Thermal, Abrasion and Dynamic-Mechanical Properties of Sugar Cane Bagasse Reinforced Polyester Resin Biocomposites

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P A P E R   I N F O

A B S T R A C T

Biodegradable composites (SCB-PES) and a control sample were developed from polyester resin (PES) matrix and sugar cane bagasse (SCB) fibre at different matrix- fibre compositions using the compression technique. The effects of fibre content (9–54 wt%) and fibre treatment on thermal and dynamic mechanical properties of polyester / sugar cane bagasse fibre biocomposites were evaluated via thermogravimetric analysis (TGA) and DMA (Model: DMA 242 E Artemis) storage modulus (E^1), loss modulus (E^{11}), change in length of modulus (Δl), tan delta (E^{11}/ E^1) and glass transition temperature (Tg). The results showed that while the weight of the pure PES matrix showed a near complete disappearance between 400 –500 ^{0}C, the 9 % FW SCB-PES composite lost nearly 50% of its weight, in the same temperature region. The results which show that SCB-PES composites demonstrated acceptable thermal and abrasion properties portrays the effectiveness of SCB as reinforcement for PES matrix and the potential of the eco-friendly composites in the fabrication of utility materials for sustainable development. The weight loss (wt%) by the composites after 6 months soil burying test ranged from 22.24 -85.34 %.

INTRODUCTION

Composite development using polymer matrix and natural fibre as reinforcement helps to boost the generally poor mechanical properties of the polymer such as its strength and stiffness while producing value-added products from low value and abundantly available lignocellulose agrowastes. Jute, coir, sisal, pineapple peels and leaves, ramie, bamboo, banana coir and pith and other natural fibres [1, 2] have variously been used as reinforcements in polymers to develop a variety of natural fibre reinforced composites with attractive engineering properties and fabricated into a wide range of articles used in many areas. High processing cost and compromised mechanical properties are reasons for the reluctance of manufacturers to fabricate articles for various uses from natural polymers. Avérous and Pollet [3] explained that the processing cost of biodegradable polyhydroxybutyrate (PHB) characterized by a comparatively short degradation time in contrast to synthetic polymers is high despite its limiting properties of brittleness and thermal instability for engineering design. To corroborate Chun et al. [2] advocacy for the incorporation of an eco-degradant in a low-density polyethylene (LDPE)/corn stalk (CS) eco-composite for the purpose of enhancing its biodegradability. Zhang et al. [1] highlighted the capacity of plant fibre reinforced polymer composite materials to facilitate the decomposition of articles developed from them. Ramarao et al. [4]; Pawar and Purwar [5]; North and Halden [6]; Razak et al. [7]; Sahari and Sapuan [8]; Nair and Laurencin [9] and Bhatia et al. [10] and many others conducted researches and carried out surveys that projected the paradigm shift from the use of persistent and harmful synthetic polymers to their biodegradable counterparts and the prospect of replacing the former with natural fibre reinforced polymer composites in the manufacture of disposable items used in medical and engineering practices as well as in arts, catering and packaging industries as a way to promote environmental sustainability and economic development in addition to hands-on on the impending run out of fossil fuel and overcome the looming challenges of petrochemicals.

Globally grown sugar cane (SC) (*Saccharum officinarium*), also known as energy cane [11] is projected to yield 550,000-625,000 tons of sugar annually from the 5.0 - 5.5 million metric tons of SC produced in Mauritius [12]. Its rising consumer demand

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and use as industry feedstock has led to increased volume of sugar cane bagasse (SCB) by-products left after extracting the sucrose-rich juice [13, 14] for sugar production [15, 16]. The over 234 million tons of SCB produced yearly by SC farmers and mills impose additional disposal challenges/costs, resulting in SCB accumulation and consequent landscape defacing through excessive SCB depositing in open fields [17]. Though currently burnt as fuel to generate energy [18] and utilized in many industry processes, over 85% of SCB is burnt away [19], thereby polluting the air [16, 17]. Moreover, the harassing of SC mill operators by law enforcement officers who demand for clean air in place of the unhealthy smoke released during SCB burning necessitates its coordinated management and harnessing for profitable utilization which will ultimately add value to this agrowaste that has become a breeding house for disease carrying rodents. It hurts that huge amounts of fibre-rich SCB are either left to litter the environment or burnt away to pollute the air where it can be exploited as fillers in polymer composites to facilitate their biodegradability.

The aim of this study is to develop biodegradable natural fibre reinforced polymer composites using SCB and polyester (PES) resin. The result of the study will help to promote the profitable utilization of SCB and minimize the number and amounts of organic wastes generated and dumped by industries. The exploitation of SCB waste in this way will help to rid the environment of used polymers, create employment and help to abate the challenges of waste management and the associated health problems.

MATERIAL AND METHOD

Materials
PES resin, starch/PLA, catalyst (ketonox), accelerator (napholite) and processing oil were purchased from Rovet Chemicals, Lagos, while masticated SCB was obtained from sugar cane stem sellers at Ama Awusa, Owerri. Sodium hydroxide (NaOH), distilled water (H_2O), aluminium foil, cellophane bag and masking tape were purchased from Idex Scientific Company, Aba, Nigeria. 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: Q15ORES), Dynamic Mechanical Analyzer (Model: DMA242 E Artemis, Perkin Elmer Thermographic Analyzer (TGA 4000) were utilized.

Preparation of bagasse
After soaking masticated and squeezed SCB from matured sugar cane stems in warm H_2O, for 4hours in order to remove retained sugar, sand and other impurities, it was rinsed with distilled H_2O, sun-dried for 3 days and shredded to < 4cm particle size before treating with 5% NaOH. 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.

Compounding of bagasse/ polyester resin/ catalyst/ accelerator
After vigorously stirring a mixture of the mercerized SCB, starch and PLA, PES resin, catalyst and accelerator to homogeneity by a manual process, the mixture was poured into a flat metal bar mould of dimension: 350 mm x 350 mm x 20 mm and compressed at 150-170 ˚C. The set up was left to cure for 5-20 minutes depending on individual compositions (SCB, PES, PLA and starch proportions were varied while catalyst and accelerator levels were kept constant in all the composites) as indicated in Table 1 below. At the end of the process, the set up was allowed to cool, the developed SCB reinforced PES resin (SCB-PES) composite removed from the mould and wrapped in a cellophane paper. It was packed and stored at laboratory conditions until it was ready for testing. By means of dumb bell cutter, the developed SCB-PES composites were cut into individual square shapes of 15 cm x l5 cm dimension from which specimen samples (according to test specification) for the various tests and investigations were cut and prepared as requisite.

Bio-composite properties determination
The determination of resistance to abrasion by the SCB-PES composites was carried out following the combined methods of ASTM D-5707 and ASTM G-65-77/G-99 using the Wallace Test Abrader (rubber wheel apparatus). For this purpose, individual cut to specification composite test samples were sequentially

TABLE 1. Formulation of PES resin/ PLA/SCB, catalyst/ accelerator/ starch composites

Filler (%)	Starch (%)	PLA (%)	PES (%)	Accel. (ml)	Catalyst (ml)
00	0	0	100	2	2
9	1	1	89	2	2
18	2	2	78	2	2
27	3	3	67	2	2
36	4	4	56	2	2
45	5	5	45	2	2
54	6	6	34	2	2

placed over a 150 mm diameter rotating drum moving a lateral distance of 42 mm. The sample was weighed to an accuracy of 1 mg before placing on the drum that rotated at 40 revolutions per minute (rpm) in order to achieve a 0.32m/s abrasion. A 60-abrasiveness value abrasive material was thereafter placed on the sample, a constant pressure of 10N applied and the determination carried out by pushing the start button. The abrasiveness of the abrasive sheet was verified by running two tests before and after each determination (test series) using rubber comparison samples. The tested sample was then removed from the apparatus and its weight determined to the nearest 1mg. The difference in weight of the composite sample before and after the determination was recorded and the A. R. deduced from the model in Equation (1):

$$A.R.\% = {(100(X-Y))}/{Y} \qquad (1)$$

where, X and Y are the initial abraded weight and final abraded weight, respectively.

The dynamic mechanical properties of the SCB-PES were studied using DMA (Model: DMA 242 E Artemis) and following the method of ASTM D7028. Prior to analyses, the test parameters: storage modulus (E'), loss modulus (E") and Tangent of delta (Tan delta; tangent loss factor E''/E') were first configured via the proteus software computer attached to the instrument. Instrument set up configured included a 3-point bending sample holder, 30 – 110 ˚C furnace temperature range, 4 N dynamic load, 1-10 Hz frequency range and a 3 K/min heating rate. Prior to the test, individual test samples, previously moulded to 60 x 12 x 5 mm dimension were cooled along with the fixture to -100 ˚C in the presence of liquid nitrogen (N_2). By means of the 3-point bending sample holder, the samples were sinusoidally loaded at a frequency of 1 Hz and an amplitude of 15μm using the temperature sweep mode from -90 to 90 ˚C after which they were tested using the tension mode. During the test the viscoelastic spectrum showing the tested parameters were recorded as a function of temperature and read from the instrument display screen.

Thermal decomposition and thermo-gravimetric properties of the unblended (serving as a control) and SCB-PES composites was determined using a Perkin Elmer TGA 4000 thermo-gravimetric analyzer operating in an inert N_2 atmosphere that flowed at 50 ml min^{-1}. For the determination, approximately 15 mg powder portion of the individual samples was placed in an alumina crucible specimen holder and heated (pyrolysis) from 50 °C to 550 °C at a heating rate of 20 °C/min. Before the commencement of TGA measurements, each sample was examined under an optical microscope to ensure that agglomerated particles invisible to the naked eye were removed. TGA measurements of mass loss fraction against temperature and time were carried out. At the end of the determination, Arrhenius plots of % mass loss by individual composites against the temperatures of decomposition were obtained from the instrument.

RESULTS AND DISCUSSION

DMA of SCB-PES composites

The DMA of a material measures the response of a material subjected to periodic stresses as a function of temperature. Changes in length, storage moduli, loss of moduli, Tg and tangent delta properties were obtained from the DMAs of the developed SCB-PES composites under investigation. The storage modulus (E'),which also gives an estimate of temperature-dependent stiffness behavior and load bearing capability of the polymer composites is a measure of the elastic response of a material, i.e. its stored energy. Loss modulus (E"), which is the imaginary part of the complex modulus and a measure of the viscous response of a material is defined as the amount of energy dissipated in form of heat by the materials during one cycle of sinusoidal load.

Tan delta (E"/E'), known as damping is the ratio of loss of modulus to the storage modulus.The glass transition temperature (Tg) of a polymeric material and its composites is the temperature at which the polymer turns from a ductile material to a hard, brittle one. T(g) is unique for every polymer material and is the property that makes a given amorphous polymer better suited for some applications over other. At the T(g), a polymer transits from a glass to the rubber-like state, marking a region of dramatic changes in the physical properties, such as hardness and elasticity.

The results of DMA investigations in Table 2 showed that the SCB-PES composites containing 18, 27 and 36 % FW demonstrated satisfactory performance outcomes in comparison with the others. 27 % FW SCB-PES composite showed a low E^{11} Mpa value of 161 but demonstrated an inflation of 68.0, the highest E^1 at 60.3 ºC and a T(g) of the same value. It has the highest E^1 of 79.6 Mpa at 66. 0 ºC though with a lower E^{11} Mpa of 161. The hardness of a polymeric material decreases simultaneously with the E^1 and E^{11}/E^1. The height of the E^{11}/E^1 peak typically lies between 0.1 and 1.2. The onset of deformation can be taken to be the T(g). The SCB-PES composite containing 36 % FW demonstrated an excellent E^{11}/E^1 value of 0.476 at 96.4ºC. Modelled by a dash pot, the 36 % FW SCB-PES composite showed a change in length (Δl) at 90ºC. The change in length (Δl) is suggested to be attributable to the chemical deformation of the SCB- PES while it contacted the environment of changing temperature. From the DMAs, the onset of E^1 in the SCB-PES composites occurred in the range: 65.8 - 99 while its inflation (inflation of the E^1) occurred between 61.5 and 71.7. The temperature of the E^{11} ranged from 56.8 – 66 ºC while the Loss Modulus fell between 154 and 208 Mpa. In E^{11}/E^1, the temperature range for the entire investigated SCB-PES composites

TABLE 2. DMA/DMTA of SCB-PES composites and the control sample

Samples	Storage Modulus (E^1) Onset; Inflation		Loss Modulus (E^{11}) Temp ºC Mpa		Tangent Delta (E^{11}/E^1) Temp ºC Peak		Change in length ($\Delta\ell$) Temp.0 ºC µm		Glass Trans Temp. (Tg) 0 ºC
00	90.7	61.5	63.5	154	90.9	0.589	81.0	-427.2	54.8
9	80.6	63.3	64.5	156	87.5	0.517	76.9	-333.0	53.0
18	91.5	66.0	63.5	188	94.5	0.487	85.1	-408.5	57.4
27	79.6	68.3	66.0	161	95.5	0.495	86.4	-326.6	60.3
36	90.0	68.0	61.5	208	96.4	0.476	90.0	-427.9	54.9
45	74.6	71.7	56.8	160	95.2	0.466	86.3	-390.1	46.8
54	65.8	65.1	61.4	151	102	0.436	90.4	-299.2	49;3

was 87.5-102 ºC while the E^{11}/E^1 peak for the samples laid between 0.436 and 0.589. The Δl occurred within the temperature range of 76.9 – 90.4 ºC and 299.2 - 429.9µm. Lastly, the Tg of the SCB-PES composites ranged between 46.8 – 60.3 º C. Evaluation of the results from the DMA/DMTA SCB reinforced PES matrix reveal the invaluable dynamic mechanical properties and consequently, the potential of the various SCB-PES composites to serve in the fabrication of articles for household/domestic use and other applications.

Abrasive resistance of SCB-PES composite

The results of the determination of Abrasive Resistance (A.R.) of SCB-PES Composites and the Control are illustrated graphically in Figure 1. Abrasion resistance, also termed the tribology refers to the ability of materials and structures to withstand abrasion and resist mechanical wearing due to contact with another surface. Wearing occurs when a hard-rough surface slides across a softer surface, causing the undesired removal of material from the surface. It is a measure of the ability of materials to withstand abrasion and is significant in applications in the fabrication and exploitation of movable and fixed parts in wear-prone settings. The result of the determination of the AR by the SCB-PES

composites showed a steady decline in AR. The observed decrease in AR values signifies that the number of rubs needed to abrade 1g of the studied composite reduced as the % Filler Weight (FW) continued to be increased. This is attributable to the increase in the number of voids in the composites as a result of the increasing filler content. Voids-rich materials are vulnerable to abrasion when they are subjected to the abrading process due to the presence of such voids in the structure. Although AR values of the composites are lower than that of unfilled matrix, yet the SCB-PES composites are suitable for use in fabrication of eco-friendly materials since polymer composites of comparable AR have reportedly been used in such areas [20].

TGA analysis

The thermograms (TG) of the SCB-PES Composites and control at different T ˚C are presented in Figure 2. The TGA is an analytical technique used to determine the thermal stability of a material and its volatile fractions by monitoring the weight changes that occur as a sample of the material is heated at a constant rate. Concisely, it measures the weight (wt) and hence the mass of a sample as a function of temperature and time. Changes in the weight of a polymeric material may be caused by decomposition and oxidation reactions in addition to physical processes such as sublimation, vaporization and desorption. TGA enables the detection of changes in the mass of a sample (gain or loss), evaluates the stepwise changes in mass (usually as a % of the initial sample mass) and determines the temperatures that characterize each step in the mass loss or mass gain curve. The results of the TGA of the new SCB-PES composites showed that while the weight of the pure PES matrix showed a near complete disappearance between 400 –500 ºC, the 9 % FW SCB-PES composite lost nearly 50% of its wt. in the same temperature region. Wt. losses in all the SCB-PES composites and pure PES matrix control showed to demonstrate a clear trend that indicated loss of stability with increasing temperature with the most significant Wt. loss occurring at 300 º C. Since SCB-PES composites in
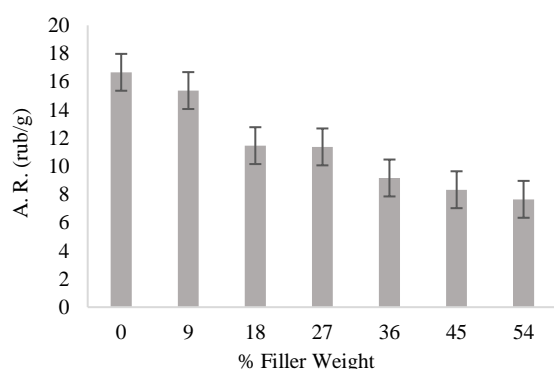

Figure 1. Abrasion resistance of SCB-PES composites and control against FW%
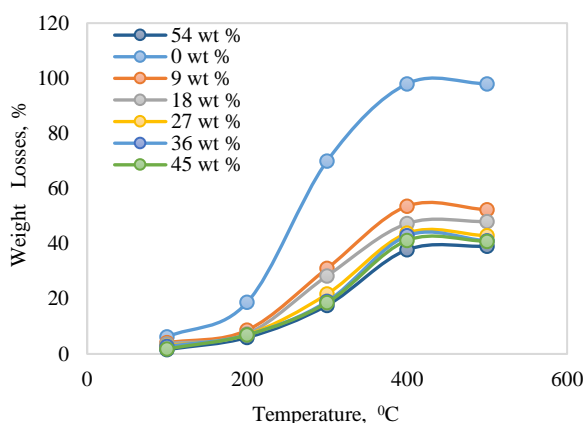
Figure 2. TG of SCB-PES composites and the control sample at different T °C

this study were compounded without any fire-retardant additive, it suggests that they can only be used in applications where the operating temperature does not exceed 300 °C; otherwise, fire retardant additives must be incorporated at the compounding stage. Wt. losses in the 9; 18; 27; 36; 45 and 54 %FW SCB-PES composites at 300 °C respectively were 31.03; 28.33; 21.73; 19.07; 18.59 and 17.63 % respectively. The result showed that 9 %FW SCB-PES composite demonstrated the least stability among the SCB-PES composites in the study. The observed trend in the TGA of the SCB-PES composites could be attributed to the influence of the reinforcing SCB on the PES matrix which is known to demonstrate and contribute appreciably to the valuable mechanical properties of T. S, flexural as well as impact strength and other mechanical properties of polymeric materials and their composites. Polymer matrix/vegetable composites usually undergo random degradation resulting to the formation of fragments with free radical ends [21]. These fragments may either depolymerize to form daughter free radicals and unsaturated products or intra-molecular transfer to form secondary free radicals. These later free radicals may form products similar to those of the previous ones. Further scission may also take place in the degradation process in the manner of the pathway suggested by Samal et al. [22].

SEM and biodegradability of SCB-PES composites and control
Figures 3a-3e show scanning electron micrographs (SEM) of the SCB-PES Composites at different filler weights (FW) after 6-months of burying in the soil. The SCB-PES composites demonstrated significant losses in weight after 6-months of burying in the soil. The observed differences between the micrographs of the developed composites before and after the soil burial

tests are due to variations in the degrees of adhesion between them. Adhesion which showed to be visibly mutilated and categorized in the buried samples signify different degrees or levels of degradation which are thought to be the result of microbial attack (by enzymatic mechanism) while the composites laid in the soil. The results showed that the degree of degradation increased as both the amount of bagasse and starch content in the composites were increased. As an -OH groups rich organic filler, SCB degrades naturally but the starch was added to boost the extent of microbial attack on it. In addition to the prevalent microbial activities at the dump site, the site was chosen to conduct the experiment in an environment that is sufficiently close or matched the end-of-life scenarios of the SCB-PES composites. The results in Figure 4 showed that the sample containing 54 % FW and that of 18 % respectively were the most and least attacked by the soil microbes at the dump site. Noticeable cracks or pores which were seen on the surfaces of the SCB-PES composites, the sizes of which appeared larger in the high FW SCB-PES composites also demonstrated a similar trend.
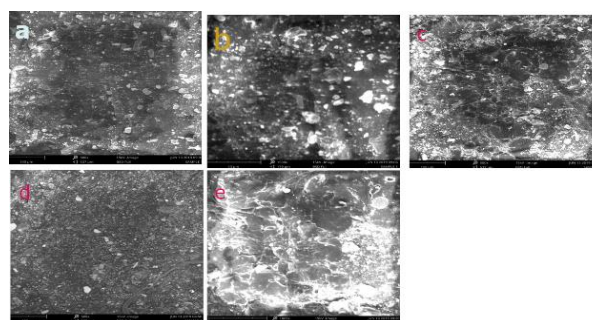

Figure 3. SEM of composite samples at the end of 6 months Soil Burial Test for (a) 18, (b) 27, (c) 36, (d) 45, and (e) 54 % filler weights repectively
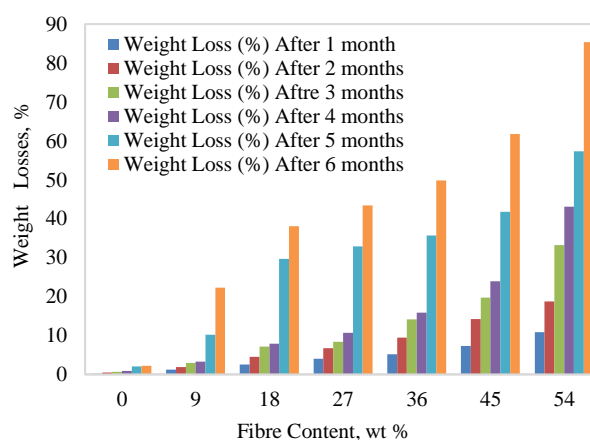

Figure 4. SCB-PES composites weight losses and control after 6 months of burying in the soil

CONCLUSION

Thermal, dynamic-Mechanical and abrasive properties of SCB-PES biocomposites with alkali treatment have been successfully evaluated. The results obtained in this study show that blending of SCB and PES resin in the presence of starch and efficient catalyst and accelerator systems can be exploited to produce eco-friendly composites with valuable end-use applications. The order and manner of introduction of the additives and the observation of the set time are necessary to guarantee the development of composites of satisfactory thermal, dynamic-mechanical as well as abrasive physical properties. The DMA of the 18, 27 and 36 % FW biocomposites demonstrated satisfactory performance in comparison with the others. Due to embedded SCB fibres with lower thermal degradation temperatures, the thermal stability of biocomposites decreased irrespective of increasing fibre content. Moreover, the demonstration of superior thermal stability by the developed SCB-PES composites against the same property in the unmodified resin attests to the potential of formation of new products. The micrographs after 6 months soil burial test show that the molecular structures of the SCB-PES composites were broken down through metabolic or enzymic processes. Engineers and others engaged in environmentally friendly product development and item fabrication can process the developed SCB-PES composites, explore and identify their end-uses as well as endorse their utilization in households and industries.

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چکیده

کامپوزیت‌های تخریب‌پذیر (SCB-PES) و یک نمونه کنترل از ماتریس رزین پلی استر (PES) و فیبر باگاس نیشکر (SCB) در ترکیبات مختلف فیبر ماتریس با استفاده از تکنیک فشرده‌سازی تهیه شد. اثرات فیبر (۵۴-۹ درصد وزنی) و فرآوری فیبر روی خصوصیات مکانیکی حرارتی و دینامیکی بیوکامپوزیت‌های الیاف پلی استر/ نیشکر باگاس با استفاده از آنالیزهای گرماسنجی (TGA) و مدول ذخیره‌ای DMA (مدل DMA 242 E Artemis) (E^1)، مدول اتلاف (E^{11})، تغییر در طول مدول (Al)، دلتا tan (E^{11}/E^1) و دمای انتقال شیشه (Tg) بررسی شد. نتایج نشان داد در محدوده دمایی یکسان، در حالی که وزن ماتریس خالص PES ناپدید شدن کامل بین ۴۰۰ تا ۵۰۰ درجه سانتیگراد را نشان داد، کامپوزیت ٪۹ SCB-PES FW تقریباً ٪۵۰ از وزن خود را از دست داد. نتایج حاکی از این است که کامپوزیت‌های SCB-PES خواص حرارتی و سایشی قابل قبول را ارائه داده‌اند، و اثربخشی SCB را به عنوان تقویت‌کننده برای ماتریس PES و پتانسیل کامپوزیت‌های سازگار با محیط زیست در ساخت مواد ابزار برای توسعه پایدار را نیز نشان می‌دهد. کاهش وزن (درصد وزنی) توسط کامپوزیت‌ها پس از ۶ ماه آزمایش دفن خاک، از ۲۲/۲۴ تا ۸۵/۳۴ درصد بود.