

Preparation and physico-chemical study of fire retardant from chicken feathers and PP/PE composites

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Abstract. Chicken feathers were used to reinforce low density polyethylene (LDPE) and polypropylene (PP). The physico-mechanical and flame retardation properties of the composites were investigated. Properties such as tensile strength, elongation at break, flexural strength, and modulus as well as water absorbency were determined and the results obtained were analyzed. The composites were further characterized by SEM and FTIR analyses to study the surface morphology and evidence of composite formation respectively. The Young modulus of reinforced composites were greater than that of virgin samples. The fiber/Polypropylene samples were observed to have the highest value of Young modulus, with a value of 133.33 GPa, and tensile strength value of 16.93 MPa. TGA and flame retardant tests were carried out to investigate the flame propagation and thermal properties of the composites. The results showed that the mechanical properties of composites from polypropylene were better enhanced than those from the composites of polyethylene, with an overall increase in mechanical properties when compared to the virgin polymers used. From the TGA and flame retardant test, it was observed that the more the fiber load the more the formation of char layers in the samples, hence retards the flame formation and reduced prolonged burning and flame propagation.

Keywords: chicken feathers; flame retardant; tensile properties; water absorption.

1. Introduction

Billions of metric tons of chickens are being consumed on daily basis around the world and feathers from the chicken end up as waste and pollutants to our environment. Recently, these and many other reasons have drawn the attention of some researchers on how best these feathers can be recycled and utilized. Chicken feathers are potential raw materials of high economical and scientific value; this is due to its low cost as waste, large availability and chemical composition. Feathers are among the most complex integumentary structure found in vertebrates and are formed in tiny follicles in the epidermis, or outer skin layer, that produce keratin proteins [1]. Chicken feathers have become of relative interest, presenting a cheap and readily available alternative to their synthetic counterparts.

Flame retardants are activated by the presence of an ignition source and are intended to prevent or slow the further development of ignition by a variety of different physical and chemical methods. To prevent polymeric materials, whether in bulk or in fiber/textile forms, from burning, the presence of a flame retardant is one of the most effective methods which improves resistance to ignition, reduces flame propagation rate, elevates ignition temperature and prevents continuous burning [2].

The major aim of using flame retardants is not to render the polymer completely non-flammable but to reduce flammability sufficiently enough to provide time for people to be saved from fire incidence and minimize the risks of death and injuries. Fire retardants are being applied to materials to minimize their inflammability or self-extinguishing. In fact, they are of different kinds that are usually applied on other materials that we come in contact-with in everyday life; such as building supplies, furniture, plastic molding materials, clothing, mattresses, bedding, and a variety of other materials. The principle of flame retardants is to decrease its propensity to burn when subjected to a heat source or open flame [3]. Flame retardants should inhibit or suppress the combustion process during particular stages of the fire process e.g. heating, decomposition, ignition, or flame propagation [2].

Chemical treatment can further improve flame retardation of chicken feather by producing a flame retardant chicken feather fiber [4]. Previous studies have been reported the use of chicken feather to optimize composites with enhanced flame retardation using epoxy [5], polymeric composites [2] and reinforced polyethylene [6].

In this present study, chicken feathers were converted into fiber reinforced composites, their mechanical properties were determined and characterization methods were carried out. A composite material is produced from two or more constituent materials that have notably dissimilar chemical or physical properties, and are mixed to form a material with properties that differ from the individual elements. In a composite, the individual elements remain separate and distinct, this properties distinguish composites from mixtures and solid solutions [7, 8].

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2. Experimental

2.1. Materials

Polypropylene (PP) and Low Density Polyethylene (LDPE) were obtained from Plasmatix Plastics Katsina, Nigeria. Ethanol (C₂H₅OH, 98%) and sodium hydroxide (NaOH) (Sigma-Aldrich) were used as received. Chicken feathers were collected from a local poultry farm at Ajiwa village, Katsina, Nigeria.

2.2. Preparation of materials

The collected chicken feathers were washed and sorted out of foreign matters. The sorted chicken feathers (2 kg) were weighed and sun-dried to a constant weight of 1.45 kg to remove water from the feathers. The fibers were manually extracted by detaching the hairy parts from the rachis (feather core).

2.3. Chemical modification of the chicken feather fiber The prepared chicken feather fiber (CFF, 950 g) was soaked in 98% ethanol (3000 cm^3) and stirred for 30 min. It was rinsed severally and dried to a constant weight of 905 g [9].

The ethanol treated fiber (900 g) was dispersed in 5000 cm³ of 1% NaOH solution and allowed to stand for 2 h at room temperature. The fiber was separated from the solution using a sieve (150 micrometer) [10].

The residue (modified chicken feather fiber) was rinsed severally with distilled water until the filtrate becomes pure and neutral to pH paper. The final residue was weighed, dried under sun for 72 h until a constant weight of 825 g was obtained.

2.4. Formulation of composites

The matrix was compounded respectively with different fillings in different ratios. The samples formulations were carried out using the weight ratio of feather to polymer matrix as: 1:5, 3:5, 1:1, 5:3, 5:1, and are designated respectively as LS1, LS2, LS3, LS4 and LS5 for the composites produced with LDPE; while PS1, PS2, PS3, PS4 and PS5 for the composites produced with PP.

Different samples of the composites were produced by melt-mixing process at a temperature of 150 °C for 3 min. Upon achieving a band formation of the LDPE on the front roll, a specific weight of chicken feather fiber (CFF) was manually added to the bank as the rolls rotate at a rate of 50 rpm and allowed to mix to homogeneity for 5 min. The composite was sheet out and labeled accordingly. The procedure was repeated for the samples containing PP matrix at a temperature of 180 °C.

2.5. Tensile strength test

The tensile test was carried out using the Hounsfield Monsanto Tensometer (model no: 386083-W9) according to ASTM D-638 [11]. The samples were subjected to a tensile force and tensile properties such as tensile strength, strain, modulus for each sample were calculated using the equations below:

$$Tensile Strength = \frac{F}{bd}$$
(MPa) (1)

where F is maximum tensile load, b is sample thickness, d is sample width;

$$Strain = \frac{L_0}{L} \tag{2}$$

where L_o is extension, L is gauge length.

2.6. Flexural test

The flexural test for the composites was carried out in accordance with ASTM D-790 [12]. The specimen measuring 100 mm x 25 mm x 10 mm was placed on a support span horizontally and a steady load was applied to the center by the loading nose, producing a three-point bending until the sample specimen failed. The maximum load (N) and the corresponding deflection (mm) were recorded accordingly. The flexural strength was calculated using Equation 3:

$$Flexural Strength = \frac{_{3FL}}{_{2bd^2}}$$
(MPa) (3)

$$Flexural Modulus = \frac{6FL}{hd^{2D}} (MPa)$$
(4)

where: L = distance between the support spans at both edge of the specimen; b = sample width; d = sample thickness; D = sample deflection; F = maximum load at break.

2.7. Thermogravimetric analysis (TGA)

A PerkinElmer Thermal analysis machine was used. A heating rate of 10 °C/min was used in this analysis, at a flow rate of nitrogen gas (inert atmosphere) of 60 mL/min and a temperature range of 20 °C to 1000 °C.

2.8. FTIR analysis

The analysis was carried out using Agilent Cary 630 FTIR spectrometer, at a range of 4,000.00 - 650.00 cm⁻¹.

2.9. Flame retardant test

The method used for the flame retardant test was adopted from Bansal and Singh [13] with slight modification. The flame retardant characteristic of the composites was tested in the horizontal burning setup. The samples with dimensions of 100 mm x 10 mm x 5 mm were held horizontally in the horizontal burning setup. The samples were ignited by the external fuel gas from one of the ends. To determine the linear burning rate of the samples, a stopwatch was used for calculating the time taken for each sample to burn from 10 mm to 100 mm. The rate of burning is calculated using Equation 5 [14-16]:

Burning rate =
$$\frac{D_p(mm)}{P_t(s) - I_t(s)}$$
 (5)

where D_p is the flame propagation distance (mm), P_t is the flame propagation time measured in seconds and I_t is the ignition time measured in seconds

2.10. Water absorption test

The specimens were prepared to a dimension of 50 mm x 50 mm x 3 mm. Water absorption test (ASTM D570) [17] was done by total immersion of specimens in distilled water at room temperature for 2 hours, then at 24 hours interval until the samples became saturated [18]. The water absorbed was determined by weighing the samples after 24 hours. The water uptake in the composite was measured using the relation [19, 20]:

Water absorbed (%) =
$$\frac{W_f - W_i}{W_i} \times 100$$
 (6)

where W_i = initial weight (g), W_f = final weight (g).

3. Results and discussion

3.1. Tensile properties

The results of the flexural properties are shown in Fig.1 and the same trend was observed for both polymers.



Figure 1. Comparison of flexural strength for pure and reinforced composites of PP/LDPE

The pure PP sample had the highest flexural value. There isn't much increase from PS1 to PS2, but it was observed that there was a decrease from PS3 to PS5 for PP composites. Similar trend was observed for LDPE samples but with a decrease in value from LS2 to LS5. Analytical models used to calculate flexural strength values assume good fiber dispersion, and the results are likely influenced by the fiber dispersion in these composites [21]. The pure samples showed a flexural strength of 21.79 MPa and 36.21 MPa for LDPE and PP respectively. LS2 and PS2 reinforced composites showed the highest flexural strength among the fibers reinforced composite with a value of 22.16 MPa and 35.78 MPa, while LS5 and PS5 showed the lowest flexural strength in both polymer samples, with a value of 15.8 MPa and 23.14 MPa. On the other hand, samples LS3 and PS3 showed a flexural strength value of 19.61 MPa and 35.12 MPa for both LDPE and PP, followed by LS1 and PS1 which were 20.08 MPa and 34.42 MPa respectively.



Figure 2. Comparison of Young modulus for pure and reinforced composites of PP/LPDE

The Young modulus value was determined from stress – strain data in accordance with ASTM – D638, for composites which were tested in uniaxial tension. As expected, Young moduli of reinforced composites were greater than young modulus of virgin samples. From Fig. 2, it was observed that PP sample PS3 (50:50) possessed the highest Young modulus value of 133.33 MPa followed by the sample PS5 with a value of 128.18 MPa. This implies over 80% enhancement in the tensile modulus compared to virgin sample that has a value of 72.55 MPa for virgin PP. Tensile modulus tends to increase as the treated fiber content increases from PS1to PS3.

This similar trend is observed in LDPE samples in LS3 (50:50 matrix to fiber loading) which possessed the highest Young modulus value of 93.33 MPa followed by sample LS5 with a value of 96.87 MPa. This implies over 90% enhancement in the tensile modulus compared to the virgin sample that has a value of 36.87 MPa. Tensile modulus tends to increase as the treated fiber content increases from LS1 to LS3 and also PS1 to PS3.

PP composites gave the best results as compared to LDPE composites. This is an indication that stiffness of fibers enhances the overall stiffness of fibers reinforced composites [18].



Figure 3. Comparison of tensile strength for pure and reinforced composites of PP/LPDE

The results of tensile properties are represented in Fig. 3. From the results, it was observed that tensile strength of the composites from PP was better enhanced than those from the composites of LDPE. Generally, it has been observed that tensile strength of composites decreased as fiber content increased from LS3 to LS5, and PS3 to PS5. The highest value is observed in sample PS1 for PP (which has 5:1 matrix to fiber loading) with a value of 16.93 MPa followed by sample PS3 with a value of 13.60 MPa. This shows that PS1 reinforcement has the optimum value for tensile strength enhancement with chicken fiber. The results showed that fibers reinforced samples gave 14.3% enhancement compared to the virgin sample that has a value of 14.80 MPa.

The tensile strength of the LDPE composite samples shows a different trend in properties compared to PP composites. It was observed that tensile strength of composites decreased as fiber content increased from LS3 to LS5. The best in this regard was composite sample denoted as LS3 (which has 50:50 fiber loading) with a value of 11.20 MPa followed by sample LS2 with a value of 10.4 MPa. This shows that LS3 sample shows the highest value for polyethylene tensile strength enhancement with chicken fiber. The results showed that fibers reinforced samples gave 14.8% enhancement compared to the virgin sample that has a value of 5.70 MPa.

3.2. SEM analysis

Figures 4.a and 4.d are the images of the virgin samples (LDPE and PP respectively) and have shown a thick coated surface of the polymeric materials. In Fig. 4.b and e, where the fiber loading is less, the samples are shown to be well dispersed in the polymer matrix as a result of good compatibility and miscibility between both materials which aid proper adhesion at the interface.



Figure 4. SEM images of (a) LDPE (b) LS1 (c) LS5 (d) PP (e) PS1 (f) PS5

On the other hand, samples containing fiber reinforcement show a rougher and porous surface (Figure 4 b, c, e, and f). These morphological changes observed in the samples show a successful impregnation and dispersion of the fibers onto the polymer matrices which in turn affects the mechanical properties of the composites [15]. The microscopic pores observed in the images are responsible for appreciable water absorbency of the composites.



Figure 5. FTIR spectra of PP (A), PS1 (B) and PS5 (C)



Figure 6. FTIR spectra of LDPE (A), LS1 (B) and LS5 (C)

Figures 5 and 6 shows the spectra of pure polymer samples and the composites. The chemical nature of individual components regulates the chemical properties of fabricated composites. On the other hand, the feathers have little effect on the chemical structure of polymers as compared with the virgin polymer. In Fig. 5 the samples show characteristic absorption peaks at 2918 and 2851 cm⁻¹ due to C-H stretching respectively and an additional peak at 1380 cm⁻¹ due to C-H bending. In Fig. 6, the absorption bands show the characteristic strong absorption peaks at 2918 and 2851 cm⁻¹ due to C-H stretching respectively, and an additional peak at 1465 cm⁻¹ due to C-H bending modes. Moreover, samples PS1 and PS5 in Figure 5 and LS1 and LS5 in Figure 6 show another important peak at 1380 cm⁻¹ and 1580 cm⁻¹ which is associated with amide group, but the peak is absent in PP.

3.3. Flame retardant test

Tables 1 and 2 show the data obtain from horizontal burning test. From the figures, it can be observed that as the amount of fiber is increased in the sample, the time required for it to burn increases and in turn, the linear burning rate decreased. Hence, at 5:1 feather to matrix loading in composite, we find the least linear burning rate. This is mainly due to the fire resistance property of the composite. The dehydration process produced substantial amount of water and char during combustion. Generally, a reasonable amount of fiber is required to form char layer while the amount of matrix is kept small. Hence, as the percentage fiber weight increases, the fire resistance property also increases. From this it can be deduced that there is synergic effect of fiber reinforcement on the polymer matrix and fire retardation activity.

 Table 1. Linear burning and weight loss rate of composites from CFF and PP

Sample	Time (min)	Linear burning rate (mm/min)
PP	1.49	33.56
PS1	0.58	86.20
PS2	1.58	31.65
PS3	2.23	22.24
PS4	3.02	16.56
PS5	3.23	15.48

 Table 2. Linear burning and weight loss rate of composites from CFF and LDPE

Sample	Time (min)	Linear burning rate (mm/min)
LPDE	1.40	35.7
LS1	0.52	96.15
LS2	1.43	34.97
LS3	1.45	34.48
LS4	2.03	24.63
LS5	2.44	20.49

3.4. Water absorption

The variation in the water absorption of the composites and the virgin samples is shown in Tables 3 and 4. From the result obtained, water absorption increases as the chicken feather fiber loading increases in the composites while it tends to increase as the immersion time increases. This implies that CFF is hydrophobic. Water absorption is low for pure sample as compared to composites sample because there have been creation of pores and reduced intermolecular forces in composites which allow aqueous substances to be trapped within the structural framework.

Table 3. Water absor	ption of com	posites from	CFF and LDPE
Initial	Final	Water	Water

Sample	weight (g)	weight (g)	absorbed (g)	absorbed (%)
LPDE	4.4210	4.4391	0.0041	0.409
LS1	4.5342	4.6175	0.0184	1.84
LS2	3.8543	3.9400	0.222	2.22
LS3	4.2007	4.4948	0.0700	7.00
LS4	2.2692	4.8690	0.1405	14.05
LS5	4.4925	5.2826	0.1759	17.59

Table 4. Water absorption of composites from CFF and PP

Sample	Initial Weight (g)	Final Weight (g)	Water Absorbed (g)	Water absorbed (%)
PP	2.5392	2.5480	0.0035	0.346
PS1	3.1281	3.2144	0.0276	2.759
PS2	2.1994	2.2679	0.0311	3.115
PS3	3.2401	3.4731	0.0719	7.190
PS4	3.4121	4.0253	0.1797	17.970
PS5	3.7711	5.1014	0.3528	35.39

As observed from Table 3, LDPE have lower percentage water absorption with 0.409% compared to the composites with 1.84%, 2.22%, 7.0%, 14.05% and 17.59% for LS1, LS2, LS3, LS4 and LS5 respectively. Same is seen in Table 4.

This signifies that the presence of feather in the composite makes it dense and contained small pores that allow the penetration of water. This result is in line with the similar works reported [17], in which the water absorption, flexural properties and morphological characterization of chicken feather fiber-wood sawdust hybrid reinforced wastepaper cement bio-composites show a similar trend with what has been observed.

3.5. Thermal analysis

The thermal behavior of the composites was examined by TGA under nitrogen heated at the rate of 10 $^{\circ}$ C/min.

Figures 7, 8 and 9 show the thermal analysis of LPDE, LS1 and LS5 which exhibits a similar thermal behavior with PP and PS1with four steps pattern. First is the onset of decomposition at 239.231 °C, 331.71 °C, 302.54 °C with about 15%, 8%, 7% weight loss for LPDE, LS1 and LS5. The second stage decomposition is 368.87 °C, 370.32 °C and 347.47°C with 32%, 31% and 34% weight loss for LPDE, LS1 and LS5 respectively. The third stage decomposition is 457.13 °C, 413.88 °C and 397.23 °C with 48%, 41% and 40% weight loss for LPDE, LS1 and LS5 respectively. The third stage decomposition is 532.64 °C, 496.73 °C and 481.47 °C with 80%, 87% and 347.8% weight loss for LPDE, LS1 and LS5 respectively.



Figure 7. TGA/DTG of virgin LDPE at 10 °C /min



Figure 8. TGA/DTG of LS1 at 10 °C/min



Figure 9. TGA/DTG of LS5 at 10 °C/minute

Figs. 10, 11 and 12 show the TGA/DTG curves for PP, PS1 and PS5 which exhibits a four-step thermal behavior. The onset is a decomposition at 332.83 °C for PP and 288.47 °C PS1 with loss in weight of 18% for PP and 4% for PS1, which is due to loss of volatile compounds and also due to dehydration of the sample. Another decomposition is observed at a second stage at a temperature of 379.15 °C for PP and 375.89 for PS1, resulting in a weight loss of 35% for PP and 34% for PS1 due to presence of volatile content in the sample. The third stage shows a decomposition at temperature of 436.33 °C for PP and 401.58 °C for PS1, with a loss in weight of 53% for PP and 30% for PS1 this can be attributed to the degradation of filler in the composite material before the final degradation of the matrix.



Figure 10. TGA/DTG of PP at 10 °C/min



Figure 11. TGA/DTG of PS1 at 10 °C/min



Figure 12. TGA/DTG of PS5 at 10 °C/min

The results shows that the composites have good thermal stability as well as the pure samples except for PS5 which can be attributed to have resulted from the disruption and decrease in the intermolecular hydrogen bonds in the composites.

4. Conclusions

The results showed that the mechanical properties of composites from PP were more enhanced physicomechanically than those from the composites of LDPE. Moreover, there was an overall increase in mechanical properties when compared to the virgin polymers used. The TGA and flame retardant test showed that the more the fiber load the more the formation of char layers in the samples, hence retards the flame formation and reduced prolonged burning. This property is attributed to the fire resistance property of the CFF. The water absorption test showed that water absorption increases with more fiber content.

Conflict of interest

Authors declare no conflict of interest.

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