

Spectroscopic Characterization of Acetylated Wood Flakes and Its High-Density Polyethylene Blends

Yakubu Azeh*, Fatima Abubakar, Monday Musah, John Tsado Mathew, Musa Tanko Umar, Yahaya Sayyadi Mohammad, Aliyu Turaki Ibrahim, Abdulkarim Mohammed Awwal, Hauwa Larai Muhammad Idris and Aishetu Ibrahim Muhammad

Received: 26 March 2022/Accepted 25 April 2022/Published online: 30 April 2022

Abstract: Acetylation is a useful reaction for the improvement of the surface properties of natural fibers in bend/composites fabrications, which are essential in the determination of their usefulness. In this study, the effects of acetylation on wood flakes/fibers of *Gmelina Arborea* was investigated. Acetylation was carried out in batches using acetic anhydride as an acetylating agent and acetic acid as a catalyst at 120°C for 1, 2, and 3 h under reflux. The efficiency of acetylation was evaluated in terms of weight percent gains (WPGs) due to acetylation. Polyethylene blends were prepared by the solution blending of acetylated/unacetylated wood flakes using casting-evaporation method in toluene. The effects of acetylated/unacetylated wood flakes on the biodegradability of blends were investigated. Acetylated wood and blends were characterized using Fourier transform infrared spectroscopy (FT-IR), Thermogravimetry analysis (TGA/DTGA) and scanning electron microscopy (SEM). Results obtained from the study revealed that modification was indicative based on the observation of C=O absorption peak at 1722 and 1640 cm^{-1} respectively. TGA results revealed blend composition of 0.5/0.5 g gave the best material's stability. We also observed that the properties of the modified wood flakes/blends were enhanced by the lower equilibrium moisture content caused by acetyl groups. The modified wood flakes could find industrial applications in fiber and particles/plywood board products for various construction purposes while unmodified blends can be used in the production of polymer-based

biodegradable products. Biodegradation results indicate that blending of acetylated wood flakes with polyethylene matrix enhanced its biodegradation. Thus, adding acetylated wood flakes into polyethylene could reduce the lifespan of PE in the environment.

Keywords. Acetylation; Wood flakes; Fourier Transform Infrared Spectroscopy; Poly-blends; Scanning Electron Microscopy

Yakubu Azeh*

Ibrahim Badamasi Babangida University, Department of Chemistry, Lapai, Niger State-Nigeria

Email: yakubuazeh@gmail.com,
azehy@ibbu.edu.ng

Orcid id: 0000-0002-3818-674X

Fatima Abubakar

Ibrahim Badamasi Babangida University, Department of Chemistry, Lapai, Niger State-Nigeria

Email: fatimaabubakar044@gmail.com

Orcid id: NIL

Monday Musah

Ibrahim Badamasi Babangida University, Department of Chemistry, Lapai, Niger State-Nigeria

Email: mkwagana@gmail.com

Orcid id: 0000-0002-3224-1664

John Tsado Mathew

Ibrahim Badamasi Babangida University, Department of Chemistry, Lapai, Niger State-Nigeria

Email: johntsadom@gmail.com

Orcid id: 0000-0002-9514-5330

Musa Tanko Umar

Ibrahim Badamasi Babangida University,
Department of Chemistry, Lapai, Niger State-
Nigeria

Email: tankoum@gmail.com

Orcid id: 0000-0002-7602-4370

Yahaya Sayyadi Mohammad

Ibrahim Badamasi Babangida University,
Department of Food Science and Technology,
Ibrahim Badamasi Babangida University,
Lapai, Niger State-Nigeria.

Email: yahsaymoh@gmail.com

Orcid id: 0000-0003-1527-2845

Aliyu Turaki Ibrahim

Ibrahim Badamasi Babangida University,
Department of Chemistry, Lapai, Niger State-
Nigeria

Email: alituraki@gmail.com

Orcid id: 0000-0002-0750-8803

Abdulkarim Mohammed Awwal

Ibrahim Badamasi Babangida University,
Department of Chemistry, Lapai, Niger State-
Nigeria

Email: abdulkarimawwal@gmail.com

Orcid id: 0000-0002-4044-4920

Hauwa Larai Muhammad Idris

Ibrahim Badamasi Babangida University,
Department of Chemistry, Lapai, Niger State-
Nigeria

Email: hauwalarai2010@gmail.com

Orcid id: 0000-0002-0426-7269

Aishetu Ibrahim Muhammad

Ibrahim Badamasi Babangida University,
Department of Chemistry, Lapai, Niger State-
Nigeria

Email: ibmaisha@yahoo.com

Orcid id: 0000-0002-4745-1453

1.0 Introduction

Polymers are highly consumed by different industries, especially in packaging. Polymeric wastes have gained close attention because of the problems of inappropriate disposal in the environment. Polyethylene represents the most consumed polymer in terms of international

trade of commercial polymers (Moreno and Saron, 2017; Nuryawan *et al.*, 2020). The global production of the highly desirable, polyethylene was estimated in 2009 as 18.4 million tons with an expected 2 % annual projected increase between 2009-2020. One of the global challenges in the polyethylene industries is on the recovery rate of polyethylene/plastic materials. LDPE is mainly used for the production of polymeric films due to its high elongation and flexibility when it is combined with wood (Guiot *et al.*, 1999; Fahim *et al.*, 2015). Wood dust is regarded as waste because it has minimal useful applications in Nigeria. It is found in almost everywhere, because of the high consumption of wood for structural utilization in homes, building and other construction outlets. Wood derived fillers have become more accepted in recent years, due to its advantages such as low density, flexibility during the processing with no harm to the equipment, acceptable specific strength properties, biodegradability, high stiffness and availability of renewable resources and low cost per volume basis (Kim *et al.*, 2007; Mosab *et al.*, 2015). Due to the very high cost of other fillers such as polylactide (PLA), polyhydroxybutyrate (PHB), polyhydroxy - butyrate-co-polyvalerate (PHB-V), and poly- ϵ -caprolactone (PCL) (Zykova *et al.*, 2021). Current research interest is now directed towards the formulation of new matrix materials based on synthetics such as low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE) and high-density polyethylene (HDPE), natural rubber, or thermoplastic starch (TPS) with natural fillers such as wood flour (Kim *et al.*, 2007; Azeh *et al.*, 2013; Nuryawan *et al.*, 2020), rice husk flour (Azeh *et al.*, 2011, 2012a,b; Ruey *et al.*, 2014; Nuryawan *et al.*, 2020), flax straw, banana, hemp, kenaf, wheat, rice, corn fibers and marine residues of animal origin (e.g., chitin from shrimp shell) (Ruey *et al.*, 2014; Nuryawan *et al.*, 2020; Zykova *et al.*, 2021), poplar fibers (Amir and Alireza, 2009).



Acetylation reaction is carried out mainly in the liquid phase. Acetic anhydride was used in the early works for the acetylation of fiber in the presence of metal salts as a catalyst. The reaction with acetic anhydride results in the esterification of the accessible hydroxyl groups in the cell wall with the formation of a by-product, acetic acid (Rowell *et al.*, 1994).

$$\text{Wood-OH} + \text{CH}_3\text{-C(=O)-O-C(=O)-CH}_3 \rightarrow \text{Wood-O-C(=O)-CH}_3 + \text{CH}_3\text{-C(=O)-OH}$$

The single-site reaction implies that one acetyl group is on one hydroxyl group and no polymerization. The weight gain in acetyl can be directly converted into units of hydroxyl group blocked. This is not true for a reaction where polymer chains are formed. Acetylation has also been carried out using gaseous ketene (Tarkow, 1946; Rowell *et al.*, 1993; Azeh *et al.*, 2013). In this case, esterification of the cell wall hydroxyl group takes place, but there is no formation of by-product, acetic acid.

$$\text{Wood-OH} + \text{CH}_2=\text{C}=\text{O} \rightarrow \text{Wood-O-C(=O)-CH}_3$$

While this is interesting chemistry that eliminates a by-product, it has been shown that reactions with Ketene gas results in poor penetration of reactive chemicals, and the properties of the reacted wood are less desirable than those of wood reacted with acetic anhydride (Rowell *et al.*, 1993). The most preferred method of acetylating wood today is the use of the limited amount of liquid; acetic anhydride without a catalyst or co-solvent (Rowell, 1991; Rowell *et al.*, 1993). Consequently, the aim of this study is to prepare acetylated wood-high-density polyethylene (HDPE) blends that could be biodegraded over-time.

2.0 Materials and Methods

2.1 Sample collection

The *Gmelina Arborea* wood flakes used in this work were collected from a local sawmill in Lapai, Niger State, Nigeria. Acetic acid, acetic anhydride, toluene, anti-bumping granules, ethanol, Water, Polyethylene (Non-printed portion of pure water sachet), n-hexane,

Potassium hydroxide. All reagents and chemicals used were of analytical grade.

2.3 Modification of wood fiber/flakes

2.3.1 Acetylation

Pre-treated wood flake was weighed (0.1 – 0.7 g) and placed in a reaction flask followed by the addition of 280 mL of a mixture of acetic anhydride and acetic acid in the ratio of 1:10 (w/v) and refluxed for 1, 2, and 3 h at 80 °C respectively. After the completion of the reaction, the acetylation product was filtered, washed with ethanol and rinsed with hot water continuously until the washed water tested neutral to pH paper. Washed samples were oven dry at 105 °C for 3 h, cooled and weighed until a constant weight was obtained and then, stored in polyethylene bags for further analysis (Azeh *et al.*, 2019). The acetylated wood flakes were ground into a fine powder and the fine powdered particles were used for the preparation of polyethylene blend at various loading content of the fibrous acetylated/unacetylated wood.

2.3.2 Preparation of Blend

The dissolution of high-density polyethylene (packaged water sachets) was done using the method described by Azeh *et al.* (2019). 0.50 g pieces of non-printed portions were weighed and placed in a beaker containing 20 mL of toluene. The content was placed on a hot plate and a thermometer was inserted and heated. The sachets swelled at 60°C and then rapidly dissolved at 80°C to give a clear solution. After the dissolution of all the PE, 0.50 g of each acetylated and unacetylated fine wood particle were separately introduced into the clear solution with stirring for a duration of 5 minutes (for homogenization) and then, spread on clean tiles to form a film. The thin films formed were peeled off the tiles to afford acetylated/unacetylated wood-polyethylene blends.

2.3.3 Water absorption tests

Acetylated-wood polyethylene blends and control were prepared and the weight of each sheet was noted. Acetylated-wood



blends/unacetylated-wood blend was subjected to a water absorption test. The blends were soaked in distilled water for 1, 2, 3, and 4 h respectively. After each immersion period, the blend was removed and excess water was wiped off using tissue paper and then re-weighed. The difference in weight was used to evaluate the water absorption according to equation 1

$$H_2O \text{ absorption } (\%) = \frac{W_2 - W_1}{W_1} \times \frac{100}{1} \quad (1)$$

where W_1 is the initial weight of blend before immersion in water and W_2 is the weight of blend after immersion in water.

3.0 Results and Discussion

3.1 Weight percentage gains (WPGs)

Weight percent gained (WPGs) in acetyl was calculated using equation 2 and the results obtained are shown in Table 1

$$Wt \% \text{ gain (WPGs)} = \frac{W_2 - W_1}{W_1} \times \frac{100}{1} \quad (2)$$

Results obtained from weight percent gained after acetylation are recorded in Table 1.

Table 1: Percent weight gained due to acetylation

Acetylation time(h)	Weight of sample before acetylation (g)	Weight of sample after acetylation (g)	WPG (%)
1	10	10.92	9.2
2	10	10.89	8.9
3	10	10.83	8.3

The results show that there was an increase in the final weight of the wood flakes (*Gmelina Arborea*) after acetylation, which indicated the success of acetylation (Fig. 1).

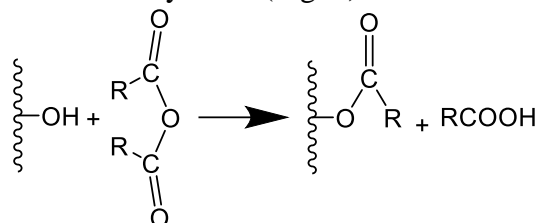


Fig 1: Anhydride and wood reaction scheme

3.2 Water absorption test

Table 2 shows the percentage absorption or the weight gained after the blends were immersed in water for 1, 2, 3 and 4 h respectively. The results of blends formulated using acetylated-wood flakes obtained after 2 h of acetylation showed that there was an initial increase in weight after the 1 h immersion in distilled water. However, weight was observed to be constant after 2 and 3 h of immersion. This indicates that the saturation point was established. After 4 h of immersion, the weight of the blends however, increased, probably due to residual swelling caused by the inaccessible -OH group in wood (Azeh *et al.*, 2012a). The

values for the percent water absorption for the acetylated-wood polyethylene blends were 12.5, 12.5, 12.5 and 25 % after 1, 2, 3 and 4 h of immersions respectively. A similar trend was observed for the blend prepared using acetylated wood that had been modified for 3 h. This was not so for the unacetylated-wood polyethylene blends as the blend showed high percent water absorption in the range of 20-40 %. This was implicated by the large presence of surface -OHs in wood. The percent water absorption was calculated using equation 1.

3.3 Thermogravimetric Analysis (TGA)

TGA/DTG of the samples was done in order to study the thermal stability of the samples for possible application areas (Figs. 2 and 3). Thermogravimetric analysis (Figs. 2 and 3) of acetylated woods, neat-PE and poly-blends revealed mass loss due to water and lignin around 100-200 °C (Ruey *et al.*, 2014). Further thermal degradation took place as a two-step process. In the first step, the degradation of hemicelluloses occurred around 300 °C. A shoulder very close to the main peak was observed in the DTG curve (Fig. 3) for all the wood samples investigated.



Table 2: Water absorption tests of blends formulated using different modified wood weights

Acetylation time (h)	Wt. of sample (g)	Wt. of sample after immersion for 1h	Wt. of sample after immersion for 2h	Wt. of sample after immersion for 3h	Wt. of sample after immersion for 4h	% Absorption for 1, 2, 3 and 4 h
Untreated sample	0.05	0.06	0.07	0.07	0.07	20.0, 40.0, 40.0, 40.0
2h	0.08	0.09	0.09	0.09	0.10	12.5, 12.5, 12.5, 25.0
3h	0.08	0.09	0.09	0.11	0.11	12.5, 12.5, 37.5, 37.5

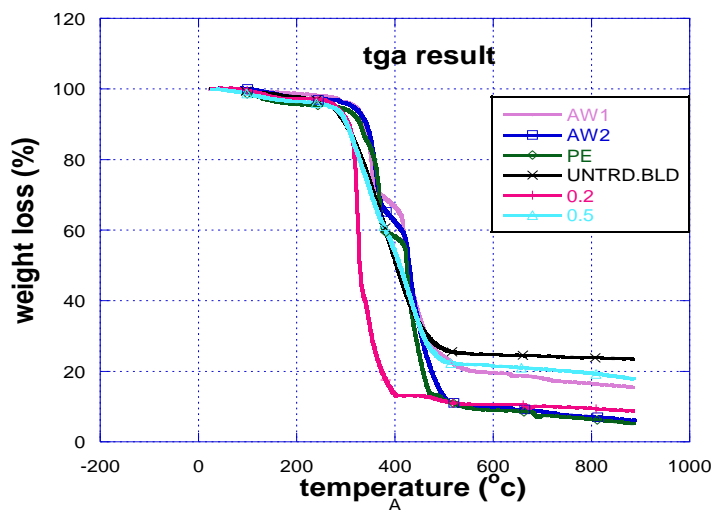


Fig 2: Thermogram (TGA) of acetylated wood (AW1 and AW2), PE, Untreated wood blends and Treated wood blends with 0.2 and 0.5 Loading Content

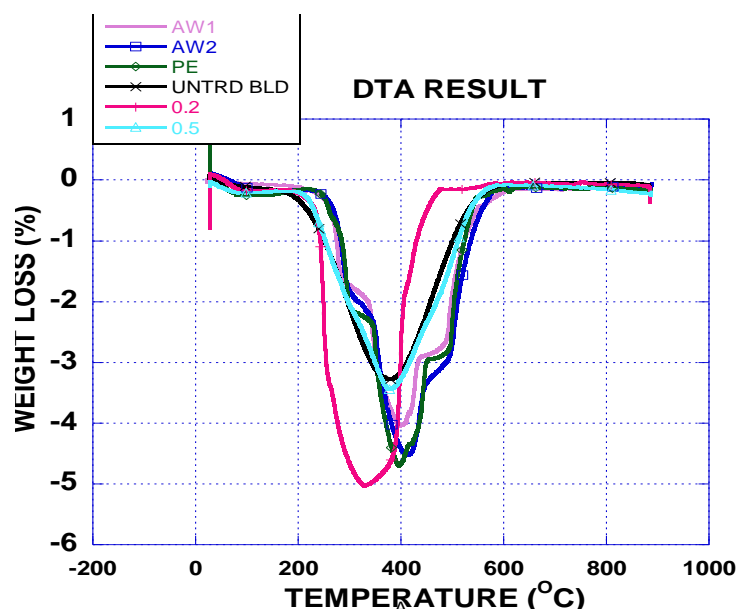


Fig 3: Derivative Curve (DTG) of acetylated wood (AW1 and AW2), PE, Untreated wood blends and Treated wood blends with 0.2 and 0.5 Loading Content



This shoulder was attributed to depolymerization of hemicelluloses in both the acetylated and unacetylated Gmelina wood blends (Ruey *et al.*, 2014).

The second degradation peak occurred around 350-400 °C is ascribed to the main degradation of cellulose structure, with the appearance of a prominent peak, which corresponds to the maximum decomposition rate similar to the findings on biocomposites reinforced with rice husks flour (Yang *et al.*, 2007; Ruey *et al.*, 2014). The depolymerization of hemicelluloses was seen around 180 °C and 350 °C, similar to the thermal properties of bio-flour-filled polyolefin composites reported by Kim *et al.* (2006). The degradation of neat-PE can be seen around 380 °C_{max}. The random cleavage of glycosidic linkage of cellulose occurred around 275 °C and 350 °C while degradation of lignin was observed around 250 °C and 500 °C similar to the findings on wood composite (Paul and Serpil, 1993; Kima *et al.*, 2006). The fast decomposition pattern of hemicelluloses is attributable to its amorphous structure (Yang *et al.*, 2007; Gao-jin *et al.*, 2010; Kajsa *et al.*, 2014). The crystalline regions in cellulose are known to improve the thermal stability of wood (Yang *et al.*, 2007). As can be seen in Fig. 2, around 180-190 °C, the 0.50 g of the blend obtained from Gmelina wood dust showed more significant weight loss. This behavior might be associated with the high content of extractives in the blend. Extractives being compounds of low molecular mass compared to cellulose, can promote ignitability of the wood at a very low temperature, due to the highly volatile nature of the compounds and thus, accelerates the degradation process. In this manner, the degradation of one component may accelerate the degradation of other wood components (Gronli *et al.*, 2002; Shebani *et al.*, 2009). Alternatively, the individual chemical components of wood behave differently, if they are isolated or closely combined within every single cell of the wood structure (Popescu *et al.*, 2009). 0.20 g of the blend was obtained

from wood dust with the extractive content of 11 %, which showed high thermal stability than the 0.50 g of the blend obtained from the same wood dust with about 100 % wood extractives. This behavior may be associated with the high content of lignin in 0.20 g of the blend. Though, the decomposition of wood is a complex process and involves a series of competitive and/or consecutive reactions. Wood decomposition is a complex mechanism and differs from one species to another, depending on structural, chemical and component composition (Yao *et al.*, 2008; Poletto *et al.*, 2010; Poletto *et al.*, 2012).

3.4. Scanning Electron Microscopy Morphology (SEM)

SEM was used to observe wood cell walls and their sub-cellular component changes due to acetylation and blending with PE. SEM images show the crystalline pattern of cellulose and the interfacial connections of acetylated samples with polyethylene (Fig. 4). Dispersibility of acetylated wood in the polyethylene matrix was ascribed to acetyl moieties on wood cellulose surface, due to similarities in surface energy of acetylated wood and polyethylene matrix. This enhanced the miscibility between the hydrophobic acetylated wood and the hydrophobic polyethylene matrix. The absence of cavities and pulled-out fibers was observed. This confirmed that the interfacial bonding between acetylated wood and the polyethylene matrix was enhanced, due to acetylation. SEM micrograph of polyethylene blends reinforced with 0.20 g of acetylated wood and 0.50 g of PE (Fig. 4), show that the acetylation of wood fiber surface had effects on blend surface as it reduced the disordered regions in the fibers. A comparison of the surface of the acetylated wood-polyethylene blend with that of the unacetylated wood-polyethylene blend showed that the blend obtained with the acetylated wood had minimal surface roughness to those made using unacetylated wood dust. A decrease in surface roughness of blends of acetylated wood was a direct indication of



interfacial compatibility of fiber and matrix surface (Samira *et al.*, 2012; Khoshkava and Kamal, 2013).

SEM images of blends reinforced with untreated wood at 0.50 g/0.50 g polyethylene loading showed numerous cavities and pull-out fiber. The blend surface was also, not smooth,

due to differences in surfaces or surface energies. Cellulose micro bundles, a localized bunch of fibers and patches were observed. This was indicative of the poor dispersion of unacetylated wood fibers within the polyethylene matrix.

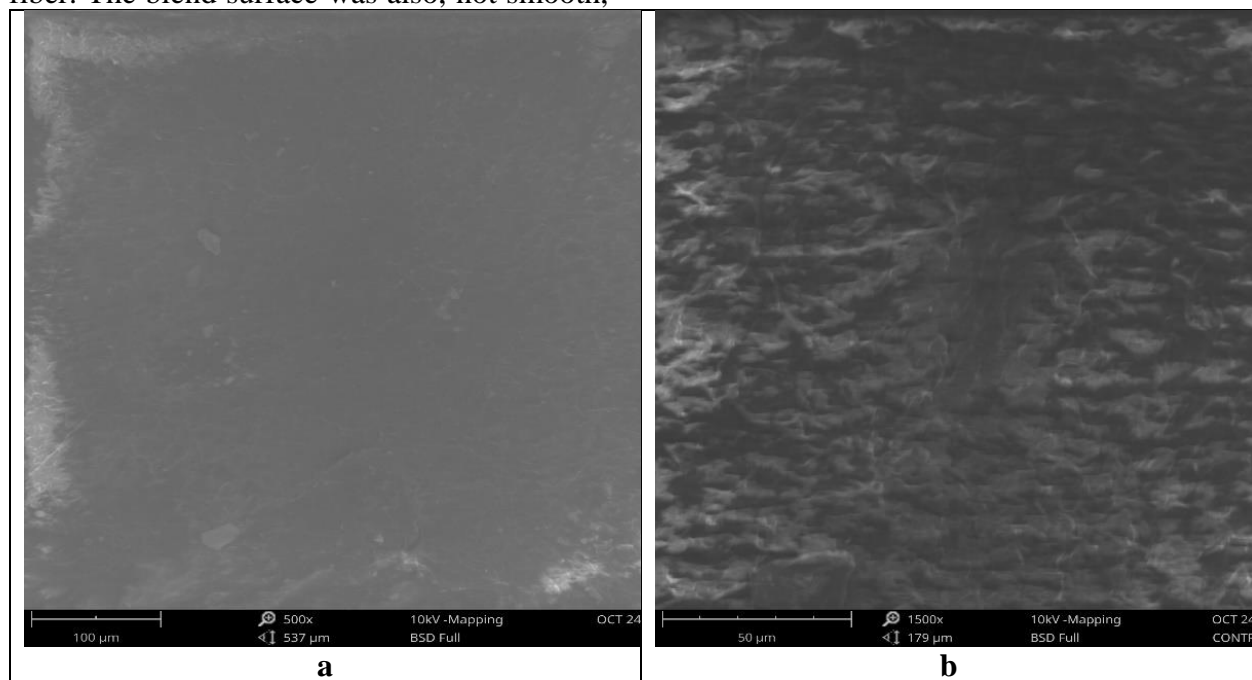


Fig 4: SEM Image of (a) acetylated-wood PE blend and (b) unacetylated-wood PE blend

3.5. FT-IR Spectroscopy

Fourier transforms infra-red spectra of the treated and untreated blend. FT-IR analysis was carried out to characterize the incorporation of wood flakes into the polyethylene and monitor the changes in FT-IR absorption bands and vibration shifts related to cellulosic wood flakes interaction (Figs. 5 and 6). The FT-IR spectra of acetic anhydride showing the major absorption bands are shown in Table 3 and 4.

The following strong absorption bands were reflected separately on the FTIR spectra of both the blends made with acetylated wood flakes and unacetylated wood flakes are characteristic broad absorptions of bonded -OH, and these include $3690\text{--}3280\text{ cm}^{-1}$ and 3697 cm^{-1} . However, a reduction in the band intensity of the blends obtained from the acetylated wood

flakes was observed. This has been attributed to acetylation. Two strong sharp bands which occurred as doublets around at $2914, 2847\text{ cm}^{-1}$ in both blends are assigned to sp^3 hybridized C-H stretch vibration. The band around $2050\text{--}1982\text{ cm}^{-1}$ and 1595 cm^{-1} are due to C=C of the aromatic ring stretch vibration. This indicates the presence of residual lignin. The peak absorption at 1722 cm^{-1} resulted from C=O of the acetate ester, due to acetylation. The band at 1640 cm^{-1} in the blends made with unacetylated wood flakes may be due to the presence of conjugated ketone in native wood. 1461 cm^{-1} and is assigned to C-O while bending vibrations of the C-H bond in blends were observed at 1379 cm^{-1} and 1267 cm^{-1} respectively (Azeh *et al.*, 2012a,b). The band recorded at 1032 cm^{-1} in both blends has been assigned to C-O. However, this band increased



in the blends made with the acetylated wood flakes. The observed increase is due to acetylation.

3.6 Burial Experiment

A burial experiment was carried out in a termite hill. Acetylated wood-polyethylene blends and unacetylated wood-polyethylene blends with an initial mass of 1.49 g for the untreated blend, 1.49 g for the treated blend, (acetylated for 3 h and 1.49 g acetylated for 2 h) were together buried in a termite hill for 1-4 weeks with monitoring. After one week, samples were

removed and re-weighed separately. Weights recorded for one week, two weeks and one month for the unacetylated blend were 1.37 g (8.05 %), 1.31 g (12.08 %), and 1.22 g (18.12 %) while in acetylated blend, 3 h and 2 h acetylation respectively had 1.41 g (5.36 %), 1.35 g (9.40 %), 1.28 g (14.10 %) and 1.44 g (3.36 %), 1.41 g (5.37 %) and 1.32 g (11.41 %). From the above results, the unacetylated polyethylene blend lost greater weight than the poly-blends obtained with the acetylated wood flakes.

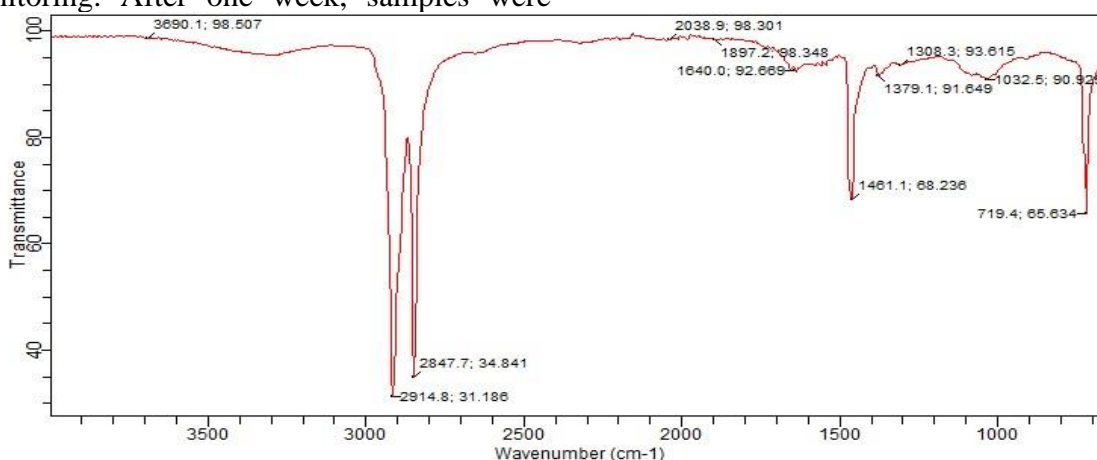


Fig 5: FT-IR result of unacetylated-wood PE blend

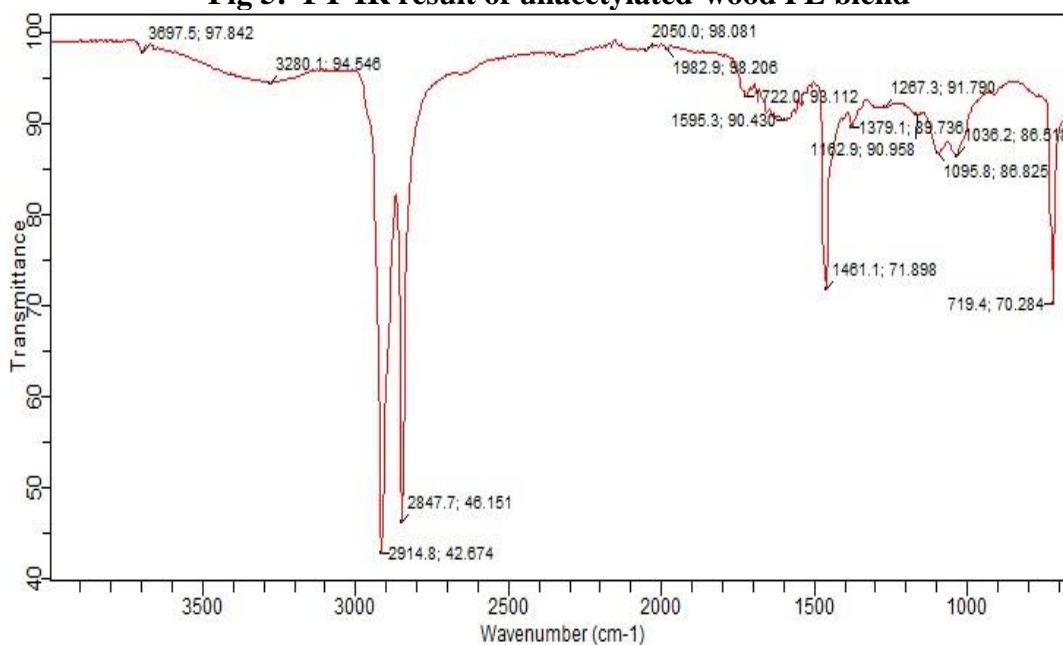


Fig 6: FT-IR result of acetylated-wood PE blend



Table 3: FT-IR results for the Acetylated-wood PE Blend

Peak (cm ⁻¹)	Functional group	References
3697-3280	–OH bond stretching	Azeh <i>et al.</i> (2012a)
2914-2847	CH stretching of methyl and methylene	Azeh <i>et al.</i> (2012a)
2050-1982	C=C stretching of aromatic ring	Azeh <i>et al.</i> (2012a)
1722-1595	C=O stretching in conjugated ketone	Popescu <i>et al.</i> (2007)
1461	C–O stretching in cellulose and hemicellulose	Azeh <i>et al.</i> (2019)
1379	C–H bending	Francesca <i>et al.</i> (2012)
1267	(νC-O) stretching band vibrations of the acetyl moieties	Azeh <i>et al.</i> (2011)
1162	C–O–C Asymmetrical stretching (cellulose and hemicellulose)	Popescu <i>et al.</i> (2007)
1095	C–O Stretching in acetyl group	Popescu <i>et al.</i> (2007)
1036	C=C stretching of the aromatic ring caused by lignin	Azeh <i>et al.</i> (2012a)
719	CH ₂ deformation and stretching in cellulose, lignin and xylan	Li <i>et al.</i> (2012); Mohamed <i>et al.</i> , (2018)

Table 4: FT-IR results for the unacetylated-wood PE blend

Peaks (cm ⁻¹)	Functional group	References
3690	–OH bond stretching	Azeh <i>et al.</i> (2012a)
2914-2847	CH stretching of methyl and methylene	Azeh <i>et al.</i> (2012a)
2038	C=C stretching of aromatic ring	Azeh <i>et al.</i> (2012a)
1640	–OH of absorbed water	Azeh <i>et al.</i> (2019)
1461	C–O stretching	Azeh <i>et al.</i> (2019)
1379-1308	CH out of plane stretching absorption of aromatic ring caused by lignin	Francesca <i>et al.</i> (2012)
1032	C=C stretching of the aromatic ring caused by the lignin	Azeh <i>et al.</i> (2012a)
719	CH ₂ deformation and stretching in cellulose, lignin and Xylan	Li <i>et al.</i> (2012)

Time variations for the acetylation also had effects on the weight loss parameter as observed with the 3 and 2 h acetylated woods. PE-blends made with the 3 h acetylated wood loss more weight than PE-blends made with the 2 h acetylated wood. This probably points to the fact that deacetylation occurred during the 3 h period of fiber acetylation, being an equilibrium reaction process. Colour change was also used as an indicator of biodegradation

(Fig. 9). The poly-blends made of acetylated wood showed no colour change while the control sample became dark-brown and had various degrees of degradation. The degradation of poly-blends was a function of wood cell wall polymers being recognized and accessed by microorganisms as food (Fahim *et al.*, 2015).



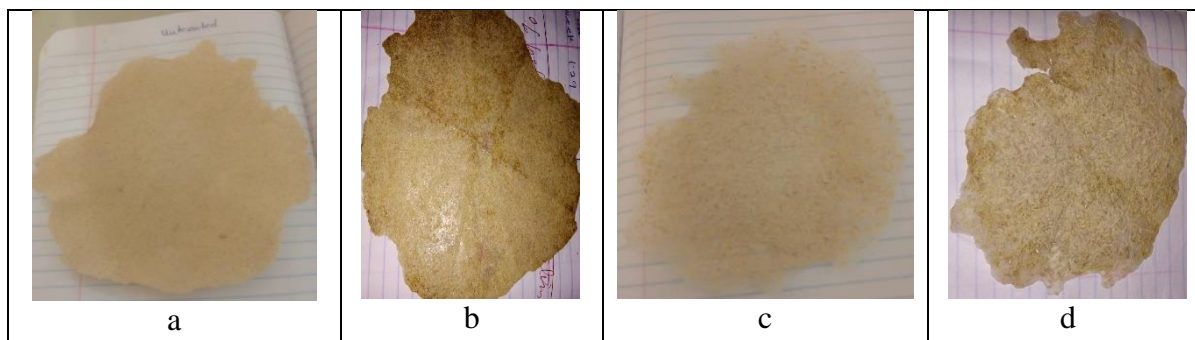


Fig. 9: (a) Unacetylated-wood PE blend before burial; (b) Unacetylated-wood PE blend after burial; (c) Acetylated-wood PE blend before burial and (d) Acetylated-wood PE blend after burial

4.0 Conclusion

Wood acetylation was successful based on the FT-IR, TGA and SEM results. Thermal analysis shows that acetylated samples were more stable thermally compared to the unacetylated samples. This trend was also observed for the blends made with acetylated samples. Biodegradation experiment shows that acetylated-wood PE blend was fairly resistance against microbial degradation. Absorption study shows that acetylated-wood polyethylene blend was more resistance to water uptake. The best loading content observed was 0.50 g of acetylated wood against 0.50 g of polyethylene.

5.0 Acknowledgements

The authors wish to acknowledge the support received from the Laboratory Technologists of the department of Chemistry, IBB University, Lapai, Nigeria.

6.0 References

Amir, N. & Alireza, A. (2009). Preparation and properties of wood plastic composites made of recycled high-density. Polyethylene. *Journal of Composite Materials*, 43, 8, pp. 877-883.

Azeh, Y., Tanko, M. U. & Sani, S. D. M. (2011). Chemical modification of microcrystalline cellulose: Improvement of barrier surface properties to enhance surface interactions with some synthetic

polymers for biodegradable packaging material processing and applications in textile, food and pharmaceutical industry. *Advances in Applied Science Research*, 6, pp. 532-540.

- Azeh, Y., Olatunji, G. A. & Mamza, A. P. (2012a). Scanning electron microscopy and kinetic studies of acetylated wood/cellulose high-density polyethylene blends. *International Journal of Carbohydrate Chemistry*, doi.org/10.1155/2012/456491
- Azeh, Y., Gabriel, A. O., Sunday, O. & Olubunmi, A. (2012b). Ketene acetylated wood cellulose for possible industrial applications in wood-base and polymer industry. *Journal of Environmental Science and Technology*, 5, 3, pp. 168-176.
- Azeh, Y., Olatunji, G. A. & Cheku, M. (2013). Acetylation of wood flour from four wood species grown in Nigeria using vinegar and acetic anhydride. *MCSER: International Journal of Interdisciplinary Studies* 2(10), pp. 147-154.
- Moreno, D. D. P. & Saron, C. (2017). Low-density polyethylene waste/recycled wood composites. *Composite Structures*, 176, pp. 1152-1157. doi: [10.1016/j.compstruct.2017.05.076](https://doi.org/10.1016/j.compstruct.2017.05.076)
- Azeh, Y., Yohanna, B. P. & Gimba, A. (2019). Chemical modification of wood using vinegar and benzoic acid against termites degradation. *Int. J. Forestry Wood Sci.*, 6(1), pp. 054-061.

- Fahim, I. S., Wae, M. & Salem, H. G. (2015). Effect of processing technique on LDPE thin films and sheets. *International Journal of Engineering Inventions*, 4(12), pp. 01-05.
- Francesca, L., Roberta, D.S., Donato, C., Giuseppe, V. & Alfonso, M. (2012). Monitoring wood degradation during weathering by cellulose crystallinity. *Taiwan. J. for Sci.*, 25(3), pp. 251- 64.
- Guiot, O., Tighzert, L. & Coqueret, X. (1999). Electron beam crosslinking of extrusion-blown LDPE films: Mechanical properties. *Eur Polym. J.*, 35, pp. 565-570.
- Grønli, M. G., Várhegyi, G. & Di-Blasi, C. (2002). Thermogravimetric analysis and devolatilization kinetics of wood. *Ind. Eng. Chem Res.*, 41, pp. 4201–4208.
- Gao-jin, L.V., Sahu-bin, W. & Rui, L. (2010). Kinetic study of the thermal decomposition of the hemicellulose isolated from corn stalk. *BioResources*, 5, pp. 1281-1291.
- Kima, H-S., Sumin, K. & Hyun-Joong, K. (2006). Thermal properties of bio-flour-filled polyolefin composites with different compatibilizing agent type and content. *Thermochimica Acta*, 451, pp. 181–188.
- Kim, H-S., Byoung-Ho, L., Seung-Woo, C., Sumin, K. & Hyun-Joong, K. (2007). The effect of types of maleic anhydride-grafted polypropylene (MAPP) on the interfacial adhesion properties of bio-flour-filled polypropylene composites. *Composites: Part A.*, 38, pp. 1473–1482.
- Khoshkava, V. & Kamal, M. R. (2013). Effect of surface energy on dispersion and mechanical properties of polymer/nanocrystalline cellulose nanocomposites. *Biomacromolecules*, 14(9), pp. 3155-163.
- Kajsa, W., Linda, P. & Markus, B. (2014). Thermal decomposition of hemicelluloses. *J. Anal. Appl. Pyrol.*, 3267-3275. [doi: 10.1016/j.jaap.2014.08.013](https://doi.org/10.1016/j.jaap.2014.08.013)
- Li, M. F., Sun, S. N., Xu, F. & Sun, R.C. (2012). Benzylation and characterization of cold NaOH/urea pre swelled bamboo. *BioRes.*, 7(2), pp. 1876-1890. doi: 10.15376/biores.7.2.1876-1890.
- Mantanis, G. & Lykidis, C. (2015). Evaluation of weathering of furfurylated wood decks after a 3-year outdoor exposure in greece. *Drvna industrija*, 66(2), pp. 115–122. doi:10.5552/drind.2015.1425.
- Mosab, K., Kotiba, H., Fawaz, D. & Young, G. K. (2015). Material properties of polyethylene/wood composites: A review of recent works. *Polymer Science, Ser. A.*, 57(6), pp. 689–703.
- Mohamed, T., Joeri, K. & Antonio, M. C. (2018). Differentiation between pine woods according to species and growing location using FTIR-ATR. *Wood Sci Technol.*, 52, 487. doi: 10.1007/s00226-017-0967-9.
- Nuryawan, A., Hutauruk, N. O, Purba, E. Y. S., Masruchin, N., Batubara, R. & Risnasari, I., et al. (2020). Properties of wood composite plastics made from predominant low-density polyethylene (LDPE) plastics and their degradability in nature. *PLoS ONE*, 15(8), pp. 1-21. [doi: 10.1371/journal.pone.0236406](https://doi.org/10.1371/journal.pone.0236406).
- Paul, T.W. & Serpil, B. A. V. (1993). *Advances in thermochemical biomass conversion springer science*, pp. 771-783. Springer Science.
- Poletto, M., Dettenborn, J., Pistor, V., Zeni, M. & Zattera, A. J. (2010). Materials produced from plant biomass. Part I: Evaluation of thermal stability and pyrolysis of wood. *Mat. Res.*, 13, pp. 375–379.
- Poletto, M., Ademir, J. Z., Maria, M. C. F. & Ruth, M. C. S. (2012). Thermal decomposition of wood: influence of wood components and cellulose crystallite size. *Bioresource Technology*, 109, pp. 148-153.
- Ruey, S. C., Mohd, H. A. B. G., Sahrim, A., Mohd, N. S. & Mou'ad, A. T. (2014). Rice husk flour biocomposites based on recycled high-density polyethylene/polyethylene terephthalate blend: Effect of high filler loading on physical, mechanical and



- thermal properties. *Journal of Composite Materials*, pp. 1–13. doi: 10.1177/0021998-314533361.
- Rowell, R. M. (1991). Changes in physical properties of wood and wood fiber resulting from chemical modification. International Symposium on Chemical Modification of Wood, Kyoto, Japan, 17–18 May. Wood Research institute, Kyoto University, pp. 75–82.
- Rowell, R. M. (1991). Chemical modification of wood. In: Hon, D. N.-S. and Shiraishi, N. (Eds.), handbook on wood and cellulosic materials. Marcel Dekker, New York, pp. 703–756.
- Rowell, R.M., Simonson, R., Hess, S., Plackett, D. V., Cronshaw, D. & Dunningham, E. (1994). Acetyl distribution in acetylated whole wood and reactivity of isolated wood cell-wall components to acetic anhydride. *Wood Fiber Sci.*, 26, pp. 11–18.
- Rowell, R. M., Lichtenberg, R. S. & Larsson, P. (1993). Stability of acetylated wood to environmental changes. *Wood Fiber Sci.*, 25, pp. 359–364.
- Shebani, A. N., Van, R. A. J. & Meincken, M. (2009). The effect of wood extractives on the thermal stability of different wood-LLDPE composites. *Thermochim. Acta.*, 481, pp. 52–56.
- Samira, S., Nor, A. I, Sanaz, A., Wan, M. W. Y. Z. & Zaki, A. R. M. (2012). Effects of fibre esterification on fundamental properties of oil palm empty fruit bunch fibre/poly(butylenes adipate-co-terephthalate) biocomposites. *International J. Mol. Sci.*, 13(2), pp. 1327–1346.
- Tarkow, H. (1946). Acetylated wood report No. 1593 USDA, Forest service, products laboratory, Madison, Wisconsin, USA 9. pp. 29.
- Yang, H., Rong, Y., Hanping, C., Dong, H. L. & Chuguang, Z. (2007). Characteristics of hemicellulose, cellulose and lignin pyrolysis. *Fuel*. 86, pp. 1781-1788. doi: 10.1016/j.fuel.2006.12.013.
- Popescu, C. M., Popescu, M. C., Singurel, G., Vasile, C., Argyropoulos, D.S. & Willfor, S. (2007). Spectral characterization of eucalyptus wood. *Appl. Spectrosc.*, 61, pp. 1168–1177.
- Popescu, C. M., Singurel, G., Popescu, M. C., Vasile, C., Argyropoulos, D. S. & Willfor, S. (2009). Vibrational spectroscopy and X-ray diffraction methods to establish the differences between hardwood and softwood. *Carbohydr Polym.*, 77, pp. 851-857.
- Yao, F., Wu, Q., Lei, Y., Guo, W. & Xu, Y. (2008) Thermal decomposition kinetics of natural fibers: Activation energy with dynamic thermogravimetric analysis. *Polym. Degrad. Stab.*, 93, 90–98.
- Zykova, A. K., Pantyukhov, P. V., Mastalygina, E. E., Chaverri-Ramos, C., Nikolaeva, S. G., Saavedra-Arias, J. J., et al. (2021). Biocomposites of low-density polyethylene plus wood flour or flax straw: Biodegradation kinetics across three environments. *Polymers*, 13, pp. 2138-2153. doi: 10.3390/polym13132138.

Consent for publication

Not Applicable

Availability of data and materials

The publisher has the right to make the data public

Competing interests

There is no competing interest

Funding

There is no source of external funding

Authors' contributions

All the authors contributed equally to the work

