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FULL LENGTH ARTICLE

# Enhancing the rheological properties and shale inhibition behavior of water-based mud using nanosilica, multi-walled carbon nanotube, and graphene nanoplatelet



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## KEYWORDS

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Drilling muds

**Abstract** Five different drilling mud systems namely potassium chloride (KCl) as a basic mud, KCl/partial hydrolytic polyacrylamide (PHPA), KCl/graphene nanoplatelet (GNP), KCl/nanosilica and KCl/multi-walled carbon nano tube (MWCNT) were prepared and investigated for enhancement of rheological properties and shale inhibition. Nanoparticles were characterized in drilling mud using transmission electron microscope (TEM) analysis. Mineralogical analysis of shale was examined by X-ray diffraction (XRD). Five shale plugs were prepared using compactor cell for the determination of shale swelling. Shale swelling was determined using the linear swell meter (LSM) for 20 hours. Results revealed that basic mud and KCl/polymer mud systems shows 30% and 24% change in shale volume. MWCNT, nanosilica and GNP were added separately in the KCl mud system. 0.1 ppb of each MWCNT and nanosilica showed 32% and 33% change in shale volume. However, when the shale was interacted with WBM containing 0.1 ppb of GNP, it was found that only 10% change in shale volume occurred. The results showed that the addition of nanoparticles in the KCl mud system improved the shale inhibition. API, HPHT filtrate loss volume, plastic viscosity (PV) and yield point (YP) were improved using GNP. It is learned from the experimental work that small concentration of KCl with GNP can mitigate shale swelling compared to the mud contains higher concentration of KCl and PHPA in WBM. Thus, GNP can be a better choice for enhancement of WBM performance.

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**Nomenclature**

API	American Petroleum Institute
FW	fresh water
FL	fluid loss
GNP	graphene nano platelet
GS	gel strength
HPHT	high pressure high temperature
KCl	potassium chloride
LSM	linear swell meter
PV	plastic viscosity
MWCNT	multi-walled carbon nano tube
NaOH	sodium hydroxide or caustic soda
OBM	oil-based mud
PAC	poly anionic cellulose
PHPA	poly hydrolytic polyacrylamide
RPM	rotation per minute
SBM	synthetic-based mud
SDS	sodium dodecyl sulfate
TKPP	tetra-potassium pyrophosphate
WBM	water-based mud
YP	yield point

*Units*

cc	cubic centimeter
cp	centipoise
ft	feet
g	gram
minutes	min
mPa s	milli pascal's
nm	nanometer
ppb	pound per barrel
ppg	pound per gallon
Pa s	pascal's
sec	seconds
μm	micrometer

*Units conversion*

$$\frac{1 \text{ lb}}{100 \text{ ft}^2} = 0.4788 \text{ Pa s}$$

$$1 \text{ cp} = 1 \text{ mPa s}$$

$$1 \text{ ppb} = \frac{1 \text{ g}}{350 \text{ cc}}$$

**1. Introduction**

Drilling operation requires an extra care in well monitoring, rig hoisting, rig power, and most importantly well control system. Proper handling of well control system is only possible by well control equipment such as blow-out preventer and proper formulation of drilling muds [1,2]. Functions of drilling mud are to maintain the hydrostatic pressure when formation pressure exceeds the drilling mud pressure, to cool drill bit when drilling in hard geological formations for longer time, and to suspend and transport drilled cuttings from subsurface to surface. However, these functions can be well performed with the proper treatment of drilling muds rheology [3]. Furthermore, rheological properties of drilling muds such as mud density, PV, apparent viscosity (AV), YP, gel strength, mud filtrate loss volume and lubricity are important to maintain for an efficient drilling operation and wellbore stability.

Shale causes world's 70% of wellbore instability problems. Shale instability is caused due to presence of clay minerals into the shale. These clay minerals in particular kaolinite, smectite and montrolite have great affinity with the water [4]. However, clay minerals start to swell after they interact with the water and as a result, clay swelling raised the wellbore instability such as shale sloughing, tight hole, caving and reduce efficiency of mud to lift the drilled cuttings. Clay swelling reduces the rate of penetration (ROP) due to bit balling with sticky clay [5]. Previously, Reid et al. [6] determined shale swelling behavior of north sea fields by interacting with different types of drilling muds. It was found that performance of tetra-potassium pyrophosphate (TKPP) was equivalent to OBM. However, TKPP muds shown mud accretion problems. Traditionally, KCl and PHPA are used to minimize the shale swelling problems. Somehow, KCl mud performance is good for shale swelling inhibition but the use of high concentration of KCl in drilling mud is strictly prohibited due to environmental concerns [7].

Beside that polymers such as acrylamide and PHPA are good heat insulators and used for prevention of mud filtrate, and inhibition of clay swelling [8]. These polymers cannot sustain high pressure high temperature (HPHT) downhole conditions [3]. Oil-based mud (OBM) and synthetic-based mud (SBM) are widely used for shale inhibition and considered as good drilling lubricants. OBM and SBM minimized the shale swelling because of less water content in their composition. Usage of OBM in environmental altered areas is considered to be illegitimate [9]. There is no doubt that OBM came up with excellent shale inhibition properties, but it raised some operational problems such as it disturbed well logging data, and sometimes it raises formation damage [10,11]. Therefore, oil and gas industry is more interested in WBM. It is used to drill almost 80% of all wells. It contains about 80% of water phase and 20% drilling additives. High water content drilling muds normally result in high friction and mud filtrate volume, low PV, and a great affinity with shale which leads to wellbore instability problems. Sehly et al. [12] found the way to minimize the concentration of KCl in WBM and reduced to environmental acceptable limit. Rodrigues et al. [13] used the multi functional polymers to modify rheological and shale inhibition properties of drilling muds. Moreover, Abdou et al. [14] evaluated Egyptian bentonite and nano bentonite as a drilling mud. It was found that use of local bentonite and nano bentonite is not suitable without using necessary drilling mud additives.

Currently, the technical challenge is faced by the oil and gas drilling sector to prepare drilling muds to improve rheological properties and shale inhibition at high temperature conditions. Conventional WBM contained shale stabilizers or conventional inhibitors are heat insulators, macro size and can not plug nanopores of shale. Therefore, water invades into the wellbore, and results in high mud filtrate volume and clay swelling. Nanoparticle can be an excellent solution to plug nanopore size of the shale. Various investigators reported the

**Table 1** Physical properties of GNP.

Properties	Typical value
Size	< 0.1–2 μm
Thickness	1.46–3.54 nm
Appearance	Gray powder
Carbon	99.5 wt%
Thinner	Distilled water and SDS
Mixing and dilution	Distilled water

**Table 2** Physical properties of MWCNT.

Properties	Typical value
Appearance	Black powder
Purity	> 97%
Surface area	230–300 m <sup>2</sup> /g
Ash	< 0.2wt%
Amorphous carbon	< 2 wt%
Thinner	Distilled water and SDS
Mixing and dilution	SDS is added for homogeneous dispersion

**Table 3** XRD of shale.

Mineral	Weight %
Montomorillonite	64
Kaolinite	11
Cristobalite	19
Quartz	4

nanoparticle based drilling muds for enhancement of rheological properties [15–17], plugging characteristics [18], and shale inhibition [19].

In this paper, experimental work has been conducted to minimize shale swelling and improve rheological performance of WBM using GNP, MWCNT and nanosilica in WBM. Behavior of nanoparticles in drilling muds was studied by TEM. Rheological and shale inhibition performance of nanoparticles based drilling muds is compared with conventional KCl and KCl/PHA mud systems.

## 2. Methodology

The methodology discussed in this paper was based on the laboratory work. All the drilling mud testing work was carried out as per recommended practice *API 13B-1* for investigating WBM [20].

### 2.1. Material selection

Drilling mud additives such as KCl, caustic soda, flowzan, PAC, PHPA, and barite were provided by Scomi Oiltools. However, GNP, MWCNT, and nanosilica were purchased from Ugent tech Sdn Bhd.

#### 2.1.1. Graphene nano platelets

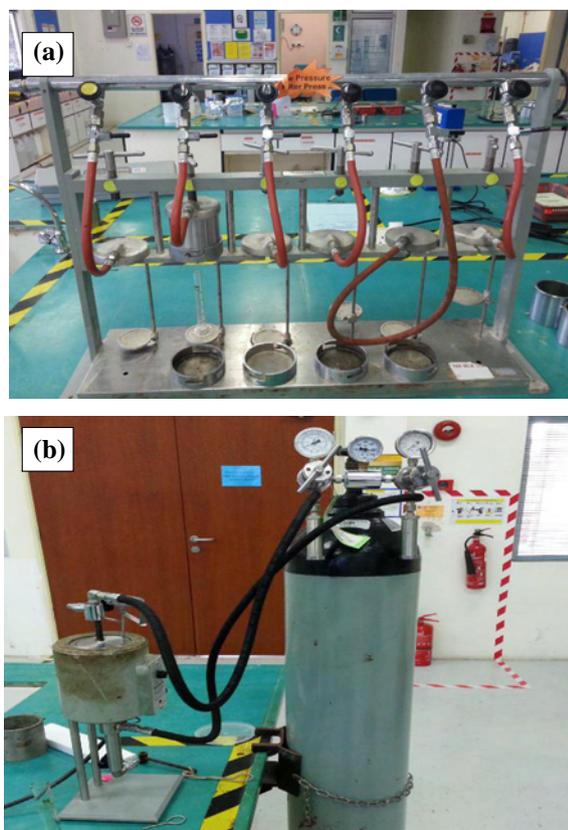
GNP is colloidal suspended particles in aqueous solution produced by the chemical modification of graphite. GNP has multi functional properties and it consists of 3–8 layers (average thickness), diameter of particle ranging from sub micron to



**Figure 1** Fann rheometer with Baroid hot cup.

**Table 4** Formulation of drilling muds.

Materials	KCl/ PHPA WBM	Basic WBM	Nanosilica WBM	GNP WBM	MWCNT WBM	Mixing time
Fresh water, ml	195	195	195	195	195	–
KCl, ppb	34.1	11.3	11.3	11.3	11.3	3
NaOH, ppb	0.2	0.4	0.4	0.4	0.4	2
Flowzan, ppb	0.3	0.6	0.6	0.6	0.6	5
PAC, ppb	3.7	3.7	3.7	3.7	3.7	5
PHPA, ppb	7	–	–	–	–	10
Barite, ppb	200	200	200	200	200	30
Nanosilica, ppb	–	–	0.1	–	–	5
GNP, ppb	–	–	–	0.1	–	5
MWCNT, ppb	–	–	–	–	0.1	5



**Figure 2** (a) API filtrate volume tester, and (b) HPHT filtrate volume tester.

50  $\mu\text{m}$  and surface area is 750  $\text{m}^2/\text{g}$ . It signifies the new class of nanocarbon. GNP can improve matrix material such as surface hardness, stiffness and strength. Such attributes of GNP fascinated to use for clay swelling inhibition and wellbore strengthening. Physical properties of GNP are shown in Table 1.

#### 2.1.2. Multi-walled carbon nanotube

MWCNT has a unique structure. It could be metallic or semiconductor depends upon the diameter and chirality of tube. MWCNT is made up of tens of graphene sheets [21]. Physical properties of the MWCNT are shown in Table 2.

## 2.2. Characterization of the materials

Nanosilica mud, GNP mud, and MWCNT mud were characterized using TEM. 0.1 g of each nanoparticles was separately added in 40 ml of diluted drilling muds and sonicated for 30 min before carrying out TEM analysis. TEM was conducted using biological-TEM Hitachi model no. HT7700. Shale was characterized using XRD. It was determined using Rigaku smart lab X-ray diffractometer R&D 100. XRD of shale is shown in Table 3.

## 2.3. Preparation of drilling muds

Basic WBM was prepared by adding fresh water, barite, KCl, NaOH, flowzan, and PAC. The composition of muds, mixing time, and amount of drilling muds additives are given in Table 4.

## 2.4. Preparation of homogeneous colloidal dispersion of nanoparticles

GNP, nanosilica and MWCNT were added at concentration of 0.1 ppb after barite in the mud formulation. Before adding nanoparticles in WBM, the nanoparticles was dispersed in



**Figure 3** Ofite lubricity tester.

**Table 5** Experimental conditions for the measurement of shale swelling, rheological properties, lubricity and API filtrate volume.

Drilling mud systems	Operating conditions
(1) Basic WBM at 11 ppb of KCl, 0 ppb of KCl, and 0 ppb of PHPA	Rheological properties and lubricity were determined at ambient conditions
(2) WBM + Nanosilica at 0.1 ppb of nanosilica, 11 ppb of KCl, and 0 ppb of PHPA	Filtrate loss volume was determined at API conditions (100 psi and ambient temperature). Shale swelling was determined at ambient temperature and pressure
(3) WBDF + GNP at 0.1 ppb of GNP, 11 ppb of KCl, and 0 ppb of PHPA	
(4) WBM + MWCNT at 0.1 ppb of MWCNT and 0 ppb of KCl	
(5) WBM + KCl+PHPA at 34 ppb of KCl and 3 ppb of PHPA	

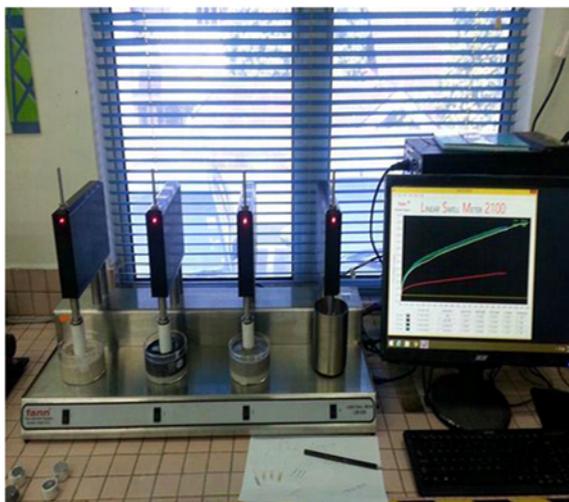


Figure 4 Fann linear swell meter.

2.5. Rheological properties

Rheological properties such as PV, YP, 10-s gel strength (10-s GS), and 10-min gel strength (10-min GS) were determined using rheometer as shown in Fig. 1. API filtrate and HPHT filtrate volume were obtained using low pressure filter press and HPHT filtrate volume tester as shown in Fig. 2(a) and (b). Total five drilling muds were investigated for rheological and shale swelling behavior. Experimental conditions for the determination of rheological and shale swelling are given in Table 5. HPHT filtrate volume was found at 500 psi, 250°F. PV and YP were calculated by Eqs. (1) and (2).

$$PV = \Phi_{600} - \Phi_{300} \tag{1}$$

$$YP = \Phi_{300} - PV \tag{2}$$

whereas,

$\Phi_{600}$  = dial reading at 600 RPM, and  $\Phi_{300}$  = dial reading at 300 RPM

Coefficient of friction (CoF) was determined by using lubricity tester as shown in Fig. 3. Lubricity was calculated by using Eqs. ((3)–(6)).

$$CoF = \frac{Torque\ reading}{100} \tag{3}$$

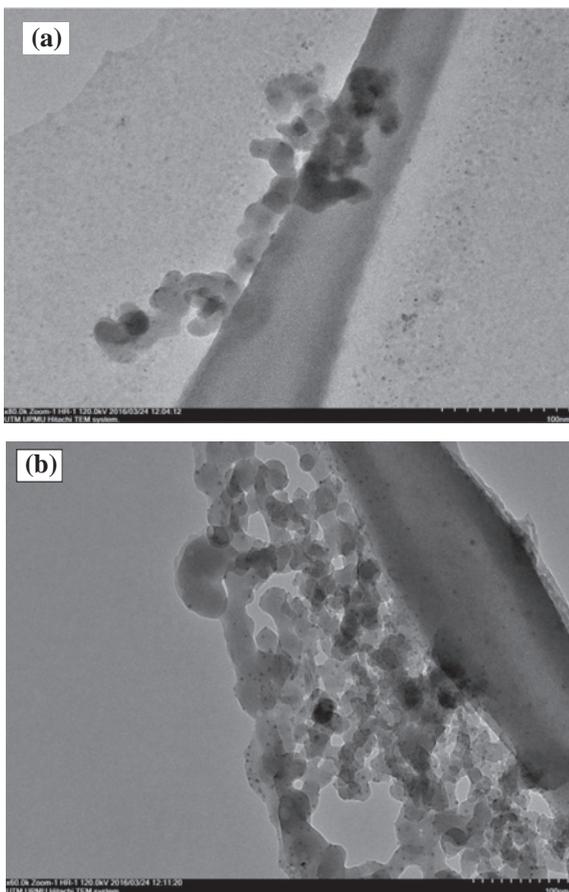


Figure 5 (a) and (b) TEM images of nanosilica in drilling mud.

SDS surfactant. 0.1 g of each nanoparticles were dispersed in the 50 ml of reagent bottle containing 20 ml distilled water and 0.1 ml of the surfactant. The reagent bottles were placed in an ultra sonicator for 30 min until homogeneous dispersion of nanoparticle in the solution can be seen.

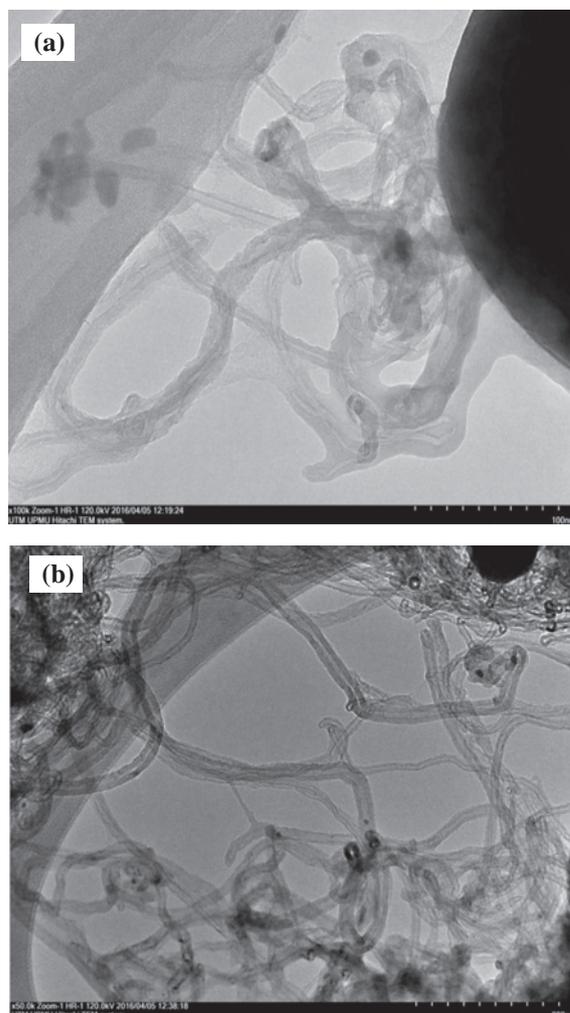
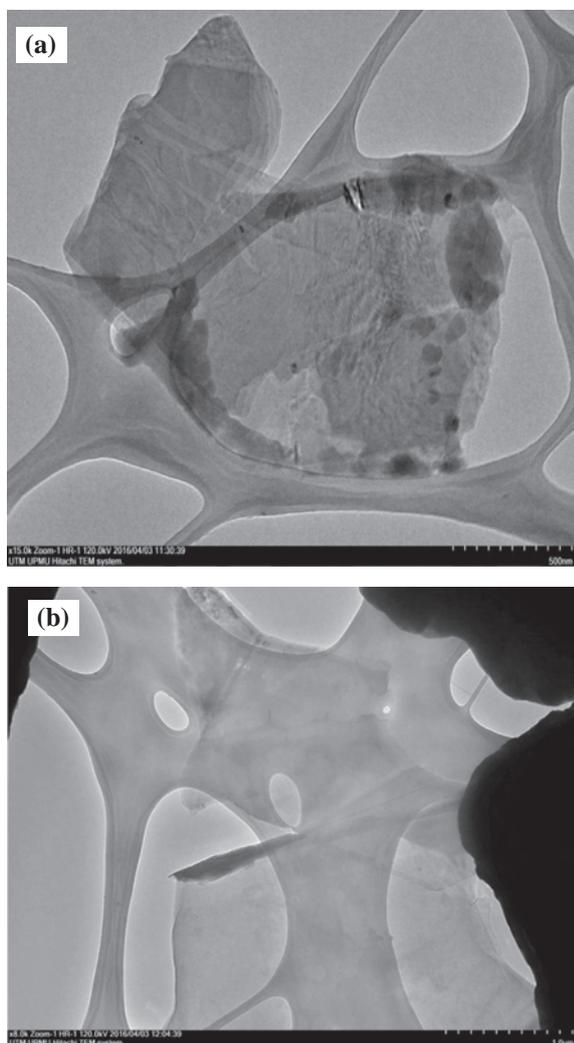


Figure 6 (a) and (b) TEM images of MWCNT in drilling mud.



**Figure 7** (a) and (b) TEM images of GNP in drilling mud.

with instrument set at 60 RPM and pressure of 100 lbs, which are

$$100 = \frac{150 \text{ inch} - \text{lbs torque wrench reading}}{1.5 \text{ inch torque shaft lever arm}} \quad (4)$$

$$CF = \frac{\text{Meter reading for water (standard)}}{\text{meter reading obtained in water calibration}} \quad (5)$$

$$CoF = \frac{(\text{Meter reading for water}) (CF)}{100} \quad (6)$$

whereas, CoF = Coefficient of friction, and CF = Coefficient factor

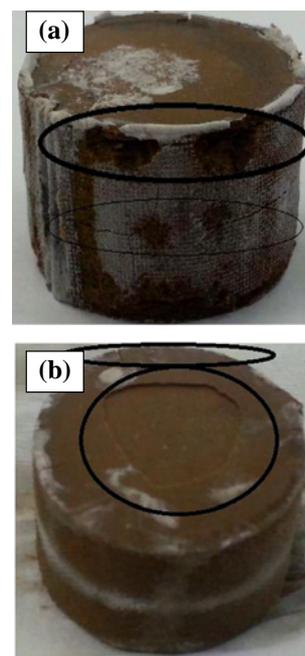
## 2.6. Linear shale swelling

Prior to start of drilling operation, it is very important to know the compatibility of drilling muds with the wellbore. The method to examine the compatibility of the shale swelling is to interact the shale with the drilling muds. In this study, shale plugs were prepared using compactor cell for swelling test. Procedure for determination of the shale swelling test is provided in linear swell meter (LSM) 2100 instructional manual [22]. Experimental set up for shale LSM 2100 is shown in Fig. 4.

## 3. Results and discussion

### 3.1. Characterization

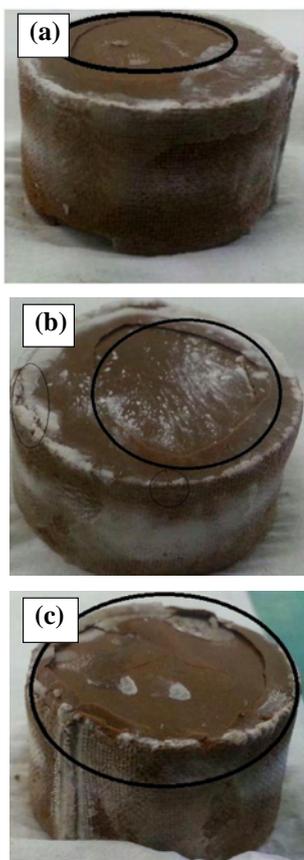
Typical TEM images for nanosilica in drilling mud are shown in Fig. 5(a) and (b) which indicates the particle nature of nanosilica and no damage in the morphology is observed in the drilling mud. The MWCNT seem to be thin porous in



**Figure 8** (a) Shale immersed in basic mud, and (b) shale immersed in KCl/PHPA mud.

**Table 6** Rheological properties and filtrate volume (API and HPHT).

Drilling muds	PV (mPa s)	YP (Pa)	10-s GS, (Pa)	10-min. GS, (Pa)	CoF	API FL (ml)	HPHT FL (ml)
WBM	22	13	4.5	5	0.2	6	16
WBM + MWCNT 0.1 ppb, KCl 11 ppb	23	14	4.5	5	0.1	5.6	15
WBM + Nanosilica 0.1 ppb, KCl 11 ppb	21	12	4.5	5	0.2	5.8	17
WBM + GNP 0.1 ppb, KCl 11 ppb	23	14	4.5	5	0.1	5.5	14
WBM + KCl + PHPA KCl 34 ppb, PHPA 3 ppb	24	15	5	5.5	0.2	5.5	15



**Figure 9** (a) Shale immersed KCl/GNP mud, (b) shale immersed in KCl/MWCNT mud, and (c) shale immersed in KCl/nanosilica mud.

morphology and the shape is retained even in the use of drilling mud as shown by TEM study in Fig. 6(a) and (b). TEM images of GNP are provided in Fig. 7(a) and (b) which shows that clear view of graphene is described by TEM analysis.

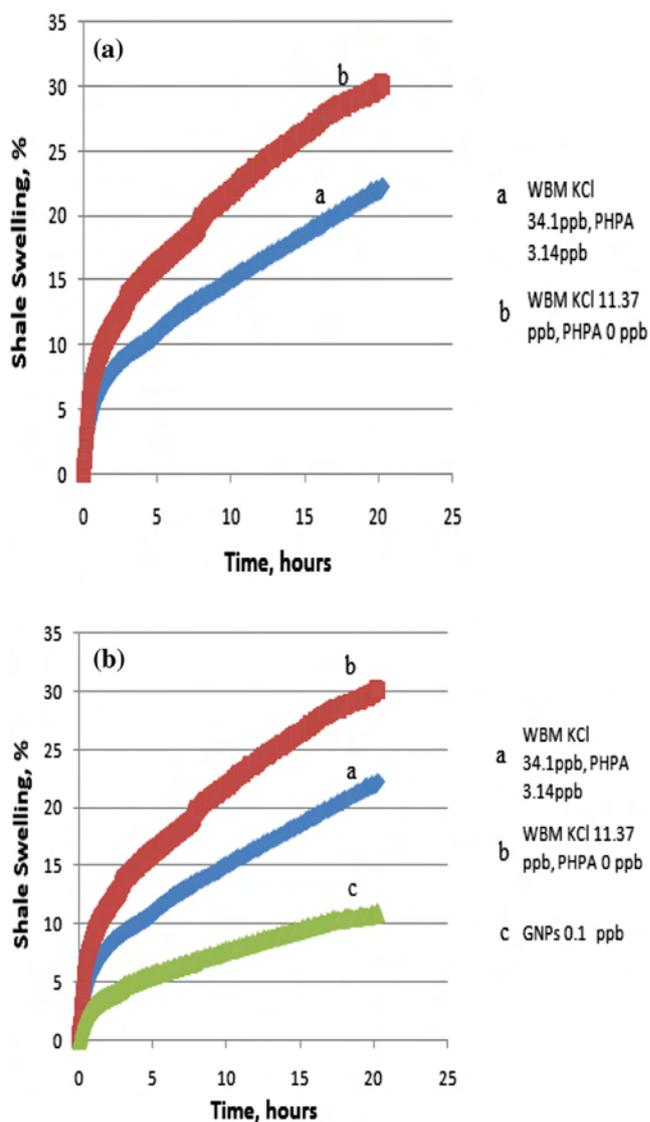
3.2. Rheological properties of drilling mud

Rheological properties such as PV, YP, 10 s-GS, 10 min, CoF, API mud filtrate, and HPHT filtrate volume of the reported drilling muds were determined and given in Table 6. It can be seen in the Table 6 that addition of nanoparticles has improved the rheological performance of WBM. However, among all nanoparticles based drilling muds, GNP mud revealed better rheological performance. PV improved with the addition of GNP as shown in Table 6. It may be GNP provides better PV because resistance in flow due to friction between the nanoplatelets, micro additives and the liquid medium of the mud. PV increases with the increase in concentration of the solids present into the drilling mud. It has been observed that API and HPHT mud filtrate volumes were slightly reduced with the addition of GNP and MWCNT as shown in Table 6. It is possible that nano size additives with enhanced structures such as nanoplatelets and nanotubes sealed the nanopore throats of the wellbore formation to prevent water infiltration [23]. GNP and MWCNT are a good conductor of heat thus mud filtrate volume reduced

compared to basic mud filtrate volume at 250°F and 500 psi conditions.

3.3. Immersion test

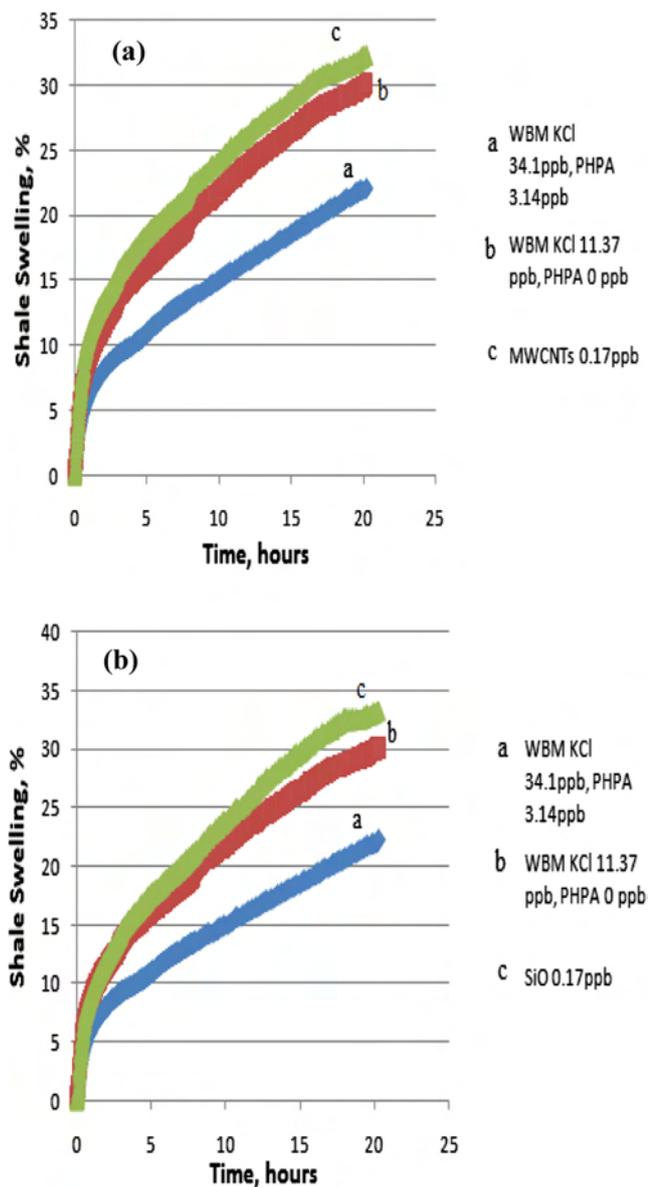
Shale was immersed in KCl, KCl/Polymer and KCl/nanoparticles mud systems for at least 20 hours to analyze the effect of drilling muds on rock-fluid interaction. Basic mud or mud contains KCl 11.37 ppb, PHPA 0 ppb showed high erosion along the boundary and some cracks on shale as shown in Fig. 8(a). The similar results were obtained when using KCl of 34.1 ppb, and PHPA of 3.14 ppb as shown in Fig. 8(b). However, nanosilica drilling mud shows that shale was slightly deteriorated at the center with erosion along the boundary as shown in Fig. 9(c). Moreover, KCl/MWCNT system shows small crack at the center of the plug as shown in Fig. 9(b). However, minimum erosion and cracks have been seen on the body of shale by using GNP shown in Fig. 9(a).



**Figure 10** (a) Shale interacted with basic mud or KCl mud, and KCl/PHPA (b) shale interacted with KCl/GNP mud and compared with basic and KCl/PHPA muds.

### 3.4. Behavior of shale swelling with different types of drilling muds

Shale was interacted with different drilling muds such as KCl, KCl/Polymer, KCl/nanoparticles mud systems. When the shale was exposed to the WBM containing 34.1 ppb of KCl, and 3.14 ppb of PHPA, it was found that 22% of volume change occurred in the shale as shown in Fig. 10(a). Change in shale volume became greater when KCl concentration was reduced to 11.37 ppb and polymer 0 ppb. It showed 31% of shale volume has been changed as shown in Fig. 10(a). Later, same shale was interacted with KCl mud containing 0.1 ppb of nanosilica, it shows that 33% of shale volume has been changed as shown in Fig. 11(b). In this study, nanosilica swelling percentage of shale was considered to be high among all designed drilling muds. When, WBM containing 11.37 ppb



**Figure 11** (a) Shale interacted with KCl/MWCNT and compared with basic and KCl/PHPA muds, and (b) shale interacted with nanosilica and compared with basic mud, and KCl/PHPA muds.

of KCl and 0.1 ppb of MWCNT was interacted with shale, it was found that 32% of the volume has been changed as shown in Fig. 11(a). Nonetheless, when shale was interacted with the similar KCl mud but with 0.1 ppb of GNP, it showed only 10% change in shale volume as shown in Fig. 10(b), which is considered to be a minimum change in shale volume among the all the reported drilling muds. GNP contains a good nanoplatelets structure as discussed in the characterization. It may be possible nanoplatelet formed a potential wall on the nanopore of the shale to prevent invasion of the water.

## 4. Conclusion

Shale plug immersed in GNP mud shows less erosion and cracking along the boundary and at the center of the shale plug compared to basic mud, KCl/PHPA, KCl/nanosilica, and KCl/MWCNT mud systems. However, KCl/PHPA mud system shows good shale inhibition compared to KCl mud system. GNP mud provides better shale inhibition and rheological properties compared to all drilling muds systems reported in this study. API and HPHT filtrate volumes were minimized using GNP mud. CoF was minimized by using GNP and MWCNT muds. However, further studies are required to investigate the effect of GNP, MWCNT, and nanosilica at higher concentrations over shale swelling and rheological behavior of the muds.

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