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ARTICLE

Impact of Mixed Fillers on the Physico-mechanical Properties of Flexible Polyether Foam

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ABSTRACT

The effect of proportional blend of periwinkle and African star apple seed shell as bio-fillers in flexible polyether foam was studied. Flexible polyether foam samples incorporated with these bio-fillers at varying percentages; 10% (S_1), 20% (S_2), 30% (S_3), 40% (S_4) and 50% (S_5) were produced respectively, while 0% (S_0) which had no filler was used as control during the experiment. The mechanical properties of the produced foam samples were determined via density, compression set, indentation hardness, tensile strength and elongation at break tests. The cream time, rise time and height of the foam as parameters for characterising the produced foam samples were determined too. Flammability test was also carried out. The microstructure of the foam samples was analysed as well by using the scanning electron microscope. The results of the experiment showed that the density of the foam samples progressively increased from 19.20 (S_0) – 26.45 (S_5) as the quantity of the filler increased. The indentation hardness result also showed an increase on addition of the filler. The foam's loading ability also increased on incorporation of the filler but S3 showed remarkable recovery after compression. The tensile strength and elongation at break of the foam decreased on addition of the filler. The morphological analysis ascertained the effect of the progressive introduction of the filler on the surface morphology of the foam. The flammability of the foam was found to decrease as the filler load increased. Since these fillers are of organic origin, readily available, cheap and eco-friendly, they provide a means of making biodegradable foam, and reducing the flammability of foam. Thus, reducing environmental pollution whilst enhancing the mechanical property of foam.

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1. Introduction

Polyurethane is any type of polymer containing urethane linkage. The urethane linkage is –NH-CO-O-. The polyurethane usually referred to as PU, is actually another name for the family of chemicals known as urethane polymers, which are composed of two principal raw materials: isocyanates and polyols, brought together with catalysts and a variety of additives [1]. Polyurethane might be hard like fiberglass, squishy like upholstery foam, protective like varnish, bouncy like rubber wheels or sticky like glue, I.e. it can exist as both rigid and flexible foams, since polyurethanes come in so many forms and can have a wide variety of properties, it therefore is used in many different applications [2]. The use of flexible polyurethane foams for cushions, furnishes, furniture and automobiles has displaced rubber foam in applications because of improved strength, lower density and easier fabrication [3].

The major raw materials used for the production of flexible polyurethane foam include Polymer Polyol, Isocyanate, Blowing agents, Catalysts, Surfactants and Additives such as Fillers which can be organic or inorganic [4]. These raw materials are mostly liquid reagents and chemicals obtained from petrochemicals and agro-products and hence the prices of flexible polyurethane foams are becoming increasingly high due to high cost of raw materials [5].

Fillers are used in the polymer matrices for various purposes such as to reduce cost, to reduce resin curing shrinkage, to control resin viscosity and to improve the stiffness and other properties of the matrix. Most commonly used filler materials in polymer matrix are calcium carbonate, alumina, and silicon carbide [6]. However, there is need for sources for renewable materials, which can serve as alternate fillers to the conventional mineral calcium carbonate ($CaCO_3$) which holds the largest market volume in the plastic section and is mainly imported. This gives room for developing countries to source out their own raw materials, reduce importation and enable the use of materials that are eco-friendly, non-toxic and biodegradable [7]. Natural fillers such as goat femur [8], egg shell [9], mixed coconut husk/corn cob [10] and rice husk/corn cob [11]. Snail shell among others has been considered as suitable substituent for inorganic fillers in polyurethane foams as a result of their various advantages [12].

Thus, this article reports an investigation on the mechanical properties, flammability and morphology of flexible polyether polyurethane foam filled with periwinkle and African star seed shell powder.

2. Materials and Methods

The quality of the end product in any manufacture process is dependent on the quality of the starting materials and the fact that manufacturing instructions are obeyed to the latter. Hence, it is pertinent to note that the raw materials used in the production of this work were sourced from Vitafoam Nigeria Plc. Bank Junction, Oba Akaran Industrial layout Ikeja, Lagos State, a company known to produce quality polyurethane materials such as pillows, mattress etc. since 1991.

Collection and preparation of periwinkle shell.

The periwinkle shells were obtained from Owerri, Imo state Nigeria. The periwinkle shells were first filtered by hand picking to remove other unwanted substances, washed thoroughly and allowed to dry for 96 hours. They were crushed to fine powder using an electric grinding machine. The crushed shells were further sieved using a muslin cloth with very small pores to obtain fine powder which had a particle size of 50 μm. It was stored in polythene bags.

Preparation of African star apple seed shell

The African apple star seed was sourced from Agulu, Anambra State, Nigeria. The seeds were then left to dry for 72 hours, so that the shell would come off easily from the seed-coat, the dry seeds were broken using a hammer and the shells were hand-picked, washed and dried for 48 hours and then crushed to fine powder using an electric grinding machine. After the crushing, the powder was sieved again to obtain smooth, fine textured powder with particle size of 50µm. it was stored in a polythene bag prior to the time of usage.

The periwinkle and African star seed shells were mixed in the ratio 50:50 and stored for production.

Foam formulation

The choice of the weights of raw materials to be used in foam production is not made arbitrarily. It is chosen on the basis of formulation. If 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 [10].

Foam production

500g of the polymer polyol was poured into an empty clean plastic jug, 400g of each of the fillers; periwinkle shell and African star apple seed shell were weighed and mixed thoroughly to obtain a homogenous mixture

Table 1. Foam Formulation.

Raw materials	PPH	S_0 (g)	S_1 (g)	S_2 (g)	S_3 (g)	S_4 (g)	S_5 (g)
Polyol	100	500	500	500	500	500	500
TDI(index:1.08)	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 catalyst	0.139	1	1	1	1	1	1
Filler(Periwinkle and African star apple seed shell)	---	0	50	100	125	200	250

Note: Pph = Part per hundred; S = sample; g = gram

containing 50% of each of the filler. 0g, 50g, 100g, 150g, 200g and 250g (0%, 10%, 20%, 30%, 40% and 50%) of the mixed fillers were weighed out respectively by using an electrical weighing balance of Model D-72336, Made in China. The quantities of the other raw materials were kept constant but the quantities of the fillers were varied according to the above percentage in each sample.

28g of water, 2g of amine, 5g of silicon oil and 1g of tin catalyst were carefully weighed out and poured into the plastic jug containing the polyol and to this mixture, the appropriate quantity of the mixed fillers for each sample was added. The mixture was stirred vigorously for 2 minutes, 325g of TDI was measured out in a separate beaker and added to the homogenous mixture and further stirring was done.

The mixture was transferred into a metal mould lined with brown paper; the brown paper was used to avoid the sticking of the foam to the body of the mould. The process was repeated for the different weights of the 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 and testing. This was also repeated for the different foam samples based on their gram weight.

Characterization of the foam samples

The following mechanical properties of the foam samples were determined using standard methods: density, while tensile strength, elongation at break, compression strength and hardness test were measured according to the ASTM-D standard specifications [13]. The surface morphology of the sample was tested via Scanning electron microscope.

3. Results and Discussion

The results of the mechanical properties of the foam samples were observed. The percentage (%) compositions of periwinkle and African Star seed shells powder in each sample varying from 0% in Sample S_1 to 50% in sample S_5 at 10% interval. The foam properties studied and their corresponding results are as shown below.

Cream time, Rise time and Curing time

The result of the cream time, rise time and curing time recorded during production of polyurethane foam is shown in the Table 2.

Table 1. Observation during the experiment

Experiment	Cream time (secs)	Rise time (secs)	Curing time (secs)	Height (cm)
S_0	3	80	24	24.20
S_1	5	90	24	24.60
S_2	5	98	24	24.80
S_3	6	118	24	25.20
S_4	8	122	24	24.80
S_5	8	122	24	24.60

From Table 2, it was discovered that as the quantity of filler load increased, the time taken for the foam reaction to commence which is the cream time also increased. In the same vein, the time taken for the foam to attain maximum height also increased with increase in filler load. The height was regained as the filler load increased. This implies that there is a direct relationship between the cream time, rise time and curing time of polyurethane foams and the filler load. This exponential relationship in this study is in complete agreement with the work of another researcher titled “effect of animal waste as filler in flexible polyurethane foam” [8].

Density

Foam density is a specific measurement of how much weight in kilograms polyurethane foam can handle per cubic meter.

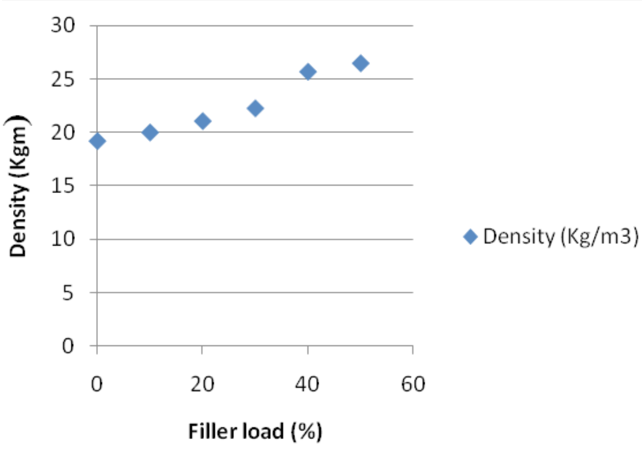


Figure 1. Effect of filler load on Density.

It was observed from the results that as the filler load increased the density of the foam samples also increased. A density of 22.00 was set for the calculations involved in this foam formulation. This could be attributed to the nature and high content of the fillers which would fill up more voids, thus increasing the foam's density [14]. The filled voids tend to make the polyurethane foam structures more compact hence increasing its weight and making it denser.

In flexible polyurethane foams, the fillers promote an increase in density and hence increase the shelf life and cushioning effect of the foam [3]. The same trend was observed in the following works [11,14-16].

Compression Set

The compression set test results were obtained by finding the difference between the initial weight of the foam sample before and after compression.

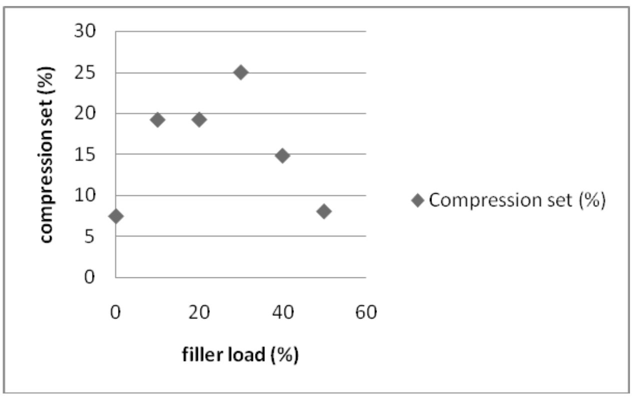


Figure 2. Effect of filler loading on compression set of flexible polyether foam

It was observed that as the filler load increased, the compression strength of the foam also increased as seen in Figure 2. However there was a sharp decline in compression strength in samples 5 and 6. This showed that the presence of the filler improved the ability of the foam to return to its normal shape after compression. This could be due to the reinforcing property of the filler as a result of its cellulosic nature. The sharp decline in compression strength of samples 5 and 6 could be attributed to the fact that though sample 5 and 6 with filler load 40% and 50% respectively have the highest density, they still had the lowest ability to return to their original size after compression. This implies that there is no direct relationship between compression and the density of a foam sample [8]. From the result, it can also be seen that the highest value for the compression set test (25%) was obtained from sample 4 with a filler load of 30%. This shows that the maximum yield for this combination of filler (periwinkle and African star seed shell) for compression set test is obtained when the sample contains 30% of the filler. These findings are in agreement with the works of some other researchers [8,11].

Tensile Strength

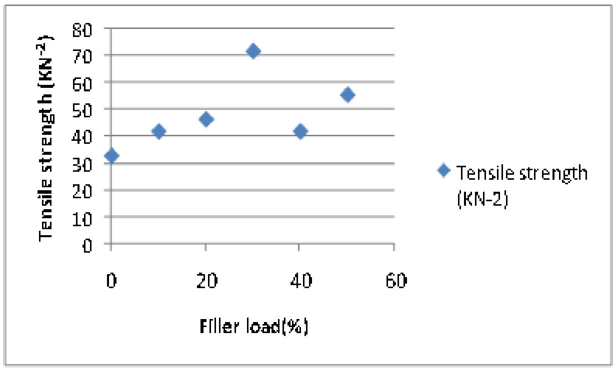


Figure 3. Effect of filler loading on tensile strength of flexible polyether foam

It was seen that the tensile strength of the periwinkle and African star seed shell filled polyurethane foam increased as the filler load increased, but there was a sharp decline in tensile strength in sample 4 and an increase in sample 5 as shown in Figure 3. The increase in tensile strength can be attributed to good interaction between filler- polymer matrix. It could also be due to nature and small particle size of the filler thereby by providing a good surface area to resist stress. Hence, this proves that the filler is suitable and can be applied in the production of polyurethane foams since it improved the mechanical properties of the foam [17]. However, the sharp decline in the tensile strength of the foam sample 4, could be attributed to poor filler interaction between the filler and polyurethane matrix. It could also be attributed to the voids created by the fillers in the matrix phase [18]. From the results, it can also be inferred that the optimum yield for the tensile strength test is obtained when the sample contains 30% of the filler load, as sample 4 has the highest value (71.7) from the experiment. The rise and fall in tensile strength in the property of this work [10].

Elongation at Break

The results were obtained from a power accurate grip Indentiometer, which recorded elongation as the distance moved by the sample before break.

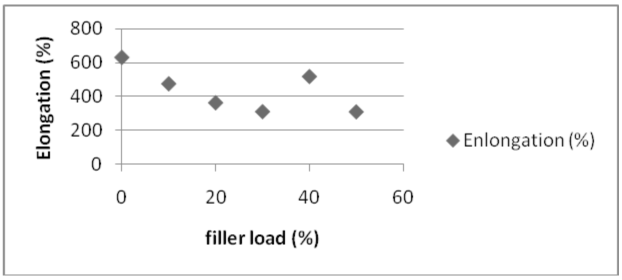


Figure 4. Effect of filler load on Elongation at break

From Figure 4, it is shown that as the filler load increased, there was a corresponding decrease in the elongation at break of the polyurethane foam samples. This inverse relation can be attributed to the nature of the filler (cellulosic) which makes the foam sample tend to elongate more with a lesser filler load than a higher one [19]. Comparing the control (sample1) with the other samples, it is observed from the results that there is an overall decrease in the % elongation at break. However, in sample 5 which has a filler load of 40%, the highest value is obtained (518.55) which although lesser than the control (indicating that there is a decrease) is still much higher in value than the sample containing 10%, 20%, 30% and 50% filler load. Hence it can be inferred from the experiment that the maximum load carrying capacity is obtained when the sample contains 40% of the filler. This trend of results has also been observed in works [16,20].

Flammability Test

The flammability of a polyurethane foam sample is dependent on the amount of oxygen available and the nature of the filler incorporated into the foam [16].

It is observed that as the filler load increased, the flammability of the foam sample decreased, as seen in Figure 5. This can be attributed to the fact that as the filler load increases, it reduces the size of the cell window in the foam structure, hereby hindering the easy passage of oxygen through the foam structure and thus, reducing the flammability of the foam. Since polyurethane products especially foams play a vital and indispensable role in everyday life because of their wide range of application in automobile, household, refrigerators, insulators, reducing the fire risk of such products is very essential [21]. Hence, the addition of biodegradable fillers such as periwinkle and African star seed shells are highly advantageous as they help to reduce the flammability of polyurethane foam. This trend of results was also obtained in the work [22].

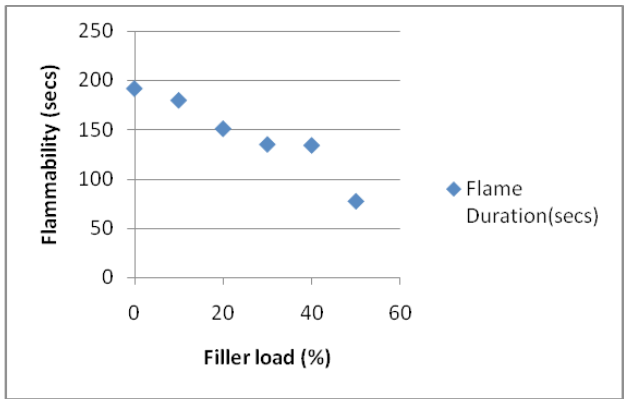


Figure 5. Effect of filler load on flammability.

Hardness test (Indentation hardness)

The firmness of the polyurethane foam is as a result of a measure of the physical property called the indentation force deflection (IFD).

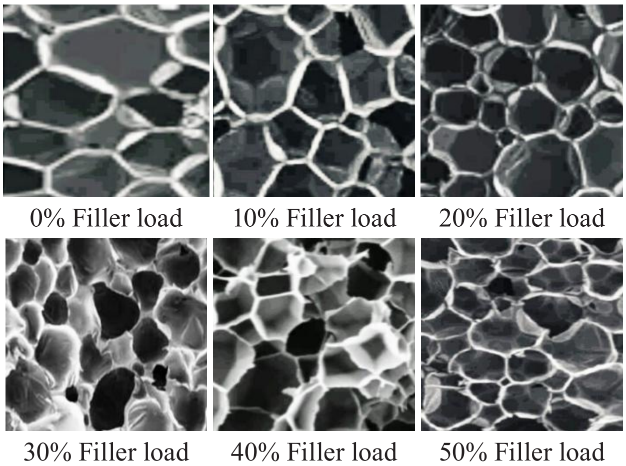
Table 3. Results for the indentation force deflection (IFD) or hardness test.

S/N	samples	Filler load(%)	25%	40%	60%
1	S_1	0	106.20	129.00	269.20
2	S_2	10	119.50	142.00	270.00
3	S_3	20	120.00	149.00	280.20
4	S_4	30	135.00	202.00	300.00
5	S_5	40	139.00	210.00	320.00
6	S_6	50	142.00	240.00	360.00

From Table 3, it was observed that as the filler load increased, the hardness of the foam also increased. This implies that increase in filler load has a positive impact on the hardness of the polyurethane foam material. The fillers exhibited hard effect on the foam and would carry a lot of weight over a long period of time without collapsing immediately. These filler materials are lingo-cellulosic materials and have been proven to be very strong and durable [18]. Furthermore, 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 interaction 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 trend of results was also obtained in works [10,23].

Surface morphology

The surface morphology of the polyurethane foam which is a function of the filler-foam interaction was observed using a scanning electron microscope.



References

[1] Mills, J.N. (2007): Polyurethane Foam: Processing and Microstructure; Polymer Foams handbook, pp.19- 37.

[2] Oertel, G. and Abele, L. (1985): Polyurethane Handbook: Chemistry, Raw Materials, Processing, Application, Properties; Hanser Publishers, Distributed in USA by Scientific and Technical Books, Macmillan. Pp.1- 25.

[3] Billmeyer, F.W. (2005): Textbook of Polymer Science, 3rd edition, John wiley and sons, Toronto, p.447.

[4] Udhayasankar, R., Karthikeyan, B. (2015): A Review on Coconut Shell Reinforced Composites; *International Journal of Chemistry And Technology Research,*8(11):624-637.

[5] Onuegbu T.U., Obi J.N., Ejikeme C. (2010): The Effects of Palm kernel Powder as Filler in Flexible Polyether Foam; *Journal of Physical Sciences, Ghana,*2(4): 126-129.

[6] Babalola, F.U. and Dominic, E.(2012): A Comparative Ananlysis of the Effects of Calcium Carbonate and Dolomite as Fillers in Polyurethane Foam Product. *Journal of Innovations in Science and Engineering,*34-40.

[7] Raju G.U., Kumarappa S., Gaitonde V.N (2012): Mechanical and Physical Characterization of Agricultural Waste Reinforced Polymer Composites; *Journal of Material and Environmental Sciences*,3(5): 907- 916.

[8] Onuegbu T.U., Obianuko, N., Mbachu, G.U., and Iloamaeke, I.M., (2010), Effect of Animal Waste (Goat Femur) As Filler in Flexible Polyether Foam, *Journal of Basic Physical Research,* Vol 1(1):5-8.

[9] Toro P, Quijad R, Yazdani- Pedram M, and Arias J.L. (2007), Eggshell, a new bio-filler for polypropylene composites, Materials Letters, Vol. 61(22): 4347-4350.

[10] Chris-Okafor, P.U., Arinze, R. U., Nwokoye, J.N. and Umar, E. U. (2017); Effects of Coconut Husk and Corn Cob as Fillers in Flexible Polyurethane Foam. *American Journal of Polymer Technology* 3(4):64-69.

[11] Chris-Okafor, P.U., Arinze, R.U., Ekpunobi, U.E., and Anugwom, M.C. (2017); Effects of Mixed Rice Husk and Corn Cob as Fillers on Some Properties of Flexible Polyether Foam. *Global Journal of Science Frontier Research:* B Chemistry 17(2): 30-37.

[12] Manjunatha, G.H. andSabeel, A.K. (2017) : Experimental Characterization of Coconut Shell Particle Reinforced Epoxy Composites; *Journal of Materials and Environmental Sciences,*8(5): 1661- 1667.

[13] Nigerian Industrial Standard 53(1979): Method of Polyurethane Production.

[14] Saliba, C.C., Oréfice, R.L., Carneiro, J.R.G., Duarte, A.K, Schneider, W.T. and Fernandes, M.R.F. (2005); Effect of the incorporation of a novel natural inorganic short fiber on the properties of polyurethane composites. *Polymer Test.;* 24(7): 819-824.

[15] Samuel, E.A. , Ganiyu, K. L., Tinuade J. A. , Semiu A. K., (2015); Model Predictive Performance of Filled Flexible Polyurethane Foam. *American Journal of Polymer Science;* 5(1): 1-9.

[16] Onuegbu, T.U., Ugwu, I.E., Ogunfeyitimi, O. (2013): Physico- mechanical Properties of Flexible Polyether foam: Comparative Effects Of Fillers; *Chemistry And Materials Research,* 3:46-52.

[17] Dalen, M.B., Ibrahim, A.Q., Adamu, H.M., and Nurudeen, A.A., (2014), Effect of calcium carbonate and kaolin filler loading on curing rates of polyurethane foams, International research *Journal of pure and applied chemistry;* Vol. 4(6):691-694.

[18] Ruijun, G., Mohim, M.S. and Samir, K.K. (2013): A Feasible Study of Polyurethane Composites Foam with Added Hardwood Pulp, Industrial Crops and Products; *Checkmate Press, New York,*42(2):273-279.

[19] Javni, I., Song, K., Lin, J., Petrovic, Z. (2011): Struc-

ture and Properties of Flexible Polyurethane Foams with Nano- and Micro- Fillers;*Journal of Cellular Plastics,*47(4):357-372.

[20] Agarry, S.E., Latinwo, G.K.,Afolabi,T.J., Kareen, A.S. (2015): Model Predictive Performance of Filled Flexible Polyurethane Foam; *American Journal of Polymer Science,* 5(1):1-9.

[21] Gharenbagh, A. and Ahmadi, Z. (2012): Polyurethane Flexible Foam Fire Behaviour, INTECH Open Access Publisher, Pp.1-10.

[22] Sant'Anna, S.S., Souza, D.A, Carvalho, C.F. and Yoshida, M.I. (2008): Morphological and Thermal Analyses of Flexible Polyurethane Foams Containing Commercial Calcium Carbonate; Ecletica, 33(2): 55-60.

[23] Agham, R.D. (2013): Performance of Coconut Husk Mixed Polyurethane Foam as an Alternative Insulating Material; *International Journal of Engineering and Innovative Technology,* 9(2): 100-104.