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Experimental Investigation of Rheological and Filtration Properties of Water-Based Drilling Fluids in Presence of Various Nanoparticles

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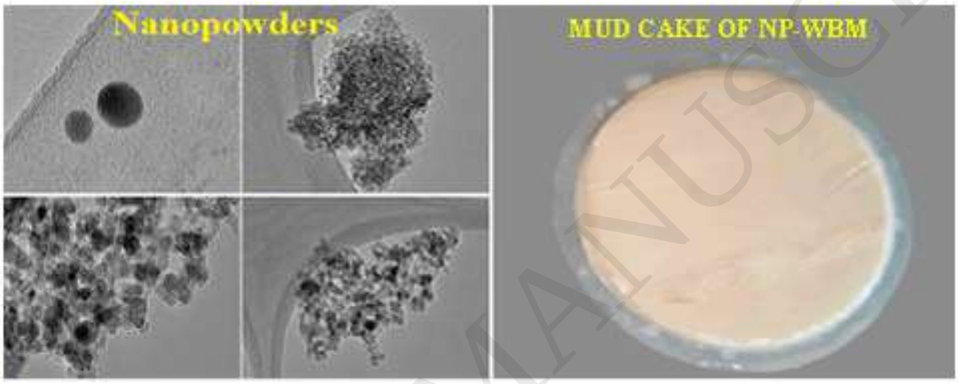
GRAPHICAL ABSTRACT



ABSTRACT

For the past decades, considerable attention has been focused on the manufacture of a high-performance, environmentally compliant water-based mud system to be a better choice than oil and synthetic-based muds (OBM/SBM). However, the improvement of WBMs has not reached the satisfactory level yet and attempts in this regard should be continued. Accordingly, in this study, an attempt was made to improve the performance of a Bentonite-WBM by adding four types of hydrophilic nanoparticles (NPs), namely aluminum oxide (Al_2O_3), titanium dioxide (TiO_2), silicon dioxide (SiO_2), and copper oxide (CuO). The NPs were dispersed in the drilling fluid with concentrations of 0.01, 0.05, 0.1 and 1wt%. The results revealed that the Al_2O_3 NPs increased the amount of mud filtration up to 80% while the mud cake quality became poorer as compared to the based mud. In contrast, the amount of mud filtration had a decreasing trend when SiO_2, TiO_2 and CuO NPs were applied especially at the concentration below 0.5wt%. Rheological properties and gel strength were also improved in the presence of TiO_2, Al_2O_3 and CuO NPs in comparison with the based mud. Overall, it was concluded that adding of the NPs at concentrations below 0.5wt% to the Bentonite-WBM has potential to improve rheological and filtration properties.

Keywords: Drilling Fluids, Water-Based Mud (WBM); Nanoparticles; Rheological Properties

1. Introduction

Globally growth in energy demand and decline in oil production from the current oil resources has renewed the interest of petroleum engineers to explore new opportunities in deep-water and unconventional hydrocarbon reservoirs. In this regard, the right choice of a drilling fluid formulation for specific drilling conditions is a key factor for the success of drilling operations, particularly in unconventional formations. Drilling fluids have various roles which include cooling drilling pipes and bits, carrying drilling cuttings from the bottom of a wellbore to the ground surface, suspending the cuttings from sedimentation during the shutdown, and stabilizing the wellbore [1-3]. Drilling fluids are mainly classified into three types, namely water-base muds (WBM), Oil-based muds (OBM) and synthetic-based muds (SBM). The OBM and SBM have higher operational efficiencies as compared to the WBM. However, the use of OBM and SBM has been declined due to the environmental issues [4-6]. This remains WBMs as the preferred ones over the other two types despite their limitations. Therefore, in order to obtain further achievements in drilling engineering, more studies on WBMs properties are in demand to improve their applications.

The main drawback of applying WBMs emerges in the course of drilling a shale formation due to shale swelling which makes the use of WBMs ineffective. This is mainly due to the fact that shale swelling brings other destructive problems such as wellbore instability, lost circulation, and pipe sticking [7-8] which lower the rate of penetration and raise the drilling operation costs. In the 19th century, OBM was utilized in shale formations. However, in the 20th century, the application of OBM was prohibited due to the environmental issues. Therefore, water became the main and unique fluid to make drilling muds. This caused the researchers focused on the modification of rheological WBM properties to solve the aforementioned problem. It is an important point that the driller of wells be able to control the rheological properties of drilling fluids by using various additives.

Several studies were carried out on the WBMs during past decades to modify their properties by adding different additives such as soda ash and calcium carbonate [9-10]. Recently, the use of nanoparticles (NPs) has been introduced for modification of drilling fluid properties [11]. Through chemical and physical processes, researchers have shown an ability to create nanomaterials with improved thermal, mechanical, electrical, and rheological properties. The positive effects of different types of NPs on the properties of drilling fluids are documented by some researchers [1,10,12-18]. For example, Hou et al. [16] and Mohammadi et al. [15] evaluated the effects of nanopowders on the rheological properties of clay based-muds at high temperature and pressure conditions. They could enhance the penetration or plastic deformation. Rosso et al. [12] determined the effect of zinc oxide NPs to remove H_2S productive on the wells to improve the WBM performance and maximizes the porosity. Abdo and Haneef [1] came up with an approach to stabilize the drilling fluid rheology in high-pressure, high-temperature (HPHT) conditions by making use of NPs. They claimed that NPs are able to retain the properties over a wide range of operating temperatures and pressure, thus ensuring efficient operation in versatile formations and operating conditions. Mohammed [10], Amanullah et al. [13], Mao et al. [14], and Aftab et al. [17] have studied the effect of various concentration Nano iron oxide on the enhancement of Bentonite-WBM rheological properties such as yield point, plastic viscosity and apparent viscosity. Chai et al. [19] and Amarfio [20] studied the effect of Al_2O_3 NPs on the WBM at varying temperature conditions that have shown increasing procedure of shear rates. Sadeghalvaad and Sabbaghi [21] and Zhou et al. [22] studied the effect of TiO_2 NPs on the WBM properties and the results indicated that the additive contributes in increasing the base mud viscosity and decreasing the fluid loss and filter cake thickness. Wang et al. [18] introduced modified Fe_3O_4 NPs with Poly (sodium p-styrene sulfonate) into water-based drilling fluids as effective additives. Their rheological tests indicated a significant improvement of the drilling fluids against salt (KCl) tolerance even under high temperature and high salinity. They, therefore, concluded that their modified water-based drilling fluids are potential candidates for drilling in deep and salty formations. A few research studies have also been conducted on thermal stability and hydrate inhibition by metal oxide NPs [23-28]. It was found that heat transfer properties have been improved extensively through the addition of metal NPs. With the use of NPs, thermal conductivity increases over 50% for some cases, and filtration loss decreases with increasing NPs concentration for nearly all studies. Accordingly, it can be concluded that NPs may have the potential of solving technical challenges associated with the drilling operation.

The performance of a nano system in water based drilling fluid has shown promising potential in shale formations stability. The main mechanism through which the nano system reduces the shale permeability is by physically plugging of the nanometer sized pores [29]. The right sized NPs in combination with the correct fluid loss system can minimize the fluid-rock interaction.

As mentioned above, although there are some studies on the effects of NPs applications on the rheology of WBM, the number of NPs utilized in the previous studies was limited and more studies are in demand in this area of research to completely understand the roles of various NPs types and concentrations on the WBM. Thus, the objective of this study is to determine the rheology properties of a Bentonite-WBM in the presence of various metal oxide NPs namely Al_2O_3, TiO_2, CuO, and SiO_2. Moreover, finding the optimum concentration of the above mentioned NPs was another task in the current study. For this purpose, the Bentonite-WBM was first made and then the above mentioned NPs were added at different concentrations to the base mud. Thereafter, plastic viscosity, yield point, gel strength, filtration loss and filter cake thickness were evaluated and the results were compared to each other.

2. Materials and methods

2.1. Materials

In this study, four commercial NP types, namely TiO_2 (40nm, purity 99.5%, specific surface area 50-100 m^2/g), SiO_2 (40nm, purity 99.5% and specific surface area 160 m^2/g), CuO (40nm, purity 99% and specific surface area 50 m^2/g), and Al_2O_3 (40nm, purity 99% and specific surface area 60 m^2/g) were Procured from SkySpring Nanomaterials, Inc., (Houston, TX). X-ray diffraction (XRD, model D5000, SIMENS) and transmission electron microscopy (TEM, model JEM-2100/HR, JEOL, Acc.200.00kV) analyses were carried out to evaluate the NPs crystalline compositions, sizes, and morphologies. Sodium carbonate (also known as soda ash, Na_2CO_3, purity >99%) was obtained from Merck chemicals. Bentonite (size 40 µm and density of 2.68 g/cm^3) powder received from SunClayTherapy, Inc. (Florida, US) and was utilized without further purification. To determine the morphology of Bentonite, Field Emission Scanning Electron Microscope (FESEM; HITACHI-SU8020) image was prepared from the powder. Furthermore, energy dispersive X-ray (EDX) analysis was also carried out to determine the Bentonite compositions.

2.2. Water-based mud preparation

To make a based mud, 22.5 gr Bentonite was slowly added to 350 mL of distilled water and put them in a mixer for 10 mins. Then, 2 gr of sodium carbonate was added to the suspension to maintain the filtration rate and increase the viscosity of the drilling fluid. The suspension was mixed by the mixer for 5 mins to achieve a uniform suspension. The prepared mud was put in the room at ambient condition (27 ^oC) for 16 hrs according to the API standard (API 1608). Accordingly, the bentonite crystallized completely in the suspension. After 16 hrs, before any testing the mud was put further in the mixer for 2 mins to recombine the mud contents. To determine the influence of NPs on the mud rheology, the base mud was placed into a balloon and the NPs in concentrations of 0.05, 0.1, 0.5 and 1 wt% were added to the prepared base mud. The muds were then agitated for 1 hr using an orbital shaker at 220 rpm and ultrasonicated by an ultrasonic bath for a period of 15 mins to obtain homogeneous muds prior to each test. It should be noted that API 13D has been meticulously used for conducting the experiments. The concentrations of mud ingredients and nanoparticles have been adopted from literature [30] and some primary experimental studies.

2.3. Determination of the rheological properties of muds

Basic rheological properties of the prepared muds such as plastic viscosity, yield point, gel strength, filter cake thickness and filtrate loss were tested. The viscosity and gel strength of muds were measured via a V-G meter (model 35, EN 61010-1:2010, CAN/CSA C22.2 No. 61010-1-2012). API RP 13B-1 was used to measure the plastic viscosity and yield point parameters. The sample initially was placed in a Thermo-cup and then was heated up to 49°C. Then, the V-G meter motor speed was set at 600 and 300 RPM to determine the values of plastic viscosity and yield point at each speed. Besides that, a filter press (Series 300 API) was applied to measure the filter loss and evaluate the filter cake. The filter press has a pressurized cell, which has been fitted with a filter medium. The schematic of filter press setup is shown in Fig.1. A Nitrogen gas cylinder was connected to the filter press equipment to raise the cell pressure to 100 psi. The prepared mud was loaded into the cell within 1⁄4 inch of O-ring groove. A suitable graduated cylinder was placed under the filtrate opening to achieve the filtrate value. The inlet valve applying pressure was then opened to the cell. Each test was carried out in a period of 30 min. once the test was finished, the cell was disassembled and the mud was discarded. It should be noted that the disassembling should be done slowly and carefully to prevent any disturbance and damage to the formed mud cake. Then, the cake was washed gently to remove excess mud. Finally, the thickness of the filter cake was measured and reported according to the thickness of 1/32 inch. It should be pointed out that all tests were carried out at room temperature (27 ˚C).

The rheology of the mud systems has been also tested using rotational rheometer (OFITE 800). Fig. 2 shows shear stress against the shear rate of the base drilling fluid without NPs at 27 ^{o}C. As it can be seen from Fig.2, shear stress was increased with the shear rate which reveals that the fluid behavior of the base mud follows the Herschel-Buckley model with a shear thinning behavior similar to Bingham Pseudoplastic fluids. However, power law fluid behavior has also been reported by other researchers [31-32].

The Herschel-Buckley model can be generalized by the following Equation:

$$\tau = \tau_0 + K\gamma^n \qquad (1)$$

Where τ is the shear stress (lb/100 ft^2), τ_0 is the yield point (lb/100 ft^2), K is the consistency index, γ is the shear rate (sec^{-1}) and n is the flow behavior index (dimensionless) which should be less than 1 for shear thinning fluids.

3. Results and discussion

3.1. Powders characterization

The average sizes of CuO, Al_2O_3, SiO_2, and TiO_2 nanopowders were determined: firstly, by X-ray diffraction (XRD) analysis, and secondly, by Scherrer's formula [33] as follows:

$$d_{hkl} = \frac{0.9\lambda}{B\cos\theta_B} \qquad (2)$$

where d_{hkl} is the mean NPs size (nm), λ is the wavelength of Cu Kα radiation (=0.1542 nm), B (in radians) comprises the full width at half-maximum of the broadened diffraction line observed at the 2θ angular range, and θ_B is the Bragg angle of diffraction.

The XRD analysis from Al_2O_3 and CuO samples demonstrated that these two NPs have alpha (α) crystalline structure and their compositions are pure (Fig. 3a-c). Furthermore, SiO_2 and TiO_2 NPs have partial amorphous (semi-crystalline) structures. The former has quartz composition while the latter has a composition of anatase as shown in (Fig.3, (b) and (d)). Besides that, from the Eq.1, the sizes of CuO, Al_2O_3, TiO_2, and SiO_2 NPs were calculated 29, 26, 11.8, and 16.1 nm, respectively. The sizes of CuO, Al_2O_3, TiO_2, and SiO_2 NPs were also measured via TEM and image-processing software (ImageJ; National Institute of Mental Health). The measured geometric means of CuO, Al_2O_3, TiO_2, and SiO_2 NPs diameter were 28 (11-120), 25 (12-138), 6 (3-65), and 13 (7-81) nm, respectively. Therefore, there is a 35% to 40% difference in the size of the nanopowders depending on whether it was measured by TEM and XRD or reported by the manufacturer. Furthermore, the morphology of NPs appeared to be spherical according to TEM images (Fig. 3). In addition, the FESEM plus EDX analyses results depict that the morphology of bentonite particles is similar to cornflake and O, Si, Al, Na, Ca, and K are the elements detected in this clay (Fig. 4).

3.2. Effect of NP type and concentration on rheological properties of Bentonite-WBM

3.2.1. Plastic Viscosity

Plastic viscosity (PV) is a parameter of the Bingham model and it represents the viscosity of mud when extrapolated to infinite shear rate on the basis of the mathematics of Bingham model [34]. Generally, a drilling fluid with high PV is difficult to pump and, therefore, is not favored by drillers for drilling operations. However, a drilling fluid should have an appropriate density to improve the hydrostatic pressure which has a direct relationship with the mud viscosity.

Accordingly, lower mud viscosity results in lower mud density and apparently lower hydrostatic pressure which is not always a good outcome. Therefore, an optimum value of PV should be obtained by considering all the operational conditions and the required characteristics of the mud for having safe drilling operations [35].

The PV of Bentonite-WBM (base mud) was measured to be 6 cP. As shown in Fig.5, the PV of Bentonite-WBM was generally increased by adding the NPs. However, the amount of PV for each type of NPs at various concentrations (from 0.01 to 1 wt%) was different. The amount of Bentonite-WBM PV was approximately doubled by adding Al_2O_3 NPs from 0.1 to 1wt%. The amount of PV was also increased to 10 cP by adding CuO NPs at 0.5wt%. However, by adding 1wt% of CuO NPs to the Bentonite-WBM mud, the PV amount was decreased to 8.5 cP. By SiO_2 NPs the amount of PV was remained constant at 8 cP at all concentrations. The results for TiO_2 NPs were totally different from other NP types. By adding 0.01wt% TiO_2 NPs to base mud, the PV amount was achieved 10 cP. With increasing concentration up to 0.5wt%, the PV was declined to 7.5 cP before reached to 9 cP at 1wt%.

NPs can improve the rheological properties of Bentonite-WBM using various mechanisms which mostly depend on the continuous phase of mud system and characteristics of NPs. SiO_2 NPs can typically enhance the apparent viscosity of water as the continuous phase of drilling fluids [5,36-38]. It has been well established that the viscosity of nanofluids is much higher than the viscosity of conventional dispersions at the same volume concentration of dispersed particles. As viscosity is defined as internal friction between two layers of a fluid under shear stress, once NPs are dispersed in the fluid, there is a possibility of increasing friction between layers of the fluid, which results in an increase in viscosity of nanofluid [11,39-40].

Generally, in absence of any reaction, physical properties of NPs such as their geometry and density along with their heat capacity play an important role in alteration of drilling fluid rheological properties. the NPs and Bentonite-WBM may be linked or bonded together directly or through certain intermediate chemical linkages to increase the PV of Bentonite-WBM [41]. Among NPs utilized in this study, Al_2O_3 NPs at a concentration of 1wt% is the best option to enhance the PV of Bentonite-WBM and CuO, TiO_2 and SiO_2 NPs in order are next options.

3.2.2. Yield Point

The yield point is a part of fluid flow resistance created by electrochemical forces within a fluid. These electrochemical forces are due to the electrical charges on the surface of reactive particles [42]. Generally, increasing yield point value leads the drilling cuttings faster to transport and carry toward the ground surface [43]. Yield point must be high enough to carry cuttings out of the hole, but not so large as to create excessive pump pressure when starting mud flow [44]. The yield point of based Bentonite-WBM was measured to be 21 lb/100ft^2. The effects of NPs on yield point of Bentonite-WBMs are demonstrated in Fig.6. The yield point of Bentonite-WBM shows different performances at the various NPs concentrations. The yield point of Al_2O_3 NP Bentonite-WBM has an increasing trend at all examined concentrations. The maximum value of yield point for Al_2O_3 NP Bentonite-WBM was achieved 45 lb/100 ft^2 at a concentration of 1wt%. In contrast, the yield point values achieved by CuO NPs were moderately decreased from 22 lb/100 ft^2 at 0.01wt% to 18 lb/100 ft^2 at 1wt%. The yield point values of Bentonite-WBMs approximately remained unchanged around 24 lb/100 ft^2 by adding SiO_2 and TiO_2 NPs at all concentrations. Generally, Al_2O_3, TiO_2, SiO_2 NPs reveal better yield point result than CuO NPs especially at a concentration of 1wt%.

3.2.3. Gel strength

Gel strength is one of the important drilling fluid properties which represents the ability of a drilling fluid for suspending drilling fluid solids and cutting [42]. The gel strength is a measurement of electrochemical forces within the fluid under static condition. Fig 7 shows the effects of NPs on gel strength at different concentrations at 10 seconds and 10 minutes, respectively. The initial measurement of base mud gel strength showed that the gel strength value for 10 seconds and 10 min was respectively 17 and 17.5. As shown in Fig. 7, the gel strength values of Bentonite-WBMs for the both tests (10 sec and 10 min) were increased around 2.5 folds by adding Al_2O_3 NPs from 0.01 to 1wt%. At 1wt% of Al_2O_3 NPs concentration the Bentonite-WBM 10 sec gel strength and 10 min gel strength values achieved 47 and 76 lb/100 ft^2, respectively. Both TiO_2 and CuO NPs Bentonite-WBMs have a similar trend in gel strength values where at concentrations between 0.01 and 0.1wt%, the gel strength values decreased and then increased till 1 wt%. The values of 10 sec and 10 min gel strength in the presence of SiO_2 NPs were remained constant from 0.01 to 0.5wt%. However, by adding 1wt% of SiO_2 NPs both 10 second and 10 mins gel strength values were declined. The high gelling characteristics of the fluid may demand a high starting torque which needs to be justified by investigating the shear thinning behavior of the fluid. Furthermore, high gel strength is essential for avoiding many severe drilling problems [45]. As a result, it can be concluded that the gelling properties of Al_2O_3 and TiO_2 NPs at concentration of 1wt% are better than SiO_2 and CuO NPs.

3.2.4. Filtration Loss

The fluid loss volume and mud cake thickness are the two measurable parameters in this type of test. The high volume of filter loss is not desirable property for drilling mud since it may have some issues like formation instability and formation damage [46]. The comparison of the fluid loss behavior of Bentonite-WBM with different concentrations of NPs is shown in Fig.8. The Bentonite-WBM had 13.4 mL fluid loss after 30 min. Adding 0.01wt% of Al_2O_3 NPs to the Bentonite-WBM, the fluid loss volume was achieved 12.5 mL and it was significantly increased to 24.4 mL at Al_2O_3 concentration of 1wt%. Generally, it can be concluded that the Al_2O_3 NPs is not a suitable additive for reducing the filter loss of Bentonite-WBM.

The filter loss of Bentonite-WBM was slightly reduced to 12.6 mL by adding 0.1wt% of CuO NPs. However, addition of 1wt% of CuO NPs to the Bentonite-WBM, mud filtrate volume raised to 14.4 mL. The results show that CuO NPs can be a candidate as an additive to reduce the Bentonite-WBM fluid loss especially at concentration below 0.1 wt%. At concentrations below 0.1wt%, SiO_2 NPs effectively reduced the Bentonite-WBM fluid loss (around 12.5 mL). SiO_2 NPs at concentration above 0.5wt%, similar to CuO NPs, caused the fluid loss increased to 14.2 wt%. However, SiO2 NPs as compared to CuO NPs is better candidate for the Bentonite-WBM fluid loss reduction. Finally, after adding different concentrations of TiO_2 NPs to the Bentonite-WBM, it was observed that the fluid loss significantly decreased to 11.4 mL. Accordingly, TiO_2 NPs as compared to the rest is the best additive for the Bentonite-WBM fluid loss reduction.

3.2.5. The quality and thickness of the filter-cake

For evaluation of filter cake quality toughness, slickness and hardness of mud should be considered. The quality of filter cake is one of the factors that should be attended in drilling operation. The quality is the representative factor of amount of filter loss and in drilling operation should be prevented from fluid loss to guarantee the wellbore stability. For this purpose, some additives should be added to drilling fluid to decrease the filter loss.

From the experiment it was noticed that increasing Al_2O_3 NPs concentration resulted in heavy filter cake thickness but low quality especially at higher concentrations. Therefore, it can be inferred that Al_2O_3 NPs cannot form an effective seal for controlling filtration loss.

CuO NPs revealed decreasing trend by increasing concentration up to 0.1wt% and then has an increasing trend in making filter-cake. And with the laboratory analyzes CuO in 1wt% has the higher fragility in the filter-cake. On the other hand, TiO_2 shows decreasing trend in making filter-cake. The quality of TiO_2 filter-cake was smoother and better particle compression also this filter-cake has not easily cracked. In the lower concentration (0.05wt%) filter-cake of TiO_2 has suitable quality. After 16 hours, filter-cake in the higher concentration twitched but in lower concentration has more flexibility. Also, it has a suitable quality rather than CuO and Al_2O_3.

SiO_2 shows increasing trend in making filter-cake by increasing concentration. In the lower concentrations (below 0.05w%), the quality of filter-cake is relatively better and more flexible with less friability.

In these tests, it was observed that with increasing in the concentration, the quality of the filter cake was improved notably only for TiO_2 NPs which have the lowest fluid loss among others. The better filter cake quality and lower fluid loss after TiO_2 belong to SiO_2, CuO, and Al_2O_3 NPs, respectively.

4. Conclusion

The effects of different concentrations of Al_2O_3, TiO_2, SiO_2 and CuO NPs on rheology of a Bentonite water based mud were determined. Generally, the applied NPs in this study were found to be suitable for the use in drilling mud due to its functional characteristics of maintaining low viscosity without compromising the density requirement and thus expected to minimize drilling problems. Plastic viscosity, yield point and gel strength of the Bentonite-WBM were increased by adding Al_2O_3 NPs that are favorable. Al_2O_3 NPs also increased the Bentonite-WBM fluid loss which is undesirable. Therefore, utilizing Al_2O_3 NPs to improve the Bentonite-WBM rheology is not highly recommended. TiO_2 and CuO NPs resulted in decreasing of plastic viscosity, yield point and gel strength. These two NPs especially at concentrations below 0.5wt% are recommended to be applied as additives to improve the Bentonite-WBM rheology. SiO_2 NPs revealed a good improvement in the Bentonite-WBM rheology and acceptable enhancement in the characteristics of the final mud product in terms of filter loss and filter cake. They are thereby highly recommended to be included in the mud formulation especially for drilling of formations which swell while contacting with WBM (e.g. shales) and those formation rocks having low shear strength (e.g. unconsolidated sands). Overall results show that NPs can be added to Bentonite-WBM to enhance the properties of drilling fluids. However, the significant point is NPs concentrations which plays a vital role in their usage.

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Fig. 1: Schematic of filter press apparatus

Fig. 2: Shear stress against the shear rate of the base mud at 27 $_o$C

Fig. 3: TEM and XRD analyses from nanopowders: (a) Al_2O_3, (b) TiO_2, (c) CuO, and (d) SiO_2

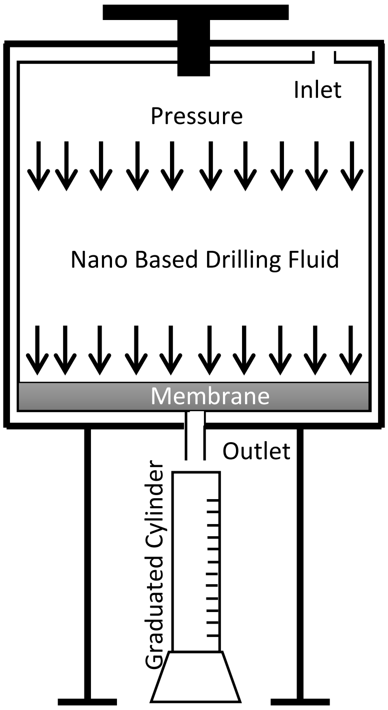
Fig. 4: FESEM (up) and EDX (down) analyses results from bentonite particles

Fig. 5: Behavior of Bentonite-WBM plastic viscosity in presence of different NPs

Fig. 6: Yield point of NPs Bentonite-WBM at different concentrations (CuO, TiO_2 and SiO_2 NPs Bentonite-WBM from left Y-axis and Al_2O_3 NPs Bentonite-WBM from right Y-Axis)

Fig. 7: Bentonite-WBM Gel strength in the presence of various NPs measured in: (a) 10 sec and (b) 10 min (CuO, TiO_2 and SiO_2 NPs Bentonite-WBM from left Y-axis and Al_2O_3 NPs Bentonite-WBM from right Y-Axis)

Fig. 8: The Bentonite-WBM fluid loss in the presence of NPs at various concentrations (CuO, TiO_2 and SiO_2 NPs Bentonite-WBM from left Y-axis and Al_2O_3 NPs Bentonite-WBM from right Y-Axis)



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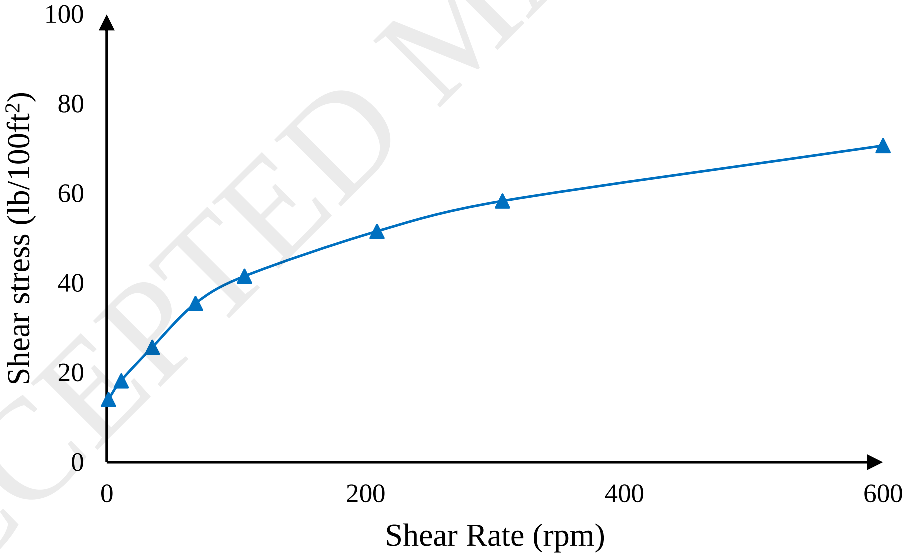

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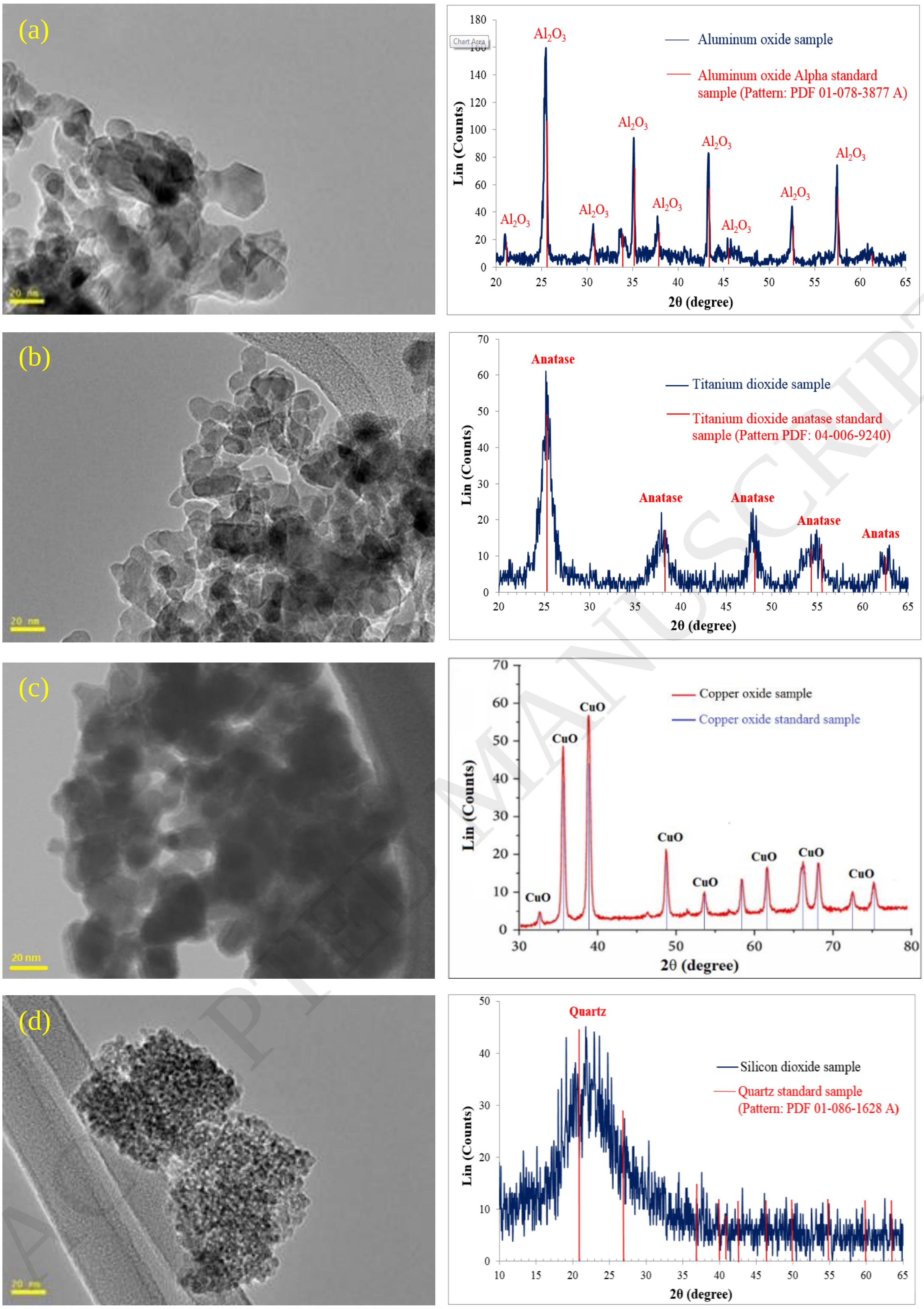

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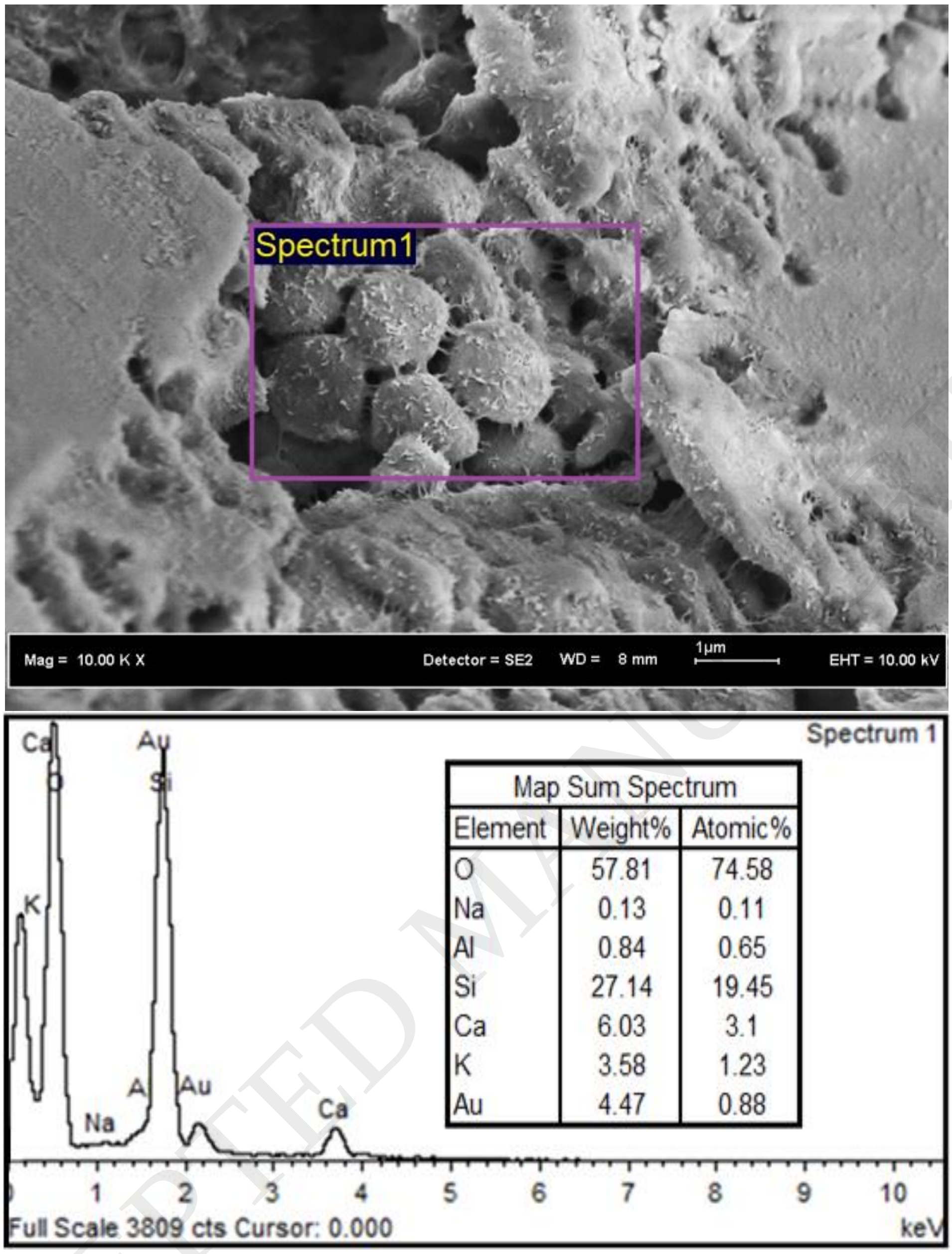
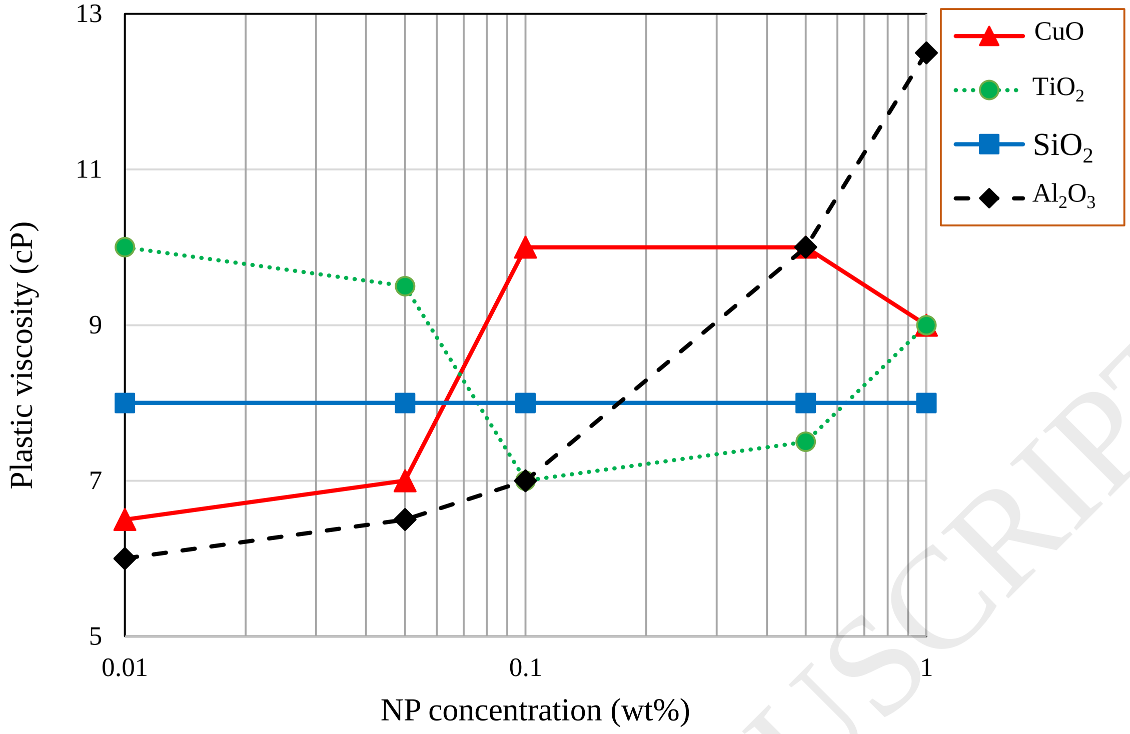


Map Sum Spectrum

Element	Weight%	Atomic%
O	57.81	74.58
Na	0.13	0.11
Al	0.84	0.65
Si	27.14	19.45
Ca	6.03	3.1
K	3.58	1.23
Au	4.47	0.88

Fig. 4: FESEM (up) and EDX (down) analyses results from bentonite particles



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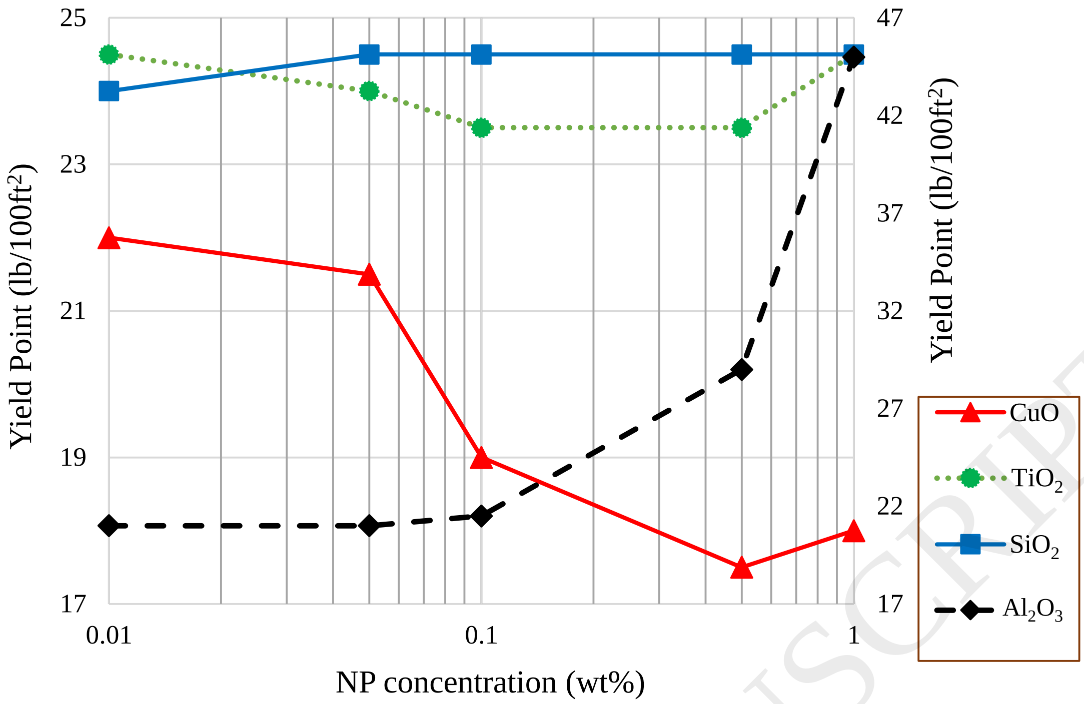
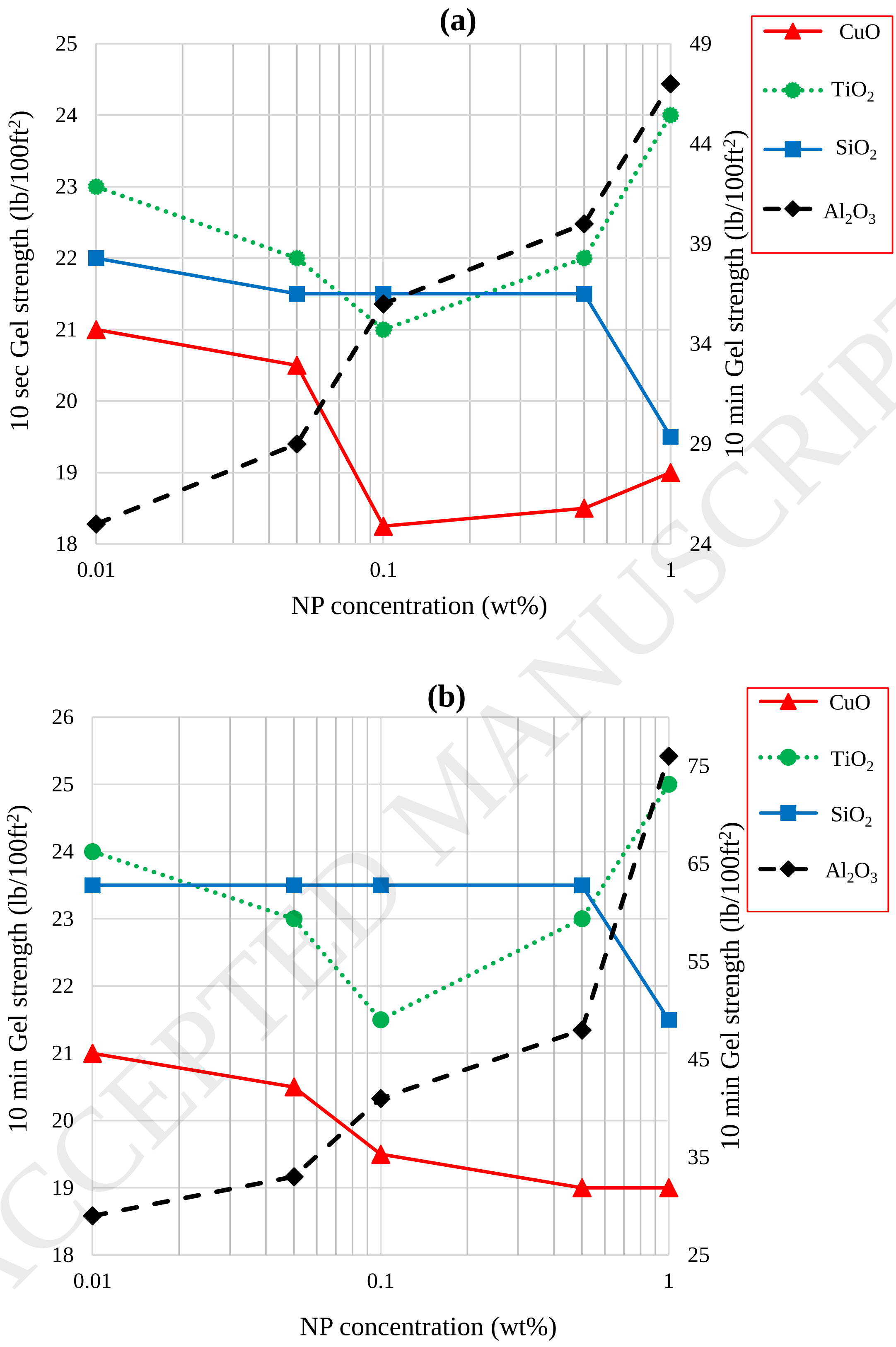


Fig. 6: Yield point of NPs Bentonite-WBM at different concentrations (CuO, TiO_2 and SiO_2 NPs Bentonite-WBM from left Y-axis and Al_2O_3 NPs Bentonite-WBM from right Y-Axis)



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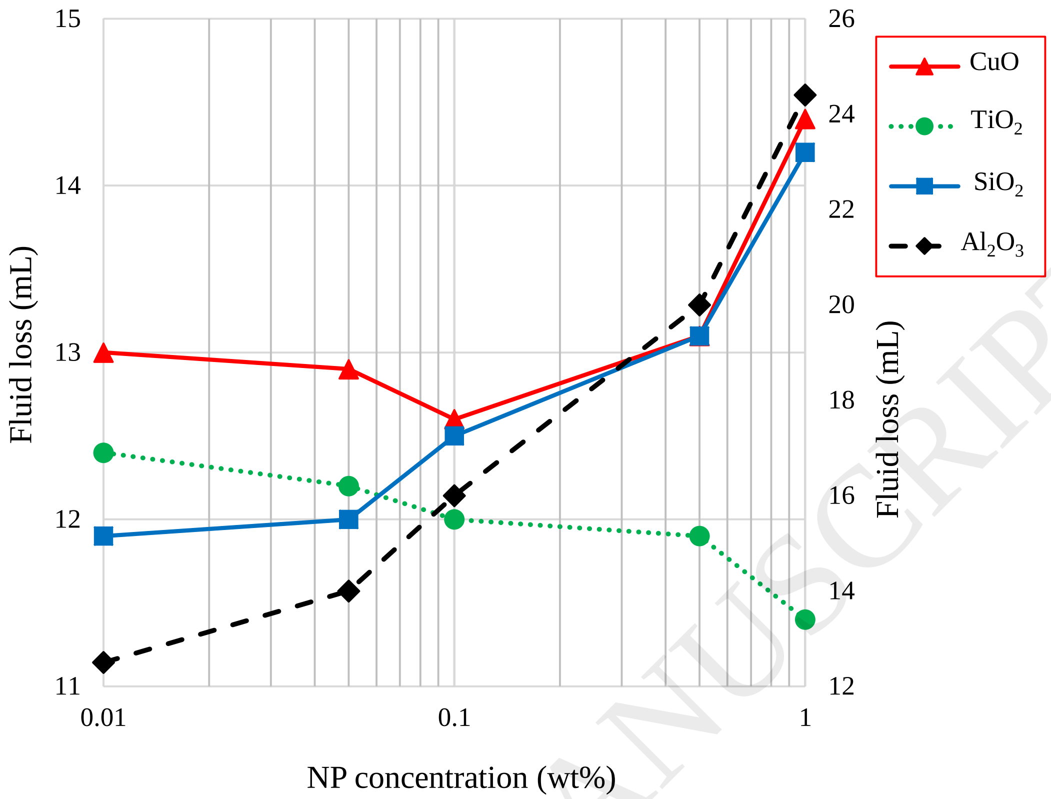

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