

THE EFFECTS OF LIGNIN PURIFICATION ON THE PERFORMANCE OF IRON COMPLEX DRILLING MUD THINNER

M. N. MOHAMAD IBRAHIM^{1*}, H. AZIAN² & M. R. MOHD YUSOP³

Abstract. Soda lignin was extracted from black liquor of oil palm empty fruit bunch (EFB) fibers by 20% (v/v) sulfuric acid. The yield of lignin, which is 3.016 grams from every 200 mL of black liquor, was then purified by *n*-pentane using soxhlet apparatus for six hours. A comparison study between purified lignin and standard lignin was then performed through physico-chemical properties and structural features using a Fourier transform infrared spectroscopy (FTIR), ultraviolet spectrometry (UV), gel permeation chromatography (GPC), high performance liquid chromatography (HPLC), and ash content test. The FTIR and HPLC results showed that there was no significant difference between the purified sample and the standard lignin. However, the UV result revealed that purified lignin gave slightly higher absorbance value than the standard lignin. The purified and unpurified samples of lignin comprised 0.345% and 5.75% ash respectively. From the GPC results, the poly-dispersity (M_w/M_n) values for the purified lignin and the standard lignin were 1.23 and 1.25, respectively. The lignin was then reacted with ferrous ion to form ferrum-tannin-lignin (FTL) complex at 105°C in the presence of formaldehyde to initiate cross-linking reaction. The rheological properties of the pure FTL (FTL_{purified}) was compared with the unpure FTL (FTL_{unpurified}) and a commercial thinner, lignosulfonate (LS). Generally, the FTL_{purified} gave comparable results with the FTL_{unpurified}, which suggests that purification process is not required to increase the performance of chelated ferrous ion drilling mud thinner.

Keywords: Oil palm empty fruit bunches, black liquor, purification, soda lignin, drilling mud thinner, rheological properties

Abstrak. Lignin soda diekstrak daripada likor hitam tandan sawit kosong (TSK) dengan menggunakan asid sulfurik 20% (v/v). Lignin yang diperoleh sebanyak 3.016 gram daripada setiap 200 mL likor hitam kemudiannya ditulenkan dengan *n*-pentana menggunakan peralatan soxhlet selama enam jam. Kajian perbandingan sifat fisiko-kimia dan perlakuan struktur antara lignin tulen dengan lignin piawai dilakukan menggunakan spektroskopi transformasi inframerah Fourier (FTIR), spektrometri ultra-ungu (UV), kromatografi ketelapan gel (GPC), kromatografi cecair berprestasi tinggi (HPLC), dan ujian abu. Keputusan ujian FTIR dan HPLC menunjukkan bahawa tiada perbezaan yang ketara antara sampel lignin yang ditulenkan dengan sampel lignin piawai. Walau bagaimanapun, keputusan ujian UV menunjukkan bahawa lignin yang ditulenkan memberi nilai keserapan yang lebih tinggi daripada lignin piawai. Peratusan ujian abu bagi lignin tulen ialah 0.345%, manakala lignin yang tidak ditulenkan mengandungi 5.75%. Daripada keputusan ujian GPC pula, nilai nisbah kebolehsbaran (M_w/M_n) bagi sampel lignin tulen dan lignin piawai masing-masing ialah 1.23 dan 1.25. Lignin tersebut seterusnya ditindakbalaskan dengan ion ferus untuk

^{1,2&3} School of Chemical Sciences, Universiti Sains Malaysia, 11800 Minden, Pulau Pinang, Malaysia
* Corresponding author: Tel: 04-6533554, Fax: 04-6574854, E-mail: mnm@usm.my

menghasilkan ferum-tanin-lignin (FTL) pada suhu 105°C dengan kehadiran formaldehid bagi memulakan tindak balas rangkai silang. Sifat-sifat reologi ferum-tanin-lignin tulen (FTL_{tulen}) dibandingkan dengan ferum-tanin-lignin yang tidak ditulenkan (FTL_{tidak tulen}) dan juga penipis komersial, lignosulfonat (LS). Secara keseluruhan, FTL_{tulen} menunjukkan prestasi yang setanding dengan FTL_{tidak tulen}, yang sekali gus membuktikan bahawa proses penulenan tidak diperlukan demi meningkatkan prestasi sesuatu penipis lumpur gerudi yang dikelatkan dengan ion ferus.

Kata kunci: Tandan sawit kosong, likor hitam, penulenan, lignin soda, penipis lumpur gerudi, sifat-sifat reologi

1.0 INTRODUCTION

The easiest and cheapest lignin resources can be found from oil palm biomass. Fibrous derivatives from palm oil industry's solid waste, especially the oil palm fiber from empty fruit bunch (EFB), present a renewable source of non-wood lignocellulosic materials. Malaysia has over 2.5 million hectares of oil palm plantations that yield more than 500 million tones of fresh fruit bunches annually. There are over 300 crude palm oil mills processing the fresh fruit bunches into crude palm oil and generate more than 8 million tones of solid waste in the form of empty fruit bunches (EFB) annually [1].

Soda lignin extracted from oil palm lignocellulosic waste especially the EFB has been successfully transformed into drilling mud thinners, i.e. ferum-tannin-lignosulfonate (FTLS) and ferum-tannin-lignin (FTL) which had been discussed elsewhere [2]. The rheological performances of these thinners, especially the FTL are comparable with the commercial thinner available in the market. In the drilling operations, FTL can be used as a thinner and fluid reducing agent in water-based mud system.

The search for the optimum performance of the FTL was carried out in this study. A drilling mud thinner, i.e. ferum-tannin-lignin (FTL), was formulated from two types of lignin, namely the purified lignin (FTL_{purified}) and the unpurified lignin (FTL_{unpurified}). The objective of this investigation is to determine whether the purification process of the lignin in the formulation of FTL is needed to increase the performance of the thinner.

2.0 EXPERIMENTAL

2.1 Material

The oil palm empty fruit bunch (EFB) long fibers material used in this study was supplied by Sabutek (M) Sdn. Bhd., a local company specializing in recycling the oil palm lignocellulosic wastes.

2.2 Extraction Process

The EFB fibers were first soaked into water for two days to remove dirt before the pulping process. Then the fibers were pulped in a 20 L stainless steel rotary digester unit with 25% (w/w) NaOH (cooking liquor) for three hours at the maximum cooking temperature of 170°C. The mixture of cooking liquor to the EFB fiber was in the ratio of 10:1.

In this pulping process, the pH of black liquor was measured as 12.45 and its density was 1.02 g/mL. The soda lignin was then precipitated from the concentrated black liquor by acidifying it to pH 2 using 20% (v/v) sulfuric acid. The precipitated lignin was filtered and washed with pH 2 water, which was prepared using the same acid in the earlier step. The soda lignin was then dried in vacuum oven at 55°C for 24 hours prior to further analysis [3].

2.3 Purification

After being dried in a vacuum oven, the soda lignin was then extracted with *n*-pentane in soxhlet apparatus for six hours to remove lipophilic non-lignin matters such as wax and lipids [4]. The precipitated lignin was filtered and washed with pH 2 water to remove the excess *n*-pentane.

2.4 Analysis of Lignin

The standard lignin sample used in this study was supplied by Aldrich (CAT No. 371076-25G). The IR spectra was recorded on Perkin-Elmer 2000 spectrophotometer. The KBr pellets were prepared containing 1% fine ground sample. For the UV analysis, a Hitachi spectrophotometer model U-2000 was used to obtain the results. Prior to the analysis, 5 mg sample was dissolved into 10 mL of 90% (v/v) dioxane-water (aliquot). 1 mL of aliquot was further diluted into 25 mL by using 50% (v/v) dioxane-water [5]. The sample was then measured for its absorbance, which should fall in the range of 200 to 350 nm [4, 6].

Nitrobenzene oxidation was carried out by adding 50 mg soda lignin into mixture of 7 mL of 2 M NaOH and 4 mL of nitrobenzene in a 15 mL steel autoclave. The autoclave was sealed tightly with a screw up fitted with Teflon gasket and heated to 165°C for three hours in a preheated thermostat silicon oil bath. After the heating period, the autoclave was cooled with ice water. The mixture was then transferred to a liquid-liquid extractor for continuous extraction with chloroform (5 × 20 mL) to remove any nitrobenzene reduction product and excess of nitrobenzene. The oxidation mixture was acidified by concentrated HCl to pH 3-4 and further extracted with chloroform (5 × 15 mL). The solvent from the second chloroform solution was removed using a rotary evaporator at 40°C under reduced pressure to obtain the nitrobenzene oxidation mixture. The mixture was then dissolved into dichloro-

methane and made it up to 10 mL. This mixture was used as a stock solution for further analysis of the oxidation mixture [3, 4].

High performance liquid chromatography (HPLC) was used to analyze the nitrobenzene oxidation mixture. 0.25 mL of stock solution was pipetted into 25 mL volumetric flask and made it up with acetonitrile-water (1:2 v/v). The resulting sample solution was filtered through a Millipore membrane (pore size 0.45 μ) to remove high-molecular weight contaminant and the 3,4,5-trimethoxybenzaldehyde was used as an internal standard. About 20 μ L of the filtrate was injected into HPLC system (Shimadzu model LC-10AD VP) equipped with Hypersil bond C₁₈ column (particle size 5 μ , 25 mm \times 4.6 mm i.d.) to determine qualitatively the oxidation products. A mixture of acetonitrile-water (1:8 v/v) containing 1% (v/v) acetic acid was used as an eluent with a flow rate of 2 mL/min. The eluent was monitored with UV (ultra-violet) detector at 280 nm [3, 4].

The molecular weight distribution of the lignin was determined using gel permeation chromatography (GPC) on a PLgel 5 μ Mixed-D column. Monodisperse polystyrene was used as the standard. 100 μ L of the sample with the concentration of 1 mg/mL was injected into the GPC. The temperature and flow rate of the column were set at 40°C and 1 mL/min respectively [3].

The ash test was done by heating the soda lignin sample in the electrical heat-Carbolite Furnaces CSF 1100 at 900°C for four hours. After heating, the sample weight was measured and the percentage of ash was calculated. If the percentage of the sample ash was less than 2%, it indicates that the lignin sample has high degree of purity [4].

2.5 Formulation of Water-based Drilling Mud Thinners (FTL)

Lignosulfonate (LS), Ressimex II, and bentonite clay used in this study were supplied by Kota Mineral (Malaysia) Sdn. Bhd. The rest of the chemicals including sulphuric acid, formaldehyde, ferrous (II) sulphate, sodium hydroxide, sodium sulphite, and dried tannin were purchased from local chemical agents and the chemicals used were of analytical grade.

Each batch of FTL sample was prepared by dissolving 10 g of lignin (either purified or unpurified) into 200 mL of hydrochloric acid (1 M). The mixture, which was a clear brown solution, needed to be stirred continuously while the pH of the mixture was adjusted to 4 by adding sodium hydroxide. To the mixture, 3.33 g of tannin was added in the presence of 4 mL of formaldehyde 37% at 105°C for two hours. After tannin-lignin (TL) complex was obtained, the mixture was further chelated with the required amount of ferrous (II) sulphate (4.17 g) for 30 minutes at the same temperature. The reacted mixture was then allowed to air dry before further drying under vacuum at 65°C. The prepared FTL was a fine dark brown powder.

2.6 Mud Properties Tests

Three sets of experiments were performed in this study. The mud property tests were performed according to the American Petroleum Institute (API) specifications [7]. Each batch of base mud used in each experiment was prepared by adding 80 g bentonite and 4 g of sodium carbonate into 1000 mL water before aging it for more than 24 hours [7].

The base mud samples were added separately with one of these three additives: LS (commercial thinner), FTL_{purified} , and $FTL_{\text{unpurified}}$. For each batch of sample, a Fann Rheometer Model 286 viscometer was used to measure the viscosity at two rotating rates of 300 rpm ($\theta_{,300}$) and 600 rpm ($\theta_{,600}$). Then apparent viscosity (μ_a), plastic viscosity (μ_p), and yield point (τ_y) were calculated as follows [8]:

$$\mu_a = \theta_{600} / 2 \quad (1)$$

$$\mu_p = \theta_{600} - \theta_{300} \quad (2)$$

$$\tau_y = 0.511(\theta_{300} - \mu_p) \quad (3)$$

where μ_a and μ_p are in unit mPa.s, and τ_y in unit of Pa.

Experiment 1 was designed to measure the basic mud rheological parameters such as μ_a , μ_p , and τ_y at different pH values. Sodium hydroxide (NaOH) solution was added into the mud system to adjust their pH values.

Experiment 2 was designed to evaluate the performance of the additives against different levels of salt contamination before and after the mud sample was heated using a Fann Roller Oven Series 2500 at 160°C for 16 hours. The rolling oven was used to simulate the actual drilling process. Two levels of salt contamination were tested in these experiments, i.e. 0.7% and 1.2%. These levels were chosen based on the previous studies [2, 9].

Finally, Experiment 3 was specially designed to evaluate the performance of the additives as a fluid reducer. In this experiment, Ressinex II (commercial fluid reducer) was used as a comparison. A Fann Filter Press model Series 300 was used to measure the API filtrate volume. Filtered volume is the amount of fluid that can be delivered through a permeable filter medium after being subjected to a 100 psi of pressure for 30 minutes.

3.0 RESULTS AND DISCUSSION

3.1 Analysis of Soda Lignin

Lignin contains three monomeric lignin units, vic p-hydroxyphenyl (H), guaicyl (V), and syringyl (S) which are capable to produce the corresponding degradation product of p-hydroxybenzaldehyde, vanillin, and syringaldehyde [3, 4]. Figures 1 and 2 depict

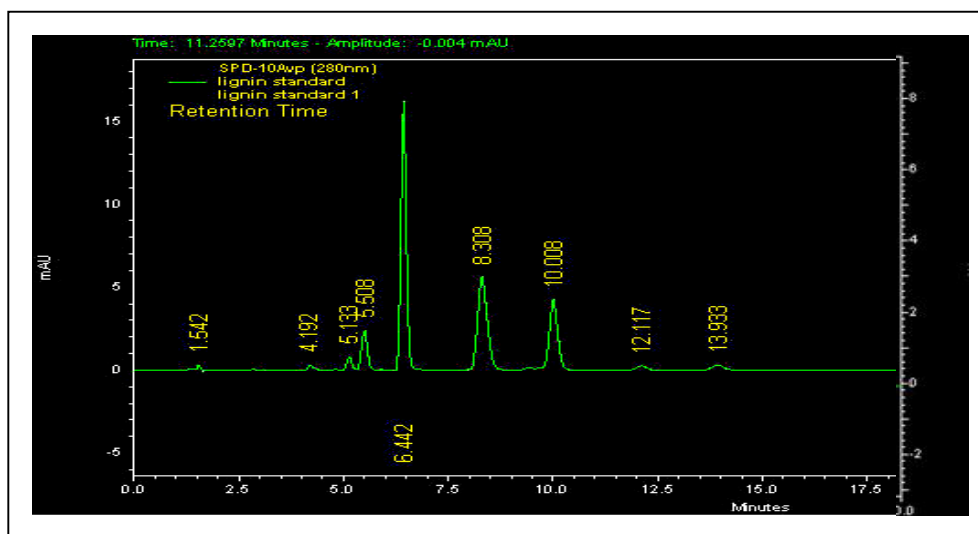


Figure 1 HPLC chromatogram for standard lignin

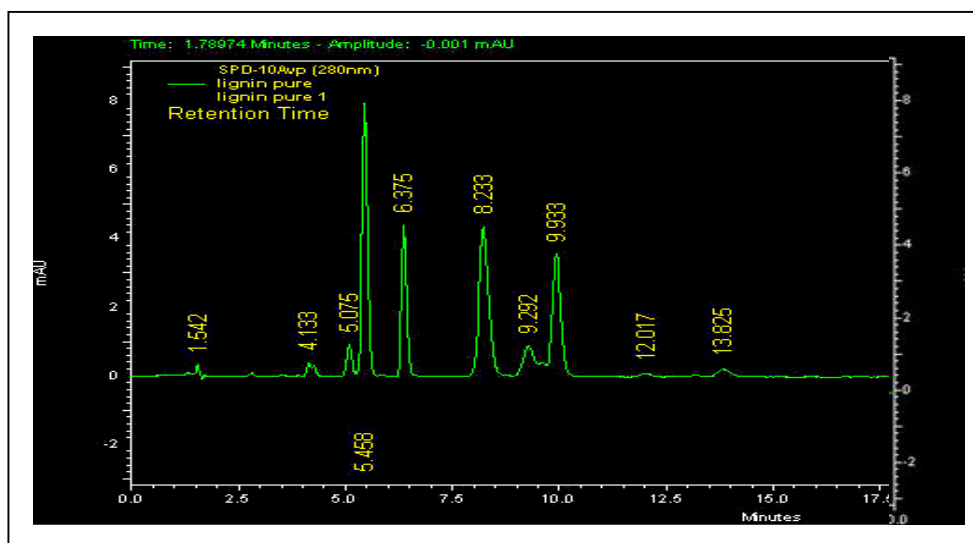


Figure 2 HPLC chromatogram for purified lignin sample

the HPLC chromatogram of the standard lignin and the purified lignin samples, respectively. The chromatograms of these lignin samples give similar peaks, however the intensity of each peak is not the same. For example, the standard lignin gives the highest peak at RT (retention time) 6.442 minute, whereas the highest peak for purified lignin happened at RT 5.458 minute. From this analysis, we can conclude that both lignin samples contained the same number of monomers, however, the concentration of each monomer was not the same. This is expected due to the

source of lignin analyzed in this study was not the same, i.e. from oil palm empty fruit bunch (purified lignin sample) and the other from the wood base lignin (standard lignin sample).

The purified soda lignin and the standard lignin sample also showed almost the same IR spectrum with each other, as shown in Figure 3. Both lignin samples indicate the presence of major peaks. The strong and broad band at 3411 cm^{-1} is the characteristic of OH group or phenolic compound. The band at $1329\text{-}1327\text{ cm}^{-1}$ is due to the bending of vibration in phenolic OH group, whereas the band at 1032 cm^{-1} is characteristic of a primary alcohol. The clear shoulder at $2940\text{-}2930\text{ cm}^{-1}$ and the band at 1464 cm^{-1} are assigned to CH stretching of methyl or methylene group. Broad medium band at 1709 cm^{-1} is due to conjugated carbonyl stretching. Moreover, 1515 cm^{-1} and 1603 cm^{-1} are characteristic of aromatic rings due to the aromatic skeletal vibrations and the band at 1115 cm^{-1} is due to the ether stretching. A band at $830\text{-}840\text{ cm}^{-1}$ indicates the C-H deformation and ring vibration [3].

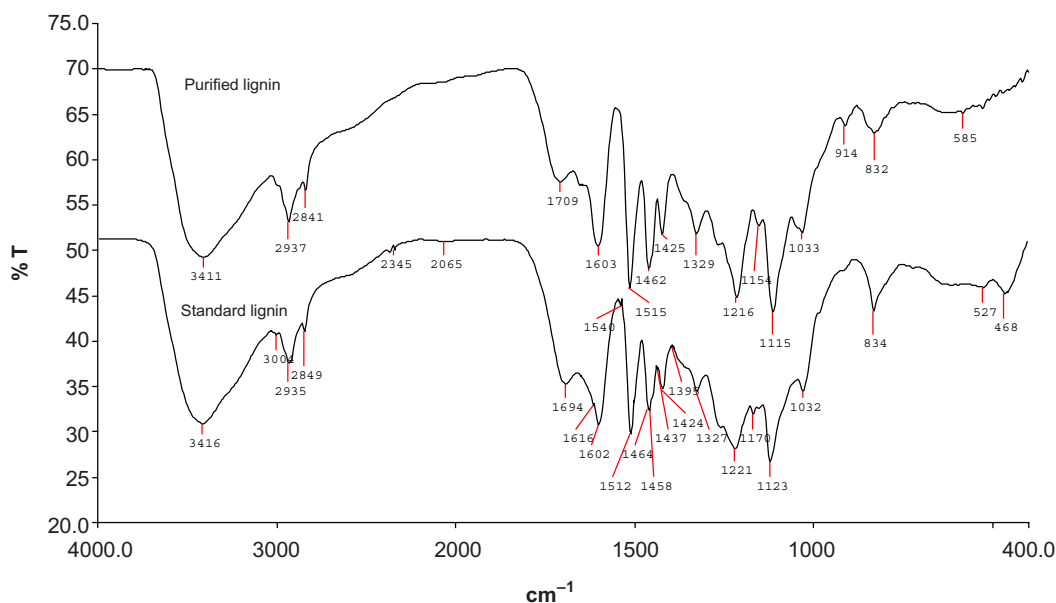


Figure 3 IR spectra for purified lignin and standard lignin samples

The observation of UV spectroscopy at $210\text{-}350\text{ nm}$ can also be used to verify the purity of the purified lignin sample. Figure 4 shows the UV spectra of two lignin samples. As shown in the spectra, the purified lignin and the unpurified lignin exhibit the same UV spectrum for the typical lignin, which have a maximum at $220\text{-}230\text{ nm}$ and a second maximum at $270\text{-}280\text{ nm}$ originating from non-condensed phenolic

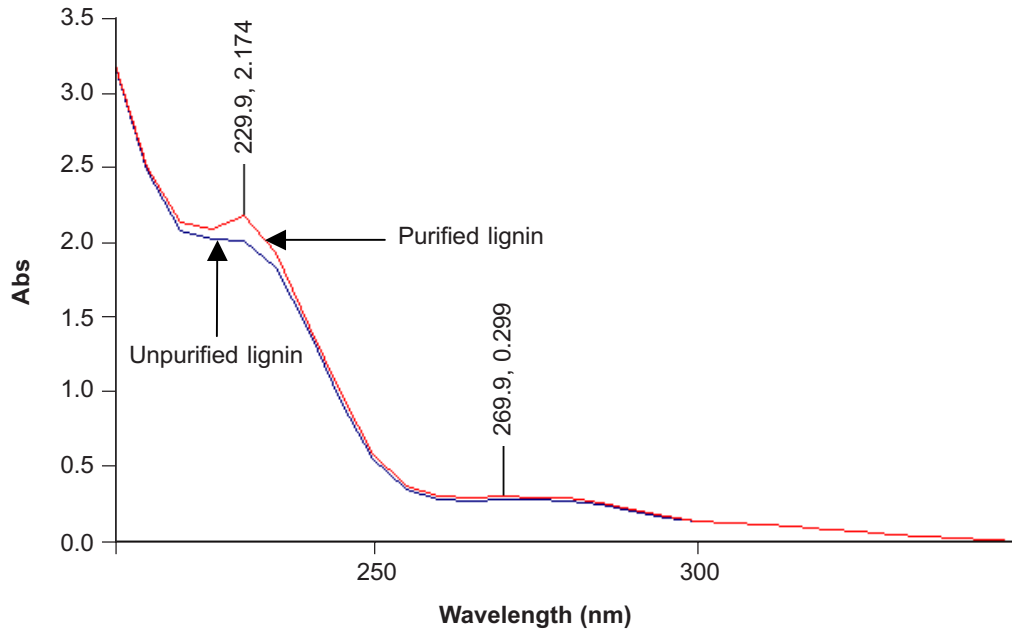


Figure 4 UV spectroscopy for purified lignin sample compared to standard lignin sample

groups (aromatic ring) in lignin [10]. The spectra also showed that the intensity of the absorbance is related to the level of lignin concentration. The absorbance of UV spectra is directly proportional to the purity level of lignin. A lower absorbance is due to the co-precipitated of non-lignin material such as polysaccharides degradation product, wax, and lipids [6]. From the GPC results, the poly dispersity (M_w/M_n) value for the purified lignin sample and standard lignin were 1.23 and 1.25 respectively. The ash test also confirmed that the purified lignin sample had a higher degree of purity where the ash contain was found to be only 0.345%, which was much lower than the unpurified sample, i.e 5.75%.

These results (IR, HPLC, UV, GPC, and ash contain test) suggested that the purification process was successfully accomplished.

3.1.1 Analysis of Mud Tests

While drilling, pH lower than 7.0 (pH of pure water) should be avoided because it will accelerate corrosion of pipeline and equipment. A pH of greater than 7.0 should be maintained throughout the drilling process because in alkaline condition, chemical thinners are more effective, corrosion is reduced, and gel strength and yield point are slightly reduced [11]. This is why acidic condition was not considered in this experiment (Experiment 1). Table 1 shows the rheological performances of FTL_{purified} and $FTL_{\text{unpurified}}$ against LS as drilling mud thinner at various pH values. Results for

Table 1 Basic mud rheological properties at various pH values for four different types of mud

Formulation of mud	pH	μ_a (mPa.S)	μ_p (mPa.S)	τ_y (Pa)
Based mud (Control sample)	8.12	27.50	8.50	18.91
Base mud + 0.3% LS [2]	9.10	22.50	14.30	8.40
	10.06	16.80	9.70	7.30
	11.13	15.10	10.00	5.30
	12.09	18.80	12.50	3.40
Base mud + 0.3% FTL _{unpurified} [2]	9.35	13.00	5.70	7.50
	10.10	21.50	13.30	8.40
	11.00	20.00	13.00	7.20
	12.08	15.70	9.70	6.10
Base mud + 0.3% FTL _{purified}	9.07	18.50	10.50	8.18
	10.02	18.25	10.00	8.43
	11.06	16.50	8.17	8.51
	12.23	13.75	7.50	6.39

All data were measured at $27 \pm 1^\circ\text{C}$

mud treated with LS and FTL_{unpurified} were obtained from previous study [2]. The experiment is important especially when drilling process advances to different types of formation where different pH values will be recorded.

Generally, the thinning abilities of FTL_{purified}, FTL_{unpurified} and LS vary with the pH of mud, as shown in Table 1. In pH range of 9 to 12, the mud treated with FTL_{purified}, FTL or LS has lower τ_y value. These results are in line with the general rule of mud preparation where if there is no unusual hole problem, the mud should be kept as thin as possible [11]. As seen in Table 1, there is no significant difference in term of τ_y value between the results for the mud treated with the FTL_{purified} and FTL_{unpurified}. Both of them gave better thinning abilities compared to LS. It is a common practice in industries that a mud engineer observe the trend of τ_y rather than the trends of μ_a and μ_p .

Generally, the results of Experiment 2 suggest that the iron complex drilling mud thinners (FTL_{purified} and FTL_{unpurified}) performed well even in the presence of salt contamination both at room temperature as well as at high temperature (160°C) for 16 hours drilling operation. This experiment is important because salt (NaCl) is the common contaminant encountered during drilling process, which is usually found in make-up water. When make-up water is very salty, bentonite neither hydrates nor disperses [12]. Bentonite particles tend to flocculate which will result in an increase in yield point and apparent viscosity values. FTL_{unpurified} also showed similar trend with FTL_{purified}. The results of this experiment also suggested that FTL_{purified} and FTL_{unpurified} could stand higher drilling temperature (160°C) compared to LS which

Table 2 Basic mud rheological properties at two different salinity levels (before and after rolling at 160°C for 16 hours)

Formulation of mud	μ_a (mPa.S)		μ_p (mPa.S)		τ_y (Pa)	
	Before	After	Before	After	Before	After
Based mud + 0.7% NaCl (Control sample)	26.08	69.00	8.17	18.00	18.31	52.12
Based mud + 0.7% NaCl + 0.3% LS	21.50	50.00	14.67	15.00	6.98	35.77
Based mud + 0.7% NaCl + 0.3% FTL _{unpurified}	21.83	45.50	11.55	17.00	10.51	29.13
Based mud + 0.7% NaCl + 0.3% FTL _{purified}	20.25	42.70	10.00	14.90	10.47	28.46
Based mud + 1.2% NaCl (Control sample)	32.80	77.20	8.70	24.70	30.80	53.70
Based mud + 1.2% NaCl + 0.3% LS	16.50	58.50	12.00	13.00	4.60	46.50
Based mud + 1.2% NaCl + 0.3% FTL _{unpurified}	10.20	49.20	12.70	10.00	8.00	24.70
Based mud + 1.2% NaCl + 0.3% FTL _{purified}	18.00	51.75	7.50	7.75	10.73	24.97

All data were measured at 27 ± 1°C

represent drilling at about 7800 m underground (1°C per 59 m depth). More complete results are given in Table 2.

The main objective of Experiment 3 is to determine whether the lignin-based drilling mud thinners (FTL_{purified} and FTL_{unpurified}) could also act as good fluid reducers. The fluid loss control is important to make sure that drilling mud will not lose to the formation. This is important to reduce the chance of contamination of drilling fluid to the formation as well as drinking water zone. Besides, it is also important to save the unnecessary cost due to losing of drilling fluid from the system. Results in Table 3 show that FTL_{purified} and FTL_{unpurified} gave smaller amount of filtrate volume compared to Ressinex II before and after rolling. The lesser the filtrate volume obtained, the better the additive acts as a fluid loss reducer. In other words, the mud sample with small filtrate volume indicated that the mud was capable to hold fluid from losing to the formation. The results of the experiment were to confirm that the formulated thinner, especially FTL_{unpurified} is not only functioning as a good drilling mud thinner but also at the same time has a potential to act as a good fluid reducer.

Table 3 API filtrate volume (before and after rolling at 160°C for 16 hours)

Formulation of mud	API filtrate loss (mL)	
	Before	After
Based mud (Control sample)	16.40	42.50
Based mud + 0.3% Ressinex II	16.10	39.40
Based mud + 0.3% FTL _{unpurified}	14.80	33.70
Based mud + 0.3% FTL _{purified}	15.10	35.00

All data were measured at 27 ± 1°C

From those three experiments, $FTL_{unpurified}$ gave comparable result as $FTL_{purified}$ which suggests that the purification process was not necessary to increase the performance of chelated ferrous ion drilling mud thinner.

4.0 CONCLUSIONS

In this study, soda lignin was successfully purified by *n*-pentane using soxhlet apparatus for six hours. The purity of the sample was verified using a Fourier transform infrared spectroscopy (FTIR), ultraviolet spectrometry (UV), gel permeation chromatography (GPC), high performance liquid chromatography (HPLC), and ash contain test. The purified lignin sample was then allowed to react with tannin and ferrous ion to form Fe-tannin-lignin ($FTL_{purified}$). This newly formulated thinner was tested against $FTL_{unpurified}$ to determine whether the purification of lignin was necessary to improve the performance of FTL. Results suggested that the purification process was not necessary to increase the performance of rheological mud test.

ACKNOWLEDGEMENTS

The authors would like to express their appreciations to Universiti Sains Malaysia and the Malaysian Ministry of Science, Technology and Innovation for the financial support of this project through a research grant (305/PKIMIA/612921). The authors would also like to thank Sabutek (M) Sdn. Bhd. for supplying EFB long fiber, and Kota Minerals and Chemicals Sdn. Bhd. for supplying bentonite and several mud additives for this study.

REFERENCES

- [1] Sabutek (M) Sdn. Bhd. <http://www.sabutek.com.my> (accessed on 24/04/04)
- [2] Mohamad Ibrahim, M. N., M. N. Nazlina, A. D. Fauzana, and S. B. Chuah. 2003. Recycling Black Liquor of Oil Palm EFB into a Drilling Mud Thinner. *Journal of Physical Science*. 24: 529-539
- [3] Mohamad Ibrahim, M. N., S. B. Chuah, and W. D. Wan Rosli. 2004. Characterization of Lignin Precipitated from the Soda Black Liquor of Oil Palm Empty Fruit Bunch Fibers by Various Mineral Acids. *ASEAN Journal on Science and Technology for Development*. 21(1): 57-67.
- [4] Lin, S. Y., and C. W. Dense. 1992. *Method in Lignin Chemistry*. Berlin Heidelberg: Springer-Verlag.
- [5] Hudgins, Jr. C. M. 1992. Chemical Treatment and Usage in Offshore Oil and Gas Production Systems. *Journal of Petroleum Technology*. 604-611.
- [6] RunCang, S., and J. Tomkinson. 2001. Fractional Separation and Physico-chemical Analysis of Lignin from the Black Liquor of Oil Palm Trunk Fibre Pulping. *Separation and Purification Technology*. 24: 529-539.
- [7] American Petroleum Institute. 1986. *Recommended Practice Standard Procedure for Field Testing Water-Based Mud Drilling Fluids*. API Recommended Practice 131-1. 2nd Ed. South Australia: Standard & Technical Publications. 1-5.
- [8] Bourgoyne, Jr., A. T., K. K. Millheim, M. E. Chenevert, and F. S. Young, Jr. 1991. *Applied Drilling Engineering*. Richardson, Texas: Society of Petroleum Engineers Text Book Series. 42-74.
- [9] Mohamad Ibrahim, M. N., S. B. Chuah, and P. Y. Cheng. 2003. Tin-tannin-lignosulfonate Complex: An Improved Lignosulfonate-based Drilling Fluid Thinner. *Jurnal Teknologi*. 38(F): 25-32.

- [10] RunCang, S., J. Tomkinson, and G. L. Jones. 2000. Fractional Characterization of Ash-AQ Lignin by Successive Extraction with Organic Solvents from Oil Palm EFB Fiber. *Polymer Degradation and Stability*. 68: 111-119.
- [11] Gary, G., H. Darley, and H. Rogers. 1980. *Composition and Properties of Oil Well Drilling Fluids*. 4th Ed. Houston, Texas: Gulf Publishing Co. 564-575.
- [12] Roland, M., A. Reimann, and K. P. Krinstad. 1988. Fractionation of Kraft Lignin by Successive Extraction with Organic Solvents. *Holzfoorschung*. 42: 111-116.