Quantification and Characterization of Interaction between Nanoparticles and Fines Migration

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Abstract— **Nanotechnology has potential in solving and managing several problems in the petroleum industry mainly in Enhanced Oil Recovery (EOR). Nanoparticles technology allows the combination of the high surface area available and the migrateable of formation fines can be fixate by using these particles with the action from the surface of material. In this paper, the experiment of quantification and characterization of interaction between nanoparticles and fines suspension was presented. Thu, the objective of this experiment is to investigate the stability and compatibility of nanoparticles used in reducing fines movement by performing zeta – potential, FESEM – EDX analysis. Another objective is to observe the effect from the migration of fines throughout the pore throat by injecting different types of nanofluid concentration in well formation. The glass beads was used in this experiment as a porous media (synthetic form) to mimic reservoir rocks where fine migration normally occurs. The glass beads are then treated with the different concentration of nanofluids where the bentonite will flow through the treated glass beads. Zeta – potential, FESEM – EDX analysis and adsorption rate were find to determine the most effective nanofluids during this experiment. As a result, MgO with concentration of 1.0% became the most effective nanoparticles that can used to prevent the fines migration because it have higher rate of adsorption.**

***Keywords*— Bentonite, EDX, FESEM, Fines suspension, Nanoparticles, Nanotechnology, Pore throat.**

I. INTRODUCTION

The use of nanotechnology in the petroleum industry is not a new thing. From the previous recent project, it has shown that nanotechnology has the potential in solving and managing several problems in the petroleum industry. In recent years, it already used in the oil and others sector for many times. For example, the rheological properties in drilling mud technology can be improve by using nano-sized clay particles and under extreme downhole conditions, nanomaterial can be used as a sensors for imaging processes [1]. One of the most general or common used of nanotechnology application is in the phase of Enhanced Oil Recovery (EOR). Due to the recent global rise in energy demand nowadays, EOR became an important choice in order to increase the productivity rate in the petroleum industry. There are a few types of nanoparticles that are commonly used including Aluminium Oxide, Zinc Oxide, Magnesium Oxide, Iron Oxide, Zirconium Oxide, Nickel Oxide, Tin Oxide and Silicon Oxide [2].

In the industry, many studies have been done in order to study the ways to control and prevent the migration or movement of fines in the reservoir formation and to avoid the concentrated of formation fines to form in the near wellbore region [3]. For example, the used of organic and inorganic clay control to minimizes the migration of fines in high-water-cut oil wells and to remove the formation of fines that can cause the pore plugging to occur in region around the wellbore, gravel packs and sand control screens for different downhole situations by using different acid systems [4]; [5]. Nanoparticles technology allows the combination of the high surface area available and the migrateable of formation fines can be fixate by using these particles with the action from the surface of material. To define formation fines, it is known as a loose solid particles appear in the formation of pore spaces sandstone with the size less than 37 microns [6]. This means the particles is small enough to migrate or move through the pore throat in the reservoir formation [7].In this research of quantification and characterization of interaction between nanoparticles and fines suspension, we use nanotechnology to enhanced oil recovery by reducing migration of fines to increase the well productivity.

In Oil and Gas Industry, the migration of fines through the pore throat in the reservoir is one of the common problem since a years. The uncontrolled fines migration through the reservoir will cause the accumulation of the fines in the pore throat hence cause the blockage in the reservoir formation which lead to the formation damage to occurs. When there has a formation damage in the reservoir formation, these scenario will prevent the hydrocarbon to migrate from the reservoir to the surface and cause the reduction in productivity. To overcome this problem, we used nanofluids that contain nanoparticles. This nanofluid will reveal the specific properties such as have a high tendency for adsorption and being a good applicants for injection into the near-wellbore region due to small sizes of nanoparticles. The purpose of this experiment is to investigate the stability and compatibility of nanoparticles used in reducing fines movement by performing zeta – potential, FESEM – EDX analysis [8]. Besides, it also to observe the effect from the migration of fines throughout the pore throat by injecting different types of nanofluids concentration in well formation.

II. METHODOLOGY

A. Materials

Bentonite, Distilled water, Glass beads, Magnesium oxide, Zinc oxide.

B. Selection of Nanoparticles.

There are two factor that was considered in selecting nanoparticles during this project which are due to its surface area and its ability in capturing and attaching fines particles [9] [10]. Hence, two different types of nanoparticles were selected known as Magnesium Oxide (MgO) and Zinc Oxide (ZnO). From previous studied, MgO have the smaller size and high total surface area compared to ZnO and our expected result is the percentage of absorption of MgO must be higher than ZnO [7].

C. Static Adsorption Test.

Then, the static adsorption test was carried out to test the ability of attachment between glass beads and different concentration of nanoparticles. This test was started by preparing 5% concentration of Magnesium Oxide (MgO). 5g of MgO was weighted and diluted with 100ml distilled water to get 5% concentration of mother solution. The diluted solution was sonicate at 30% amplitude (amp) for 25 minutes to make sure the diluted solution is well dispersed. The mother solution are then diluted into five different concentrations, 0.05%, 0.1%, 0.5%, 1.0% and 3.0%. All the nanofluids were sonicated again to ensure the solution is well dispersed.

0.5g of glass beads was weighted and transferred into five different of 20ml sample bottle and nanofluids (0.05%, 0.1%, 0.5%, 1.0%, 3.0%) was added into each sample bottle until the volume reached 20ml. The five samples bottle were labelled based on its different concentration. The samples was shaked slowly for 30 seconds to make sure all area of glass beads in contact with nanofluids. Shaking process are then repeated for five times with a gap of one hours each. The samples was leave for overnight. On the next day, the samples were dicanted with deionized water. The samples bottle was shake slowly and the process was repeated until the deionized water is not change its transparency which indicated no more unattached or excessive nanoparticles.

5 ml of each samples that contained glass beads was take out from the sample bottle for zeta – potential analysis. The remaining glass beads has been dried in 100°C humidity oven for 24 hours before it is ready for FESEM – EDX analysis. The whole process with the same steps was repeated for another nanoparticles, Zinc Oxide.

D. Characterization of Naniparticles.

Zeta – potential was carried out to test the stability of the samples [11]. For the zeta – potential analysis, 5 ml samples from the static adsorption test was used to analyze the zeta – potential values of samples for each five different concentrations. 20 ml of deionized water that contained 0.5g of glass beads without nanoparticles was used as a reference case. This analysis was started by transferred 5ml solution into the bottle test by using pipette. Then, 1ml of the reference samples solution was taken by using syringe and put in the zeta – potential's cell. The cell contain the samples must be observed first to ensure the sample in the cell are bubbles free [12]. After that, the cell are placed in the zeta – potential machine which then will be run and measure by using computer. All the data were displayed on the computer. The same methods was repeated for all different concentration of samples for both MgO and ZnO.

The remaining glass beads that has been dried was used for FESEM – EDX analysis to observe ability of attachment between different concentrations of nanoparticles and glass beads [13]. From the FESEM analysis, the image of attachment between nanoparticles and glass beads can be observe to study further about their attachment. This is verified by using EDX analysis where, the actual weight of the samples will be measured to prove the results from the FESEM analysis.

E. Soaking Test Experiment.

In the second phase, soaking test experiment was performed to determine the adsorption rate hence the effective nanoparticles can be identified. This test is started by measured 50g of glass beads and put in the glass column. The glass beads then was flushed by using deionized water until all the clear effluent was observed to ensure all the impurities was removed from the glass beads. After that, 50ml of deionized water was poured in the glass column containing glass beads and leave for overnight. On the next day, the deionized water was removed from the glass column. 0.25g of bentonite in 50ml deionized water was prepared and sonicate at 30amp for about 30 minutes to ensure the bentonite solution is well dispersed. The dispersed bentonite solution are then put in the 250ml separatory funnel. The separatory chamber are then open slowly to ensure the bentonite solution from the separatory funnel flow into the glass beads by gravitational forces. The bentonite solution will passed through the glass beads and filter paper. Bentonite particles trapped on the filter paper will be dried and the weight was measured. From the previous studied, the rate of adsorption was measured by using the formula in Equation 1 below [12]. All the steps was repeated by using sample that has been soaked with 0.05%, 0.1%, 0.5%, 1%, and 3% concentration of MgO nanofluid and ZnO nanofluid. Figure 1 below shown the experimental set up for soaking test experiment.

$$\% \text{ Adsorption} = \frac{\text{Initial weight of bentonite (g)} - \text{Final weight of bentonite (g)}}{\text{Initial weight of bentonite (g)}} x\ 100 \ldots\ldots (1)$$

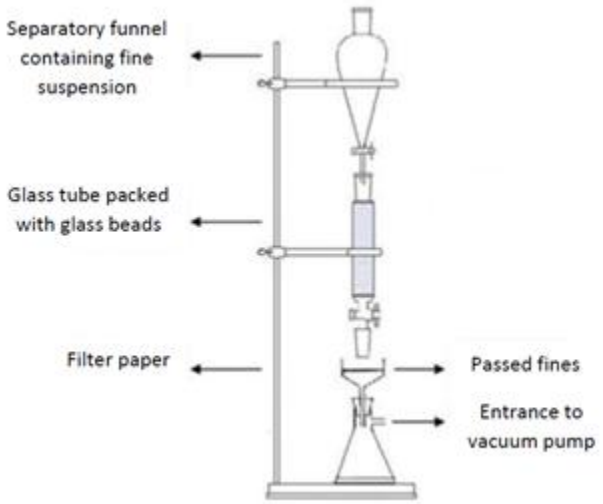


Figure 1 Experimental set up for soaking test experiment.

III. RESULTS AND DISCUSSION

A. Characterization of Nanoparticles.

Field emission scanning electron microscopy (FESEM – EDX) analysis was carried out to test stability and ability of attachment of the sample from static adsorption test with the raw glass beads as sample for reference [11] [14]. The results are shown in Figure 2 and Figure 3 respectively.

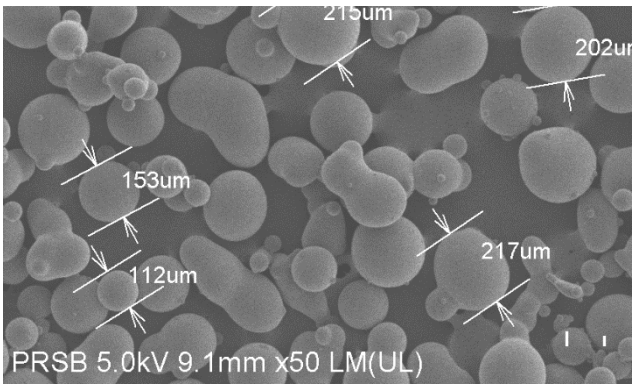


Figure 2 Raw glass beads with 50X Magnification.

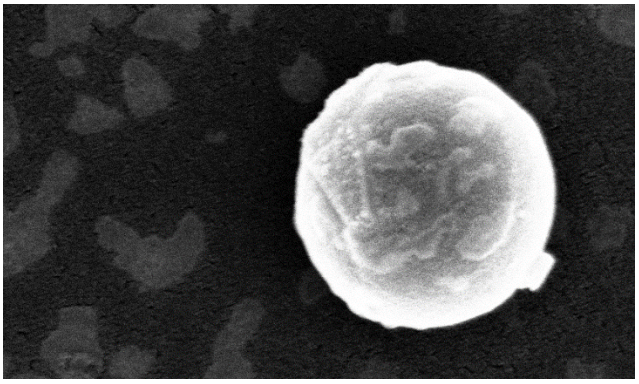
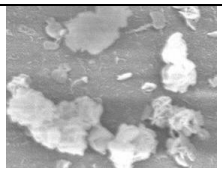
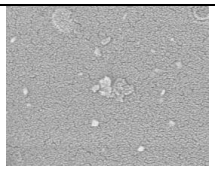
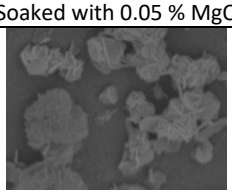
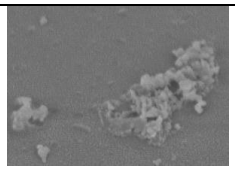
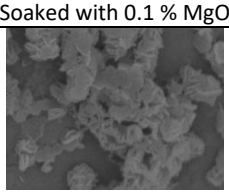
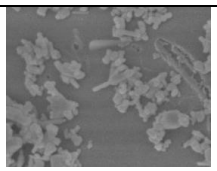
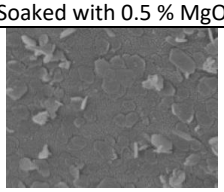
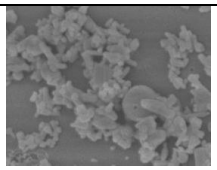
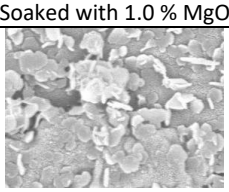
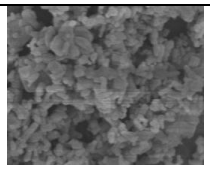


Figure 2 Raw glass beads with 50,000X Magnification.

Figure 2 and Figure 3 shows the FESEM images for the sample of raw glass beads without soaking with any nanoparticles for the magnification of 50X and 50,000X respectively. From the FESEM – EDX analysis, the average size of the glass beads is between 100 – 250 nm. The raw glass beads will further soak with the different concentration of nanoparticles (MgO and ZnO) to observe the ability of attachment between nanoparticles and glass beads. The result of glass beads soaked with nanoparticles were tabulated in the Table 1.

Table 1 Glass beads soaked with nanoparticles at 10,000X Magnification.

Magnesium Oxide (MgO)	Zinc Oxide (ZnO)
 Soaked with 0.05 % MgO	 Soaked with 0.05 % ZnO
 Soaked with 0.1 % MgO	 Soaked with 0.1 % ZnO
 Soaked with 0.5 % MgO	 Soaked with 0.5 % ZnO
 Soaked with 1.0 % MgO	 Soaked with 1.0 % ZnO
 Soaked with 3.0 % MgO	 Soaked with 3.0 % ZnO

From the Table 1, the FESEM analysis showed the attachment of nanoparticles to the glass beads was started at the concentration of 0.05% for both glass beads soaked with MgO and ZnO respectively. The attachment of nanoparticles for the glass beads soaked with MgO is nearly covered all the surface of the glass beads at the concentration of 0.5% compared to lower concentration at 0.05% and 0.1%. At the 1.0% concentration, the result showed that the nanoparticles are fully covered or attached around the surface of the glass beads and at the highest concentration which is 3.0%, the double layer of nanoparticles was formed surround the surface of the glass beads. The double layer occurred due to very high concentration of nanoparticles used to soak the glass beads.

On the right side in the Table 1, all the results were tabulated to show the attachment of different concentration of nanoparticles (ZnO) with glass beads. From the result, it showed that the ZnO are fully covered on the surface of glass beads at the concentration of 1.0%. Comparing to the three lowest concentration of ZnO, the attachment of ZnO at 0.1% is higher than 0.05% but it is lower compared to 0.5%. It seems that the situation at the highest concentration for both nanoparticles were same where, the double layer of attachment also occurred at the 3.0% concentration of ZnO eventhough the percentage of attachment for the ZnO is maybe slightly lower compared to MgO. Figure 4 and Figure 5 below shows the summarization from the FESEM analysis for both MgO and ZnO respectively. The red dotted line showed the line scale for nanoparticles to fully covered the glass beads. The fully attachment of MgO to the glass beads was started at the concentration of 1.0% whereas for the ZnO, it is started to fully attach the glass beads at the concentration of more than 1.0%.

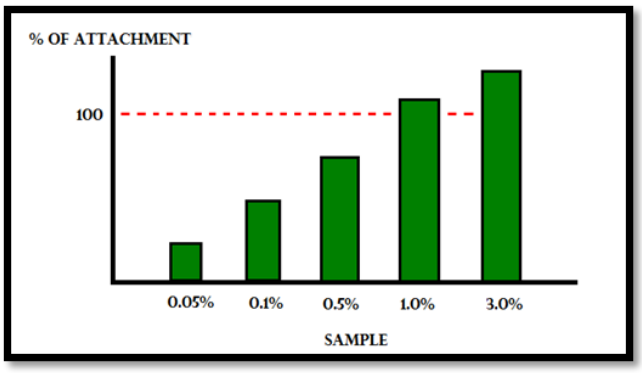


Figure 4 Percentage of Attachment between Glass Beads and MgO.

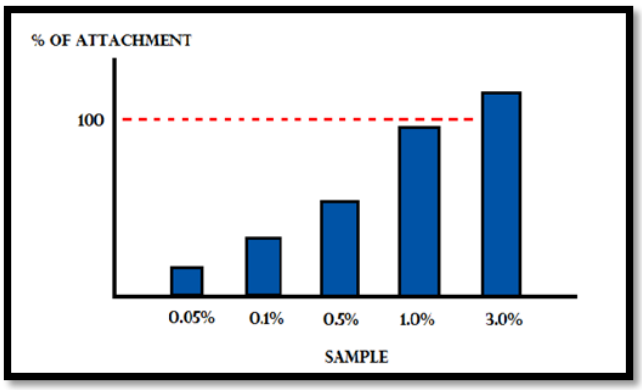


Figure 5 Percentage of Attachment between Glass Beads and ZnO.

The EDX analysis give the results in term of weight percent of silica particles for the reference case, soaked MgO and soaked ZnO [15]. The results presented in table 2, shows that the glass beads surface are successfully treated with nanoparticles.

Table 2 EDX Analysis of the Result.

Experiment	Wt.% (actual)

	0.05%	0.1%	0.5%	1.0%	3.0%
Reference case	11.6				
Soaked with Mgo	13.24	22.95	48.72	31.28	32.71
Soaked with Zno	35.36	33.77	79.32	68.18	81.19

The zeta – potential was used in this project to test the stability of suspension. Based on the literature studied, the stability of any suspension can be determined by measuring its zeta – potential and the suspension are considered as a stable when the zeta – potential value is below than -30mV and above than +30mV and vice versa [12]. The stable suspension cause the particles of the suspension tend to repel to each other. Different with unstable suspension, the particles of the suspension are tend to attract each other and promoted the particles to coagulate and flocculate [11]. The sample of bentonite diluted with distilled water, raw glass beads with distilled water and glass beads soaked with different concentration of MgO and ZnO were test in the zeta – potential equipment to study further about its stability condition. The sample of raw glass beads soaked in distilled water was used as a reference case and the bentonite was used as a fines in this project. All the zeta – potential results were tabulated in the Table 3 below.

Table 3 Zeta – Potential (mV) results for different cases.

Concentration (%)	**0.05 %**	**0.1 %**	**0.5 %**	**1.0 %**	**3.0 %**
Reference Case	-42.3	-42.3	-42.3	-42.3	-42.3
Soaked with MgO	-17.3	-4.5	-4.5	-16.1	-2.7
Soaked with ZnO	-25.6	-27.6	-26.1	-16.1	-2.7
Bentonite Solution (Fines)	-43.6	-43.6	-43.6	-43.6	-43.6

Based on the zeta – potential value in Table 3, the glass beads are unstable at all concentration when soaked with MgO. When the glass beads soaked with ZnO, the concentration at 0.05%, 1.0% and 3.0% become the most unstable compared to the concentration at 0.1% and 0.5%. Hence, regarding to this result, the concentration of the nanoparticles at 0.05%, 1.0% and 3.0% for both nanofluids were used in the soaking test represent the low, medium and high concentration to measure the rate of bentonite adsorption. The rate of bentonite adsorption will indicates the ability of the nanoparticles to prevent the fines movement through the glass beads which act as pore throat in the real reservoir conditions.

B. Soaking Test Experiment

The soaking test experiment was performed by using three selected concentrations which are 0.05%, 1.0% and 3.0%. These three different concentrations were selected based on the previous test, FESEM – EDX and zeta – potential analysis. Soaking test was run to identify and validate which type of nanofluids and at what concentration, the nanofluids become the most effective and efficient to prevent the migration of bentonite which act as fines through the glass beads. In this cases, the reference case was used by soaking the glass beads with distilled water without present of any nanofluids. From the soaking test, the efficiency of nanofluids can be determined by calculating the rate of adsorption using Equation 1 below and all the results were tabulated in Table 4.

$$\% \text{ Adsorption} = \frac{\text{Initial weight of bentonite (g)} - \text{Final weight of bentonite (g)}}{\text{Initial weight of bentonite (g)}} x\ 100 \ldots\ldots (1)$$

Table 4 Rate of adsorption for different cases.

Concentration (%)	**0.05 %**	**1.0 %**	**3.0 %**
Reference Case	20	20	20
Soaked with MgO	32	56	Blocked
Soaked with ZnO	28	48	Blocked

Based on the result above, the most effective of the nanofluids to prevent the migration of fines through the glass beads is at the concentration of 1.0% for both cases. The rate of adsorption at 1.0% is higher compared to the concentration at 0.05% for both types of nanofluids. This is because the zeta – potential value at concentration 1.0% is higher compared to the zeta – potential value at concentration 0.05%. This situation was verified by soaking test result when it is shown that the rate of adsorption at 1.0% is higher compared to the rate of adsorption at concentration of 0.05%.

When the percentage of concentration for both nanofluid were increased to 3.0%, the result from the soaking test shown that the rate of adsorption is undefined. This is due to the situation when the nanofluids cannot flow through the glass beads when the stopper is opened after the glass beads was soaked overnight with 3.0% concentration for both MgO and ZnO. So, it is considered the flow of bentonite solution was blocked. The very high concentration of nanofluids caused the agglomeration of nanoparticles occurred hence prevent the nanofluid from flowing down through the glass beads.

This situation was verified by previous test, FESEM analysis where the results show that the nanofluids form a layer by layer or called as double layer at concentration of 3.0% for both types of nanofluids. So, the MgO and ZnO at concentration of 3% is not efficient to use to control the fines migration in the pore throat because it can caused more problem when the pore is blocked by this nanoparticles. To be conclude, based on the static adsorption test, FESEM – EDX analysis, zeta – potential and soaking test, the most efficient and effective nanoparticles that can be used to control and prevent fines migration in the pore throat is by using MgO at the concentration of 1%.

IV. CONCLUSION

The aims of this study is to find the most effective nanoparticles to be used to reduce the movement of fines throughout the pore throats in the reservoir area by calculating the rate of adsorption of bentonite through the glass beads. Based on the static adsorption test, FESEM – EDX analysis, zeta – potential and soaking test experiment, Magnesium Oxide with the concentration of 1% became the most effective nanoparticles that can be used to prevent the movement of fines in the reservoir.

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