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Research paper

Rheological properties measurement of Mucuna solannie as cement slurry extender: characterization and verification using rheological models

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Abstract

Rheological properties of lead cement slurry with Mucuna solannie admixture as an extender was measured in accordance with API standard. Bentonite extender was used as a control. The elemental and oxide compositions of Mucuna solannie were determined using Scanning Electron Microscope and X-Ray Florescence (XRF) methods, and rheological properties were obtained using rheometer after conditioning. The rheological data from Mucuna solannie and bentonite lead slurries were validated using Bingham Plastic and Herschel-Bulkley models. The result showed that Mucuna solannie contains high carbon atomic concentration and is responsible for its high rheological properties values. Lead slurry prepared with Mucuna solannie gave higher plastic viscosity, yield point and gel strength than that of bentonite. Herschel-Bulkley model described the rheological properties better than Bingham Plastic model. Due to high rheological properties values of the slurry prepared with Mucuna solannie, dispersant is needed for the optimization of the yield point and gel strength.

Keywords: *Bentonite; Elemental Composition; Lead Slurry; Mucuna solannie; Oxide Composition; Rheological Properties.*

1. Introduction

The rheology of conventional cement paste is not as complicated as that of cement slurries used in oil well operations. Rapid gel formation, collapse under stress which depends on inter-particle forces, and destruction of gel fragments are elementary factors that describe cement slurry rheology [1]. Also, with particle size distribution (PSD) analysis, another work modified Krieger – Dougherty model, and showcased particle diameter, surfactants and clusters effects on effective viscosity of nanofluids [2]. Similarly, other researchers investigated the effects of temperature on water-based mud properties like Mucuna solannie mud and Detarium microcarpum, Brachystegea eurycoma, Pleurotus mud [3], [4]. They discovered that rheological properties decreased with temperature increase, and fluid loss increased with temperature increase. Both drilling mud and cement slurry are all pseudo-plastic fluids; they have similar behavior in terms of rheological properties because they are all pseudo plastic fluids. Also, another researcher investigated chemical admixtures effects on rheology mechanisms in oil/water cement slurries at varying temperatures where advanced shear-strain/shear-stress rheometer was applied [5]. Cement paste morphology and hydrated products phase compositions were studied with Scanning Electron Microscope and X-ray diffraction analysis. As admixtures dosage increased, adsorption on cement particles increased and cements particles negative charge increased. The result was greater repulsion which enhanced the cement paste rheological properties [6]. Rheological properties optimization, such as plastic viscosity and yield stress, can be achieved with statistical design if cementitious materials supplements are applied at varying temperatures. Also, it could be used to better understand the trade-offs with key mixtures parameters, such as superplasticizer's dosage, and supplementary cementitious materials level [7]. It was presented that rheological properties of oil/water cement slurries depend highly on additive used, water/cement ratio and temperature. Rheological behavior of cement is important for the drilling process; it will be optimum to predict correctly about slurry placement. Controlling the rheological behavior of cement slurries can help to improve the relevant movement between particles, the stability and substitutability according to the API standard [8]. A study of oil well cement slurries rheological behavior presented that inter-particle attraction is suppressed by dispersant molecules by cement particles' surface saturation which results in sedimentation [9].

Also, experiments have been carried out on the use of ilmenite plant dusts as substitutes to barite as densifier in cement slurries. Rheological investigation showed that proper dispersion could guarantee application of one of the ilmenites [10]. Various admixtures usually added to cement slurries always depend on purpose of the cementing hole conditions for a successful cementing operation. The process suggested that low-shear-rate dynamic viscosity would govern sedimentation. Using different additives during the formulation of cement slurry gives has economic advantage and environmental benefits. Hardened cementations matrix quality and end-use performance predic-

tion, combined with physical properties when processed are determined by rheological properties. Laboratory measurement of cement-based materials rheology is still a challenge. However, Newtonian model and non-Newtonian model are used to describe rheology. The orientation or distortion of particles that oppose Brownian motion effects, caused by shear stress transmission are attributable to non-Newtonian flow behavior [2].
In summary, optimization of the rheological behavior of oil well cement slurry should be achieved for optimized cementing. Unfriendly environments necessitate having good durability and mechanical properties in the well life cycle. A material could exhibit yield stress based on test conditions. It has been shown that geometry affects yield stress of cement pastes [11]. Viscosity of a sample varies since shear rate is the determining factor Thus, apparent viscosity is used for any shear rate specified. Bingham Plastic and Herschel-Bulkley models are the two rheological models applied in this research where cement slurry was prepared with Mucuna solannie as extender, while cement slurry prepared with bentonite on equal concentration was used as a control.

1.1. Characterization of Mucuna solannie

Mucuna is found worldwide both in Africa and Asia; they are leguminous and are used for culinary purposes [12, 13]. Its use in industrial sector for rheology control has been tested. Mucuna solannie can be used as a cash crop. Planting of the seed in square pattern, with 10,000 plants every hectare, can yield 63.6 metric tons in a year if no filler crops are planted, and a plant for every hill is used [14]. That was proposed to ascertain that the crop would be available when required in oil and gas drilling activities.

2. Materials and method

The elemental and oxide compositions measurement of Mucuna solannie were carried out, followed by slurry rheological properties laboratory measurements prepared using Mucuna solannie as an extender and bentonite as a control.
This section is divided into 6 parts, namely; (a) Collection of the Mucuna solannie (b) Physical pre-treatment and processing of the Mucuna solannie (c) Elemental and Oxide compositions measurements (d) Cement slurry formulation (e) Conditioning of cement slurry (f) Cement slurry rheological properties measurement. These measurements were carried out in accordance with API [15].

2.1. List of apparatus/materials

Apparatus and materials used were X-Ray Fluorescence (XRF) Cu-Zn, Scanning Electron Microscope, electronic weighing device, variable speed mixer, variable speed viscometer, measuring cylinders, spatula, stop watch, bentonite, Mucuna solannie, Class G cement, defoamer and fresh water.

2.2. Collection of Mucuna solannie

The Mucuna solannie utilized for this study were obtained from Ogbaete Enugu market, Enugu state of Nigeria.

2.3. Processing of Mucuna solannie

The sorted seeds were cracked open with a sledge hammer to allow for water to seep into the seed. The water helps in softening the seed and allow for easy removal from the seed coat. For the optimization of carbohydrate, the seeds were boiled, allowed to cool, and thereafter dried in a moisture extractor oven at 149^{O}F. The dried seeds were ground. The flour was screened through a standard test BS sieve (0.63mm) to obtain flour particle size.

Fig. 1: Scanning Electron Microscope (SEM).

2.4. Elemental composition measurement using SEM/EDS method

The Scanning Electron Microscope – Energy Dispersion Spectroscopy (SEM) Phenom Prox (by Phenom World Eindhoven) was used to carry out the morphology and elemental analyses. With a sticky carbon tape, sample was placed in the Aluminium stub. The sample was insulated using gold and then grounded electrically. Each sample was then labeled on their stub, and then dried in the oven at 140^{O}F for 3 hours. Nitrogen line was opened at 50 psi and the vent button was pressed to fill the area with nitrogen for proper purging of the chamber. The sample holder stub was then placed in the sample chamber holes, the door was shut and rotary pump picked. At about 35 minutes, vacuum of 5 x 10^{-5} Pa was created. The filament light was switched on and the monitor automatically switched on. At this stage, the peak accelerator voltage read 15kV and the filament burned out. Specific wavelengths of X-rays are emitted when electron beams excite surface atoms which characterize the elements atomic structures. Dispersive energy detector analyzes the emissions, assigning elements, which yields atomic composition. The lowest scan mode of 10x was picked and the TV scan clicked. The magnification was then taken to 1000x at a slow scan, 2000, 3000 to 10,000. Image was then saved. This is known as energy dispersive X-ray spectroscopy (EDS), and it is used for specimen surface composition analyses, with diagram shown in Figure 1, and results in Figures 2 to 4. The elemental and oxide compositions determined using X-Ray Fluorescence (XRF) Cu-Zn is in Tables 5 and 6.

2.5. Cement slurry formulation

Two formulations were carried out using Mucuna solannie and Bentonite extender additives, conditioned at both bottom hole circulating temperature of 200^{O}F and 150^{O}F respectively as shown from Table 1 to Table 4.

Table 1: Bentonite Extender Slurry Composition

Slurry name	Lead slurry
Cement sack weight	94.0 lb/sk
Slurry density	12.0 lb/gal
Slurry yield	2.397 ft^3/sk
Cement type	Conventional cement
Mix water type	Fresh water
Mix water required	14.175 gal/sk
Mix fluid required	14.334 gal/sk
Slurry composition	Class G cement + 0.0313-gal ASP-742 + 3% BWOC Bentonite

Table 2: Bentonite Extender Slurry Formulation

Additives	Function	Concentration	Weight (gram)	Volume (ml)
Class G cement	Cement	100 % BVOB	377.66	120.27
ASP-742	Defoamer	0.0313 gal/sk	0.95	1.05
Bentonite	Extender	3% BWOC	11.33	4.28
Fresh water	Mix water	14.175 gal/sk	474.40	474.40
Total			864.35	600.00
Mix fluid		14.334 gal/sk	486.35	479.79

Table 3: Mucuna solannie Extender Slurry Composition

Slurry name	Lead slurry
Cement sack weight	94.0 lb/sk
Slurry density	12.0lb/gal
Slurry yield	2.357 ft^3/sk
Cement type	Conventional cement
Mix water type	Fresh water
Mix water required	13.741 gal/sk
Mix fluid required	14.033 gal/sk
Slurry composition	Class G cement + 0.0313gal/sk ASP-742 + 3% BWOC Mucuna solannie

Table 4: Mucuna solannie Extender Slurry Formulation

Additives	Function	Concentration	Weight (gram)	Volume (ml)
Dyckerhoff G cement	Cement	100 % BVOB	384.11	122.23
ASP-742	Defoamer	0.0313 gal/sk	0.97	1.07
Mucuna solannie	Extender	3% BWOC	11.52	8.86
Fresh water	Mix water	13.741 gal/sk	467.74	467.74
Total			864.35	600.00
Mix fluid		14.033 gal/sk	480.23	477.65

2.6. Atmospheric pressure conditioning

The formulated cement lead slurry using Bentonite and Mucuna solannie was respectively conditioned at BHCT of 150^{O}F and 200^{O} F in accordance with API procedure.

Within 1 minute after mixing, the slurry container of the atmospheric pressure consistometer was filled to the marked line. The test fluid was heated from ambient temperature to the desired temperature. The slurry container preheated to the desired temperature was placed in the atmospheric consistometer cell. After the slurry reached test temperature, the test temperature was held for +/-30 minutes to allow the test fluid temperature to reach equilibrium. The paddle was removed and the test was stirred briskly with a spatula to ensure consistency.

2.7. Rheological properties and gel strength measurements

The conditioned test slurry extended with Bentonite and Mucuna solannie was respectively poured into the preheated viscometer cup to a level adequate to raise the fluid to the scribed mark on the rotor without the rotor or bob touching the bottom of the cup. The initial dial reading was taken after 10 seconds of continuous rotation at 3 RPM. The remaining dial readings at other speeds were taken, first at ascending order and then in descending order. The highest speed used was 300 RPM. The ratio of the dial readings was also taken during ramp up to ramp down at each speed. The ratio was used to qualify the fluid properties as shown in Table 8 in the attachment.

3. Results

3.1. Results presentation

These include the results obtained from the determination of the elemental and oxide compositions using both X-Ray Fluorescence (XRF) Cu-Zn and SEM methods as shown in Tables 5, 6, 7 and Figures 2 and 3 of plot view runs and SEM map.

Table 5: Elemental Composition of Sample Using X-Ray Fluorescence (XRF) Cu-Zn Method

Element	Run 1	Run 2	Run 3
Mg	< LOD	< LOD	< LOD
Al	0.985	0.967	0.937
Bal	95.746	94.126	95.073
Si	2.564	2.251	2.771
P	0.314	0.318	0.312
S	0.281	0.279	0.280
Cl	0.012	0.013	0.013
K	1.449	1.455	0.454
Ca	0.414	0.416	0.397
Ti	0.018	0.018	0.017
V	< LOD	< LOD	< LOD
Cr	< LOD	< LOD	< LOD
Mn	< LOD	< LOD	< LOD
Fe	< LOD	< LOD	< LOD
Co	< LOD	< LOD	< LOD
Ni	< LOD	< LOD	< LOD
Cu	< LOD	< LOD	< LOD
Zn	0.001	0.002	0.002
As	< LOD	< LOD	< LOD
Se	< LOD	< LOD	< LOD
Rb	< LOD	< LOD	< LOD
Sr	< LOD	< LOD	< LOD
Zr	< LOD	< LOD	< LOD
Nb	< LOD	< LOD	< LOD
Mo	< LOD	< LOD	< LOD
Pd	< LOD	< LOD	< LOD
Ag	< LOD	< LOD	< LOD
Cd	0.001	0.002	0.001
Sn	< LOD	< LOD	< LOD
Sb	< LOD	< LOD	< LOD
Ba	< LOD	< LOD	< LOD
W	< LOD	< LOD	< LOD
Au	< LOD	< LOD	< LOD
Pb	< LOD	< LOD	< LOD
Bi	< LOD	< LOD	< LOD

LOD: Low Detection

Table 6: Oxide Composition Result Using X-Ray Fluorescence (XRF) Cu-Zn Method

Oxide	Run 1	Run 2	Run 3
CuO	0.01	0	0
NiO	0	0	0
Fe_2O_3	0.011	0	0.103
MnO	0.013	0	0.019
Cr_2O_3	0	0	0
TiO2	0.0167	0.029	0.022
CaO	0.608	0.583	0.518
Al_2O_3	1.991	1.828	1.908
MgO	0	0.243	0
ZnO	0.01	0.003	0.008
SiO_2	4.047	4.816	4.21

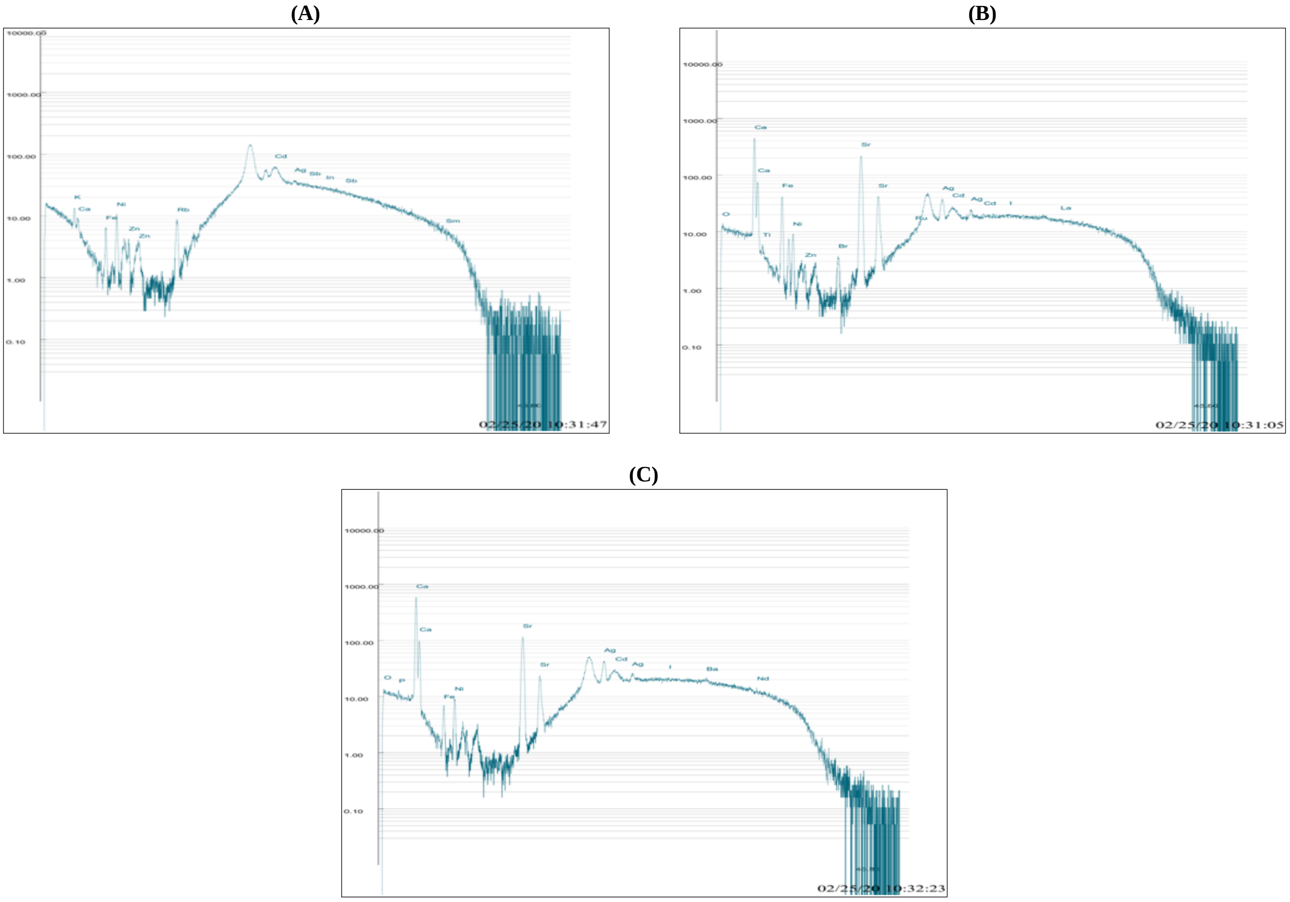

Fig. 2: A, B, C: Plot View of Run 1, Run 2 and Run 3 Using X-Ray Fluorescence (Cu-Zn) Method.

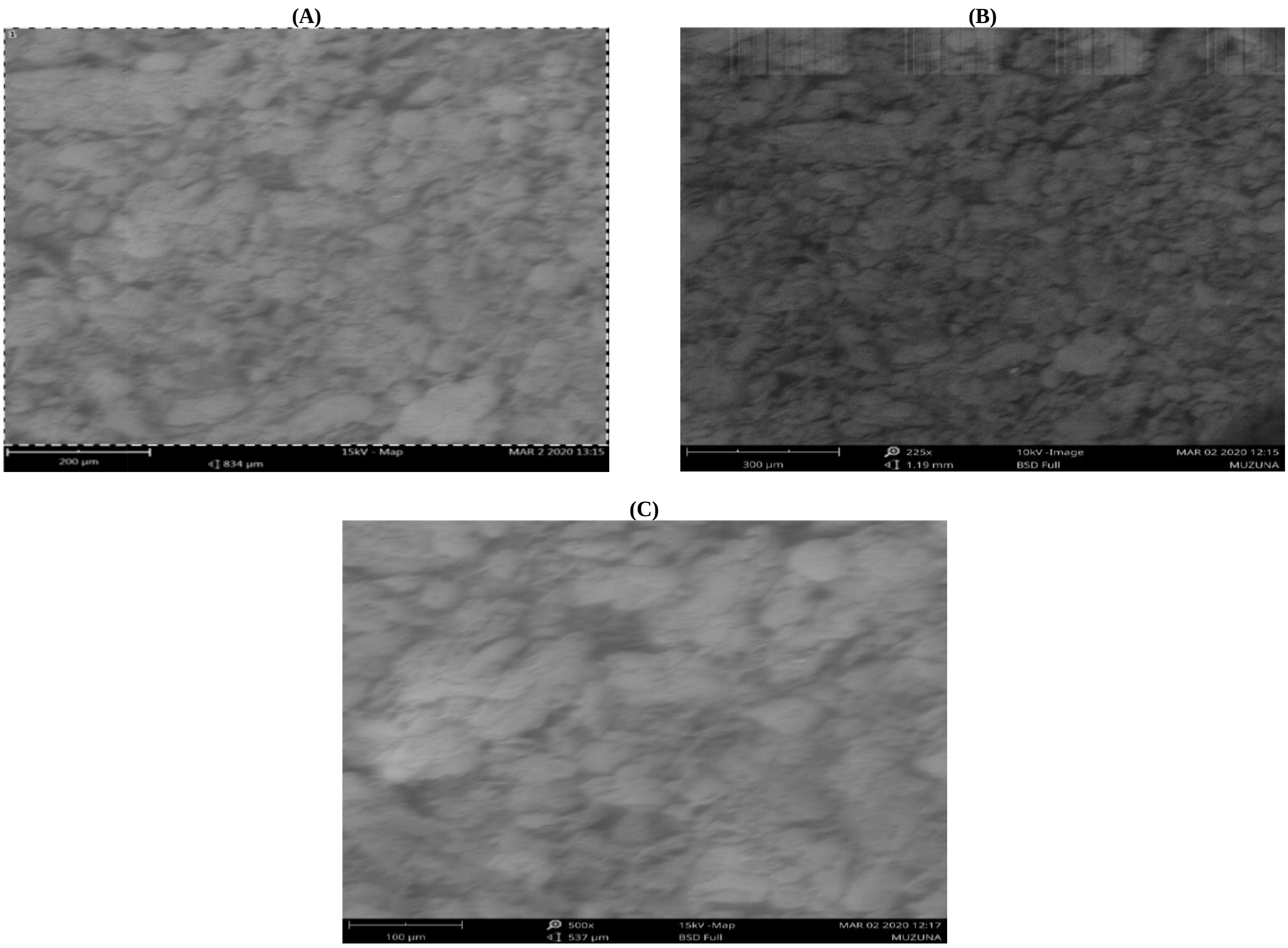

Fig. 3: A, B, C: SEM Map of Mucuna solannie.

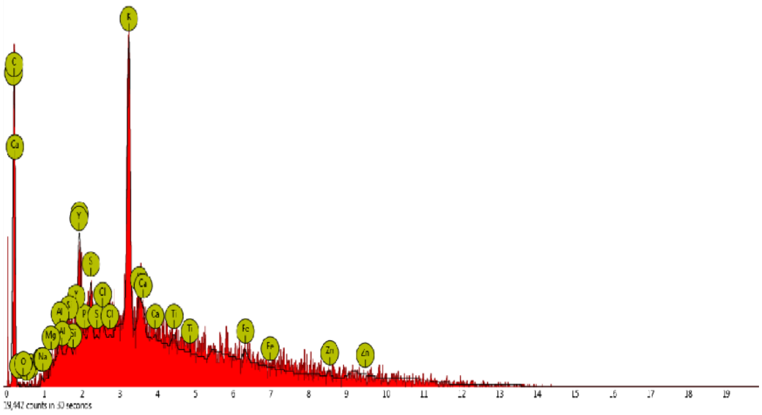

Fig. 4: Plot View of SEM Run Result.

Table 7: Elemental Composition of Mucuna solannie

Number	Symbol	Name	Atomic Conc.	Weight Conc.
19	K	Potassium	28.86	39.18
6	C	Carbon	35.27	14.71
15	P	Phosphorus	7.34	7.89
30	Zn	Zinc	2.81	6.37
26	Fe	Iron	2.63	5.11
16	S	Sulfur	4.54	5.05
20	Ca	Calcium	3.27	4.55
39	Y	Yttrium	1.15	3.54
22	Ti	Titanium	1.84	3.06
17	Cl	Chlorine	2.19	2.70
8	O	Oxygen	3.74	2.08
14	Si	Silicon	2.00	1.95
13	Al	Aluminium	1.89	1.77
12	Mg	Magnesium	1.39	1.17
11	Na	Sodium	1.08	0.86

Table 8 (Appendix) shows the rheological data obtained from the rheological measurements of the cement slurries prepared with both Mucuna solannie and bentonite respectively as extenders. Their results were then simplified (Table 9).

Table 9: Lead Slurry Rheological Properties Obtained from Mucuna solannie and Bentonite as the Extenders

Parameters	Mucuna solannie Design at $150^{O}F$ BHCT	Bentonite Design at $150^{O}F$ BHCT	Mucuna solannie Design at $200^{O}F$ BHCT	Bentonite Design at $200^{O}F$ BHCT
Plastic Viscosity, cP	89	11	114	8
Yield Point, $lb/100ft^2$	67	56	66	46
10 sec. Gel strength, at $77.5^{O}F$	13	13	15	10
10 min. Gel strength, at $150^{O}F$	16	16	22	16

3.2. Results discussion

The basic reason for determination of rheological properties is to predict plastic viscosity, gel strength and yield point values of the cement slurries in order to qualitatively evaluate the suitability of Mucuna solannie for cementing operations. From Fable 9, it can be seen that cement formulated with bentonite has lower plastic viscosity and also yield point compared to that of the cement formulated with Mucuna solannie. Cement formulated with Mucuna solannie recorded a high plastic viscosity of 89 cP, 114 cP as compared to 11cp, 8 cP for that of Bentonite at BHCT of $150^{O}F$ and $200^{O}F$ respectively. This showed that lead slurry prepared with Mucuna solannie has more resistance to flow, although it has a better transport capacity than that of lead slurry prepared with bentonite. This shows that it will require a significant amount of dispersant in the lead slurry for mud prepared using Mucuna solannie to work effectively. The high rheological properties values of lead slurry prepared with Mucuna solannie is due to its high carbon atomic concentration obtained from high concentration of carbohydrate as shown in Table 7.

Also, lead slurry prepared with Mucuna solannie gave excessive gel strength and will lead to high pump initiation pressure to break circulation after cement slurry is in a static condition for a period of time. High pump pressure may also result in formation fracture and lost circulation of cement slurry. However, both extenders are suitable for cementing operations due to their appreciable gel strength values obtained, but Mucuna solannie lead slurry has to be dispersed for the good rheological properties to be obtained.

3.2.1. Rheological model performance evaluation

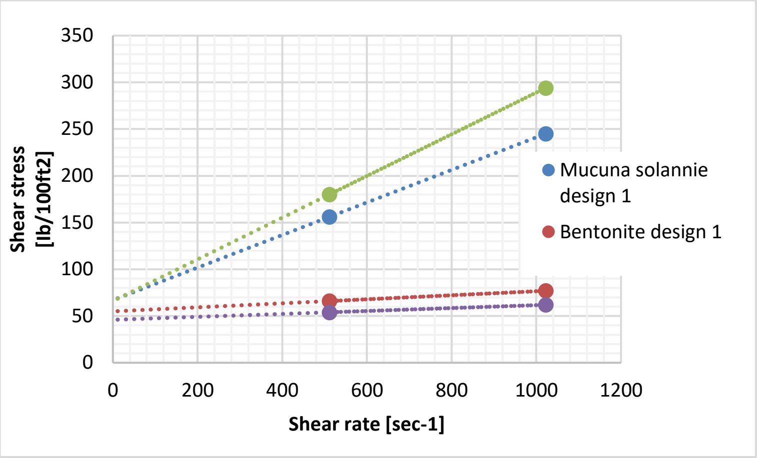

Fig. 5: Mucuna solannie and Bentonite Rheological Plots at 150^{O}F and 200^{O}F Using Bingham Plastic Model.

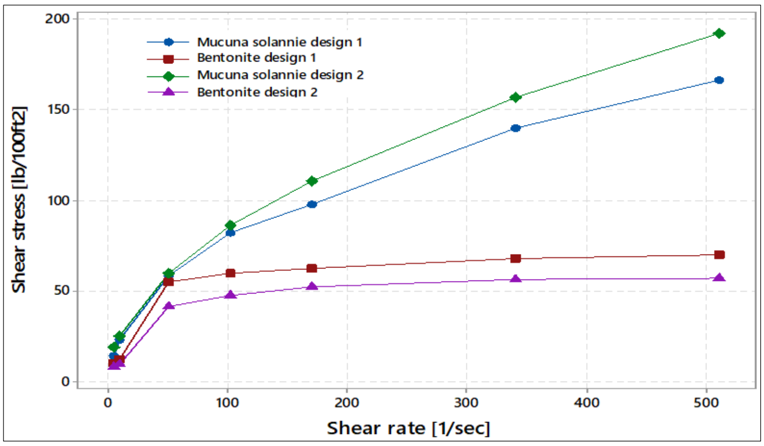

Fig. 6: Mucuna solannie and Bentonite Rheological Plots At 150^{O}F and 200^{O}F Using Herschel-Bulkley.

Both slurries prepared with Mucuna solannie and Bentonite (Table 8) were evaluated and compared using Bingham Plastic and Herschel-Bulkley rheological models. Herschel-Bulkley model, using the yield stress, highlighted lead cement slurry of better rheology at both designed BHCT. Herschel-Bulkley model predicts rheology more accurately and characterizes in the entire shear rate range accurately compared to Bingham plastic. Low shear rate yield stress values are indicated, that serve as indicator of annular slurry performance. Compared to Mucuna solannie, bentonite showed greater capacity to be stable at high temperature from the data generated.

4. Conclusion

From the performance evaluation, it could be inferred that Mucuna solannie contain high carbohydrate and carbon contents. Lead slurry prepared with Mucuna solannie gave higher rheological results than that of the lead slurry prepared with bentonite extender. Also, dispersant would be needed for enhancement of the rheology of the slurry when using Mucuna solannie as an extender. Herschel-Bulkley model gave a better representation than Bingham plastic model.

Nomenclature/designation

BWOC = by weight of cement
BVOB = by volume of blend
BHCT = bottom hole circulating temperature
Design 1 = experiment at 150^{o}F
Design 2 = experiment at 200^{o}F

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Appendix:

Table 8: Slurry Rheological Properties of Mucuna solannie Extender and Bentonite Extender at 150OF and 200OF BHCT

Rotational Speed (RPM)	Mucuna solannie and Bentonite for 150OF BHCT		Mucuna solannie and Bentonite for 200OF BHCT	
	Dial Readings (Mucuna solannie)	Dial Readings (Bentonite)	Dial Readings (Mucuna solannie)	Dial Readings (Bentonite)
3	14	10	18	8
6	22	12	24	9
30	55	52	56	39
60	77	56	81	45
100	92	59	104	49
200	131	64	147	53
300	156	66	180	54
10 sec. gel at 77.6OF	13	13	15	10
10 min. gel at 190OF	16	16	22	16