Ekebafe et al. Nigerian Journal of Materials Science and Engineering. Vol. 10(2):109 - 118 (2020)

Preparation and Assessment of Biodegradable Polyurethane Foams from Sugar Bagasse

*Ekebafe Lawrence, [1]Akpa F. A. Omolade, and [2]Olugbemide David A.

Industrial Chemistry Research Unit, Department of Chemistry,
University of Lagos, Akoka, Lagos, Nigeria
[1]Department of Science Laboratory Technology, Auchi Polytechnic, Auchi, Nigeria
[2]Department of Basic Sciences, Auchi Polytechnic, Auchi, Nigeria
Corresponding Author: lawekebafe@gmail.com

Abstract

This study is focus on producing polyurethane foams that would have same or better properties compared to the conventional and still be able to undergo biodegradation upon disposal. Microfibers were isolated sequentially using nitric acid, sodium hydroxide and hypochlorite bleaching under different extraction conditions. The cellulose microfibers extracted were characterized for their composition, structure, and properties using prescribed procedures. Polyurethane foams were made from the biopolyol containing as much as 25 ml liquid cellulose. Samples of the foams were characterized based on morphology, density, modulus and compression test using standard methods. The biodegradation integrity of the foams was assessed in this study using short-term accelerated laboratory experiments including microbial analysis, and soil burial experiments. The results were compared with the conventional polyurethane foams. The results showed that the cellulose based polyurethane foam sample with 10-15 ml liquid Fibers have about 75 ± 0.50% cellulose for the acid treated and 79 ± 1.05% for the alkaline treated. The results revealed that the optimum conditions to remove most of non-cellulosic materials from the sugar bagasse were 1M HNO_3, 7M NaOH, 3 h and 100°C for both the alkaline and the acid procedure, and 10 ml/L NaClO, 45 minutes and 95^{oC} for bleaching treatment. The results showed that the studied foams biodegraded under anaerobic conditions. Differences in the properties of the foams produced in comparison with the conventional foams were not significant. However, significant difference was observed in the biodegradation of the foams produced with the conventional foams.

Keywords: Biodegradation, Cellulose, Foam, Polyurethane, Sugar Bagasse

1.0. INTRODUCTION

Environmental concerns are driving the effort to find new methods for producing many common items using renewable and biodegradable resources as opposed to the synthetic and often petrol-based materials commonly used (Furtwengler and Averous, 2018). The major downfall of the majority of today's foams is that they are typically produced from non-renewable, non-recyclable, and non-biodegradable materials.

Issues facing the polymer foam industry are that of waste disposal, recyclability, flammability and the effect of blowing agents on the environment. The restrictions on the use of CFC's (chloro-fluoro-carbons) have become very important in making polymer foams more environmentally friendly. Advances in biodegradable foam materials are also helping to improve the recyclability and waste disposal.

The large abundance of natural cellulose from wood pulp, along with its excellent mechanical and thermal properties and biodegradability, make it an attractive alternative to such environmentally harmful materials (Zhang *et al.* 2017).

Biomass consists mainly of cellulose (30–35%), hemicellulose (15–35%) and lignin (20–35%), which contain two or more hydroxyl groups in the molecule and form a stable three-dimensional network structure (Lu *et al.* 2016, Rapra, 2018;).

The conventional polyurethane foam is non-biodegradable, thus constituting much environmental pollution upon disposal. The problem so created is what these research endeavors to address by incorporating liquefied bio-polyols from sugar bagasse to the formulation to stimulate biodegradation upon disposal after usage. Sugar bagasse must therefore be converted into a liquid form by liquefaction process (Zhang *et al.* 2017, Rapra, 2018, Ashida, 2007, Kržan *et al.* 2005; Zhang *et al.* 2007; Kunaver *et al.* 2010).

As a cheap resource, sugar bagasse offers great potential as an alternative to wood. Since uncared sugar bagasse also cause environmental problems. Increased research during the recent years has considerably contributed to the understanding of sugar bagasse as well as to improved processing technologies for broader uses. Sugar bagasse is presently super abundant in Nigeria with so much untapped potentials.

The use of natural fibers as reinforcements in polyurethane polymer matrices has been previously reported (Ge *et al* 2018). Also, sisal fibers have been used to reinforce polypropylene compounds as an ecological alternative to the automobile industry (Ge *et.al*. 2018). The use of short organic fibers as reinforcement for polyamide thermoplastic polymer matrices was also studied (Tan *et al.* 2011). Regarding the polyurethane polymeric matrix, studies conducted by Gama *et al.* reported the incorporation synthetic materials derived from renewable sources into other components from agriculture, for improving the properties of the resulting composite material and – at the same time – for increasing the biodegradability of the matrix, a desirable characteristic in many polymers (Gama, *et al.* 2014, Gama, *et al.* 2017). Other studies have focused on the preparation and determination of the physical properties of polyurethanes derived from molasses (Yao *et.al.* 1995, Gandini *et al.* 2010).

Polyol-based copolymer poly(ethylene glycol) (PEG)/glycerol was synthesized (D'Souza and Yan 2013) and the liquefied biomass was characterized by a high degree of modified bark components. The polyols were analyzed for their composition, structural features and performance. Polyurethane films had also been obtained by copolymerization between liquefied wood and polymeric methylene diphenyl diisocyanate (PMDI) (Kurimoto et al. 2001). In their research, they carried out a liquefaction process for three types of softwood and three hardwood species in solvents such as glycerol and poly(ethylene glycol) and assessed the influence of the wood species on the mechanical properties of the obtained materials.

Japanese cherry blossom (Sakura) had also been liquefied to obtained polyurethane resins (Mori, 2015) liquefied. He also carried out modifications of the biodegradable resins obtained using tetraethoxysilane and found that the thermal stability of the obtained resins based on liquefied wood was better than for commercial polyurethanes. In addition, the introduction of an inorganic Si network was at the molecular level and positively influenced the mechanical properties.

The large abundance of natural cellulose from wood pulp, along with its excellent mechanical, thermal properties and biodegradability, made it an attractive alternative to such environmentally harmful materials. The impact of non-biodegradable foams on the environment is increasing and there is urgent need to provide sustainable solution through the production of foams that will biodegrade in the environment using a technologically feasible and cost effective method. Solving an evolving environmental problem through sustainable cost effective technology for the production of biodegradable polyurethane foams that would conveniently replace the conventional non-biodegradable ones hence this study.

2.0 MATERIALS AND METHODS

2.1 Materials

Sugar Baggase and molasses were sourced locally from Aviele in Etsako west LGA, Edo, Nigeria. Chemicals used in the study include: toluene diisocyanate (TDI), Diethylamine, Polyethylene glycol PEG-300, Sodium hydroxide, dibutyltin dilaurate, Methanol, Ethanol, Acetic acid, Nitric acid were products of Sigma Aldrich, Germany and were used as supplied.

2.2 Isolation of the Cellulose Microfibers from the Sugar Bagasse

500 g milled dried sugar bagasse powder was placed on a round bottom flask and a varying concentration of the nitric acid (1-10M) were added and in a separate setup 1-10M NaOH solution was added to eliminate traces of lignin with heating at 100°C for 0.5-3 hr. The resulting material was then treated with an excess of sodium hypochlorite, NaClO (1-10mL/L) for 0.5-4 hr at 25-100°C temperature to eliminate pectins, proteins, hemicelluloses, and some mineral components (Alemdar and Sain, 2008).

2.3 Preparation of polyurethane foams

The methodology for the preparation of polyurethane foams is similar to that mentioned elsewhere (Gao *et al.* 2010, Vega-Baudrit *et al.* 2011). Table 1 shows the amounts used to synthesize polyurethane foams (PUF) and the nomenclature used to identify each mixture.

Characterization: The ash content was analyzed according to ASTM D 1102-56, at a temperature of 500 °C, and moisture content determined in a moisture balance. Solubility in cold and hot water was determined according to ASTM D 1110-56, and solubility in organic solvents was analyzed according to ASTM D1107-56, modified by

Table 1: Quantities used for the preparation of the biodegradable polyurethane foams (CPUF) from sugar cane molasses and bagasse.

Sample	Fiber(ml)	Mix(g)	Fiber(g)	TDI(g)
1	0	25.97	-	30.00
2	5	13.00	1	15.16
3	10	13.08	2.0	15.02
4	15	13.11	5.0	15.70
5	20	13.05	10.0	15.05
6	25	13.85	15.0	14.50

*Catalyts: 0.08 g

replacing benzene with cyclohexane. The hemicellulose content was determined according to ASTM D 1104-56, the lignin content was analyzed according to ASTM D1106-56 and, finally, the determination of the cellulose yield was carried out according to ASTM D 1103-60. The bulk density was determined by the tapping method. The tap density was measured from the final volume of the tapped sample. Characterization of molasses in terms of their pH, conductivity, moisture content, ash content, protein content, minerals and heavy metals was carried out adopting methods described elsewhere (Ge *et al.,* 2003, Hu *et al.* 2012). Fourier transform infrared (FTIR) spectroscopy was carried out to quantitatively identify the constituents of the various extracted fibers. X-ray diffractions were conducted over a 5-45° 2θ range using X-ray radiation. The Diffractometer was used to identify the crystalline peaks. The morphology of the various extracted fibres samples was investigated by using the scanning electron microscopy (SEM). To measure bulk density, the foams were cut into 2.00 cm cubes, mass being determined with an analytical balance. The compression stress at 10% strain was measured by the method described by ASTM-D695M. CPUFs surface morphology was examined by SEM.

2.4 Biodegradation Experiments

The ability to undergo biodegradation was assessed using method designed for testing biodegradation of plastics under simulated environmental conditions (ASTM, 1993, EN ISO 846, 1997).

A series of batch cultures was set up to screen polyurethane (PU) biodegradability and to monitored degradation of PU. The weighed biodegradable polyurethane (BPU) was enclosed in a lidless plastic bottle of known weight, and the bottle buried in the waste dump, with a slope of 45^o. The bottle was opened and turned downwards, buried in the soil. The change in weight was recorded at 2 weeks interval.

The degradation percentage (DP) of the CPUF was calculated as described in ASTM, (1993), and EN ISO 846, (1997). The microbiological degradation and deterioration test was carried out following already described method (Gu and Gu, 2005 and Zhao *et al.* (2012).

3.0 RESULTS AND DISCUSSION

The results of the processing and characterization of the sugar cane bagasse, acid and alkaline extracted cellulose microfiber, revealed that the chemical treatments removed the majority of the lignin and other non-cellulosics in order to release the fibers as shown in Table 2. The treatments with the acid and alkaline with sodium hypochlorite promoted a decrease in the diameter as shown in the morphology micrographs which did not exhibit significant differences with increment in acid/alkaline concentration or the exposure time. The results details have been reported in Akpa, *et al.* (2016) and Ekebafe, *et al.* (2017).

The FTIR results confirmed the isolation of the cellulose microfiber from bagasse and the production of the biodegradable polyurethane foams, as reported in in Akpa, *et al.* (2016) and Ekebafe, *et al.* (2017).

The morphology of different CMF materials is shown in Figure 1. The commercial powders were formed by particles of an irregular shape. The XRD result is as presented in Figure 2. Wide angle XRD was used to determine the effect of the cellulose on the structural changes of the polyurethane foams. Figure 3 shows the XRD patterns of cellulose, control, and the cellulose based polyurethane foams from 15–40, because all the characteristic peaks were in this range. Cellulose had three peaks at 20, 25, 30 corresponding to the diffractions of amorphous cellulose and crystalline cellulose respectively.

3.1 Preparation of the Cellulose based Biodegradability Polyurethane Foams

The isocyanate group (-N=C=O) is an unsaturated and highly reactive group containing two cumulative double bonds. It can react with both electron acceptor and electron donor functional groups (Bhavesh, *et al.* 2018). The main groups reacting with isocyanate are hydroxyl, carboxyl and amino groups. Isocyanate undergoes two types of reactions in the foaming process - primary and secondary as shown in scheme I and II (Szycher, 2006).

Table 2a: Physico-chemical properties of the samples

SAMPLE CODES	LIGNIN (%)	CELLULOSE (%)	HEMI-CELLULOSE (%)	CHO (%)	FAT (%)	MOISTURE CONTENT (%)	ASH CONTENT (%)
A	17.982	57.527	24.490	NA	N.A	7.67	1.908
A	17.626	57.551	24.823	NA	N.A	7.67	1.990
B	NIL	100	NIL	NA	N.A	4.09	3.671
B	NIL	100	NIL	N.A	N.A	4.09	3.628
C	7.192	75.401	17.407	N.A	N.A	8.33	0.683
C	7.205	75.432	17.363	N.A	N.A	8.33	0.672
D	2.997	79.371	17.632	N.A	N.A	8.58	0.828
D	3.039	79.274	17.687	N.A	N.A	8.58	0.851
E	N.A	N.A	N.A	83.853	2.500	11.322	0.098
E	N.A	N.A	N.A	83.729	2.523	11.412	0.104

Table 2b: Physico-chemical properties of the samples

SAMPLE CODES	FMĂŐŐÖÅ	Ì Ō (ppm)	ĞÑ (ppm)	Í Ò (ppm)	FÞ (ppm)	ĶŌ (ppm)	FÕ (ppm)	ĬÎ 2 (ppm)	ĨĪÎ İ ĜĦ (%)
A	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
A	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
B	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
B	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
C	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
C	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
D	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ
D	ČĆĖĆĆĆ	ĆĖĈĊĊ	ĆĖĈČĆ	ĆĖĆĈÇ	ĆĖĆĈĊ	ĆĖĆĆE	ČĖĆED	ĈĎĆĆĖĆĆ	ČĖČČĐ
E	ČĈĖĆĊÇ	ĆĖĆĐE	ĆĖĈĈĐ	ĆĖĆĈĊ	ĆĖĆĈĊ	ĆĖĆĈĆ	ČĖĆĐĆ	ĈĎĈĆĖĆĆ	ČĖĊČĎ
E	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ	Í ĖĖ

A=Sugar Bagasse; B=Commercial Cellulose; C=Acid-Extracted Cellulose,;D = Alkaline Extracted Cellulose; E= Molasses; N.A (Not Applicable)

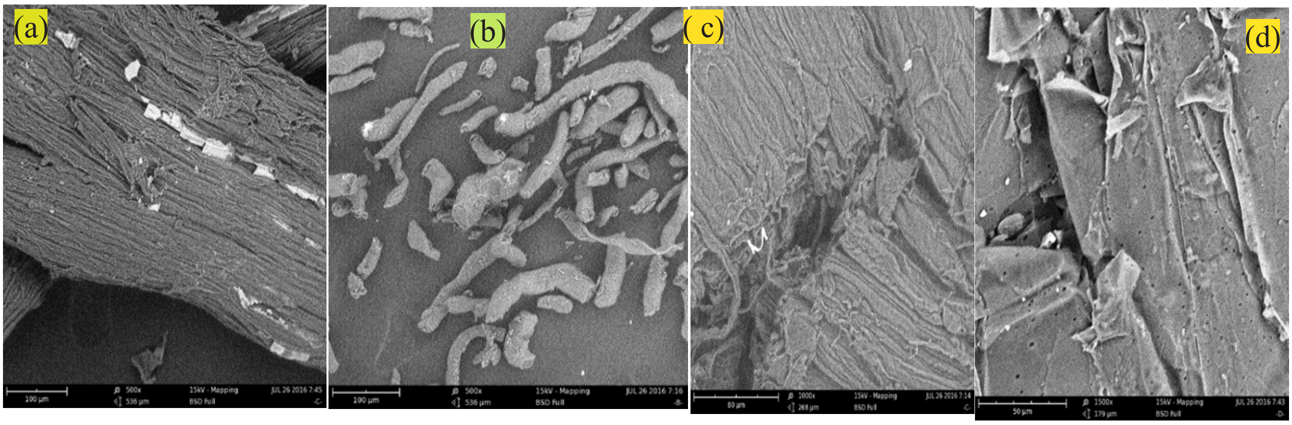

Figure 1: CMF micro-photo plates obtained from (a) bagasse, (b) commercial cellulose (c) acid extracted cellulose, (d) alkaline extracted cellulose.

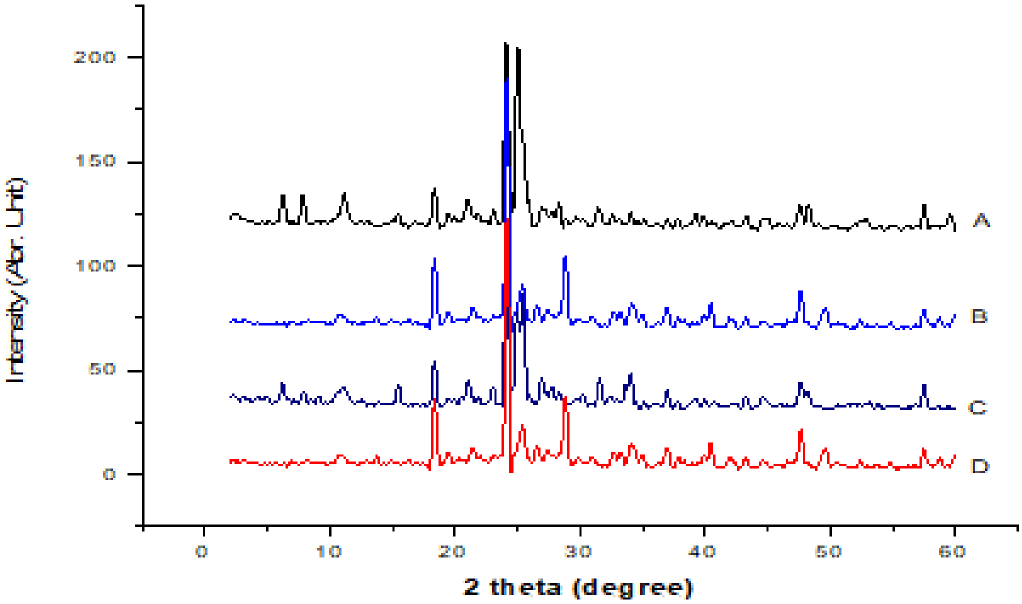

Figure 2: XRD for the various cellulose samples.

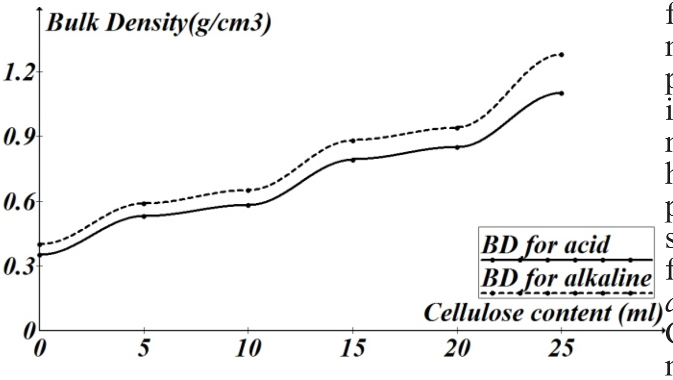

Figure 3: Effect of cellulse contet of foam on its bulk density

Scheme 1: `

R-NCO + R'-OH → R-NH-CO-O-R' (Urethane)
R-NCO + H_2O → R-NH-CO-OH (Carbamic Acid)
R-NCO-CO-OH → R-NH_2 + CO_2
R-NCO + R-NH_2 → R-NH-CO-NH-R (Urea)

PU foam reaction is a unique process in which polymerization and foam blowing occurs simultaneously. Polymer structure forms rapidly in order to support the fragile foam, but not fast enough to burst the bubbles.

Scheme II:

R-NH-CO-O-O-R' + R-NCO → R-NH-CO-NR-CO-O-R'
(Urethane) (Allophanate)
R-NH-CO-NH-R + R-NCO → R-NH-CO-NR-CO-NH-R
(Urea) (Biuret)

Figure 3 showed that the incorporation of cellulose polyol increased the bulk density of the composite foam significantly (up to 65%) compared to the neat foam. The effect of polyol on density is more pronounced for high dosage of polyol. The increase in foam density might be attributed to cell nucleation and heavier mass of the polyol. It is hypothesized that the presence of fiber led to the process of heterogeneous nucleation, producing smaller homogeneous cell sizes within cellular foam and thus increasing the cell density (Xia, *et al.* 2005, Ema, *et al.* 2006, Ting, *et al.* 2011, Gayathri *et al.* 2013). Surfaces promote nucleation because of wetting. As density was determined by mass over volume, the increase in foam mass was proportional to the fiber content. The bulk density of polyurethane foam is an essential feature, because it allows obtaining more low-weight material, especially if it is to be used as a packing material. The samples with lower cellulose content (5.0 and 10.0ml) had the lowest bulk density. 15.0. 20.0 and 25.0ml showed higher density and lower resistance to mechanical traction.

3.2 Effect of cellulose micro fiber on polyurethane foam compression mechanical properties

The synthesis of polyurethane foam from sugar bagasse indicated that the lignocellulosic materials incorporated as reinforcement provide a polyurethane matrix with a greater ability to withstand compression tests and increase the modulus. Compression analysis, involving the exertion of a certain force on a sample; showed (Figure 4) the compressive stress (σ) and the modulus (E) aimed at observing the trends in deformation under test conditions. When a load is applied on a foam material, it will gradually be compressed, and the stress-strain curve experiences three regions of deformation: linear elasticity under 5% of strain was observed at the initial level of deformation and as the load is increased, the foam plateau and densification occurs (Valášek, *et., al.* 2013).

The initial deformation behavior is linear elastic and as load increased, the cell walls begin to collapse to form the plateau region, until the opposing walls in the cells meet one another to elevate stress rapidly forming the densification region. Under the load, the urethane soft segments stretch, while the urethane hard segments carry the load and release it when the stress is removed. The compressive strength of the composite foam system showed improvement with incorporation of cellulose fiber. The cellulose fiber containing a large number of hydroxyl groups could form hydrogen bonds or other chemical bonds with the matrix, contributing to the foam enhancement.

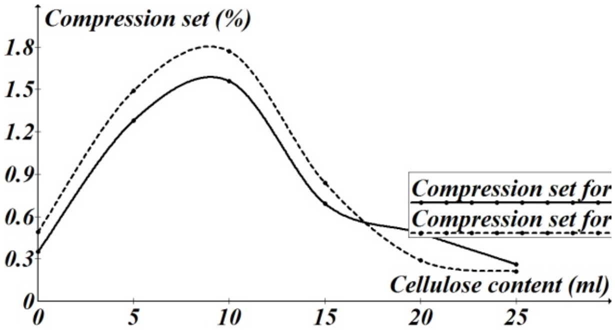

Figure 4: Effect of cellulose content of foam on its compression set

The modulus provides information on the material's ability to withstand an applied load before reaching the breaking point. The results obtained are shown, in Figures 4 and 5.

A maximum value of both compressive stress (σ) and modulus (E) was achieved in samples containing 15 ml cellulose microfiber. At higher microfiber concentrations, the synthesized materials showed lower values of compressive strength and modulus, probably in response to the poor dispersion of fibers in the polymer matrix, generated by a poor interaction between the fibers and the polymer chains.

Similarly, Agrawal, *et al.* (2017) reported that the ends of the microfibers incorporated into polymeric materials act as sources of stress concentrations, which produce cracks in the material, when subjected to stress. Zhang *et al.* (2014) have shown that composite materials contain voids, due to an incomplete impregnation of the fibers with resin, appearing as weak spots in the material, as a result of the high concentration of efforts that promote spreading of cracks. All these indicated that the addition of microfiber can generate a larger percentage decrease in the compression properties.

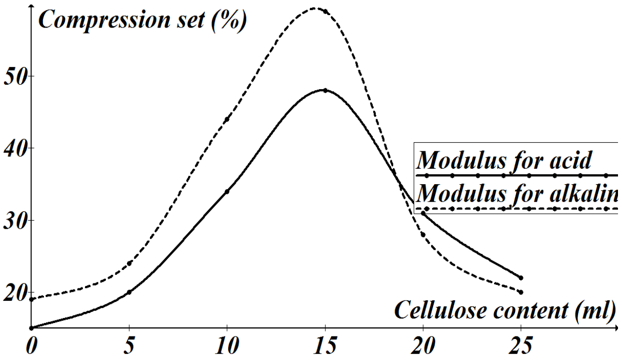

Figure 5: Effect of cellulose content on the modulus of the foam

3.3 Biodegradation Analysis

Literature reports revealed the susceptibility of biodegradable polyurethane foams to fungal and bacterial attack (Allen *et. al.*1999). These studies revealed that biodegradable PU foams are more susceptible to fungal attack than other forms which corroborate the outcome of this study as shown in Tables 3-5. Table 3 showed the mean bacterial and fungal count which is irregular as the foam cellulose content increases. However, there was an initial increase as the cellulose content of the foam to a maximum 15-20 ml cellulose content of the foam, after which there was a decrease in the cumulative weight. Also there was an obvious increase in the cumulative weight loss as degradation continues from first week to the eight week, Table 4.

Cosgrove *et al.* (2007) reported in a recent study of the involvement of soil fungal communities in the biodegradation of bio-polyurethane, the study showed that fungal communities on the surface of the bio-polyurethane were compared to the normal native soil communities using culture-based and molecular techniques. This was also revealed in the result as shown in Table 7-8.

From tensile strength analysis of the foams, it can be concluded that there is deterioration on the surface of the foams during these bioassays.

3.4 Soil Burial Biodegradation Test

The biodegradable polyurethane foams were highly susceptible to degradation in the soils as reported in Table 6 and 7, losing up to 84.2% of its tensile

Table 3: Total bacterial and heterotrophic fungal counts in the foams culture

Foam cellulose content (ml)	Mean aerobic bacterial count (cfu/g)	Mean fungal count (cfu/g)
0	8.9×10^{3}	2.9×10^{3}
5	5.2×10^{3}	7.8×10^{2}
10	1.3×10^{4}	2.4×10^{4}
15	1.9×10^{4}	4.9×10^{4}
20	9.7×10^{3}	1.4×10^{4}
25	4.8×10^{3}	8.1×10^{3}

Table 4: Weight losses of the foams during lab microbial culture biodegradation test

Samples of foams filled with cellulose content (ml)	% Cumulative weight loss After: 2wks	4wks	6wks	8wks
0	1.25	1.10	0.98	0.76
5	18.62	25.83	30.12	36.91
10	39.37	49.05	60.85	66.28
15	45.11	59.02	68.55	70.49
20	49.94	55.68	65.28	72.99
25	30.54	44.98	40.27	43.85

Table 5: Tensile strength (MPa) measurements for the foams during laboratory test

Samples of foams filled with cellulose content (ml)	After: 2wks	4wks	6wks	8wks
0	84.2	79.6	72.8	67.4
5	45.5	40.1	35.9	30.8
10	51.6	40.7	35.2	30.8
15	33.8	30.5	26.4	22.8
20	40.8	40.5	36.2	29.9
25	51.8	48.5	39.1	40.8

Table 6: Weight losses of the foams during lab microbial culture biodegradation test

Foams cellulose content (ml)	% Cumulative weight loss After: 2wks	4wks	6wks	8wks
0	1.19	1.00	0.81	0.51
5	24.61	34.98	42.92	46.76
10	46.98	55.25	69.35	78.42
15	56.91	68.66	79.75	85.66
20	68.85	71.53	78.63	85.89
25	41.09	49.01	49.89	52.58

Table 7: Tensile strength (psi) measurements for the foams during laboratory test

Foams cellulose content (ml)	After: 2wks	4wks	6wks	8wks
0	84.2	79.6	72.8	67.4
5	38.1	35.9	31.3	27.5
10	35.5	31.9	30.0	25.3
15	25.1	24.8	21.5	19.0
20	30.5	30.9	27.9	22.8
25	59.2	60.0	65.5	66.9

strength. The report showed the ability of the soil microcosm in the biodegradation of the foams. Cosgrove *et al.* (2010) study corroborated the outcome of this investigation on soil microcosms that were biostimulated with the PU dispersion agent the result showed specific fungi were enriched in soil following biostimulation; however, few of these fungi colonized the surface of buried PU.

Table 6 showed the cumulative weight loss in test foams during soil burial experiments. Dry weight analysis of 8 weeks treated samples showed an average of 58% weight change. The results indicate statistically significant weight loss in the foams, within the 5% probability level.

Based on the results of the weight loss measurements, and tensile strength measurements, buried in the soil and the control samples, it can be concluded that there was significant biodeterioration of the foams due to potential microbial activity in the field soil under anaerobic conditions during these short-term experiments.

3.5 Morphology of the Cellulose Based Biodegradable Foam

Scanning electron microscope (SEM) images of polyurethane foam samples are shown in Figures

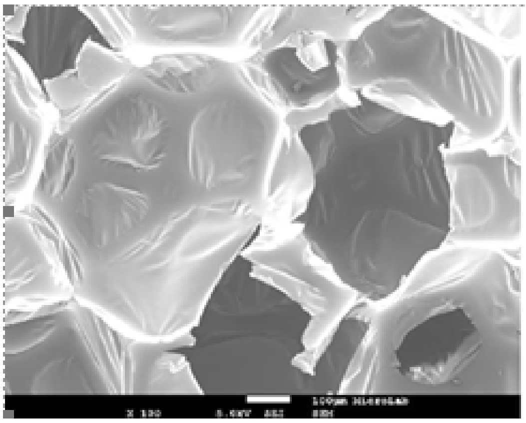

Figure 6a: 10 ml acid Treated cellulose filled foam

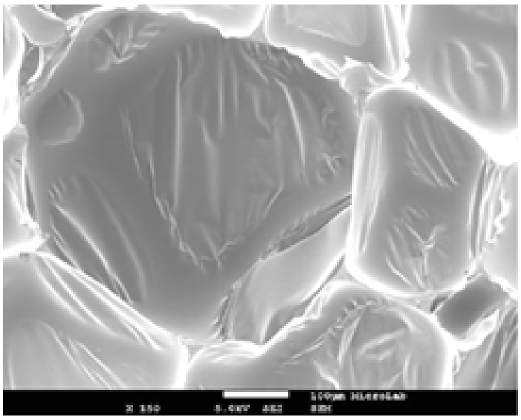

Figure 6b: 15 ml acid cellulose foam

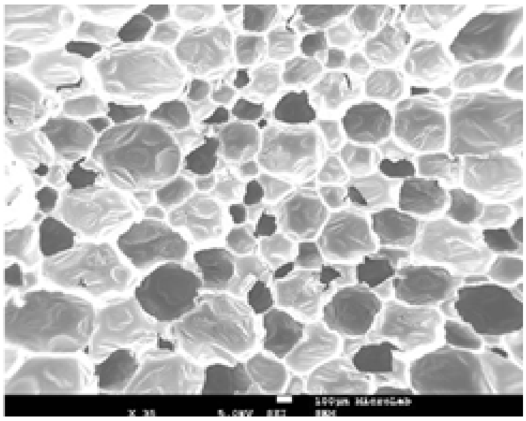

Figure 7a: 10 ml alkaline treated cellulose filled foam

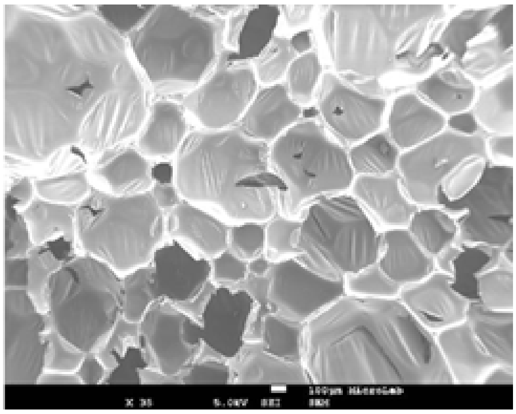

Figure 7b: 15 ml alkaline cellulose foam

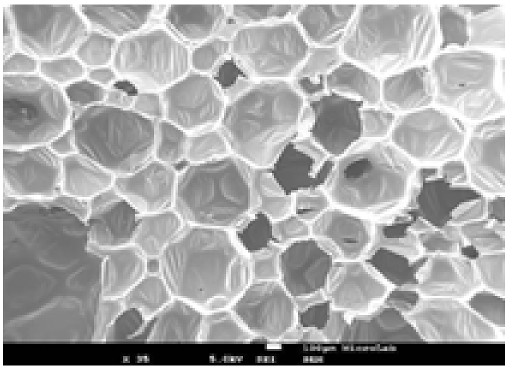

Figure 7c: 20 ml alkaline cellulose foam

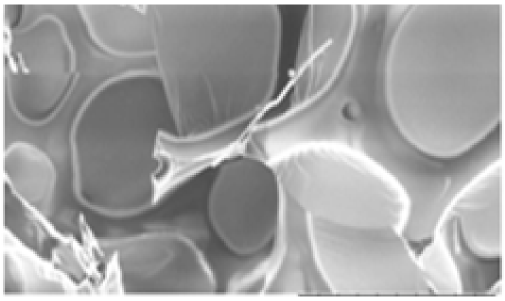

Figure 8: Control (No cellulose) foam

6-8. It can be seen that the 15 ml and 20 ml acid and alkaline cellulose exposed the open cells inherent in the polyurethane foams. The SEM images of the cross section of the different polyurethane foams showed the cells arrangement in the structure. The pore surface was mostly regular and smooth, but with different sizes of the cells (particularly in the 20 ml alkaline cellulose foam) (Figure 7C). The size distribution is approximately 200–500 µm diameter. The ideal structure is composed by homogeneous network of dodecahedra cells, where gas grows inside during the foam formation (Yan *et al.* 2010, Jin *et al.* 2011).

The quality of the foams can be related to their microscopically cell structure. It is well known that the cell size of the rigid foam has an important effect on mechanical properties (Gao *et al.* 2010, Fidan *et al.* 2014,). The excellent honeycomb structure obtained during foam formation made it possible for a considerable amount of still air to be trapped, thus, leading to an increased passive insulation (Yan *et al*. 2010, Jin *et al.* 2011). It can be observed in Figures 7-9 that formulation with the 20 ml alkaline cellulose showed best properties. This formulation has the maximum score in most of the properties evaluated. With the SEM images it is possible to appreciate that its cells are smaller and more homogeneous, with orderly arrangement of the wall surface.

4.0 CONCLUSION

The research showed the ability of the cellulose based polyol used to induce biodegradation in the polyurethane foams prepared. The results revealed that the conventional foams could not biodegrade due to the synthetic nature of the polyol used being from petroleum based chemicals. The results also revealed the better compressibility recovery of the polyurethane foams prepared when compared with the conventional foams. These polyols are an environmentally conscious alternative to the depleting fossil resources. The catalytic transformation of the constituents of lignocellulose to this value-added chemical presents an environmentally attractive and energy efficient process compared to the currently used high temperature and energy consuming gasification or pyrolysis technologies. Based on the results the effect of fiber on density is more pronounced for high dosage of fiber. The compressive strength of the composite foam system showed improvement with incorporation of cellulose fiber when compared with the conventional. It follows therefore, that the 15 ml cellulose microfiber fiber sample showed better compression properties.

REFERENCES

Agrawal, A; Kaur, R; Walia, R.S. (2017): "PU Foam Derived From Renewable Sources. Perspective on Properties Enhancement": An overview. European Polymer Journal 95: 255-274

Akpa, F. A. O.; Olugbemide, D.A.; Ekebafe, L. O. (2016): "Isolation and Characterization of Cellulose Microfiber from Sugar Cane Bagasse for the Production of Biodegradable Polyurethane Foams". Nigerian Journal of Polymer Science and Technology, 11: 109-114

Alemdar, Ayse and Sain, Mohini. (2008): "Isolation and Characterization of Nanofibers from Agricultural Residues-Wheat Straw and Soy Hulls". Bioresource technology. 99:1664-71.

Allen A, Hilliard N, and Howard G. T (1999) "Purification and Characterization of a Soluble Polyurethane Degrading Enzyme from Comamonos acidovorans". International Biodeterioration & Biodegradation 43:37–41

Ashida, K. (2007): "Polyurethane and Related Foams Chemistry and Technology": Taylor & Francis Group: Boca Raton, Fl, USA. 11-27

American Society for Testing and Materials (ASTM) (1993): In: Annual book of ASTM standards. vols. 08.01, D638; 08.03, D5247 D5292; 08.03,D5509-96; 08.03, D5338-98. Philadelphia, Pennsylvania:

Bhavesh Padhiyar, Hitkam Pandya, Jigisha Modi, Sandeep Rai (2018): "Synthesis and Characterization of Polyurethane in Powder Form". International Journal of Chemical Synthesis and Chemical Reactions 1(2): 22-29.

Cosgrove L, McGeechan P. L, Robson G. D, and Handley PS (2007) "Fungal Communities Associated with Degradation of Polyester Polyurethane in Soil". Applied Environmental Microbiology 73:5817–5824

Cosgrove L, McGeechan PL, Handley PS, & Robson GD (2010) "Effect of Biostimulation and Bioaugmentation on Degradation of Polyurethane Buried in Soil". Applied Environmental Microbiology 76:810–819

D'Souza J. Ning YanNing Yan (2013): "Producing Bark-based Polyols through Liquefaction": Effect of Liquefaction Temperature. ACS Sustainable Chemistry & Engineering 1(5):534–540

Ekebafe, L. O.; Olugbemide, D.A.; Akpa, F. A. O (2017): "Biodegradation Studies of Cellulose-Based Polyurethane Foams", Macromolecules, 12 (2): 106-125

Ema, Y., Ikeya, M., Okamoto, M. (2006): "Foam Processing and Cellular Structure of Polylactide-Based Nanocomposites". Polymer, 47: 5350-5359.

EN ISO 846, (1997): "Specifies Methods for Determining the Deterioration of Plastics Due to the Action of Fungi and Bacteria and Soil" Handbook of biodegradable polymers, 57-65

Fidan, M.S.; Alma, M.H. (2014): "Preparation and Characterization of Biodegradable Rigid Polyurethane Foams from the Liquefied Eucalyptus and Pine Wood". Wood Research 59:97-108

Furtwengler P, and Averous L. (2018): "Renewable Polyols for Advanced Polyurethane Foams from Diverse Biomass Resources". Polymer Chemistry 9: 4258

Gama, N.V.; Coasta, L.C.; Amaral, V.Barros-Timmons, A.; Ferreira, A (2017): "Insights into the Physical Properties of Biobased Polyurethane/Expanded Graphite Composite Foams". Composite Science and Technology 138: 24-31

Gama, N.V.; Soares, B; Freire, C.S.R.; Silva, R.; Neto, C.P.; Barros-Timmons, A.; Ferreira, A. (2014): "Rigid Polyurethane Foams Derived From Cork Liquefied at Atmospheric Pressure". Polymer International 64: 250-257

Gandini, A.; Pinto, C.; Coasta, J. J.; Pascoal, N. C. (2010): "Process for the Production of Liquid Poliols of Renewable Origin by the Liquefaction of Agro-Forestry and Agro-Food Biomass". WO Patent 2010020903 A1

Gao, L. L.; Liu, Y. W.; Lei, H.; Peng, H.; Ruan, R. (2010): "Preparation of Semi-Rigid Polyurethane Foam with Liquefied Bamboo Residues". Journal Applied Polymer *Science* 116: 1694-1699

Gayathri, R. Vasanthakumari,. R., Padmanabhan C. (2013): "Sound Absorption, Thermal and Mechanical Behavior of Polyurethane Foam Modified with Nano Silica, Nano Clay and Crumb Rubber Fillers". *International Journal Science Engineering Research,* 4: 301-308

Ge, C. Priyadarshini, L. Cormier, D. Pan, L. Tuber, J. (2018): "A Preliminary Study of Cushion Properties of a 3D Printed Thermoplastic Polyurethane Kelvin Foam". *Packaging Technology Science* 31: 361-368

Ge, J., Shi, X., Cai, M., Wu, R., Wang, M. (2003): "A Novel Biodegradable Antimicrobial Pu Foam from Wattle Tannin". *Journal of Applied Polymer Science*, 90: 2756-2763.

Gu J. G, Gu J. D. (2005): "Methods Currently Used In Testing Microbiological Degradation And Deterioration of a Wide Range of Polymeric Materials with Various Degree of Degradability: A Review". *Journal Polymer Environment*; 13:65.

Hu, S.; Wan, C. Li, Y. (2012): "Production and Characterization of Biopolyols and Polyurethane Foams from Crude Glycerol Based Liquefaction of Soybean Straw". *Bioresource Technology* 103: 227-233

Jin, Y.; Ruan, X.; Cheng, X; Lu, Q. (2011): "Liquefaction of Lignin by Polyethyleneglycol and Glycerol". *Bioresource Technology* 102:

3581-3583

Kržan A, Kunaver M, and Tišler V. (2005): "Wood Liquefaction using Dibasic Organic Acids and Glycols". *Acta Chim Slov* 52:253–258

Kunaver M, Jasiukaityte E, Čuk N, Guthrie J. T. (2010) "Liquefaction of Wood, Synthesis and Characterization of Liquefied Wood Polyester Derivatives". *Journal Applied Polymer Science* 115:1265–1271.

Kurimoto, Y.; Koizumi, A.; Doi, S.; Tamura, Y.; Ono, H. (2001): Wood Species Effects on the Characteristics of Liquefied Wood and the Properties of Polyurethane Films Prepared from the Liquefied Wood. *Biomass and Bioenergy* 21(5): 381-390.

Mori R (2015): "Inorganic–organic Hybrid Biodegradable Polyurethane Resin Derived from Liquefied Sakura Wood". *Wood Science Technology* 49:507–516.

Rapra S. (2014): "Polymer Foams Market Forecast to 2019"; Smither RAPRA: Shabury, UK 09-15

Rapra S. (2018): "High Performance Polymer Foams to 2021" – Market reports: Smither RAPRA: Shabury, UK 15-18

Szycher Michael (2006): "Szycher's Handbook of Polyurethanes". 2nd ed. *CRC Press:* New York, NY, USA 20-38

Tan S. Abraham, T; Ference, D; MacOsko, C.W. (2011): "Rigid Polyurethane Foams from a Soybean Oil-Based Polyol. *Polymer*, 52: 2840-2846

Ting, L., Liang, M., Fuwei, L., Wuzhou, J., Zhaobo, H., and Pengfei, F. (2011): "Preparation, Structure, and Properties of Flexible Polyurethane Foams Filled with Fumed Silica". *Journal of Natural Sciences*, 16(1): 29-32.

Vega Baudrit, José K,, Delgado and S, Madrigal. (2012): "Biodegradable Polyurethanes from Sugar Cane Biowastes". *Cellulose Chemistry and Technology.* 45: 507-514.

Valášek, P. Žarnovský, J. and Müller M. (2013): "Thermoset Composite on Basis of Recycled Rubber" P. *Advance Materials Research*, 801: 67-73

Xia, C., Lee, J. L., Widya, T. and Macosko, C. (2005): "Polyurethane/clay nanocomposites foams: Processing, structure and properties". *Polymer,* 46: 775-783.

Yan, Y.; Hu, M. and Wang, Z. (2010): "Kinetic Study on the Liquefaction of Corn Stalk in Polyhydric Alcohols". *Industrial Crops Production* 32: 349-352

Yao, W. Chen, B, and Wu, K (2003): "Smart Behaviour of Carbon Fibers Reinforced Cement-Based Composite". *Journal of Material Science and Technology* 19(3): 239-242

Yao, Y., Yoshioka, M., and Shiraishi, N. (1995): "Rigid Polyurethane Foams from Combined Liquefaction of Wood and Starch", *Mokuzai Gakkaishi,* 41(7): 659-668

Zhang T, Zhou Y, Liu D, and Petrus L (2007): "Qualitative Analysis of Products Formed During the Acid Catalyzed Liquefaction of Bagasse in Ethylene Glycol". *Bioresource Technology* 98:1454–1459.

Zhang, H: Fang, W. Z: Li, Y. M. and Tao, W. Q. (2017): "Experimental Study of the Thermal Conductivity of Polyurethane Foams". *Applied Thermal Engineering* 115: 528-538

Zhao Y, Yan N, Feng M (2012): "Polyurethane Foams Derived from Liquefied Mountain Pine Beetle-Infested Barks". *Journal Applied Polymer Science* 123:2849–2858.