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PHOSPHORYLATION AND CHARACTERIZATION OF LIGNIN AND ITS APPLICATION ON MILITARY UNIFORM AS A FLAME RETARDANT

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ABSTRACT

The use of coconut fiber an agricultural waste was considered as an environmentally friendly and affordable alternative in the formulation of an environmentally friendly bio-based phosphorus containing flame retardant to be used on military uniforms in Nigerian defence system to mitigate the impact of fire hazards for soldiers during high-risk situations. This was achieved by extraction and modification of lignin from coconut husks using phosphoric acids. Strips of fabrics were treated with 30% aqueous solutions of virgin lignin (V-Lig) and phosphoric acid modified lignin (PM-Lig). The structures of the virgin lignin extracted(V-Lig) and the phosphoric acid modified lignin (PM-Lig) were characterized by Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM). The characterizations showed that phosphate groups were covalently bonded to the lignin structure indicating the success of the lignin modifications. Strips of fabrics were treated with 30 % aqueous solutions of the virgin lignin (V-Lig) and the phosphoric acid modified lignin (PM-Lig). The thermo-gravimetric analysis in nitrogen atmosphere performed at 950 °C with heating rate of 10 °C/min. For the flame retardant treated fabrics showed slow rate of weight loss with high char residue and high ultimate degradation temperatures (57 % weight loss with 42% char for V-Lig and 65 % weight loss with 50 % char for PM-Lig) against the untreated fabric sample which degraded completely with no char residue (100 % weight loss with 0 % char) indicating high thermal stability for the treated fabrics. Flammability characteristics of the treated fabric samples investigated by UL-90 horizontal burning test showed very high flame resistance (109.0 mms^{-1} burning rate with 94.071 % weight loss for V-Lig and 45 mms^{-1} burning rate with 66.4 % weight loss for the PM-Lig) when compared to the untreated fabric sample (127.2 mms^{-1} burning rate and 94.662 % weight loss) confirming the PM-Lig to be a good flame retardant for fabrics with an ease of application.

Key words: *Coconut husks, Extraction, modification, characterization, flame-retardant, fabrics.*

1. INTRODUCTION

Lignin is a complex organic polymer found in the cell walls of many plants, particularly in wood and bark. It is one of the most abundant organic polymers on earth, second to cellulose. It is composed of various phenolic compounds with a highly irregular and complex structure, consisting of cross-linked phenolic monomers such as coniferyl, sinapyl, p-coumaryl alcohols. Lignin provides structural, rigidity and strength to plant cell walls. It also plays an important role in water transport within the plant by reinforcing the vascular tissues, thereby preventing collapse of the plant under tension. Lignin as complex organic polymer has gained attention as a potential flame retardant due to its unique properties. It has also been employed as an alternative to antioxidants and surfactants. Lignin exhibits high thermal stability which is of great importance for flame retardants. It has the ability to form char when exposed to high temperatures, which acts a barrier that slows down the transfer of heat and reduces the release of flammables volatiles. During combustion, lignin decomposes to form a stable char layer that insulates underlying material from heat and oxygen thereby inhibiting the combustion process. Lignin can be used in combination with other substances or flame retardants to enhance their effectiveness. Unlike the synthetic flame retardants, lignin is biodegradable and does not produce toxic products during combustion. This makes it a more environmentally alternative for flame retardants. The phosphorylation of lignin increases its thermal behavior and produces effective and environmentally flame retardants. phosphorous-containing flame retardants are widely used as an alternative to halogenated flame retardants, and can suppress fire in a polymer in two different ways (Beata *et al.*,2020**).** In this work, a different phosphorylation route was used to modify lignin. coconut husk, an agricultural waste with rich lignin content was used for lignin extraction. A novel flame retardant was formulated and characterized. The bio-based flame retardant is economically and environmentally friendly and serves as a better alternative to halogen-based flame retardants which are known for their environmental hazards.

2. MATERIALS AND METHODS

2.1.Reagents

Dried coconut husks, Hydrogen peroxide (50%, 1.200g/cm^3), Sodium Hydroxide (98%, 2.13g/cm^3), Copper Oxide (99%, 6.31g/cm^3), Phosphoric acid(H_3PO_4=85%,1.88g/cm^3), Methanol (99.9%, 0.792g/cm^3), Dimethyl Sulphoxide ($DMSO_4$ =99.5%, 1.1gcm^3)
Iron (ii) sulphate ($Fe_2 (SO_4)_3$=98%, 1.89g/cm^3), Sulphuric acid (H_2SO_4 =98%, 1.83g/cm^3), Urea (CH_4N_2O= 98.5%, 1.32 g/cm^3), Water.

2.2. Sample Collection

Natural biomass resources (coconut husk) was collected from station market located at Nigerian railway co-operation Kaduna junction, Kaduna south local government area of Kaduna state and transported to the laboratory in an enclosed container for proper storage

2.3. Extraction of Lignin

Dried coconut husks were washed, dried, cut into parts of approx. 5 cm, crushed in to powder using a hammer mill and sieved to particles sizes of 1.25 cm using a standard test sieve. An aqueous 2 % by wt of NaOH solution was prepared. The mixture of dry husk (5 g) and NaOH (115 cm^3) was allowed to digest in a digester for 2 hrs at 160 ^{o}C to carry out extraction as reported by Pandharipande *et al.,* (2018). The solution obtained was filtered and the filtrate was acidified to pH 1 using Concentrated H_2SO_4. The acidified mixture was heated to 100 ^{o}C and then followed by dilution with water to a volume ratio of 1:5, and allowed to precipitate overnight. The solution was filtered using whatman filter paper of size 46×57 cm and the solid mass aggregate was oven dried at 105 ^{o}C. The product obtained was analysed for the presence of lignin using FTIR (Pandharipande *et al.,* 2018).

2.4. Chemical modification of lignin using phosphoric acid

Exactly 2.00 g of lignin and 16.8 cm^3 phosphoric acid was mixed and ŵpreheated at 80 ^{0}C for 15 min in a 200 cm^3 round bottomed flask as reported by Rungrodnimitchai *et al.,* (2014). It was then heated on an oil bath at 150 °C for 2 hrs. After cooling to room temperature, the mixture was washed with tap water to neutral pH, rinsed with acetone, and dried at 80 °C. The equation of the reaction is presented in equation 2.1.

$$\text{Lignin-OH} + H_3PO_4 \xrightarrow[150^{o}C,\ 2hr]{\triangle} \text{Lignin-O-P-}(OH)_2 + H2O \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.1$$

2.5. Catalytic oxidation of the phosphoric acid modified lignin (PM-Lig)

Exactly 0.3 g of PM-lignin in 50 % aqueous solution of methanol (20 cm^3) followed by 0.08 g of CuO, 0.004 g of $Fe_2(SO4)_3$, 2.4 g of NaOH and 2 cm^3 of 30 % H_2O_2 were placed in a 100 cm^3 of stainless steel autoclave. The catalytic reaction was carried out at 150 °C for 60 min with a stirring speed of 400 rpm as reported by Xinping *et al.,* (2015). The autoclave was cooled down to room temperature with cooling water. The depolymerized products were acidified with sulphuric acid (Xinping *et al.,* 2015).

2.6. Fabric Treatment

Military uniform fabrics of length 100 mm and 30 mm width with 0.4 mm thickness were prepared and treated with 30 % aqueous solutions of the virgin lignin(V-Lig) and phosphoric acid modified lignin (PM-lig) flame retardant respectively. The samples were immersed in the aqueous solutions for 24 hours for thorough wetting and dried in the air.

2.7. Evaluation of the flame retardancy of the treated military uniform fabrics

The thermal stability and combustibility study of the treated and untreated cotton fabrics was characterized in terms of thermo-gravimetric analysis (TGA) and UL-90 Horizontal burning test

2.8. Thermo-gravimetric analysis (TGA)

The thermo-gravimetric analysis was performed with a Perkin Elmer Pyros-Thermo-gravimetric analyser instrument. Fabric samples of weights 14.309 mg were heated from 30 °C to 950 °C at a heating rate of 10 °C/min under nitrogen atmosphere and the thermal stability and decomposition of the treated and untreated fabric samples were determined.

2.9. UL-90 Horizontal burning test

The ignitability and flammability of the treated and untreated fabric samples were determined using the UL-90 horizontal burning test which evaluated the flammability of the materials in ambient temperature. The flammability tests were carried out on a horizontal burning test with a burning time of 30 seconds. The ignition time, extents of burning and weight loss were determined. A visual observation in terms of drippings as well as the rate of burning was also calculated using the formula Rb= Lb/Tb and the results were summarized in Table 3.0 and Figure 5.0.

Note: Rb= rate of burning, Lb= length of burning and Tb= time of burning.

3. Results and Discussion

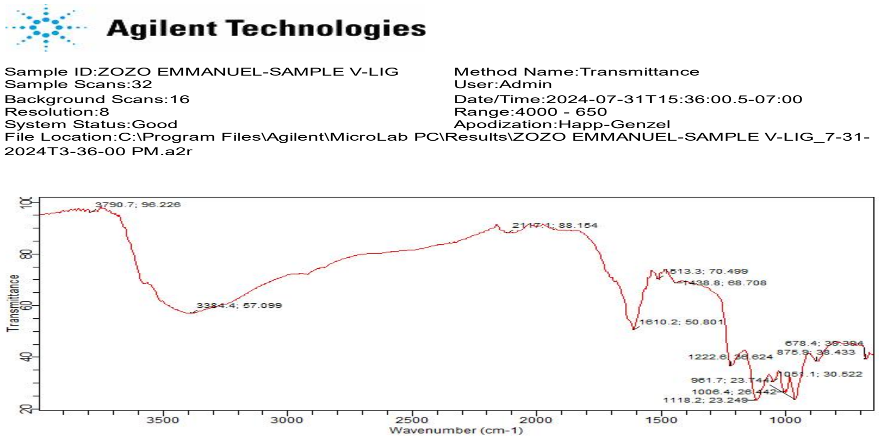
Characterization of the V-lig, and PM-lig

Fourier Transform-infrared (FT-IR) Spectroscopy

The virgin lignin extracted was first characterized by Fourier transform infrared spectroscopy (FT-IR) followed by the structure of the phosphoric acid modified lignin to ensure that the modification was successfully achieved. The virgin lignin (V-Lig) spectrum exhibited classical patterns as described in different literatures such as Prieur *et al.,* (2017), Pandharipande *et al.,* (2018) and Dereca *et al.,*(2014). An absorption band centered at $3384.4cm^{-1}$ was observed and attributed to hydroxyl groups from both the aliphatic and aromatic chains of lignin. The C-H vibration of CH_2 was detected at 2117.1 cm^{-1}. Many absorption bands were observed below 2000 cm^{-1} and were presented in Figure 1A. very weak and low-intensity peaks in the fingerprint region of 900 to 650 cm^{-1}, are responsible for the poly-substituted phenolic structure of lignin. The many absorption bands observed in the range of 2000 to 650 cm^{-1} reveal the complexity of the lignin structure.

The phosphorylation process on the other hand did not degrade the lignin structure, as a broad absorption band centered at 3365.8 cm^{-1} was observed and attributed to the –OH stretching vibration from the aromatic and aliphatic moieties of lignin. The appearance of a peak at 2105.9 cm^{-1} was attributed to stretching vibrations of the $–CH_2$ group from the aliphatic chains. a major change occurred at the large and intense band centered at 1095.8 cm^{-1} and was attributed to the absorption of the phosphate P-O-C ester linkages present in the phosphate groups. Stretching of the P-O bond was also detected at 1364.2 cm-^{1} in the PM-Lig spectrum. The phosphates groups observed in PM-Lig may be bonded to the lignin's structure with ester linkages. See Figure 1B.

A



Agilent Technologies

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Background Scans:16
Resolution:8
System Status:Good
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User:Admin
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B

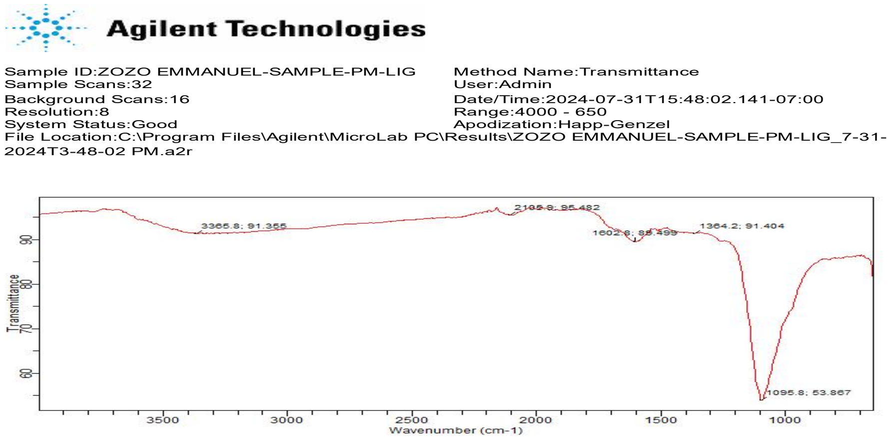

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Figure 1 FT-IR spectrums of the V-Lig (A) and PM-Lig (B)

It can be seen from Fig 1A and B that peaks appearing in the range of 1600–1400 cm^{-1} correspond to the aromatic C-C ring vibrations in phenyl-propane structures. Both the unmodified and modified lignin have shown a broad absorption band in the range of 3384–3365 cm^{-1} , which can be assigned to the –OH stretching vibration, due to the presence of either alcohol, hydroxyl or phenolic hydroxyl groups in the aromatic and aliphatic moieties of lignin. The modification process may have affected the extent of hydrogen bonding in the PM-Lig hence the –OH absorption peak has been shifted from 3384.4cm-^{1} in the V-Lig to 3365.8cm-^{1} in the PM-Lig spectrum. Appearance of peaks in the range of 2909–2842 cm^{-1} can be attributed to aliphatic C-H stretching vibrations of the-CH_2-and-CH_3 groups of the phenyl-propane side chains in both the unmodified and the modified lignin (V-Lig and PM-Lig).

Absorption bands below 2000 cm^{-1} (1500-600) cm^{-1} are attributed to the existence of both the conjugated and unconjugated carbonyl ester or acetyl groups in the unmodified and the modified lignin (V-Lig and PM-Lig) respectively.

Morphology of the V-Lig and PM-Lig

The morphology and structural characteristics of the V-lig, and PM-Lig particles were analyzed by scanning electron microscopy (SEM) in 500×537 μm magnification. The SEM images shown in Figure 2A and B Compare V-Lig and PM-Lig particles. After modification of the virgin lignin, physical properties such as color and particle size were observed. Lignin appeared to be light camel in color with a smooth surface. After modification, it was observed that PM-Lig, became darker with an increased particle size much bigger than those of the virgin lignin. The V-Lig particles are observed to be small spherical particles which clew together with a smooth surface. The PM-Lig particles on the other hand are not clewed together as in the case of the V-Lig but rather look as a chunk with many holes all over, exhibiting a porous surface. These changes in morphology and structures of the PM-Lig when compared to the V-Lig showed that indeed modification was successfully carried out.

A

B

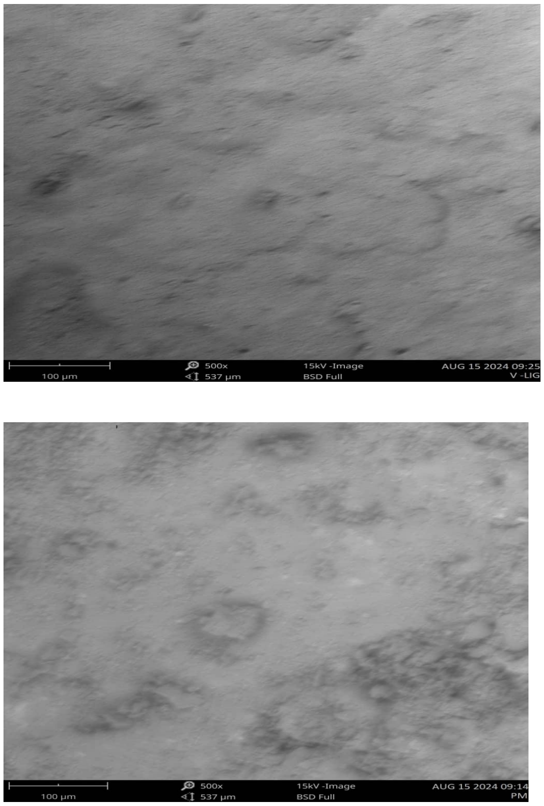

Figure 2: SEM images of the V-Lig(A) and PM-Lig (B)

Table 2 Degradation and char content of the untreated and treated fabric samples by Thermo- gravimetric analysis in Nitrogen atmosphere

Sample	Onset of degradation		Second stage of degradation		End set of degradation		Char Residue (%)
	Temp(0C)	Weight loss (%)	Temp(0C)	Weight loss(%)	Temp (^0C)	Weight loss (%)	
Control	200 -895	96%	-	-	-		0
V-Lig	140-270	15%	270-350	2%	350-717	57%	42%
PM-Lig	180-398	15%			400-715	65%	50%

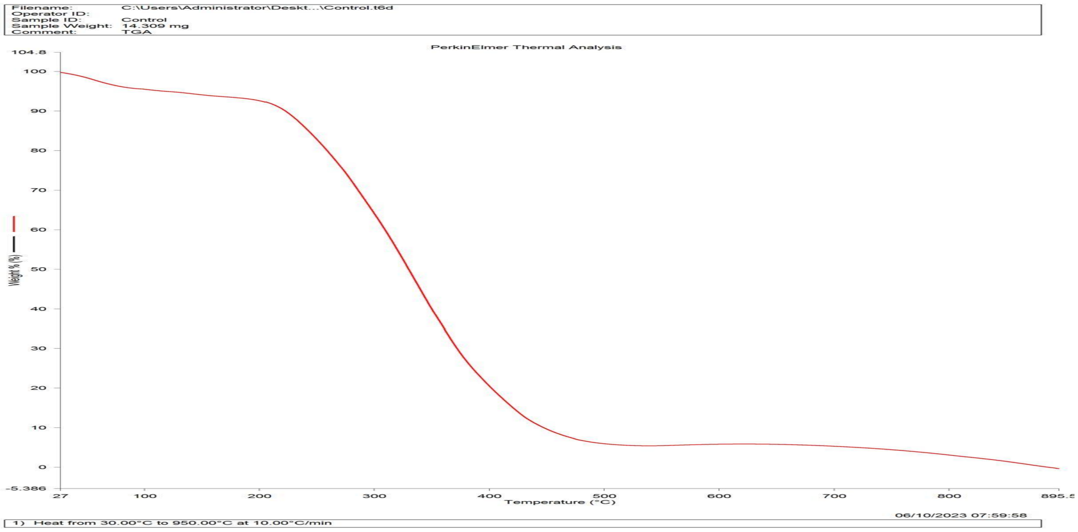

Figure 4A: TGA graph of the untreated fabric material (control sample)

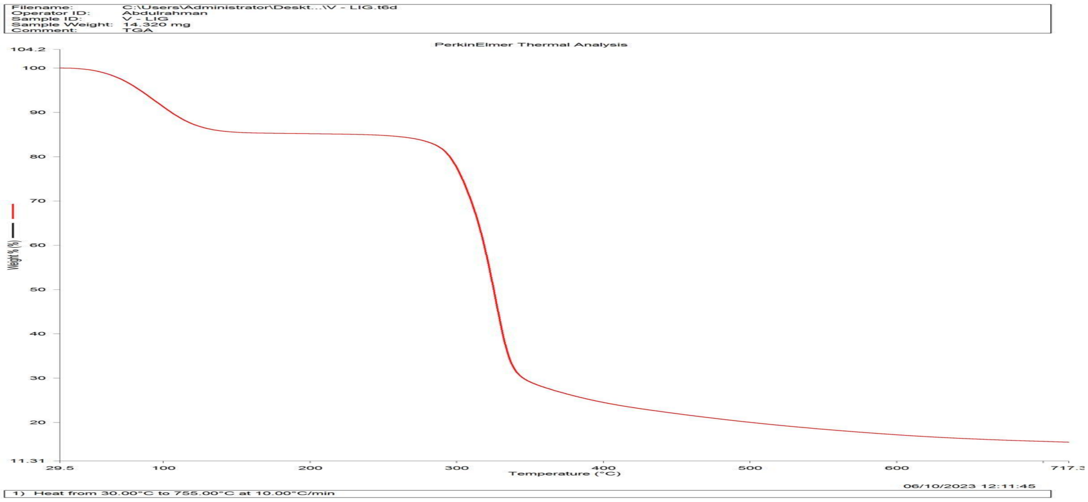

Figure 4B: TGA graph of the military fabric treated with the aqueous V-Lig solution

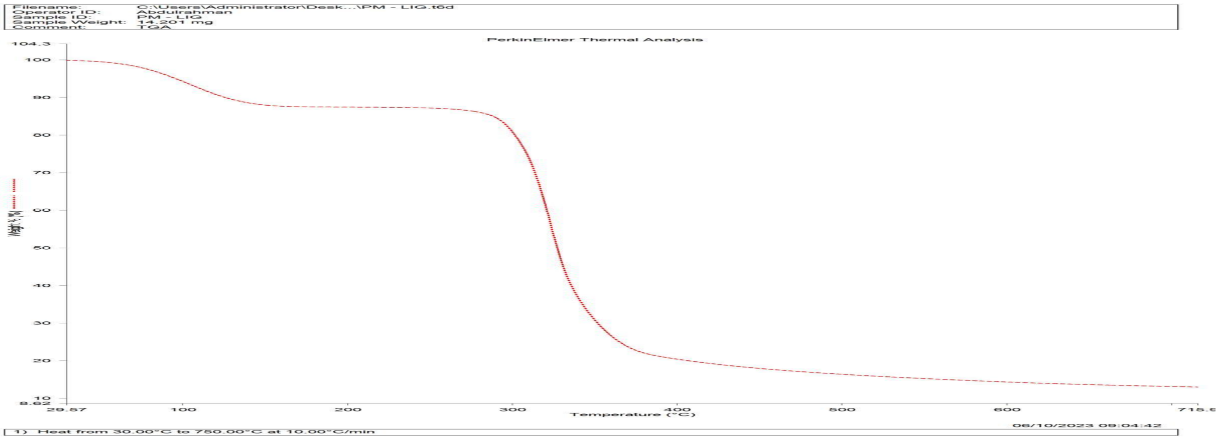

Figure 4C: TGA graph of the military fabric treated with aqueous PM-Lig solution

The TGA curves reveal the weight loss percentage of the materials with respect to the temperature of thermal degradation. Decomposition temperature with the corresponding weight losses at each stage of degradation and the amount of residual char formation were determined and summarized in Table 2. The thermal degradation of the untreated fabric material (control sample) started at 200 °C and degraded completely to 895.5 °C with no char residue formation. See Table 2 and Figure 4A.

The degradation of the fabric material treated with the aqueous solution of the virgin lignin (V-Lig) occurred in three stages as reported in other literatures. In stage one, the initial weight loss of 15 % occurred at 140 °C due to the evaporation of water. The second stage of the degradation occurred at 270 °C with a weight loss of 2 % attributed to the degradation of the components of carbohydrates in the lignin sample, which are converted to volatile gases such as CO, CO_2, and CH_4. The end set degradation of the material occurred at 350 °C and continued to 717 °C with a weight loss of 57 % and char residue formation of 42 %. (see Table 2 and Figure 4B).

The PM-Lig treated fabric material on the other hand had its onset of degradation between 180 °C-398 °C with a weight loss of 15 %. The end set of the degradation began at 400 °C and continued to 715 °C with a weight loss of 65 % and char residue of 50 %. This result is very similar to other works done on the phosphorylation of lignin and its thermal decomposition, See Table 2 and Figure 4C.

It can be seen from Table 2 and Figures 4A, B and C that the onset of degradation temperatures for the treated fabric samples (150 °C and 180 °C for V-lig and PM-lig) were low when compared to the control sample (untreated fabric material) which started degrading at 200°C. This early onset of temperature degradation may be due to the evaporation of water and the degradation of the components of carbohydrates present in lignin and the decomposition of the phosphoric acid molecules present in the samples. It can be observed from the TGA curves that thermal degradation did not commence until the materials had absorbed certain amount of heat energy thereby initiating the degradation processes and the subsequent break down of the structures causing molecular chains to be broken. The amount of char residue formed for the samples appeared to be higher in the fabric sample treated with the aqueous solution of PM-lig flame retardant (50 %), followed by the V-lig treated fabric material with 42 % char formation. There was no char formation for the control sample (untreated fabric material) which degraded completely to 815 °C. The reason for the char formation in the V-lig and PM-lig treated fabric materials is due to the nature of lignin as a char forming agent while The phosphates groups which were covalently bonded to the lignin structure were meant to promote dehydration and decarboxylation reactions thereby increasing the amount of carbonaceous char residue which becomes more stable at high temperatures. The stabilization observed in the PM-lig treated fabric material demonstrates that the presence of phosphorus increases char formation and enhances thermal stability of lignin at high temperatures.

Flammability of the untreated and the treated fabric samples.

The untreated (control sample) fabric and the fabric material treated with 30 % aqueous lignin solution (V-Lig) showed high flammability with intense dripping, igniting the samples and burning with flame spreads up to the holding clamps. The burning rate of the untreated military fabric was observed to be slightly higher (127.2 mms^{-1} with a weight loss of 94.662 %) when compared to the fabric sample treated with the virgin lignin (V-Lig) aqueous solution (109.0 mms^{-1} and 94.071 % weight loss), suggesting the flame retardant action of lignin due to its ability to form char thereby creating a protective barrier between volatile gasses released during combustion. Similar results were obtained by Prieur *et al.*, 2016 in their study on modified lignin as flame retardant for polymeric materials, where lignin was reported to exhibit interesting flame retardant properties in the polymers.

The fabric sample treated with phosphoric acid modified lignin flame retardant (PM-Lig) on the other hand showed a great improvement in flame resistance over the control sample and the sample treated with aqueous lignin solution. The rate of burning for the PM-Lig treated sample was calculated to be 45mms^{-1} lower with a great reduction in weight loss of 66.4 % when compared to the control sample whose burning rate was 127.2 mms^{-1} higher with a weight loss of 94.662 %. This was because phosphorus or the phosphate groups grafted to the lignin structure accelerated the charring of lignin and formed a significant amount of char which acted as a protective layer or barrier and insulated the cotton fabric from the effect of heat and flammable gases. The result obtained was compared with that of Mendis *et al.,* (2016), where it was reported that phosphorylation of lignin decreased the duration of flaming for the modified samples.

Table 3.0: UL-90HB Flammability test results

S/N	Sample	Ignition time (S)	Extent of burning (mm)	Rate of burning (mms1)	Weight loss (%)	Visual observation
1	Control	0.74	92.69	127.2	94.662	Smoky flame with dripping
2	V-LIG	0.85	84.00	109.0	94.071	Smoky flame
3	PM-LIG	1		84.0	74.524	Less flaming and dripping

Figure 5.0: Samples after UL-90HB test.

4. CONCLUSION

A novel bio-based and nontoxic phosphorus containing flame retardant was successfully formulated and used to impart flame resistance on military uniforms. It was formulated from lignin extracted from coconut husks and chemically modified using phosphoric acid. Fabric samples were treated with 30 % aqueous solutions of virgin lignin (V-Lig) and phosphoric acid modified lignin (PM-Lig) flame retardants at room temperature and air dried for 24 hours. The structure of the lignin extracted as well as that of the phosphoric acid modified lignin flame retardants were characterized by FT-IR and SEM . Flame retardant treated fabrics exhibited similar thermal stability in nitrogen atmosphere at 950 ^{0}C in TGA, showing slower weight loss with high char yields than the untreated fabric which was completely consumed. Flammability in UL-90HB test showed that the fabric sample treated with PM-Lig flame retardant exhibited excellent flame resistance than the control sample and the sample treated with virgin lignin solution which burnt very fast and most intensively. Thus thermal degradation and flammability studies of the samples proved that PM-Lig is a good flame retardant for military uniforms with an ease of application.

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