PERFORMANCE EVALUATION OF *DELONIX REGIA* SAWDUST AS CEMENT RETARDER IN OIL AND GAS WELL

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Abstract

Sawdust, a byproduct of wood exploitation and processing, poses environmental pollution risks if not managed appropriately. Its substantial carbon footprint can lead to pollution, habitat disruption, and fire hazards. However, repurposing sawdust as a chemical additive in cement slurry within the oil and gas industry offers an environmentally friendly solution. This practice aligns with sustainability goals, enhances cement slurry properties, promotes wellbore stability, and replaces more hazardous additives, thereby reducing environmental impact. This study is aimed at the production of a retarder (sodium lignin) from sawdust waste obtained from the *Delonix regia* species, and examining its effect on the thickening time/consistency, compressive strength, and rheological properties on the slurry of a Class G cement At a Bottom Hole Circulating Temperature (BHCT) of 90°C, thickening time tests conducted on the slurry samples revealed that as the concentration of the locally synthesized retarder increases, the thickening time of the concrete also increases, with minimal effect on compressive strength. The optimal thickening time result of 6 hours and 13 minutes was achieved with 0.5% Sodium Lignin (Retarder from *Delonix regia* sawdust) replacing a portion of the cement. At a bottomhole static temperature (BHST) of 100°C, increasing the concentration of the formulated sample led to higher Plastic Viscosities (PV) and yield points in the slurries. The findings indicate that slurries formulated with sodium lignin maintain viscosities within recommended values, making them suitable for pumping.

Keywords: *Delonix regia* Sawdust, Class G Cement, Thickening Time, Compressive Strength, Rheological Properties.

1.0 INTRODUCTION

Cementing in the oil and gas industry is a not only a delicate process but very important operation. Well cementing is the process of pumping a cement slurry into a wellbore to provide various functions such as sealing the wellbore, supporting the casing, and preventing fluid migration between subsurface formations. Anaele et al (2019) defined cementing as operation of placing of oil cement slurry in between the formation and casing to achieve adequate zonal isolation of the well, which is the process of preventing

fluids such as water or gas from mixing with the fluids e.g oil in another zone . Incomplete zonal isolation and weak bonding between the cement, casing, and/or formation can impede well productivity, leading to oil spill.

The improper management of sawdust, a byproduct of wood exploitation and processing, can pose environmental pollution risks. The considerable carbon footprint associated with sawdust may result in pollution, habitat disruption, and fire hazards. However, a sustainable solution is found in repurposing sawdust into a chemical additive in cement slurry within the oil and gas industry. This environmentally friendly practice aligns with sustainability goals, enhances cement slurry properties, ensures wellbore stability, and replaces more hazardous additives, ultimately reducing the overall environmental impact.

Lately, the possibility of sourcing for oil field additives locally from biomass is now gaining wide recognition, as wastes which used to constitute environmental nuisance in form of pollution are now been converted into useful materials. Wood shavings and sawdust which are by products of wood processing are among the wastes in the list of biomass causing environmental concerns. These materials are of considerable concern in Nigeria's various wood industry sectors due to its substantial volume. Nigeria generates an estimated 5.2 million tons of wood wastes and 1.8 million tons of sawdust annually (Owoyemi et al., 2016). Despite advancements in sawdust utilization methods, a significant portion of this waste faces improper disposal, leading to environmental hazards. Addressing these concerns through the recycling and reusing of wood wastes is vital, transforming them into valuable resources. One such sustainable practice involves converting waste sawdust into chemical products, such as sodium carboxymethyl cellulose. While sawdust poses a risk of environmental pollution when not stored under controlled conditions, it also serves as a crucial biomass source for generating solid fuels. These fuels find applications in various heating systems, co-generation installations, decentralized residential setups, and traditional boilers for thermal energy production.

Unfortunately, observed improper disposal practices, such as burning sawdust and other agricultural waste or allowing them to decompose in Nigeria (Jekayinfa&Omisakin, 2005), have led to environmental challenges, rendering affected areas unsuitable for certain economic activities and creating breeding grounds for disease-causing agents. The *Delonix regia* commonly recognized as the Royal Poinciana, Flamboyant, or Flame Tree, is a flowering plant from the Fabaceae family, originating in Madagascar and it is widely cultivated for its ornamental beauty, distinguished by its expansive, umbrella-like canopy adorned with intricate, feathery green leaves, the tree maintains year-round vibrancy, except in regions experiencing dry seasons or mild winters. Adapting well to challenging urban environments and exhibiting salt tolerance, the tree is suitable for coastal planting. It thrives in diverse soil types, including clay, loam, and sand, and can endure both acidic and alkaline soil pH. Marked by a rapid growth rate and drought resistance, *Delonix regia* is a resilient and versatile species. Beyond its aesthetic allure, *Delonix regia* serves practical purposes. Its wood is employed in constructing canoes, seeds are fashioned into accessories such as necklaces or bracelets, and the elongated pods, containing seeds, find use as "maracas." The young stems double as a natural toothbrush, reputedly aiding in the treatment of gingivitis. Its wood is employed in constructing canoes, seeds are fashioned into accessories such as necklaces or bracelets, and the

elongated pods, containing seeds, find use as "maracas". Imohiosen, and Akintola (2021) investigated the production of CMC from sawdust waste, specifically from the *Delonix regia* tree, which is highly underutilized. The production process involves the Williamson ether synthesis method in a slurry medium, comprising mercerization and etherification reactions. The synthesized CMC products are characterized using FTIR Spectroscopy. The study finds that the synthesized CMC products exhibit favorable filtration and rheological properties suitable for drilling fluid applications. By adding low concentrations of these products to the mud, filtration volume can be reduced by 11.4% to 32.9% under low temperature and pressure conditions. Consequently, the synthesized CMC products offer a promising local substitute for low viscosity foreign CMC products in drilling operations.

Anaele et al. (2019), in their study investigated the use of sawdust as a chemical additive (retarder) in the oil and gas industry. Their primary aim was to explore improved methods for addressing the environmental challenges posed by wood waste. Their study involved analyzing cement slurry with sawdust as a retarder, and the results revealed a significant retardation of over 50%. Specifically, the setting time of the cement slurry increased from 4 hours and 31 minutes to 7 hours and 25 minutes with 2ml of the sample produced, and further extended to 8 hours and 35 minutes at 3ml by volume. Akintola and Akintola (2021) investigated the impact of Rice Husk Ash (RHA) and other additives on the compressive strength, consistency, and rheological properties of Class G cement slurry. The additives include Guinea Corn Husk Ash as a retarder, along with liquid additives like fluid loss additive, antifoam, dispersant, and water. The research compares the effectiveness of these additives against a control using pure Class G cement slurry. Results indicate that increasing RHA concentration enhances compressive strength over time, with the most significant increase observed at 13.01% RHA replacement. Additionally, the thickening time decreases with higher RHA percentages. At a high temperature of 700°C, increasing RHA concentration reduces plastic viscosities while increasing yield points of the slurries, with viscosity levels falling within recommended values for pumpability. At various conditions of temperature and pressure, fluid loss increases with higher RHA percentages but remains acceptable. Ogbonna (2009) investigated the application of retarder to increases slurry thickening time only. The result from the study while using different concentration of retarder concentrations shows that not only was the thickening time affected but the compressive strength, rheological as well as the free fluid properties. This study is aimed at developing a more cost effective and environmentally friendly locally sourced cement retarder for cementing oil and gas wells, using sawdust obtained from the processing of *Delonix regia*.

2.0   MATERIALS AND METHOD

For the evaluation of *Delonix regia* sawdust as a potential cement retarder, the materials and equipment used in the study are as follows:

Table 1.0: Materials and Method

Delonix regia sawdust	Chandler Engineering Warring blender
Digital Weighing Balance	Chandler Engineering HPHT Consistometer Model 7222
Electrical weighing balance	Chandler Engineering HPHT Consistometer Model 7222
Clean syringes	Chandler-Model 4265 Ultrasonic Cement Analyzer
Measuring cup	Chandler API Viscometer
Measuring bowl	Centrifuge tubes (50ml)
Measuring cylinder	Pressure vessel
Sieve	Centrifuge
Dispersant	Oven
Fluid loss additive	Antifoam additive
Sodium Lignin	Class G cement
Water	Sulphoric Acid
Sodium Hydroxide (NaOH)	Sodium Bisulfite

The chemicals used were analytical grade purchased from agent of Sigma-Aldrich.

2.1   Sample Sourcing and Preparation

The *Delonix regia* (Figure 1.0) was collected from Ebrohime Road, University of Ibadan, Oyo State. This particular species is readily available in Southwestern Nigeria and was chosen for this study to see how best it could be utilized.The sawdust was sorted and impurities such as leaves, large chunks of wood etc, were taken out to ensure uniformity of the sample. The sawdust was pulped to

extract the cellulose from wood by dissolving the lignin that binds the cellulose fibers together. The sawdust was boiled in the presence of water and sodium hydroxide for 90mins to separate lignin from cellulose.

Figure 1.0 :Delonix regia Sawdust Sample

2.2. Preparation Lignin Liquor

100g of the *Delonix regia* sawdust was measured using an electric weighing balance and transferred into a 4000ml pressure reactor (Kolajo and Onilude, 2016) and 10g of sodium hydroxide (NaOH) and water was added. The sawdust was completely covered with the alkaline solution, before being heated up for 90mins at 120□ this was done to increase the lignin yield as adapted from Kolajo*et al* (2023). The solution was filtered to separate the solid component and to obtain a lignin solution with a pH of 11. To neutralize the alkalinity, sulfuric acid was added little by little until a pH of 2 was obtained.   3g of yeast was then added to the mixture to ferment the sugar content of the sawdust to which was present in the solution and left for 24hours. Using a centrifuge, the solid precipitate was obtained from the mixture and separated from the alcohol and water content. The solid precipitate was then dried in an oven to remove the remaining water content @ 70□ for 12hours. The black dry powder is the grinded into a fine powder which is lignosulphate used as a retarder.

2.3   Characterization of the Lignin Liquor Sample

Characterization plays a key role in identifying the makeup of a material, such as its chemical, physical, and structural properties. The lignosulphate proximate and ultimate analyses were determined using the ASTM 3174-76 procedure. The lignosulphate sample was also characterized by X-ray Powder Diffraction (XRD) using the 1.3000-1.7000 Automatic Laboratory Digital ABBE Refractometer,

Scanning Electronic Microscopy (SEM) using the Thermo Scientific Phenom Desktop SEM and the Fourier Transform Infrared (FTIR) using the using the Nicolet Apex KBr FTIR Spectrometer. The characterization experiment was conducted in the Multidisciplinary Research laboratory of the University of Ibadan and the Rolab Research and Diagonistic Laboratory, Ibadan.

2.4 Preparation of Cement Slurry Sample

The 3 slurry samples are Class G cement, Class G cement treated with 0.3% BWOC (By Weight of Cement) of Sodium Lignin and another treated with 0.5% BWOC (By Weight of Cement) of Sodium Lignin, with composition as presented in the Table 2.0. The varying concentration of the synthesized retarder is to ascertain its effect on cement slurry with increase in concentration. The following slurry samples were prepared according to the API 10A recommended standards. The entire process including formation and testing was carried out in the laboratory of SOWSCO Well Services, Port Harcourt, Rivers state. The class G cement was obtained from SOWSCO Well Services and kept in a plastic bag to prevent moisture and stored in a cool area to prevent it from caking or having lumps in it.

Table 2.0: Cement Slurry Samples

	Materials	Grams	Percent (%)	Specific Gravity	Pounds	Gal/Sk	Abs. Vol
	Cement	789.94	100	3.15	94	3.591	0.0382
	Sodium Lignin	2.37	0.3	0.00	0.282	0.000	0.1082
	Antifoam	3.39	0.43	0.97	0.404	0.050	
	Fluid loss	11.73	1.49	1.12	1.398	0.150	
Neat cement	Dispersant	0.00	0.00	0.00	1.260	0.000	
	Retarder	0.00	0.00	0.00	1.210	0.000	
	Water	335.24	42.29	1.00	39.936	4.800	
					135.73728	8.5908	
Slurry with 0.3% Sodium Lignin	Dispersant	0.00	0.00	0.00	0.000	0.000	
	Water	333.13	42.22	1.00	39.936	4.770	
					135.76968	8.5913124	
Slurry with 0.5% Sodium Lignin	Dispersant	0.00	0.00	0.00	0.000	0.000	
	Water	332.03	42.13	1.00	39.6032	4.760	
					135.87448	8.601654	

2.5 Evaluation of the Synthesized Cement Retarder Extracted from Delonix Regia

Several tests were conducted to evaluate the performance of the synthesized cement retarder which was obtained from *Delonix regia* sawdust by determination of the thickening time, rheological properties and compressive strength properties of Class G cement and a water content of 46% By Weight of Cement (BWOC). The testing conditions are specified in Tables 3.0 and 4.0.

Table 3.0: Well Data

	MD= 3550m	TVD= 11646ft		
S/N		1	2	3
BHST	□/□	90□/194□	90□/194□	90□/194□
BHCT	□/□	100□/212□	100□/212□	100□/212□
Bottom Hole Pressure (BHP)	psi	7000	7000	7000
Heat up Time	min	58	58	58

Table 4.0: Slurry Data

S/N		1	2	3
Slurry Density	ppg	15.80	15.80	15.80
Slurry Yield	cuft/sk	1.15	1.15	1.15
Total Fluid Fraction	%	44.40	44.14	44.05
Total Fluid Volume	gal	5.00	4.97	4.96

2.5.1 Thickening (Setting) Time Test

According to Alp et al, (2013), the thickening time or setting time test determines how long the cement slurry will be fluid before hardening or losing its pumpability. A High-Pressure-High-Temperature (HPHT) Consistometer with maximum pressure of 154 MPa (22 000 psi) and temperatures up to 204□ (400□) was used to conduct this test.

2.5.2   Consistency Test

The consistency test was carried out using a HPHT Consistometer (Chandler-Model 7222 & 8340). The slurry sample is poured into a consistency slurry cup (pressure vessel) and placed inside the chamber of the HPHT Consistometer which can turn at speeds up to 150 rpm. The HPHT Consistometer measures the cement slurry thickening time under high pressure and temperature even at downhole conditions.

2.5.3   Compressive Strength Test

The Compressive Strength test was performed using the Ultrasonic Cement Analyzer (Chandler-Model 4265) with maximum pressure and temperature of 20,000psi (138 MPa) and 204.4□ (400□) to determine the setting time and the Wait-On-Cement (WOC) time. The cement slurry was prepared according to API specifications and placed in the unit's temperature and pressure-controlled cell where the curing conditions expected simulate downhole conditions.

2.5.4   Rheology Test

The rheological property of cement slurries is necessary in the prediction of the frictional pressures that occur when pumping the different fluids down the well was determined using the Chandler API Viscometer. The rheology of fluids also has a major effect on solids setting and frees fluid properties and also on the friction pressures (Joel, 2009). Since rheological testing is typically conducted at atmospheric pressure, the maximum temperature is limited to about 190°F (Anon,1997) After blending, the slurry was conditioned using atmospheric consistometer to simulate the well condition of 120□ for 20 minutes. The conditioned slurry was poured into the viscometer cup and the bob of the viscometer was inserted into the cup and the thermometer of the viscometer was also inserted to record the temperature of the slurry. The viscosity was measured using the Fann viscometer and the readings were recorded.

3.0 RESULTS AND DISCUSSION

The characterization experiment was conducted in the Multidisciplinary Research laboratory of the University of Ibadan and the Rolab Research and Diagonistic Laboratory, Ibadan.

3.1 Characterization Result of the Lignosulphate Powder
### 3.1.1 The Sample Proximate and Ultimate analysis

The proximate and Ultimate analysis were performed in triplicate and the results presented in the Table 5.0. The determination of moisture content, Volatile content, Fixed Carbon and Ash contents, were obtained according to ASTM standard. The Ultimate analysis results was expressed in dry basis, though the sulfur and nitrogen contents completed 100%. their quantities were negligible considering the other elements.

Table 5.0: Proximate and Ultimate Analysis of the Lignosulphate powder Sample

| Sample	Moisture Content (%)	Volatile Content (%)	Fixed Carbon Content (%)	Ash Content (%)	C (%)	H (%)	N (%)	O (%)	S (%)	Hhv (Mjkg^{-1})
Lignosulphate powder	7.90 + 0.4	62.82+ 0.7	25.64 + 0.4	3.66 + 0.2	41.80 + 0.3	8.34 + 0.3	0.40 + 0.1	40.55+ 0.2	0.15 + 0.3	38.30

3.1.2: The SEM

The SEM images (Figures 2.0 a, b and c) clearly shows the shape and size distribution of the lignosulphate powder, with the Figures 2.0 b and c. presenting the larger magnified SEM images
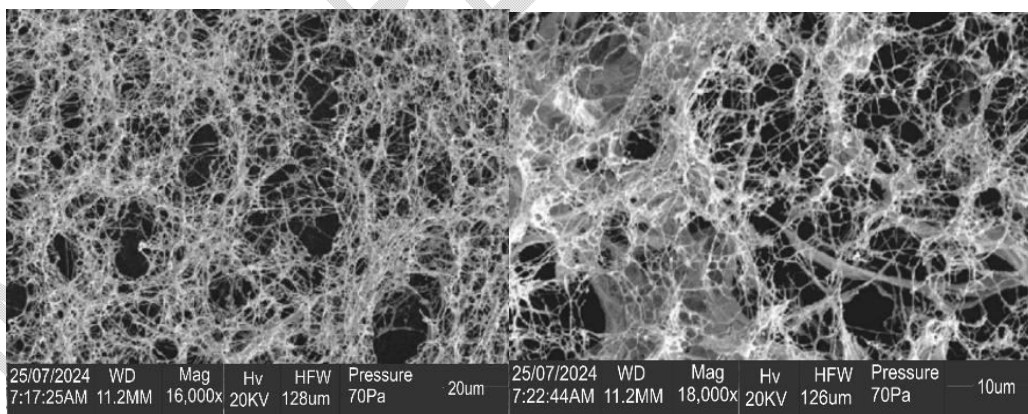
(a)   (b)
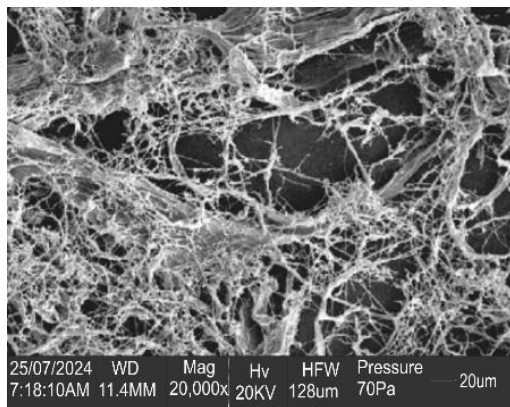
(c)

Figure 2.0 Lignosulphate powder SEM image of the lignosulphate powder at magnifications of (a) 1600 (b) 18.000 (c) 20.000.

3.1.2: The XRD

To investigate the intercalation of lignosulphate powder saw dust XRD analysis was used. The Figure 3.0 show the XRD pattern of the lignosulphate powder. From the result, it was observed that the lignosulphate powder had a diffraction peak at 10.900o is the peak, indicates that the poly (AAc-co-AAm) do not penetrate in the interlayer space efficiently and only covered the surface of SD. It may be due to the large molecular structure of poly (AAc-co-AAm). Similar results are obtained by Huang, et al. (2011) from their study, observed that when HACC was used for medication of bentonite, surface of bentonite was covered by the HACC.
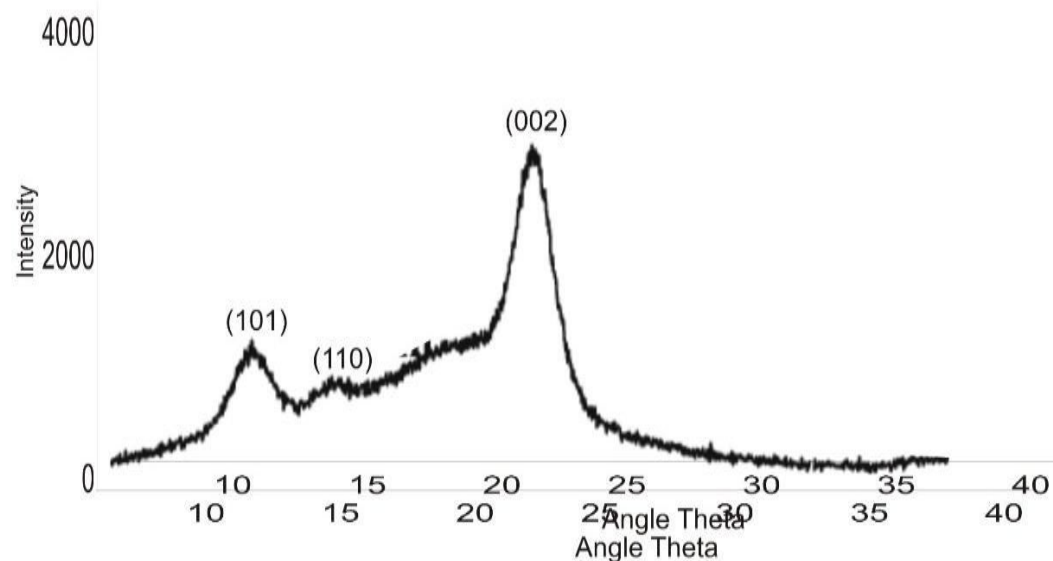
Figure 3.0: XRD Spectrum for the lignosulphate powder

3.1.3   Fourier-Transform Infrared (Ftir) Spectroscopy

The FTIR spectrum of the lignosulphate powder had 9 peaks that were observed in the result as seen in Figure 4.0. The Figure shown suggest a complex chemical nature with a variety of functional groups.
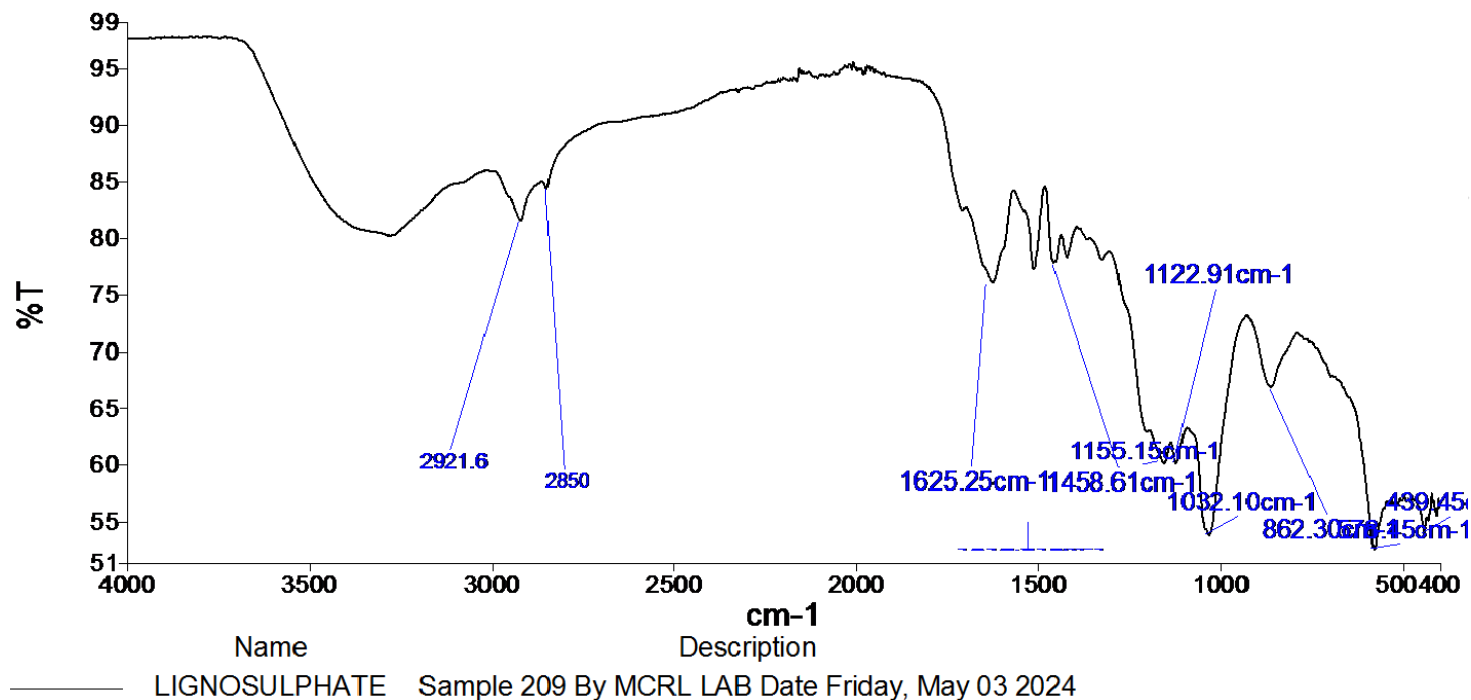
Figure 4.0: FTIR Spectrum of Lignosulphate powder

3.2   Thickening Time Test

The result of the setting time of the cement slurries shows that the neat slurry setting approximates the setting time proposed by the America Concrete Institute for neat slurry at 80□ which is around 4hrs. For this experiment, the obtained setting time for the neat slurry was 2hrs 04mins. Also, other slurry samples show that at different concentration addition of the formulated sample; the setting time increases progressively as seen in Table 6.0. The Figures 4.0-6.0 shows the thickening time test results for the neat slurry, slurry with 0.3% bwoc and slurry with 0.5% bwoc, with slurries set at 2hrs 04mins, 4hrs 04mins and 6hrs 13mins, respectively while Table 6.0 highlights the consistency values (in Bearden Unit of Consistency), concentration and the setting time.

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Table 6.0: Thickening Time

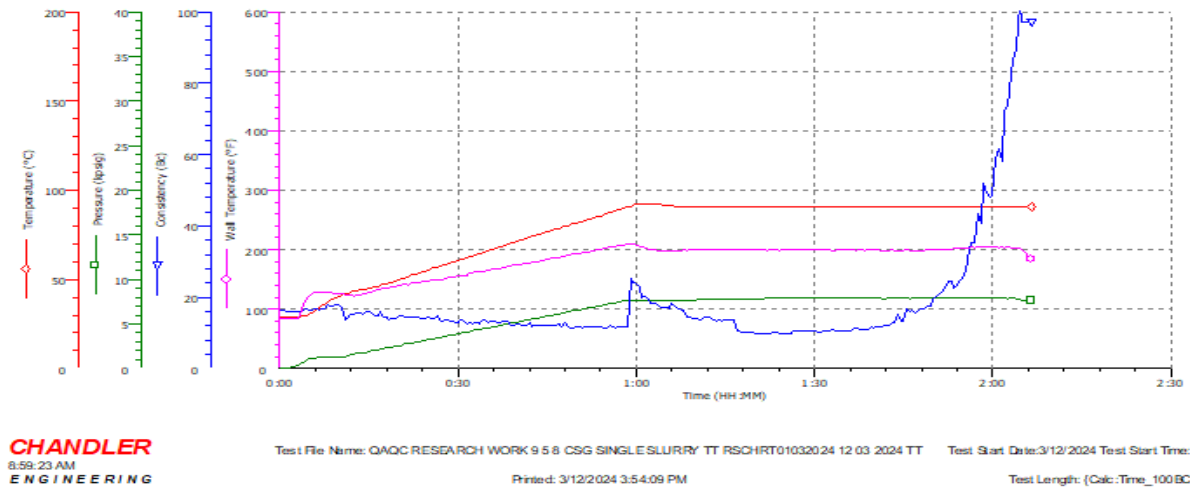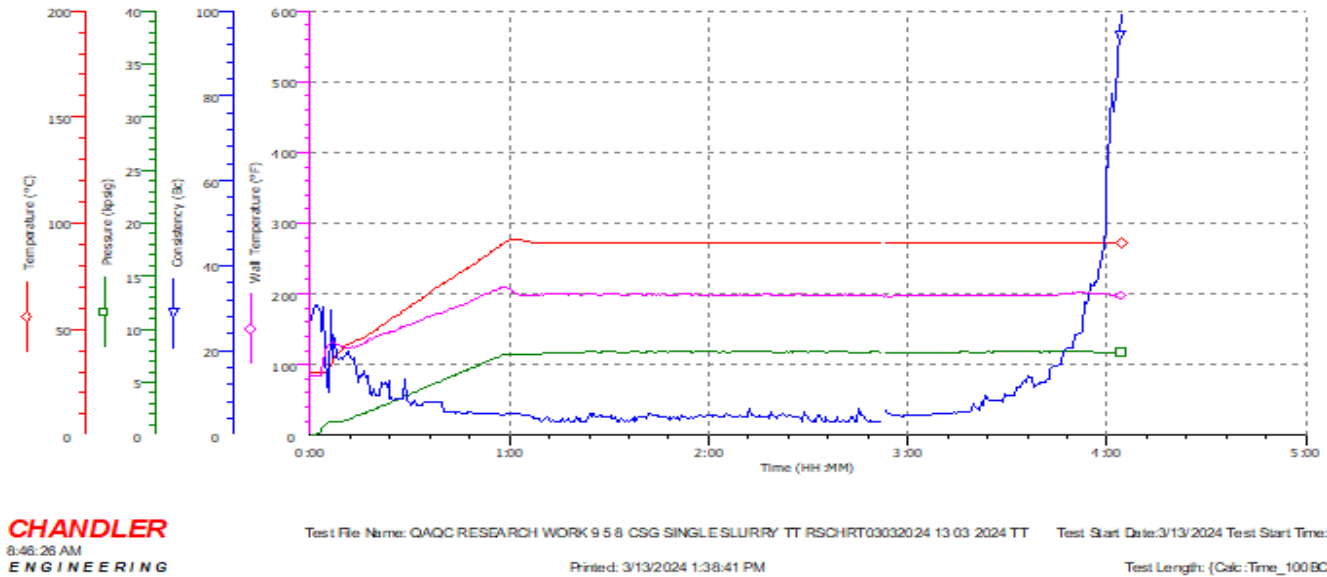| CONSISTENCY (BC)	CONCENTRATION (gal/sk)	SETTING TIME (hrs: mins)	NEAT SLURRY SETTING TIME (hrs:mins)
40BC	0.031	03:58	01:58
	0.051	05:50	
70BC	0.031	04:01	02:02
	0.051	06:09	
100BC	0.031	04:04	02:04
	0.051	06:13	

Figure 5: Thickening Time for Neat Slurry

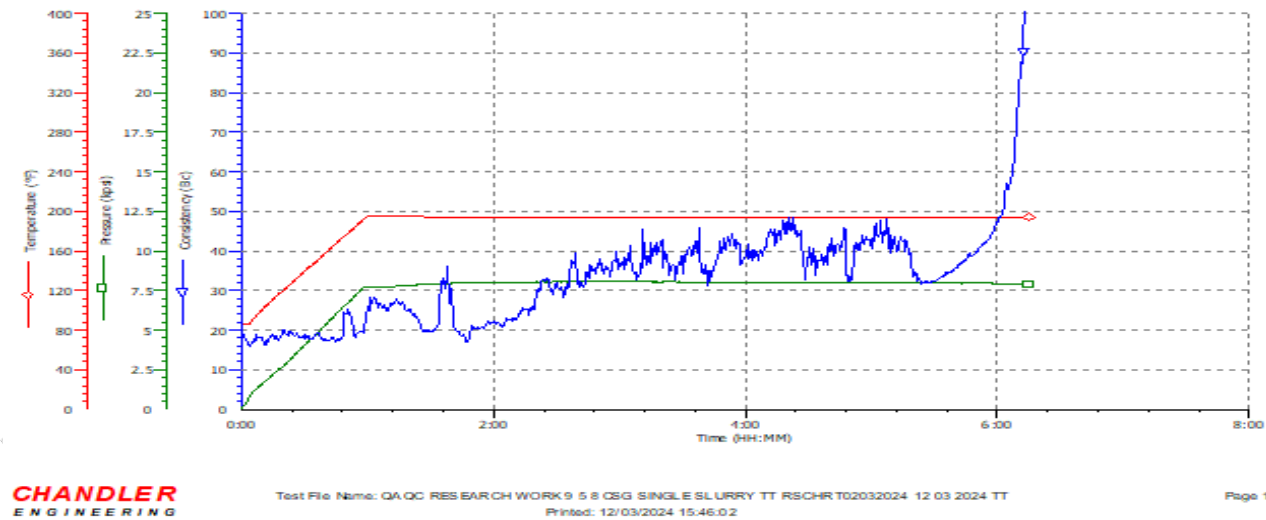**Figure 6: Thickening Time for 0.3% bwoc of locally synthesized retarder**

Figure 7: Thickening Time for 0.5% bwoc of locally synthesized retarder

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3.4 Rheology Test

The Table 7.0 shows the rheology of the slurry at 90☐/194☐, it was observed that as the concentration of the retarder increases the yield point decreases, which was in agreement with the findings from Joel, (2009) and Anaele et al. (2019). For good cement slurry from operational point, the slurry yield point must not be less than 6 lbf/100ft^2. This is very important to avoid settling down of the cement particles.

Table 7.0: Rheology Test Results

SPEED (rpm)	NEAT SLURRY		0.3% BWOC SAMPLE		0.5% BWOC SAMPLE	
	AMBIENT	BHCT	AMBIENT	BHCT	AMBIENT	BHCT
600	187	140	160	169	148	158
300	103	78	69	103	55	115
200	70	55	46	76	38	84
100	34	28	23	43	17	57
60	20	18	14	29	9	42
30	10	9	8	17	6	26
20	7	7	5	13	4	23
10	4	4	3	8	3	11
6	4	3	3	6	2	10
3	3	2	2	4	2	5
10sec Gel	2	-	2	-	2	-
10min Gel	3	-	2	-	2	-

PV, cp	84	62	71	66	64	68
YP, lbf/100ft^2	19	16	-2	37	-3	40

Table 8.0 Cement Slurry Composition

Dry Components	Neat						0.3% BWOC SAMPLE						0.5% BWOC SAMPLE					
	Grams	Percent %)	Specific Gravity	Pounds	Gal/Sk	Abs. Vol	Grams	Percent %)	Specific Gravity	Pounds	Gal/Sk	Abs. Vol	Grams	Percent %)	Specific Gravity	Pounds	Gal/Sk	Abs. Vol
Cement	789.99	100	3.15	94	3.591	0.0382	789.94	100	3.15	94	3.591	0.0382	788.99	100	3.15	94	3.591	0.0382
SodiumLignin	0.00	0.00	0.00	0	0.000	0.1082	2.37	0.3	0.00	0.282	0.000	0.1082	3.97	0.5	1.11	0.47	0.051	0.1082
Liquid Components																		
Antifoam	3.39	0.43	0.97	0.404	0.050		3.39	0.43	0.97	0.404	0.050		3.38	0.43	0.97	0.404	0.050	
Fluidloss	11.73	1.49	1.12	1.398	0.150		11.73	1.49	1.12	1.398	0.150		11.72	1.49	1.12	1.398	0.150	
Dispersant	0.00	0.00	0.00	1.260	0.000		0.00	0.00	0.00	0.000	0.000		0.00	0.00	0.00	0.000	0.000	
Retarder	0.00	0.00	0.00	1.210	0.000		333.13	42.22	1.00	39.936	4.770		332.03	42.13	1.00	39.6032	4.760	

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Water	335.24	42.29	1.00	39.936	4.800				135.76968	8.5913124				135.87448	8.601654	
				135.73728	8.5908											

4.0 CONCLUSION

The results obtained from various tests demonstrate that sawdust can effectively reduce the setting time of cement slurries, particularly observed with Class G cement, thus acting as a retarder in the oil and gas industry. Sawdust, a byproduct of wood processing that typically contributes to environmental pollution, can be repurposed efficiently, potentially serving as a revenue source and offering an alternative to imported cement retarders in the industry.

Converting sawdust into a retarder represents a significant step in addressing the environmental threats posed by this waste material, thereby contributing to cleaner and healthier surroundings. Moreover, the tree utilized in this study is readily available and easily accessible, making it a viable alternative for retarders in the industry. The locally synthesized retarder demonstrates the capability to retard Class G cement with a density of 15.80 ppg and a water content of approximately 42% bwoc. Additionally, the study reveals a decrease in the yield point of the cement slurry as the concentration of the locally synthesized cement retarder increases.

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