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Experimental investigation of the role of hexamethylenediamine in controlling fine migration in clay-rich sandstones

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This research investigated the effect of hexamethylenediamine (HMDA) on the fine migration phenomenon in sandstones containing kaolinite clay. Fine migration, which refers to the movement of fine particles during oil and gas extraction, can lead to decreased production efficiency and severe problems in hydrocarbon reservoirs. Therefore, understanding and controlling this phenomenon is of great importance. In this study, zeta potential (ζ) values were measured for clay and sand in different fluids with different concentrations of HMDA. The results showed that HMDA effectively reduces the ζ of both materials. This reduction is due to hydrogen bonds and electrostatic interactions between HMDA and the surface of clay and sand particles. Considering that the ζ is a measure of the tendency of particles to accumulate or disperse, its reduction can lead to an increase in the system's stability and a decrease in the possibility of fine migration. Wettability measurement and clay-rich core flooding tests were performed to verify the results. These experiments showed that HMDA at an optimal concentration of 1 wt% alters the wettability of sandstones from oil-wet to neutral and significantly reduces fine migration. Wettability alteration is caused by the neutralization of surface charges of particles and the increase of interactions between particles and fluid. XRD analyses also showed that the concentration of kaolin particles in the studied sandstone is 3.2%, with an original permeability of about 62 md. Conducting five core flooding experiments on these sandstone plugs confirmed that HMDA, due to ζ compensation, prevents fine migration and inhibits pressure from increasing during the core flooding experiment. Under controlled fine migration conditions (HMDA solution), the magnitude of core permeability remained almost constant at the level between 50 and 60 md. However, low salinity water reduced it to below 10, while pressure increased and fluctuated.

Keywords Fine migration, Zeta potential, Wettability, Hexamethylenediamine

Abbreviations

BET	Brunauer-Emmett-Teller
DLS	Dynamic Light Scattering
HMDA	Hexamethylenediamine
HS	High salinity

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LS	Low salinity
LSH	Low salinity-HMDA
MMT	montmorillonite
XRD	X-ray diffraction
XRF	X-ray Fluorescence

As one of the most essential energy sources in the world, the oil industry always faces many challenges^{1–4}. One of these challenges is the phenomenon of fine migration^{5–7}, which refers to the movement of fine particles in oil reservoirs⁸. This phenomenon can reduce the performance and efficiency of oil extraction and block pores in oil formations. Especially in sandstone reservoirs, the accumulation of fine particles can significantly decrease permeability and, ultimately, a drop in oil production^{9–13}.

The salinity and composition of fluids in reservoirs play an essential role, especially in particle behavior and movement^{14–18}. Studies have shown that an increase in salinity can lead to an increase in the electric charge on the surface of particles and a decrease in the repulsion between them^{19–22}. This can quickly help the movement of fine particles and the occurrence of fine migration. In contrast, water with low salinity can significantly increase fine migration^{23–26}. The reason for this is related to the electric charge of the particle surface. In low salinity conditions, the particle surface's electric charge decreases, reducing the repulsive force between the particles^{27–30}. In other words, in low salinity conditions, fine particles can be closer to each other, and their movement becomes more accessible, which ultimately leads to fine migration and pore blockage^{31–34}.

Several methods and techniques are used to prevent the phenomenon of fine migration. One of these methods is the use of surfactants. Surfactants can help reduce the surface tension of fluids and thus control the behavior of fine particles. By changing the surface characteristics of particles, these materials can increase the repulsion between them and prevent the accumulation and movement of fine particles^{35–38}.

Another method is using clay stabilizers, which improve the stability of fine particles during the oil extraction process^{39–44}. These materials can help strengthen the structure of minerals and prevent fine migration. In addition, additives such as HMDA have been considered an effective option in controlling the behavior of particles in different salinity conditions. HMDA can change the electrical charge of the particle surface by creating appropriate chemical bonds and helping to increase their stability. In recent years, research has been done on using nanoparticles and new technologies to control fine migration. For example, nanoparticles can be used as catalysts or additives in fluids to help improve oil recovery performance^{45–50}.

The phenomenon of fine migration, as one of the most critical challenges of the oil industry, has attracted the attention of researchers in recent years. Studies have shown that fine migration can significantly affect oil extraction performance. Many researchers investigated the effect of the movement of fine particles on the permeability of oil reservoirs and showed that the movement of fine particles can lead to a significant decrease in permeability^{51–54}. The salinity and composition of fluids are also known as influential factors in the behavior of particles in oil reservoirs. This can help to increase the movement of fine particles and the occurrence of fine migration^{55–58}.

This research aims to investigate salinity and HMDA's effect on the behavior of kaolinite and quartz particles in oil extraction processes. Core flooding, Brunauer-Emmett-Teller (BET), and zeta potential tests have been performed on different samples. The results of this research can help to understand the phenomenon of fine migration better and provide practical solutions in the management of oil resources.

Methodology

Materials

This section presents the materials required for the experiments performed in this research. The materials utilized include HMDA, sandstone core plugs rich in clays, and salts such as NaCl, CaCl₂, and MgCl₂ purchased from Merck.

HMDA

Hexamethylenediamine (HMDA), the primary chemical used in this study, was sourced from Merck, a reputable supplier known for high-quality chemicals. The HMDA utilized in this study was of 98% purity, ensuring high consistency in its chemical properties. Its chemical composition comprises a primary diamine functional group with the molecular formula C₆H₁₂N₂. The material is highly soluble in water, and its structure allows it to interact effectively with the clay particles in the sandstone samples, thus preventing fine migration.

Clay particles

The kaolinite needed in this research was obtained from Pars Ore Company. Kaolinite is one of the essential minerals in sandstone oil formations, primarily found in clay-rich sandstones. Due to its unique characteristics, such as crystalline structure and water absorption, this clay can significantly affect the lubrication behavior and fluid flow in these formations. As a tecto silicate mineral, kaolinite has solid and stable bonds that can lead to dense structures resistant to fluid flow^{59–61}.

Sand particles

Quartz is one of the most common and stable tectonic minerals, widely occurring in petroleum formations and sandstone sediments. This mineral is known by the chemical formula SiO₂, and due to its unique physical and chemical properties, it plays a vital role in geological and petroleum engineering processes⁶². Quartz was used as a reference material in batch experiments in this work. The quartz used in this work had a purity of 99%.

Minerals composition	Mass%	Chemical composition	Mass%
Quartz	93.1	SiO ₂	97.5
Kaolinite	4.7	Al ₂ O ₃	1.7
Muscovite	1	Fe ₂ O ₃	0.6
Cristobalite	0.8	MgO	0.06
Anorthite	0.4	TiO ₂	0.03

Table 1. Semi-quantitative mineral and chemical compositions of montmorillonite (MMT) obtained from XRD and XRF, respectively.

No	Length (cm)	Diameter (cm)	Porosity (Vol%)	Permeability (md)
1	11.1	3.81	22.95	61
2	11.2	3.81	22.91	61
3	10.9	3.81	22.88	61
4	11	3.81	22.97	61
5	11	3.81	22.95	61
6	11.2	3.81	22.95	61

Table 2. Specifications of core plugs used in this study.

Core plugs

In this research, core plugs taken from outcrop formations of oil fields related to southeast Iraq were used to perform core flooding tests, which contained 5% by weight of kaolinite. This combination has been chosen to simulate natural conditions in sandstone oil formations. XRD (X-ray Diffraction) and XRF (X-ray Fluorescence) tests effectively confirmed the presence of 5% by weight of kaolinite and showed that this mineral is significantly present in the structure of the samples. The results of these analyses are shown in Table 1.

Flooding of six core plug samples was used to study the effect of HMDA on fine migration in clay-rich sandstones, the specifications of which are given in Table 2. These studies provide the possibility to accurately evaluate the effect of kaolinite on the behavior of fluids and oil extraction processes.

Salts

In this experiment, different salts, including NaCl, CaCl₂, and MgCl₂•6 H₂O, were prepared with high concentrations from Merck. These salts were used to investigate the effect of salinity and ionic composition on fluid behavior in different conditions of oil formations. These salts were selected due to their particular characteristics in creating simulated conditions for core flooding experiments and evaluating their effects on fluid displacement and fine migration in kaolinite and quartz in sandstone formations.

Experimental procedure

Zeta potential measurement

Quartz and kaolinite particles were ground separately to reach micro sizes in this experiment. Then, two brine samples were prepared for ζ measurement: one with high salinity with a salt concentration of 250,000 mg/L and the other with low salinity with a concentration of 1,000 mg/L. This selection of different salinity conditions was done to investigate salinity's effect on ζ and particles' behavior in different environments.

In the test process, the milled particles were first suspended in both types of brines and stirred well to create a uniform distribution for 24 h. Then, the zeta electrophoresis device was used to measure the ζ . By applying an electric field, the movement of particles in the measuring liquid and the ζ were calculated, which indicated the electric charge of the surface of the particles and their interaction with the environment.

In addition, the particle size was also investigated using Dynamic Light Scattering (DLS). This device determines their size and distribution by analyzing the pattern of light scattered by suspended particles^{63,64}. The results of these tests can help better understand the behavior of quartz and kaolinite particles in different salinity conditions and their effect on oil extraction processes.

Brunauer-Emmett-Teller (BET) test

In this experiment, the BET method was used to investigate the adsorption of HMDA on clays and quartz. This experiment was designed to determine this material's specific surface and absorption capacity on two different types of minerals. At first, kaolinite and quartz samples were prepared separately and then treated with HMDA at a particular concentration under control conditions.

The BET method is specifically used to measure the specific surface area of solids and allows us to investigate the extent of HMDA adsorption on minerals. In this experiment, the changes in strain and the volume of absorbed gas were measured after exposing the samples to nitrogen gas. These data allow us to calculate the specific surface area and adsorption capacity of HMDA on clays and quartz.

The results obtained from this experiment can help to understand better the interaction of HMDA with minerals and its effect on the physical and chemical properties of kaolinite and quartz soils. This information is precious in preventing fine migration and optimizing oil extraction processes.

Core flooding test

The primary test in this research was core flooding, which was done using the Vinci device. The schematic of this device is given in Fig. 1. This experiment is designed to investigate the phenomenon of fine migration in sandstone oil formations containing clay. In this process, samples of core plugs containing 5% by weight of kaolinite were used to investigate the behavior of fluids and interactions between particles in different conditions.

At first, the samples of core plugs were prepared in certain conditions and accurately placed in the core holder of the Vinci machine. These samples included a mixture of quartz and kaolinite, which have essential characteristics in the behavior of fluids in petroleum formations. The Vinci device was prepared with precise pressure and temperature settings, and the initial pressure or confining pressure was applied to the samples to create suitable simulation conditions of the actual situation in oil tanks.

In the next step, fluid with a specific salinity (high or low salinity) was continuously injected into the samples. This experiment used different fluids, including high-salinity and low-salinity brine samples, to investigate different effects on the fine migration phenomenon. At the same time as the fluid was injected, the pressure was continuously measured along the length of the samples. These measurements allow researchers to observe changes in pressure over time in response to fluid injection.

In addition, the output fluid from the samples was collected and analyzed to investigate its composition and characteristics. This analysis includes measuring the concentration of fine particles and the chemical changes in the fluid. After the experiment, the collected data, including pressure, flow rate, and composition of the outlet fluid, were analyzed in detail. These analyses help to identify the flow patterns and behavior of particles in response to pressure and salinity changes and can lead to the identification and investigation of the fine migration phenomenon.

The results obtained from the core flooding test can provide valuable information about the interactions between minerals and fluids and help better understand particles' behavior in petroleum formations⁶⁵. This information is especially critical in optimizing oil extraction processes and resource management in sandstone formations. With a more detailed understanding of fine migration, it is possible to provide solutions to control and improve oil extraction performance.

Results and discussion

In this section, the results of the experiments are fully presented, and points obtained from experiments are thoroughly discussed. The composition of brines used in ζ and core flooding experiments is presented in Table 3.

In Table 3, HS denotes high salinity, LS is low salinity, and LSH is shortened to low salinity with HMDA.

Zeta potential test

A zeta potential experiment in the first phase was conducted to find the optimal concentration of HMDA to manipulate clay particles' surface charge in distilled water. As presented in Table 4, increasing HMDA concentration to 1 wt% strongly affects the surface charge of clay particles; however, further increments have no more impact on this parameter.

The ζ measurement test results show the effect of salinity and the addition of HMDA on kaolinite and quartz particles. As illustrated in Table 5, ζ for kaolinite particles in LS reached -40 mV and quartz -27 mV. These

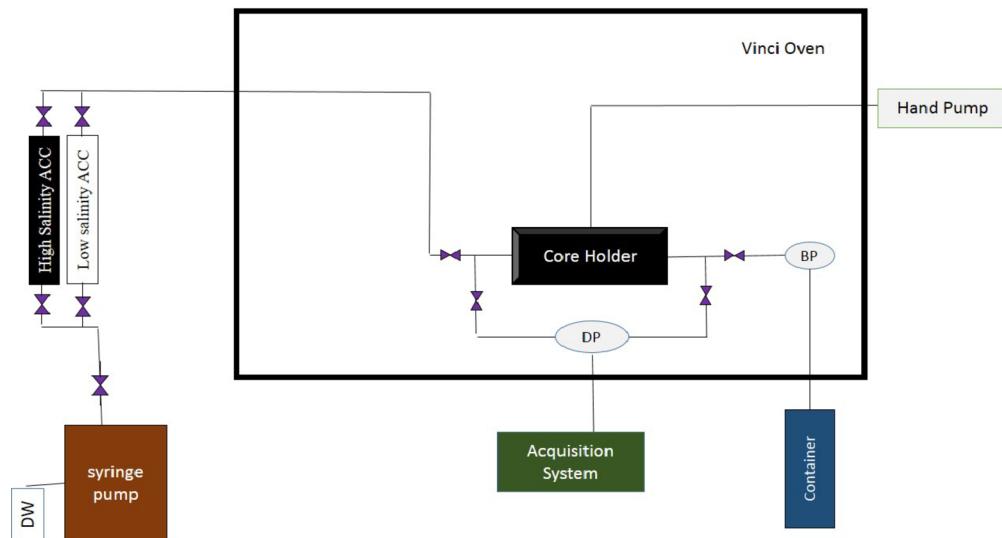


Fig. 1. The schematic of the Vinci device utilized for core flooding experiments.

Brine	HMDA (wt%)	NaCl (mg/L)	CaCl ₂ (mg/L)	MgCl ₂ (mg/L)	TDS (mg/L)
HS	0	180,000	60,000	10,000	250,000
LS	0	800	150	50	1000
LSH	1	800	150	50	1000

Table 3. The composition of different Brines used in this study.

HMDA wt%	0.25	0.5	0.75	1	2	4
Clay ζ (mv)	-36	-19	-12	-4	-3	-2

Table 4. Zeta potential measurements for kaolinite particles in various HMDA solutions.

Particle type	ζ in HS (mv)	ζ in LS (mv)	ζ in LSH (mv)
Quartz	-1	-27	-2
Kaolinite	-3	-40	-8

Table 5. ζ values measure for kaolinite and quartz particles in high and low-salinity Brine.

values indicate a high negative charge on the surface of the particles in LS conditions, which can lead to the repulsion between particles and thus increase the probability of movement of fine particles.

As the salinity increased to 250,000 mg/L, the ζ for both minerals decreased significantly to -3 mV for kaolinite and -1 mV for quartz. This decrease indicates a decrease in the electric charge of the surface of the particles, by which the repulsive force between the particles decreases. In HS, kaolinite and quartz particles can quickly get closer to each other, increasing the possibility of fine migration. These findings indicate that an increase in salinity can lead to an increase in the movement of fine particles in oil formations.

By adding HMDA to the samples at a concentration of 1%, the ζ for kaolinite and quartz decreased to -8 and -2 mV in brine. This significant decrease in ζ indicates the positive effect of HMDA in reducing the repulsion between particles. By reducing the repulsion, the particles can come closer together and form stable aggregates or flocs, which can help prevent fine particles from moving under certain conditions.

These results are significant for controlling fine migration in oil formations because fine particles in extraction fluids can decrease the efficiency and performance of oil extraction processes. Using HMDA makes it possible to effectively reduce particle repulsion and provide conditions where fine particles move less, thus creating more stability in the extraction process.

Finally, these investigations show that understanding the effect of salinity and additives such as HMDA on ζ and particle behavior in petroleum formations can help optimize oil extraction processes and resource management. Researchers and engineers can use this information to design effective strategies to control fine migration and improve oil extraction performance.

BET test results

BET test was performed to investigate kaolinite and quartz's surface area and pore characteristics, focusing on nitrogen adsorption as the adsorbed material. BET results related to kaolinite, quartz, and their modifications with HMDA are presented in Fig. 2A and B. The results of this experiment show that adsorption of HMDA alters the surface characteristics in kaolinite and quartz particles.

In the test results, the adsorption rate of nitrogen on virgin kaolinite was higher than that of quartz. In the test conditions, nitrogen adsorption on kaolinite reached 120 mg/g, and on quartz reached 80 mg/g. This difference indicates the higher surface area of kaolinite particles. This phenomenon can be attributed to kaolinite's surface structure and chemical characteristics.

In addition, the desorption test results also provide interesting information. In the desorption test, it was observed that 90% of nitrogen adsorbed on kaolinite was not quickly released after pressure reduction. However, only 60% of nitrogen adsorbed on quartz was released under these conditions.

After modifying the surface of kaolinite and quartz particles with HMDA, the surface characteristics for both particles were changed. The adsorption capacity for nitrogen gas in kaolinite particles dropped from 120 to 61 mg/gr-rock. However, it was reduced from 80 to 65 in quartz particles, indicating that HMDA reduces the specific surface capacity of kaolinite particles filling its pores.

These findings are significant in the field of management and control of the fine migration phenomenon. Considering that HMDA can act as an effective additive in oil extraction processes, its use in appropriate conditions can help to increase extraction efficiency and reduce the problems caused by the displacement of fine particles. As a result, understanding the adsorption and desorption behavior of HMDA on kaolinite and quartz can help optimize oil extraction methods and natural resource management.

Furthermore, the wettability study of sand slice surfaces in a water/oil system showed that adsorption of HMDA alters the sandstone surface from oil-wet to neutral status, presented in Fig. 3.

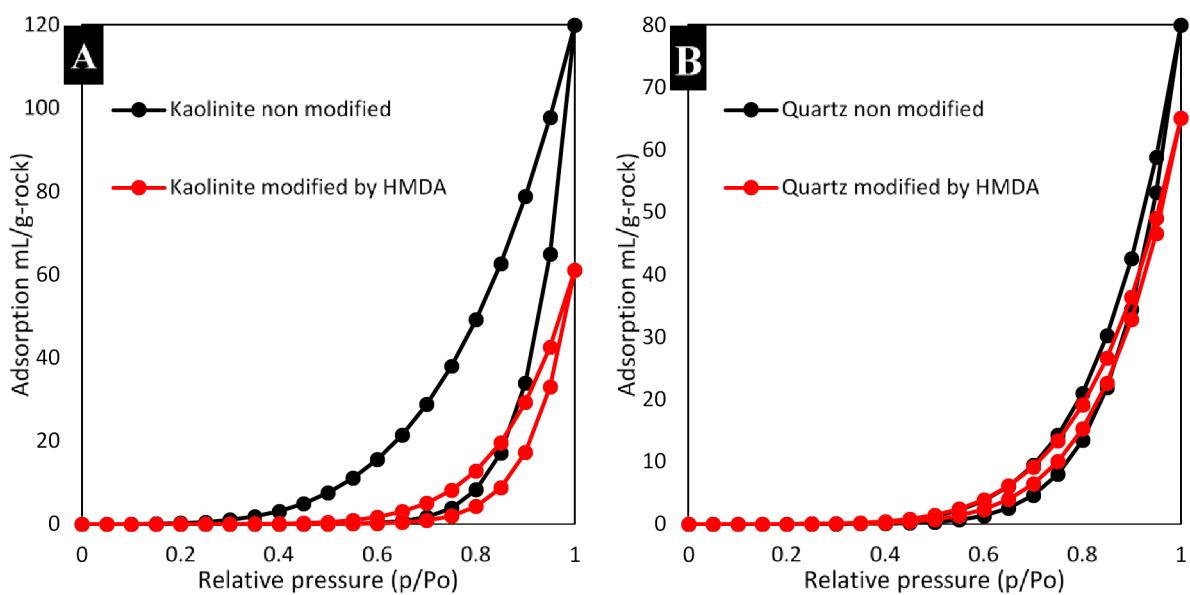


Fig. 2. BET results obtained from pure and modified (A) kaolinite and (B) quartz particles.

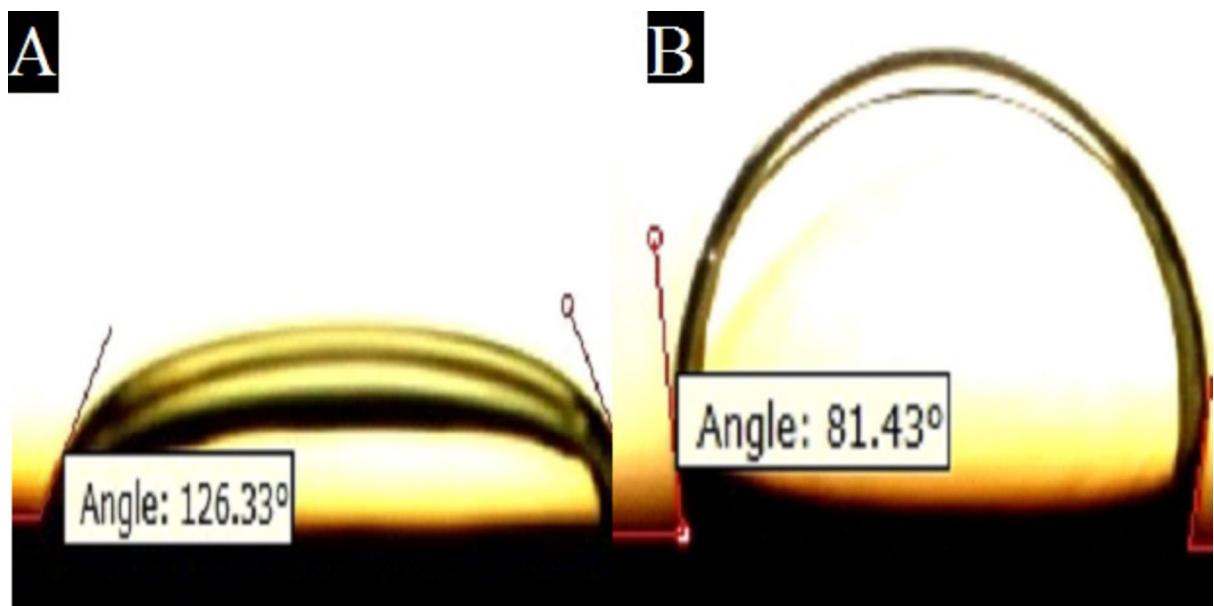


Fig. 3. Status of an oil droplet on an oil-aged sandstone rock in (A) formation brine (B) in HMDA solution after 48 h.

Results of core flooding tests

In this part, six core flooding tests were performed in the following order, and the graphs and explanations of each test are explained separately and in detail.

First experiment: injection of HS Brine and low injection rate

This experiment injected HS brine of 250,000 mg/L into the plug at a low injection rate of 0.1 mL/min, which resembles 1.3 ft/day, simulating areas far from the wellbore. As illustrated in the obtained data in Fig. 4, one of the key results of this experiment was that the injection pressure remained constant from the beginning to the end, and no significant increase was observed. This pressure stability indicates the absence of fine migration, which means that the fine particles did not move under these conditions, and the pore structure in the plug was maintained.

This situation can be due to the high electric charge on the surface of kaolinite and quartz particles in HS brine. In HS conditions, the particle surface's negative charge increases significantly, leading to repulsion between particles and preventing their movement. This phenomenon shows that, under appropriate conditions,

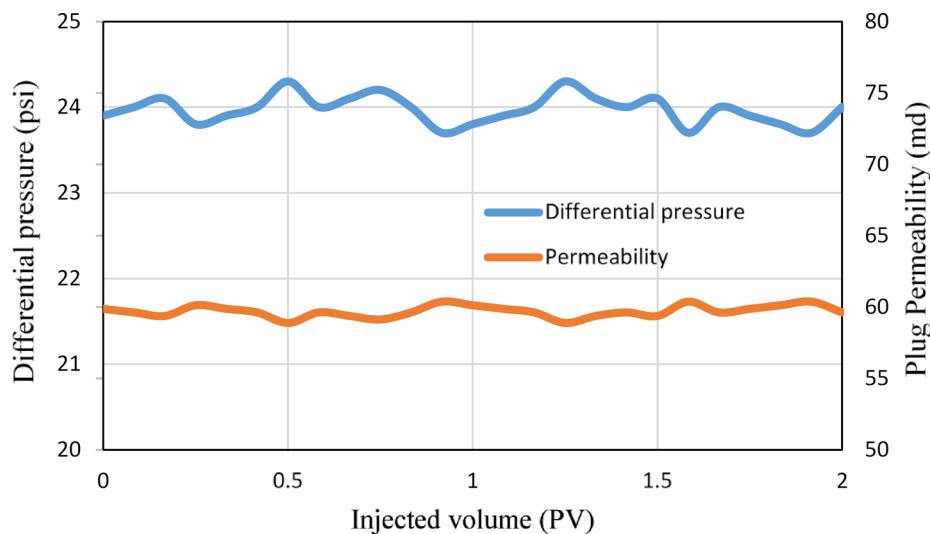


Fig. 4. Core flooding data related to injecting HS brine in 0.1 mL/min rate in plug#1.

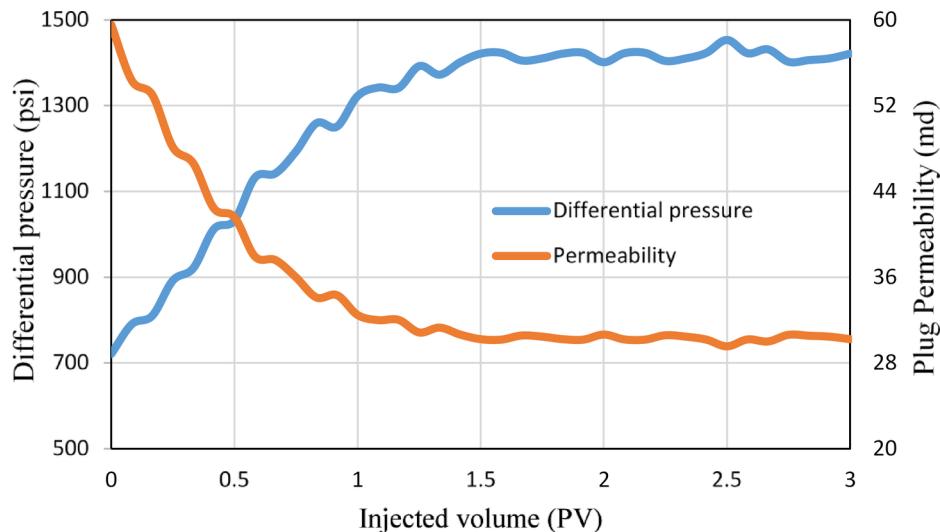


Fig. 5. Core flooding data obtained from flooding plug#2 by HS brine in 3 mL/min.

HS can be used to control the behavior of particles in oil formations, and it is especially effective in preventing the accumulation of fine particles.

The second experiment: injection of HS Brine and a high injection rate

This experiment injected HS brine into the plug at an injection rate of 3 mL/min, equal to 40 ft/day, simulating near-wellbore areas. Based on data illustrated in Fig. 5, the injection pressure started to increase, and the permeability of the plug reached 31 mD, while in the previous experiment, this value was 60 mD. This significant decrease in permeability indicates the positive effect of a higher injection rate on the movement of particles and their behavior in HS.

This phenomenon can be because, with the increase of the injection rate, the drag force on the particles increases, which can lead to the movement of fine particles. However, at the same time, due to the HS, the electric charge of the surface of the particles is such that it limits their movement. As a result, the balance between drag force and electrical repulsion in these conditions can decrease permeability.

Third experiment: injection of LS Brine and low injection rate

LS was injected into the plug, and in this condition, fine migration occurred, and the permeability decreased to 13 mD. According to Fig. 6, due to the lower electric charge on the surface of the particles, the repulsion between them is reduced, and this causes the tiny particles to move quickly and create blockages in the pores.

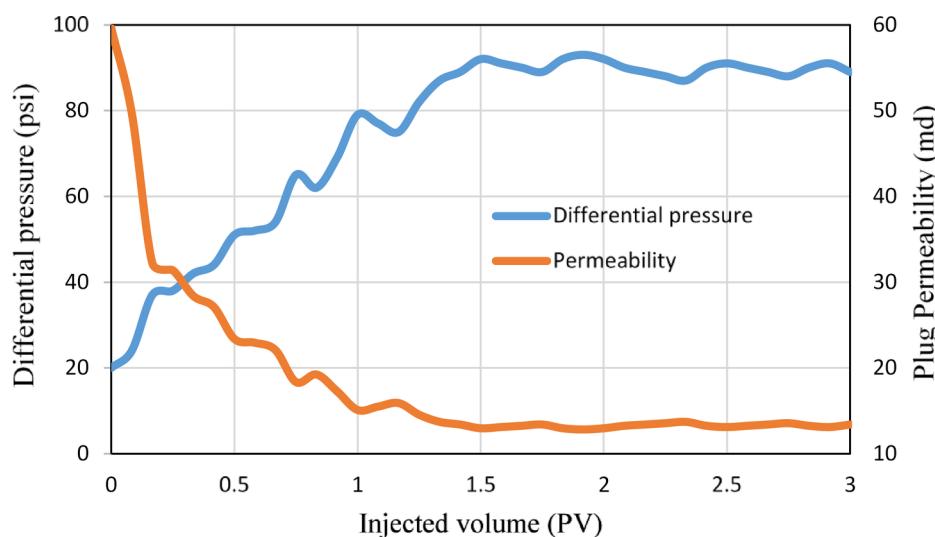


Fig. 6. Core flooding data obtained from injecting LS in plug#3 in low rate condition.

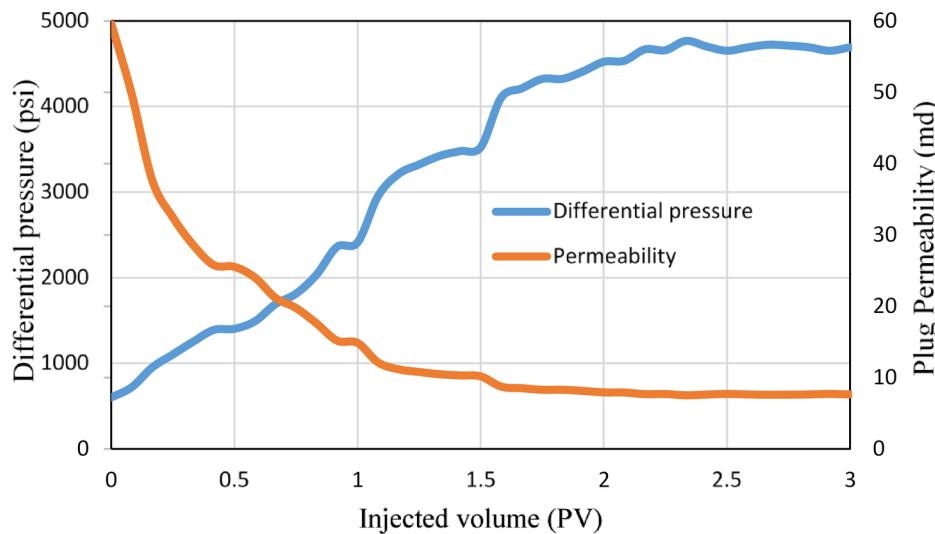


Fig. 7. Core flooding data obtained from injecting LS brine in plug#3 in high-rate condition.

These findings show insufficient repulsive force between particles in LS, which can lead to particle accumulation and permeability reduction. Additionally, these results point to the importance of salinity control in petroleum formations, as LS can significantly affect oil recovery efficiency.

Fourth experiment: injection of LS Brine and high injection rate

In this experiment, LS brine was injected into plug#4 at a high injection rate, and the permeability drop was strongly noticeable and reached seven mD. Based on Fig. 7, the sharp decrease in permeability indicates the negative effect of LS and high injection rate on particle movement and fine migration.

In this case, due to insufficient repulsion between particles in LS, the drag force caused by the high injection rate can lead to the displacement of fine particles and severe blockage of pores. These findings point to the importance of paying attention to the environmental conditions and the behavior of particles in oil extraction processes and show the necessity of controlling the injection rate in LS.

Fifth experiment: injection of LSH Brine at a low injection rate

In this experiment, LS brine containing one wt% HMDA was injected into the plug at a low injection rate. Based on data illustrated in Fig. 8, permeability decreased to only 52 mD, which does not indicate a noticeable decrease. These results indicate that HMDA, as an effective additive, can increase the stability of particles and prevent the displacement of fine particles.

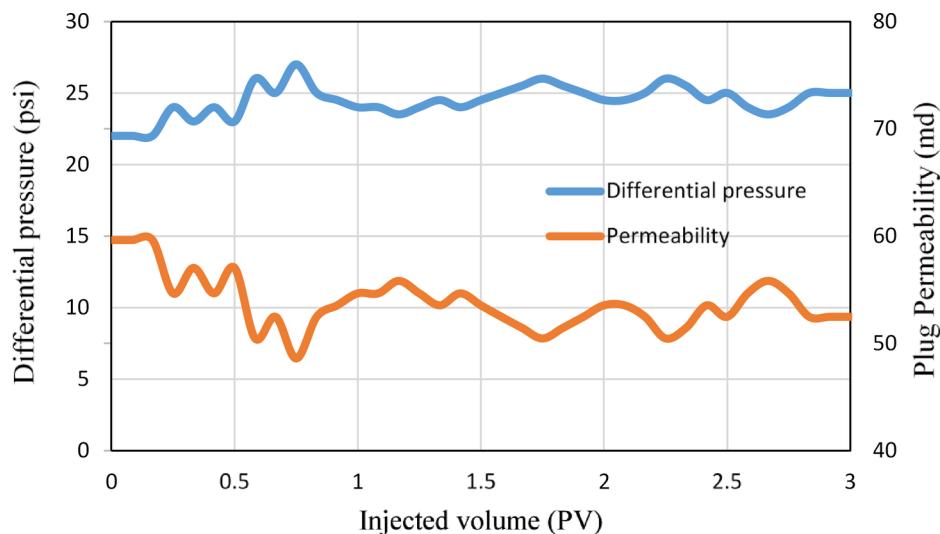


Fig. 8. Core flooding data obtained from injecting LSH into plug#5 with a low rate.

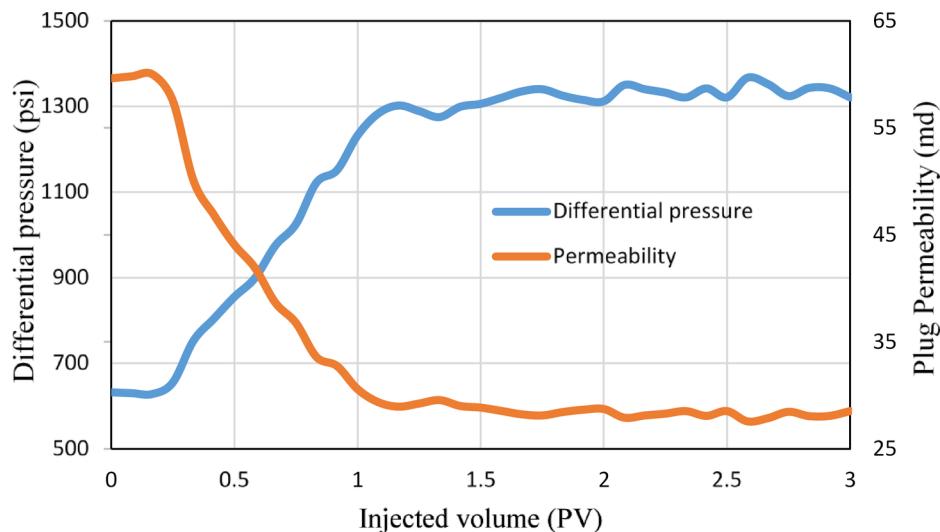


Fig. 9. Core flooding data obtained from injecting LSH into plug#6 with the high rate.

By creating chemical and physical bonds with kaolinite and quartz particles, HMDA can change their surface's electric charge and help increase the repulsion between particles. These findings show that adding HMDA can be an effective strategy to control fine migration in oil formations.

Experiment six: injection of LSH at a high injection rate

LSH was injected at a high injection rate in this experiment, and the obtained data are plotted in Fig. 9. In this state, permeability decreased from 60 mD to 27 mD. This sharp decrease in permeability was caused by fine detachment due to the high drag force.

The results of this experiment show the challenges that can arise in low salinity conditions and high injection rates. In this situation, the drag force can overcome the electrical repulsion and lead to the separation of fine particles. These findings emphasize that the use of HMDA should be carefully controlled, and environmental conditions should be considered to prevent problems caused by fine detachment.

In general, the results of these tests show the significant effect of salinity and injection rate on particle behavior and fine migration phenomenon in oil formations. Also, using HMDA as an additive can help improve permeability performance and stability in different conditions. As a result, a detailed understanding of particle behavior under different salinity conditions and injection rates helps to optimize oil extraction processes and natural resource management.

Limitations of the study

While the findings presented in this study offer valuable insights into the effect of hexamethylenediamine (HMDA) on fine migration in sandstone reservoirs, several limitations must be acknowledged:

1. **Single Fluid System:** The study primarily focuses on a single fluid system to investigate the effects of HMDA on zeta potential, wettability alteration, and fine migration. However, multiple fluids are present in real-world reservoir conditions, and their interactions with the additive may differ. The effects of HMDA on fine migration in more complex fluid environments, such as those containing varying salinity or different surfactants, have not been explored in this study.
2. **Environmental and Economic Considerations:** Although HMDA shows promise in controlling fine migration, the potential environmental impact and economic feasibility of using HMDA as an additive in large-scale operations remains to be assessed. Future studies should consider the environmental safety and cost-effectiveness of HMDA to ensure its practicality in real-world applications.
3. **Scale-Up:** While laboratory-scale experiments provide valuable insights, there are significant differences between laboratory-scale and field-scale operations. The results observed in core flooding tests may not directly translate to large-scale reservoir conditions due to factors such as heterogeneity of the reservoir, fluid flow dynamics, and the scale of fine migration. Future studies should aim to validate these findings in larger-scale field tests to ensure their applicability in real-world scenarios.

Conclusion

This research investigated the effect of salinity and additives on the behavior of kaolinite and quartz particles in oil extraction processes using core flooding, BET, and zeta potential tests. The results clearly show the significant effects of salinity conditions and injection rate on oil formations' fine migration and permeability performance.

The results of core flooding tests showed that in HS and low injection rate, the magnitude of permeability barely reduced from 60 to 50 md, which indicated that fine migration did not occur. In conditions of low salinity and high injection rate, fine migration led to a sharp decrease in permeability from 60 to 4 mD, while pressure increased with fluctuation. However, adding HMDA to LS brine and the injection rate prevented fine migration and reduced the permeability from 60 to 52 mD.

In the BET test, it was also observed that HMDA is effectively adsorbed on kaolinite and quartz, which can help improve the performance of extraction processes and control particle behavior. These findings suggest that HMDA can act as an effective additive in various salinity conditions and help prevent fine particle migration and pore plugging in petroleum formations.

This research shows the importance of understanding the behavior of particles in different conditions in oil extraction processes. The obtained results can help engineers and researchers design and optimize extraction processes and provide solutions for better control and management of oil resources. Considering the challenges in the oil industry, this information can be used as a basis for future research and the development of new solutions for managing and exploiting energy resources.

Data availability

Data will be made available on academic request from the corresponding author.Khaled HeratiHarat University, AfghanistanKh.h1992huni@gmail.com.

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Declarations

Competing interests

The authors declare no competing interests.

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