



Rheological Properties Improvement of Treated Palygorskite Drilling Mud by Adding Prepared Nano Magnesium Oxide

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Abstract

Attapulgit clay is a hydrated magnesium aluminum silicate mineral. Attapulgit clay stone in the Bahr al-Najaf region contains calcite and quartz minerals (43.4% and 13.9% by weight), respectively. This work is devoted to develop the attapulgit clay found in Bahr Al-Najaf to be suitable for oil wells drilling. The goal of this project is to develop attapulgit mud that meets the American Petroleum Institute (API) for the application of oil well drilling mud in Bahr Al-Najaf. To achieve this objective, unwanted materials (calcite and quartz particles greater than 75 μm in diameter) should be removed and the attapulgit rods dispersed. To make attapulgit mud appropriate for oil well drilling mud. Wet sieving was used to remediate Iraqi attapulgit mud in this study. XRD and SEM tests were done to determine the metals and shape of the prepared Nano MgO to improve the rheological properties. The rheological parameters of attapulgit were measured using an ofite viscometer. Nano MgO was added to attapulgit in three proportions (0.02, 0.05, and 0.08) gm wt. Magnesium oxide showed a great contribution to apparent viscosity and plastic viscosity by 16% of attapulgit aqueous suspensions. The higher viscosity will provide better cuttings carrying capability during drilling fluid circulation especial at low velocity.

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1. Introduction

Clay minerals are utilized to improve the flow's stability and viscosity solutions because of their exceptional rheological qualities [1]. The continuous phase of the drilling mud's base fluid carries all of the mud's components and has a major influence on its performance and technological aspects [2]. Base fluids are selected depending on their availability, cost, composition requirements, technical performance, biodegradability, environmental cleanup, and health safety concerns. The three types of alkaline fluids are oil base, water base, and alkaline fluid synthesis [3]. Fresh water, salt water, brine, or saturated brine can all be used as the drilling mud's water foundation [4]. Attapulgit ($\text{Mg, Al}_2\text{Si}_4\text{O}_{10}$) is a mineral with the general formula $((\text{Mg, Al})_2\text{Si}_4\text{O}_{10}(\text{OH}))$. $\text{Mg}(\text{Al}_{0.5-1}\text{Fe}_{0-0.5})\text{Si}_4\text{O}_{10}$ or $4(\text{H}_2\text{O})(\text{OH})$. $4\text{H}_2\text{O}$. Is a chain of magnesium-aluminium silicate typical of natural fibrous clay material, with some aluminium ions replaced by iron ions at equal values. Attapulgit is employed in a range of industries, including drilling mud, petrochemicals, construction materials, chemistry, papermaking, medicine, agriculture, food, and environmental protection, due to its unusual absorptive, colloidal, rheological, and catalytic properties. Alexander and Arie [5] Sacalum (Yucatan) attapulgit was proposed as a drilling mud at first. Attapulgit has the advantage

over other clay minerals (such as bentonite) in that it is less sensitive to salts and temperature, i.e., the desired rheological properties remain constant even at high-electrolyte concentrations, which is why it is widely used in both marine and salt formation drilling [6] paraphrased and formalized Palygorskite forms gels in both fresh and salt water by creating hydrogen bonds that link particles in a lattice structure. These properties allow large, dense particles to be suspended in the clay of drilling cuttings while using relatively little water circulation in the pump and drilling sector [7]. Palygorskite, unlike bentonite, generates salt water gels and is used in a unique salt-water drilling mud for drilling salt-contaminated stratum [8]. Particles of palygorskite can be thought of as positively (+) and negatively (-) charged particle zones. In both salt and fresh water, it is the intertwining of these alternating patterns that creates the relationship. Charges that permit them to form gel suspensions. Although most clay minerals disperse in water to produce stable and viscous suspensions, the gel-forming mechanisms vary for each mineral in clay due to differences in structure, particle size shape, and content. [9, 10] Formalized paraphrase because viscosity is more important than suspended palygorskite stability, it is apparent that high pricing and toxic solvents in their operations will limit the value of industrial applications. Nanomagnesium oxide (nano-MgO) is a highly functional material that has been employed in a variety of applications, including catalysts [11], superconductors [12], antibacterial compounds [13], as well as others nano-MgO particles found in a wide range of shapes and sizes, including nano spheres and nano cubes. The form and size of nano-MgO particles are influenced by the magnesium salts used, the precipitant utilized, and the reaction circumstances. The most frequent method for producing nano-MgO is to layer several magnesium salts or magnesium hydroxide on top of each other [14,15]. Ceramic materials have been produced at low temperatures via wet chemical processing, which entails the generation of a colloidal solution gel, the formation of a gelatinous solution, and the removal of the liquid contained in the micro-interconnected channels within the gel [16]. In this study cubic shaped Magnesium oxide nanoparticles were successfully synthesized by sol-gel method using magnesium nitrate and sodium hydroxide at room temperature. Advantages of Sol Gel: Improved adhesion between the substrate and the topcoat because of the gel state, the materials can be shaped into complex geometric shapes due to their gel condition. Precursors of ceramic oxides are dissolved in a solvent suitable for converting a sol-gel solution to produce high purity products. It is a simple cost-efficient, and effective approach to make high-quality coatings by using a low-temperature process (200-600°C) [17]. One of the most recent advancements in the oil and drilling industries is the use of nanoparticles in drilling fluid technology [18]. Drilling fluid issues can be overcome [19]. Using NPs with unique features such as high thermal conductivity and a large surface area [20]. Controlling fluid loss and mud cake removing hazardous chemicals, improving heat transfer [21], and rheological qualities such as viscosity [22]. The target of this work is reaching attapulgite clays of Bahar Al-Najaf properties specifications to the American Petroleum Institute (API) standard. In this project, Iraqi attapulgite clay will be acid treated and wet sieved to make it acceptable for oil well drilling. Mud Drilling operations will be successful when suitable drilling fluids are used to minimize or control hole problems. Numerous studies and researches have been carried out to improve the drilling fluid used in drilling the well. Recently, nanotechnology applications have shown a positive impact on drilling fluid technology so the objective of this study was to formulate locally drilling fluids with nanomaterials to improve the rheological properties of drilling fluid system.

2. Experimental Procedure

2.1. Raw Materials

The materials used in the preparation of the Attapulgite mud are shown in Tables 1 and 2.

Table 1: Material used in attapulgite separation processes.

Material	Chemical formula	Specification	Source
acetic acid	CH ₃ COOH	Assay 99.5-100%	Alpha chemika-India
Distilled water	H ₂ O	Ph= 7	Al-Mansoura co-Iraq

Table 2: Material used in the manufacture of MgO- Nanomaterials.

Material	Chemical formula	Secificationp	Source
Magnesium Nitrate	Mg NO ₃ . 6H ₂ O	-----	Aldrich Chemical Corporation
Deionized water	H ₂ O	PH= 7	
Sodium hydroxide	NaOH	-----	Aldrich Chemical Corporation
Methanol	CH ₃ OH	-----	Aldrich Chemical Corporation

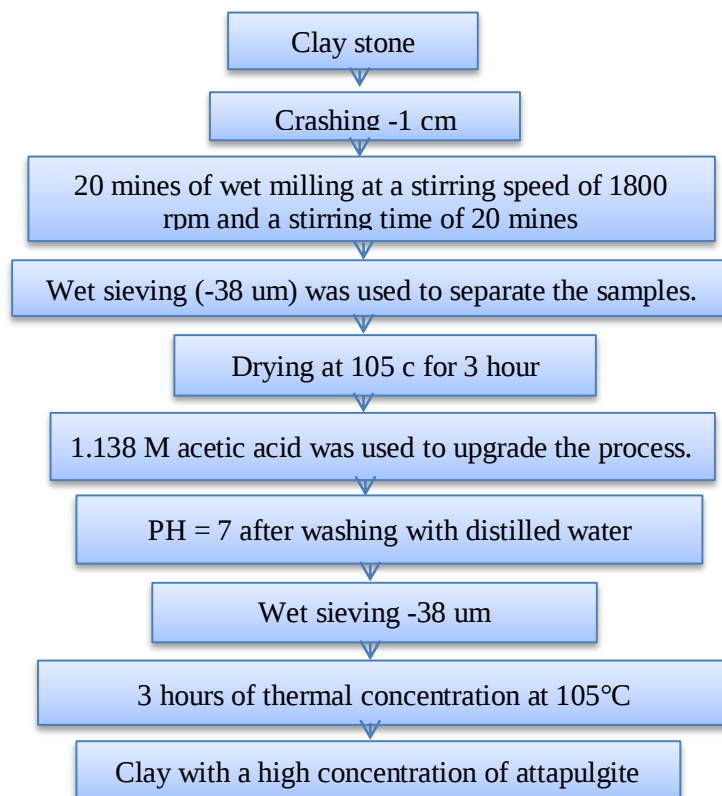
2.2. Preparation of Attapulgite Mud

Materials Selective samples of attapulgite rock were collected from Bahar AL Najaf region at meddlepartof Iraq. The mineral analysis of the rocks is shown in Table 3. Samples were crushed by using Retch jaw crusher machine (type BB200 rostoffrei –Germany) to obtain small cubic particles with size $\approx 1\text{cm}^3$.

Table 3: Theminal analysis for attapulgite rock

Minerals	Calcite	Quartz	Palygroskite	Antigorite-T	Vermiculite	Chlorite	Phengite
%	43.4%	13.9	6.2	3.0	0.1	5.6	4.2

Sample Preparation: Figure1 shows the flow chart for purification Process for sieved attapulgite was devoted to remove calcite mineral. This process was implemented as following sequence, first, adding sieved attapulgite clay to 1.138 M acetic acid at liquid /solid ratio 10 to1 v/w in glass container under mechanical stirring (800 rpm) at 28 C for 1 hour. 2. washing with distilled water until pH values be 7. 3. wet sieving process was applied at the studding mesh size. 4. Achieved attapulgite clay was dried at 105°Cfor (3hour).

**Figure 1:** The flow chart for attapulgite acid treatment.

2.3. Nanomaterial

Preparation of nano-magnesium oxide

Magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was used as a source material, while NaOH was used to make experimental magnesium oxide nanoparticles. The Aldrich Chemical Corporation provided all of the ingredients utilized in this synthesis, which were employed without additional purification. In a typical experiment, 0.2 M magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was dissolved in deionized water (100 ml). With steady stirring, a (0.5 M) sodium hydroxide solution was added drop by drop to the prepared magnesium nitrate solution ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$). A crystalline white precipitate after a few minutes, magnesium hydroxide appeared in the beaker, stirring went on for thirty minutes. The solutions had a pH of 12.5 and the precipitate had been filtered and washed three to four times with methanol to remove ionic contaminants. After re-crystallization and grain development, the material was centrifuged for 5 minutes at 5000 rpm/min before being dried at room temperature. The dry white powder samples were annealed in the air at 500°C for 2 hours shown in Figure 2.



Figure 2: Steps of synthesis MgO using Sol-gel technique

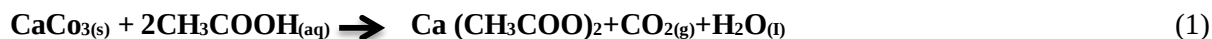
2.4. Experimental Steps

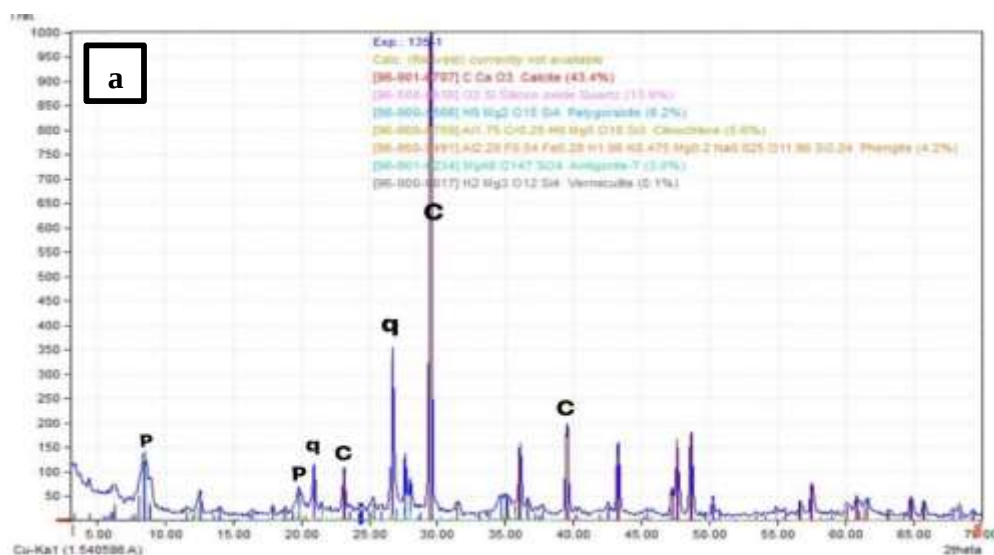
The drilling mud was prepared as explained below: 1. Mix (22.5) g Attapulgate with (350) ml of water for 30 min using multi mixer (Hamilton Beach mixer). 2. Leave the mud for 24 hours to hydrate. 3. Mix the hydrated mud for 10 minutes to break the gel. 4. Add the desired amount of MgO nanomaterials and mix with (Hamilton Beach mixer) for 10 minutes. 5. Use ultra-sonic bath for good mixing and dispersion for 10 minutes. 6. Now the water based mud is ready for testing.

3. Result and Discussion

Figure 3-a The primary component of attapulgate is revealed by X-Ray diffraction (XRD) of the raw material (Calcite, Quartz, Attapulgate, Illite, Chlorite).

Mineral analysis of upgraded attapulgate with acetic acid is shown in Figure 3-b. The results demonstrate that 1.138M acetic acid was enough to remove all of the calcite from the separated clay. The acid used in the upgrading procedure was determined using the chemical reaction equation for acetic acid with calcite in this study.





P: Palygorskite, q: Quartz, C: Calcite.

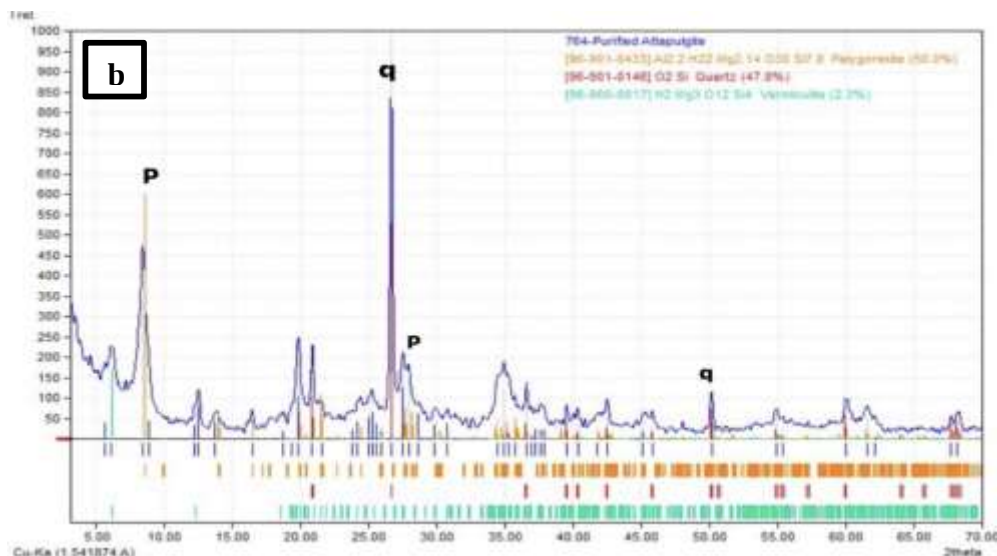


Figure 3: Mineral analysis **a)** for untreated (with calcite) clay **b)** for treated attapulgite clay.

Figure 4 shows the X-ray diffraction pattern [23]. Of the MgO nanoparticle that annealed at 500°C for two hours in air, XRD analysis is performed in the range of (20–80°) (2θ) using CuKα radiation. It clearly exhibits the peaks at angles 36.96°, 42.98°, 62.36°, 74.71° and 78.66° corresponds to (111), (200), (220), (311), and (222) planes[24]. The intense peaks shows that the powders are highly crystalline. The powders obtained from the MAS precursor are composed of MAS cubic structure (JCPDS card, No. 21-1152) which is similar to those reported by Ganesh and Pati et al [24,25]. No other impurity phase is found in the XRD pattern. XRD pattern shows high intense (220) orientation peak revealing the high crystallinity of the synthesized material. The mean crystallite size is calculated using (200) reflection and found to be (20-30) nm [26].

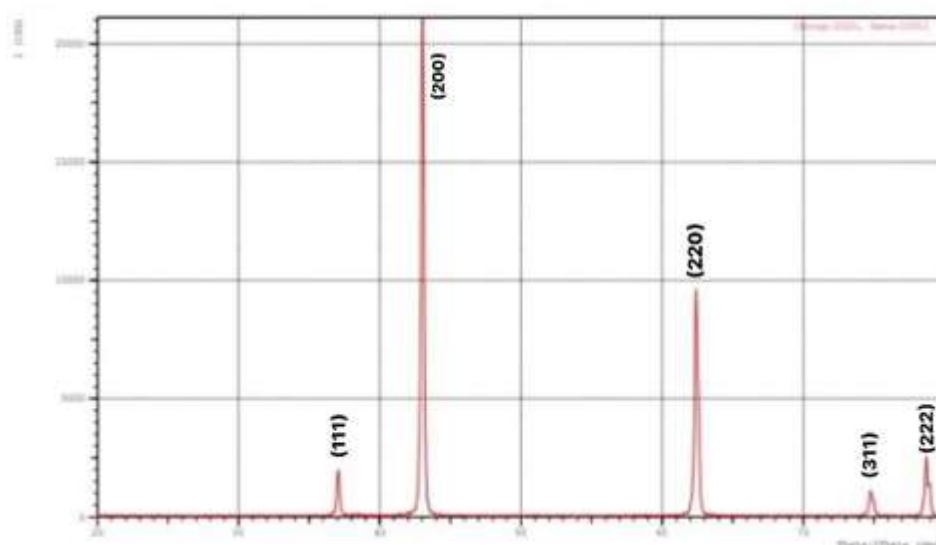


Figure 4: XRD Analysis of MgO nanoparticles

Reveals the FE-SEM [27]. Images of the obtained MgO nanoparticles, Figure 5a-b illustrates that small amount of agglomeration is present in the as-synthesized sample due to annealing at 500°C, results in further agglomeration of the particle as well as deterioration of the spherical shape. The FE- SEM images are shown in Figure 5c-d, where spherical and cubic shaped nanoparticles of magnesium can be seen, with particle size of about 20-30 nm [28].

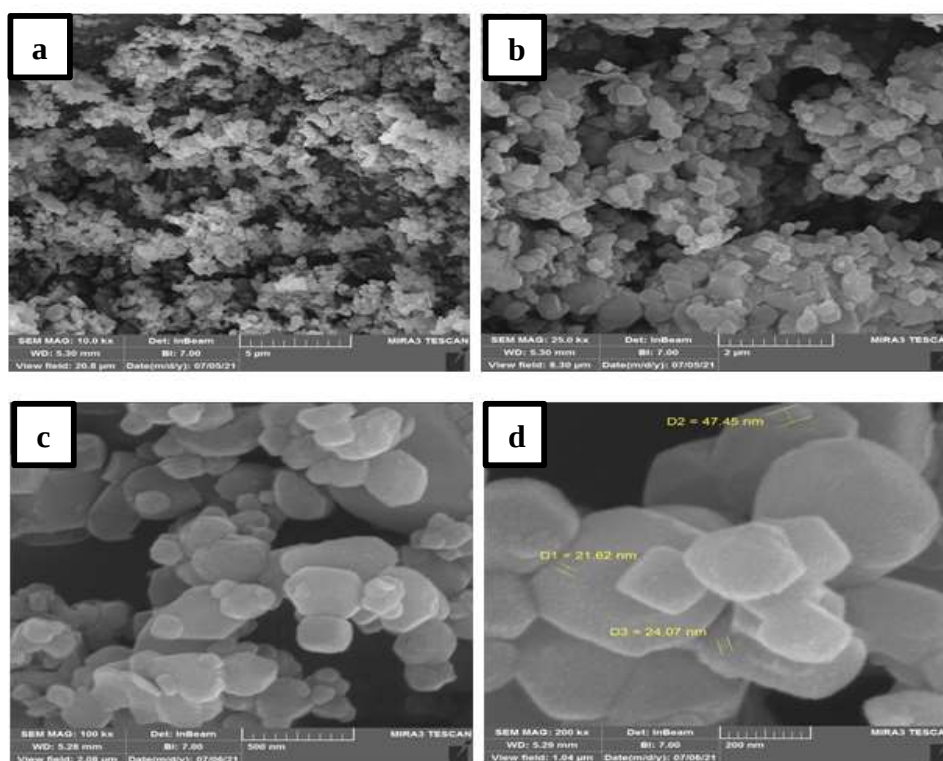


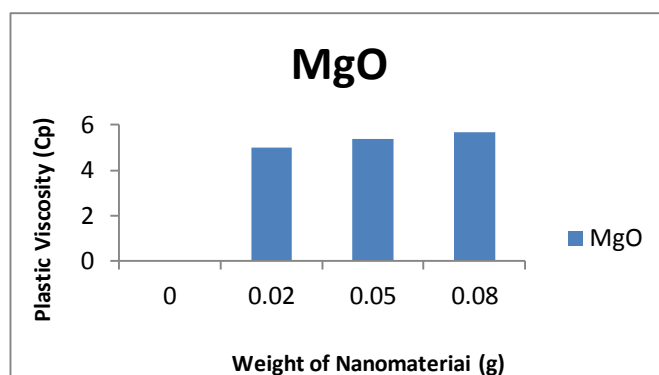
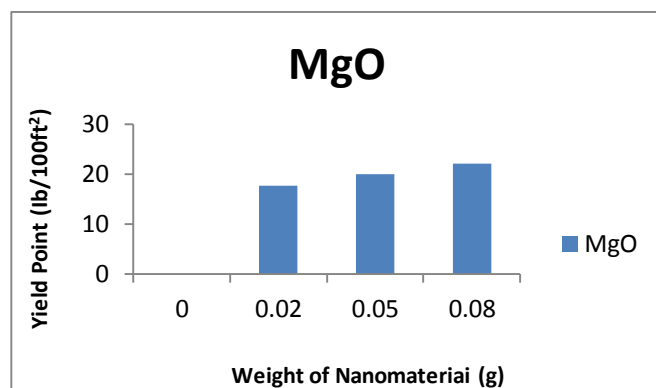
Figure 5: FE-SEM images of the synthesized MgO nanoparticles synthesized using extract where the scale bar is **a)** 5µm, **b)** 2µm, **c)** 500 nm, and **d)** 200 nm.

Table 4 shows the Rheological Properties of the attapulgite water-based mud with different concentration of MgO nanoparticles.

Table 4: Rheological properties of attapulgite water-based mud with different concentration of MgONanoparticles.

Wt (g)	PV (Cp)	YP (Ib/100 ft²)	AV (Cp)	Gel Strength (Ib/100 ft²)	
				10 Sec	10 Min
Attapulgite water-based mud					
22.5	4.9	16.1	13.7	19.6	20.8
Attapulgite water-based mud + MgO NPs					
0.02	5	17.6	14.3	22.5	23.8
0.05	5.4	20	15.9	25.6	26.9
0.08	5.7	22.1	17.2	26.4	27.0

The effect of adding (0.02, 0.05, 0.08) gm of magnesium oxide nanoparticles to attapulgite drilling mud are shown in Figures (6,7,8) the effect of MgO nanoparticles weight on (PV,YP,AV), which gave the improvement percentage (2%),(8%), (16%) in Plastic Viscosity. Through adding (0.08) g, the highest PV , YP and AV increased to (5.7 , 22.1, 17.2) Cp respectively, while the gel strength (10 sec and 10 min) increased to (26.4 and 27.0 lb/100 ft²) respectively. It is concluded that Magnesium oxide nanoparticel revealed great contribution to viscosity enhancement. The main mechanism was the electrostatic attractive interaction between magnesium oxide particles with positive charges and the palygorskite rods with negative charges [29].

**Figure 6:** Effect of Nano-magnesium oxide Weight on Plastic Viscosity of attapulgite water-based Mud.**Figure 7:** Effect of Nano-magnesium oxide Weight on Yield point of attapulgite water-based Mud.

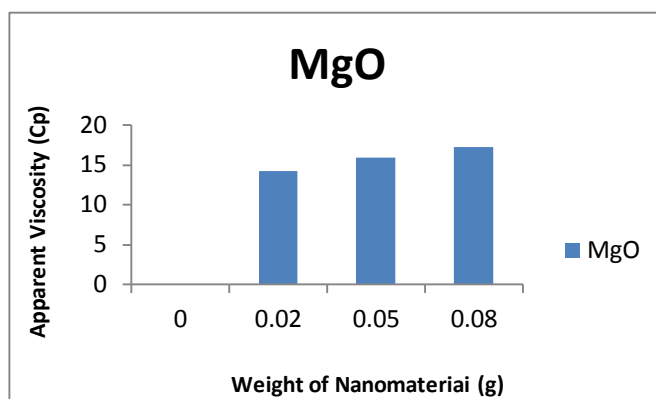


Figure8: Effect of Nano-magnesium oxide weight on apparent viscosity of attapulgite water-based Mud.

4. Conclusions

The findings of an acid treatment for separated attapulgite using a weak acid (acetic acid) showed that the full calcite mineral could be removed without causing any damage to the attapulgite morphology. Therefore, upgrading attapulgite clay with acetic acid yields excellent rheological qualities. By using the sol-gel method and magnesium oxide nitrate, the brucite and perclase phases were created from magnesium oxide nanoparticles. In which a dried white gel was annealed the air at 500°C for 2 hours. The cubic shape of the individual nanoparticles of magnesium oxide with a size of around 20-30 nm, as shown by SEM, leads to assemble large particles as well as spherical shape. XRD for bulk MgO, measurements indicated the normal mono crystalline periclase phase, which makes it a molecule and makes it a good site. The effect of adding 0.08 g of Magnesium oxide NPs to attapulgite water-based mud, which gave an improvement in Rheological properties compared to the blank sample increased to 16% in plastic viscosity. It is concluded that Magnesium oxide nano particle demonstrated a significant contribution to the improvement of viscosity. The main process was electrostatic interaction between positively charged magnesium oxide particles and negatively charged palygorskite rods. The Palygorskite rods have been structurally reversed as a result of this combined action, resulting in flocculation reinforcement.

Conflict of Interest

There are no conflicts of interest regarding the publication of this manuscript.

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