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Effects of Mixed Rice Husk and Corn Cob as Fillers on Some Properties of Flexible Polyether Foam

P. U. Chris-Okafor [α], R.U. Arinze [σ], U.E. Ekpunobi [ρ] & M.C. Anugwom [ω]

Abstract- This work studied the effects of local fillers; rice husk and corn cob, on flexible polyether foam. These fillers were mixed in the ratio of 50:50. The foam samples were produced using polyol and other recipes for foam production in the appropriate formulations. Varying loads of the mixed fillers of particle size of 0.25mm, such as, 5%, 10%, 15%, 20% and 25% were incorporated into the foam. The physico-mechanical tests carried out on the foam samples were; density, hardness, compression, tensile strength and elongation at break. The creaming time, rising time, ignition time, char formation, char temperature and char duration of flexible polyether foam were also observed. The results showed that all the filled foams have higher densities than the unfilled foams as increase in filler loads increased the density of the foam samples. The fillers also modified other properties more than the unfilled foam used as a standard. The results of the flame tests showed that with increase in filler loads, the flammability of the produced foam was observed to be reduced. Hence, mixed rice husk and corn cob can be used as flame retardant in foam production and to improve other properties of the foam like density, load bearing ability and durability. More so, these fillers are agricultural wastes, completely organic and eco friendly, thus, can be used to produce biodegradable polyurethane foams.

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I. Introduction

Polyurethane products are everywhere, it is found in every area of human activities. Flexible polyurethane foams are one of the most important classes of cellular plastics and can be applied in the fabrication of a wide range of materials for different uses such as foam mattresses, pillows, furniture, cushioning materials for automobiles, packing, recreation, shoes, [1]. The versatility of polyurethane chemistry permits the production of a great variety of materials such depending on the initial ingredients used in the synthesis [2]. Its usefulness has displaced rubber foam in these applications because of improved strength, lower density and easier fabrication.

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This in turn necessitated the incorporation of a variety of fillers into foam samples. Filler denotes any material that is added to polymer formulation to lower its cost or to improve its properties. The use of fillers to modify properties of composition can be dated back to at least middle of 19th Century in Roman era when artisans used ground marble, calcium carbonate ($CaCO_3$) in lime plaster, frescoes and pozzolanic mortar, paper and paper coating [3]. The higher the degree of the surface area of the filler, the higher it's stiffening ability on the polymer. Fillers can be classified in many different ways ranging from their shapes to specific characteristics: Extender fillers and functional fillers are classes based on performance [4].

In general, industries that produce flexible polyurethane foams use fillers to modify the material's properties in some way, such as: dimensional stability, retraction from the mold and density [5-6]. When adding filler to a polymer to form a conjugated biphasic material, the tension applied to the polymeric matrix will be transferred in part to the disperse phase, the filler, since it presents properties superior to the pure polymer [7]. Efficient reinforcement is achieved by interactions of the filler polymer matrix via mechanisms of adhesion, which could be: adsorption, electrostatic attraction, chemical bonding and mechanical adhesion [8]. Accordingly, it is necessary to know the end-use of the material in order to use the correct concentration in the polymer matrix, obtaining a product of reliable quality.

Many research works have been done on petrochemical based polyurethane systems using inorganic fillers like calcium trioxocarbonate (IV), talc, kaolin etc. But the quest for local, cheap, organic, readily available, bio-degradable and eco-friendly materials has spurned many researchers into finding materials that can used as fillers for polyurethane foams. Some of these researches include the use of agricultural materials especially wastes as fillers incorporated into polyurethane foams. And the effects of various agricultural and organic materials as fillers in flexible polyether foam have been studied, which include: effects of coconut husk and corn cob as fillers in flexible polyurethane foam [9], effects of mixed organic materials as filler on the physico-mechanical properties of flexible polyether foam [10], the effect of coconut and palm kernel shells on density, porosity index and tensile

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properties of flexible polyether foam [11], palm kernel shell powder as filler in flexible polyether foam [12], etc. Chemicals used for the production of flexible polyether foam are: toluene diisocyanate, polyol, amine, stannous octoate, silicon oil and additives such as colourants, fillers, flame retardants, water and auxiliary blowing agents are also used [13]. Flexible polyether foams of different types have been produced using these chemicals. Such foams are used in various fields of life such as bedding, upholstery, laminated clothing and packaging. It also has good acoustic properties due to its structure [14].

Therefore, this study is to provide additional information on the effects of mixed rice husk and corn cob as fillers. These materials are locally available agro-wastes, lingo-cellulosic and relatively inexpensive as such that they can lower resin costs, improve stiffness and also can serve as eco-friendly materials to reduce the over dependence on petroleum-based plastics and reduce land-fills.

II. Materials and Methods

a) Samples collection and preparation

The polyurethane chemicals; polyol, toluene diisocyanate (TDI), silicone, stannous octoate, amine and water, used were sourced from Winco Foam Manufacturing Company in Awka, Anambra State. The local agricultural materials used as fillers; rice husk was sourced from Rice Mill in Abakaliki, Ebonyi State, while corn cob was sourced from Ifite, Awka, Anambra State both in Nigeria.

The rice husk and corn cob were washed with clean water to remove dust and unwanted impurities respectively. These were then dried under the sun for seven days, ground to fine powder and sieved by Gilson Automatic Sieve Tester, SS-15 model to pass through mesh sizes of 2mm, 1.6mm, 1.4mm, 0.8mm, 0.4mm and 0.25mm. The particle mesh size of 0.25mm was used for the two fillers to enhance easy homogeneity of the fillers.

b) Preparation of flexible polyether foam

125g of each of the fillers; rice husk and corn cob were weighed and mixed thoroughly to obtain a homogenous mixture containing 50% of each of the filler. 25g, 50g, 75g, 100g and 125g (5%, 10%, 15%, 20% and 25%) of the mixed fillers were weighed out respectively by using an electrical weighing balance of Model D-72336 Made in China. The percentage was based on the quantity of the polyol used. Polyol and toluene diisocyanate were weighed out and poured into separate mixing containers. Appropriate quantities of water, amine, silicone oil and glycerin were weighed separately using syringes for the purpose of an accurate stoichiometric reaction.

The measured silicon, water, amine and glycerin were poured into the beaker containing the polyol and mixed filler, this was stirred properly. Stannous octate was poured into the mixture and stirred for 10seconds. Then the measured toluene diisocyanate was poured into that same mixture and stirred. At this stage the creaming time was taken.

The mixture was transferred into mould with dimensions of 8 x 8x 8cm and the rising time (time taken to attain full height) was also taken with a stop watch. This process was repeated for the different weights of filler.

Ten minutes after full rise was attained, the foam samples were removed from the mould and allowed to cure for 24 hours before characterization. This was also repeated for the different foam samples based on their gram weight.

c) Foam Formulation

The choice of the weights of the raw materials to be used in foam production is not made arbitrarily, it is chosen on the basis of formulation (Biodun, 2000). If a high –density foam is desired, the tendency is that the volume of water used will be reduced; this is due to the fact that density is universally related to volume. Table 1 below showed the formulation of the produced foam samples.

Table 1: Foam Formulation

Material	Pphp	F_0	F_1	F_2	F_3	F_4	F_5
Polyol (g)	100	500	475	450	425	400	375
Filler (g)	0	0	25	50	75	100	125
TDI (g)	53	265	251.76	238.50	225.25	212	198.75
Silicone oil (mL)	0.90	4.50	4.28	4.05	3.83	3.60	3.38
Amine (mL)	0.20	1.00	0.95	0.90	0.85	0.80	0.75
Tin (mL)	0.15	0.75	0.71	0.68	0.64	0.60	0.56
Water (mL)	4.30	21.50	20.43	19.35	18.28	17.20	16.12
Glycerin (mL)	1.00	5.00	4.75	4.50	4.25	4.00	3.75

Note : pphp= parts per hundred of polyol

F_0, F_1, F_2, F_3, F_4 and F_5 = 0%, 5% (25g), 10% (50g), 15% (75g), 20% (100g) and 25% (125g) filler loads respectively.

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d) Characterization of Foam Samples

The following mechanical properties of the foam samples were determined using standard methods: density [15], while tensile strength, elongation-at-break, compression strength and hardness test were measured according to the ASTM-D standard specifications [16]. Its flammability tests were analyzed via flame duration, ignition time and char formation.

III. Results and Discussion

The results of the physico-mechanical properties of the foam samples were observed. The particle size of the filler has a dominating effect on these mechanical properties. Fillers become increasingly reinforcing as their particle size decreases in order to enable a large surface area for the polymer matrix-filler interaction.

a) Results of the mechanical properties tests

Table 2: Readings for the density test

Filler Load (%)	Filler load(g)	Density(kg/m^3)
0	0	20.03
5	25	20.06
10	50	21.98
15	75	21.22
20	100	31.89
25	125	34.48

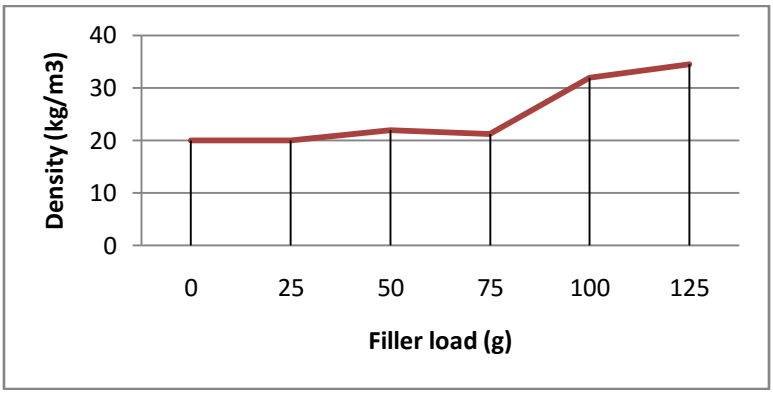

Fig. 1: Effect of filler load on density of the foam samples

From Table 2 and Fig.1, it is observed that increase in filler load causes increase in density of the foam samples. This could be attributed to the nature and high content of fillers which would fill up more voids, thus increasing its density. In flexible polyurethane foams, the fillers promote an increase in density and resistance to compression [14]. Density test helps to predict the performance of foam in their cushioning application as it predicts its durability and its ability to support and to push-back against weight and prevent foam collapse. This is in agreement with some other works [11, 12].

Table 3: Readings for the compression set test on the foam samples

Filler Load (%)	Filler Load(g)	Compression Set (%)
0	0	3.7
5	25	7.69
10	50	4.17
15	75	7.63
20	100	8.70
25	125	4.55

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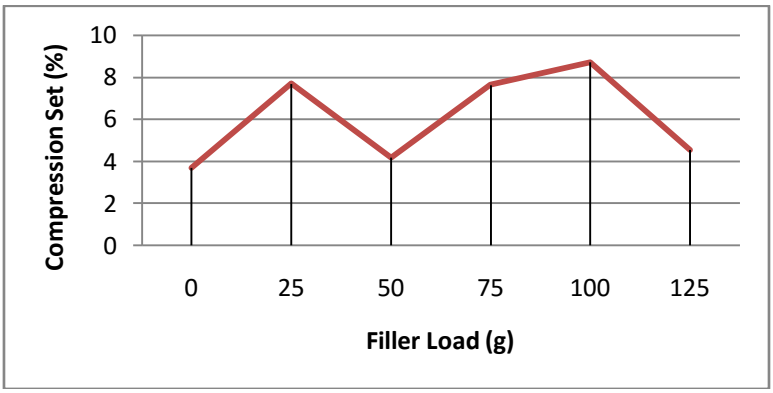

Fig. 2: Effect of filler load on compression set of the foam samples

It is observed that the fillers increased the compression strength of the foam, as shown in Table 3 and Fig. 2. Although, there was a varying trend in the decrease at filler loads of 50g and 125g respectively. This showed that the presence of filler improved the possibility of foam to return to its original size. This is one of the qualities of good and durable foams. It could be due to reinforcing property exhibited by the filler as a result of its cellulosic nature. From the result above, it is also observed that the decrease in compression strength at 125g filled foam, inferred that though125g filled foam sample has the highest density, still it has a low ability to return to original size after compression, hence it is evident that the density of a foam sample has no direct effect on its compression [13]. The increased compression set also agrees with the works of some other authors [10,11,12].

Table 4: Readings for the tensile strength test on the foam samples

Filler (%)	Filler load (g)	Tensile strength (MPa)
0	0	83.21
5	25	114.34
10	50	116.37
15	75	125.05
20	100	106.48
25	125	91

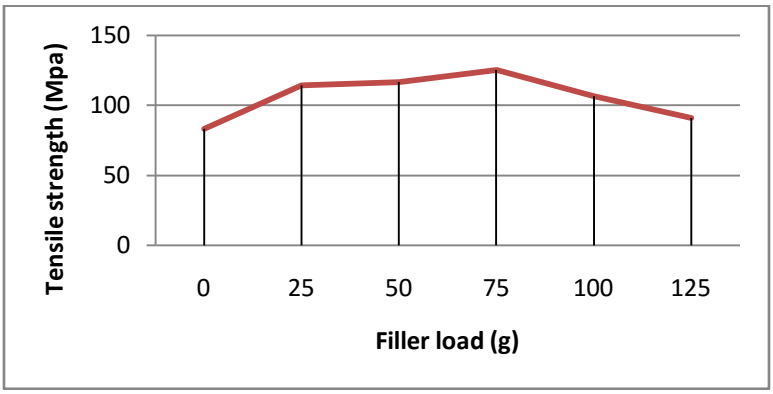

Fig. 3: Effect of filler load on tensile strength of the foam samples

It is observed that the tensile strength increased as the filler load increased as evident in Table 4 and Fig.3. This enhanced property could be attributed to good interaction between filler-polymer matrix. It could also be due to nature and small particle size of the filler thereby providing a good surface area to resist stress. Since, additives and fillers are added to improve the mechanical properties of foam [11,13].

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Table 5: Readings for % Elongation

Filler (%)	Filler Load (g)	% Elongation
0	0	130.10
5	25	128.23
10	50	116.37
15	75	109.12
20	100	106.48
25	125	94.43

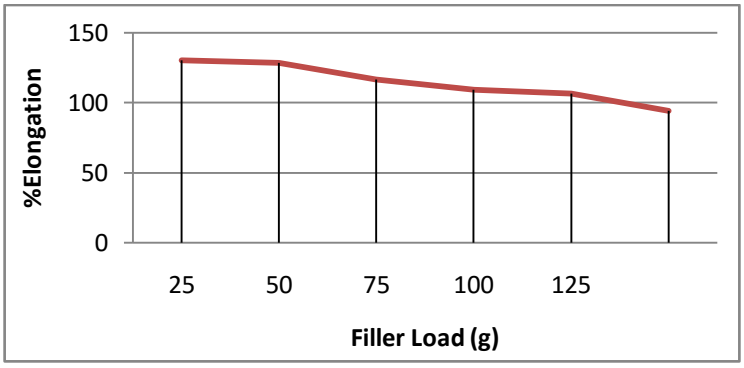

Fig. 4: Effect of filler load of %elongation of the foam sample

It is evident that the fillers decreased the % elongation-at-break of the foam samples as seen in Table 6 and Fig. 4 above. The cellulosic nature of the filler could be attributed to this effect that led to its tendency to elongate with a lesser filler ratio than a higher one. This also agrees with other works [10, 13].

Table 6: Readings for indentation force deflection (IFD) or hardness test

Filler Load (%)	Filler Load(g)	25%	40%	65%
0	0	28.3	38.0	71.5
5	25	32.0	40.6	84.3
10	50	43.2	52.6	101.5
15	75	47.4	61.4	109.8
20	100	35.9	46.6	91.1
25	125	58.8	69.9	123.5

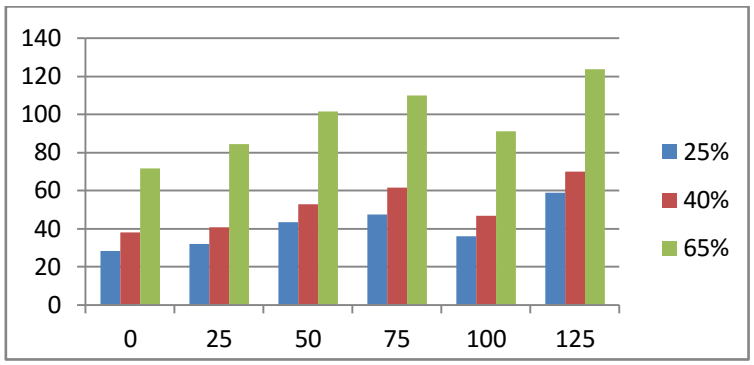

Fig. 5: Effect of filler load on IFD of the foam sample

It is evident that the hardness of the produced foam increased as filler load increased. This is as shown in Table 6 and Fig.5. The 65% IFD exhibited the highest hardness and this could be due to the nature and particle size of the fillers as well as the interactions between the polymer matrix-filler phase. 25% and 40% showed values less than 65% though the increase in hardness was displayed. The filler added in foam formulation modify the foam's hardness, improving the dimensional stability. Hence the increase in filler load increases the load bearing properties of the foam sample. This also agrees with the works of some other authors [11, 17].

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Table 7: Readings of creaming, rising and curing time

Filler Load (%)	Filler Load(g)	Cream time (sec)	Risingtime (sec)	Curing time(hr)
0	0	13	30	24
5	25	15	28	24
10	50	16	24	24
15	75	10	20	24
20	100	18	17	24
25	125	22	11	24

From Table 7 above, the creaming time of the foam samples increased as the filler load is increased. This shows that as the filler load is increasing, the creaming time is been delayed. Delay in creaming time is often essential for the production of complex parts particularly for multiple hardness foam cushions. Also, the variation in the rising time of the foam samples showed a uniform decrement in rise time as the filler load increases. This implies that the foam rises faster when the filler composition increases indicating that the blowing / gas production reaction between toluene diisocyanate and water occurs faster. It could therefore be deduced that the introduction of fillers in foam formulation greatly influences the reaction time which is a positive development; while the curing time was observed to be constant for all the filled samples.

Table 8: Readings of flammability test of the foam samples

Filler Load (%)	Filler Load(g)	Ignition Time(sec)	Flame Duration(sec)	Char Formation(sec)	Char Temp(^{0}C)
0	0	2	196	2	49
5	25	3	161	3	53
10	50	5	162	5	61
15	75	3	165	3	69
20	100	6	147	6	62
25	125	8	88	8	58

The ignition time was observed to increase as filler load increased as seen in Table 8 above. This implies that increase in filler load increases the ignition time of the foam samples and this is the same for the char formation except for 15% foam which has the same time as the 5% foam sample. The flame duration decreased as filler load increased. This shows that the presence of filler reduces the flame duration. Thus, this entails that mixed rice husk and corn cob can be used as a flame retardant in foam production.

IV. Conclusion

The results of the effects of the mixed filler; rice husk and corn cob on the flexible polyether foam samples showed the reinforcing abilities of the fillers. This could be attributed to the small mesh size of the mixed filler; that created a good surface area for the filler- polymer matrix interaction thereby enhancing the foam's mechanical properties. The filler also exhibited the tendency of flame reduction and can be used since polyurethane foams are highly inflammable.

The research showed that at 75g-100g concentrations of the filler gave the best reinforcing properties. Thus, moderate incorporation of filler can be used to give desired quality of products. Density of a foam sample has no direct effect on its creaming time, rising time and compression set. The load bearing properties of foam increases with increase in filler load. Good quality and flame retarding foam can be cheaply produced using easily accessible fillers like rice husk and corn cobs.

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