Morphological, Mechanical, and Multifunctional Properties of Sugar Cane Bagasse/Polyester Composites

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ABSTRACT

Various fiber-matrix combinations were used to create biodegradable composites using sugar cane bagasse (SCB) fiber and a polyester resin (PES) matrix. SEM, FTIR, mechanical testing, thermogravimetric analysis (TGA), and the method of soil burial were employed to assess the effects of the fiber content (9-54 wt%), PLA (1-5.5 wt%), starch (1-5.5 wt%), and fiber treatment on the morphology of the surface, flexural, thermal, tensile as well as biodegradable qualities of polyester/sugar cane bagasse fiber biocomposites. The outcomes demonstrated that the optimal tensile strength of biocomposites, impact, and flexural capabilities were attained by the 37 wt% treated sugar cane bagasse fibers. Tensile, flexural, and impact strength measurements for the 9, 18, 27, 37, 45, and 54 wt% biocomposites, respectively, ranged from 10.21 to 18.00, 21.33 to 28.12, and 12.21 to 15.67 Mpa. The results show that sugarcane bagasse-polyester composites demonstrated acceptable mechanical and multifunctional properties. This portrays the effectiveness of sugarcane bagasse as reinforcement for Polyester matrix and its potential for eco-friendly fabrication of components.

INTRODUCTION

Numerous synthetic polymers/plastics have long life cycles, which interfere with soil nutrients and degrade the soil after being dumped or buried in landfills [1]. This leads to an intensified campaign for the addition of biodegradable substances to polymers and their composites [2] and a growing understanding of the importance of preserving our environment from plastic pollution. The potential for modern environmental friendliness and their extensive applications for materials in the biomedical, mobile, and automotive sectors are driving up demand for natural fiber reinforced biocomposite materials. Natural fibers like jute, sisal, pineapple peels and leaves, ramie, bamboo, hemp, coconut coir, sugar cane bagasse, and other fibers are degradable, non-toxic, and readily recyclable. To improve the fiber surface’s quality and interfacial bonding to matrices through chemical modification [6]. It is generally necessary to pretreat natural fibers using conventional alkali treatment [3], treatment approaches using permanganate [5], or peroxide [4]. Depending on the specific chemical makeup of the material under consideration, biodegradable additives assist in the decay of matter into water (H2O), inorganic substances, carbon dioxide (CO2), and methane (CH4).

Research on and use of sugarcane bagasse fiber residues as a source of polymer reinforcement is extensive. It attracts a lot of interest because it has inherited qualities like biodegradability and renewability [7]. Hemicellulose (20.0-27.5 wt%), lignin (18.0-26.3 wt%), and cellulose (41.0-55.0 wt%), and other components (7.0 wt%) make up sugar cane bagasse [8-10]. Sugar bagasse nanocrystals to strengthened starch was created by Slavutsky and Bertuzzi [11] and improved interfacial interaction, dispersion, and mechanical properties were observed. As a result, the strength was increased from 2.8 to 17.4 MPa and Modulus increased from 112 to 520 MPa.. Ashes from the burning of sugar bagasse can be used for polymer reinforcement in addition to their traditional use as an energy source for the ethanol and sugar industries [7]. Sugarcane bagasse waste

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can be used to generate or produce a variety of fillers, including cellulose, micro/nanofibers, nanocrystals, fibers, and ashes from sugarcane bagasse (SBA). (MFC or CNF) were infused into a number of polymer matrices, including thermosets [16, 17], nylon [15], low density polyethylene (LDPE) [13], polyethylene oxide (PEO) [14], and polypropylene (PP) [12]. To address the recycling issues, a new paradigm ranging from artificial polyesters to a novel breed of resins that degrade naturally has occurred [18]. Polyester is an extensively utilized commercial biodegradable material. They are produced by polycondensation reactions between aliphatic succinic and adipic acid, and glycols, like 1,4-butanediol and ethylene glycol [19]. Their biodegradation rates [19] and properties [20] are influenced by their structure. The common uses for this type of biopolymer include tissue engineering and healthcare surgical intervention [21, 22], automobile parts [23], packaging materials, degradable grocery bags in supermarkets, and other uses [24]. The emergence of reinforced polyester fiber biocomposites can provide an alternative to their synthetic fiber reinforced counterparts in terms of the environment and energy [25, 26]. The studies of biocomposites reinforced with sugar cane bagasse fibers have been extensively covered in research literature. When SBA was used to reinforce natural rubber, de Paiva et al. [27] discovered that there was a powerful interfacial interaction. Thermal and structural characteristics of SB-based biocomposites were characterized by Vilay et al. [28], Mandal and Chakrabarti [29]. The fibers’ thermal degradation behavior was altered by surface modification [30]. According to reports, the chemical treatment decreased the hydroxyl groups, improving the adhesion of the fibers to the polymeric matrix. It primarily reacts with lignin components, specifically the lignin-associated step in the thermal degradation of the fibers (i.e., phenolic syringyl and guaiacyl) [16]. The alkali treatment enhance the thermal the strength of the fibers by removing their wax and hemicellulose, which are thermally unstable components.

The current study examines the effects of fiber content and fiber pre-treatment on the mechanical, thermal, and biodegradable properties of PES/SCB fiber biocomposites produced through compression molding. When concentrating on the fully biodegradable PES/SCB fiber composites, only their mechanical properties [11] or mechanical and thermal properties [29] were experimentally investigated on an individual basis. It is anticipated that this work will be able to provide further elaborate insight into the production and characterization of any potential commercial products made from PES/SCB fiber fiber biocomposites.

MATERIAL AND METHOD

Materials

The following items were purchased from Rovet Chemicals in Lagos: PES resin, starch/PLA, catalyst (ketonox), accelerator (napholite), and processing oil. Masticated SCB was obtained from Ama Awusa, Owerri, sugar cane stem vendors. Idex Scientific Company, Aba, Nigeria, sold the following items: sodium hydroxide (NaOH), distilled water, aluminum foil, cellophane bags, and masking tape. The following equipment were used; Compression moulding machine (WZ Shoemaking Machine Co. S/No: 03577-86365889), sieve shaker (WZ Shoemaking Machine Co. S/No: 03577-86365889), weighing balance (Mettler AE 200), Wallace test abrader (Brooks® S/No: 084025/1), impact testing machine (Resil Impactor; Pieniza Torino, Italy S/No: 6957), abrasion shape cutter (Dump bell Shape Cutter. S/No: EL 695/EL692), Scanning Electron Microscope (SEM) (Phenon Prox. S/No: Q150RES), Fourier Transform Infra- Red (FT-IR) spectrometer (Agilent Technologies Nicolet 6700 (4000 – 650 cm-1), Dynamic Mechanical Analyzer (Model: DMA242 E Artemis), hardness testing machine (Asker Shore D Model GS-719L), Perkin Elmer Thermographic Analyzer (TGA 4000) and universal testing machine/tensometer (Mossantos Tenstometer, UK, Mat. Testing Machine S/No: 9875), Micrometer gauge (Mitutoyo).

Method

Figure 1 shows Schematic illustration of the composite preparation procedure. Masticated and squeezed SCB from mature sugar cane stems was soaked in warm water for 4 hours to remove any remaining sugar, sand, or other impurities. It was then rinsed with distilled water, sun-dried for 3 days, and ground into 3–4 cm-sized particles before being treated with 5% NaOH [30, 31, 32]. Using magnetic stirring, 5g of NaOH was dissolved in 100 ml of distiller water to create the alkali solution. In the end, a homogeneous 5 % NaOH solution was produced. The fibres were pre-treated with the 5 % NaOH solution for four hours (4h). After pre-treatment, the fibers were washed with distilled water until all untreated sodium hydroxide was removed. After washing, the fibres were pre-dried in sunlight, and oven dried at 80° C for 4 hours to ensure that percentage of moisture was reduced to a minimal level. The fibres were bow milled to get a sieve size of 70µm prior to compounding. The alkali treated (mercerized) bagasse was subsequently ground to a fine powder in a laboratory mill and stored in a dry plastic container at laboratory conditions until it was used for compounding. A mixture of the mercerized SCB, starch and PLA, PES resin, catalyst, and accelerator was vigorously stirred until it was homogeneous using a mixer process. The mixture was then poured into a flat metal bar mould with the following dimensions: 350 mm x 350 mm x 20 mm and compressed at 150–170 °C. Depending on the individual compositions, the set up was left to cure for 2 hours. As shown in Table 1, different ratios of SCB, PES, PLA, and starch were used in all of the composites,
Figure 1. Schematic illustration of the composite preparation procedure

while catalyst and accelerator levels remained constant. After the procedure, the setup was left to cool. Out of the mold, the SCB reinforced PES resin (SCB-PES composite) was removed, packaged, and kept in a lab environment until it was time for testing.

A Lloyd’s EZ50 universal testing machine was used to perform tests for flexural and tensile strength. Strip-like specimens from biocomposite were cut in accordance with ASTM D790 and ASTM D638, respectively. A 2 mm per minute speed was used for both tests using a gauge length of 115 mm for tensile tests. For each material batch, more than four specimens were used in order to guarantee good testing reproducibility. To quantify the impact of thermal degradation on PLA/coir fiber composites, a Seiko SII Exstar 6000 (TG/DTA 6200) was used for thermogravimetric analysis (TGA). Functional groups present were identified using FT-IR spectroscopy in untreated and mercerized SCB, while physical properties (surface morphology, water absorption (WA), density, and specific gravity) were used to characterize the various developed SCB-PES composites. Mechanical properties (flexural, tensile strength, impact strength, hardness, elongation at break, and Young’s modulus), abrasion resistance, dynamic mechanical properties (glass transition temperature (Tg), loss of modulus, storage modulus, and Tangent delta), thermal properties (weight loss), chemical resistance (acid, base, oxidizing agents, and detergent), and biodegradability (soil burial test) were all tested using the equipment and methods specified for each test. Using a SEM with a sputter coater and operating at an accelerating voltage of 5.0 kV and 1000 magnification, the morphology of the SCB-PES composites was examined. According to Opara et al.’s [33] description of the ASTM D 638 M method, the T. S. of the SCB-PES composites was assessed on individual test samples that had been cut into dumbbell shapes. The stress-strain curve was used to calculate the elongation at break (E), modulus of elasticity (MOE), and tensile modulus using the same instrument. Using the Tensometer Universal Testing Machine and the ASTM D 790 M method, the flexural strength of each SCB-PES composite was evaluated on five identical samples with dimensions of 40 x 10 x 3 mm. 200 N and 25 mm were the load cell’s support span and capacity, respectively, while the test speed was 5 mm/min. Using a Resil impactor and the ASTM D0256 method, the SCB-PES composites’ impact strength or resistance was tested. Using an Asker Shore D Durometer Hardness tester and adhering to the ASTM D 2240 method as described by Opara et al. [33], the SCB-PES composites were subjected to a hardness test. Using a Perkin Elmer TGA

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<tr>
<th>Filler (Wt. %)</th>
<th>Starch (Wt. %)</th>
<th>PLA (Wt. %)</th>
<th>PES resin (Wt. %)</th>
<th>Accelerator (naphtolite) (ml)</th>
<th>Catalyst (ketonox) (ml)</th>
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4000 thermo-gravimetric analyzer operating in an inert N\textsubscript{2} atmosphere flowing at 50 ml min\textsuperscript{-1}, the thermal decomposition and thermo-gravimetric characteristics of the unblended (serving as a control) and SCB-PES composites were determined. [34].

An outdoor/natural soil burial experiment that lasted six months was used to investigate the SCB-PES composites’ biodegradability. The experiment was carried out using Kim et al.’s [35] method with a few modifications in a compost soil in an open but marked area at the city dump site. Individual dumbbell-shaped specimens of predetermined sizes were cut from the blends for this purpose, and they were first engraved before being weighed and buried in the ground 12 cm or so below the surface. Every 30 days at the end, the samples were meticulously picked up, cleaned with distilled water, and oven-dried at 80\textdegree C for 24 hours before being tested for weight loss and morphology [36].

RESULTS AND DISCUSSION

Morphological structures

Figure 2 (a-f) shows the scanning electron micrographs (SEM) of the SCB-PES Composites at various fibre weights (FW) and the control. According to the amount of filler present, the micrographs depict the pictorial effect of the various levels of adhesion in the individual SCB-PES composites. Figure 2 (f) shows poor fiber distribution at the higher fibre content of 45 wt\%. It demonstrates that the presence and quantity of the fiber in SCB-PES 18, 27, and 36% increased the effective surface area that the polymer matrix was able to wet. This is a result of the fiber’s improved interface adhesion with the matrix of the polymer. The NaOH treatment encouraged interfacial bonding between the fibers and PP matrix. In terms of surface flatness and homogeneity, the composites that had pleasing physical and morphological appearances suggested that those with 18, 27, and 36% fiber had better fiber/matrix adhesion.

Tensile Strength

The results of the determinations of the tensile properties of the SCB-PES fibre biocomposites and the control are graphically represented in Figure 3. At fiber content levels ranging from 9 to 36 wt\%, the developed SCB-PES composites’ tensile strength is only marginally better than that of neat PES. The least Tensile Strength (TS) measurement was 9.33Mpa for the neat PES or the unfilled polyester matrix. With an increase in fiber content, the values of Tensile Strength in all SCB-PES composites significantly increased. This demonstrates that adding SCB as a filler to PES resin improved the TS of SCB-PES composites. Due to the SCB particles’ rough (even) surfaces, which prompted an improved polymer matrix-filler interface and, as a result, an increase in TS, the tensile properties of the material were improved.

The SCB-PES composites’ elongation at break tests as shown in Figure 3 revealed that as filler content increased, the energy at break of the composites decreased in all cases. The greater and more uniform dispersion of the filler particles in the polymer matrix, which ultimately led to improved interaction and adhesion between both constituents of the composite, was credited by James and Ronald [38] as the cause of the observed tendency. According to Trodec et al. [39], the presence of additives increased the energy at break of all composites at the filler content under consideration. When compared to Hull and Clyne’s [40] findings on the interaction between MA and -OH groups in cellulose fiber/PE composites, the recorded overall decrease in elongation/energy at break of the studied SCB-PES composites is imperative.

![Figure 2. SEM micrographs of PES-based biocomposites reinforced with (a) 0% weight (Control), (b) 9 weight percent treated SCB fibers, (c) 18 weight percent treated SCB fibers, (d) 27 weight percent treated SCB fibers, (e) 36 weight percent treated SCB fibers, (f) 45 weight percent treated SCB fibers](image-url)
Flexural strength
It can be seen from Figure 4 that as filler content at the investigational particle size increased, the developed composites' flexural strength increased as well. According to Da et al. [41], tests of mechanical properties and flexural strength of PP-mica composites increased as the composite's mica content increased. Similar findings and reports were made by Tasdemir et al. [42] and Feiyu et al. [43] regarding the mechanical properties of extruded HDPE and PP wood flour decking boards and the properties of PP composites produced with waste silk and cotton fiber as reinforcement. The flexural strength of SCB-PES fibre biocomposites is shown in Figure 4.

The catalyst (ketonax) and accelerator (napholite), which interacted strongly with the PES matrix and SCB filler, may also have increased the flexural strength of the composites. In the influence of coupling agent on the properties and structure of PVC/clay nanocomposite, Ge and Jia [44] found that addition of a coupling agent improved the properties and decay resistance of hazel nut husk filled polymer composites. The SCB-PES composites' flexural strength, which increased as SCB content increased, was also found to decrease at 45 and 54%, following a similar pattern to that seen for tensile strength. It's likely that the excessive amounts of SCB filler in the aforementioned composites overcame the PES matrix's ability to bind, decreased the degree of adhesion between it and the SCB filler, and ultimately compromised the flexural strength property. The elongation at break properties of SCB-PES fibre biocomposites is shown in Figure 5.

Impact and Hardness Strength
As shown in Table 5 the impact strength analysis showed that the neat PES resin has an impact strength of 9.23 (J/m) and that the impact strength of the SCB-PES composites increased as the amount of SCB filler was increased, with the 36% FW composite showing the greatest impact resistance. When the amount of filler in the composites exceeded the binding strength of the matrix, as was the case with flexural strength and TS, the magnitude of the increase in impact strength decreased. The adhesion-promoting catalyst (ketonax) and accelerator (napholite) added during the compounding process may have increased the impact strength of the newly developed SCB-PES composites, similar to most polymer composites. Enhanced impact strength of the composites is the effect of compatibilization on
Plasticized Polylactic Acid/TPS Blends were published by Moghaddam et al. [45]. The finding regarding the SCB-PES composites' impact strength is comparable to that made by Troedec et al. [39] regarding the impact of different chemical treatments on the composition and structure of hemp fibers. In every instance, the hardness increased as filler was added as shown in Table 5. It revealed that as the filler content increased, the composites displayed improved stiffness, rigidity, and firmness. Increased adhesion between the PES polymer and the SCB fiber may be the cause of the behavior. The outcome, which also showed a gradual increase in Shore D hardness, indicated a higher degree of interfacial adhesion between the SCB and matrix. Similar findings regarding the impact of alkalization on sponge fiber (luffa cylindrical) on the mechanical and WA of reinforced composites were made by Mohanta and Acharya [46].

The impact strength of SCB-PES Composites based on different weight percent of fibers is shown in Figure 6. Also, the hardness of SCB-PES Composites with respect to fiber content is shown in Figure 7.

**TGA thermal analysis**

In Figure 8, the thermograms (TG) of the SCB-PES Composites and control at various temperatures are displayed. The weight of the pure PES matrix showed a nearly complete disappearance between 400 and 500 °C, whereas the 9 weight percent SCB-PES composite lost nearly 50 % of its weight in the same temperature range. The most significant weight loss occurred at 300 °C. Weight losses in all of the SCB-PES composites and neat PES matrix showed a clear trend that indicated loss of stability with increasing temperature. It suggests that SCB-PES composites can only be used in applications where the operating temperature does not exceed 300 °C because no fire-retardant additives were added during the compounding process for these composites in this study. At 300 °C, the corresponding weight losses for the 9; 18; 27; 36; 45; and 54 %FW SCB-PES composites were 31.03; 28.33; 21.73; 19.07; 18.59; and 17.63 %. According to the findings, the least stable SCB-PES composite in the study was the 9 weight percent version. The SCB fiber reinforcement of the PES matrix contributed significantly to the valuable properties of Tensile Strength, Flexural and Impact Strength, and other mechanical properties of the composite, as was observed in the TGA of the SCB-PES composites.

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<th>Table 5. Hardness of SCB-PES Composites</th>
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<td>Filler (wt. %)</td>
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<td>Hardness Shore D</td>
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![Figure 6. Impact strength of SCB-PES Composites](image1)

![Figure 7. Hardness of SCB-PES Composites](image2)

![Figure 8. TGA of SCB-PES Composites and the control sample at different Temperatures °C](image3)
Biodegradable effect
Micrographs of composites after six months buried in the ground is shown in Figure 9. SEM micrographs of PES reinforced composite at end of 6 months soil burial test with (a) 18 wt. % SCB fibers, (b) 27 wt. % SCB fibers, (c) 36 wt. % SCB fibers, (d) 45 wt. % SCB fibers, (e) 54 wt. % SCB fibers.

Before the soil burial tests, the micrographs of the developed composites as shown in Figure 2 differed from Figure 9, and this difference was due to variations in the levels of adhesion between the composite materials. In contrast to the composites before burial in the soil, the buried samples' adhesions showed signs of visible mutilation, indicating varying degrees or levels of degradation that are thought to be caused by microbial attack. The findings demonstrated that as the composites' starch and bagasse contents increased, so did the degree of degradation. As an organic filler with a high concentration of -OH groups, SCB naturally breaks down, but the starch was added to increase the level of microbial attack on it. The biodegradability of neat Polyester and Polyester / sugar cane bagasse fibre is shown in Figure 10.

According to the SEM findings, the soil microbes at the dump site attacked the samples with 54% filler weight the most and those with 18% the least. These changes are primarily caused by the impact of extra-cellulose enzymes and biosurfactants on the chemical components of the SCB-PES composites, which the composites interacted with in the soil. Leaching of the SCB filler particles may have also resulted in the formation of the pores on the composites' surfaces, weakening or otherwise reducing the strength/hardness of the SCB-PES composites with relatively higher filler weight compositions. The molecular structures of the SCB-PES composites were disassembled through metabolic or enzymic processes, as evidenced by the micrographs taken after a 6-month soil burial test. Figure 5 illustrates how the weight loss of material samples after 1, 2, 3, 4, and 6 months burial test was used to characterize the biodegradability level of biocomposites. Nearly no weight loss is observed for neat PES until 6 months with 2.22% weight loss. The greatest amount of weight loss (85.34%) occurred at a fibre content of 54 % after 6 months. The fact that treated fiber biocomposites lost more weight during compost burial tests may be because there was still some residual NaOH present, which caused the PLA polymeric chains to break more quickly. Regardless of fiber treatment, the 54 % (wt) of fiber appears to be the biodegradable effect's saturation point, above which an excessive amount of fibers tends to form individual fiber bundles or aggregates rather than actively interact with PES for any further rapid biodegradation process brought on by bacterial growth. In their study, Oliaei et al. [47] observed that degradation of neat c-PCL samples begins at the outer surface and progresses more slowly inward. Different mechanisms will be used as part of the biological degradation process by microorganisms and their enzymes, which could alter the rate of degradation in comparison to the current findings.

Preeti et al. [48] described elements that affect the ultraviolet (UV) stability of biopolymers, including techniques to increase photoresistance and their effect on biodegradation. They talked about the fate of biopolymers in the soil and how residues affect the health of the soil. A small number of studies have also investigated the adverse effects (such as microbial toxicity) of composite and biopolymer degradation in soil [49, 50].

![Figure 9. SEM micrographs of PES reinforced Composite at end of 6 months soil burial test with (a) 18 wt. % SCB fibers, (b) 27 wt. % SCB fibers, (c) 36 wt. % SCB fibers, (d) 45 wt. % SCB fibers, (e) 54 wt. % SCB fibers](image-url)
CONCLUSIONS

In this study, composite materials made of polyester (PES) and sugarcane bagasse (SCB) resin with varying amounts of SCB fiber as filler have been produced in the presence of starch and an effective catalyst and accelerator. The findings show that SCB and PES resins can be combined with starch and efficient catalyst and accelerator systems to produce composites that are advantageous for end-use applications and environmentally friendly. Developing composites with acceptable morphology, mechanical, and thermal properties, as well as other physical properties, requires procedures. These procedures help to control the introduction of additives and their timing and ensure that the designated time is adhered to. The morphology of the composites demonstrated the skillful blending of polyester resin and sugarcane bagasse to produce homogeneous materials. The demonstrated superior thermal stability of the developed SCB-PES composites in comparison to the same property in the unmodified resin attests to its enhanced performance for end-use applications. Mechanical characteristics show a noticeable improvement. The tensile strength of polyester resin is also improved by the addition of sugarcane bagasse filler. As the filler loading increased, the composites’ values of elongation at break generally decreased. The flexural, tensile, and elongation at break values were all significantly improved by the addition of sugarcane bagasse (SCB). Because of the proper interfacial bonding and alignment between the matrix and the filler, the effect of impact strength increased with an increase in filler loading. Sugarcane bagasse filler stiffens the polymer core, making it more resistant to indentation, which may be related to the hardness properties of the composite with increased percentage filler loading. The study showed that the degree of degradation was increased by the use of suitable catalyst/accelerator systems. According to micrographs taken six months after the composite was buried in soil for testing, the level of starch increased the degree of microbial attack on the composite, making it more biodegradable.

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REFERENCES


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