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International Journal of Petrochemical Science & Engineering

Research Article

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Cement slurry set retarding potential of oil palm kernel shell wastes for oil well operations

Abstract

One major problem of the agro –industries in Nigeria is the management of its wastes generated from its processes. These wastes can be converted to useful products and are attractive for being an alternative to solving the disposal issues in the industry. This study assessed the synthesis of lignosulfonates from Oil Palm Kernel Shells (OPKS) and evaluated its performance as a retarder additive for oil well cementing operations. OPKS was synthesized using the pulping process and filtered to isolate the lignin from cellulose and hemicellulose. Sulfonating process was carried out using Sodium Bisulfite ($NaHSO_3$) to produce sodium lignosulfonate. Thereafter, Fourier Transform Infrared spectroscopy (FTIR) was applied to determine the functional groups present in the synthesized product. The product was evaluated for its performance as a retarder through the thickening time test using an automated HTHP Cement Consistometer at different temperatures, concentrations and consistency (Bc). A linear increment of cement slurry thickening time up to 40% with increase in concentrations was observed in relation to a reference (commercial retarder). The results also showed that lignosulfonates from OPKS has the potential to retard the set of cement slurry, however to maximize its retardation function, more investigations needs to be carried out to purify and modify the synthesized lignin. Its utilization as a cement retarder will promote the preservation of the environment and also reduce the overdependence on expensive cement additives.

Keywords: retarders, additives, cement slurry, wastes, thickening time

Volume 6 Issue 1 - 2023

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Received: March 21, 2023 | **Published:** April 14, 2023

Abbreviations: OPKS, oil palm kernel shells; FTIR, fourier transform infrared spectroscopy; HTHP, high temperature, high pressure; OPK, oil palm kernel; PKO, palm kernel oil; PKC, palm kernel cake; FFB, fresh fruit bunch; OPEFB, oil palm empty fruit bunch; OPMF, oil palm mesocarp fibre; OPF, oil palm fronds; OPT, oil palm trunks; POME, palm oil mill effluent; API, american petroleum institute; CPO, crude palm oil; $NaHSO_3$, sodium bisulphite; Bc, bearden consistency; SDGs, sustainable development goals; NaOH, sodium hydroxide; H_2SO_4, sulphuric acid; KBr, potassium bromide

Introduction

A major problem in the agricultural industries in developing countries like Nigeria is the management of wastes. Agro wastes in Nigeria are mostly subjected to open air burning which is associated with environmental pollution.[1] Inefficient and improper disposal of these solid wastes create serious hazards to public health, including pollution of air and water resources and increase in rodent and insect vectors of disease, creates public nuisances as well as interfere with community life and development.[2] The failure or inability to salvage and re-use these natural materials economically results in unnecessary waste and depletion of natural resources.

The oil palm industry for instance is one of the major agro businesses in Nigeria and has been recognized for its contribution towards economic growth and rapid development.[3] Oil palm tree is found in about 27 states in Nigeria, making Nigeria the fifth largest producer of oil palm in the world with domestic production of 930,000 metric tons which accounts for about 1.5% of global output. Oil palm have found application in food industry,[3,4] industry (an active ingredient for making soap, detergents, cosmetics, lipsticks, pharmaceutical products.[5]

The main products of the oil palm industry are crude palm oil (CPO) and oil palm kernel (OPK), which yields another type of oil known as palm kernel oil (PKO), and residue known as palm kernel cake (PKC). The wastes generated from processing the Fresh Fruit Bunch (FFB) are known as FFB wastes. Almost 60% –70% of the volume from the processing of FFB is removed as wastes. The solid wastes include Oil Palm Empty Fruit Bunch (OPEFB), Oil Palm Mesocarp Fiber (OPMF) and Oil Palm Kernel Shell (OPKS), Oil palm Fronds (OPF) and Oil Palm Trunk (OPT) which represent important biomass in the oil palm industry.[5,6] Another important waste of the industry is Palm Oil Mill Effluent (POME), which comprises of all the liquid wastes generated in the palm oil mills.

As the demand for palm oil increases, the wastes generation also follows a similar trend. These generated wastes from the oil palm industry in developing countries like Nigeria are seldom in use except for use as boilers fuel. These wastes are basically burnt in the palm oil mills resulting to atmospheric pollution which impacts on air quality.[6] However, naturally occurring materials can be turned to value added resources for applications in oil and gas sector as well. The use of these materials has become necessary due to the environmental requirements that are associated with the disposal of oilfield chemicals after use, sustainability of raw materials for producing the chemicals, and the need for reducing the overall cost of oil and gas production.[7]

Basically, the oil and gas industry in its quest to contribute to sustainable development goals has over the years encouraged the use of green materials in its operations. Economically, the cost of these materials is low thereby achieving various targets in low cost of production. In drilling fluid design, the effectiveness of various mud formulations composed of by products from agro wastes has been reported.[8–10] For instance, the use of date palm fiber as a sorbent for clearing oil spills has also been explored. Also, surfactants developed from wood pulping have been used in enhanced oil recovery jobs.[6] These wastes can be modified and used as alternatives to inorganic chemicals in the oil and gas production processes.

Oil and gas production processes involves the building of oil wells based on cycles of drilling, casing and cementing job going down section by section until the target or pay zone is reached. For each section cement slurry is designed and placed to fill the annulus space between formation and case. Oil well cement is applied to fill the annular space between the pipe and rock formation, displaces the drilling fluids, support the casing and protect it against corrosion and impact loading, restricts the movement of fluids between formations, and isolates the productive and none productive zones.[11] However, the cement slurry must withstand higher pressures and temperatures encountered in down hole applications.[12]

During the movement along the drilled hole for cementing operation, there is a big temperature difference between the top and bottom of cement slurry column.[9] This large temperature difference would cause retardation and super-retardation in the top of cement slurry column, which not only delays the process of drilling operation, but also increases the security risks of subsequent drilling operation.[8] Hence, accurate control of the setting or thickening time of the cement slurry is necessary. That is the time after initial mixing when the cement can no longer be pumped, is crucial in this process. If the thickening time is too short, the cement fails to reach its required placement, whilst too long a thickening time leads to costly delays.

Operational problems as a result of short thickening times are especially dramatic because the cement can set prematurely in the casing or pumping equipment.[13,14] Cementing operations are mostly carried out at high temperatures and high pressures (HTHP) in wells and this requires cement formulation that are properly design to withstand these conditions. Hence, chemical additives are used to modify cement slurry's behavior in order to meet those requirements. Over the last decades, various types of additives are used to design cement slurry for successful filling of the annulus space between the casing and geological formation. One of such additives is known as retarders which are used to increase the thickening time of cement slurry, thus postponing cement set and allowing more operational time for placement of slurry in the annulus space.[15]

Currently, the retarders used in the industries are mostly imported and the costs keep increasing year by year[16] as compared to the price of crude in the international market. Therefore, researchers are encouraged to continuously look for potential additives from locally sourced materials such as oil palm biomass to harness the chemical properties of these resources. This will increase the energy index of the country especially in the area of oil and gas exploration and production and adequately use the opportunities available in the oil industries.[1] Again, The Nigerian Content Act, seeks to achieve the promotion of the development of local (Nigerian) content (both personnel and resources) in the oil industry as one of its major goal, with this in view, the need for the development of local additives as substitute for foreign additive is paramount.[17,18]

Hence, in a bid to encourage the conversion of natural resources such as oil palm kernel shells into useful products, this study is set up to experimentally synthesize, characterize and evaluate the performance of the Oil Palm Kernel Shell (OPKS) in cement slurry formulation as slurry set retarding additive. Oil Palm kernel shell (OPKS) as a major by-product of palm oil production has in recent times become an important renewable biofuel source because of the global concerns about the environmental effects of extensive use of fossil fuel. It is currently exploited for on-plantation energy generation, and also being exported by some industrial-scale palm oil producers in Asia.[19]

The usage and industrial applications of the palm kernel shells could be categorized into three major sections such as:

- Energy/fuel production,
- Aggregates for concrete reinforcement, and
- Water purification.[19]

The material however remains under - exploited in Nigeria, since it comprises of the following components from investigations (Table 1). One of such components is Lignin, this can be explored for different industrial applications, particularly for its retarding potential for cement slurry set. OPKS has lignin content of about 50%,[18,20] thus there is need to explore new technologies for the conversion of lignin to Lignosulphonate which is a known natural retarder.[21] Adoption of new technologies will not only create channels for innovative oil palm waste management but also create jobs in the region.

Table 1 Oil palm chemical composition

Biomass	Chemical composition (% dry wt)				
	Cellulose	Hemicellulose	Lignin	Extractives	Ash
Oil Palm Empty Fruit Bunch (OPEFB)	38.3	35.3	22.1	2.7	1.6
Oil Palm Mesocarp Fiber (OPMF)	33.9	26.1	27.7	6.9	3.5
Oil Palm Kernel Shell (OPKS)	20.8	22.7	50.7	4.8	1
Oil Palm Fronds (OPF)	30.4	40.4	21.7	1.7	5.8
Oil Palm Trunk (OPT)	34.5	31.8	25.7	3.7	4.3

Source: Norfadhilah et al.[18]

Hence, this study would focus on the synthesis, characterization and evaluation of the performance of the synthesized retarder from OPKS. Also, a critical look at the Sustainable Development Goals (SDGs) shows that a lot depend on a wholesome environment, hence, this study also seeks to address the following SDGs: Goal 7: Affordable and clean energy which is to ensure access to affordable, reliable, sustainable and modern energy for all. Goal 8: Decent work and economic growth which promote inclusive and sustainable economic growth, employment and decent work for all. Goal 9: Industry, Innovation and infrastructure which builds resilient infrastructure, promote inclusive and sustainable industrialization and foster innovation and Goal 12: Responsible consumption and production which ensures sustainable consumption and production patterns. That is to achieve the sustainable management and efficient use of natural resources so as to substantially reduce waste generation through prevention, reduction, recycling and reuse in order to minimize their adverse impacts on human health and the environment.[22]

Materials and methods

Materials required for this study include but not limited to the following: Portland cement, Oil palm kernel shells (Plate 1), Extractive additives: NaOH, $NaHSO_3$ and H_2SO_4, Imported retarder, Defoamer. And some of the major equipment include but not limited to the following: speed blender (Plate 2), viscometer (Plate 3), Atmospheric consistometer (Plate 4), HTHP consistometer (Plate 5).

Plate 1 Oil Palm kernel shells.

Plate 2 Warring Blender.

Plate 3 Fann Viscometer.

Plate 4 Atmospheric Consistometer.

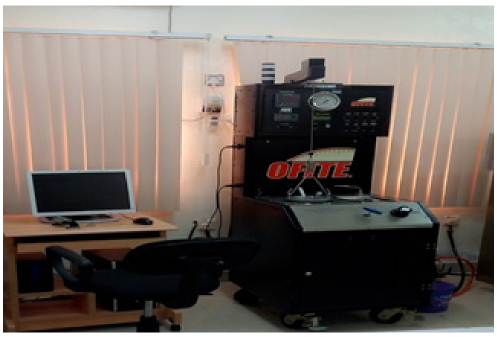

Plate 5 HTHP Consistometer.

Research design

The study was carried out in three sections, namely:

a. Synthesis or formulation of the retarder from OPKS

b. Characterization of the synthesized retarder

c. Determination of thickening time.

The research design for this study is represented in the flowchart of (Figure 1).

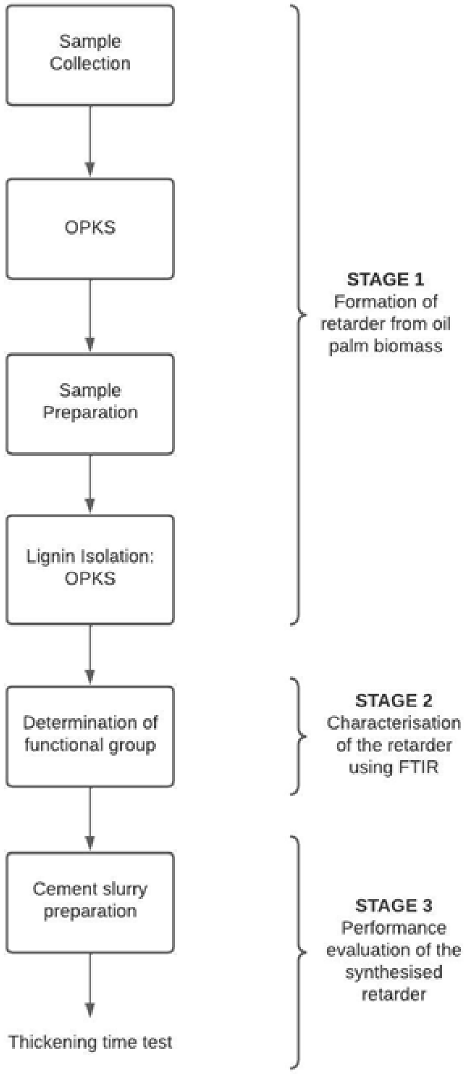

Figure 1 Flowchart for the Research Design.

Synthesis or formulation of the retarder from OPKS: lignin isolation

Oil palm kernel shells (OPKS) were collected stored in polyethene bags at room temperature until ready for use. They were sun dried to remove moisture, drying was completed in a moisture extractor oven. The samples were selected to remove debris and unwanted particles and thereafter crushed and milled using an industrial crusher to achieve reduction in particle size. The milled samples were sieved with a sieve of mesh size 0.25mm to get uniform samples for the extraction of lignin. The process of extraction of Lignin from the selected biomass followed the procedures as recorded in.[23]

40 grams of the sieved biomass (OPKS) was measured and put into a 2000ml round bottom flask. 2M of Sodium Hydroxide (NaOH) solution was dripped into the flask until the biomass is submerged. The sample was mixed thoroughly and fixed in a heating mantle at 120^{o}C for extraction time of four hours. Upon completion of extraction, the sample in the flask was filtered to remove cellulose and hemicellulose components from the lignin and the filtrate was neutralized using 98% sulphuric acid until the pH reach a value of about 2.[24] The extracted lignin was oven dried at a temperature of 70^{o}C for 3 hours to remove the moisture content in the lignin before sulfonation; this is to avoid water interference in the reaction.

Sulfonation reaction was carried out to produce lignosulfonate. 1M Sodium bisulfite ($NaHSO_3$) solution was prepared and 200ml of the prepared solution was added to the extracted lignin in the reactor. This was heated at a regulated temperature of 100^{o}C for 5 hours to get the product (lignosulfonate) from the oil palm kernel shells (OPKS).

The procedure for the formulation of the retarder from OPKS is represented in the flowchart of (Figure 2) and the formulated retarder is shown in Plate 6.

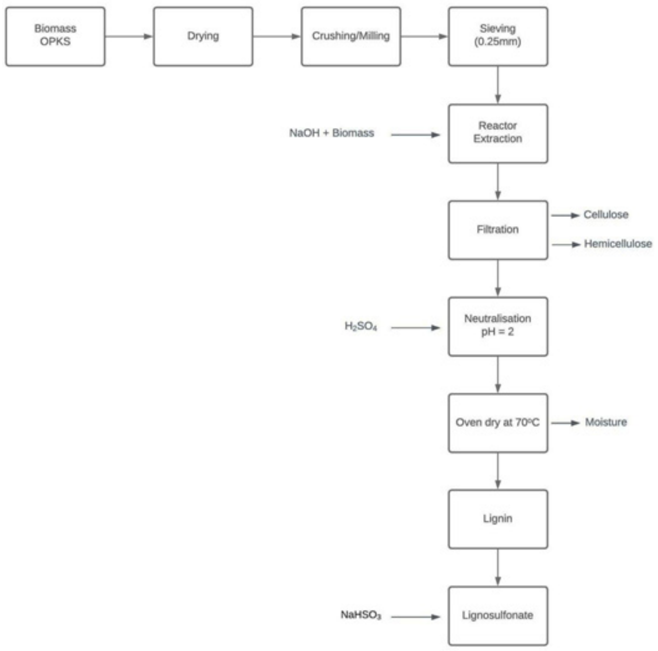

Figure 2 Flowchart of the formulation of the Lignosulfonate from OPKS.

Plate 6 Synthesized Lignosulfonate from OPKS.

Characterization of the synthesized retarder

The Fourier Transform Infrared spectroscopy (FTIR) was applied to determine the functional group present in the synthesized retarder. FTIR analysis was performed for all samples isolated to have a result regarding the bio minerals. A few crystals were mixed with KBr (Merck for spectroscopy) and pulverized in an agate mortar to form a homogenous powder from which, under a pressure of 7 tons, the appropriate pellet was prepared. All spectra were recorded from 4000 to 400 cm^{-1} using the Pelkin Elmer 3000 MX spectrometer. Scans were 32 per spectrum with a resolution of 4 cm^{-1}.The spectra were analyzed using the spectroscopic software Win-IR Pro Version 3.0 with a peak sensitivity of 2cm^{-1}.

Evaluation of the synthesized retarder

The performance evaluation of the synthesized retarder was carried out following the procedures described thus:

Cement slurry preparation

Quantities of G –cement, fresh water, deformer and the synthesized retarder from OPKS was weighed and blended according to API recommended practice 13B-2 standard using a warring blender, liquid components of the additive was also measured by its volume equivalent using syringe while the solid components were measured by their mass equivalent using electronic weighing balance.[25] The warring blender was turned on and maintained at a speed of 4000±200rpm, the liquid components and water were poured into the blending cup, thereafter the solid components were added and finally the cement was added within 15 seconds and the speed of the blender was increased to 12000rpm for 35 seconds to ensure homogeneity.

Thickening time testing

The API thickening time test is the accepted method for measuring how long cement slurry should remain pumpable under simulated down-hole temperature and pressure conditions. The test was performed in an Ofite Model 2040 Automated HTHP Cement Consistometer that is usually rated at pressure up to 30,000 psi and temperatures up to 400^{o}F (204^{o}C). HPHT consistometer plots the consistency of a slurry over time at the anticipated temperature and pressure conditions as precisely as possible. The end of the thickening time test is considered at 40Bc or 70Bc for most applications.[7] The test involved mixing the cement slurry according to current API procedures, placing the slurry into the slurry cup, and then placing the slurry cup into the Consistometer for testing.[26]

The HPHT Consistometer was started and the cup containing the mixed cement slurry was gently placed in the Consistometer cup and rotated slowly until the bottom cell cap engages to the drive. This was done within 5 minutes of preparing the cement slurry, after which the consistometer motor was turned on and the drive started rotating at 150rpm. The potentiometer was then placed on the rotating slurry to engage correctly, an initial consistency reading proportional to the viscosity of the cement slurry was observed. The Consistometer head was carefully lowered into the HPHT chamber and spine down to lock. The gland nut fitting was inspected and the thermocouple was inserted through the port in the center of the consistometer head. This was tightened by hand, the air exhausted and pressure release valve were closed and the air supply valve opened (allowing air to pass from the reservoir into the Consistometer pressure chamber). The controller was thereafter programmed using the laboratory calculation sheet to perform the test in line with the design parameters). The heater was switched on and the timer clock reset and the test started. The test was also periodically checked to ensure it was running as programmed. The controller was stopped at the end of the test.

In this study, the test was carried out with the prepared slurry and different concentrations of the synthesized retarder: 0.03gal/sk, 0.07gal/sk and 0.10gal/sk. The testing temperature and pressure of 100°F and 30000psi respectively (the chosen parameter condition value as a reference point) were maintained and controlled to mimic the down-hole condition of the well that the slurry will be subjected in the well formation. The test was stopped as soon as the cement slurry attained a consistency that showed that the cement slurry was unpumpable and had lost its hydrostatic property. The time taken to reach this consistency is known as the thickening time. The test was concluded when the consistency of the slurry reached 70Bc (Bearden Consistency).

Results and discussion

Characterization using FTIR spectroscopy

The synthesized retarder from OPKS was characterized using FTIR spectroscopy and the result is presented in Figure 3. Also, the spectrum for the imported retarder used for comparative studies is also represented in Figure 4.

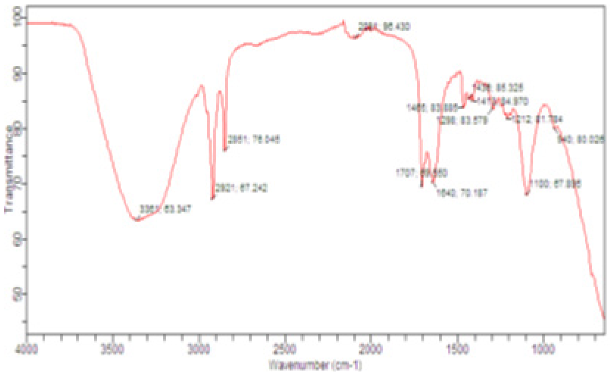

Figure 3 FTIR analysis result for synthesized retarder.

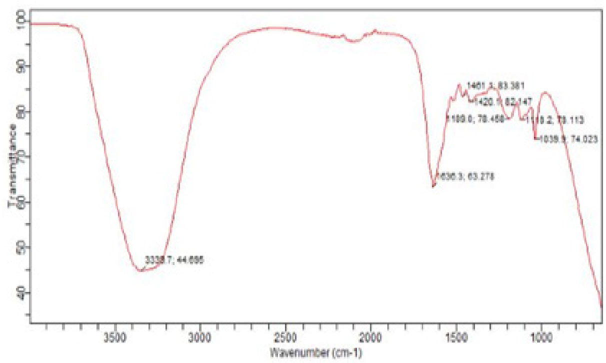

Figure 4 FTIR analysis result for imported retarder.

From Figure 3 (FTIR spectrum of formulated retarder from OPKS), the wavelength band is in the neighborhood of 3300cm^{-1} – 3400cm^{-1} with peak at 3357.7cm^{-1} this could be attributed to phenolic and aliphatic OH groups and those between 1900cm^{-1} – 2200cm^{-1} wavelength shows multiple peaks approximately at 2821.6cm^{-1} and 2854.7cm^{-1}, this might be associated with the aliphatics C-H and aromatics stretch groups. The wavelength band between 1700cm^{-1} – 1400cm^{-1} with peaks at 1640.6cm^{-1} and 1711.6cm^{-1} indicates the presence of alkenes C=C stretching while the multiple peaks at 1413.8cm^{-1}, 1436.8cm^{-1} and 1465.8cm^{-1} indicates the presence of ketone C=O group in the sample. Again, the wavelength number of 1212.8cm^{-1} - 1298.8cm^{-1} and 940cm^{-1} –1097.6cm^{-1} might indicate the presence of the amine C-N groups and the carboxylic acids group respectively. Also the complex vibration associated with the C-O, C-C stretching and C-OH bending in polysaccharides. Finally the band at approximately 600cm^{-1} – 740cm^{-1} might be associated with the ester S=OR and the sulfonic S=O groups which were formed as a result of sulphonation reaction of sodium with the OH group of the aliphatic side chain of the lignin, this is similar to that reported by Anaele et al.,[13] Setiati et al.,[14] Watkins et al.,[23] and Heradewi.[24]

Furthermore, Figure 4 is the FTIR spectroscopy analysis of the imported cement retarder; which shows a deep and broad peak at the functional group zone having a wave number of 3333.7cm^{-1} showing the presence of OH^- group in the phenolic and carboxylic acid compounds. Also, there is a variable intensity in the region of wave number between 1900cm^{-1} – 800cm^{-1}, in the region there is a shallow peak of 1636.3cm^{-1} showing aromatic group, the peak with wave number of 1039.9cm^{-1} represent the complex vibration associated with the C-O, C-C stretching and C-OH bending in polysaccharides. finally the band at approximately 640cm^{-1} represent the sulfonic groups which are formed as a results of sulphonation reaction of sodium bisulphite with the OH^- group of the aliphatic side chain of the lignin. Again, this is similar to that reported by.[20–22,27]

Hence, from the FTIR analysis, it can be deduced that the synthesized retarder has the basic components of a typical lignosulphonates: the phenols O-H, aliphatics and aromatics C-H, Ketones C=O and Esters S-OR.[17]

Performance evaluation: slurry preparation

Tables 2 & 3 represents the standard laboratory quantity used for the investigation of thickening time for neat slurry (cement and water only) and the slurry with 5ml of the formulated retarder respectively. The prepared slurries (neat and slurry with retarders) were allowed to set at surface temperature. From the laboratory experiment, the obtained thickening or setting time for the neat slurry was 300mins, this is similar to that reported by[14] and the setting time for the slurry with 5ml of OPKS retarder was 420mins while that of the imported retarder was 530mins as represented in Figure 5. The synthesized retarder from OPKS showed a 40% increase in thickening time compared to that of the neat slurry.

Table 2 Standard laboratory quantity for neat slurry

Components	Weight (gram)	Volume (ml)
Class G cement	770	243.04
Fresh water	364	356
Deformer	0.87	0.96
Total	1134.87	600
Mix fluid		356.96

Table 3 Cement slurry recipe for laboratory test with 5ml of formulated samples

Components	Weight (grams)	Volume (ml)
Class G cement	770.13	243.04
Fresh water	360.03	351
Deformer	0.87	0.96
Samples	5.66	5
Total	1136.69	600
Mix fluid		356.96

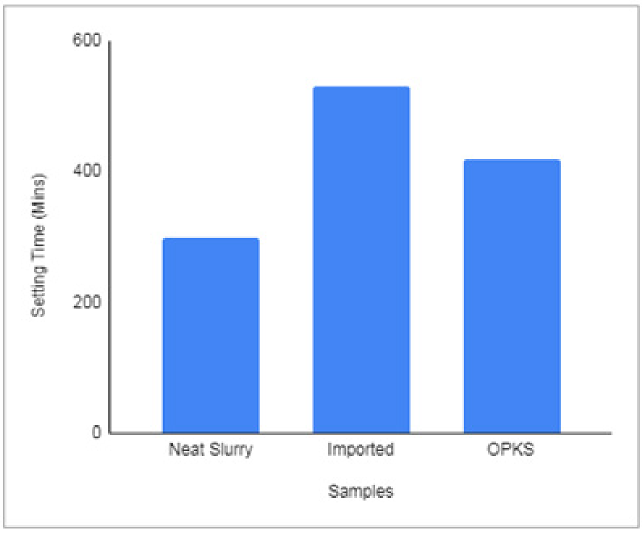

Figure 5 Thickening time for the different samples at surface temperature.

Comparing the performance of the imported retarder to the synthesized retarder showed that the imported retarder performed better by retarding the cement slurry for 530mins at surface temperature. However, the synthesized retarder from OPKS has the potential to increase the setting time of cement slurry for about 120mins as compared to the setting time of the neat slurry. Hence, more investigations can be carried out to improve the performance of this retarder as it is biocompatible with the environment.

Thickening time at different temperatures, concentrations and consistency (Bc)

From (Figures 6–9), it is observed that as the concentration of the synthesized retarder from OPKS increased from 0.03gal/sk to 1.00gal/sk, the thickening time also increased for 40Bc and 70Bc. It was also observed that as the temperature of the cement slurry increased, the thickening time reduces for a particular retarder concentration; this is similar to that reported by.[28] Also, this shows that temperature has a significant effect on thickening time. Again, it was observed that as the temperature of the cement increases the rate of change of the thickening time with respect to retarder concentration tend to increase faster at higher retarder concentration. This indicates that the thickening time increases rapidly at a higher temperature when the concentration of the retarder is high, this is similar to that reported by.[13,29]

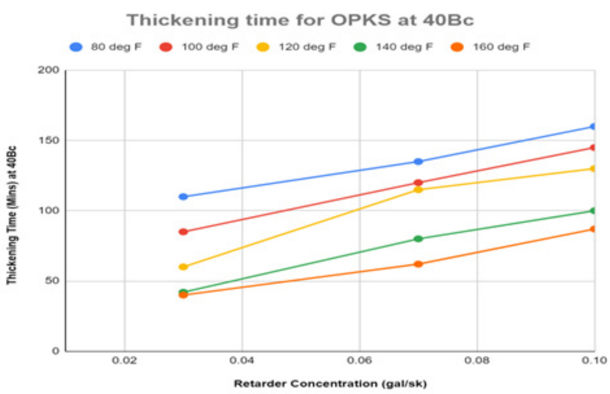

Figure 6 Thickening time for OPKS at 40Bc.

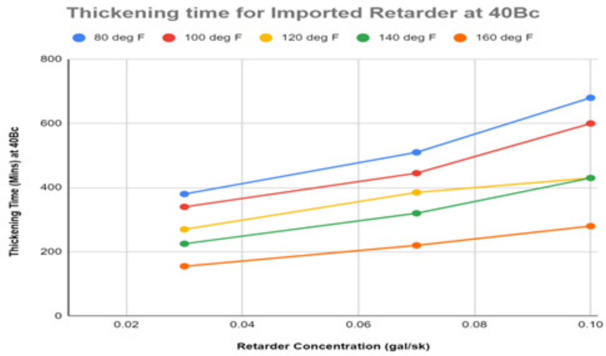

Figure 7 Thickening time for Imported retarder at 40Bc.

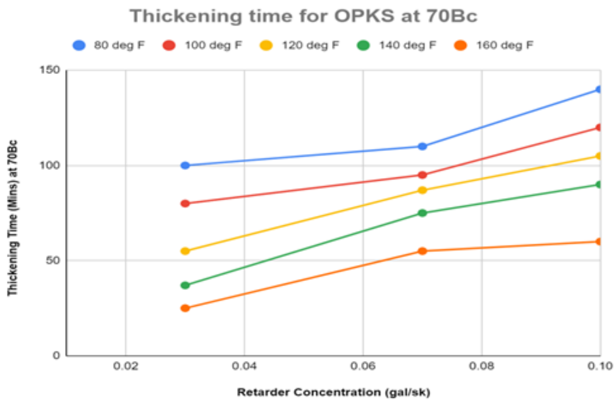

Figure 8 Thickening time for OPKS at 70Bc.

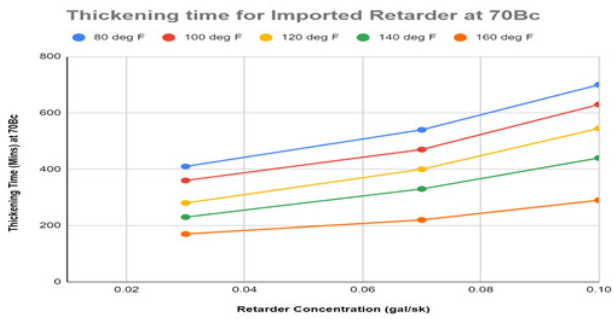

Figure 9 Thickening time for Imported Retarder at 70Bc.

In addition, it was observed that although the lignin content of OPKS is above 40% by dry mass,[18] its performance is low as compare to the imported retarder. This might be as a result of the physical and chemical properties of the OPKS lignin which is highly depended on the extraction or isolation technique applied.[27] Again, several other components were identified in the lignin from OPKS as shown by the multiple peaks in the FTIR spectroscopy analysis (Figure 3). One of such component is the amine group, generally, calcium and alkali chlorides are the main kinds of chloride based accelerators, while nitrates, nitrites, thiocyanates, formats and alkanoamines are chemicals commonly used as non-chloride accelerators. Alkanoamines which might be a product of phenols OH and the amine C-N groups present in OPKS could be the reason for the poor performance of the OPKS because it could act as an accelerator at certain concentration.[30]

Actually, there are three types of lignosulfonates: filtered, purified and modified,[21] the synthesized retarder from OPKS is only filtered, no modification or purification was carried out on it while the imported retarder is synthetic or inorganic and hence modified to fit its purpose of retarding cement slurry for oil well application.

Conclusion

Naturally occurring materials in form of wastes especially from the agro - industries can be turned to value-added resources for applications in oil and gas production. The use of these materials has become necessary due to the environmental requirements that are associated with the disposal of oilfield chemicals after usage, sustainability of raw materials for producing the chemicals, and the need for reducing the overall cost of hydrocarbon production. OPKS possess valuable potential to supplement the energy supply of the oil and gas industry through sustainable renewable energy technologies.[31,32] It is a major by-product of palm oil production and has become an important renewable bioresources. This study has shown that although the commercial retarder performed better in terms of thickening time of cement slurry, OPKS has the potential to be utilized as a retarding additive in cementing operations because of the reasonable amount of lignin (>40%) in the kernel shells. Sulphonation was carried out on the product and characterized using FTIR. The synthesized product is shown to contain the basic components of lignosulphonates from analysis. An increase in the concentration of the synthesized product causes a corresponding increase in the thickening time of the cement slurry. Thus this study has shown that lignosulphonate can be formulated from isolated lignin from oil palm kernel shells. And it has a huge potential to be cement slurry set retarding additive but more investigation are necessary to purify and modify the lignosulphonates to enhance its performance as a retarder for oil well cementing applications.

Acknowledgements

None.

Conflicts of interests

The authors declare that there is no conflicting interest.

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Citation: Altraide I, Oji A, Ogbonna JF. Cement slurry set retarding potential of oil palm kernel shell wastes for oil well operations. *Int J Petrochem Sci Eng.* 2023;6(1):1–8. DOI: 10.15406/ipcse.2023.06.00126