THERMAL AND MECHANICAL PROPERTIES OF HIGH-DENSITY POLYETHYLENE (HDPE)/LEATHER COMPOSITE

E. T. MUSA[1], A. HAMZA[2], A. S. AHMED[3], U. S. ISHIUKU[4] AND E. G. KOLAWOLE[5]
[1]Department of Chemical Science, Federal University, Kashere
[2]Department of Chemistry, Ahmadu Bello University, Zaria
[3]Department of Chemical Engineering, Ahmadu Bello University, Zaria
[4]Department of Textile Science and Technology, Ahmadu Bello University, Zaria
[5]Department of Chemistry and Industrial Chemistry Bowen University, Iwo
[musatesther@gmail.com; 07084372016]

ABSTRACT

The possibility of using solid tannery waste in HDPE was studied by examination of the mechanical properties, morphology and thermal properties of the composites produced. Composite was manufactured by compression moulding technique for varying fibre contents from 10% to 60%. Mechanical and physical tests were conducted on composite specimens in accordance to ASTM D638. A partial replacement of 10-40 wt% of HDPE (petroleum-derived plastics) was successful with tensile value of 118.868, 100.971 MPa at 10 wt% for composite of HDPE filled chrome waste and HDPE filled vegetable re-tanned waste, while HDPE (control sample) at 10 wt% gave 25.338 MPa only, The optimal value for tensile strength, and elastic modulus was obtained at filler loads of 10wt%. The morphology and thermal properties of the matrix and composites were studied by scanning electron microscopy, thermogravimetric analysis, thermal differential analysis and differential scanning calorimetry. The TGA and DTA results of composites were more thermally stable with additives than the control or it's composite without additives. The thermal differential analysis of composites without additives displays two steps weight losses while composites with additives display a single step weight loss. In conclusion, the additives does improve the mechanical properties of HDPE/Leather solid waste, it greatly helps to stabilize the composite against thermal and photo degradation. HDPE90/Vegetable waste10 were shown to have better properties than those of Chrome tanned and Hide un-tanned waste.

Keywords: *Leather waste; Mechanical properties; Thermal stability; Composite; Additives.*

1.0 INTRODUCTION

No plastics can reliably function even briefly at a temperature above the softening or heat distortion temperature of the base polymer. The large coefficient of linear thermal expansion (CTE) of polymer is much larger than those of metals and ceramics in the range of 20 ppm K^{-1}, this at present is suppressing the utility value of polymer. It was reported by Koshi and Masayoshi, 2014 that the coefficient of linear thermal expansion (α) of polymer is within 30 to 40 ppm K^{-1} or at most over 100 ppm K^{-1}. In most cases, polymer matrix composites (PMCs) filled with low-expansion inorganic materials control thermal expansion in polymers. Modification of properties and process ability of polymers through addition of other substances is a field huge in scope and fast moving in character (John and Richard, 2005). To enable polymer (plastics) compete with or even overshadow light metal and alloys in stress and safety sensitivity in engineering and domestic applications, and be biodegradable, the prospective polymer is modified or/and compounded with additives, or/and make composites through fibre reinforcement with judicious incorporation of suitable additives (Hollaway, 1993). Where a desired balance of thermal and mechanical properties is the basic requirement for the material, thermal analysis, a branch of material science where the properties of materials are studied as they change with temperature are employed. The effect of the many additives used (including stabilizers) can be determined using differential scanning calorimeter (DSC) ASTM D3418 standard method. Thermogravimetric analysis and/or thermal differential analysis are employed to determine thermal stability and the percent weight loss of a test sample. The loss in weight over specific temperature ranges provides an indication of the composition of the sample, including volatiles and inert filler, as the sample is being heated at a uniform rate in an appropriate environment.

The improved properties by addition of particles can be achieved when there is good interaction/ dispersion between the fibre and the matrix. Additives are, therefore, often added by melt processing under control temperature to promote intercalation between fibre-matrix bonds and to protect them from thermal degradation. Mechanical properties of polymer can be improved by mixing the polymer with some form of rigid filler (Mascia, 1974; Lutz and Richard, 2005). Composites, such as fibre reinforced polymer are materials that consist of at least two different phases (Gowariker *et al*., 2007) combined together, of which each retained its properties to create new material with properties that cannot be achieved by any one component acting alone. For example without the addition of antioxidants, few plastics would have a brief life; most plastics would have none, because articles of commerce could not be fabricated without irreversible destruction. Polyethylene, for which cross-linking predominates over chain scission, require 0.025 to 0.05 percent (%) of either calcium stearate hydrotalcite or blend (an acid catalyst scavenger) as anti-degradants (Mascia, 1974; Richard, 2001). Plasticizers are used in polymer to promote movement of chains and segments of amorphous polymer while flame retardants are used to minimize the susceptibility of polymer materials to inflammability (Mascia, 1974), renewable materials are added to polymer to make them biodegradable. The non-gradation of synthetic polymer after its service life (Mohanty *et al*., 2005) is a major environmental issue that has led to the need in recent years, to develop materials that are environmentally friendly, 'green' as a partial or full replacement of petroleum-derived plastics (Mohanty *et al*, 2005) and the need for material properties to meet exacting end-use requirement (Musa *et al*., 2014) by scientists and manufacturers. Composite research development has become one way of harmonizing the need of the present day-technology with material property requirement and one of the way of controlling urban and industrial waste and/or residue. This study is aimed at converting solid waste in the tannery into usable material which would be of domestic and industrial value.

2.0 MATERIALS AND METHODS

2.1 Materials

Leather waste was collected locally from the tannery at the Nigerian Institute of Leather and Science Technology (NILEST), Zaria. High-density polyethylene (HDPE) commercial grade (EIPIN) was of Indorama Chemical Company Port-Harcourt, Nigeria. Trimethyl quinoline (TMQ), Magnesium hydroxide [$Mg(OH)_2$], acrylic acid, sodium hydroxide (NaOH), titanium dioxide (TiO_2) and ethylene vinyl acetate (EVA) copolymer, were analytical-reagent grade of (M&B), GMS and Aldrich Chemical. Natural rubber (NR) was also locally obtained from commercial market (Samaru), Zaria.

2.2 METHODS

2.2.1 Hide preparation

Tannery waste collected were sorted out first according to chemical substance of tanning used, those tanned by vegetable tanner(*acacia nilotical*) were collected, cleaned, sun and oven dried then ground to 0.5 mm particle size using an Arthur Thomas Wiley laboratory Mill (model 4) . The ground specimen as preserved in clean dried thermoplastic transparent bucket with lid as the bulk reinforcement. In like manner, the chrome tanned waste and hide un-tanned waste were prepared and preserved and kept as the bulk reinforcement. The high-density polyethylene (HDPE) and additives were used as received. The different proportions of the matrix/reinforcements are as shown on Table 1 and Table 2

Table 1: The formulation used in making the various matrix/reinforcement composites (Turu *et al*., 2014)

Matrix	**Reinforcement**		
High Density poly ethylene (HDPE) (wt%)	1st Expt. Hide powder (wt%)	2nd Expt. Chrome tanned and re-tanned with syntax (wt%)	3rd Expt. Chrome tanned and re-tanned with vegetable (wt%)
100	0	0	0
90	10	10	10
80	20	20	20
70	30	30	30
60	40	40	40
50	50	50	50
40	60	60	60

Table 2: The Formulation used in making the various matrix/reinforcement with additives (A) (Turu *et al*., 2014)

Matrix	**Reinforcement Plus additives**		
High Density poly ethylene (HDPE) wt%	1st Expt. Hide powder (wt %) A	2nd Expt. Chrome tanned and re-tanned with syntax (wt%)A	3rd Expt. Chrome tanned and re-tanned with vegetable (wt%)A
100	0	0	0
90	10	10	10
80	20	20	20
70	30	30	30
60	40	40	40
50	50	50	50
40	60	60	60

where A = 0.5g trimethylquinoline (TMQ), 3.5g$Mg(OH)_2$, 0.3g TiO_2, 10g natural rubber (NR), 2g ethylene vinyl-acetate copolymer (EVA) and 2ml of acrylic acid respectively.

The wet/raw or green hide obtained from National Animal Production Research Institute (NAPRI) was washed in a salt solution and then in distil water. It is then limed, bated, degreased and dehydrated with acetone. The treated sample was cut into150 mm² size and dried at a temperature of 50 °C in an atmosphere of 25 ^{0}C and 65 ^{0}C before it is ground into powder using a Thomas Wiley laboratory mill (model 4), in accordance to SLC 04-07 (Sarker, 2005). Few pieces of the sample were grinded and discarded to clean the apparatus before proceeding. The ground sample was stored in a clean, dry and air tight container. This was the bulk sample for analysis. From it, samples were drawn to maintain the same state of moisture content whenever a portion is taken for analysis. These operations remove fats and other proteins normally present in the hide leaving only collagen. For instance, according to Sarkar (2005), soaking and washing remove albumin, washing in salt dissolves globulins which are removed later by washing; liming removes keratin and some extent mucoids while bating removes the remaining inter-fibrillary matters and any probable elastin; while treatment with acetone removes fats and dehydrates the hide.

2.2.2 Compounding

Each composite formulation on Table 1and 2 were picked one after the other and compounded at 180°C for 7 to 9 minutes using Two-Roll Mill. The rolls speed for the rear and front were 48 and 5 revolutions per minute (rpm). The compounding was done in Zaria- Nigeria at atmospheric room temperature (27 ^{0}C) in accordance with ASTM D639. The compounded sample was stored in a labelled black polythene bag. To each formulation on Table 2, 0.5 g trimethylquinoline (TMQ), 3.5 g $Mg(OH)_2$, 0.3 g TiO_2, 10 g natural rubber (NR), 2 g ethylene vinyl-acetate copolymer (EVA) and 2ml of acrylic acid were respectively added first to the matrix on the bank formed between the two rolls and mix for 3 minutes before the appropriate quantity of fibre was gradually added until the mixture uniformly mixed in other to modify the formulation on Table1.

2.2.3 Compression moulding

A thin aluminum sheet was used as the mould through which film of composite were pressed. Each compounded formulation, 1.5g in weight, was measured out, and then wrapped in a cut foil-paper. The wrapped sample was then placed in an aluminum sheet (mould) then placed on the moveable platens of the hot press. With the help of the hydraulic controlled ram, the lower platen containing the compounded sample moved up to the upper platen closing the mould and exerting moulding pressure (Musa *et al.,* 2014). The films were compressed at a compression temperature and pressure of 180 to 185 ^{o}C and 3×10^3 N/m² within 3 minutes preheating and 5minutes pressed respectively. After 8 minutes, the pressure was released on the lower platen falling by gravity. For each compounded formulation, 10 samples were pressed into thin film of 0.4 to1.0 mm thickness. In addition, 15 more samples, Dog-bone in shape were produced having dimension 100 mm × 40 mm × 3 mm for the different type of waste (HDPE/Hide (un-tanned) waste, HDPE/Vegetable (*Acacia Nitotica*) (*Bagaruwa*) tanned waste and HDPE/Chrome tanned waste with trimethyl quinolene (TMQ), titanium oxide (Ti_2O), magnesium hydroxide [$Mg(OH)_2$] acrylic acid, ethylene vinyl-acetate copolymer and natural rubber (NR). the additives are collectively designated as A for simplicity.These samples are stored separately in different containers, drawn later for analysis.

2.3 CHARACTERIZATIONS OF SAMPLES

2.3.1 Tensile test

The strength of the composites, which is a measure o force required to break the composite and the extent to which the composites stretches or elongates before breaking was carried out using a Hounsfield Tensometer (Model W3179) in accordance with ASTM D638.

2.3.2 Impact test

The composite sample was clamped rigidly at one end of a Ceast Izod impact machine (model 957) and struck at the other end by a pendulum weight, to break the sample in according to ASTM D 256-05 recommendation. The kinetic energy of the pendulum (read on the machine and recorded) was the energy used to break the composite sample. The impact was calculated from the equation given below.

Impact = Energy of pendulum/thickness composite (J/mm^2)

2.3.3 Thermo gravimetric analysis (TGA) (ASTM E1131)

Thermogravimetric analysis (TGA) was the method used to study the thermal stability of resulting composites using a thermogravimetric analyzer TGA-50; (Shimadzu Corporation (Japan)). The heating rate was 10 ^{0}C/min, decomposition temperature under nitrogen gas atmosphere was from 0 - 800 ^{0}C.

The composite samples were enclosed in a 160 µl aluminum crucible for testing. The mass of the sample was determined first by weighing the crucible before and after the addition of the sample.

2.3.4 Morphological test

The crystallizing behaviour of waste from hide un-tanned, chrome tanned and vegetable tanned of the composites was measure using a differential scanning calorimeter (DSC). Samples of 5.6 to 6 milligrams were taken and placed in aluminum capsules and were heated from 0 - 200 ^{0}C at rate of 10 °C/minute. Plots of heat flow versus temperature were produced from which the resulting thermal scan is then analyzed.

2.3.5 Scanning electron microscopy (SEM) test

The morphology of the composites was studied using a JSM-5600 LV (Jeol, Tokyo, Japan) scanning electron microscope. The fracture surfaces of the composites after tensile test were mounted on a metal stub and sputter-coated with gold in order to make the sample conductive before analysis. Morphologies of the composite samples before and after modification with additives were studied. Scanned images were taken at an accelerating voltage of 2.0 and15.0 kV.

3.0 RESULT AND DISCUSSION

3.1 Tensile Strength

From the result of Figure 1a it shows that the breaking strength for HDPE/Chrome tanned waste, HDPE/Vegetable tanned waste and HDPE/Hide un-tanned waste decrease with an increase in waste content and indicating that strength of the control is higher with value of 25.338 MPa at 10 wt% than the different of composite samples. This agrees with research work carried out by Justin *et al* (2005); Ambrósio *et al*, (2011) without modifying the natural fibre used. The decrease in tensile strength could be due to void formation during processing, which lead to micro crack formation under loading and thereby reduces breaking strength or due poor dispersion of the filler in the matrix leading to poor interaction between fibre and matrix as stated by Sukhdeep *et al* (2014) when there is imcompatibity between fibre and matrix.

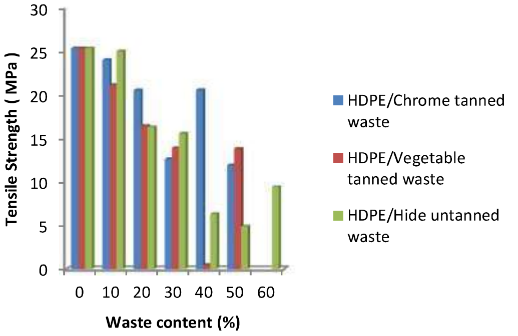

Figure 1a: Tensile strength of composites without additives

The result on Figure 1b shows that additives modified both HDPE/Chrome tanned waste and HDPE/Vegetable tanned waste content from 0 - 30 wt% and 0 - 40 wt% with HDPE90/Chrome tanned waste10 having the highest value of 118.868 MPa. The composite samples tensile properties are better than control sample and the unmodified formulation in Figure 1a. HDPE/Hide un-tanned waste shows a decrease in tensile strength even with additives and increase in waste content. The tensile strength of HDPE90/Chrome tanned waste10 (A) and HDPE90/Vegetable tanned waste10 (A) indicates that the additives had improved the interaction between the waste (fibre) and matrix. This observation agrees with results of Daniella *et al* (2009) and Ku *et al* (2011). The decrease in tensile strength at higher waste content could be attributed to poor adhesion between waste (fibre) and the matrix due insufficient wetted surface area that lead to micro crack formation at the interface.

3.2 Yield Stress

The ability of composite sample to undergo reversible deformation or carry stress without suffering from a permanent deformation is describe by the measure of its yield strength. The yield strength in Figure 2a for HDPE/Chrome tanned waste and HDPE/Vegetable tanned waste generally was lower than the control sample, meaning the control has better yield except the yield of HDPE90/Hide un-tanned waste10. The low yield strength could be attributed to the cohesive forces existing in the filler i.e. the fiber as a result of strong hydrogen bonding holding the fibre together more than the adhesive forces existing (mechanical bonding) at the fiber-matrix interface. However, the yield strength of HDPE90/Hide un-tanned waste10 could be as a result of irreversibly hydrolyzed gelatin or waxy found in raw hide and a skin adding to its wettability, thereby improving interfacial bonding, suggesting that mechanical interlocking exists within the fibre-matrix interface making the adhesion forces stronger than cohesive force. However this wetted area was available for only fiber-matrix of 0-10 wt% beyond which, a weak interface that cannot effectively transfer stress to the fibres by the matrix resume. Insufficient wetted area results in interfacial defects which act as stress concentration. Fibre wettability has been shown to affect the toughness, tensile and flexural strength of composites as reported by Pickering *et al* (2015).

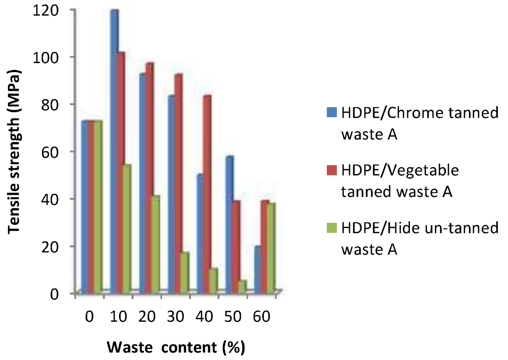

Figure 1b: Effect of additives on composites

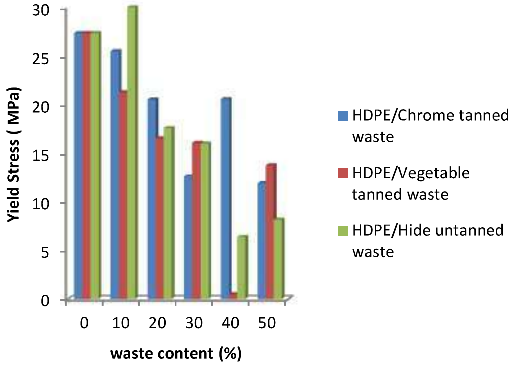

Figure 2a: Yield stress of composites without additives

Result of Figure 2b shows that the incorporation of additives modifies the yield strength of HDPE/Chrome tanned waste and HDPE/Vegetable tanned waste similar to what was shown on Figure 1b. The increase in yield strength was from 10-30 wt% and 10-40 wt% waste content respectively. The best formulation was observed with HDPE90/Chrome tanned waste10A with yield strength of 156.316 MPa, indicating good ability to undergo reversible deformation or carry stress without suffering from a permanent deformation. The improved interaction between the waste (fibre) and matrix is suggesting that the additives act as chemical groups bonding the fibre surface and the matrix to form a bond as reported by Ambrósio *et al* (2011). In addition, the reduction at higher waste contents could be attributed to poor wetting as discussed earlier with respect to Figure 1a, 1b and 2a which leads to reduced stress transfer across the fibre-matrix interface and thereby increases porosity leading weak concentration as reported in Pickkering *et al* (2015). The low yield strength of HDPE90/Hide un-tanned waste10A could be due to over-treatment on the formulation with additives that may had destroyed the mechanical bond existing within the interface and cause cracking of fibre.

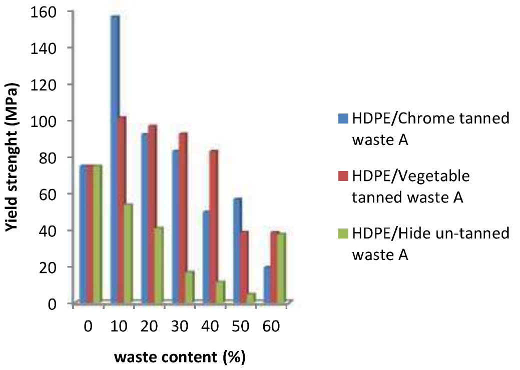

Figure 2b: Effect of additives on the yield strength of composites

3.3 Impact Strength

The result on Figure 3 shows that the impact strength increases with increased in waste content from 10 wt% to 30 wt%, for HDPE/Chrome tanned waste, and 10 wt% to 40 wt%, for HDPE/Vegetable tanned waste before it later decreases with further increased in waste content. All the composites show better impact at 10 wt% waste content except impact of HDPE/Hide un-tanned waste. However, HDPE/Vegetable tanned waste shows exceptional impact strength of 0.636 Jmm^{-2} at HDPE70/Vegetable tanned waste 30A. The high impact strength observed is implying excellent toughness of the formulation.

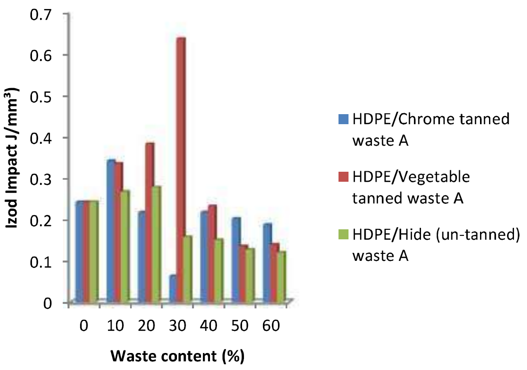

Figure 3: Impact strength of composites with additives.

3.4 TGA

Figures 4a and 4b describe the changes in weight in relation to change in temperature on composites without and with additives. The corresponding data for Figures 4a and 4b are listed on Table 3. The thermal differential analysis of composites without additives displays two steps weight losses as shown in Appendix 1a, 1b, 2a, 2b, 3a and 3b respectively. Weight loss in the first step was due to volatile organic molecule present during uncoiling as heat is gradually applied or as due to impurities (decomposition of inorganic compound present as additives). The second step weight loss occur due to the degradation of the composites within range of 347 – 447 ^{0}C, while composites with additives display a single step weight loss within the range of 447 – 550 ^{0}C.

The degradation of HDPE (Control) began at about 293 ^{0}C with a weight loss of about 1.283 %, and the maximum decomposition rate appeared at about 440 ^{0}C with residual weight of about 15 %. The onset degradation temperature of HDPE90/Chrome tanned waste10 without additives started at 50^{0}C with weight loss 0.447 % but with additives, it began at 300 ^{0}C with weight loss 2.205 %. The maximum decomposition rate appeared at 500 ^{0}C with residual weight of 25 %. The summary of the TGA results for the various composites are presented in Table 3.

3.5 DSC

Table 4 shows the melting temperature for HDPE without and with additives, the peak height and peak area for composites. The melting temperature of the composites as seen on Table 4 was higher than the melting temperature of the control sample. There was a broadening of the peak area as additives were added (See Appendix 4a, 4b, 5a, 5b, 6a, 6b, 7a and 7b respectively). The sharpness of the peak area indicates the level of crystalline and purity of composite. Peak height decreases with the incorporation of waste into the control sample.

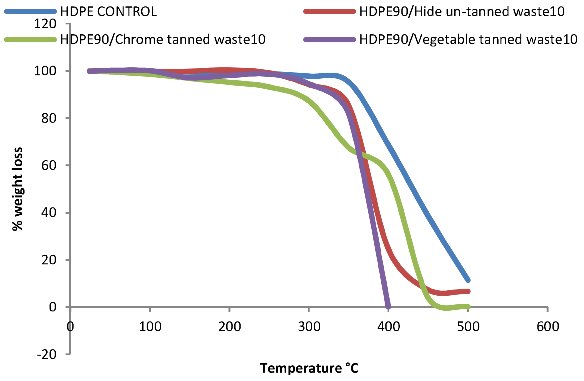

Figure 4a: Thermo gravimetric analyses of the control sample, HDPE90/Hide un-tanned waste10, HDPE90/Chrome tanned waste 10 and HDPE90/Vegetable tanned waste 10 without additives

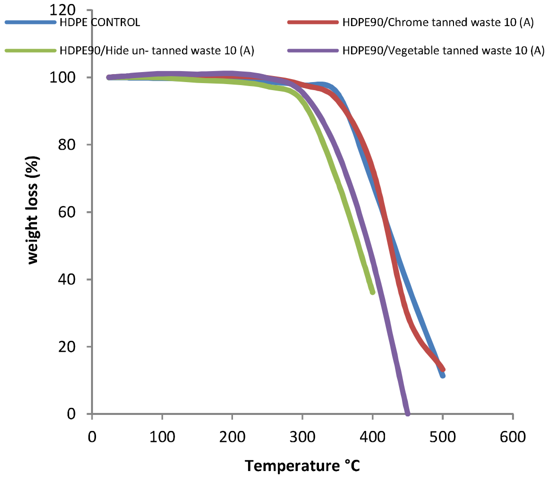

Figure 4b: Effect of additives on the thermo gravimetric analyses of control sample, HDPE90/Hide un-tanned waste10A, HDPE90/Chrome tanned waste10A and HDPE90/Vegetable tanned waste10A.

4.0 CONCLUSION

The use of solid tannery waste on HDPE is a viable alternative for the production of new materials and can therefore be used in many new applications where HDPE is applicable. If the end-user applications require high tensile strength (toughness), then HDPE90/Chrome tanned waste10 and HDPE90/Vegetable waste10 (A) are recommended to be used. Thus, this work had contributed to the reduction of the tannery solid waste in the environment. Composites of HDPE/leather waste are safe because from an environmental point of view, tons of chrome is kept inside the polymer as Cr3+, without the risk of oxidizing to Cr6+ under the action of sun light and heat, thereby reducing the risk of water and soil contamination. A partial replacement of 10-40 wt% of HDPE (petroleum-derived plastics) with biodegradable leather (natural fibre) was successful when it is modified with suitable additives. NR, TMQ, Mg $(OH)_2$, Ti_2O, EVA and acrylic acid have modify and enhanced the mechanical properties of HDPE-natural fibre (leather waste). The optimal value for tensile strength, and impact strength was obtained at filler loadings of 10-30 wt%.

Table 3 TGA temperatures and residue weight of HDPE and its composites.

Composites	T_o Onset tempt. T^0C	T_d Max decomposition tempts. T^0C	Weight loss T_o (%)	Weight loss T_d (%)	Residual weight (%)
Virgin HDPE	293	440	2.01	49.744	15
HDPE/Hide un-tanned waste	265	417	0.148	14.661	12
HDPE/Chrome tanned waste	290	453	1.0	52.136	44
HDPE/Vegetable tanned waste	263	395	1.977	98.948	0
HDPE/Hide un-tanned waste A	237	421	1.982	87.723	11
HDPE/Chrome tanned waste A	297	452	2.004	74.47	25
HDPE/Vegetable tanned waste A	262	414	0.991	69.26	30.7

Table 4: DSC analysis result for HDPE (control) and its composites

S/No.	Composite name	Peak height Wm	Melting temperature (T °C)	Peak Area JK/(mg)s
1	Control (100% HDPE)	17.7	162.21	26.58105
2	HDPE90/Chrome tanned waste 10	12.02	159.51	26.60427
3	HDPE90/Vegetable tanned waste 10	16.31	160.68	30.63222
4	HDPE90/Hide un-tanned waste 10	12.25	162.02	28.25667
5	Control plus additives (100%HDPE	17.57	161.12	33.54034
6	HDPE90/Chrome tanned waste10A	15.43	163.89	30.48755
7	HDPE90/Vegetable tanned waste10 A	12.11	162.25	34.69698
8	HDPE90/Hide un-tanned waste10 A	10.64	161.3	26.27539

The TGA and DTA results indicate that the composites are more thermally stable with additives than the control and it's composite without additives. Micrograms obtained shows that a ductile fracture surface of HDPE/Chrome tanned waste was the mode of tensile deformation as shown in Appendix 8 and 9 respectively.

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