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Retention Effect of Air Saturation and Concentration of Nanoparticles in porous Media as applied to Petroleum Engineering

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ABSTRACT

Nanoparticles are often used for the purpose of stabilizing emulsion or foam in enhanced oil recovery. It is also sometimes used in mobility control for displacement purposes. However, irrespective of the particular application, the nanoparticles have to be injected and transported through the porous media. The DLVO and colloidal filtration are two retention-causing theories considered in this research work. This paper investigates the retention parameters in five runs of laboratory experiments. Effect of air saturation and concentration of nanoparticles were investigated. Cabo-o-sil (fumed silica) of approximately 0.2 - 0.3 µm size was used. The laboratory experiments were based on injection nanoparticle dispersion through sandpack (porous media) using syringe pump, and were carried out under room temperature. PVC pipe filled with unconsolidated Johor sand was used to represent (porous media). Sandpack used has an internal diameter of 0.5 in and a length of 1 foot. The UV spectrometer was used to quantify the amount of particles retained in the porous media. The relevant parameters monitored include retention concentration, (R_{conc}), nanoparticle recovery (R_{NP}), arrival time ($t_{arrival}$), and monolayer coverage ($R_{monolayer}$); which were used to determine the favorability of the varied parameter. Experimental findings have shown that presence of air saturation causes more retention of nanoparticles, while lower injected nanoparticle concentration causes less retention; and vice-visa.

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INTRODUCTION

Despite the fact that a nanoparticle is capable of passing through the pore throat of the porous media due to its small size (µm), retention of certain percentage of the injected particles are still observed in porous medium Caldelas *et al*., (2011).

Nanoparticles have been employed in emulsion stabilization purposes, due to their small particle size (µm) and ability to withstand harsh conditions like high temperature and pressure Zhang *et al*., (2010). Some of the physiochemical theories guiding particle transport in a porous media are Derjaguin- Landau-Verwey-Overbeek (DLVO) theory and the colloidal filtration theory.

Derjaguin-Landau-Verwey-Overbeek (DLVO) was established since 1945, and it describes the force between charged particle surfaces interacting within a liquid medium. It combines the effects of the Van der Waals forces of attraction and electrostatic repulsion due to the so called double layers of counter ions. These forces also act between the particles and the sand grain. This is related to the charges carried by the particles and charges of the sand grain in which they are being transported, Torkzaban *et al*., (2007).

Colloidal filtration theory explains how the colloids strike and attach to the collector surface during its transport through porous media Torkzaban *et al*., (2007).The theory involves physiochemical filtration and straining. The filtration involves particle attachment to the pore surface of the medium and the latter involves the permanent physical trapping of particles in pore throat that are too narrow to allow passage due to strain and filtration effect.

Colloidal filtration theory and DLVO theory are two main principles that play a huge role in entrapment of nanoparticles in the porous media.

Nanoparticles have been very useful in EOR and reservoir engineering applications. It has successfully been used to stabilize oil-in-water and water-in-oil emulsion by modifying the surface coating Zhang *et al*., 2010. CO_2-in-water foam has successfully been created by the use of similar coated nanoparticles for stabilization purposes Espinosa *et al*., 2010. In Rodriguez *et al*., (2010), two different nanoparticles were used to conduct laboratory experiment. One formulation used 5-nm bare silica nanoparticles and the other, 5-nm

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silica nanoparticles coated with polyethylene glycol (PEG). The PEG coated nanoparticle can stabilize CO2-in-water foams and n-octane-in-water emulsion. It was then suggested that appropriate coating of nanoparticle surfaces can reduce the aggregation and retention of nanoparticles in a porous media. This is because the coating enhances transportability of nanoparticles through the porous media by reducing the aggregation. Caldelas *et al*., (2011), in his work, suggested that larger salinity increases nanoparticle retention lightly and delayed nanoparticles arrival. Velocity, residence time and sandpack length are three parameters which have a minor effect on nanoparticle retention. Temperature had marginal effect, with 2% points greater retention at 80 ^{o}C compared to 21 ^{o}C. Both surfaces were coated making bare silica to be successfully transported. This shows that surface coating can be used as a criterion for transporting particle in rock system studies.

Ryan *et al*., (2012), experimented by using fluorescent nanoparticles in a Nano fluidic flow cell packed with glass beads. After 40 PV of injection at a lower ionic strength condition (pH 7.0), fewer fluorescent nanoparticles were attached to the center of the flow cell, where the pore scale velocity is relatively higher.For a longer injection period (300 PV), more nanoparticles were attached to the centre of the flow cell, and particles were attached to both the upstream and downstream sides of the glass beads. Nanoparticles attached under a higher ionic strength condition (100 Mm NaCl, pH 7.0) were found to be mobilized when flushed with DI water. The mobilized particles were later reattached to some favorable sites.

The attachment efficiency factor was found to reduce with an increase in flow velocity in another experiment conducted by Caldelas *et al*., (2011). Boise sandstone and Texas cream limestone were crushed into single grains and sieved into narrow grain size fractions. In some cases, clay was added to the Boise sandstone samples. These grains were packed into (1 ft- 9 ft) slim tubes (ID = 0.93 cm) to create unconsolidated sandpack columns. The columns were injected with aqueous dispersion of silica nanoparticles (with and without surface coating) and flushed with brine.

The results obtained by Federico showed that larger salinity increased nanoparticle retention slightly and delayed nanoparticle arrival. Velocity residence time and sandpack length have minor effect on retention. Temperature has a marginal effect with 2 % point greater retention at 80 ^{o}C compared to 21 ^{o}C. Surface coated and bare silica nanoparticles were successfully transported, showing that surface coating might not a prerequisite for successful nanoparticle transport and rock system as studied in Caldelas *et al*., (2011).

Caldelas *et al*., 2011, also experimentally investigated the effect of the nanoparticle concentration on its retention using salt tolerant 3M. His results showed that air saturation contributes to particles' retention during their transport. This suggests that retention is affected by the air water interface present in the porous media. In other words, retention increases as air filled saturation increases in the sandpack.

MATERIALS AND METHODS

The methodology employed in this study is based on sandpack injection using unconsolidated sand collected from Johor Bahru as porous media (unconsolidated) and Nanoparticle dispersion as injection fluid.

Materials:

The sandpack contained sand grains within the range of 250-355 μm in size. The Sandpack model was used to replace the actual core. A 3 wt % brine was used to prepare the dispersion with nanoparticle of 200-300nm. Other materials used are 1 ft long PVC plastic sandpack with 0.5 inch diameter and kaolin. Equipment used includes UV spectrometer, vacuum pump and Syringe pump.

Methods:

A series of laboratory tests were conducted. The experimental procedure involves sand pack injection filled with unconsolidated sand. Initially, a calibration curve was plotted using different nanoparticle concentration and the absorbance was measured at a wavelength of 480 nm. The nanoparticle absorbance was measured using the UV spectrometer, which in turn is translated to the concentration using the calibration curve. The calibration curve is as shown in Figure 1, while the calibration curve data are shown in Table 1.

The sand pack porosity was then determined by weight using Equations 7 – 9. This was later saturated with 3 wt% brine and finally injected with the particle dispersion. A total of 5 experimental runs were performed and analyzed. It should be noted that in each case, the sand pack was pre-flushed with several pore volumes of 3wt% brine solution using the brine saturation setup in shown Figure 2 and also a post-flush with brine was also carried out. The BET measurements for the surface area of the sand and kaolin are shown in Table 2.

Air Saturation:

The air saturation was varied in Experiment 1 by running the vacuum pump for about 1 hour to suck out air from the sandpack. Sucking out air allowed me to achieve an-air free porous media in order to test for nanoparticle retention in absence of air. This was achieved by the aid of the vacuum saturation apparatus shown in Figure 3.

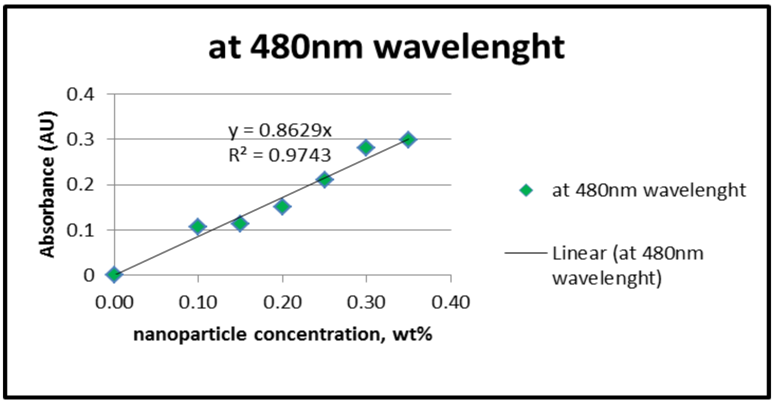

Fig. 1: Calibration curve obtained using the cab-o-sil nanoparticle dispersion.

Table 1: Calibration curve data.

% wt nanoparticle	Absorbance (AU)
0.00	0
0.10	0.107
0.15	0.113
0.20	0.151
0.25	0.211
0.30	0.282
0.35	0.299

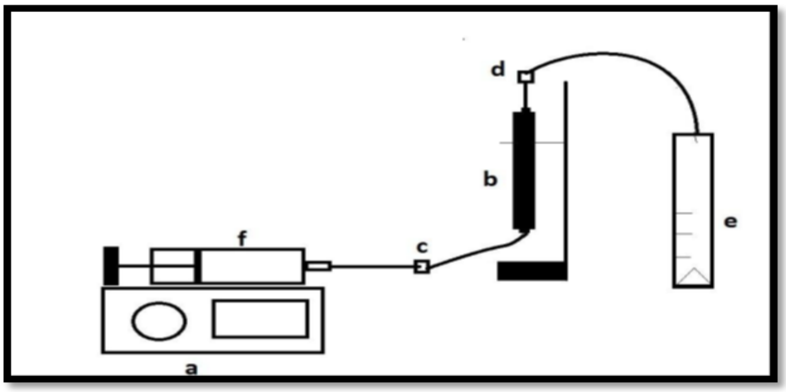

Fig. 2: Brine saturation apparatus (Setup 2) (a) syringe pump (b) sand pack (c) downstream valve (d) upstream valve (e) measuring cylinder.

Table 2: BET Measurement.

S/N	Specimen	Measurement (m^2/g)
1	Sand	2.04
2	Kaolin	14.51

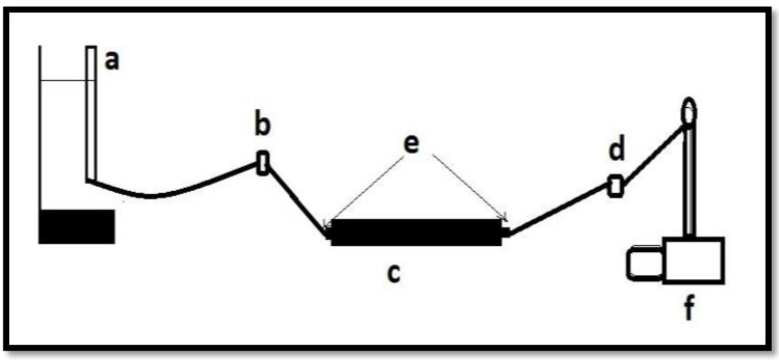

Fig. 3: Vacuum Saturation apparatus (Setup1) (a) graduated cylinder (b) downstream valve(c) sand pack (d) upstream valve (e) quick connectors (f) vacuum pump.

The pore volume of the sandpack was about 26.30 cm^3. The sandpack was saturated with 3 wt% brine after the vacuum saturation was carried out. This was subsequently injected with 3.3 PV, 0.2 wt% concentration nanoparticle dispersion (fumed silica) at 1cc/min rate using injection apparatus setup shown in Figure 4.

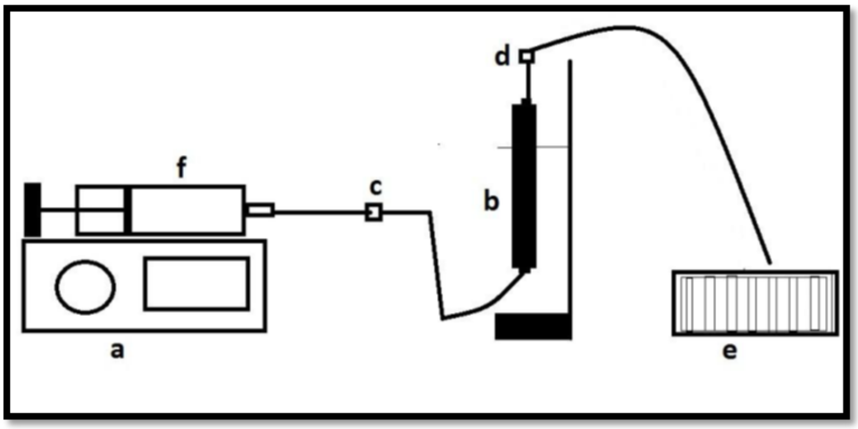

Fig. 4: Injection apparatus (Setup 3) (a) syringe pump (b) sand pack (c) downstream valve (d) upstream valve (e) fractional collector/set of test tubes in a rack.

The effluent was measured and the dimensionless concentration was plotted against PVI (i.e. the effluent history). In Experiment 2, the sandpack was brine saturated (rather than being vacuum saturated) in order to study the effect of presence of air on retention. About 3.5 PV of dispersion was injected, followed by brine postflush. All the experiments were conducted at room temperature. Such important parameters, as Retention concentration, (R_{conc}), nanoparticle Recovery (R_{NP}), Arrival Time ($t_{arrival}$), Monolayer Coverage ($R_{monolayer}$), were used to judge the NP retention in each case. The favorability pattern of the varied parameters was determined by using the aforementioned parameters. The equations describing these parameters are written out in Equations 1 – 9, Murphy, (2012).

Equations (Murphy, 2012)

R_{NP} = (Area under the effluent history curve / PV) [1]

$R_{conc} = (R_m/S_A)$ [2]

$R_m = ((1- R_{NP}) * PVI * C_{inj} * V_P)$ [3]

$R_{cap}= (t_{arrival} - 1)$ [4]

$R_{monolayer} = R_{conc} * 3 * 3^{0.5}/ (\pi * DP * \rho_P)$ [5]

$C_D = C_{effl}/ C_{inj}$ [6]

$\Phi_{brine}= W_{brine}/(1.02V_{bulk})$ [7]

$\Phi_{grain}= 1- (W_{sand}/(\rho_{sand}*V_{bulk})$ [8]

$\Phi_{air} = \Phi_{grain} - \Phi_{brine}$ [9]

d. Nanoparticle Concentration:

Experiments 3,4 and 5 were carried out with 0.1 wt%, 0.5 wt% and 0.05 wt% Nanoparticle concentrations respectively. The objective is to study the effect of NP dispersion concentration on the NP retention. The pore volume was 26.30cm^3. The sandpack for each experiment was brine saturated and subsequently injected with respective concentration of NP dispersion. The effluent was measured and relevant parameters were calculated and recorded. About 3.3 cm^3, 3.5 cm^3 and 3.45 cm^3 dispersions were injected in Experiments 3, 4 and 5 respectively. All of the experiments were followed by 3 wt% brine post-flush.

RESULTS AND DISCUSSIONS

Data obtained from the 5 runs of experiments performed, was used to compute other important parameters like R_{conc}, R_{NP}, $t_{arrival}$, and $R_{monolayer}$. The complete experimental results (data) are summarized in Table 3. Below are the results discussion of the air saturation and NP dispersion concentration effect.

Table 3: Result Summary of Experiments 1 to 5.

	Saturation Effect	Air saturation Effect	Concentration Effect	Concentration Effect	Concentration Effect
	(no air)	(with air)	0.1wt% conc	0.5 wt% conc	0.05 wt% conc
Experiments	1	2	3	4	5
V_P(cc)	26.306	26.306	26.306	26.306	26.306
Brine porosity(%)	37.75773	28.58155	28.58155	28.58155	28.58155
Sand type	100wt% sand	100wt% sand	100wt% sand	100wt% sand	100wt% sand
S_A (m^2)	160.956	160.956	153	153	154
q(cc/min)	1	1	1	1	1
PV (PVs)	26.3	26.3	26.3	26.3	26.3
C_{inj} (wt%)	0.2	0.2	0.1	0.5	0.05
Nanoparticle	cab-o-sil	cab-o-sil	cab-o-sil	cab-o-sil	cab-o-sil
R_{NP}(%)	72.72727	57.79048	93.71981	98.40833	86.12903
R_{conc}(mg/m^2)	29.41847	48.28988	3.725251	4.378602	3.557101
$R_{monolayer}$(%)	7.685854	12.61619	0.973257	1.143951	0.959499
Experimental setup	setup1 &3	setup2 &3	Setup 2&3	Setup 2&3	Setup 2&3
D_P(μm)	205-355	205-355	205-355	205-355	205-355
R_m (mg)	4.73508	7.772546	0.569963	0.669926	0.565579
$t_{arrival}$ (pv)	1.3	2.4	0.98	1.25	1.5
PVI	3.3	3.5	3.45	3.2	3.1
Parabolic base(pv)	4	3.7	5	4.82	4.5
Parabolic height	0.9	0.82	0.97	0.98	0.89

(a) Effect of air saturation on retention:

From the results of Experiment 1 and 2 shown in Table 3 and the effluent histories in Figure 5, the result suggests that that air saturation caused larger retention in the sandpack. Air filled porosities in Experiments 1 and 2 were 4 % and 13.2 % respectively. A significant difference was observed in the two results, which is due the the presence of air-water interface in the porous medium. The value of retention concentration in Experiment1 was 29.4mg/m^2 (with no air saturation) and that of Experiment 2 was 48.289mg/m^2, (with significant air saturation). The presence of air-water interface in Experiment 2 has led to much nanoparticles retention in the sandpack column. It can therefore be concluded that the presence of air saturation may be the major cause of the variation in retention, since all other conditions were kept same in both Experiments 1 and 2. Recall that the only exception was the quantity of air saturation presence. This result in conformance to the one obtained from previous works of Caldelas *et al*., (2011) and also in Murphy, (2012).

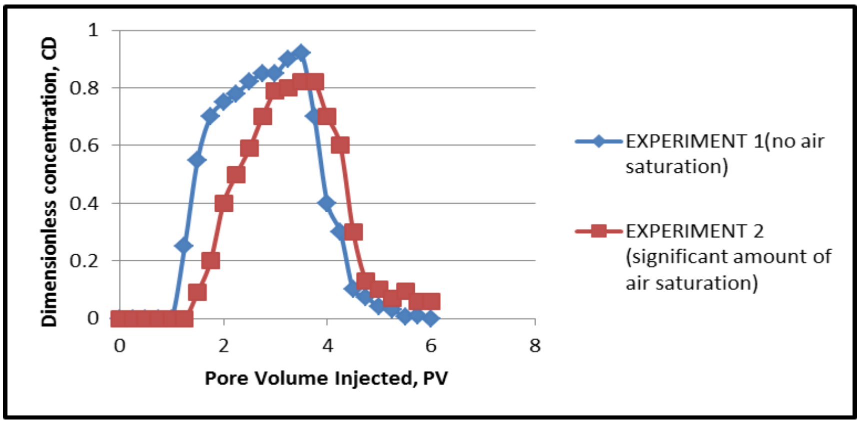

Fig. 5: Air Saturation Effluent Histories for experiments 1 & 2.

Furthermore a reasonable conclusion can also be drawn from the retention capacity, which is calculated using Equation [4]. This yields approximately twice the retention when compared with Experiment 1. This result also agrees with result in Murphy, (2012).

(b) Effect of nanoparticle concentration on retention:

The objective of this experiment is to see how the retention concentration R_{conc} is affected by different NP dispersion concentration of fumed silica. Therefore, the variable parameter here is the NP concentration. The retention concentration R_{conc} was plotted against the injected concentration in wt%, as shown in Figure 6.

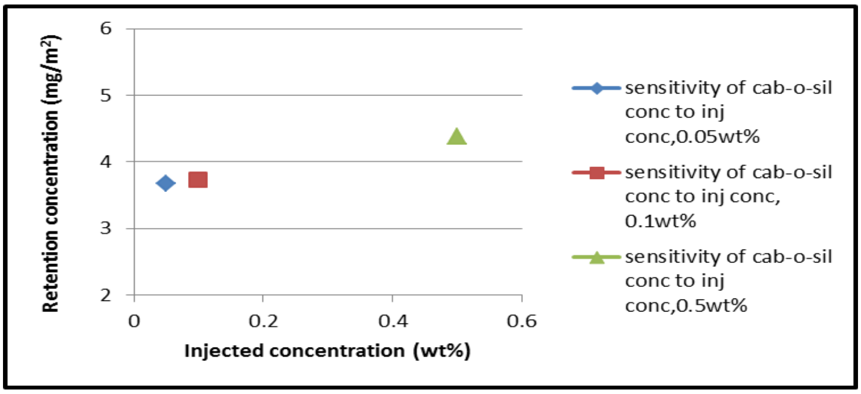

Fig. 6: Cab-o-sil retention concentrations to injected concentration.

It can be seen that, the higher concentration of 0.5wt% gave the highest retention, which is unfavourable and undesirable. However, the lower concentration of 0.1wt% gave a lower retention (3.7mg/m^2) while the lowest concentration of 0.05wt% which gave a much lower retention concentration of 3.6mg/m^2). This shows that the higher the nanoparticle concentration, the more the retention concentration.

The following suggestions can also be made from the effluent histories plots shown in Figures 7 – 9. When 0.5wt% concentration dispersion was injected, a time was reached when the dimensionless concentration reached unity and remained there until it starts to drop when nanoparticle injection stopped and brine post flush began. But, for the 0.1wt% concentration dispersion, its dimensionless concentration almost reached unity; while for 0.05 wt% concentration dispersion, its dimensionless concentration was still a bit far away from unity. These results suggest that the sandpack porous media has a threshold for retention capacity and above which no further retention can take place. Then at this point, the injected concentration will be equal to the effluent concentration ($C_{inj} = C_{eff}$); which is similar to the results obtained in the previous work of Caldelas *et al*., (2011) and Murphy, (2012).

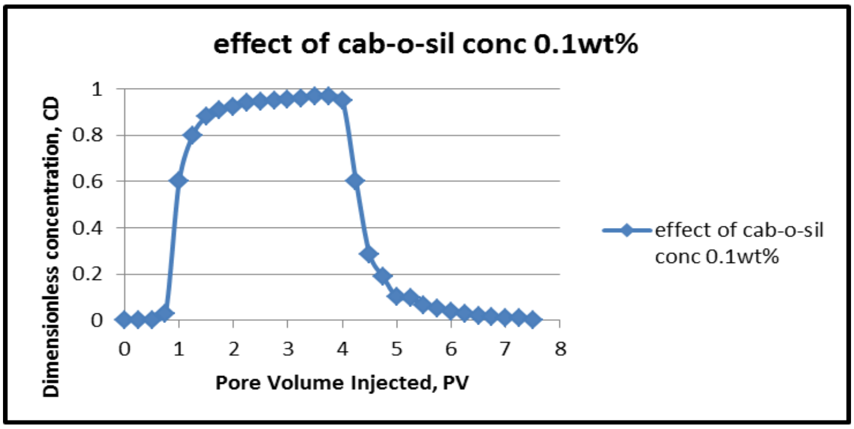

Fig. 7: Effluent history of Experiment 3 using cab-o-sil.

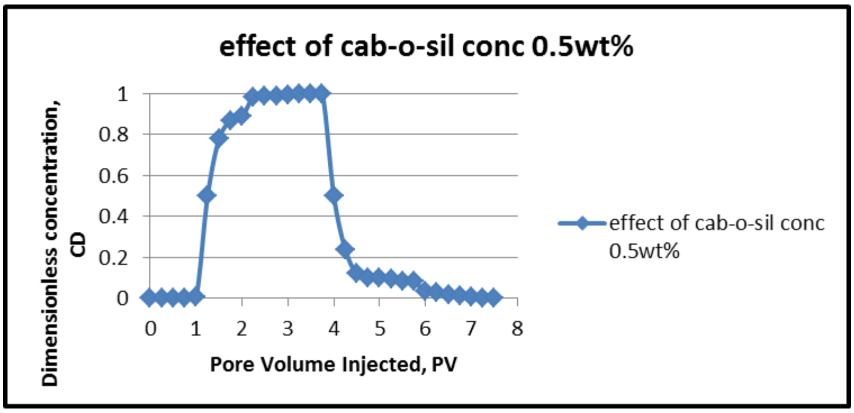

Fig. 8: Effluent history of Experiment 4 using cab-o-sil.

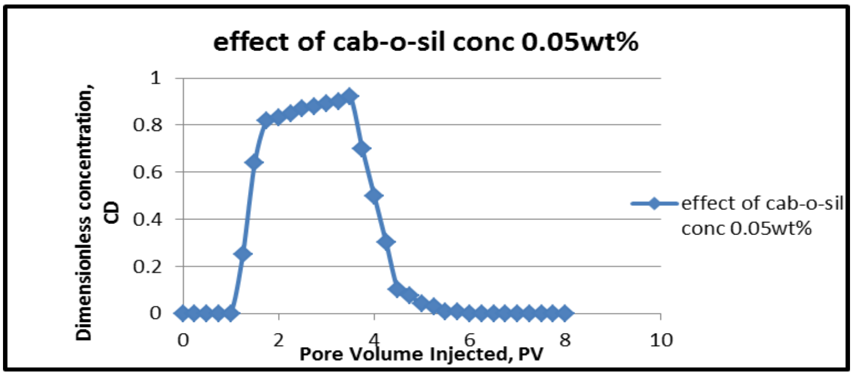

Fig. 9: Effluent history of Experiment 5 using cab-o-sil.

Conclusions:

Based on the conducted study, the following conclusions can be made. Air saturation has been confirmed to affect nanoparticle (cab-o-sil) retention, due to the air/water interface formed in the porous media, which definitely has affinity for attracting nanoparticle retention. This has been proven in Experiment 1 and 2. Moreover, for experiments with constant flow rate, it was seen that the dimensionless concentration of the effluent reaches unity. This suggests that the porous media has a fixed retention capacity, after which it is filled; there won't be no space for further retention. In other words, all additional PV injected will pass through the sand pack without any significant retention on the sand surface. The result obtained in this work has shown that fumed silica exhibit same retention characteristics as the nanopartilces used in Murphy and Caldelas.

Nomenclature:

$t_{arrival}$ = Nanoparticle arrival time
NP = Nanoparticles
R_{conc} =Retention concentration
R_{NP} =Nanoparticle recovery
$R_{monolayer}$=Monolayer coverage
PV =pore volume
S_A =Surface area of sand
Q =flowrate of injection
C_{in} =injected concentration
C_{eff} =effluent concentration
Dp =Sand particle diameter
R_m = Retained mass.
ρ = Nanoparticle density
R_{cap}= Retention Capacity

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