

Evaluation of Polyacrylamide grafted Starch/Clay Nanocomposite as a potential Drilling Fluid additive in Inhibited Water Based Drilling Fluid System

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Abstract

The wellbore instability on account of high shale activity has to be mitigated to optimise the problem. In this research, Polyacrylamide grafted Starch/Clay Nanocomposite (PSGCNC) was synthesized and used as drilling fluid additive. The mechanism adopted for the synthesis of the nanocomposite was the free radical polymerization and it was characterized by Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis and XRD. During its synthesis, the ratio of clay to grafted PASG monomer were varied and the effects of the synthesized product on the filtration and rheology of the drilling fluid system was observed and studied rigorously. Optimistically at last, the most suitable grade of PSGCNC was used as a drilling fluid additive and its effects were studied. The experimental investigations revealed that PSGCNC exhibited superior shale encapsulation property than partially hydrolysed polyacrylamide (PHPA), an often-used clay encapsulator, and also has better control on HPHT wells. Hence, the synthesized nanocomposite may be utilized as a drilling fluid additive in inhibitive water-based drilling fluid system.

Key Words: Polyacrylamide Grafted Starch, Clay Nanocomposite, Inhibited water based drilling fluid system, Thermogravimetric analysis, HPHT well, XRD, FTIR.

1. INTRODUCTION

Shales have an affinity towards water and has spaces in their matrix that causes fluid-rock interactions and the invasion of fluid may accelerate physio-chemical reactions which causes unessential swelling of the clay blocks leading to wellbore instability problems and is the prime reason behind opting for oil-based muds for shale formation rather than water-based muds. But regulations regarding oil-based muds is that it has unrecoverable damage on environment. So, the most cited way to mitigate wellbore instability problem is the use of inhibited water-based mud.

Choline Chloride (CCI) salt has been widely used as a successful shale inhibitor in water-based mud systems. Polyacrylamide (PAA), a polymer, has a much wider use due to its property of shale encapsulation. Presence of salt (CCI) increases the encapsulation effect of PAA. However, the negative effects of salinity increase on polymer is portrayed in its mechanical degradation as the polymer chain coils up and break. In addition, it also undergoes damage under high temperature. Hence, it is necessary to increase the mechanical and thermal characteristics of the PAA for using it in drilling mud. Also, there were citations by several acknowledged researchers, about the use of nano-fluid based mud drilling systems which also has a much higher well strengthening capacity. Hence the nano-composites have now become the centre of research and captured attentions by companies for use in drilling fluid as an effective mud-system.

Nano-clays are nanoparticles with mineral silicates layered structure which are further categorised as montmorillonite, Kaolinite, illite, attapulgite, smectite, sepiolite, hectorite etc depending on their chemical morphology. As of now the nanoparticles are used to prepare nano-composite which can be used as a rheology modifier etc. So, it is now coined about the incorporation of nano-clay into the polymer matrix to improve the desired properties of mechanical strength and high temperature resistance in the polymer which can be done by mixing, rigorous stirring, and sonication. The polymerization of the grafted structure of polyacrylamide and starch may result in an exfoliated nano-structure which is desirable due to its versatile properties and may be utilized as a drilling fluid additive.

In this study, polyacrylamide and starch grafted clay nano-composite was developed by free radical polymerisation technique with potassium persulfate (KPS) as an initiator. The characterization of the nano-composite was done using Fourier transform infrared spectra (FTIR) and Thermogravimetric analysis (TGA). The synthesised nano-composite was used as one of the additives in a water based inhibited drilling mud system. Further, for more authenticity the shale recovery performance test was carried out for the evaluation of the prepared Polyacrylamide and starch grafted/ clay based nano-composite as a drilling fluid additive in inhibited mud system.

Higher temperature at deeper depth shows chemical changes which may lower the drilling fluids inner integrity. To prevent this, degradation, smart fluids for drilling operations are being conducted worldwide on utilizing nanoparticles, used water-based drilling fluid by addition of synthesized nanocomposite with different concentration and monitored the improvement on rheological, filtration and thermal conductivity through standard methods for both deionized water and salty water. Nano-technology's application in the oil and gas diving is extremely diverse and irreplaceable and may show new benchmark in the domain of zones. TiO₂/polyacrylamide nano-composite can be used as an effective additive in the water based drilling fluid [4]. It is projected that the dissolve nanoparticle based drilling fluids have considerable operational performance under downhole conditions.[11] TiO₂/polyacrylamide nanocomposite were used as an effective additive in the water based drilling fluid for the purpose of rheological property improvement, addition of these particles also results in decrease in fluid loss and mud cake thickness [19]. **Rajat et al.** use polyacrylamide- grafted- polyethylene glycol/ silica nanocomposite as potential additive in water based drilling mud for reactive shale formation [14]. Polymeric nano composite carbon nano tube and grapheme nanoplatelets were used as additives to enhance the rheological and filtrate loss properties [17]. Polyacrylamide- grafted- polyethylene glycol/ silica nanocomposite were used as an additive in water based mud for reactive shale formation [14]. Nanoparticles can be used in improving the wellbore instability due to swelling / hydration of reactive shales [8]. Drilling fluid properties like viscosity, yield point, gel strength, density can be improved using clay nanoparticles in drilling fluid [9]. In the recent years several studies on the effect of nanoparticles on the properties of drilling fluid were performed needs to be improved is drilling fluid. High pressure high temperature conditions results in the deterioration of fluids rheological and filtrate loss properties, limiting tool and down hole equipment selection, determining downhole pressure becomes difficult, chances of lost circulation increases.

2. MATERIAL AND METHODS

The preparation and formulation of Inhibited Water based Drilling Fluid System and PSGCNC depends solely on the composition of the agents required in an accurate and correct manner.

In the first hand, according to generalised measuring manual guidelines different muds samples were formulated for PSGCNC based polymer holding the other components as constant using the Mettler Electronic Precise ion Balance to measure the mass of the different component so as to provide their exact composition in the mud system to be formulated and Hamilton Beach Mixer for proper stirring and mixing of the components so as to choose the optimum concentration.

The materials and chemicals required for the formulation has been enlisted in the **TABLE 01**.

2.1. Formulation of Nano-composite with polymers

Clay Nano-composite was synthesized in an inert nitrogen atmosphere. Prior to the polymerization, dispersion of clay is required to achieve the best combination of matrix-nanoparticle properties. This can be achieved by stirring an aqueous solution of Nano-clay. The reaction may took place in a 3-neck round bottom flask kept on a magnetic stirrer, for constant stirring. Then the reaction mixture needs to be cooled and observe the precipitation of product. Afterwards, it is dried and specific amount of synthesize Nano-composite can be obtained.

2.2. Development of water based drilling fluid with Nano-composite

The base fluid was prepared by using Hamilton Beach Mixer. Then the desired amount of Nano-composite need to be added to this fluid to evaluate its effect on the various properties of the system as per standard procedures recommended for field testing of the drilling muds. The rheological parameters and fluid loss control property of the developed system will be analyzed with Fann VG meter, model 35, and Fann API filter press, Fann Instrument Company (Houston, Texas), respectively. The water analyzer will be used to evaluate the pH and the electrical conductivity.

2.3. Characterization of the PSGCNC

2.3.1. Fourier transform infrared spectra (FTIR)

Initially, before the FTIR synthesis, the polyacrylamide and starch grafted/ clay nanocomposite was oven dried and the stored in air tight micro-centrifuge tubes. The FTIR SPECTRA (450-4000 cm^{-1}) were measured with Perkin-Elmer Spectrum Two, FT-IR instrument (USA). Also, the KBr pellets were prepared using a hydraulic pressure press with 100psi pressure for 60 seconds over a known weight of KBr (1 mg). The IR spectrum of the KBr pellet was then prepared and recorded and scanned

2.3.2 Thermal Stability

Thermogravimetric analysis of the nanocomposite was done to determine its thermal stability in a Netzsch-STA 449 Jupiter (Germany). In total a set of 15 mg samples sealed in aluminium pans was scanned from 30°C to 650°C at a rate of 10°C/min in the protective nitrogen gas environment.

2.3.3. X-ray Diffraction:

X-ray spectra of the polyacrylamide and starch grafted/ clay nanoparticle were obtained using a Carl Zeiss FESEM analyser with an attached energy dispersive identification X-ray software X-MAX (Oxford instruments, SDD X-MAX 50, U.K) for elemental analysis.

TABLE 01: Agent Specifications for formulation of Conventional and PSGCNC based Inhibiting Water based Drilling Fluid System	
Agent Specification	Composition of PSGCNC based Inhibiting Water based Drilling Fluid System
Base Fluid (Fresh Water)	1500 ml
Weighing and Bridging Material (Calcium Carbonate)	FCC: 3.4%(W/V) MCC:5.1%(W/V)
Viscosifier & Fluid Loss Control Agent (PSGCNC)	3% (W/V)
Clay/Shale Inhibitor or Stabilizer (Choline Chloride)	2.5% (W/V)
pH Control Agent (Caustic Soda)	0.025% (W/V)
Non- Biodegradation Agent, Biocide (Formaldehyde)	0.15% (V/V)
Lubricating Agent (Linseed Oil)	0.3% (V/V)

2.4. Characterization of Inhibitive Water-based Drilling Fluid System:

2.4.1 Rheological and filtration properties of the drilling fluid system:

The base fluid system was developed using potassium chloride being added at a concentration of 4.0 wt./v%, xanthan gum (0.3 wt./v%) and pregelatinized starch (3 wt./v%) and was thoroughly stirred and mixed in Hamilton Beach Stirrer at high speed. Based on the American Petroleum Institute (API) recommended procedures, fixed and calculated amounts of nanocomposites was added to the mud to learn its effect on the various mud properties. Then inspection of the rheology was done using the Fann VG meter, model 35, Fann Instrument Company (HOUSTON, TEXAS). The Fann API filter press was then used to determine the filtration properties. The following formulae were used to develop the rheological parameters as per API recommended practice of standard procedure for field testing of drilling fluids:

$$\text{Apparent viscosity } (u_a) = \frac{\Delta\theta_{600}}{2} \quad (\text{mPa}\cdot\text{s})$$

$$\text{Plastic viscosity } (u_p) = \Delta\theta_{600} - \Delta\theta_{300} \quad (\text{mPa}\cdot\text{s})$$

$$\text{Yield point } (Y_p) = (\Delta\theta_{300} - u_p) * (0.5) \quad (\text{Pa})$$

2.4.2 Salt Resistance of the drilling fluid system:

A certain amount of synthesized nanocomposite (0.7 wt./v%) was mixed with various concentrations of CCl salt and added to the base fluid after aging the base fluid at 95° C and then evaluate the salt resistance capacity of the drilling fluid system. The effect of salt addition on the rheology and filtration properties of the mud system was studied up to a certain specified optimum saturation point as recommended by the API.

3. RESULTS AND DISCUSSIONS:

3.1. Results of Characterization of the PSGCNC

3.1.1. FTIR characterization of the nano-composite:

FTIR analyses were sought to clarify the grafting of polyacrylamide on starch/ clay nanoparticle composite. The FTIR spectra of the clay (montmorillonite), polyacrylamide (PAM), starch (St), and polyacrylamide and starch grafted/ clay nanoparticle composite are shown in **Fig 01**.

The characteristic vibrational bands of Na-MMT at 3480 and 1654 cm⁻¹ are indicative of stretching and bending of OH groups, respectively. The spectrum of PAAm shows two bands at 3464 and 2935 cm⁻¹, which are due to the N-H stretching vibration of the NH₂ group and C-H stretching vibrations. The bands at 1676 and 1440 cm⁻¹ are attributed to stretching of C=O group in amide and CH₂ scissoring, respectively. The characteristic absorbance bands at 1024, 941 and 517 cm⁻¹ are assigned to bending vibrations of Si-O-Al (octahedral Al), Al-Al-OH and Si-O-Si, respectively.[13] For starch, a characteristic absorption peak at 1029 cm⁻¹ is attributed to C-O stretching vibration. Characteristic peaks at 1678 and 1098 cm⁻¹ are due to the presence of water and C-O stretching of the anhydrous glucose ring [22]. However, in the FTIR spectrum of the PAAm-g-St/ MMT, three additional peaks at 3420, 2945 and 1680 cm⁻¹ were enlightened which are due to the amine group (-NH), stretching of C-H bond and amide-I (C=O stretching) of the amide group of graft PAAm chains, respectively [25], indicating the presence of MMT and starch. Thus, the FTIR spectra put forwards that the Polyacrylamide Starch/ Clay Nanocomposite was synthesized.

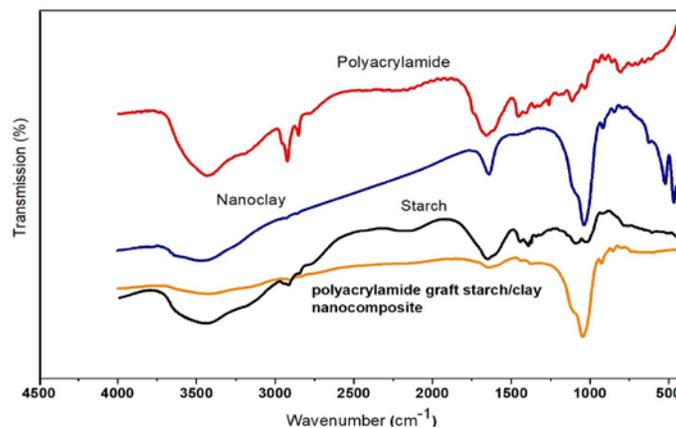


Fig. 01. FTIR Characterisation of the PSGCNC

3.1.2. Thermogravimetric analysis of the nano-composite:

Thermogravimetric analysis (TGA) and differential thermogravimetry (DTG) patterns of the dried polyacrylamide and starch grafted/ clay nanoparticle composite (0.5wt.% polyacrylamide and starch grafted/ clay nanoparticle, 0.4 wt.% crosslinker and 2000ppm montmorillonite) were thoroughly gone through and a thermogram was generated.

The thermogram of the polyacrylamide and starch grafted/ clay composite shows that on heating the composite more, another weight loss occurred randomly at which suggested that the nature of the nanoparticle composite has almost degraded. The thermogram of the polyacrylamide and starch grafted/clay nanoparticle composite shows a

remarkable extension of the degradation temperature which may be due to the presence of clay in the polymer matrix. Hence, the polyacrylamide and starch grafted/clay nanoparticle composite can be used in high temperature reservoirs.

3.1.3. X-Ray Diffraction analyses on the nanoparticle composite:

The XRD graph structures of the clay (montmorillonite), polyacrylamide and starch grafted/ clay nanoparticle composite are plotted. A basal peak for the clay (montmorillonite) was identified at $2\theta \sim 10^\circ$ in **Fig. 02** which shows the diffraction from the regular periodicity pattern of the stacked silicate layers. The basal peak vanished when we used the polyacrylamide and starch grafted/ clay nanocomposite material with the clay (montmorillonite). This indicates that clay layers either were exfoliated or have a lower concentration level of it's in the polymer.

It has been evident from the figures describing the rheological properties of the Drilling Fluid System as a function of PSGCNC concentration that they vary according to the varied concentrations differently. The funnel viscosity increases substantially with the increase in concentration of PSGCNC which is clear from **Fig. 03**.

The other rheological parameters, apparent and plastic viscosity, yield point and gel strength increases in a unregular fashion which is due to long chain of the nanocomposite. The fluid loss decreases with increasing concentration which in terms a good indication for the composite drilling fluid system providing high durability with enhanced performance even in adverse subsurface conditions. [**Fig 04-07**]

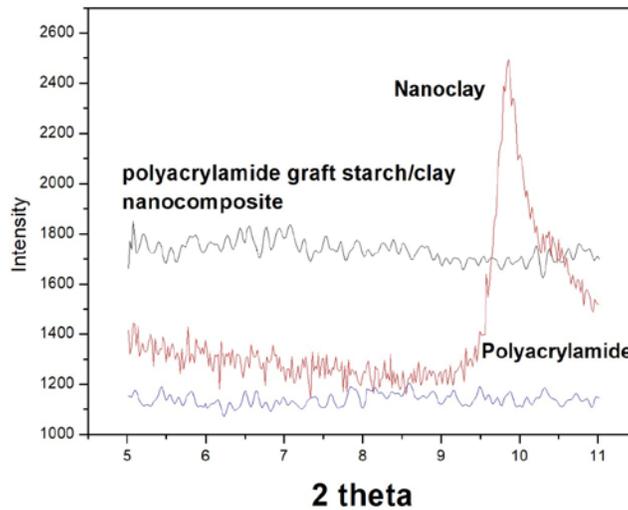


Fig. 02. XRD analysis of the PSGCNC

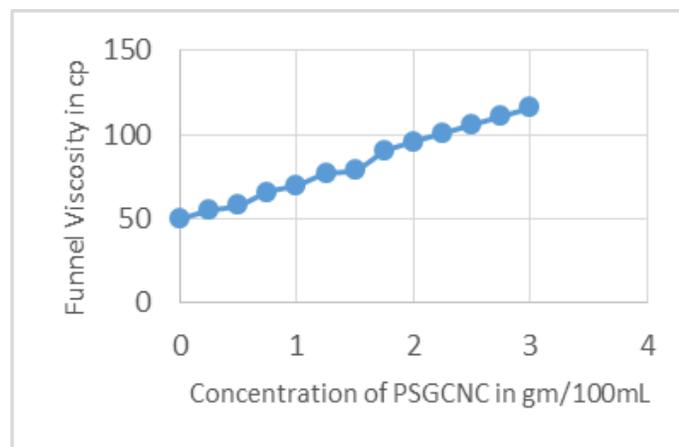


Fig 03. Funnel Viscosity vs. PSGCNC concentration

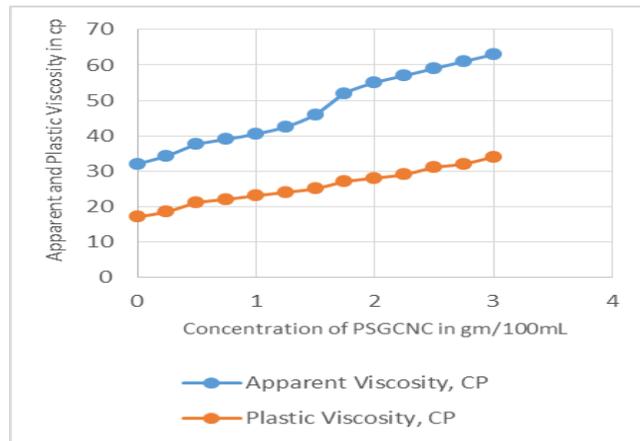


Fig 04. Apparent and Plastic viscosity vs. PSGCNC concentration

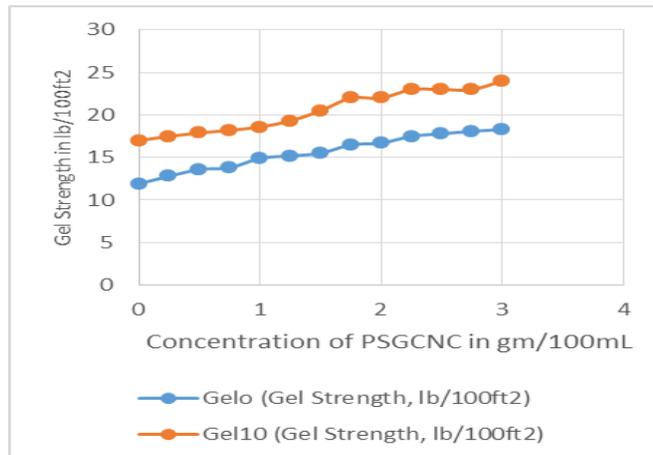


Fig 05. Gel Strength vs. PSGCNC concentration

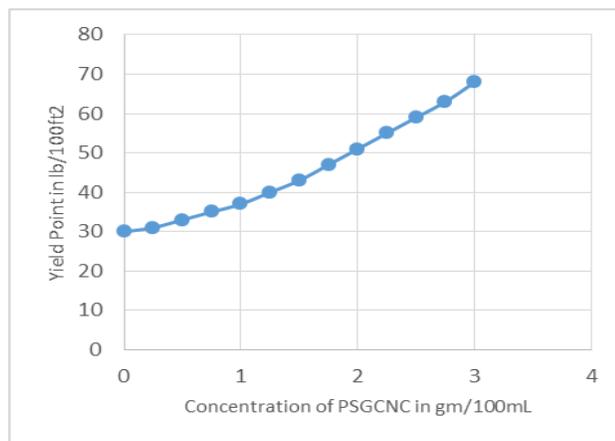


Fig 06. Yield Point vs. PSGCNC concentration

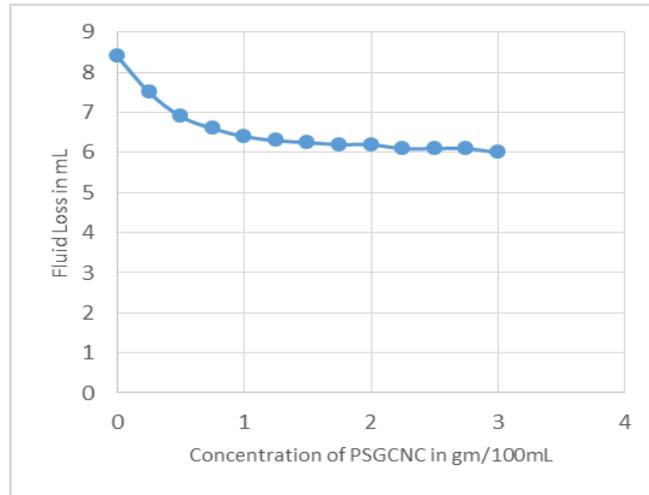


Fig 07. Fluid Loss vs. PSGCNC concentration

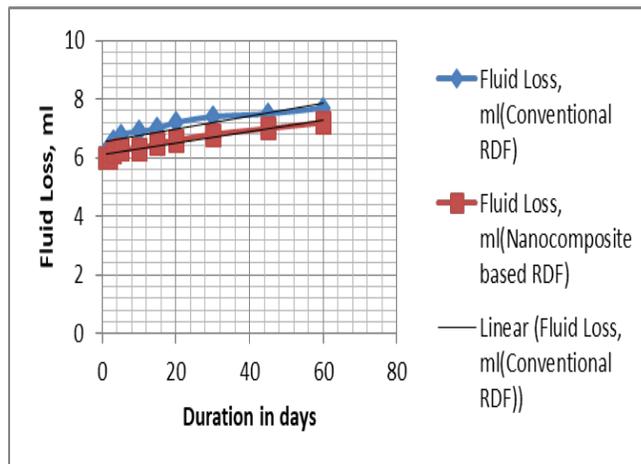


Fig. 08. Fluid Loss vs. Duration in Days for Biodegradation Study

3.2. Effect of PSGCNC based Nanocomposite on the Biodegradation and Thermal Degradation of Inhibitive WBM

Biodegradation and Thermal Degradation are two important properties which needs to be analysed. To study the effects of the Nanocomposites on the biodegradation nature of the designed PSGCNC based nanocomposites in Inhibitive WBM, the samples were kept upto 60 days in the laboratory and investigated the mud properties in a regular routine manner basically to investigate the durability of the mud samples against the biodegradation with increasing time. To examine the temperature durability of the nanocomposites based designed Inhibitive WBM basically the hot roller oven and Grace M3600 Viscometer for increasing the temperature of the mud samples and to measure their rheological properties has been used. The mud properties have been investigated by increasing the temperature of the mud samples from 85 °F to 300°F in the laboratory in regular routine manner with a temperature gap of 15 °F.

TABLE-02: Lubricity and Rheological properties of PSGCNC samples at different composition of Linseed Oil				
Linseed Oil %	Properties of the samples			
	Plastic Viscosity, cp	Yield Point, lb/100ft ²	Gel, lb/100ft ²	Lubricity Co-efficient
0%	19	23	5	0.287
0.1%	18.5	23	5	0.275
0.2%	18.5	23	5	0.270
0.3%	18	23	6	0.264
0.4%	18	22	6	0.253
0.5%	18	22	6	0.243
0.6%	18	21	7	0.235
0.7%	18	21	7	0.232
0.8%	18	21	7	0.229
0.9%	18	21	7	0.229

From the experimental investigations represented in **Fig. 08.**, it has been observed that all the rheological properties and fluid loss properties degrades with increasing time and temperature due to the degradation of the components of PSGCNC based nanocomposite Inhibitive WBM which much lower than other drilling fluids as the aspect ratios of the rheological properties have been maintained. [26]

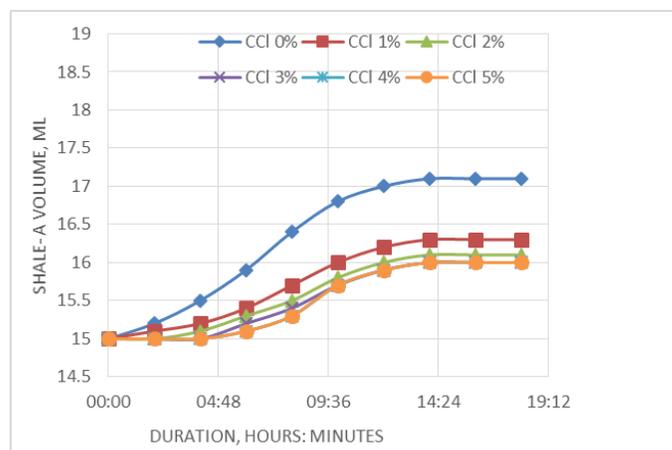


Fig. 09. Expansion of Shale-A Volume vs. Duration

3.3. Effect of Linseed Oil (as Lubricating Agent):

HPHT wells experience high torque and drag, henceforth use of lubricants in the drilling fluid is primarily required for which Linseed Oil, a vegetable oil has been selected as a lubricating agent. Lubricity is measured with the help of lubricity

tester which measures the Lubricity coefficient as it indicates the performance of the lubricant used in the drilling fluid. Lower the Lubricity coefficient, higher is the performance. It has been observed from laboratory results that a very small composition of linseed oil can increase lubrication by significantly decreasing lubricating coefficient thereby keeping the other mud properties constant. It has been found that 0.8% of Linseed Oil composition can be recommended for optimum lubricity of the drilling fluid and above this value the rheology of the mud might be disturbed which is undesirable. **Table 02** describes a decreasing trend of rheological parameters taken into consideration till 0.5% of Linseed Oil concentrations and remains constant thereafter.

TABLE-03: Properties of Ten (10) numbers of WBM samples at different composition of Choline Chloride (Fresh Water: 1.5 liter, Clay (Bentonite): 10% and Temperature: 81 °F)

% of Choline Chloride	Properties of Samples		
	Plastic Viscosity, cp	Yield Point, lb/ 100 ft ²	Gel ₀ , lb/ 100 ft ²
0	1.9	7	1
1	2.6	3.2	2
2	0.3	2.6	1
3	0.1	1.6	1
4	0.1	1.6	1
5	0.1	1.6	1
6	0.1	1.8	1
7	0.1	1.8	1
8	0.1	1.8	0
9	0.1	1.9	0

3.4. Results of Bentonite Inhibition Test with Choline Chloride [(2-Hydroxyethyl) Trimethyl Ammonium Chloride (C5H14ClNO)]:

For Bentonite Inhibition Test, a solution has been formulated with fresh water, bentonite with varying composition of Choline Chloride concentrations. The test was carried out on the basis of the fact that bentonite is a smectite clay and if Choline Chloride inhibits the smectic clay in the laboratory condition, then it would act accordingly in the reservoir condition. The mud properties were calculated at different concentrations of Choline Chloride and it was observed in **Table 03** that from 0 to 3%, the decreasing trend was very sharp, from 3% to 5% there was slight decrement of the values, however after 5% the formulated system exhibited an increasing trend. Thus, it was evident that at 5 % Choline Chloride concentration the values were minimum which in turn signified that the 5% Choline Chloride will inhibit the swelling of smectite(bentonite) when it comes in contact with and consequently there will be no change in rheological properties of the PSGCNC nanocomposite based Inhibitive WBM. The Choline Chloride will decrease the gel strength and yield point due to the mechanical interactions of the dispersed solids.

3.5. Evaluation of the results of Shale Inhibition Test with Choline Chloride:

For Shale Inhibition Test with Choline Chloride the following results were graphed for varied concentrations of Choline Chloride: CCl 0%, CCl 1%, CCl 2%, CCl 3%, CCl 4% and CCl 5% solutions.

From **Fig. 09 and 10**, it can inferred that the volumes of Shale A and B increases significantly with increase in time in first hand but the increase is not significant thereafter which is much improvised in case of PSGCNC nanocomposite based Inhibitive WBM.

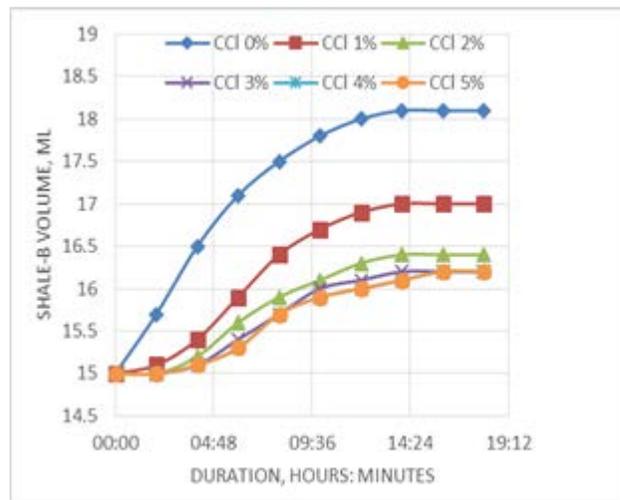


Fig. 10. Expansion of Shale-B Volume vs. Duration

4. CONCLUSION

The synthesis of polyacrylamide and starch grafted/ clay nanoparticle composite by polymerization has been accomplished successfully. On further morphological and rheological studies, it has coined the fact that the presence of clay nanoparticles within the polymer matrix has altered the morphology of the nanoparticle composite.

The synthesis of polyacrylamide and starch grafted/ clay nanoparticle composite by polymerization has been accomplished successfully. On further morphological and rheological studies, it has coined the fact that the presence of clay nanoparticles within the polymer matrix has altered the morphology of the nanoparticle composite.

- The presence of nanoparticles has endeavoured the thermal strength and extended non-biodegradability of the same.
- The intensity of filtration of this new synthesised composite is reduced at higher monomer ratios.
- Also, the grafted nanoparticle composite has much higher resistance for salinity damage.
- It also has better handling capacity of high swelling formations as compared to the previously used polymer drilling systems.
- The presence of reactive clays has successfully been reported by XRD and FTIR analysis.
- The lubrication performance is high for PSGCNC as compared to other additives to conventional WBMs.

Hence, the synthesized polyacrylamide and starch grafted/clay nanoparticle composite may be used as an additive in water-based drilling fluid system for the drilling of water sensitive formations profusely and additionally indicate a positive pathway of incorporation of Nanotechnology in the Drilling Fluid domain.

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