

Flame Retardant Effects on Flexible Polyurethane Foam Treated with Potassium Aluminum Sulphate

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ABSTRACT

The effectiveness of potassium aluminum sulphate (alum) as flame retardant was studied by incorporating varying concentrations of it: 0%, 0.1%, 0.2%, 0.3%, 0.4% and 0.5% in the foam recipes. Fire characteristics of the foam samples produced were investigated and the results compared with the control. Results of the investigation showed that as the concentration of potassium aluminum sulphate increases, the ignition time, add-on, and char formation increases while the flame propagation rate and afterglow time decreases. Thermal behavior of the foam samples were also determined using thermo gravimetric analyzer. Results show that the novel flame retardant in the foam samples delayed the onset of degradation time and also high activation energy was required for the burning of the foam samples

Keywords: *Flame retardant, flexible, polyether, foam*

1. INTRODUCTION

Recently, much loss of life and properties caused by the fire hazards due to the use of polymeric materials has aroused much concern among consumers, manufacturers and government regulatory bodies [1,2]. Attempts have been made to minimize the fire hazards associated with polymer materials by the use of flame retardant additives. Flame retardants are materials that are used as coating on or incorporated into a combustible product to raise the ignition point or to reduce the rate of burning of product [3]. Generally, the interiors of homes, offices, vehicles and packages are decorated with polymeric materials in form of foamed plastics. Inherent flame retardant polymers such as polyurethane foams in which the flame retardant elements or groups are chemically bonded into the molecular chains of the original polymers, not physically added into the polymers matrices have been investigated and these have received more interest both in academic and industrial fields[4].

Flame retardant polyurethane foam has been prepared with diethyl N,N'-diethanol amino methyl phosphate and isocyanate[5]. The flame retardant polyurethanes with higher flame retardant properties have been synthesized with phosphorus-containing polyurethane by a novel reaction of phosphorus containing diisocyanate and diols [6]. A new flame retardant compound of polyether polyol has been synthesized from melamine formaldehyde, phosphorus oxychloride, and a compound with epoxy structure [7]. This polyether polyol was formed with polyisocyanate, water and catalyst by one step method, producing new structural flame retardant polyurethane flexible foam with good flame retardancy.

The effectiveness of halogen compounds depend on the ease of liberation of the halogen. Again, the nature

of the group to which the halogen atom is attached is very important because it determines the carbon-halogen ratio and carbon-halogen bond energy and hence the amount of halogen released during burning. Generally, alicyclic or aliphatic halogen compounds are more effective than aromatic halogen compounds for most polyolefin's burning at low temperature due to the lower carbon-halogen bond energies and hence relatively easier halogen-release [8]. The lower carbon halogen bond energy decreased their wider application in most engineering plastics and polymer materials burning at high temperatures. Halogen-containing flame retardant especially the bromodiphenyl ethers posed some problems during applications due to their high toxicity and potentially carcinogenic brominated furan and dioxins formed during combustion [9]. Most of the flame retardants are toxic and are not readily available; it becomes necessary to look for environmentally friendly and readily available local raw materials. The raw material chosen for the research is alum (potassium aluminum sulphate, $K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$). This work is aimed at investigating the effect of local raw material as flame retardant on flexible polyether foam.

2. MATERIALS AND METHODS

The raw materials used were obtained from Winco foam Nigeria Ltd, Awka. Potassium aluminum sulphate used as flame retardant was sourced from Anambra State Water Corporation, Awka, Nigeria.

3. PREPARATION OF MATERIALS

The alum was ground into fine powder using electric milling machine and was sieved to pass through laboratory test sieve of 63 μ m U.S.A. Standard Testing

Sieve, (ASTM) E. II Specification and kept in air tight polyethylene bag.

4. FORMULATION OF FOAM SAMPLES

The formulations in Table 1 were used for the production of flame retardant flexible polyether foam:

Table 1: Formulation of Foam Samples

Raw material	Pph(%)	A ₀ (g)	A ₁ (g)	A ₂ (g)	A ₃ (g)	A ₄ (g)	A ₅ (g)
Polyol	100	500	500	500	500	500	500
TDI	54.8	274	274	274	274	274	274
Water	4	20	20	20	20	20	20
Amine	0.14	0.7	0.7	0.7	0.7	0.7	0.7
Silicon	0.8	4	4	4	4	4	4
Stannous Octoate	0.16	0.8	0.8	0.8	0.8	0.8	0.8
Alum	Varied	0	0.5	1.0	1.5	2.0	2.5
	Percentage	0%	0.1%	0.2%	0.3%	0.4%	0.5%

Note: A₀ = 0%, A₁ = 0.1%, A₂ = 0.2%,

5. PRODUCTION OF FLEXIBLE POLYURETHANE FOAM

Flame retardant concentrations used in the production of flexible polyether foam are 0%, 0.1%, 0.2%, 0.3%, 0.4% and 0.5%. Measured quantity of alum was added to the polyol in a mixing bowl and stirred vigorously, followed by sequential addition of other raw materials including silicone, stannous octoate, 2-dimethylamino ethanol and water [10]. The mixture was stirred and TDI (toluene diisocyanate) was finally added and stirred continuously until when there was a sign of rising in the mixture which was immediately poured in the mould. The foam was allowed to solidify for about 10 minutes in the mould before being removed. The foam was allowed to stay for 20- 24 hours for its total curing after it had been removed from the mould before testing. The raw materials were measured based on 100g parts by weight of polyol [11]. The choice of the weights of the raw materials in the foam production was based on foam formulation while the samples were weighed on the triple beam balance.

6. CHARACTERIZATION OF THE FOAM SAMPLES

The following fire characteristics of the foam samples were analyzed: ignition time, burn length, flame propagation rate, flame duration, char formation, afterglow and add-on. [12, 13]. Thermo gravimetric analyses of the foam samples were also carried out.

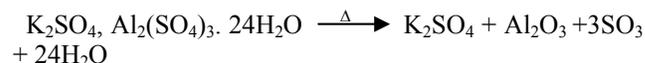
7. RESULTS AND DISCUSSION

The results of ignition time, burn length, flame propagation rate and add-on are shown in Fig.1, the results indicate that as the concentration of flame retardant increases, the ignition time increases. Ignition time is the time taken by the sample to catch fire. As the combustible material receives heat, there is increase in temperature

when it receives and redistributes the heat by conduction. While the temperature becomes very high, volatiles are formed and preceded to the surface but as the temperature of the materials increases decomposition reactions occur which release combustible product. Ignition occurs if sufficient heat is supplied. Potassium aluminum sulphate (alum) raises the ignition temperature of the foam.

Again, also the results show that the burn length decreases with increase in flame retardant concentration. Burn length determines the distance from the original sample edge to the farthest point showing evidence of damage due to combustion [14]. At 0%, the foam sample gave the highest burn length probably because of no flame retarding effect on the foam.

As can be seen in Fig.1 again, the flame propagation rate decreases as the concentration of flame retardant increases, incorporation of potassium aluminum sulphate has some effect on burning ability of foam samples. The equation below show the flame retardant action of alum



The flame retardant action of alum can be attributed to a number of factors. The non-combustible composition of gaseous products SO₃ and H₂O from alum dilute the concentrations of ignitable fuel pyrolysates thereby reducing their effectiveness. Al₂O₃ an excellent heat conductor deflects heat from the combustion zone [15]. Water has a cooling effect. Thus, the reduction in flame propagation rate must be due to two principal factors; evolution of water and non combustible gases which cool the flame as well as dilute the concentration of flammable pyrolysis product. Add-on depends on the concentration of the flame retardants used. In the result, it

can be seen that as the concentration of flame retardant increases, add-on increases.

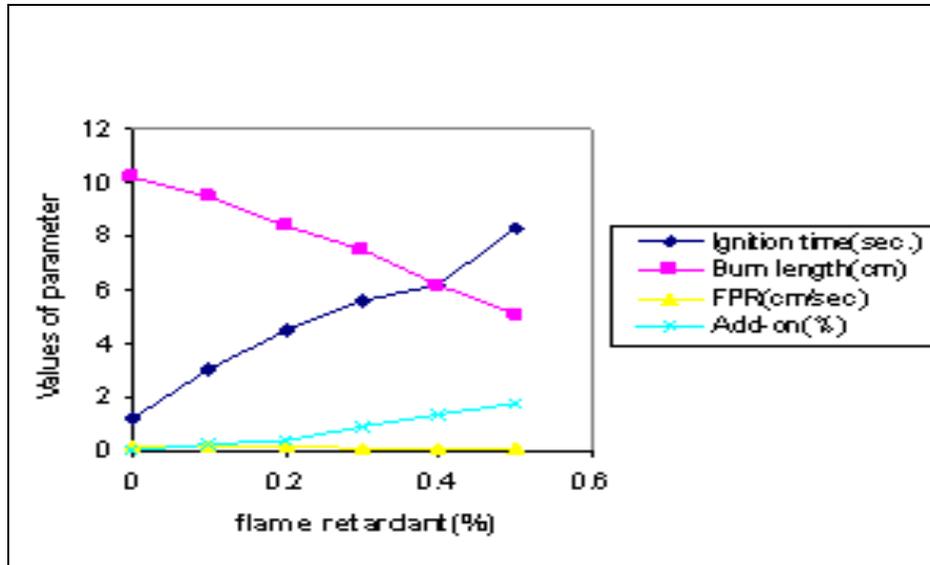


Fig 1: Ignition time, burn length FPR and add on of foam retarded with album.

The results of char formation, flame duration and after glow time are shown in Fig. 2. From the results, it was observed that as the concentration of flame retardant increases, the flame duration decreases sharply. At 0% concentration, the foam sample gave the highest value of flame duration.

On the other hand as the concentration of flame retardant increases the char formation increases. From literature, flame retardant promotes char formation at the polymer surface and this reduces flammability by acting as a barrier to inhibit gaseous products from diffusing to the flame, and to shield the polymer surface from the heat flux [16]. Polymer decomposition by heat entails firstly, pyrolysis, and then oxidation/combustion processes. At high temperature pyrolysis tends to proceed more violently with more disorderly disruption of chain structures to form flammable volatiles and with less condensation of carbonaceous entities to form char.

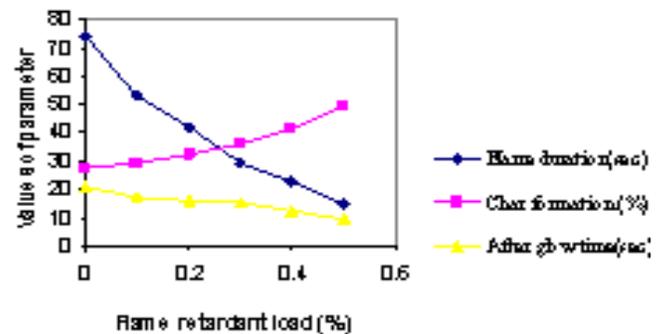


Fig 2: The result of flame duration, char formation and afterglow time

The results also indicate that as the concentration of the flame retardant increases, after glow time decreases. Glow is a surface oxidation process that depends on the quality of char left at the end of heating as well as available oxygen [17]. Thus, the higher the char, the longer the afterglow time (AGT) values. The significance of this observation becomes clear if it is realized that in fire situation, what is most striking is the flame and that attracts fire combatants. There have been countless cases when after the flame have been dowsed and everybody

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gone home, and the fire starting all over later. This scenario is because; the usually not perceptible glow was not extinguished. The longer the afterglow time, the higher the risk of re-enlightenment of a fire.

8. THERMAL BEHAVIOUR

Thermal analyses of flexible polyether foam filled with different percentages of the novel flame retardants were tested. It was observed that weight loss in the first and third degradation regions were lesser than the weight loss in the second degradation region. However, the initial

degradation is important and more representative of the formulation behavior as compare to that in the second and third degradation regions. In the case of the retarded foam the higher weight loss shows that when disposed after its life time, it can be environmentally friendly. Weight loss may arise from evaporation of residual moisture or solvent but at higher temperatures it results from polymer decomposition [18]. The TGA results of weight loss at different temperature and different concentrations/energy profiles of flame retardant flexible polyether foam are shown in Table 2.

Table 2: TGA results showing changes in weight loss (mg) with increase in temperature(°C)

Temperature decomposition stage	Weight loss of control sample(mg)	Weight loss of foam treated with different concentrations of Potassium Aluminum Sulphate (mg)				
		0%	0.1%	0.2%	0.3%	0.4%
First decomposition temperature(5-280°C)	0.5	0.5	0.2	0.5	0.3	0.2
Second decomposition temperature(281-420°C)	17.5	17.0	23.0	17.5	23.0	20
Third decomposition Temperature(421-560°C)	0.2	0.3	0.2	0.2	0.2	0.3

From ambient temperature to 280°C, the weight loss is 0.5mg for untreated sample but there is a variation in weight loss as the concentrations of flame retardant increase. The order of weight loss is shown as follows: 0.1% and 0.3% gave 0.5mg, 0.2% and 0.5% gave 0.2mg while the weight loss in 0.4% is 0.2mg. At the second

degradation region, that is, from 281 to 420°C, the weight loss is in increasing order 0.15(17.0mg) < 0.3% (17.5mg) < 0.5%(20mg) < 0.4% which show that sample treated with 0.4% is more environmentally friendly. In the third degradation region, from 421 to 560°C, the weight loss for the sample is 0.2mg almost in all concentrations.

Table 3: Energy Profile of flexible polyether foam retarded with Alum

Event	Thermo gram parameter	Pure sample (0%)	0.1%	0.2%	0.3%	0.4%	0.5%
Max. Temp(°C)	Stable up to	306.84	304.84	305.31	239.82	314.67	306.76
On set of thermal degradation time(min)		3.02	6.40	7.68	2.80	8.78	7.87
1 st step degradation Temperature(°C)	Start temp	62.22	112.19	134.72	66.10	152.58	136.86
	Mid temp	306.84	304.25	306.31	239.82	314.42	306.75
	End temp	356.98	328.04	358.83	338.59	340.42	369.04
2 nd step degradation Temperature(°C)	Start temp	370.14	372.82	434.64	-	-	-
	Mid temp	431.67	397.99	470.51	408.81	472.43	-514.72
	End temp	486.66	481.06	537.42	513.64	525.86	

Duration of degradation in (min)		11.22	11.13	14.84	17.21	13.81	12.85
1st step Energy req.(uV)	Start Energy	-19.01	-8.73	-15.98	-12.39	-13.72	-18.19
	Mid Energy	12.07	19.84	23.22	11.82	23.72	21.31
	End Energy	46.40	22.28	12..95	55.19	49.81	17.73
2nd step Energy req.(uV)	Start Energy	24.23	-0.73	32.75	-	-	-
	Mid Energy	33.14	17.50	79.07	29.64	24.44	-
	End Energy	72.11	20.97	43.06	58.35	29.04	94.12

9. CONCLUSION

In conclusion, the results of the fire properties of the flexible polyether foam samples studied showed decrease in burn length, flame propagation rate, after glow time and flame duration while ignition time, char formation and add-on increase with increase in concentration of flame retardant. The results of TGA show that less weight was lost at first and third degradation region while much weight was lost at second that degradation region. The energy profile indicates that Thermal degradation of materials requires energy supply for the breakage of bonds. This first stage of degradation at any stage has three sub-temperature steps, viz, the start, the mid and the final temperatures. In fact, at higher concentrations, it required more than 12 mins for degradation to commence. It is concluded that potassium aluminum sulphate can be used as flame retardant in flexible polyether foam.

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