



Synthesis of Manganese Dioxide (MnO₂) Nanostructures Using a Modified Chemical Method as a Fluid Loss Control Additive for Water-Based Drilling Fluids (WBDF)

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تخليق هياكل نانوية من ثاني أكسيد المنغنيز (MnO₂) باستخدام طريقة كيميائية معدلة كمضاف للتحكم في فقد السوائل في سوائل الحفر القائمة على الماء (WBDF)

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ABSTRACT

Background:

Water-based drilling fluids (WBDFs) remain the most widely used drilling fluids because they are less expensive and more environmentally acceptable than oil-based and synthetic-based fluids. Their performance depends heavily on rheological and filtration characteristics, which are highly sensitive to the chemical additives incorporated into the mud. This study explores the influence of manganese dioxide (MnO₂) nanoparticles on the rheological behavior and filtration performance of WBDF.

Materials and Methods:

MnO₂ nanostructures were synthesized using a modified chemical route and characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM) to confirm their crystalline phase, bonding structure, and morphology. WBDF samples were then prepared with and without MnO₂ nanoparticles. Key parameters, including plastic viscosity, yield point, density, filtrate volume, and mud cake thickness, were measured to assess the effect of nanoparticle incorporation.

Results:

XRD analysis confirmed the formation of crystalline MnO₂, while FTIR spectra revealed strong Mn–O vibrations characteristic of MnO₂ structures. SEM imaging showed well-defined nanostructures suitable for fluid modification. Compared with the base WBDF, MnO₂ enhanced fluids demonstrated improved rheological stability, higher yield point and viscosity consistency, and a notable reduction in fluid loss. Additionally, mud cakes formed in the presence of MnO₂ exhibited reduced thickness and signs of structural modification, indicating improved sealing efficiency.

Conclusion:

The addition of MnO₂ nanoparticles significantly enhances the rheological and filtration properties of WBDF. These improvements suggest that MnO₂-modified WBDF can effectively minimize filtrate invasion into formations and provide more stable drilling performance.

Keywords: Water-based drilling fluid (WBDF), manganese dioxide nanoparticles (MnO₂), rheological properties, filtration control, plastic viscosity, yield point, mud cake thickness.



INTRODUCTION

Most drilling operations, such as coal mining and hydrocarbon exploitation, now rely on drilling fluid, often known as drilling mud. Its essential functions include bringing cuttings to the surface from the borehole, protecting the formations from damage, supporting the wellbore, lubricating drill pipes, preventing corrosion, and sealing off permeable formations with a fine, low-permeability filter cake [1], [2]. It is common practice to classify drilling fluids as either water-based (WBDF), oil-based (OBF), or gas-based (GBF) systems, depending on the base fluid type. In these complicated systems, there are three main parts: the base fluid, which is continuous and makes up most of the volume; the non-reactive solids, such as drill cuttings, weighting agents, and lost circulation agents; and the reactive part, which changes the physicochemical properties of the drilling fluid, and comprised of additives or treatment agents [3].

WBDFs are adopted extensively in the whole world, as they have both economic and environmental benefits [4]. Nevertheless, WBDFs are also able to dissolve salt particles and this leads to changes in density and destabilisation of clays, especially in shale formations. Fluid loss gives rise to the invasion of the geological formation by the drilling fluid rather than recovering via the annulus. It can occur both in the case of static (e.g., tripping) and dynamic (circulating) drilling [5]. This kind of invasion may cause damage to the formation as well as cause severe operational issues in the oil and gas wells. The solid remains that are deposited upon the borehole wall constitute a mud cake the thickness of which is directly related to the torque, drilling time, and the cost of drilling [6]. In high permeability formations, the pressure difference between the mud column and the formation may lead to different sticking of the drill pipe in a thick mud cake. To alleviate this, fluid loss control additives are added to the drilling fluids in order to create a protective low-permeability and thin coating around the wellbore [7].

Lignin, asphaltite and bentonite, as typical fluid loss control agents have been used in the past [8]. Nevertheless, most of these additives are restricted such as lack of chemical stability, low thermal resistance and a lack of plugging capability in nanoporous formations. The traditional additives with particle sizes between 0.1 and 100 μm do not work effectively in the formation exhibiting pore throats less than 0.1 μm like in shale [9]. The restrictions together with the increased environmental and economic issues have guided research to nanomaterials, which possess better mechanical strength, high surface area, and better interaction with clay and polymeric constituents in WBDFs.

Recent years have seen the use of nanoparticles in crystalline sand, e.g. metal oxides, carbon-based nanomaterials, in drilling fluid application in the areas of rheology, density, stability and filtration control [10]. These enhancements are important in harsh downhole conditions of pressure, temperature and depth. By adopting a thin and compact and impermeable layer of nanoparticles, the incorporation of nanoparticles can reduce clay swelling, spurt loss, and circulation loss by applying a coating on the surface of the wellbore [11]. In addition, even low concentrations of the nanoparticles are sufficient to make considerable improvements, and the total mud cost becomes lower [12].

Manganese dioxide (MnO_2) is a transition metal oxide with structural stability, and a high specific surface area and is widely compatible with the environment. It has multiple polymorphic forms ($\alpha, \beta, \gamma, \delta$ and λ), with controllable physicochemical characteristics. MnO_2 nanostructures



have been widely used in catalysis, batteries, sensors, and water purification systems because of their high adsorption capacity and great reactivity of the surface [13]. As a nano-additive in WBDF, MnO_2 can be a good bridge and sealer of nanopores in the filter cake, leading to a greater control of fluid loss and a stable rheological behavior even when used in the high-temperature and high-pressure environment. It is further more thermal resistant than other substances, which makes it useful in down hole conditions where most organic additives breakdown [14].

The knowledge of the connection between MnO_2 nanostructures and the rheological and filtration behavior of WBDF makes it possible to derive the drilling fluid formulations to perform more efficiently and less harmful to the environment. Thus, in this research, manganese dioxide (MnO_2) nanoparticles prepared through an adapted chemical process can be suggested as an effective additive to improve rheological and 3 characteristics of a water-based drilling fluid.

METHODOLOGY

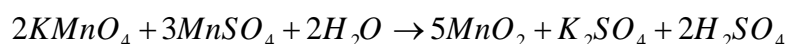
• Chemicals and materials

Hydrochloric acid (37% HCl), potassium permanganate ($KMnO_4$) (99%), sodium hydroxide (NaOH) (97%), and manganese (II) sulfate monohydrate ($MnSO_4 \cdot H_2O$) (96.3%) were bought from R&M Chemicals. No additional purification was performed on the chemicals used, and they maintained their analytical purity. Distilled water was used for the mud preparation and synthesis operations.

• Synthesis of manganese dioxide (MnO_2)

The process for synthesizing manganese dioxide (MnO_2) was as follows: A mixture of 3.04 g of $KMnO_4$ and 4.0 g of manganese sulfate solution ($MnSO_4$) was prepared in 10.00 ml of water. After that, 1.00 ml of hydrogen sulfate was added to the solution and then stirred well in a cushion flask. Manganese Dioxide (MnO_2) Synthesis Get Ready. As illustrated in Figure 1, the controlled oxidation-reduction reaction between potassium permanganate and manganese sulfate was utilized to synthesize MnO_2 nanostructures. This procedure involved a modified version of a chemical precipitation method. Following conventional procedure, a solution of 0.1 mol $MnSO_4 \cdot H_2O$ was prepared by dissolving the ions in 100 mL of distilled water and then stirring the mixture with a magnet for 30 minutes. Under continuous stirring at 60 C, 0.2 mol of $KMnO_4$ dissolved in 100 mL of distilled water was added to the $MnSO_4$ solution in a separate beaker. After adding a 0.5 M NaOH solution, the reaction mixture's pH was brought up to 7-8. We maintained the reaction duration at 3 hours until a dark brown MnO_2 precipitate formed.

The mixture was then left to age at room temperature for 2 hours in order to increase crystallinity. The precipitate was centrifuged after aging and mixed with 1 M HCl and distilled water several times until a neutral pH of the supernatant was obtained. The dried product was dried in a hot-air oven at 80 °C over a period of 24 hours and then calcined at 400 °C over a period of 2 hours to get crystalline MnO_2 nanostructures. The general equation is as follows:



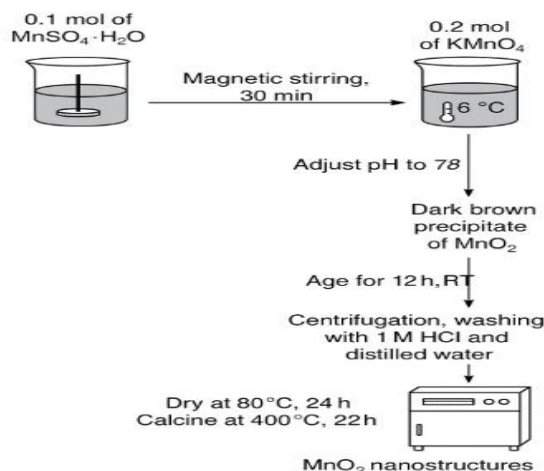


Figure 1. Schematic diagram depicting the synthesis of MnO₂.

- **Water-Based Drilling Fluid (WBDF) preparation**

WBDF was prepared as 5 samples using a Hamilton Beach Mixer as per the formulation shown in the table below (Table 1). Each sample was to be composed of:

Table 1. MnO₂-Modified WBDF Preparation.

Sample	Mn O ₂ (mg)	Fresh water (mL)	Soda Ash (g)	Sodium Chloride (g)	Caustic Soda (g)	Xanthan Gum (g)	Barite (g)	Bentonite (g)	Hydro-star (g)
MnO ₂ -0	0	318	0.15	31.9	0.20	1.00	114	10.0	3.0
MnO ₂ -3	3	318	0.15	31.9	0.20	1.00	114	10.0	3.0
MnO ₂ -6	6	318	0.16	32.0	0.22	1.05	114	10.0	3.1
MnO ₂ -9	9	318	0.16	32.1	0.22	1.05	114	10.0	3.1
MnO ₂ -12	12	318	0.17	32.2	0.23	1.10	114		

Xanthan gum was also used as a high molecular weight biopolymer to improve the rheological characteristics of the WBDF. Barite was incorporated to enhance the density of the mud whereas bentonite was used to give the mud some viscosity and colloidal solids to control filtration. Rheological characteristics of the base WBDF have been determined before nanoparticles are added.

MnO₂ nano-additives effects were examined by adding different quantities of MnO₂ nanoparticles (3 mg to 12 mg) in the samples of WBDF. The high-speed mixer was used to homogenise each and every mixture of 10 minutes to ensure even dispersion of the nanoparticles.

- **Characterisation of MnO₂**

The following methods were used to study the morphological and structural properties of the synthesized MnO₂:

- The X-Ray Diffraction Method (XRD): The crystal structure was examined using a 40k-alpha (1.5406 Å) radiation-equipped rigaku D/MAX 2200 V/PC diffractometer (Japan) operating at



40kV and 40mA. At a rate of 2°/min, the diffraction patterns were observed over a 2-theta range of 10 o to 80 o.

- Perkin Elmer's Spectrum One FTIR spectrometer confirmed functional groups and Mn-O bonding in the 400–4000 cm region, using Fourier Transform Infrared Spectroscopy (FTIR).
- Thermogravimetric Analysis (TGA): Starting at room temperature and increasing by 100°C/min in a nitrogen environment, a TGA analyzer was subjected to a thermal stability test.
- Scanning Electron Microscopy (SEM): The MnO₂ nanostructures were analyzed using a Molineweis Supra 35 VP (Germany) microscope to determine the morphology, particle size, surface texture, homogeneity, and shape.

• Rheological and filtration assays

The experiments with filtrations in the current research were carried out under ambient temperature and pressure conditions and were conducted in the API low-pressure, low-temperature (LPLT) filtration procedures. Although these conditions offer a great understanding of the fluid loss control characteristics of MnO₂ modified WBDF systems, the experimental findings are not entirely applicable in high-pressure, high-temperature (HPHT) downhole conditions. That is why the given decrease in the volume of filtrate and increase in the quality of the mud cakes may be understood in the context of surface and near-surface drilling conditions.

At room temperature (30 °C), the rheological and filtration characteristics of the WBDFs such as the density, plastic viscosity (PV), yield point (YP), and mud cake thickness, were determined.

- Density: Calculated with the help of a mud balance in the agreement with the API RP 13B-1 regulations and stated in pounds per gallon (ppg). The bubble indicator was used to level the mud balance beam and so counterbalanced it.
- Measured Viscosities: Viscosity of plastics (PV) and yield-point (YP) were all measured by using Fann M3600 viscometer at 300 and 600 rpm rotational speeds respectively. Recording of the readings was done after steady-state values were achieved. The following equations were used to calculate the parameters [15]:

$$PV = \text{Reading at } 600 \text{ rpm} - \text{Reading at } 300 \text{ rpm} \quad (1)$$

$$YP = \text{Reading at } 300 \text{ rpm} - PV \quad (2)$$

A typical API filter press was used to determine the loss of fluids and mud cake thickness. The sample of the WBDF was pressed in a filter press cell and a pressure of 100 psi was applied. Volume of filtrate was measured at 7.5 minutes and 30 minutes. Depressurisation was followed by the removal of the filter paper under great care and the thickness of the mud cake was measured with the help of a digital caliper.

All the experiments were conducted thrice so as to make the results as reproducible and accurate as possible.

RESULTS AND DISCUSSION

• Properties of MnO₂

The XRD analysis was used to examine the crystalline structure of the obtained MnO₂. The obtained pattern of XRD diffraction of MnO₂ nanostructures was compared with the standard reference pattern as shown in Figure 2. The 200, 211, and 220 planes of the tetragonal 2-MnO₂ phase were also sharp and appeared at 25, 28.7, 37.4, 42.0, 56.4, and 59.8. The existence of these typical peaks verifies the effective crystallization of the MnO₂ [16], [17].

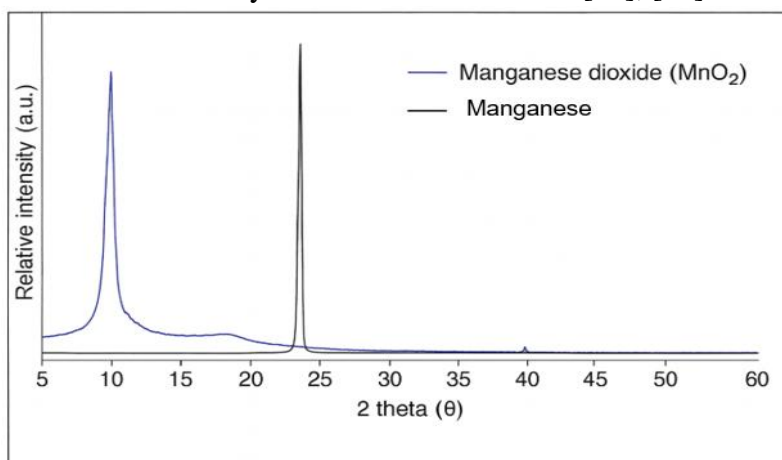


Figure 2. XRD patterns of manganese and MnO₂.

No extra peaks are found, which means that it is pure in terms of phase without the presence of additional manganese oxides like Mn₂O₃ and Mn₃O₄. The minimum size of crystallites was determined based on the Debye Scherrer equation and found to be approximately 45 to 60 nm in diameter, which indicates the nanoscale of the synthesized MnO₂.

The average crystallite size was estimated using the Debye–Scherrer equation:

$$D = (K\lambda) / (\beta \cos \theta) \quad (3)$$

D is the crystallite size, K is the shape factor (0.9), 1.5406 Å is the X-ray wavelength of the Cu K α radiation, β is the FWHM of the selected diffraction peak (measured in radians), and θ is the Bragg angle. Computation of the size of crystallites was conducted based on the highest peak of the diffraction, which was a (211) plane and had a value of $2\theta = 28.7$. The determined crystallite size was between about 45 and 60 nm, which showed the nanoscale of the synthesized MnO₂.

FTIR spectroscopy was also used to verify the existence of metal oxygen bonds and surface hydroxyl groups. According to a graph (Figure 3), a good absorption band was observed at 530580 cm⁻¹, which is related to the Mn-O vibration, signifying the effective preparation of MnO₂. The weak and broad band at 3400 cm⁻¹ is ascribed to the vibration of the stretching wave of the surface hydroxyl groups (O-H), whereas the band at 1630 cm⁻¹ is ascribed to adsorbed molecular water (H₂O bending). These data are in agreement with the literature accounts of MnO₂ nanostructures [18], [19].

The oxidation-reduction reaction between KMnO_4 and MnSO_4 occurred to produce a dark brown precipitate, which is typical of MnO_2 . The product was a fine gel-like slurry that was easily separable upon centrifugation. The dry powder of MnO_2 was dark brown-black in colour, which is differentiated by the light pinkish colour of the starting matter of MnSO_4 . This change, along with the results of the FTIR and XRD, supports the creation of nanostructured MnO_2 .

Figure 5 shows the synthesised MnO_2 using SEM micrographs. The pictures showed agglomerated nanorods and nanosheets of homogeneous morphology and particle size distribution. The MnO_2 nanostructures had a porous, rough surface texture, which increases the surface area and increases their capacity to interact with the water-based drilling fluid matrix. The size of the nanostructures was averaged at 80-50 nm. Its porous structure allows the MnO_2 to be a good candidate in fluid loss control related uses as it is capable of bridging nanopores and creating a dry filter cake on the surface of the wellbore [20].

- **Rheological properties of MnO_2 -WBDF**

The density is another important property of the drilling fluids formulation because it should be able to counteract the formation pressure and avoid instability of the wellbores or shale shying [21]. The density of WBDF is indicated in Table 2 when it is with and without MnO_2 added. Density was slightly higher with MnO_2 concentration with the base mud being 9.10 ppg and 9.85 ppg with 12 mg of MnO_2 added showing that the addition of a low level of nanoparticles did not much affect the overall fluid density.

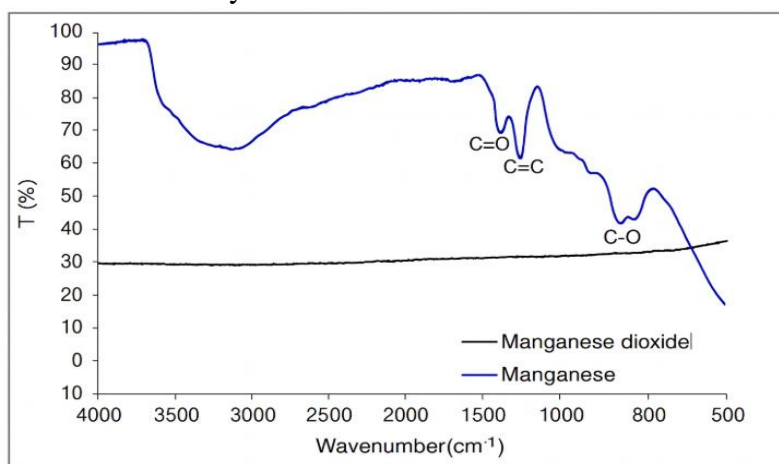


Figure 3. FTIR of manganese and MnO_2 .



Figure 4. Photograph of synthesised MnO₂ gel.

Table 2. Density of MnO₂ modified WBDF samples.

WBDF Samples	Density (ppg)
MnO ₂ -0	9.10
MnO ₂ -3	9.35
MnO ₂ -6	9.55
MnO ₂ -9	9.70
MnO ₂ -12	9.85



subsistence cuttings transportation capacity. In this regime, it was found that the MnO₂-modified WBDF can have a better flow behavior without affecting the suspension performance [15].

Figure 7 shows the shear-thinning nature of the MnO₂-WBDF system. It was not a Newtonian fluid because shear stress was not proportional to the shear rate. At low shear rates, the apparent viscosity declined at an alarming rate with shear rate but at higher shear rates, shear stress changes were not as significant. This pseudoplastic nature has benefited the drilling operation as the fluid has the ability to sustain higher viscosity at low shear rates to sustain the cuttings when the pump is not running, and lower viscosity at high shear rates to limit pumping energy during drilling circulation [23].

• Filtration characteristics of MnO₂-WBDF

The check of filtration performance is one of the critical factors to assess the effectiveness of drilling fluids. Again, excessive filtrate loss may cause formation damage and differential sticking and bad wellbore stability [24]. Figure 8 represents the influence of MnO₂ concentration on the filter cake thickness and the filtrate volume. The loss of filtrate, as well as mud cake thickness, was significantly reduced due to the addition of MnO₂ nanoparticles. When 9 mg MnO₂ and 12mg MnO₂ were added, the base WBDF (without MnO₂) showed a filter cake thickness of 2.1 mm and 1.3 mm respectively.

One can explain this decrease by the fact that the nanoscale particle size of MnO₂ and the large surface area allow it to fill the micro and nanopores in the filter medium creating a dense and resistant barrier. Also, the hydrophilic nature of MnO₂ improves contact with the aqueous phase, leading to homogeneous distribution of the particles, and better cake compaction [25]. As a result, the level of fluid loss was reduced to 11.5 mL (base WBDF) and then to 6.8 mL (at 12 mg MnO₂), which proves the enhanced sealing property of MnO₂.

Figure 9 shows SEM micrographs of the WBDF filter cakes without and in the presence of MnO₂ (MnO₂-0, MnO₂-6, MnO₂-12). The microstructure of the filter cake that was not packed with MnO₂ showed coarse and irregularly shaped pores in which the particles were loosely packed. However, the modified samples, the MnO₂ samples, have a more compact and uniform microstructure, having far fewer voids and less connectivity between the pores. When using higher concentrations of MnO₂, the nanoparticles were used to fill the interstitial cavities between the clay and barite particles to create a low-permeability network. This enhanced packing minimizes intrusion of filtrate besides increasing the sealing power of the mud cake.

These findings are consistent with previous research that found nanostructured MnO₂, TiO₂, and ZnO to enhance thermal stability and fluid loss management when added to drilling fluid [26], [27]. The impressive performance boost of the MnO₂-WBDF is due to its nano-bridging effect, which decreases pore infiltration and enhances the development of a thin and compact filter cake. This, in turn, improves the level of filtration control and rheological stability.

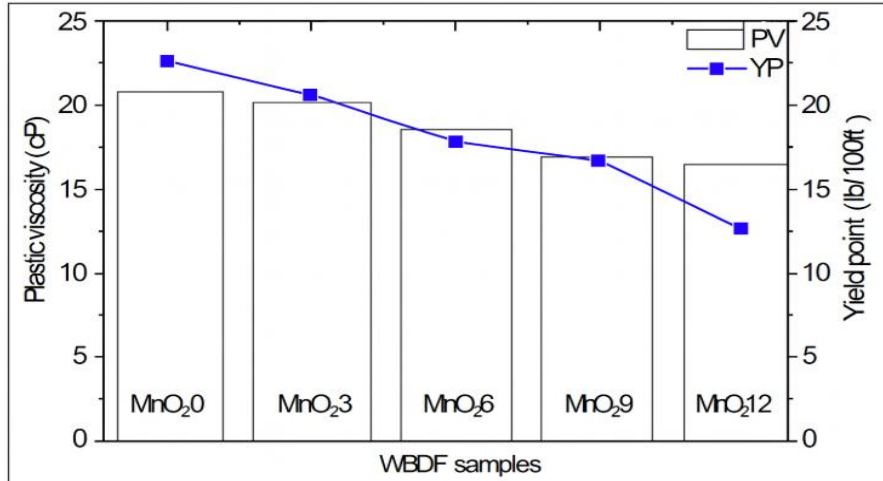


Figure 6. Plastic viscosity and yield point.

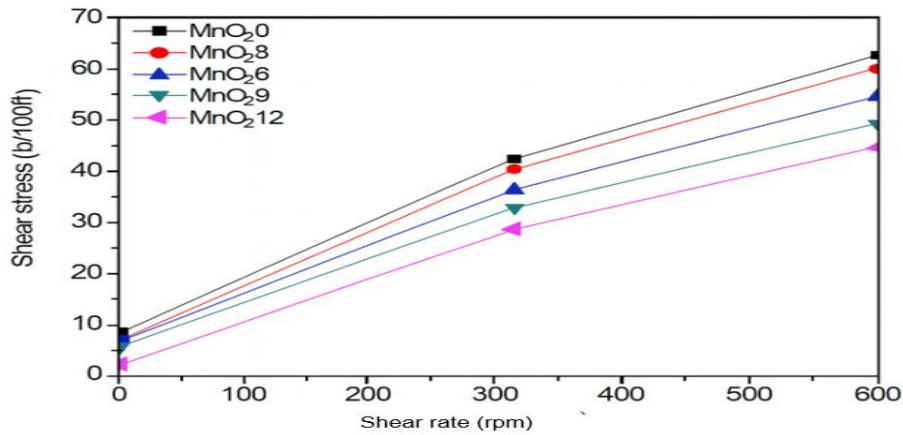


Figure 7. Flow curve of MnO₂-modified WBDF samples.

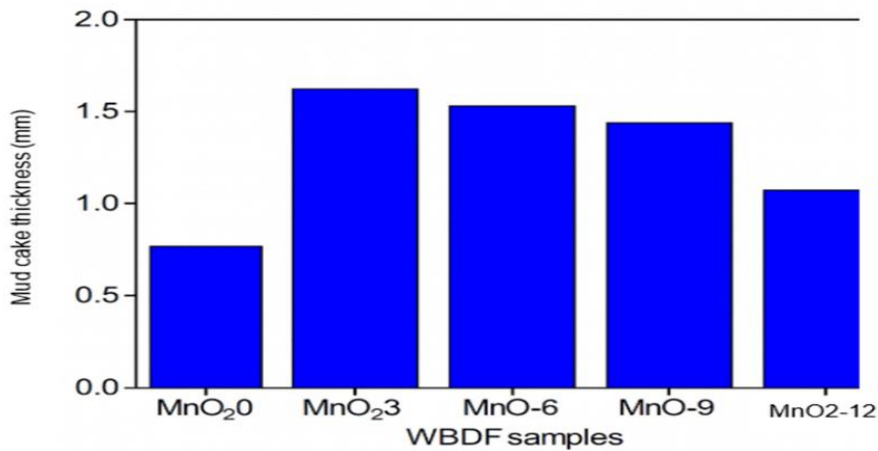


Figure 8. Mud thickness of MnO₂-modified WBDF samples.

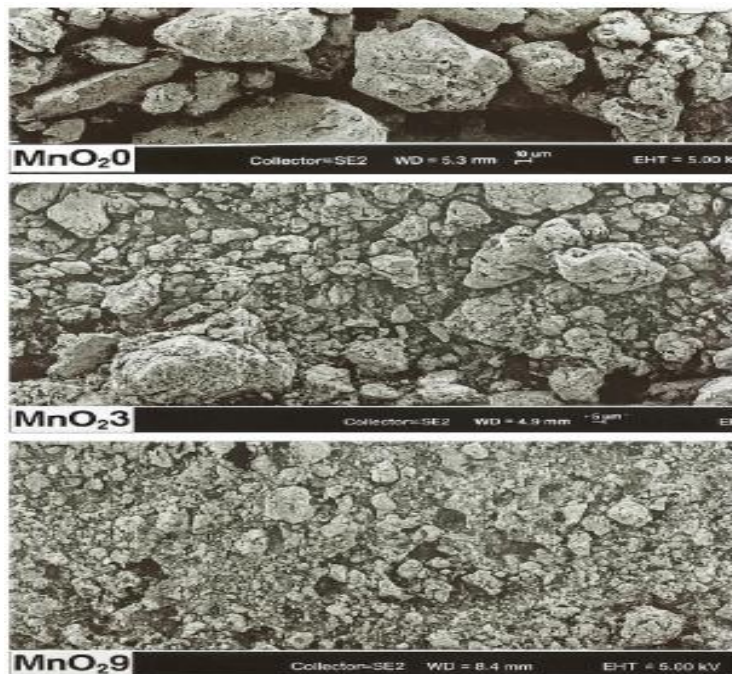


Figure 9. Scanning electron micrographs of WBDF filter cakes containing 0, 3, and 9 milligrams of MnO₂.

CONCLUSIONS

A modified chemical procedure was used to successfully manufacture manganese dioxide (MnO₂) nanostructures. These nanostructures were then examined and were found to be an effective nano-additive in water-based drilling fluids (WBDF). The rheological and filtration properties of WBDF were thoroughly investigated in relation to the MnO₂ concentration. The results of the experiment suggest that the addition of MnO₂ nanoparticles improved the performance of the WBDF. The optimization of the plastic viscosity (PV) and yield point (YP) suggests enhanced flow behavior and the capacity to suspend particles. In addition, when the concentration of MnO₂ increased, both the filtrate volume and the filter cake thickness dropped, demonstrating MnO₂'s excellent efficiency as a fluid loss control agent. Due to its hydrophilicity, tiny particle size, and large surface area, MnO₂ can significantly improve the performance of drilling fluids with a very small amount and this, in turn, reduces the additive's overall cost. Furthermore, MnO₂ is a chemically stable, eco-friendly, and reagent-free substance; as a result, it could be a potential material for next-generation nano-additives in the creation of sustainable, high-performance WBDF systems.



Conflict of interests.

There are non-conflicts of interest.

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الخلاصة

المقدمة:

تظل سوائل الحفر المائية (WBDF) أكثر سوائل الحفر استخدامًا نظرًا لانخفاض تكلفتها وملاءمتها البيئية مقارنةً بالسوائل النفطية والصناعية. ويعتمد أداؤها بشكل كبير على خصائصها الريولوجية والترشيحية، وهي خصائص حساسة للغاية للإضافات الكيميائية المُضافة إلى الطين. تستكشف هذه الدراسة تأثير جسيمات ثاني أكسيد المنغنيز النانوية (MnO_2) على السلوك الريولوجي وأداء الترشيح لسوائل الحفر المائية (WBDF).

طرق العمل:

تم تصنيع هياكل نانوية من MnO_2 باستخدام طريقة كيميائية معدلة، ووصفت باستخدام حيود الأشعة السينية (XRD)، ومطيافية تحويل فورييه بالأشعة تحت الحمراء (FTIR)، والتحليل الوزني الحراري (TGA)، والمجهر الإلكتروني الماسح (SEM) للتأكد من طورها البلوري، وبنية الرابطة، وشكلها. ثم حُضرت عينات WBDF مع وبدون جسيمات نانوية من MnO_2 . وقُيست المعلمات الرئيسية، بما في ذلك اللزوجة اللدنة، ونقطة الخضوع، والكثافة، وحجم الترشيح، وسمك كعكة الطين، لتقييم تأثير دمج الجسيمات النانوية.

النتائج:

أكد تحليل XRD تكوين MnO_2 بلوري، بينما كشفت أطراف FTIR عن اهتزازات Mn-O قوية مميزة لهياكل MnO_2 . أظهر تصوير المجهر الإلكتروني الماسح هياكل نانوية واضحة المعالم ومناسبة لتعديل السوائل. وبالمقارنة مع WBDF الأساسي، أظهرت السوائل المُحسنة من MnO_2 استقرارًا ريولوجيًا مُحسّنًا، ونقطة خضوع أعلى، واتساقًا أعلى في اللزوجة، وانخفاضًا ملحوظًا في فقدان السوائل. بالإضافة إلى ذلك، أظهرت كعكات الطين المتكونة بوجود أكسيد المنغنيز (MnO_2) انخفاضًا في السمك وعلامات على تعديل هيكلية، مما يشير إلى تحسن في كفاءة الختم.

الاستنتاجات:

تُحسن إضافة جسيمات أكسيد المنغنيز النانوية بشكل كبير الخواص الريولوجية والترشيحية لسائل الحفر المائي (WBDF). وتشير هذه التحسينات إلى أن سائل الحفر المائي (WBDF) المُعدّل بأكسيد المنغنيز (MnO_2) يُمكنه تقليل تسلسل المُرشح إلى التكوينات بفعالية، وتوفير أداء حفر أكثر استقرارًا.

الكلمات المفتاحية:

سائل الحفر المائي (WBDF)، جسيمات أكسيد المنغنيز النانوية (MnO_2)، الخواص الريولوجية، التحكم في الترشيح، اللزوجة اللدنة، نقطة الخضوع، سمك كعكة الطين.