Evaluation of the Flexural Strength, Sorption, Rheological and Thermal Properties of Corncob Plastic Composites

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***Abstract*— *Plastic composites were made from corncobs and high density polyethylene (HDPE) by extrusion and evaluated. The composites were manufactured using two different screened corncob particle size fractions (<2 mm and <0.5 mm) and tested for flexural properties, water sorption, melt flow and thermal properties. The melt viscosities at 190°C were 18.0 ± 0.8 kPa·s (<2 mm) and 24.0 ± 0.6 kPa·s (<0.5 mm). The results obtained indicated that the composites made with the smaller particle size fraction had higher flexural strength (31.7 ± 1.7 MPa) and modulus of elasticity (1.4 ± 0.1 GPa) than those made with the larger particle size fraction (21.2 ± 1.4 MPa and 1.1 ± 0.1 GPa). Also, the composites made with the smaller particles and were more dimensionally stable. Corncob composites had thermal stability range of 259 – 274°C (onset degradation temperature). The corncob composites made with smaller sized particles possessed better properties in comparison with those made from the <2 mm. Particle size and density significantly affected the mechanical, physical and thermal properties of the composites evaluated.*

***Keywords*— Corncobs, flexural properties, particle size, plastic composites.**

I. INTRODUCTION

The scarcity of timber supply in Nigeria, due to the increasing demand for solid wood and wood products, has necessitated the use of agricultural fibers for composites production [1,2]. This is because agricultural fibers such as cotton and maize stalks, groundnut and coconut shells, rice and corncobs are renewable natural resources, which are abundantly available and low-cost alternatives. Their advantages over traditional reinforcing materials such as glass fibers and mica are acceptable specific strength properties, low density, good thermal properties, reduced tool wear, thermal and respiratory irritation, ease of separation and biodegradability [3,4]. Incorporation of agricultural fibers in composites production can enhance complete material utilization, offset wood shortage and foment the development of indoor and outdoor building components in developing countries like Nigeria [5,6].

Amongst the several agricultural crops cultivated in Nigeria, corn, a cereal crop remains the most popularly grown and consumed in all ecological zones [7]. Corn has an estimated cob residue of 3.3×10^6 ton per year, which is used as fuel but most times litter the environment especially during the harvesting season from February to September [8,9]. The chemical composition of corncobs (35 - 46% hemicellulose, 18 - 40% cellulose, 12 - 23% lignin and 1 - 4% ash) makes it a suitable candidate as a candidate furnish for plastic composites [4,6,9-11]. Furthermore, corncob based polyethylene composites have been shown by several researchers to have good flexural strength (25 - 45 MPa) and modulus (1.3 - 2.7 GPa), tensile strength (11 - 36 MPa) and water absorption (0.75 - 6.0%) properties [3,4,6].

However, literature is sparse on the effect of corncob particle size on properties of high-density polyethylene (HDPE) composites with a coupling agent. This is because the incorporation of small sized particles (100 - 200 mesh) can increase the melt flow rate (MFR), a measure of processability, and flexural strength but resulted in reduction in toughness, stiffness and crystallinity of the composites [12,13]. This effect has been attributed to interfacial interaction between the plastic matrix and the lignocellulosic fibers [12,13]. Low interfacial bonding may sometimes occur between the hydrophilic fibers and hydrophobic plastic matrix. Therefore, coupling agents, such as maleated polyethylene (MAPE) is typically incorporated in the production process to enhance fiber-matrix compatibility and thus improve properties [14,15].

The aim of this study was to evaluate the use of corncobs particles of various size in HDPE based plastic composite by extrusion. The corncob particles were partially characterized (size and composition) and the composites strength, thermal, rheological and physical properties were determined.

II. MATERIALS AND METHODS

2.1 Production of corncob plastic composites

Corncobs collected from a local market in Oyo state, Nigeria were Wiley milled and screened to <2 and <0.5 mm particle size fractions. The milled particles were oven dried at 70°C to <0.5% moisture content (MC) prior to extrusion. HDPE (48% w/w) (Equistar Petrothene, LB 0100-00, MFI = 0.3 g/10 min, and density = 0.950 g/cm^3), corncob particles (50% w/w) and MAPE (Polybond 3029, Crompton) coupling agent (2% w/w) were blended in 500 g batches and then compounded at 0.5 kg/h on an 18 mm co-rotating twin-screw extruder (Leistritz, L/D ratio of 40, 200 rpm) and extruded into a ribbon (3.6 x 50 mm^2) [16]. The barrel and die temperature were 140 - 160°C.

2.2 Properties characterization of corncob reinforced plastic composites

2.2.1 Corncob particle characterization

Air-dried samples of the corn cob particles (4-5 g) were Soxhlet extracted with dichloromethane (CH_2Cl_2, 150 mL) for a period of 20 h according to ASTM D1108-96 [17]. The CH_2Cl_2 extract was then evaporated to a constant weight and extractives yield determined gravimetrically. The lignin content was determined on extractive free corncobs using the Klason lignin method according to ASTM D1106-96 [18]. Specifically, the dried extractive free corncobs (200 mg) was incubated in 72% H_2SO_4 (2 mL) for 1 h at 30°C, then diluted into 4% H_2SO_4, and subjected to secondary hydrolysis in an autoclave (117 kPa and 121°C) for 30 min. The hydrolyzate was filtered to obtain Klason lignin content gravimetrically. The ash content was determined gravimetrically by furnacing the samples at 600°C for 16 h. All analyses were done in duplicate.

Optical microscopy was performed on screened particles on an Olympus BX51 microscope at 40× magnification equipped with a DP70 digital camera. Particle measurements (length and width) were performed on 160 particles for each sample using the Olympus MicroSuite software [19].

2.2.2 Sorption, mechanical and melt flow properties of composites

Water absorption (WA) and thickness swell (TS) tests were conducted following a modified ASTM D570-95 [20] procedure. Five replicate specimens (3 × 20 × 50 mm^3) for each sample were immersed in water at 23°C for 61 days and dimensions periodically measured. Weight gain and thickness swell were measured on a total composite basis for determination of WA and TS respectively. The diffusion coefficients (D_f) of the composites were calculated using equation 1 [21]:

$$D_f = \pi \, (h/4M_\infty)^2 \, (M/\sqrt{t})^2 \qquad (1)$$

Where, M_∞ is the maximum moisture content (MC) (%) measured at the end of the test, h is the sample thickness (m) corresponding to M_∞, t is the time (s) and $\delta M/\delta\sqrt{t}$ is the initial slope from the graph of MC versus $\delta\sqrt{t}$ relation. Three point flexural tests (strength and modulus) were performed on extruded specimens (3.56 × 20.1 × 110 mm^3, 5 replicates) according to ASTM Standard D 790-07 [22] with a crosshead speed of 1.31 mm/min and a span of 57 mm until specimen failure or 5% strain, whichever occurred first on an Instron 5500R-1132 universal test machine equipped with a 5 kN load cell. Data were collected and processed using Bluehill v3 software (Instron). Melt flow rate (MFR) and melt viscosity of molten plastic composites were measured in triplicate using a CEAST Model 7024.000 melt flow indexer according to ASTM D 1238-04C [23] through a 2.0955 mm Ø × 8 mm die, at 190°C, and load of 15 kg. Dynamic rheological measurements (elastic modulus (G′), viscous modulus (G″), and complex viscosity (η*)) were performed on a Bohlin CVO 100 rheometer, using serrated parallel plates (25 mm Ø), in an oscillating mode with an extended temperature control module on disc (3 mm × 25 mm Ø) samples. Experiments were performed in the linear viscoelastic region. Measurements were carried out at 180°C in the frequency range of 0.01 to 100 Hz at an applied strain of 0.5% [24]. Data were analyzed using the Bohlin rheology v6.51 software.

2.2.3 Thermal characterization

Thermo gravimetric analysis (TGA) was conducted using a Perkin Elmer TGA 7 instrument. Specimens (4-5 mg) were analyzed at a heating rate of 20°C/min from 50 to 800°C in a N_2 atmosphere (30 mL/min) and analyzed using Pyris v8 software (Perkin Elmer). Differential scanning calorimetry (DSC) was performed on samples (4-6 mg, in duplicate) using a TA Instruments model Q200 DSC with refrigerated cooling. The samples were (i) equilibrated at 70°C (3 min) then ramped to 180°C at 10°C/min, held isothermally for 3 min, (ii) cooled to 70°C at 10°C/min and held isothermally for 3 min and the cycles repeated [16]. Data were analyzed using TA Universal Analysis v4.4A software. The degree of crystallization of HDPE was calculated from the ratio of the melting enthalpy (ΔH^o_f, 105-145°C) of the sample to ΔH^o_f = 293 J/g of 100% crystalline HDPE [25].

Dynamic mechanical analysis (DMA) was performed in 3-point bending mode (15 mm span) on rectangular samples (3 × 6 × 20 mm^3) using a Perkin Elmer DMA-7 instrument (1 Hz and 0.1% strain) with refrigerated cooling from -50 to 115°C at a ramp rate of 3°C/min. Interfacial adhesion was evaluated by an adhesion factor

(*A*) determined from DMA data at 25 and 40°C according to Kubat et al. [26] as follows:

$$A = (1/(1-V_f))\ (\tan \delta_c/\tan \delta_m) - 1 \qquad (2)$$

where, c and m subscripts represent composites and HDPE matrix, and V_f is the fiber volume fraction:

$$V_f = (W_f\, \rho_m)/(W_f\, \rho_m + W_m\, \rho_f) \qquad (3)$$

Where, W_f is weight of corncob, W_m is the weight of HDPE, ρ_f is the density of fibers and ρ_m is the density of HDPE (0.95 g/cm^3). The ρ_f (<2 mm = 1.458 g/cm^3 and <0.5mm = 1.456 g/cm^3) was determined by gas pycnometry (1.3 g, degassed for 1 h at 104 °C) using an Ultrapycnometer 1000 (Quantachrome) with N_2.

III. RESULTS AND DISCUSSION

3.1 Properties of corncob particles

The corncob samples had a CH_2Cl_2 extractives content of 0.82% which is higher than that obtained by Pointner et al. (2014) at 0.3% but lower than 16% recorded by Ogah et al. (2015) who used a polar solvent system. Extractives can act as plasticizing agents and can reduce the MFR of the plastic composites [27], however, at this low level it is not likely to greatly influence MFR. The corncob lignin content was 15.2% while the ash content was 9.3%. The lignin value compared favorably with 11 - 23% obtained by Ogah et al. [11], Opara et al. [4], Pointner et al. [10] and Luo, et al. [6]. The ash content of 9.3% obtained in this study was higher than 1 - 4% recorded in the literature [4,6,10,11].

The lengths of the <2 mm and <0.5 mm screened corncob particles were 539 ±500 µm and 271 ±250 µm, respectively. The widths were 298 ±290 µm and 140 ±130 µm, respectively (Fig. 1). The calculated aspect ratios for the <0.5 and <2 mm fibers were comparable at 2.1 and 1.8, respectively.

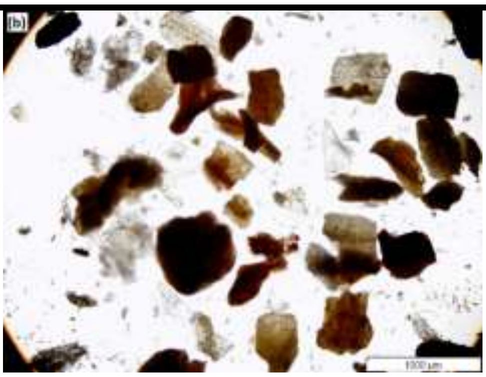

Fig. 1: Optical micrographs of screened (a) <2 mm and (b) <0.5 mm corncob particles (40×, scale bar 1 mm)

3.2 Density and flexural properties

The densities of the composites are shown in Table 1. As shown, composites made from <2 mm particles was 17% lower than that made from <0.5 mm particles (1056 kg/m^3). A similar decrease in density was observed in wood plastic composites (WPC) made with <2 mm versus <0.5 mm screened particles [19]. The lower density could be attributable to incomplete filling of the voids and/or lumens in the larger screened particles during extrusion. The results of the flexural tests on the corncob HDPE composites are shown in Table 1. As shown, the flexural strength of the <2 and <0.5 mm corncob composites were respectively, 21.2 ± 1.4 and 31.7 ± 1.7 MPa. While the flexural moduli were 1.1 ± 0.1 GPa (<2 mm particles) and 1.4 ± 0.1 GPa (<0.5 mm particles). The flexural properties were comparable to the literature (strength of 25 - 45 MPa and modulus of 1.8 - 2.7 GPa) for corncob-HDPE composites [4,6]. Composites made from <0.5 mm particles recorded significantly higher flexural strengths and moduli than those made from <2 mm particles. This observation could be ascribed to the higher density of the composites made from <0.5 mm particles which could have enhanced interfacial bonding between the cob particles and the plastic matrix (Table 1). This is more so since composites made from <0.5 mm particles have larger surface area and low stress concentration and will thus have higher strength properties [12,13]. The energy at maximum load (EML) of the composites were significantly different at 0.34 J versus 0.41 J (Table 1).

Table 1: Flexural properties and density of corncob plastic composites made with <0.5 and <2 mm particles

Composite	Density (kg/m^3)	Strength (MPa)	Modulus (GPa)	Energy (J)
<0.5 mm particles	1056.4^a (38.1)	31.7^a (1.7)	1.4^a (0.1)	0.406^a (0.005)
<2 mm particles	879.7^b (23.0)	21.2^b (1.4)	1.1^b (0.1)	0.343^b (0.005)

Means with the same letters and columns are not significantly different. Standard deviations are given in parentheses.

3.3 Melt flow and rheological properties

The melt processing behavior (MFR and melt viscosities) of the composite blends were determined using a 15 kg load in order to get a reasonable flow rate. The MFR were 3.1 and 4.0 g/10 min and melt viscosities were 24.0 and 18.0 kPa·s, for the composites made with <0.5 and <2 mm particles, respectively (Table 2). These values compared favorably with those reported for WPC (0.35 - 2.90 g/10 min; 12.7 - 107 kPa·s) [13]. Composites made from <2 mm particles recorded significantly higher MFR and lower viscosity than those from <0.5 mm particles. A similar observation was seen in WPC [19] and bamboo HDPE composites [24] made with <2 mm versus <0.5 mm screened particles. This phenomenon could be because of the lower density and an increase in the unfilled regions in the composites as observed by Stark and Berger [28].

Table 2: Melt flow rate (MFR) and melt viscosities of corncob plastic composites made with <0.5 and <2 mm particles

Composite	MFR (g/10min)	Melt viscosity (kPa·s)
<0.5 mm particles	3.1^{b} (0.10)	24.0^{a} (0.60)
<2 mm particles	4.0^{a} (0.17)	18.0^{b} (0.75)

Means with the same letters and columns are not significantly different. Standard deviations are given in parentheses.

Dynamic rheological measurements were also obtained on the corncob composites and HDPE melts at 180°C. Fig. 2 shows the G', G" and η* as a function of frequency for the composites and HDPE at 180°C. For all melt samples G' and G" were shown to increase with frequency. The addition of <0.5 mm corncob particles to HDPE increased G' 5-fold and G" 4-fold (at 0.1 Hz). At lower torsional frequency, the G' was lower than G", indicating a viscous response and the molten samples were more fluid. Incorporation of corncob particles in HDPE increased G' and G" and narrowed the gap between them at low frequency, because of the rigid character of the corncob fibers. Corncob particle size had a distinct effect on the dynamic rheological behavior of the composite melts. The cross-over point of G' and G" (G_c = G' = G") was shown to occur at a lower frequency for the corncob composites than for HDPE. The G_c decreased from 0.37 Hz for HDPE to 0.06 Hz for <2 mm corncob composites to 0.02 Hz for <0.5 mm corncob composites (Fig. 2), indicating that particle addition lowered the frequency at which the material transferred from viscous to elastic behavior. This phenomenon was also observed in rheological measurements on WPC [29] and bamboo HDPE composite [24] melts.

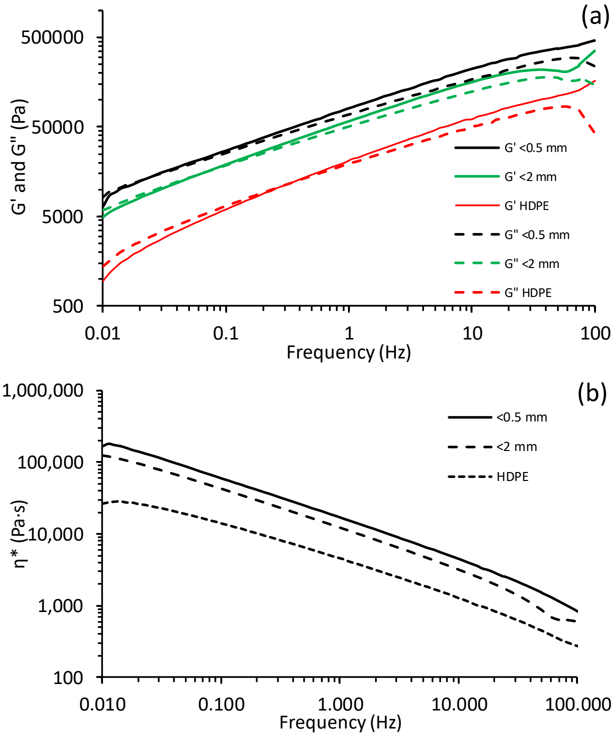

Fig. 2: Dynamic rheology showing (a) elastic (G') and viscous moduli (G") and (b) complex viscosity (η) as a function of frequency for HDPE and corncob composites made with <0.5 and <2 mm particles at 180°C.*

The flow curves showed that η* decreased with an increase in frequency, showing a shear thinning behavior, for HDPE and the composite melts (Fig. 2). The η* (at 1 Hz) was also increased from 4.54 kPa·s for HDPE to 12.2 kPa·s for the <2 mm corncob composites to 16.9 kPa·s for the <0.5 mm corncob composites. This observation of higher η* for the <0.5 mm corncob composite melt is consistent with those obtained by MFR. The higher viscosity could be explained by a higher particle-matrix interaction in the melt due to a larger surface area. These same trends in η* were observed for bamboo HDPE composites made with different particle sizes [24].

3.4 Water absorption and thickness swelling

The water soak (WA and TS) properties of the composites were measured with time (Fig. 3 and Table 3). Fig. 3 shows the WA of the corncob composites reaching a pseudo-equilibrium state in accordance with Fickian behavior [30]. The D_f for the composites made with <2 mm corncob particles (4.84×10^{-9} m^2/s) was about 3-fold higher than that for the <0.5 mm particles. The respective WA of the <2 mm and <0.5 mm corncob composites was shown to increase from 4.04 to 33.6% and 3.82 to 15.1%

over 61 d. The TS of the <2 mm and <0.5 mm corncob particle composites was shown to increase 2.23 to 6.26% and 1.98 to 5.33%, respectively. These WA and TS values are similar to temporal increases from 1.9 to 22.4 % and 1.7 to 14.1% for WPC [23] and 1.3 to 28.0% and 0.36 to 4.9% obtained by Adefisan and McDonald [19], respectively. Corncob composites made with smaller particle size had the least sorption properties. This may be possibly due to the higher density and improved interfacial interaction with the plastic matrix which resulted in less moisture ingress. Also, the composites made with larger sized particles had 17% lower density and thus had more voids which could result in higher water content in the composites [31].

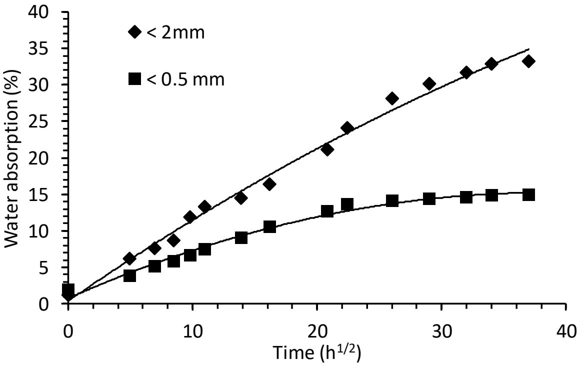

Fig. 3: Water absorption (WA) of corncob HDPE composites with time$^{1/2}$

Table 3: Water soak properties of corncob HDPE composites made with <0.5 and <2 mm particles

Composite	WA (%)		D_f (10^{-9} m²/s)	TS (%)	
	1 d	61 d		1 d	61 d
<0.5 mm particles	3.82 (0.91)	15.01 (4.99)	1.46	1.98 (0.18)	5.33 (0.54)
<2 mm particles	4.02 (0.19)	33.63 (1.04)	4.84	2.33 (0.41)	6.28 (1.15)

Standard deviations are given in parentheses.

3.5 Thermal Analysis

The thermal stability of the corncob HDPE composites was evaluated by TGA (Fig. 4 and Table 4). All thermograms show a small weight loss before 100°C, associated with water loss. The degradation stage <400°C of composites can be mainly attributed to the decomposition of chemical components such as hemicelluloses (150-350°C), lignin (250-490°C), extractives and cellulose (275-350°C) [16,18]. From the differential thermogravimetric (DTG) thermograms, the decomposition of HDPE occurred at > 460°C. Composites made with larger sized particles generally degraded at higher temperatures (274°C) in comparison with those made from smaller sized particle (259°C) indicating higher thermal stability possibly due to mass transfer effects of the larger particles as observed in pyrolysis kinetic studies [34].

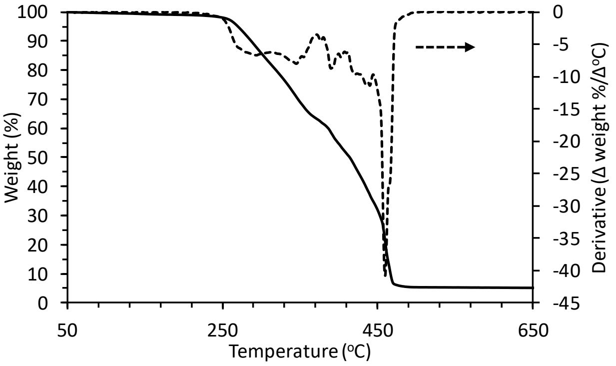

Fig. 4. TGA and DTG graphs of corncob plastic composites made with <0.5 mm particles

Table 4: Thermal degradation behavior of corncob plastic composites made with <0.5 and <2 mm particles

Corncob composite	onset (°C)	1st peak (°C)	2nd peak (°C)	3rd peak (°C)	4th peak (°C)	Final decomp-sition (°C)
<0.5 mm particles	259	274	343	392	460	469
< 2 mm particles	274	301	360	457	494	494

The degree of HDPE crystallinity and transition temperature in the corncob plastic composites was determined by DSC and data presented in Table 5. These values were comparable to WPC [13,16,19]. The HDPE crystallinity in the composites made from <2 mm particles was 6% higher than that made from the <0.5 mm particles (65.9%). This may suggest that the larger particles induced more nucleation of HDPE crystals than the smaller particles. Gallagher and McDonald [13], observed a similar increase in crystallinity from 35 to 41% for 50% maple based WPC going from <73 µm to 150-178 µm sized particles. It is speculated that the composites made with <2 mm particles, with a lower packing density than the <0.5 mm fibers, may have more interstitial space to form spherulites and thus a higher crystallinity [33]. The melt temperature (T_m) for the composites were the same at 132°C. The crystallization temperature were similar at around 119°C.

Table 5: Crystallization and melt temperatures and crystallinity of corncob plastic composites made with <0.5 and <2 mm particles

Corncob composites	T_c (°C)	T_m (°C)	Crystallinity (%)
<0.5 mm particles	118.4 (0.1)	132.2 (0.1)	65.9 (5.4)
<2 mm particles	118.9 (0.2)	132.1 (0.3)	70.3 (1.1)

Standard deviations are given in parentheses.

The storage modulus (E') of the corncob composites and HDPE was determined by DMA and the thermograms are shown in Fig. 5 and values at 25°C are given in Table 6. The corncob composites made with <0.5 mm particles had the highest E' (at 25°C) value at 56.8 MPa, followed by the composites made with <2 mm particle at 25.1 MPa, and HDPE had the lowest at 19.3 MPa. The damping factor (tan δ) is also measured by DMA on viscoelastic materials and can be used to give information about the interface between different phases (particle-plastic) in composite materials that affect energy dissipation. The effect of the corncob particle size on the strength of the particle-HDPE interface was evaluated using *A* and determined from DMA data (tan δ) according to Kubat et al. [26]. The *A* values for the corncob-HDPE composites at 25 and 40°C are given in Table 6. Low *A* values means a high degree of interaction (improved adhesion) between the two phases [24,26]. The corncob composites made with <0.5 mm corncob particles had the lowest *A* (0.28 at 25°C), or good interfacial interaction, compared to composite made with <2 mm corncob particles (0.33 at 25°C). These results are consistent with the flexural properties and storage modulus (E') data. This trend of getting better adhesion (lower *A*) using smaller particles was also observed in bamboo-plastic composites [24]. This could be attributed to an increased surface area with the smaller particles and not likely attributable to aspect ratio of the particles since they were similar (~2). However, these results are in odds with an increase in HDPE crystallinity for the composites made with the larger particles and cannot be explained.

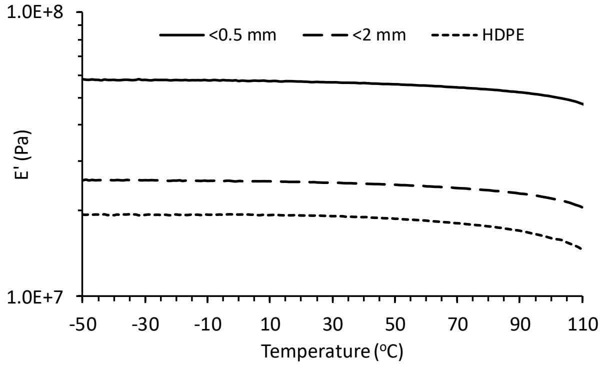

Fig. 5: DMA thermograms of storage modulus (E') of HDPE and corncob plastic composites made with <0.5 and < 2 mm particles

Table 6. Adhesion factor (A) and storage modulus (E'), determined by DMA, of corncob plastic composites made with <0.5 and <2 mm particles.

Corncob composite	*A* at 25°C	*A* at 40°C	E' (Pa) at 25°C
<0.5 mm particles	0.28	0.25	5.67×10^7
<2 mm particles	0.33	0.29	2.51×10^7
HDPE			1.93×10^7

IV CONCLUSIONS

Extruded natural plastic composites were successfully produced from different sized particle fractions of corncobs. The extruded composites possessed moderate strength and sorption properties and were thermally stable. Differences in the densities of the composites and fiber size appeared to affect the properties of the fabricated corncob plastic composites. Composites produced with smaller particle size (<0.5 mm) had superior strength and sorption properties attributable to enhanced interfacial bonding with the plastic matrix. The fabricated plastic composites corncobs are suitable as building components in Nigeria and the region. For higher performing natural plastic composite materials smaller corncob particles should be used.

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