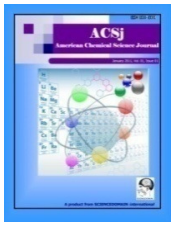

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The Effect of *Acacia senegal* Leaf Extract on the Flammability of Flexible Polyurethane Foam

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Authors' contributions

This work was carried out in collaboration between all authors. Author BA designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Authors POI and UZF managed the analyses of the study. Author HMM managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Health and environmental consequences of conventional flame retardants necessitated the incorporation of new plant based flame retardants. According to Australlian Plant Society all *Acacia* species can be used as fire retardants which lead to the selection of the leaves of *Acacia senegal* as a flame retardant on flexible polyurethane foam. GC-MS analysis revealed the presence of phosphorus and nitrogen compounds. Add on percentage ranged from 10-20%, Ignition time from 8-17 seconds, flame propagation rate ranged between 0.33-0.10 cm/s , after glow time ranged from 25-5 seconds and char formation ranged from 23-30%. The flammability characteristics of the flexible polyurethane foam was improved upon doping with the extract of the leaves of this plant.

Keywords: Add on; after glow; Char; flame propagation rate; flame retardants; ignition time; polyurethane; GC-MS.

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1. INTRODUCTION

Public demand for increased safety has led to greater interest in fire retardant materials in the last 30 years. Polymers find many uses and add greatly to the quality of modern day life. However, a major problem arises because most of the polymers on which these materials are based are organic and thus flammable [1]. At present, natural and synthetic polymer materials have been used worldwide in almost every industry.However,fire hazards associated with the use of these polymeric materials which result in the loss of life and properties are of particular concern among Government regulatory bodies, consumers and manufacturers alike.To reduce combustibility of the polymers, smoke or toxic fume production, the incorporation of halogen free flame retardant to polymers becomes an effective method for developing novel flame retardant materials [2].

Coupled with this is the fact that flexible polyurethane foams are found in every corner of our society and are highly combustible. This sad situation makes the use of fire retardant materials imperative in the Nigerian society in order to reduce fire accidents, injury and death. In this regard, extract of the leaves of *acacia senegal* was incorporated into the flexible polyurethane foam formulation to ascertain it's suitability for use as flame retardant.

A deciduous shrub or shrub tree with a flat to rounded crown. The tree has many branches and erect twigs spreading within the upright part. The branchlets have thorns just below the nodes. Leaves are small, grey green, alternate, and bipinnate. Flowers are white or cream coloured. Pods are dehiscent and the tree flowers during the rainy season [3].

The aim of this study is to develop novel plant based fire retardant for flexible polyurethane foam that is free from the shortcomings of the conventional flame retardants.

2. EXPERIMENTAL

2.1 Materials

2.1.1 Sample collection and identification

Two (2) kg of representative sample of *Acacia senegal* leaf was collected, identified and authenticated at the Harbarium of the Botany unit, Usmanu Danfodiyo University, Sokoto, Nigeria.

The polyurethane foam materials which comprisedof a Polyether Polyol (polyoxyalkylene) with an initiator that has a functionality of 2. Their costs are cheaper than Polyester Polyols, resulting foams are hydrolysis resistant and the functionality and equivalent weights can be varied, TDI (toluene disocyanate), Methylene Chloride, Dimethylethylamine, Tin (II) Isooctaoate and Silicon Oil were obtained from Concord Foam and Allied Chemicals Ltd opposite Sa'ad Petrol Station along western bye pass and Latex Foam Company in Rinjin Sambo industrial layout, all in Sokoto.

2.2 Methods

2.2.1 Preliminary sampletreatment

One (1) kg of the plant leaf material (representative sample) was thoroughly washed with clean water, dried at room temperature and then powdered using a grinder. About 200 g of the powdered plant material was soaked in water (200 ml) for 48 hours.At the end of the extraction the extract was filtered using Whatman filter paper.The filterate was concentrated to dryness in an oven at 70℃ and stored until further use [4,5].

2.2.2 Sample preparation

2.2.2.1 Preliminary screening of the fire retardant plant

Preliminary screening of the plant leaf extract for flame retardancy was done by soaking the extract of this plant in commercial polyurethane foam obtained from Latex Foam Factory and it' s efficacy tested based Add on, Ignition time, Flame propagation rate, After glow time and Char formation. Flame retardant activity was observed which prompted the final selection of this extract as a flame retardant in flexible polyurethane foam

2.2.2.2 Flexible polyurethane foam preparation

Flexible polyurethane foam was produced by mixing the TDI (2.5 ml) with the mixture of the polyol (12 ml), tin (II) isooctaoate (0.1 ml), silicon oil (0.2 ml), distilled water (0.5 ml) and dimethylethanalamine DMEA (0.1 ml). The mixture was thoroughly mixed with a glass stirrer to get a good dispersion of the reagentsso as to get a foam which will be used as a blank sample in the experiment [6].

2.2.2.3 Incorporation of the plant extract in the foam production

Fiveseries of different weight (0.2, 0.4, 0.6, 0.8,1.0 g) of the plant extract (dried) were measured and incorporated into the above formulation as a monomer. The resultant foams produced were left to cure for 48 hours in order to get a foam of good cell structure.

2.2.3 Analysis

2.2.3.1 Add on %

The flexible polyurethane foams produced after inoculation with the flame retardant was weighed and the one without flame retardant (blank) was also weighed and the difference divided by the weight of the untreated foam (blank) multiplied by 100. The same dimension was used for all the samples, that is, 2 cm × 2 cm × 5 cm [7,8].

$$\text{Add on \%} = \frac{Weight of Treated Foam - Weight of Untreated Foam}{Weight of Untreated Foam} \times 100$$

2.2.3.2 Afterglow time

The afterglow times were calculated using a stop watch to find out the time in seconds between the time the flame extinguished and the time the material stopped glowing [7,8].

2.2.3.3 Flame propagation rate

The sample was clamped vertically at a distance of 5 cm from a Bunsen burner flame and ignited and the time in seconds it takes the fire after being ignited to travel across the substrate were recorded and the flame propagation rate were calculated by dividing the distance by the time obtained using a stop watch clock The same dimension was used for all the samples, that is, 2 cm × 2 cm × 5 cm [7,8].

$$\text{Flame Propagation Rate} = \frac{Distance Travelled (cm)}{Time\ (s)}$$

2.2.3.4 Ignition time

The sample was clamped vertically at a distance of 5 cm and ignited at the base. The ignition time was calculated by recording the time in seconds after the Bunsen burner flame stroke the sample surface to the time when the sample caught fire [7,8].

2.2.3.4 Char formation

The amount of char formed was obtained by first crushing the sample in a crucible and weighing the content. It was then put in an electrically heated muffle furnace at 700℃ for 20 minutes and weighing the samples. It was calculated by dividing the weight of the material after burning by the weight of the material before burning multiplied by 100 [7,8].

$$\text{Char \%} = \frac{Weight of Material After Burning}{Weight of Material Before Burning} \times 100$$

2.2.4 Column chromatography

Acacia senegal leaf extract was subjected to column chromatographic technique to separate the fractions that may be responsible for the observed flame retardancy of the plant. Twelve fractions were collected from the column. This was achieved by packing the column with glass wool followed by silica gel using the wet method where the tap of the column was left open while the mixture of the silica gel and the solvent (hexane) were introduced into the column to prevent the formation of air bubbles. After that, soft rubber was used to tap the column so that the mixture settled very well in the column. It was then followed by the introduction of a mixture of the extract and silica gel. The top was covered by glass wool.

The elution started using hexane to elute the compound. After some time, gradient elution was introduced in to the process by varying the ratio of the solvent system where 10% toluene was used, then 20%. The ratio of the solvent was increased gradually up to 100%.

Furthermore, a mixture of toluene and acetyl acetate was introduced gradually by raising the ratio of the solvent system up to 100%. Then ethanol was introduced at a later stage of the elution so as to elute the compounds with the highest polarity in the same manner as the previous procedure.

2.2.5 Treating foam with the fractions

Flexible polyurethane foam of equal dimensions 2 cm × 2 cm × 5 cm were soaked in the fractions obtained from the column for 48 hours after which they were dried in an oven. The treated foams were tested to find out which fraction has the highest flame retardant action.

2.2.6 GC-MS analysis of the fractions

The most effective fraction was then taken for GC-MS to tentatively identify the compounds present.

3. RESULTS AND DISCUSSION

3.1 Result

The results of the various tests conducted are shown below and explained by Fig. 1 to 5. These are add on %, ignition time, flame propagation rate, after glow time and char formation. Extraction yield and elemental analysis of the active compounds were done.

3.1.1 Extraction yield

The extraction yield is 42.76 g which is equivalent to 21.38%

3.1.2 GC-MS result and elemental analysis

The result for the GC-MS and elemental analysis are given below:

1 2-Nitrohexane: Retention time 6.643 minutes, molecular formula $C_6H_{13}NO_2$, peak area 49.23% and the elemental analysis of Nitrogen is 10.6921%
2 1- [[[(2- aminoethoxy) hydroxyl phosphinyl] oxy] methyl] – 1,2 –ethanediyl ester: Retention time 23.200 minutes, molecular formula $C_{37}H_{74}NO_8P$, peak area 0.61% and the elemental analysis of Nitrogen is 2.0270% and that of Phosphorus is 4.4825%
3 Palmitin, 1,2–di- , 2 – aminoethyl hydrogen phosphate: Retention time 23.200 minutes, molecular formula $C_{37}H_{74}NO_8P$, peak area 0.61% and the elemental analysis of Nitrogen is 2.0270% and that of Phosphorus is 4.4825%

4. DISCUSSION

The incorporation of flame retardant into a flexible polyurethane foam formulation can result to some extent in combustion modified urethane foams [9]. Addition of a flame retardant may also alter the rise time of the flexible polyurethane foam depending on the flame retardant type and the amount of the flame retardant used [9]. Furthermore, the concentration of *Acacia senegal* leaf extract affect the rise time in increasing order of doping which resulted in low rising throughout the series. The above observation indicated that since the rise time is inversely proportional to the dope concentration, the flame retardant will be needed in small quantity.

From Fig. 1 *Acacia senegal* leaf extract has a good add on % with the highest as 20% and the lowest as 10% it was observed that add on depended on the dope concentration of the flame retardant material and the specific gravity of the material. It is the amount of the flame retardant material imbibed by the substrate [6]. The result showed that add on % increases as the concentration of the dope increases in all the foams. This is in agreement with the cited literature [6] because they follow the same pattern despite the fact that the result varies which could be due to the difference in the dope concentration of the flame retardants used in both cases.

Fig. 2 depicted the ignition time of the extract. It has a good ignition time. The lowest ignition time recorded in the series was 8 seconds and the highest was 17 seconds while that of the blank sample was 1 second. According to Ashida (2007) among the isocyanate based foams, polyurethane both flexible and rigid are more inherently flammable, thus, increasing the ignition time, that is, the time before the foam is ignited is important. Moreover, the ignition time increases as the dope concentration increases in the subsequent concentrations. This is in concert with (Ikeh, 2011).

Fig. 3 showed the flame propagation rate of the sample analysed. It has a good flame propagation rate. The highest flame propagation rate recorded was 0.10 cm/s and the lowest was 0.33 cm/s while that of the blank sample was 0.98 cm/s indicating good flame retarding activity the spread of a flame along a substrate takes place in the following manner; piloted or radiant heat raises the temperature of the material to pyrolysis level. The resultant volatile and combustible pyrolysates ignite at the right air or oxygen concentration. Some of the exothermic heat of combustion is lost to the surrounding while some parts are re-channeled back for further pyrolysis of the substrate, hence, combustion is sustained. The rate of this pyrolysis or combustion scheme along the substrate determines the rate of flame propagation [7].

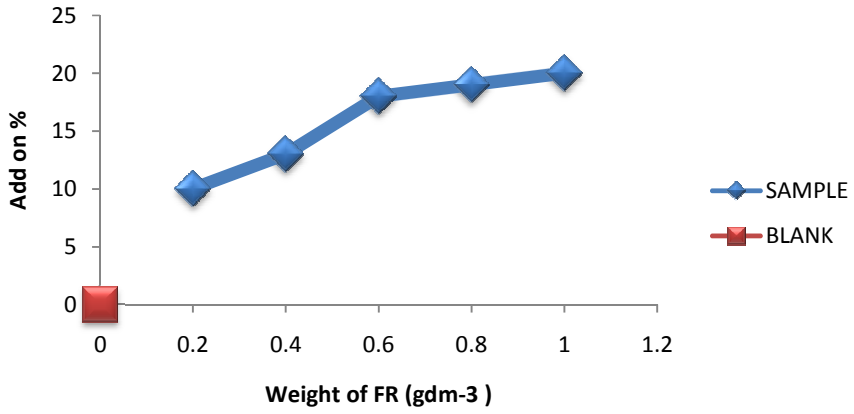

Fig. 1. Effect of Concentration on Add on

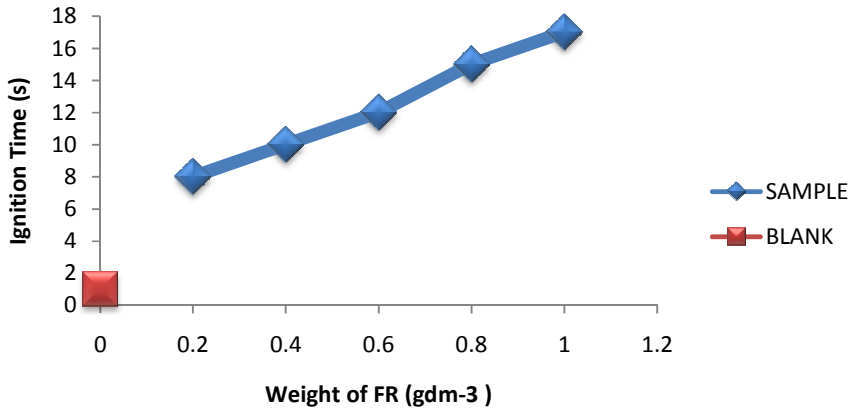

Fig. 2. Effect of Concentration on Ignition Time

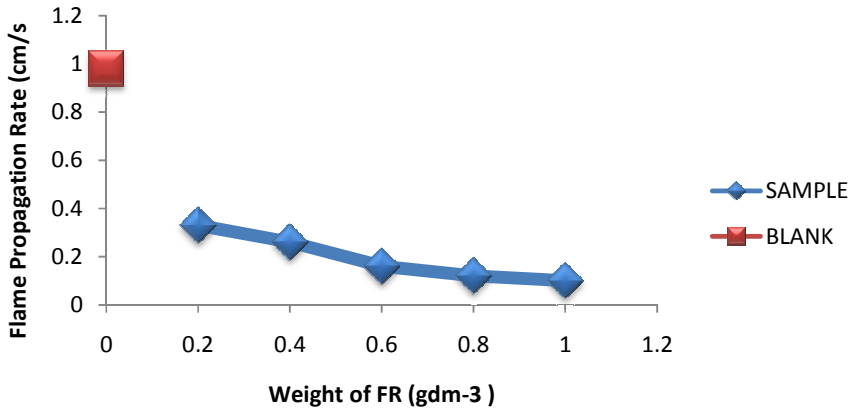

Fig. 3. Effect of Concentration on Flame Propagation Rate

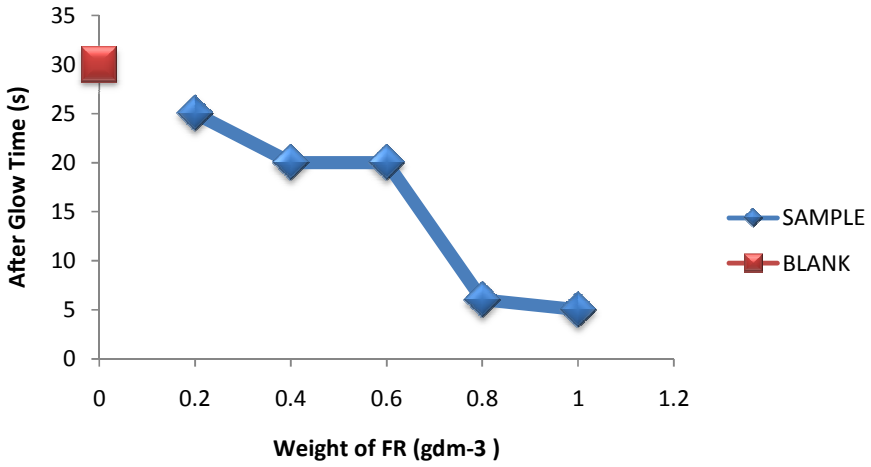

Fig. 4. Effect of Concentration on After Glow Time

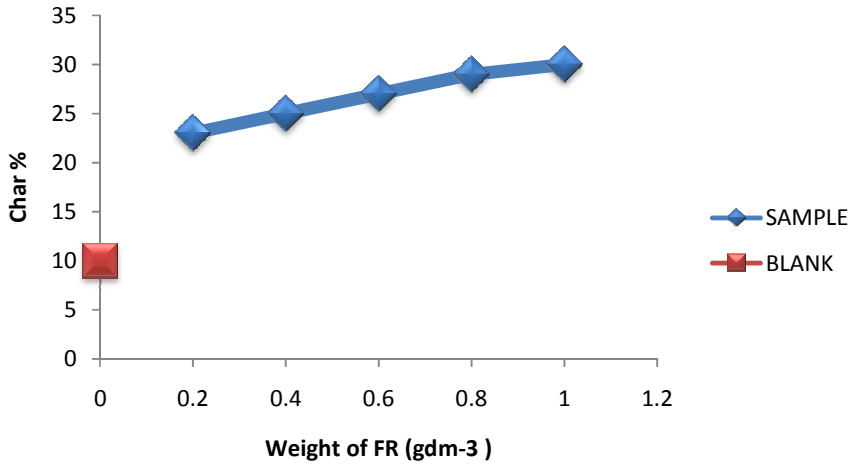

Fig. 5. Effect of Concentration on Char %

The flame propagation rate is inversely proportional to flammability resistance of foams. If the flame propagation rate is increasing the flammability resistance is decreasing and vice versa. This assertion is true for the extract as the flame propagation rate decreases as the dope concentration increases along the series. The blank sample burn while dripping the burning portion continuously.

Fig. 4 showed the after glow times for all the extracts analysed. *Acacia senegal* leaf extract has a good after glow time. The lowest after glow time was 5 seconds and the highest was 25 seconds while the blank sample took 30 seconds before the glowing stopped. Glow is a heterogeneous oxidative surface reaction and depends on the amount of burnt material and oxygen available [7,8,10]. Therefore, it can be seen that the treatment decreases the after glow time as you go up the series. For all the materials investigated, there are definite after glow times.

Finally, Fig. 5 showed the char % of *Acacia senegal* leaf extract. It has a moderate char forming ability. The lowest char formation was 23% and the highest was 30%.The char formed by the blank sample was 10%. Char formation is the formation of an impervious layer between the burning and the unburned part turning the material into a carboneceous char. The formation of an insulating barrier between these parts is brought about by dehydrating agents or char forming agents which hinder heat transfer to

unburned parts thereby reducing the reaction rate.

The difference in char percentage between the cited literature and the present work is significant although it is higher than the blank sample which may indicate that the sample analysed has char forming ability.

The GC-MS result showed the presence of 3 compounds which may be responsible for the flame retarding action of the *Acacia senegal* leaf extract. These compounds contain nitrogen and phosphorus. Phosphorus compounds act both in the condensed phase and vapour phase.

The condensed phase flame retardant action of phosphorus culminate in to the production of water which cools the flame by absorbing heat and char which is an insulator that slows the rate of polymer degradation and fuel formation and subsequent quenching of the flame [11]. The physical or chemical vapor-phase mechanism may be reasonably hypothesized in cases where a phosphorous flame retardant is found to be effective in a non charring polymer and especially where the flame retardant or phosphorous containing breakdown products are capable of being vaporized at the temperature of the pyrolyzing surface [11]. There is evidence that phosphorous-containing additives can act in some cases by catalyzing thermal breakdown of the polymer melt, reducing its viscosity and favouring the flow or drip of molten polymer from the combustion zone [11]. Certain nitrogenous compounds such as melamines, guanidins, ureas, and other amides are found to enhance or synergize the flame retardant action of phosphorous. This may be the mechanism that brought about the flame retardant activity of the extract.

5. CONCLUSION

Inoculation of flexible polyurethane foam with *Acacia senegal* leaf extract modified it's combustion behaviour by reducing the ignition time, after glow time, flame propagation rate and increase the char % indicating a possible vapour phase mechanism. This is important because reducing the flammability characteristics of flexible polyurethane foam will increase escape time during fire outbreaks, reduce fire injury and death and may act as an alternative to halogenated flame retardants due to stricter laws and regulations on their use which affect both humans and the environment.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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