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Mechanical properties and drop-weight impact performance of injection-molded HDPE/birch fiber composites

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ABSTRACT

Natural-fiber-reinforced composites offer various advantages over synthetic composites, including low density, useful mechanical properties and environmental friendliness. In spite of the progress achieved in the field, the mechanical performance of these composite materials has yet to be fully characterized, particularly in terms of impact resistance. In this study, we measured the drop weight impact, Izod impact strength, hardness, tensile strength and elastic modulus of birch-fiber-reinforced HDPE obtained by injection molding. Drop weight impact energy absorbed was constant and independent of fiber content whereas impact strength was inversely proportional to fiber content. Material toughness decreased slightly at 40% fiber. The Shore D hardness of virgin HDPE increased from 50.6 at 0% fiber to 74.6 at 30% fiber. The improvement of the elastic modulus of a composite containing 40% fiber was 27.2% superior to that reported for similar material made by compression molding. The corresponding improvement in tensile strength was superior by 19.7%. Birch-fiber-reinforced HDPE could be an adequate alternative to technical polymers widely used in several industrial sectors.

1. Introduction

Composites of thermoplastic and natural fiber are a relatively new group of materials. After decades of development of man-made high-tech fibers such as carbon, aramid and glass, it is remarkable that natural fibers such as linen, jute, hemp, sisal, kenaf and so on are now arousing interest, especially as substitutes for synthetic fiber and technical polymers in the automotive industry. Natural fiber composites were developed to meet the demand for affordable eco-friendly materials. The advantages of natural fibers over glass and synthetic or artificial fibers include certain improved mechanical properties, lower material costs, less abrasion of manufacturing equipment, lower energy consumption and health risks, less skin irritation, easier recycling and increased biodegradability [1–5]. The global demand for plastic composites reinforced with wood fibers is projected to increase by 144% over the period of 2016–2024, from $4.46 billion to $10.89 billion [6]. Although many businesses (e.g. construction, sporting equipment, automotive parts) are using natural fiber composite materials, their development, especially from short fibers, is still limited due to insufficient understanding of their mechanical behavior and sensitivity to environmental factors [7–10]. Although the mechanical properties of wood-fiber composites (WPCs) appear not to depend on wood type [11], the lignocellulosic fiber and lignin type and the cellulose and hemicellulose content have strong influences [12].

Natural fibers are obtained from various natural sources such as plants, animals and minerals [13]. Plant fibers are further classified based on the source and their physiological properties. Fibers obtained from the stem are called bast fibers (flax, hemp, kenaf, jute, isora, etc.), leaf fibers (sisal, abaca, curaua, palm, etc.), seed fibers (cotton, soya, kapok, calotropis procera, etc.), fruit fibers (coir, luffa, etc.), grass fibers (bamboo, wheat straw, baggase etc.) and wood fibers such as hardwood and softwood (teak wood, rosewood, birch, ect.) [13–18]. Choosing one fiber over another can be motivated by mechanical, physical and chemical properties of fibers. But, the availability of a local fiber which exhibits interesting properties can be considered as a determining factor for the choice of a fiber.

The appropriate natural fibers extraction represents a major challenge faced during the processing of plant fibers. The most common methods to separate the plant fibers are dew retting and water retting process. Depending on the fiber category, these methods require approximately 14–28 days for the degradation of waxes, pectin, hemicellulose and lignin. To reduce long processing time, alternative

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Table 1
Birch fiber quality analysis results.

Dimension	Measured
Mean length: L (mm)	0.49
Mean width: D (μm)	24.7
Aspect ratio: L/D	19.79
Fiber count	5000

methods such as mechanical extraction and chemical treatments have been introduced [19].

For use in composite materials, thermoplastics must melt or soften at 200–220 °C, that is, below the temperature at which the filler degrades [20]. Polyethylene, polypropylene, polystyrene and polyvinylchloride are therefore suitable. However, satisfactory dispersion of natural fillers in a thermoplastic matrix has been difficult to achieve, since the former are hydrophilic whereas the latter is hydrophobic, making a durable interface difficult to obtain and lessening the transfer of stress from one component to the other. To increase the affinity of the components for each other, the surface properties of at least one must be modified. Compatibilizers are bifunctional compounds that can be used to increase wood/plastic mutual adhesion. Their use as coupling agents increases the tensile and flexural strengths of wood/polymer composites [21–23]. A common example is maleated polyethylene (MAPE), which has been used widely in polyethylene/wood fiber composites [24,25] and is effective at concentrations as low as 2–3% by weight [24–28].

Polyethylene (PE) is one of the most widely used thermoplastics in the world because of its toughness, near zero moisture absorption, chemical inertness, strong dielectric character, low friction and ease of processing [29,30]. Pipes, containers, electrical insulation and numerous other items are made of PE. The mechanical and physical properties of PE depend significantly on variables such as crystalline

Table 2
Physical and mechanical properties of the birch fibers.

Fiber	Density (g/cm^3)	thermal conductivity (W m-1 K^{-1}	specific heat measured at 17 °C (J Kg^{-1} K^{-1}	shear modulus G	Poisson coefficient ν	elastic modulus E_1 (GPa)
Yellow birch	0.62	0.15	1300	0.94	0.45	13.9

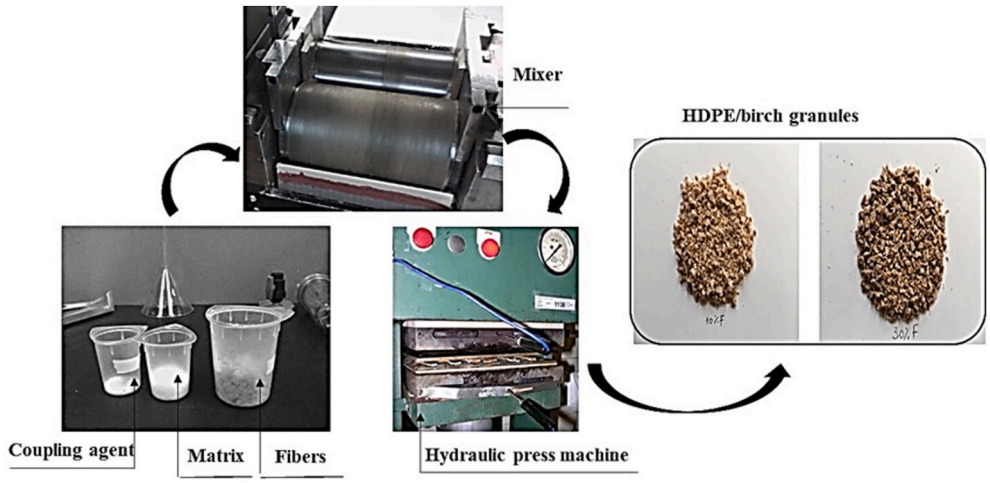

Fig. 1. Summary of the molding process used to obtain HDPE/birch fiber composite granules (left: 10% fiber; right: 30% fiber).

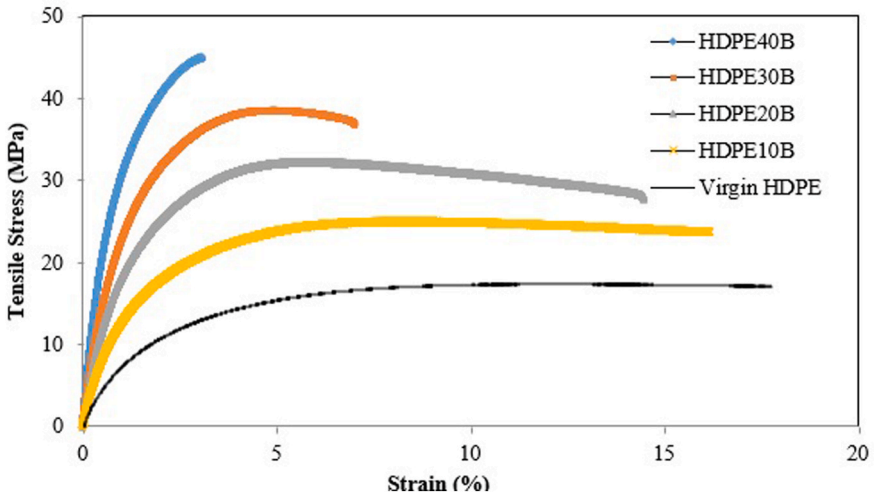

Fig. 2. Tensile test: stress-strain behavior of HDPE/birch composites.

Table 3
Tensile properties of HDPE/birch fiber composites.

Material - Nomenclature	Property			
	Fiber content (%)	Young's modulus (MPa)	Tensile strength (MPa)	Strain failure (%)
Virgin HDPE	0	1510 (±0130)	18.8 (±1.64)	No break
HDPE10B	10	1660 (±90)	25.08 (±0.11)	No break
HDPE20B	20	2670 (±130)	32.47 (±0.19)	12.74 (±1.08)
HDPE30B	30	3370 (±160)	38.33 (±0.47)	7.60 (±0.4)
HDPE40B	40	4390 (±140)	45.54 (±1.024)	3.19 (±0.46)

Table 4
Young's modulus of HDPE/wood composites, as measured in tensile tests and by impulse excitation.

Nomenclature	Birch fiber content (%)
Virgin HDPE	0
HDPE10B	10
HDPE20B	20
HDPE30B	30
HDPE40B	40
Tensile test (MPa)	Impulse excitation (MPa)
1510 (±130)	1720 (±38)
1660 (±90)	2340 (±28)
2670 (±130)	3140 (±77)
3370 (±160)	4060 (±72)
4390 (±140)	4430 (±48)

structure and molecular weight. Composites made with PE as a matrix have better mechanical and physical properties than the pure plastic and are used as packaging materials, in electrical storage devices, and in thermal energy, automotive, biomedical and space applications. They can be extruded, injection-molded, compression-molded or rotational-molded [29]. Birch is a widespread hardwood tree that grows well in cool climates with abundant precipitation, such as in the province of Quebec, Canada [31,32]. Birch fibers, a byproduct of the paper industry, are among the many wood fibers used to make composite plastic materials. Birch fiber/polyethylene composites can be produced in the laboratory using thermo-compression equipment [31,33]. However, studies using test specimens made of HDPE/birch fiber composites produced by injection molding under industrial conditions are very limited [5]. This new material has already been characterized by different forms through mechanical tensile and bending tests, thermo-mechanical tests (DMA) and thermal degradation tests (TGA) but not under low velocity impact stress and especially drop-weight impact tests [33]. Low velocity impacts are defined as events which can occur in the range 1–10 m/s depending on the target stiffness, material properties and the projectile mass and stiffness [34]. A low velocity impact event can occur in-service or during maintenance activities and can be considered one of the most dangerous loads on composite material. For low velocity impact events, the usage of pendulums like the ones present in the Charpy test, the Izod test and drop towers or drop weights have become standard. A drop weight impact testing unit enables the simulation of a wide variety of real-world impact conditions and collects detailed performance data [35]. One of the advantages of this test with respect to the Charpy and Izod tests is that a wider range of test geometries can be examined, thereby enabling more complex components to be tested. Its main function is to test the impact behavior of composite plates. Izod and Charpy impact testing can provide a large amount of data since they are easy to set up and can collect a large amount of data quickly. However, the results obtained from these tests are not in depth such that they will show more of the characteristics of the material. Natural short fiber composites are subject to cracks or perforation when they are impacted. The quantification of the energy required to create these defects is very important, this is to better understand the resistance and the damage resilience of the material [36]. Very little work has investigated composites of short natural fibers and a regular thermoplastic to see its behavior on impact. Most works assess Charpy or Izod resilience. Hardness tests on these composites are also rare in the literature.

The purpose of this study is to test the drop-weight impact shock-absorbing capacity, Izod impact resilience and hardness of injection-molded HDPE/birch composite and also to compare injection-molding to compression-molding in terms of material mechanical properties, primarily by testing tensile characteristics.

2. Materials and experimental testing

2.1. Compounding

2.1.1. Materials

High-density polyethylene (HDPE, 0.953 g/cc based on ASTM D-1505, melt index 18 g/10 min based on D-1238) from Raplast Inc. Yellow birch thermomechanical wood pulp fiber was prepared at the Innovations Institute in Ecomaterials, Ecoproducts, and Ecoenergies Biomass Based (I2E3) at University of Quebec at Trois-Rivières (UQTR). The thermomechanical pulping process (TMP, a mechanical extraction process) [19] was used to make the fibers. This process uses wood chips subjected to a temperature above 100 °C in steam in order to soften the fibers. Pressurized defibration ensues in a refiner fitted with two rotating discs rotating at high speed. It is the effect of successive cycles of compression and decompression that produces the dough at a yield rate of approximately 90%. Wood fiber was dried at 80 °C in an air-circulating oven for 24 h and then ground to 20–60 mesh size before use. The fiber aspect ratio (mean length divided by average diameter) classes were obtained by mechanical refining and screening and characterized using an OpTest fiber quality analyzer (Table 1).

Physical and mechanical properties of the birch fibers used are mentioned in Table 2.

High-density polyethylene (HDPE, 0.953 g/cc based on ASTM D-1505, melt index 18 g/10 min based on D-1238) was provided from Raplast Inc and the coupling agent, maleic anhydride grafted polyethylene coupling (MAPE) from Addcomp company.

2.1.2. Compounding

The materials were prepared by blending the components in a Thermotron mixer (C.W. Brabender, model T-303, Fig. 1). HDPE and MAPE at a mass ratio of 20:3 were melted on rollers at 170 °C. Wood fiber and the remaining HDPE were then blended in for 7 min at 60 rpm. The blend was peeled off the roller and re-blended five times for 3 min each to obtain a uniform composite sheet, which was removed from the roller and cut into strips with a knife to fit into the samples mold. To obtain granule for injection molding, the sheet was cooled and granulated in an industrial grinder. Wood fiber content was set at 10%, 20%, 30% or 40% by weight (Fig. 1). A 100-ton-capacity Zerus 900 press (ZHAFIR Plastics Machinery GmbH) was used to mold test specimens.

2.2. Mechanical characterization

2.2.1. Tensile test

Specimen tensile strength at room temperature was measured on an Instron device (Model LM-U150) equipped with a 50 kN load cell. A 25 mm extensometer connected to the data acquisition system was fixed to the specimen gauge length section to record the strain. The test was conducted at 2 mm/min. The strength at maximal load, elastic modulus and strain at break were determined in compliance with ISO 527–1:20 [37]. Five replicates were tested for each material composition.

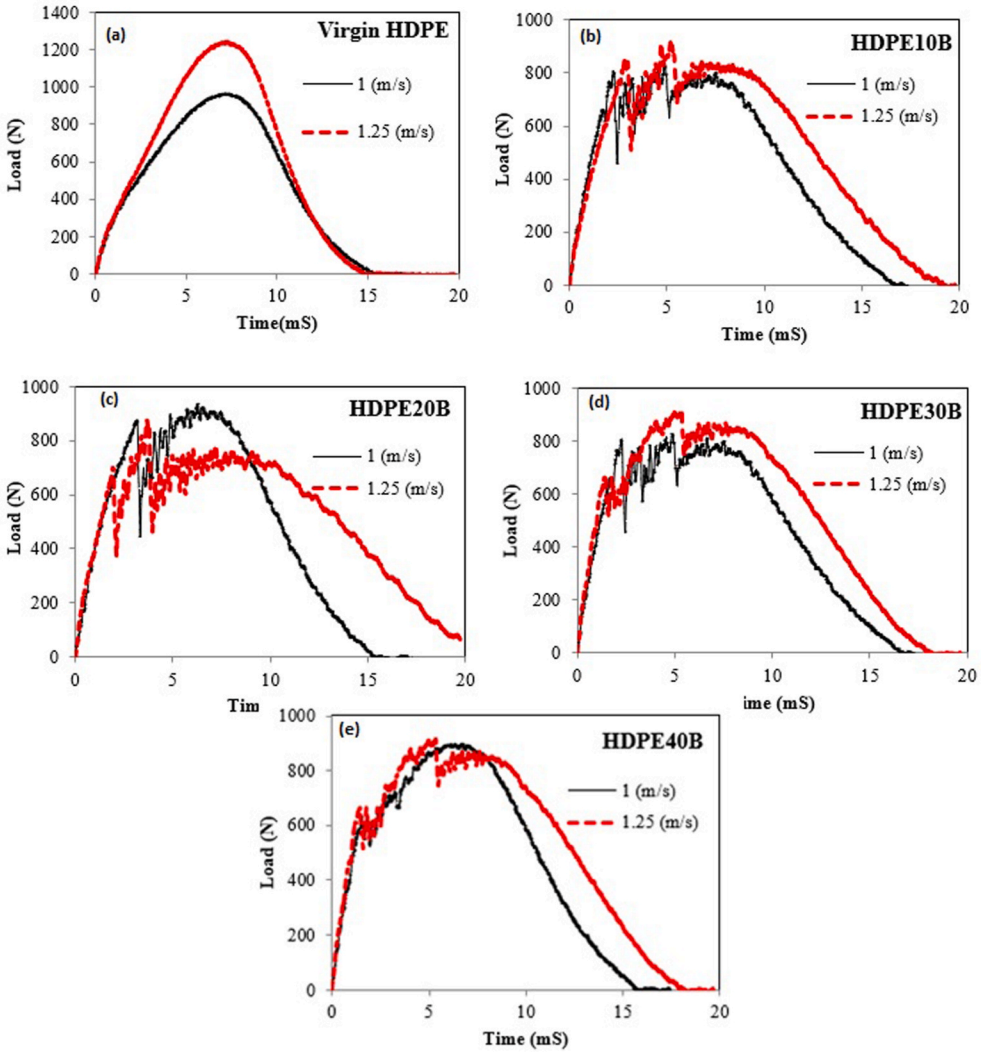

Fig. 3. Low-velocity impact tests at 2.56 J: (a) virgin HDPE, (b) 10% birch fiber, (c) 20% birch fiber, (d) 30% birch fiber, (e) 40% birch fiber.

2.2.2. Impulse excitation measurements

Young's modulus was measured using non-destructive impulse excitation technique according to ASTM E1876 – 09 [38]. Five Samples were excited mechanically by automated tapping. Vibration was recorded using a microphone and analyzed using the "resonant frequency and damping analyzer" (RFDA) software (IMCE, Belgium). Young's modulus (E) was calculated from the acquired resonant flexural frequency f_f (in Hz) as per ASTM standard E 1876-09 using the following equation (Eq. (1)):

$$E = \left(m \cdot f_f^2 / b\right)\left(L^3 / t^3\right) T_1 \quad (1)$$

where m is the mass of the specimen in kg, b, L and t are respectively the width, the length, and the thickness of the specimen in meters and the constant T_1 is the correlation factor for the fundamental flexural mode needed to account for finite thickness and the Poisson ratio of the specimen [38–40].

2.2.3. Drop weight impact test

An Instron CEAST 9350 free-fall drop-dart machine was used with the 40 mm specimen support, 22 kN load cell capacity and 12.7 mm diameter hemispherical tup type according to ASTM standard-D5628-10 [41]. The impact energy was 2.75 J at a falling height of 51 mm. Five specimens of each composition (virgin HDPE, the four wood/HDPE blends) were tested, four at 1 m/s impact velocity one at 1.25 m/s.

2.2.4. Izod impact test

Izod tests were carried out on an Instron CEAST 9050 impact pendulum equipped with a 0.5 J hammer. The pendulum is designed to determine the resilience, ductile and/or brittle fracture. As stipulated by ASTM D256 - 10e1 [42], the breaking energy of the material tested must be between 10% and 90% of the capacity of the hammer used for the Izod tests. Five specimens of each composition (virgin HDPE, the four wood/HDPE blends) were tested.

2.2.5. Hardness test

Hardness of neat HDPE and wood-fiber composite in disk form (62.5 mm diameter) was measured using an analog durometer Shore D scale as per ASTM D2240-15e1 [43] with the specimens treated as semi-hard plastics. Five specimens of each composition (virgin HDPE, the four wood/HDPE blends) were tested. The test was repeated at least 8 times for each specimen. This type of device is used to test the hardness of rubbers, semi-rigid and rigid plastics.

3. Results and discussion

3.1. Tensile test

Fig. 2 shows the effect of fiber loading on the tensile stress-strain

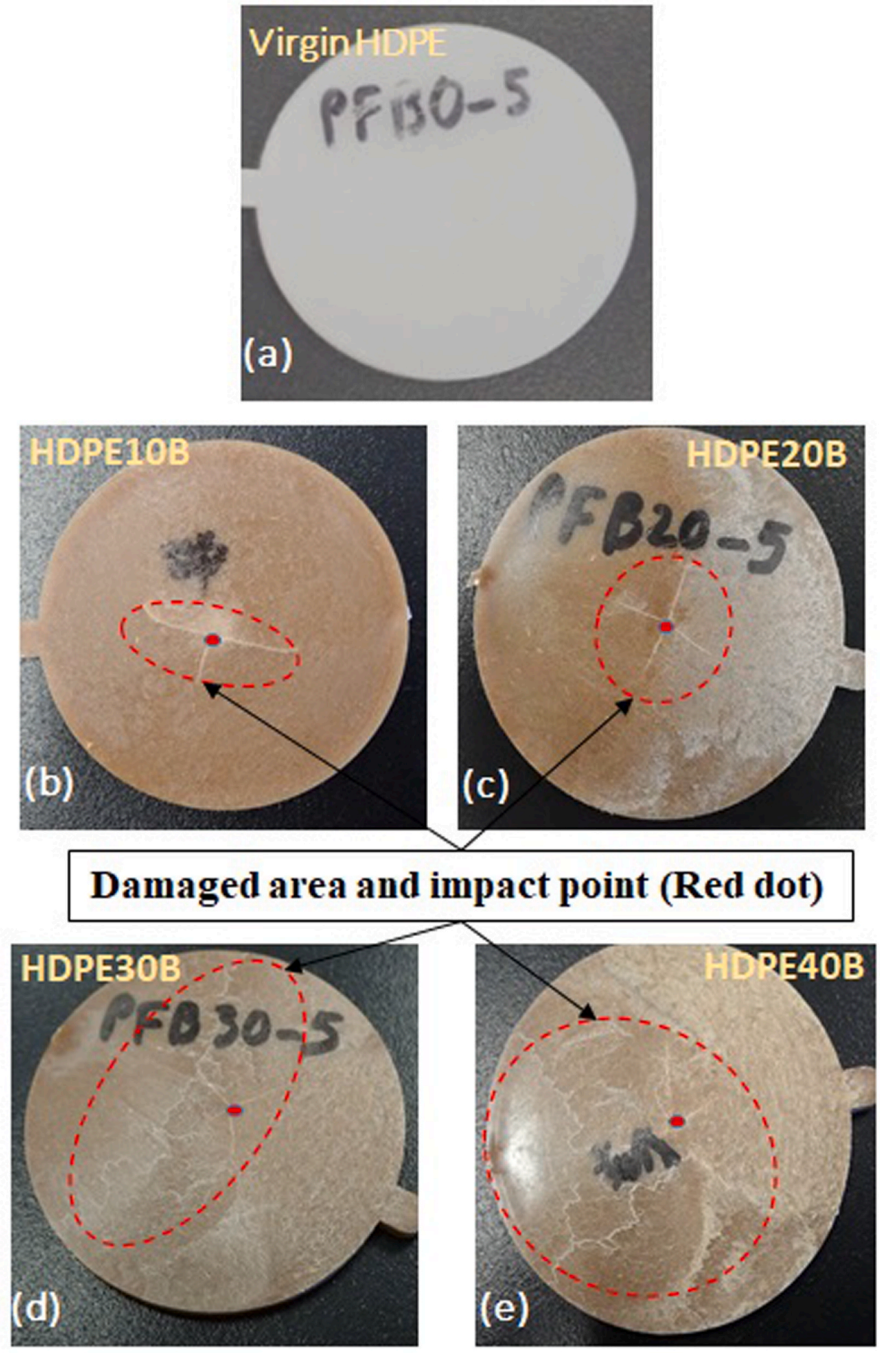

Fig. 4. Appearance of specimens after impact tests: (a) virgin HDPE, (b) 10% birch fiber, (c) 20% birch fiber, (d) 30% birch fiber, (e) 40% birch fiber.

curves of HDPE/wood composites. Increasing the amount of fiber increased the maximal stress and stiffness of the materials. It also decreased the strain at failure, which is the consequence of less mobility of the molecules in the polymer matrix (Fig. 2). In view of these curves, the following is noted:

- A linear first phase characterized by an elastic Young's modulus E;
- A non-linear second phase reflecting the beginning of damage and onset of permanent strain (plasticity) until the stress maximum;
- A final phase in which the stress decreases until the final fracture.

The static strength properties are given in Table 3. Results show the effects of fiber loading on the elastic modulus and the tensile strength of HDPE/birch composites containing 3% MAPE. At 40% birch fiber, Young's modulus increased linearly by 190.7% and the tensile strength by 142%. These significant increases are due not only to the fiber but also to better adhesion between fiber and matrix [44] with the addition of the coupling agent.

Previously reported increases of the Young's modulus and tensile strength of HDPE/birch fiber composites produced by compression molding are smaller, for example 3450 MPa and 38.05 MPa respectively relative to base values of 1510 MPa and 18.8 MPa [33], compared to 4390 MPa and 45.54 MPa in the present study, for gains of 27.2% and 19.7% respectively over the previous improvements. Overall, the results are interesting even though the bio-composite loses ductility as the fiber content increases, as indicated by the decrease in the strain at failure [32]. Tensile strength of HDPE/20% birch fiber composite in this study is better than that of HDPE/40% yellow pine fiber (23.52 MPa) [45],

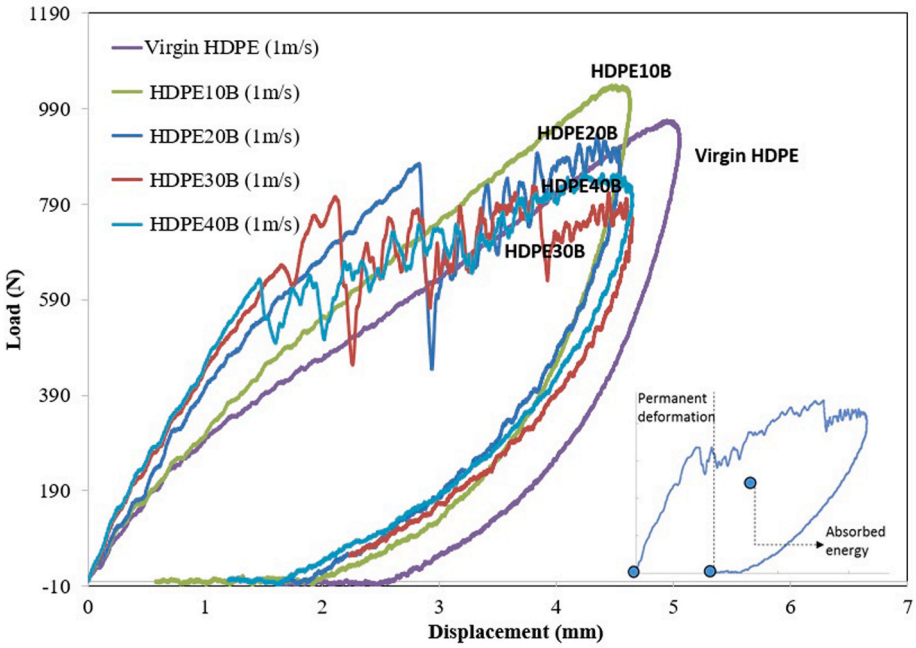

Fig. 5. Force/displacement curves obtained for the five materials.

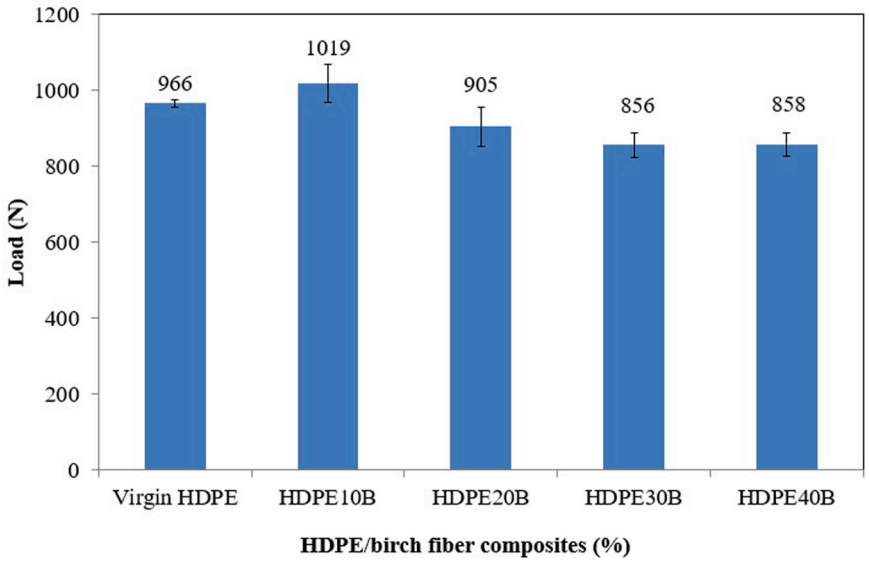

Fig. 6. Maximal drop impact force withstood by five HDPE/wood composite materials (at 1 m/s impact velocity).

HDPE/20% Palm empty fruit Bunch fiber (25 MPa) [46], HDPE/30% flax fiber (24 MPa) [47], HDPE/kenaf fiber (27 MPa) [48]. Tensile strength and Young's modulus of HDPE/40% birch fiber composite have almost the same values as that of HDPE/50% modified poplar wood fibers (44 MPa, 4300 MPa) [49].

3.2. Impulse excitation measurements

Young's modulus based on impulse excitation measurement was in overall agreement with that measured in tensile tests (Table 4). The modulus increased by 157.6% (from 1720 to 4430 MPa) as the birch fiber content increased from 0 to 40%.

3.3. Drop-weight impact tests of material strength

The force absorption curves in Fig. 3 show the phenomena of free fall, stop and rebound described by G. Belingardi and R. Vadori [50], who point out that if the energy absorbed by the specimen is not too great, a rebound occurs without saturation or perforation. The "end of contact" time is the instant when the force between specimen and dart returns to zero. For drop tests, five specimens of each composition (virgin HDPE, the four wood/HDPE blends) were tested, four at 1 m/s impact velocity one at 1.25 m/s. In the case of HDPE/wood composite (Fig. 3 b–e), the time course features two thresholds, a first one where the rising curve drops off sharply followed by a rise marked by large oscillations, and a second similar drop followed by a rise with a lower slope. These features indicate that initial damage to the material was reversible and that recovery from the second damage was only partial

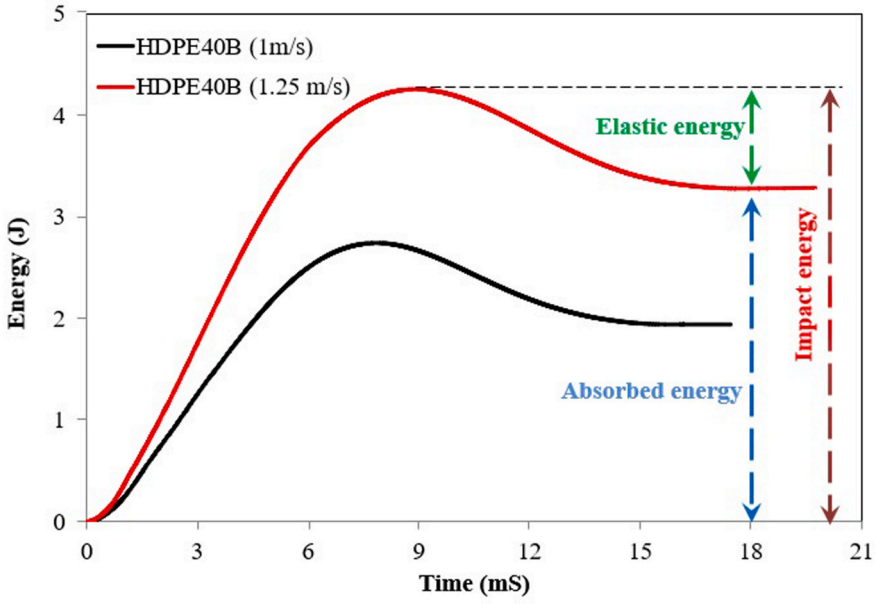

Fig. 7. Energy balance during the drop impact test (40% birch fiber).

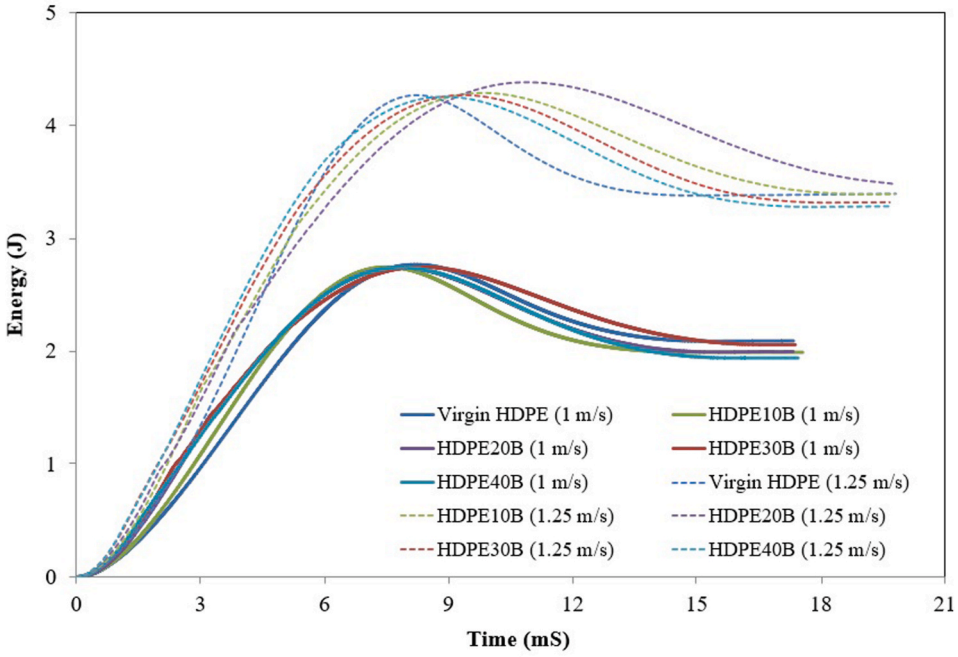

Fig. 8. Energy absorption by composite materials during the drop impact test.

Table 5
Energy maximum, absorbed energy, permanent deformation and degree of damage (at 1 m/s impact velocity).

Nomenclature	Birch fiber content (%)	Energy Max (J)	Energy Absorbed (J)	Permanent deformation (mm)	Degree of damage, μ
Virgin HDPE	0	2.75	2.09	2.62	0.76
HDPE10B	10	2.75	2.04	1.94	0.74
HDPE20B	20	2.75	2.01	1.89	0.73
HDPE30B	30	2.75	2.04	1.81	0.74
HDPE40B	40	2.75	1.97	1.66	0.71

[50]. The curves obtained for virgin HDPE have no such features (Fig. 3 a), which means the material underwent no damage. Composite containing 10% birch fiber also underwent no damage at velocity 1 m/s (Fig. 3b). The initial damage occurred at forces of about 850 N, 700 N, 660 N and 600 N in composites containing respectively 10%, 20%, 30% and 40% birch fiber.

The patterns of the graphed data are consistent with the appearance of the samples in the photographs (Fig. 4). Cracks are visible in the composites (Fig. 4b–e), whereas the virgin HDPE appears intact (Fig. 4 a).

The maximal strength value can be obtained also from the force-displacement diagram (Fig. 5). It increases with the impact energy until the second damage occurs then drops suddenly. It represents the

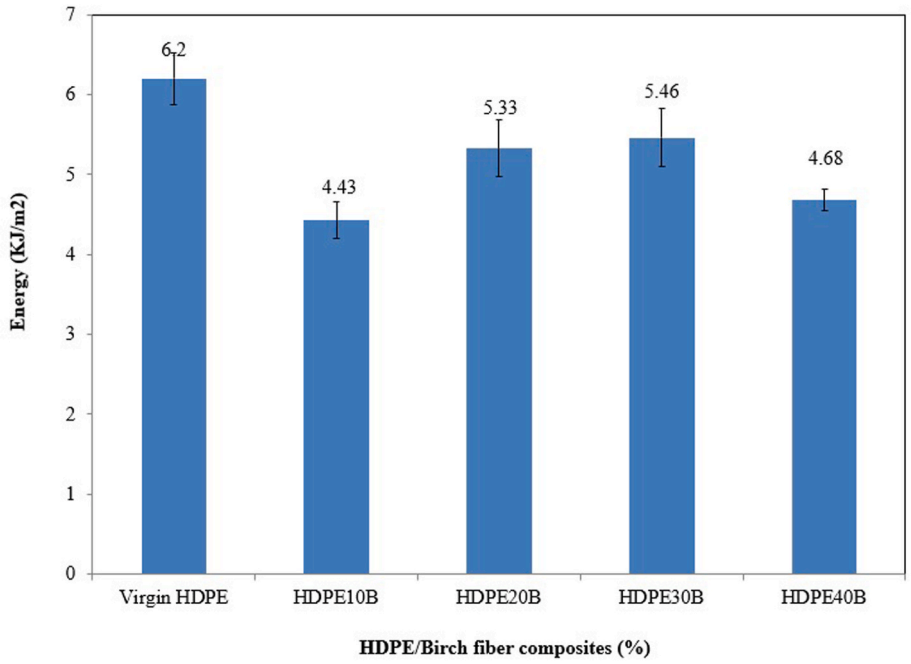

Fig. 9. Izod impact energy of HDPE/birch fiber composites.

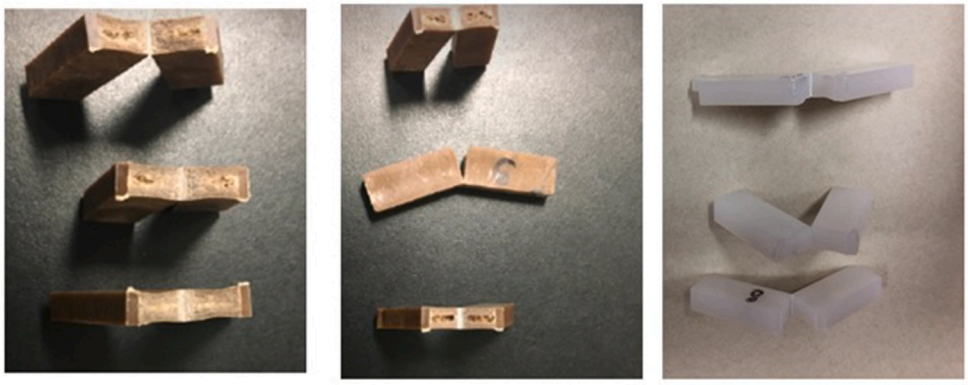

Fig. 10. HDPE/wood composite specimens after the Izod impact test, (a) 40% fiber, (b) 20% fiber, (c) virgin HDPE.

maximum load-carrying capacity of the material [50]. The separation between the unloading and loading curves indicates that all specimens absorbed a significant fraction of the impact energy [51]. A visual comparison of the maximal strength obtained for the materials tested is shown in Fig. 6. The composite containing 10% birch fiber has the best maximum load carrying capacity, followed by virgin PE.

Fig. 7 shows the overall behavior of the composite materials during the impact test. The ascending portion represents the absorption phase and the descending portion represents the partial restitution phase in terms of the kinetic energy required for rebound [52]. Table 3 and Fig. 8 show that the energy absorbed is greatest in the case of virgin PE followed by composite containing 10% birch fiber. Composite containing 40% birch fiber absorbed the least energy [52].

Based on fracture mechanics theory, the total fracture energy absorbed during impact should be proportional to the damage sustained. The classical definition of impact resistance is the ability of a material to absorb energy without failure, material toughness being proportional to the energy absorbed before fracture. In this study, toughness was maximal for virgin PE and minimal for 40% birch fiber composite.

The impact energy E_{max} (Table 5), that is, the maximum amount of energy that the specimen can store as internal elastic deformation energy or dissipate via plastic deformation or fragmentation (i.e. the energy required to stop the dart in the rebound cases) and permanent deformation were also highest for virgin PE [50–54]. Permanent deformation was lowest for composite containing 40% birch fiber.

The ratio μ of the dissipated (hence unrecoverable) energy to E_{max} indicates the degree of damage. It was always less than 1.0, proving that rebound occurred in all specimens.

3.4. Izod impact test

The results of the Izod impact test are shown in Fig. 9. Impact strength of pure HDPE slightly decreased after the addition of 10% wood fiber, decrease already observed for wood fiber composites by some authors [55]. The impact strength of the composites was found to increase with fiber content but never exceeded 88.1% of the value for pure HDPE. Birch fiber thus decreased the impact strength of the plastic. Impact strength of HDPE/30% birch fiber composite in this study is higher than that of HDPE/40% yellow pine fiber (3.93 KJ/m2), but much lower than that of HDPE/50% modified poplar wood fibers (23 KJ/m2) [45,49]. [][][].

Fibers absorb energy of impact through three mechanisms: de-

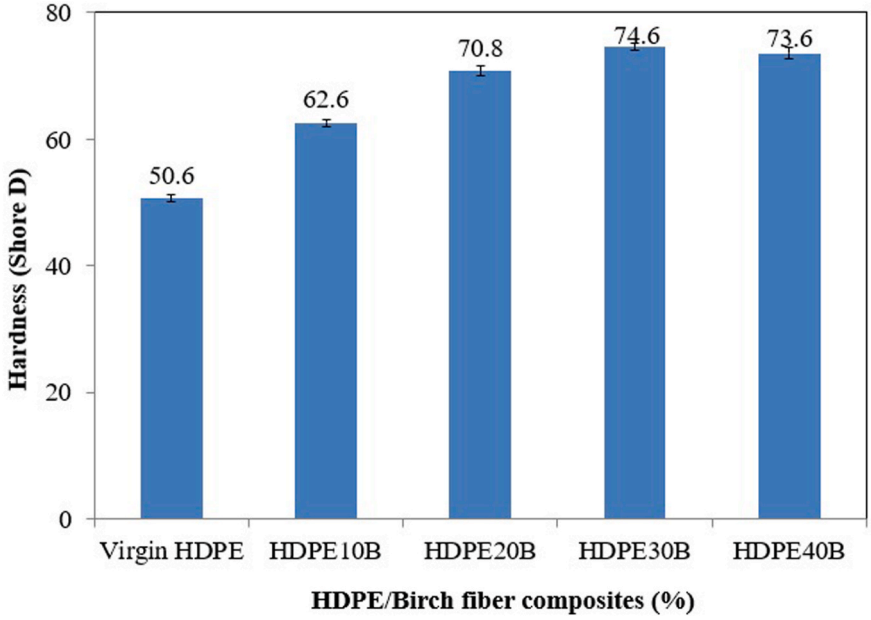

Fig. 11. HDPE/birch fiber specimen hardness.

bonding, pull-out and fracture. The strain energy released by de-bonding and fracture is proportional to the de-bonded length. Weak adhesion between matrix and fiber thus predisposes the material to greater energy absorption [56,57].

The decrease in the impact strength as the fiber content passed from 30% to 40% shows that there is an optimal concentration of fiber, which differs from one composite type to the next [56]. Above this concentration, no further improvement of mechanical properties is possible, since fibers are incompletely surrounded by matrix material and in contact with each other, and less energy can be absorbed [56,57].

The decrease in the impact strength of HDPE/birch fiber composite compared to pure HDPE can be explained by the appearance of defects in the samples. Porosity induced during injection is up to 40% greater in material containing fiber (Fig. 10).

3.5. Hardness test

Hardness test results showed that adding birch fiber to virgin HDPE increased the Shore D hardness, from 50.6 to 73.6 for 40% fiber, an increase of 46.64%. The highest value, 74.6, an increase of 48.62%, was achieved at 30% fiber (Fig. 11). The slight decrease at 40% fiber is attributed to deterioration of mechanical properties due to the influence of temperature, pressure and possibly other injection-molding parameters. To obtain material without defects at 40% fiber, the temperature and pressure were increased to reduce the viscosity and improve flow, but birch fibers char under these conditions. In addition, 40% is likely above the optimal concentration of fiber, as mentioned above [56].

Modulus and hardness are positively correlated, since hardness is a function of the relative fiber volume and the modulus. In this study, tensile and flexural modulus increased with fiber loading, as reported previously [58,59].

4. Conclusion

The birch fiber used in this work presents very interesting morphological and mechanical properties. The resilience of polyethylene/birch fiber composite materials made by injection molding under industrial conditions was examined using the drop weight and Izod impact tests. Material hardness was also measured.

For injection-molded HDPE/birch fiber composite, the improvement of the elastic modulus of material containing 40% fiber was 27.2% superior to the improvement reported previously for similar material produced by compression molding. The corresponding improvement of the tensile strength was 19.7% superior.

In drop weight impact tests, the energy absorbed by the whole specimen was not too high; rebound occurred in all cases. Neither saturation nor perforation was noted.

Virgin polyethylene sustained no damage whereas all-composite blends were fractured. The composite containing 10% birch fiber had the best maximum load-carrying capacity, followed by virgin PE. Absorbed energy was maximal in virgin PE followed by 10% wood fiber composite. In this study, composite material toughness was greatest at 20% fiber.

Izod impact strength of the composites increased with fiber content but was at best 12% below the value for pure HDPE. The use of birch fiber thus decreased the impact strength of the plastic. Shore D hardness increased by 48.62% from 50.6 for pure HDPE to 74.6 for composite containing 30% fiber and was lower at 40% fiber. Increasing the amount of fiber increased material stiffness and maximum stress and lowered the strain at failure.

Author statement

Agbelenko Koffi: Investigation, Methodology, Writing - original draft.

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Research data for this article

Data will be made available on request.

Human/Animal rights

This article does not contain any studies with human or animal subjects performed by any of the authors.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

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Appendix A. Supplementary data

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