids. [5]

Designation			Drainage processes		Water/Nanofluid processes			Oil recovery	
Types of water/Nanofluid	**Conc. (%P)**	**Core**	**Sw_{irr}**	**So_i**	**VPI**	**Sw_f**	**So_r**	**Recovered oil (%)**	**Recovered oil differential (%)**
Stage 1									
AF 30.000 ppm	0	W1	0,219	0,781	2,406	0,474	0,526	32,640	-
SiO_2	0,01	A1	0,246	0,754	2,607	0,537	0,463	38,570	5,903
	0,05	A2	0,223	0,777	3,139	0,445	0,555	28,558	-4,082
	0,1	A3	0,285	0,715	2,974	0,501	0,499	30,312	-2,327
Al_2O_3	0,01	B1	0,191	0,809	3,098	0,452	0,548	32,324	-0,316
	0,05	B2	0,292	0,708	3,034	0,566	0,434	38,657	6,018
	0,1	B3	0,316	0,684	4,436	0,581	0,419	38,703	6,064
NiO	0,01	C1	0,253	0,747	3,339	0,495	0,505	32,317	-0,322
	0,05	C2	0,332	0,668	2,341	0,542	0,458	31,415	-1,189
	0,1	C3	0,206	0,794	2,372	0,494	0,507	36,199	3,560
TiO_2	0,01	D1	0,117	0,883	2,831	0,421	0,579	34,421	1,782
	0,05	D2	0,302	0,698	2,072	0,539	0,461	33,883	1,244
	0,1	D3	0,174	0,826	3,171	0,303	0,697	15.693	-16,947
Stage 2									
SiO2 /Al_2O_3	0,01	E1	0,283	0,717	3,033	0,571	0,429	40,163	7,524
	0,05	E2	0,317	0,683	2,341	0,606	0,394	42,286	9,647
	0,1	E3	0,220	0,780	2,879	0,414	0,586	24,854	-7,786
Stage 3									
AF 30.000 ppm	0	F1	0,231	0,769	2,189	0,474	0,526	31,647	-
SiO_2 /Al_2O_3	0,05		-	-	2,959	0,479	0,521	32,605	0,958
Stage 4									
AF 160.000 ppm	0	G1	0,272	0,728	2,435	0,450	0,550	24,469	-
SiO_2 /Al_2O_3	0,05	G2	0,298	0,702	3,317	0,505	0,496	29,364	4,895
Stage 5									
AF 160.000 ppm	0	H1	0,159	0,841	3,818	0,437	0,563	32,991	-
SiO_2 /Al_2O_3	0,05	H2	0,218	0,782	5,251	0,662	0,338	56,715	23,724
Conc.= concentration of nanofluids (%P); So_i = Initial oil saturation; So_r = Residual oil saturation; Sw_{irr} = Irreducible water saturation.									

The viscosity of the displaced fluid is an important parameter in the enhanced oil recovery, in which the viscosity increase of the displacement fluid improves the mobility ratio, which increases the displacement of the oil by increasing the sweep efficiency.

Table-4 shows the results of the emulsion before and after the addition of FW and nanoparticles, as indicated by Farah *et al* (2005).

Table-4. Experimental information of oil-water emulsion viscosity with nanoparticles at different concentrations. [5]

Concentration of nanoparticles (%P)	**Viscosity of emulsion** (cp)	**Change in viscosity (Δn)** (%)
Dynamic viscosity of the crude at 45°C		
0	65,81	0
Dynamic viscosity of the crude + 5 % P de FW		
0	71,3	8,3
Dynamic viscosity of the crude + 5 % P de FW y nanoparticles		
SiO_2		
0,01	69,25	5,23
0,05	79,99	21,55
0,1	76,69	16,53
Al_2O_3		
0,01	73,39	11,52
0,05	63,37	-3,71
0,1	69	4,85
NiO		
0,01	70,82	7,61
0,05	70,87	7,69
0,1	70,43	7,02
TiO_2		
0,01	70,83	7,63
0,05	72,39	10
0,1	61,8	-6,09
$\Delta\eta = \left[\frac{\eta_{oil(45°C)} - \eta_{oil+FW+nano)}}{\eta_{oil(45°C)}}\right] * 100$		
$\boldsymbol{\eta_{oil(45°C)}}$ = Dynamic viscosity of crude without nanofluid. $\boldsymbol{\eta_{oil(FW+nano)}}$ = Dynamic viscosity of crude oil with nanofluids (FW+ nanoparticles).		

Figure-4(a) shows the size (16, 65 um) of the drop of 5% by weight of FW in oil, but when injected with 0, 05% by weight of Al_2O_3 nanoparticles its size decreased by 38, 44% that is, at 10,25 um, which is reflected in Figure-4(b).

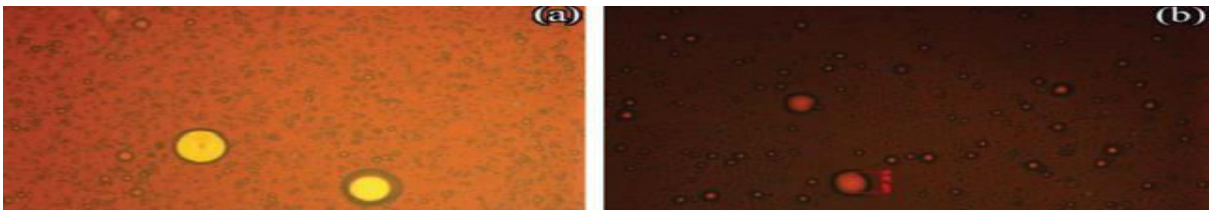

Figure-4. Analysis of crude oil particles: (a) Injection of 5% by weight of FW at 30.000ppm (b) injection of 5% by weight (4, 95% FW y 0, 05% by weight of Al2O3) nanofluid. Source: SPE 164106.

These indicate that Al_2O_3 nanoparticles tend to destabilize the water droplets, which facilitates the coalescence of the water droplets and thus reduces the viscosity of the emulsion. [5]

In the study developed by Osamah and company it is observed that TiO_2 at a concentration of 0, 1 %P provided a viscosity reduction of -6, 09 %, but in reference to the percentage of oil recovery was not favorable because it showed a differential of -16,947%.

A difference of Al_2O_3 at 0, 05 % by weight that showed favorable behavior in the change of viscosity and

in the oil recovered differential, being -3, 71% and 6, 018%.

In the study that was developed by Egolo and company, the experiments with oil of 22, 44 °API were carried out, also having a viscosity of 53,27735cp and a density of 0,9114g/cc @ 27 °C, in which they were used 9 type of nanoparticles for the enhanced oil recovery, which were diluted in treated formation water (30.000 ppm of salinity), the properties of the nanoparticles are shown in Table-5, the concentration of the nanoparticles was 3 g/L. [6]

Table-5. Nanoparticles properties. [6]

S. No	Nanoparticle	Particle size (mm)	Superficial area (m2/g)
1.	Aluminum Oxide (Al_2O_3)	40	~ 60
2.	Magnesium Oxide (MgO)	20	~ 50
3.	Iron Oxide (Fe_2O_3)	20 - 40	40 - 60
4.	Nickel Oxide (Ni_2O_3)	100	6
5.	Tin Oxide (SnO)	50 -70	10 - 30
6.	Zinc Oxide (ZnO)	10 - 30	90
7.	Zirconium Oxide (ZrO_2)	20 - 30	35
8.	Silicio Oxide treated with Silane (SiO2)	10 - 30	>400
9.	Hydrophobic Silicon Oxide	10 - 20	100 - 140

The behavior due to each nanoparticle in the oil recovery is shown in Table-6, where it is observed that the best oil recovery was given due to Al_2O_3.

In the study carried out by Nares and company in the improvement of heavy oil in the Gulf of Mexico through the use of transition metals, the density of said crude was altered from 12,5 to 23,5°API. [7]

Table-6. Oil recovery due to nanofluids. [6]

S. No	NANOPARTICLE	Formation Water	
		% Total Recovery	% Recovery due to Nanoparticles
1.	Experimental Control	35,0	0,0
2.	Aluminum Oxide (Al2O3)	40,0	5,0
3.	Magnesium Oxide (MgO)	32,5	-2,5
4.	Iron Oxide (Fe2O3)	35,0	0,0
5.	Nickel Oxide (Ni2O3)	36,7	1,7
6.	Zinc Oxide (ZnO)	30,8	-4,2
7.	Zirconium Oxide (ZrO2)	31,7	-3,3
8.	Tin Oxide (SnO)	31,7	-3,3
9.	Silicio Oxide treated with Silane (SiO2)	39,2	4,2

The physical and chemical properties of said oil were measured by ASTM standards; the values obtained are shown in Table-7.

Table-7. Physical and chemical properties of heavy crude oil. [7].

	Method	Content
Gravity API	**ASTM-D -287**	**12,5**
Water by distillation, Vol.%	ASTM-D- 4006	0,2
Viscosity, cst	ASTM-D- 445	
298,0 K		18.130
310,8 K		6.250
327,4 K		1.490

Due to the use of Al_2O_3 it was possible to alter the density of the oil simple already named, the results are shown in Table-8.

Table-8. Results of the comparison between comercial catalysis MoCoP/Al2O3 y MoWNiCoP/Al2O3. [7]

	Heavy crude oil	MoCoP/Al_2O_3	MoWNiCoP/Al $_2O_3$
Gravity API	12,5	21,2	23,5
Water by distillation, Vol.%	0,2	0,1	0,1
Viscosity, cst			
298.0 K	18.130	117	78,0
310.8 K	6.250	60,9	45,3
327.4 K	1.490	27,3	24,9

As it is possible to observe the Al_2O_3 it manages to obtain favorable behaviors or alterations for the production of heavy oil.

Table-9. Properties and characteristics average of the formation "U Superior", Ecuadorian East.

Sand	U Superior
°API	21
Total Rock Volume (acre-ft)	80.000,00
Porosity	15
Sw (%)	30
Reservoir temperature (°F)	185
Salinity (ppm ClNa)	15.000
Oil viscosity at Pi (cp)	7
Recovery factor	12,5

4. CONCLUSIONS

The use of nanoparticles in the production of heavy oil has good results improving the viscosity and/or recovery of oil such as nanoparticles of Al_2O_3 that managed to reduce the viscosity and increase the percentage of oil recovery, similar behavior had the TiO_2 that managed to reduce the viscosity but cannot improve he percentage of recovery, also taking into account the concentration of Al_2O_3 and TiO_2 which were 0, 05 y 0, 1% by weight respectively, the best economic feasibility is that of Al_2O_3 because it is used in lower concentration and the comparison in price between the 2 is relatively equal.

The use of Al_2O_3 nanoparticles in other studies altered the physical properties such as the density that by means of catalytic processes an increase 12, 5 to 23, 5 °API was obtained.

RECOMMENDATIONS

- Use filtered and diluted water formation as this presents greater efficiency in the behavior of enhanced oil recovery compared to untreated water.
- Do not use high concentrations of nanoparticles in the preparation of nanofluids because these at low rates of operation could cover the throat of the pores of the formation.
- Apply Al_2O_3 nanoparticles because it shows better effectiveness in viscosity reduction, °API increase and oil recovery, in addition to presenting prices similar to TiO_2 that presents only efficiency in viscosity reduction.
- Perform a laboratory test of nanoparticle injection of Al_2O_3 with 0, 05% by weight in formation water and then carry out pilot test in a determined well that produces the "U Superior" sand located in the Ecuadorian East, where they meet some similar characteristics to the studies analyzed.

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