

## ARTICLE

# Investigation of the Mechanical Properties of Flexible Polyether Foam Filled with Eggshell and Groundnut Husk Powder

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## ABSTRACT

The study investigated the effect of eggshell and groundnut husk as fillers on flexible polyether foam. The fillers with the particle sizes of 50µm each were mixed in the ratio of 50:50. Diverse percentages of the two combined fillers ranging from 10%, 20%, 30%, 40% and 50% were incorporated into the polyether recipes in the appropriate foam formulations and foam samples were produced. The unfilled foam (0%filler) served as the standard control sample. The physico-mechanical test carried out on the foam showed increase in the properties such as density, compression set test, indentation hardness test, tensile strength test and decrease for elongation at break test as compared to the unfilled foam. The scanning electron microscopy result showed the cell sizes became smaller and concentrated with an irregular shape as the filler load increased, thereby making it denser and thicker. Eggshell and groundnut husk fillers may be used in the making of flexible polyether foam since these materials are organic in natures. They can improve the mechanical properties and biodegradability of polyurethane product. The utilization of these fillers in polymer composites production will help in sanitizing the environment by reducing landfills and producing eco-friendly waste and also it influences cost positively.

## 1. Introduction

Polyurethanes are polymers that consist of units joined by urethane links <sup>[1]</sup>. Flexible polyurethane (PU) foams are main products from urethane materials. They are obtained by polymerizing multifunctional isocyanates and polyol. Flexible polyurethane foams can be in the applications of the following; as cushion materials for automotive seat, mattress, refrigerators, insulations furniture, and in packaging

<sup>[2]</sup>. Flexible polyurethane material has become such an extensively used material because of its exceptional light weight, vigor to weight ratio performance and principally, is the amount of comfort, safety and value not matched by other sole materials <sup>[3]</sup>. This usefulness prompts the increase in the prices of polyurethane products consistently over the years which in turn necessitated the incorporation of variety of fillers into foam samples.

Also, the costs of flexible polyurethane foams are

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becoming progressively high due to the elevated price of raw materials [4]. Flexible polyether foams are classified as low density, medium density and high density [5]. The worth of foam depends mostly on its density. Consequently, for the high price of raw material, it is important to source for cheaper, easily accessible and eco-friendly material that can be used as filler. Fillers are used in plastic and rubber industries as finely divided solid materials which are incorporated into the liquid, semi-liquid or solid composition to transform the physical properties of the composition and to lessen cost. Primarily, fillers are used to lower end products, thus, they are called extenders. Along with over twenty most key fillers, calcite ( $\text{CaCO}_3$ ) holds the largest market volume and is chiefly used in plastic sectors. Other fillers include dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ), kaolin and talc [6].

In recent times, there is growing efforts to expand novel classes of bio-inspired composite materials. The major benefit of these types of materials is that they are environmentally friendly and do not add to the depletion of energy resources because they are derivatives of renewable resources [7].

However, earlier studies have shown that chicken eggshell (ES) is an agricultural by-product that has been listed globally as one of the most awful environmental problems. This eggshell contains about 95 % calcium carbonate in the form of calcite and 5 % organic materials such as type X collagen, sulphated polysaccharides, and other proteins, which makes it a rich source  $\text{CaCO}_3$  as well as being environment friendly, biodegradable, available and low dense. This uniqueness succeeds eggshell as a superior entrant for bulk quantity, economical, lightweight and low load-bearing composite uses, such as the automotive industry, trucks, homes, offices, and factories. Even though, there have been numerous attempts to exploit eggshell components for diverse applications, its chemical composition and accessibility makes eggshell a prospective resource of filler in polymer composites [8].

Thus the aim of this study is to harness the potentials of these mixed organic waste materials as fillers incorporated into flexible polyether foam.

## 2. Experimental

### 2.1 Materials and Chemicals

The foam chemicals; toluene diisocyanate, TDI, polyol, silicon oil, stannous octate and amine used for the production of the foam samples were obtained from Vita

Foam Nig. Plc. Ikeja, Lagos State, Nigeria; while the fillers; egg shells and groundnut husks were sourced from Chrunches and Eke- Awka market respectively all in Awka, Anambra State, Nigeria.

### 2.2 Preparation of the Fillers

The egg shells were washed carefully and sun desiccated. They were then pulverized into fine powder, using an electric grinding appliance. Further sieving was done to obtain a fine smooth powder with a mesh of  $50\mu\text{m}$  and then stored in polyethylene bag.

The groundnut husks were hand-picked, washed, sun dried and milled into fine powder using an electric grinding appliance. Further sieving was done to obtain a fine smooth powder with a mesh of  $50\mu\text{m}$  and then stored in polyethylene bag. The egg shells and groundnut husks powders were mixed homogeneously in the ratio of 50:50 and stored in a polyethylene bag.

### 2.3 Preparation of Flexible Polyether Foam

The polyol, toluene diisocyanate and fillers with particle size of  $50\mu\text{m}$  were precisely weighed into different beakers using triple beam weighing balance. The supplementary raw materials required in minute quantities were measured using syringes. Polyol and the mixed fillers were poured in a plastic container, and the blend was stimulated very well until total homogenization was achieved. About 500g of polyol was poured into an empty clean jug while 50g of the mixed fillers (groundnut husk and eggshell mixture) was added to the polyol and stirred vigorously. 28g of distilled water and 2g of amine was added while mixing. 5g of the silicon oil and 1g of stannous octate was also added to the jug while stirring. Lastly (325g) of TDI was poured into the jug and the mixture was stirred until it was fully homogenised. The whole mixture was then smartly poured into a prepared metal mould. This procedure was repeated 2, 3, 4 and 5. Sample zero ( $X_0$ ) has no addition of fillers (eggshell and groundnut husk). The measurements of these recipes were based on parts per hundreds of polyol [9].

A stopwatch was used to monitor the rise time of each foam sample, and foam sample was allowed to cure for 24 hours. Then, the cured foam blocks were cut to standard sample sizes for analysis. Thereafter, the foam samples produced were subjected to mechanical properties test.

Table 1 shows the foam formulation with its recipes and appropriate quantities.

**Table 1.** Foam formulation

Raw materials	Pph	X <sub>0</sub> (g)	X <sub>1</sub> (g)	X <sub>2</sub> (g)	X <sub>3</sub> (g)	X <sub>4</sub> (g)	X <sub>5</sub> (g)
Polyol	100	500	500	500	500	500	500
TDI	58.99	325	325	325	325	325	325
Water	4.36	28	28	28	28	28	28
Amine	0.422	2	2	2	2	2	2
Silicon oil	0.822	5	5	5	5	5	5
Tin	0.139	1	1	1	1	1	1
Filler load		00	50	100	150	200	250

**Note:** Pph = Part per hundred; X=sample; g=grams; X<sub>0</sub> = 0% filler; X<sub>1</sub> = 10%; X<sub>2</sub> = 20%; X<sub>3</sub> = 30%; X<sub>4</sub> = 40% and X<sub>5</sub> = 50%

### 3. Results and Discussion

The results of the physico-mechanical properties of the produced foam samples were discussed. The unit mesh size of filler has a dominating impact on all the properties.

#### 3.1 Creaming Time

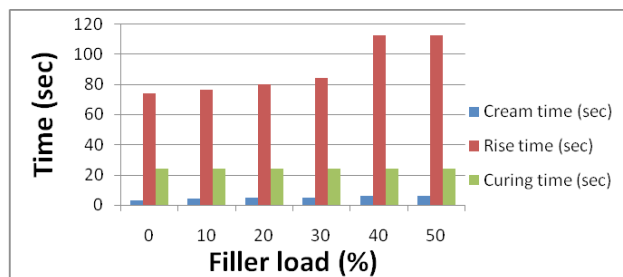
This is the first incident measured and occurs usually a short time after incorporation when the mixed liquid turned cloudy in appearance and liquid starts to rise from its initial stable stage. The cream time was observed to be higher than that of the unfilled foam. As soon as the generation of carbon (iv) oxide starts, the creaming time is dependent upon the quantity of amine used. High amount of amine starts the cream time.

#### 3.2 Rising Time

This occurs when the reacted foam has reached its biggest level or utmost height. At this stage, the foam increase has taken place such as cell structures are foamed, gas reaction accompanied by generation of carbon (iv) oxide takes place and exploration of auxiliary blowing agent is present in formulation.

#### 3.3 Curing Time

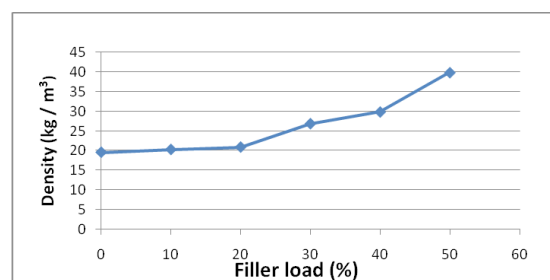
This also occurs when the foam is completely risen, the foam is firstly soft gelatinous mass and the phase between the accomplishment of full rise and whole solidification of the produced foam is the curing time. This is basically the moment main cross linking occurs. The results of creaming, rising and curing times recorded during the production of flexible polyether foam are as shown below.



**Figure 1.** The effects of the mixed fillers on creaming time, rising time and curing time of filled flexible polyether foam

The filler was observed to increase the creaming and rising time as shown in Figure 1. This means the filler increased the reaction time, giving room for complete nucleation of the foam recipes.

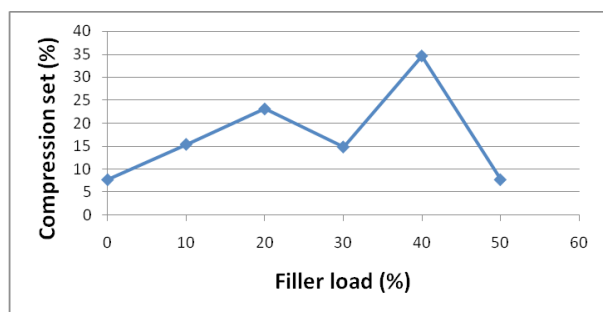
#### 3.4 Density



**Figure 2.** Result of filler load on density of the produced foam samples

From the result above, it is seen that the density of the polyether foam was improved by the filler load. As the filler load increased, the density also increased from 10%, 20%, 30%, 40% and 50% filler loads as shown above in Figure 2. A formulation of 22kg/m<sup>3</sup> density was used to prepare the entire laboratory mixing and it was discovered that the densities increased as the quantity of fillers (egg shell and groundnut husk) were increased and this could be due to the better mechanical properties exhibited by the fillers. Hence, the increase in the density can be said to be due to the filler particles filling the voids in the flexible polyether foam structure which tend to compact the materials thereby reducing the foam porosity and make it denser. As the filler content increased, the hardness of the foam samples also increased. It would not simply collapse or descend after weight is positioned on it. In other words, the produced foam samples will be durable and not sink nor collapse when sat upon. Hence, additives and fillers are used to increase foam density and it is the ability of the foam to provide support. This is in accordance with other related researches [10-12].

### 3.5 Compression



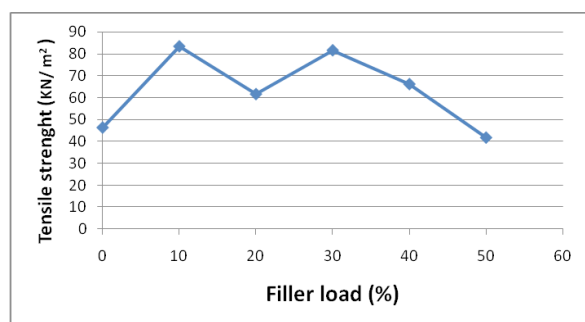
**Figure 3.** Result of filler load on the compression set of the produced foam samples

The compression set result as shown in Figure 3 was seen to increase as the filler load increased. This implies that the foam sample would easily regain its original height after a bulky mass is lifted from the foam. This is one of the qualities of superior and resilient foams. This could be accredited to the enhanced mechanical properties exhibited by the fillers.

The filler increased the compression set test by almost 100% except at 50% filler load which had the same value with the unfilled foam. This enhanced compression set could be due to the reinforcing nature of the mixed filler, thereby improving the mechanical property of the foam sample. So the optimum filler load for the compression set is at 40% and adding more filler could affect the property of the foam. This is in agreement with the results obtained by some other authors [4,13,14].

### 3.6 Tensile Strength

This is one of the most important mechanical tests for any polymeric material. It shows the stress-strain curve in tension.

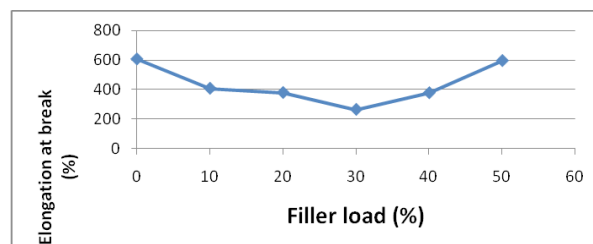


**Figure 4.** Result of filler load on the tensile strength of the produced foam samples

It is seen from Figure 4, that the result of the tensile strength of the produced foam samples improved as the filler load increased but a sharp decrease was observed at 50% filler content with the value 41.8 %. This increment could

be attributed to cellulosic nature of the groundnut husk and reinforcing nature of the eggshell. These combined together improved the tensile strength of the foam samples. The foam samples tend to be strong. This is in accordance with the result of some order related works [14,15].

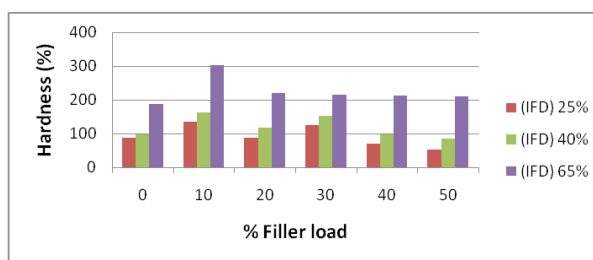
### 3.7 %Elongation at Break



**Figure 5.** Result of filler load on the %elongation at break of the produced foam samples

The result of the filler on the %elongation at break of the flexible polyether foam is as shown in Figure 5. It is experimental that elongation slightly decreased as the filler load increased. This decrease was lower than that of the unfilled foam (0% filler load) with the highest value (609.0%). 50% filler load experienced a greater value for elongation at break (598.2%) than other samples with value of 409.0%, 380.6%, 265.3% and 378.7%. This could be as a consequence of its greater tendency to extend with a smaller filler ratio than a higher ratio. That shows the flexibility of the produced foam samples, that is its ability to return to its original shape after bending or folding, which is part of a good quality of foam. This is in agreement with the results of some other researchers in the past [14,16].

### 3.8 Hardness



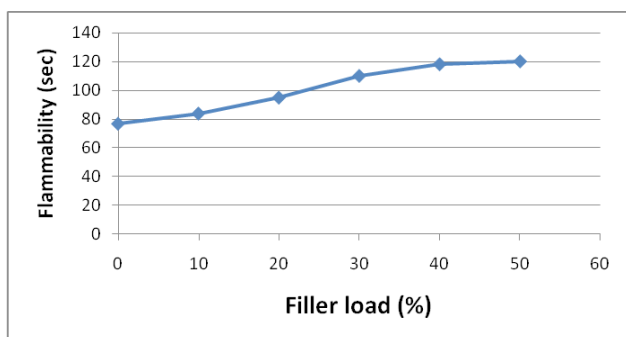
**Figure 6.** Result of filler load on the hardness (IFD) of the produced foam samples

The stability of polyurethane foam is considered as a physical property called indentation force deflection (IFD). The boost in the filler load was observed to have a positive effect on the produced foam samples by its increase on the hardness properties of the foam samples as shown in Figure 6. This implies that the filler will have a hard result on the



foam and will bear a lot of heaviness over an extended period of time without failing immediately. The crystalline nature of the egg shell and cellulosic nature of the groundnut husk have been proved to be very tough and resilient. The hardness of foam is seen by its ability to withstand heavy weights without collapsing. And this was exhibited by these fillers, so the produced foam samples could carry heavy loads. This result is in agreement with other related works <sup>[13,14]</sup>.

### 3.9 Flammability



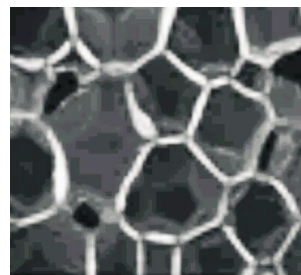
**Figure 7.** Result of filler load on the flammability property of the filled polyether foam

From Figure 7, the flame duration time are 77sec, 84sec, 95sec, 110sec, 118sec and 120sec for 0%, 10%, 20%, 30%, 40% and 50% filler loads respectively. This simply shows that the filler increased the flame duration of the foam sample. This may be due to the interaction between the fillers and foam matrix phase which fills up the pores in the foam making it harder for the flame to go off if it ignites. This could also be attributed to the fact that one of the filler (groundnut husk) is flammable and that the filled foam constitutes more residues after ignition. So, it is advised that local materials that can reduce the flammability or act as flame retardant should be incorporated into the foam in order to reduce its flammability.

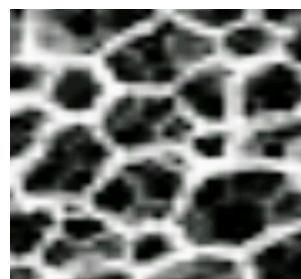
### 4. Results of the Surface Morphology of the Filled Polyether Foam via the Scanning Electron Microscope



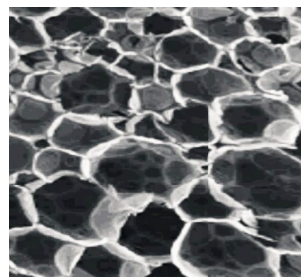
**Figure 8a.** 0% Filler



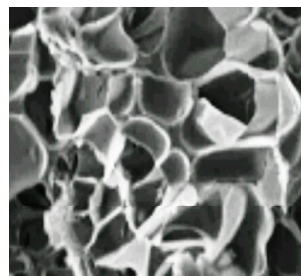
**Figure 8b.** 10% Filler



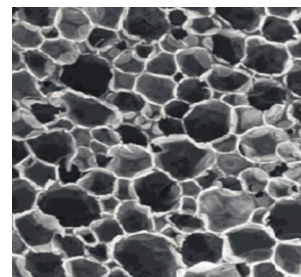
**Figure 8c.** 20% Filler



**Figure 8d.** 30% Filler



**Figure 8e.** 40% Filler



**Figure 8f.** 50% Filler

**Figure 8.** Effects of the filler load on the surface morphology (SEM micrographs) of the filled polyether foam

The result obtained from the SEM analysis of the flex-

ible polyether foam produced showed that the control experiment (0% filler load) displayed a cell morphology that basically appeared to be hexagonal or heptagonal in shape, with thicker strut on the walls. The produced foam has many pores/voids which makes it flexible polyether foam.

The increased mechanical properties exhibited by the foam sample can be explained by the surface morphology result. It could be seen that as the filler loads increased, the sizes of the pores were reducing as shown in the micrographs Figures 8b-f. As the filler was observed to compact and distort the cell walls of the polyurethane foam, thereby making the foam denser, thicker and harder. Hence, the SEM results could be used to deduce the improved mechanical properties of the filled flexible polyether foams.

## 5. Conclusions

This study has shown that the incorporation of mixed organic fillers (eggshell and groundnut shell) in foam formulation has impacted good qualities on the produced flexible polyether foam samples. This was due to the strengthening nature of the fillers and small mesh size of the mixed fillers; that created a good surface area for the filler-polymer matrix interaction thereby enhancing the foam's mechanical properties. The increase in quantity of fillers resulted in the progressive increase in density, hardness indentation and compression thus affecting cost positively. The use of organic fillers will have a positive impact on the environment. It brings to rest the challenges of waste disposal by polyurethane foam manufacturing company due to its biodegradable property. The optimum mechanical property was achieved at 40% filler load.

Hence, foams produced with these fillers are strong, dense, flexible and can return to its original size and shape after being bent or folded. Foam manufacturers are encouraged to harness these organic waste materials in their production of foams due to their availability, cost effectiveness, processability, economic and environmental impacts; as they can replace conventional materials like calcium trioxocarbonate (iv) in foam production.

## References

- [1] Kugler, P.M., Wiltz, E.P., Wuilay, H.. A New Era for MDI Flexible Polyurethane Slab Stock Foam, Cellular plastic, 1997, 33(2): 159-184.
- [2] Usman, M.A., Adesun, S.O., Osifeso, G.O.. Optimum Calcium Filler Concentration for Flexible Polyurethane Foam Composite, A Journal of Materials and Materials Characterization Engineering, 2012, 11(3): 311-320.
- [3] Klempner, D., Sendjaveric, V.. Handbook of Polymeric Foams and Foam. Technology. Hanser Publishers. Munich, 2004: 339-378.
- [4] Onuegbu, T.U., Obianuko, N., Mbachu, G.U., Iloa-maeke, I.M.. Effects of Animal Waste (Goat Femur) as a Filler in Flexible Polyether Foam, Journal of Basic Physical Research, 2010, 1(1): 5-8.
- [5] Ajiwe, V. I. E, Ulakpa, C. M, Onuegbu, T. U.. Additive Effects On Production of Polyurethane Flexible Foams, World Journal of Biotechnology, 2005, 6(1): 957-967.
- [6] Hans, G.E.. Plastics, General Survey in Ullmann's Encyclopedia Industrial Chemistry, Wiley, VCH, Weinheim, 2005.
- [7] Toro, P., Quijad, R., Yazdani-Pedram, M., Arias, J.L.. Eggshell, a New Bio-Filler for Polypropylene Composites, Materials letters, 2007, 61(22): 4347-4350.
- [8] Abdullah, A.S.. Water adsorption mechanical properties of high density polyethylene/egg shell composite, Journal of Basrah Researches (Sciences), 2011, 37(3): 36-42.
- [9] Vitafoam Plc Nigeria; Manual Guide.
- [10] Saliba, C.C., Orefice, R.L., Carneiro, J.R.G., Duarte A.K., Schneider, W.T., Fernandez, M.R.F.. Effect of the Incorporation of a Novel Natural Inorganic Short Fibre on the Properties of Polyurethane Composites; Polymer Test, 2005, 24(7): 819-824.
- [11] Dalen, M.B., Ibrahim, A.Q., Adamu, H.M., Nurudeen, A.A.. Effects of calcium carbonate and Kaolin filler loading on curing rates of polyurethane foams. International research Journal of pure and applied chemistry, 2014, 4(6): 691-694.
- [12] Chris-Okafor, P.U., Arinze, R.M., Nwokoye, J.N, Ukpai E.U.. Effects of Coconut husk and Corn cobs as Fillers in Flexible polyurethane foam. American Journal of Polymer Science and Technology, 2017, 3(4): 64-69.
- [13] Ruijun, G., Mohini, M.S, Samir, K.K.. A Feasibility Study of Polyurethane Composite Foam with Added Hardwood Pulp, Industrial Crops and Products, Checkmate press, New York, 2013, 42(2): 73-279.
- [14] Chris-Okafor, P.U., Arinze, R.U., Ekpunobi, U.E., Anugwom M.C.. Effects of Mixed rice husk and Corn cob as Fillers on Some Properties of Flexible Polyurethane Foam. Global Journal of Science Frontier Research B: Chemistry, 2017, 17(2): 2249-4626.
- [15] Onuegbu, T.U.. A Study On the Effects of Coconut and Palm Kernel Shells On Density, Porosity Index and Tensile Properties of Flexible Polyether Foam, ARPN Journal of science and Technology, 2012, 2(6): 529-532.
- [16] Billmeyer, F.W.J.R.. Text Book of Polymer Science. New York: John Wiley and Sons. 1984, 246.
- [17] Onuegbu, T.U., Ugwu, L.E., Ogunfeyitimi, O.. Physio-mechanical Properties of Flexible Polyether Foam: Comparative effects of fillers, Chemistry and material research, 2013, 3(9): 48.