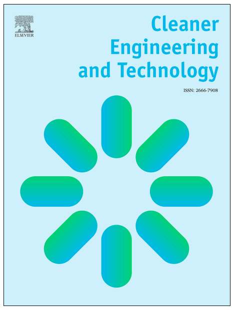

Properties of polylactic acid and biochar-based composites for environment-friendly plant containers

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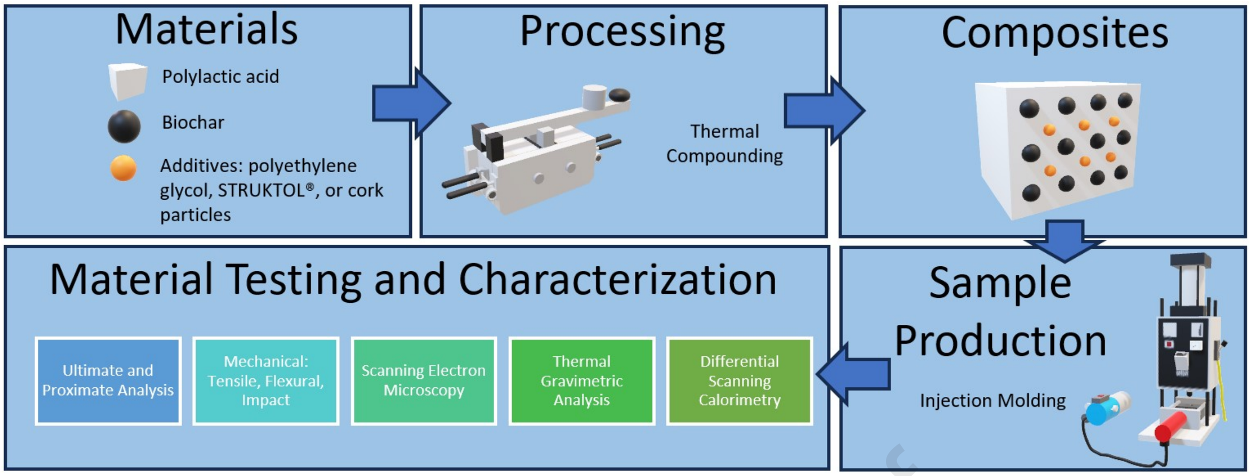
Materials
Polylactic acid
Biochar
Additives: polyethylene glycol, STRUKTOL®, or cork particles
Processing
Thermal Compounding
Composites
Sample Production
Injection Molding
Material Testing and Characterization
Ultimate and Proximate Analysis
Mechanical: Tensile, Flexural, Impact
Scanning Electron Microscopy
Thermal Gravimetric Analysis
Differential Scanning Calorimetry

Properties of polylactic acid and biochar-based composites for environment-friendly plant containers

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Abstract

Traditional, petroleum-based plastics are known to linger in the environment for years due to their non-biodegradability and non-sustainability, eventually fragmenting into microplastics and contaminating waterways and agricultural systems. Therefore, many researchers have focused on bio-based and biodegradable polymers, such as polylactic acid (PLA). Biochar, a byproduct of the bio-fuel production process, has been investigated as a filler to reduce the amount of PLA used and improve mechanical properties. This research focuses on effect of biochar amount on PLA composites and the impact of individual additives consisting of polyethylene glycol, STRUKTOL® (a plasticizer), and cork particles. It was found that the cork-based composites (51-54 MPa) improved tensile strength more than both of the plasticizer-based composites (41-46 MPa). While the scanning electron microscopy images showed some aggregation in the 10 wt.% biochar composite, the cork composites (1 and 3 wt.%) showed slightly less aggregation than other composites. Additionally, analysis revealed high carbon (81%) and fixed carbon (74%) concentrations, respectively, in the hardwood-derived biochar, indicating potential for carbon sequestration.

Key Words: polyethylene glycol; STRUKTOL®; cork; plasticizer; injection mold

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1. Introduction

According to the United States Environmental Protection Agency, the US alone produced over 35 million tons of waste plastic in 2018 (the most recently available data), but only ~8.7% reached recycling sites. This leaves much of the plastic waste to either unsustainable disposal methods (landfills, incineration, etc.) or improper disposal methods (littering, etc.) [1]. Without intervention, unsustainably disposed plastic waste could reach over 200 million metric tons per year globally by 2060 [2]. Traditional, non-biodegradable plastics have been studied not only for their environmental contamination [3], but for their potential health problems, including complications with the immune system, genetic abnormalities, and fetal defects [4-6]. The consumption/usage of plastic-based products shows no sign of reduction, though, especially in single-use plastic goods. Therefore, many researchers have begun studying alternative biodegradable, bio-based polymers as opposed to the traditional petroleum-based ones.

In such studies, several polymers have appeared as potential replacements, with an emphasis on polylactic acid (PLA) due to its promising mechanical properties and current commercial implementation. Classified as a biodegradable polymer, PLA faces two main problems in its widespread application: its brittle nature and expensiveness [7, 8]. PLA must be able to mechanically and financially compete with traditional polymers in order for industries to have an incentive to replace conventional materials. While potentially an environment-friendly polymer due to being both biobased and biodegradable, there are conflicting reports regarding its environmental impact. A life cycle assessment revealed the main drawback of PLA is its production process, in which much of the carbon dioxide (CO_2) is released into the environment [9]. However, Kane et al. (2022) examined the environmental impact of adding up to 40% biochar filler to the PLA polymer matrix, with results indicating a reduction in global warming effects [10]. Biochar (BC), a byproduct of the biofuel production process, offers a potential solution in the form of filler material to cut costs and assist with CO_2 management due to its high carbon concentration. In small quantities (1-5%), biochar has shown some improvement in specific PLA properties due to its porous structure and surface properties [11-14]. However, these concentration percentages are small in terms of the overall polymeric matrix.

However, biochar research has yielded conflicting viewpoints on results at times. Aup-Ngoen and Noipitak (2020) found that adding 0.25% of biochar to PLA polymer matrices had a negative impact on both tensile strength and elongation at break, while Huang et al. found improvement with 1% grapevine-based biochar in PLA composites [11, 13]. Zhang et al. investigated biochar microspheres in several studies (2023 and 2024), in concentrations up to 5 wt.% of PLA films, citing increases in tensile strength and modulus [14, 15]. At higher percentages, though, there is significantly less discrepancy as biochar aggregates, leading to poorer mechanical properties, as seen in a study by Zouari et al. (2022) for 20% biochar/PLA composites [12]. Zhang et al. (2022) investigated 40 wt.% of biochar in PLA composites and found a decrease in tensile, flexural, and impact properties. However, they determined that additives could improve the mechanical properties of biochar composites [16]. Therefore, additives (such as either plasticizers or natural fibers) are often researched in order to improve these properties.

Additives within composites were investigated to determine what may yield the most useful properties for a particular application. These additives have ranged from natural fibers (hemp, cellulose, cork, etc.) [12, 14, 17-19] to plasticizers including polyethylene glycol (PEG) or glycerol [19-21]. PEG and STRUKTOL® were found to improve mechanical properties of pristine

PLA and non-biochar-based PLA composites (dried grains, wood flour, lignin etc.) [19-22], though they have not been tested in PLA/biochar (BC) composites with an emphasis on injection molding. Additionally, cork particles have been tested within pristine PLA and were found to have improved impact strength and elongation at break [17, 18]. Cork/PLA/BC based composites have not been studied up until this work. Both plasticizers and cork-based materials were compared in this study for an examination on property changes resulting from different classifications of additives.

This research examines PLA with a higher concentration of biochar (10 wt.%) with each of the three additives tested for future commercial plant containers. The idea behind the plant containers is to find a durable, inexpensive, and biodegradable/compostable material for the replacement of traditional polymers. Upwards of 4 billion plant containers are produced per year, with vegetables, herbs, and annuals being the most popular plants for biodegradable containers [23]. This indicates that biodegradable plant containers may be best applied to agricultural fields where the container biodegrades in the ground with the plant, while biochar filler could sequester carbon underground after the container degrades. Often, these plant containers are made with polypropylene (PP), providing our work with target mechanical and thermal properties. Investigations of PLA/biochar/additive composites are further highlighted in their applicability to plant containers. Within this work, PLA/biochar composites were developed and tested with the inclusion of three additives: PEG, STRUKTOL®, and cork particles. These three additives were chosen as cork and STRUKTOL® combined with PLA and biochar have not been tested, while PEG, biochar, and PLA have limited literature and have not been tested using injection molding methods. The additives were added in two lower concentrations, 1 wt.% and 3 wt.%. These percentages were chosen based on previous literature studies, while keeping high amounts of biochar at the forefront of the investigation. The properties tested included tensile, flexural, and impact strength as well as properties of the biochar including composition. Furthermore, scanning electron microscopy, differential scanning calorimetry, thermogravimetric analysis, and the physisorption analysis were done to analyze morphology and thermal properties of the composites.

2. Experimental

2.1 Materials

Polylactic acid (3251D Ingeo Biopolymer), was purchased from NatureWorks (Minnetonka, MN, USA). Hardwood-based biochar was produced using a pilot scale fluidized bed gasifier at 850°C [24]. Polyethylene glycol (Spectrum 6000 Distearate, Spectrum Chemical, New Brunswick, NJ, USA), STRUKTOL® (TR451, Struktol Company of America, Stow, Ohio, USA), and untreated cork (size 7 stoppers, Manton Industrial Cork SE, Hauppauge, NY, USA) were purchased. The cork stoppers were ground and sieved to under 850 µm as found to be an appropriate size range through experimental observation. A concentration of 10 wt.% of biochar was chosen based on previous experimental trials.

Table 1. Weight percentages of composites and labels used in this work. The abbreviations correspond to the filler or additive as well as the percentage.

Composites	Materials (wt. %)				
	PLA	**Biochar**	**PEG**	**STRUKTOL®**	**Cork**
PLA	100	0	0	0	0

10BC	90	10	0	0	0
1PEG	89	10	1	0	0
3PEG	87	10	3	0	0
1S	89	10	0	1	0
3S	87	10	0	3	0
1C	89	10	0	0	1
3C	87	10	0	0	3

2.2 Composite Production

A horizontal planetary ball mill (MTI Corporation Richmond, CA, USA) was used to produce smaller, more uniform particle sizes for the biochar. The biochar was milled for 2 hours (1 hour forward and 1 hour reverse) at 800 rpm. Both the biochar and the PLA were then dried at a minimum overnight at 70°C in a drying oven to remove moisture before thermal compounding [25].

The material was taken as needed from the drying oven and added to the mixer (2128 C.W. Brabender internal mixer, Brabender, Inc., Hackensack, NJ, USA) for the thermal compounding process. For the composites and pristine PLA, the mixer worked well between 170-175°C, with previous literature suggesting a time of 5 minutes at a speed of 100 rpm [25]. Compositions, along with their respective labels can be found in Table 1. The material was then removed from the internal mixer and placed in a granulator. The top feed granulator (Shini USA, Willoughby, OH, USA) ground the polymer and composites for placement into an injection mold barrel. The shredder composite pieces were first placed in a drying oven at minimum overnight (105°C) before use in an injection mold. The injection mold (Proto-ject 150HP, with a Zinko Hydraulic Jack) was purchased from Manning Innovations (Halls, TN, USA). The barrel was found to be best set at 185°C, while the nozzle was best at 190°C, based on experimental trials. The air pressure used was 60 psi.

2.3 Biochar Analysis

Ultimate analysis of the biochar sample was done using an Elementar Vario MICRO (Ronkonkoma, NY, USA) and run in triplicate. Proximate analysis was done as well, in accordance with ASTM D1762.

2.4 Mechanical Testing

Tensile specimens of Type 4 were produced according to ASTM D638. For tensile calculations, the width and thickness of each specimen were taken to be 6 mm and 3.2 mm (respectively) for all calculations. Little variation in width and thickness was found, making the ASTM standard width and thickness acceptable. The samples were tested using a Mark-10 Digital Force Tester (Model F1505, Mark-10 Corporation, Copiague, NY, USA) with a force sensor maximum load of 6.7 kN. Flexural specimens were produced according to the ASTM D790. The samples were tested using a Mark-10 ESM750S motorized test machine (Copiague, NY, USA) with a maximum load of 500 N. Flexural strength and modulus of elasticity were calculated according to ASTM standards. Similarly, Izod notched impact samples were produced according to ASTM D256. They were measured using a Trinocular Stereo Zoom Microscope Model 420T-430-PHF-10 (National Optical & Scientific Instruments Inc., Schertz, TX, USA) and tested using XJUD Digital

Charpy Izod Impact Testing Machine (Deli Group Co. Ltd., Ningbo, China). The pendulum energy load was 2.75 J. All mechanical composites were tested in sample sizes of ten. One-way ANOVA testing was performed to determine statistical difference ($\alpha < 0.05$). Least Significant Difference tests were done as well, and each composite was grouped accordingly.

Before each mechanical test (tensile, impact, and flexural) an environmental chamber (Model AA-5460A, Espec Corp., Hudsonville, Michigan, USA) was used to condition the samples. All samples were maintained at 23°C and 50% following ASTM Standard D618.

2.5 Morphology Characterization

Images were taken of the biochar and composites using a ZEISS EVO50 Scanning Electron Microscope (Carl Zeiss Microscopy, New York, USA) at an accelerating voltage of 20 kV. The composites were sputter coated beforehand using gold with an EMS 150R ES sputtering system from Electron Microscopy Sciences (PA, USA). The magnifications used were 1000x for the composites and 1500x for the biochar. Charging did occur on several composite images due to inconsistent contact of the sample with the SEM stub. These appear as small bright spots in a few images.

Additionally, physisorption analysis was done to determine the biochar surface area, average pore size, and pore volume using a Tristar II Plus Model (Micrometrics, Norcross, GA, USA). The biochar was placed in a vacuum at 200°C for two days, after which it was degassed at 90°C for 2 hours and 150°C for 24 hours. The surface area was measured using Brunauer-Emmett-Teller (BET) equation. The average pore size and pore volume were determined according to BJH

(Barrett-Joyner-Halenda) desorption values. For both SEM imaging and BET analysis, cork particles were not able to be analyzed due to the oils emitted when these processes were run.

2.6 Thermal Properties Testing

Thermogravimetric analysis was done using a TGA 5500 (TA Instrument, Newcastle, DE, USA) with a nitrogen mass flow rate of 25 mL min^{-1}. The temperature range tested was up to 1000°C with a heating rate of 20°C min^{-1}. Additionally, differential scanning calorimetry (DSC) was done using TA Instruments Q200 (Newcastle, DE, USA). A heat-cool-heat cycle was used with temperatures ranging from 20 to 250°C with heating and cooling rates of 10°C min^{-1}. The samples were repeated in triplicate and statistically analyzed using one-way ANOVA.

3. Results and Discussion

3.1 Biochar Composition

Table 2. Ultimate and proximate analysis of biochar.

Property	**Percent Composition (wt.%)**[a]
Carbon	81.12
Hydrogen	1.48
Nitrogen	0.26
Sulfur	0.47
Oxygen (by difference)	5.86
Moisture Content[b]	5.82
Volatile Matter	14.36

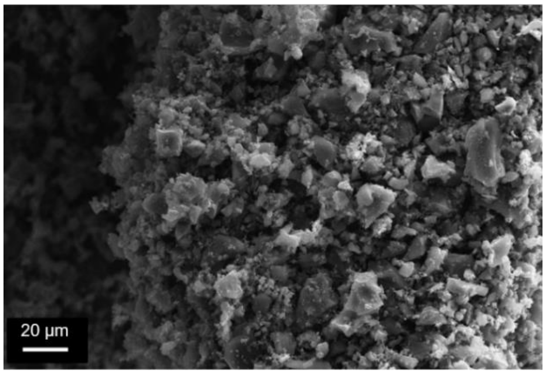
Fixed Carbon	74.83
Ash	10.81

[a] dry basis. [b] wet basis

Table 2 shows the results of the ultimate analysis for the biochar. The resulting composition was found to be 81.12% carbon, 1.48% hydrogen, 0.26% nitrogen, 0.47% sulfur, and 5.86% oxygen. Carbon content is important as one of the goals is the sequestering of significant amounts of carbon. Previous biochar/polymer composite studies cite a carbon percentage between 67-76%, with feedstocks including cassava rhizome [11], date palm waste [26], and tree bark [27]. The higher carbon content of the material adds to its usefulness in potential environmental remediation, with high fixed carbon content promoting stable carbon. Nitrogen addition to soil offers potential improvement in plant growth and limits nitrogen-enriching soil amendments (such as fertilizers) [28]. An advantage of a soil remediating plant container could be the additional plant growth due to added nutrients with a simpler route for deposition into the soil. It was also investigated that sulfur-enriched biochar could assist in plant growth within high salt-containing soils [29]. Therefore, some sulfur content within the biochar could improve certain soil types for increased agricultural yield.

In terms of the proximate analysis results (moisture, volatile matter, fixed carbon, and ash contents), biochar's low moisture content could prevent water from disrupting the polymer matrix, thereby decreasing incompatibility. Additionally, it was found that high ash content from municipal waste-based biochar caused more aggregation as opposed to the lignin-based biochar from woody biomass that showed less aggregation [30]. This could influence the dispersion of biochar in this work as it is made from hardwood pellets, rich in lignin, to help minimize aggregates. Currently there is limited work on how both ultimate and proximate analysis results can affect polymer/biochar interactions and is an area that should be investigated further in the future.

Table 3. Results for the BET characterization of biochar.



BET - Biochar	
Surface Area (m^2/g)	219.67
Average Pore Size (nm)	5.24
Pore Volume (cm^3/g)	0.075

Figure 1. SEM image of ball-milled biochar at 1500x magnification.

Figure 1 above was taken of the ball-milled biochar at 1500x magnification under SEM. The scale bar is defined at 20 µm, with many of the particles being close to this value or smaller. Since the biochar was ball milled to increase dispersion and decrease the possibility of aggregation, it appears as a fine powder. Table 3 indicates that there was an average pore size (5.24 nm) and volume (0.075 cm^3/g), as well as available surface area for interactions to take place (219.67 m^2/g). Upon review of literature, it was determined that the surface area of this study's biochar falls within range of previously reported values 185-300 m^2/g [26, 27, 31-34]. This indicates that biochar may have produced a good interlocking mechanism between itself and the polymer, which is promising for this study.

3.2 Mechanical Properties

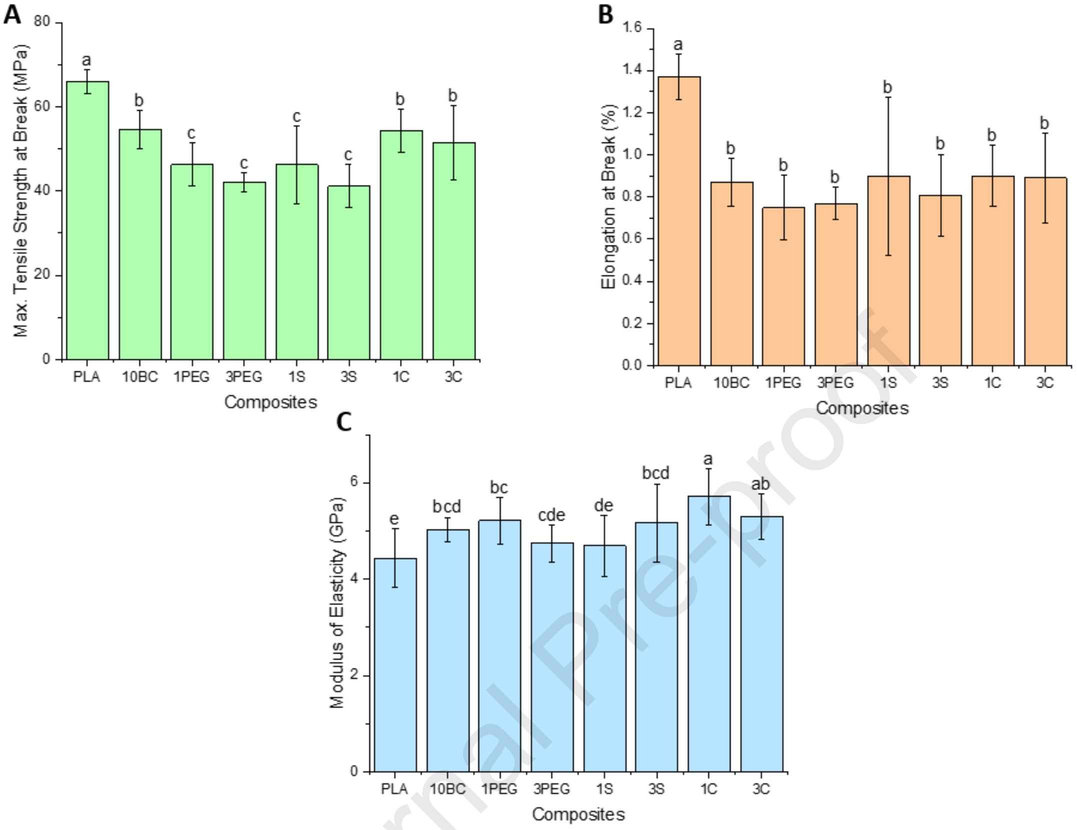


Figure 2. Maximum tensile strength (A), elongation at break (B), and modulus of elasticity (C) calculated from the results of the tensile tests for pure PLA and biochar composites.

Figure 2 demonstrates the results of the tensile tests done for all composites where maximum tensile strength, elongation at break, and modulus of elasticity were calculated. Least Significant Difference testing is denoted by the letters directly above each bar. Analyzing the results of Figure 2A, the maximum tensile strength at break was highest for neat PLA (65.9 MPa). Previous literature has found that PLA falls within a range of 38-70 MPa [11-13, 21, 22, 25, 35, 36], with differences possibly being due to PLA grades or processing methods. However, when compared to PLA of the same grade, Li et al. (2020) determined their pristine PLA to be ~70 MPa in tensile strength and was processed according to injection molding methods as well [37]. Plant containers are often made from polypropylene, reported to have a tensile strength of ~34 MPa [38]. Though PLA exceeds this, values for tensile strength will decrease as biochar content increases. Therefore, it is important to find an additive that maintains strength at the lower biochar concentrations, before exploring higher concentrations (>10 wt.%).

The 10BC, 1C, and 3C composites were second highest, with comparable tensile strength values (51.5-54.6 MPa). Cork is able to replace some of the polymer matrix and maintain the high tensile strength to that of 10BC composite. It should be noted that it has been found that injection molding could damage the porous structures of the cork particles during processing [36], possibly leading to poorer connectivity with the PLA. It was found by Vilela et al. (2013) that when cork was added to PLA, the tensile strength decreased from ~70 MPa to ~60 MPa. Furthermore, the authors see a continuous decrease in tensile strength as cork content is added to the PLA matrix [18]. This work sees a similar trend of a ~10 MPa decrease between the pristine PLA and the PLA/BC/cork composites.

The lowest tensile strengths were found for both plasticizers. The composites for 1PEG, 3PEG, 1S, and 3S were found to be statistically similar (41.2-46.3 MPa) as well as exhibiting a decrease in tensile strength compared to neat PLA. This may be due to polymer-plasticizer compatibility as PEG is known to be hydrophilic, while PLA is hydrophobic. However, it was found that PEG improved tensile strength in PLA/ 3 wt.% PEG at ~54 MPa [39]. Therefore, the PEG may be incompatible with the biochar rather than the polymer. STRUKTOL® TR451 is used for polyolefin compatibility, making it an oleophilic/hydrophobic substance. Though STRUKTOL® TR451 is limited in research, it was tested for tensile properties with PLA and lignin composites, in which it improved the PLA/lignin strength. It should be noted that this strength was not greater than pristine PLA [40, 41]. Wasti et al. (2021) further determined that the STRUKTOL® did not improve composites to that of neat PLA [19].

For Figure 2B, elongation at break was found to be highest for neat PLA (1.8%), but similar for all tested composites (1.0-1.1%). PLA is typically considered a brittle material and is not expected to experience much change in length. This is one aspect where the material differs from the traditional PP, where elongation has been noted to be 115-350% [38]. Typically, literature reports an elongation at break of 2-14%, though this again depends on PLA grade, processing methods, etc. [11, 12, 21, 42]. However, it is interesting to note that all composites decrease to similar values. Therefore, it is advantageous that the elongation at break decreases little to none as additives replace the PLA matrix. Previous studies on PLA/cork composites found that the elongation at break was similar to that of neat PLA (100% infill), at ~6.4% [17], or only slightly lower (a decrease of ~0.10%) [18]. For PEG composites, high amounts (10-30 wt.%) have been found to increase elongation at break [21, 31]. This indicates that it may be possible that not enough plasticizer was used or the PEG was incompatible with the biochar. Furthermore, it was found that composites (PLA/lignin) with STRUKTOL® had decreased elongation at break [40, 41].

For Figure 2C, modulus of elasticity calculates the stiffness of the pristine PLA and its composites. PLA was found to be the lowest of all tested composites (4.44 GPa) but statistically similar to 3PEG (4.7 GPa) and 1S (4.7 GPa). PLA itself was found to fall within a range of 0.8-4 GPa according to previous literature [11-13, 22, 37, 42]. It is possible that some of the additives increased the stiffness of the material, but statistical testing revealed that many were similar, leading to biochar being a common property. Reduced polymer chain movement is cited as a result of the biochar addition and theorized that this is affected by the carbon content of the biochar. Therefore, the high carbon content in biochar could influence a high modulus [11]. Overall, tensile properties are an important aspect in the design of plant containers as it is important to consider how consumers will handle the containers. It is valued that the material has a high strength to resist breakage (and subsequently be classified as having good quality by consumers), however, it cannot be brittle.

Figure 3 provides the results of the notched Izod impact testing. Overall, many of the composites showed similar values (22-23.9 J m^{-1}) with slight exceptions being between 1PEG and 3S. PP has been found to have an impact strength of 18-32 J m^{-1} [38, 43]. As previously discussed for the tensile strength, though PLA is higher in some cases, it is important to maintain this value as biochar is added. Both Huang et al. (2023) and Mohapatra et al. (2014) tested Izod impact and determine their values for pristine PLA to be ~16 J m^{-1} and ~26 J m^{-1}, respectively [13, 42]. The results of this study fall between these values at 23 J m^{-1} for neat PLA, and mostly remain consistent as biochar filler and the additives are included. Across literature, the addition of some biochar was found to improve the impact strength of PLA composites. Aup-Ngoen and Noipitak (2020) were able to slightly improve pristine PLA (0.017 J mm^{-2}) using 0.25 wt.% of biochars (0.019-0.03 J mm^{-2}) derived from various pyrolyzed feedstocks [11]. Kane and Ryan (2022) saw a significant increase with 2.5 wt.% (~13 kJ m^{-2}) biochar when added to PLA (~7 kJ m^{-2}) [44]. In most cases, small concentrations of biochar contribute to higher impact strength values of PLA composites. However, an exception can be found in a work by Huang et al. (2023). This study saw increases from neat PLA at ~16 J m^{-1} up to ~22.7 J m^{-1} for 1 wt.% biochar and ~17 J m^{-1} for 10 wt.% biochar, though the authors varied biochar particle size [13]. Yet slightly lower impact strengths than the results of the current study, both the neat PLA and 10 wt.% biochar were close in value, with a difference of ~1 J m^{-1} [13]. The results of this study had a similar outcome in which the 10BC value (23.3 J m^{-1}) was numerically higher than PLA (23.0 J m^{-1}), though not statistically different.

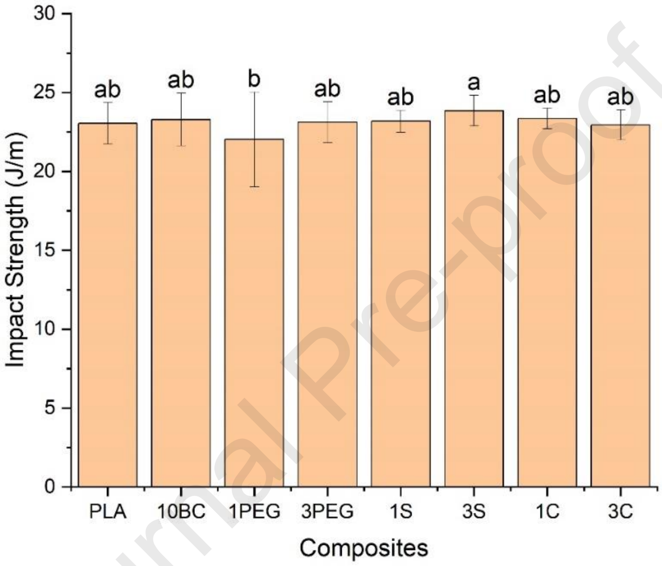


Figure 3. Results of impact testing for PLA and all composites.

PEG has been found in literature to improve impact strength to ~27 J m^{-1} (10 wt.% PEG in PLA) and ~832 J m^{-1} (20 wt.% PEG in PLA) [42]. However, significantly less PEG was used for this current study, and literature has found that a 3 wt.% of PEG could be added to a PLA matrix to improve impact strength (from 43 to 47 kJ m^{-2}) [39]. Therefore, it is possible the PEG was incompatible with the biochar more so than the PLA. For the STRUKTOL® plasticizer, little information on impact is available for this specific plasticizer grade with PLA, though one study found a significant decrease from pristine PLA (~23 kJ m^{-2}) for Charpy impact strength when

STRUKTOL® was added in 1 wt.% (~4 kJ m^{-2}). However, the authors note that the PLA/lignin/STRUKTOL® composite improved from the PLA/lignin composite by ~13% [40, 41]. In this work, the STRUKTOL®-based composites (23.2 J m^{-1} for 1 wt.% and 23.9 J m^{-1} for 3 wt.%) were comparable to that of neat PLA (23.0 wt.%) and the 10 wt.% biochar composite (23.3 wt.%).

Cork particles in particular were chosen as additives for this study because they have been found to have improved impact strength previously. Mazur et al. (2022) determined that the addition of 30 wt.% cork particles improved the impact strength to 22.3 kJ m^{-2} from that of neat PLA (7.9-10.4 kJ m^{-2}), with fiber length being noted as a possible mechanism for improved impact [17]. Similar to the STRUKTOL®, the cork particles were found to replace portions of the PLA matrix while maintaining similar levels of impact strength. Overall, impact strength can be an influential factor in designing plant containers as they will need to withstand sudden impacts with consumers handling the containers as well as potential damage during transport.

Figure 4 depicts the results of the flexural testing, including strength at break and modulus of elasticity. For PLA and PLA/biochar/additive composites, there is limited information available on flexural testing. In this study, pristine PLA strength was found to be 90.7 MPa. In comparison, Li et al. (2018) determined that the flexural strength of PLA was ~83 MPa, while ~120 MPa was determined for PLA in a work by Li et al. (2020) [21, 37]. Most noticeably, all composites decrease significantly in strength, with all the additive-based composites (40.2-69.0 MPa) being incomparable to 10BC (80.2 MPa). Ho et al. (2015) investigated flexural testing of PLA/bamboo-based biochar composites and determined that at 10 wt.%, small structural cracks formed resulting in mechanical failure [45]. This could be a possible mechanism for breakage in the flexural samples for this work. Investigations for the addition of 20 wt.% of biochar to PLA, resulted in a decrease from ~120 MPa to ~60 MPa [37].

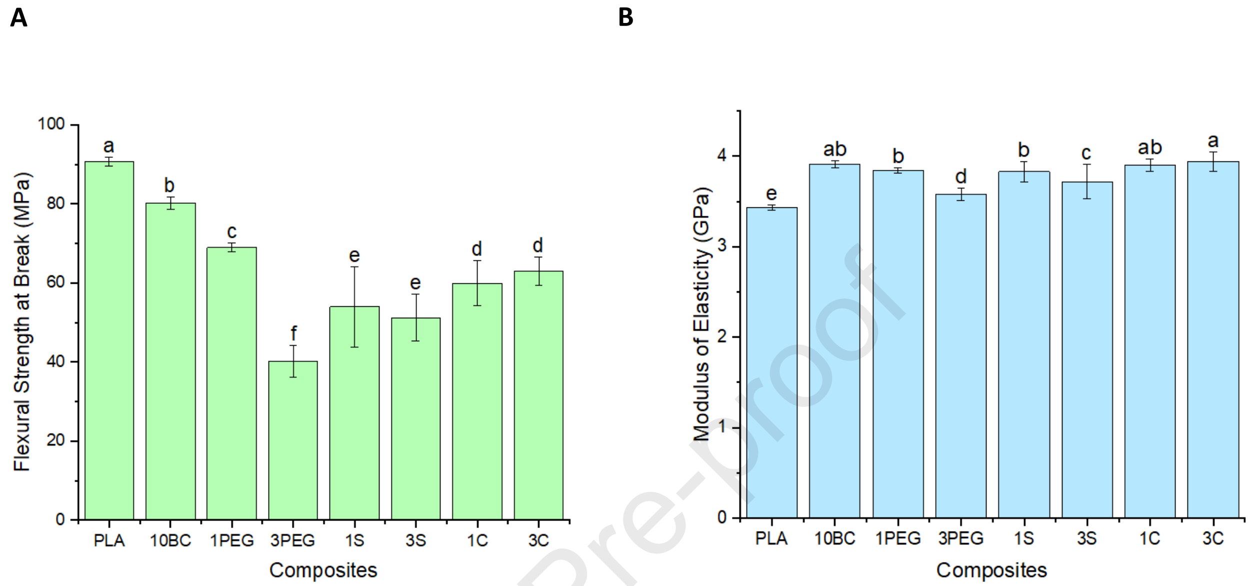


Figure 4. Flexural testing results of all composites, including strength at break (A) and modulus of elasticity (B).

Out of all additives, 1PEG was able to maintain the third highest value of strength (69.0 MPa). Likewise, Li et al. (2018) saw decreases to 55-60 MPa for flexural strength (~82 MPa neat PLA) and to 2.3-2.7 GPa for flexural modulus (3.2 GPa neat PLA) for all tested PEG composites. The authors indicate that this is a sign of the PEG plasticizing effects [21]. Moreover, PLA/lignin/STRUKTOL® composites (~58 MPa) were found to have a decreased flexural strength to that of the pristine PLA (~82 MPa), consistent with this work's results for PLA/biochar/STRUKTOL® (51-54 MPa) [40, 41]. For cork particles, Mazur et al. (2022) determined that a decrease in flexural strength and modulus occurred when 30 wt.% of cork particles was combined with a PLA matrix (~45 MPa and ~1.5 GPa, respectively), compared with the pure PLA (~105 MPa and ~3.2 GPa, respectively). The authors attribute this to fiber orientation rather than polymer adhesion [17]. In this work, all flexural modulus values were found to increase significantly with respect to pristine PLA, possibly due to the biochar filler. This is well demonstrated in a study by Ho et al. (2015) where flexural modulus increased with increasing biochar content [45]. The second lowest modulus values were found for 3PEG. This may be an indicator of the plasticizing effects to increase chain movement, as both PEG and STRUKTOL® experience a decrease from 1 wt.% to 3 wt.%. Further restrictions on chain movement are emphasized by the modulus for 3C, where values were highest, possibly due to bulky cork particles restricting chain movement. Biochar is the most likely explanation of the increase in modulus, especially when studies without it show decreases when additives are included [17, 21, 45].

3.3 Morphology

Figure 5 shows the SEM images taken of all samples at 1000x magnification, highlighting the surface consistencies. These images were taken along the cross-sectional breakage of the tensile samples. Notably, PLA shows the smoothest surface of all composites and is attributed the to its brittle nature [46]. Unlike neat PLA, all samples containing biochar experienced a rough surface at the point of the tensile sample breakage. This is attributed to biochar in the composite, where aggregation occurs and causes breakage. The material experiences a jagged fracture, possibly due to the inconsistent stress concentration sites as a result of BC aggregation [45]. Mohapatra et al. (2014) were able to take SEM images of their PLA/PEG composites, with the texture being similar to this work's PLA/BC/PEG composites. However, the texture is slightly more consistent than Figures 5A and 5B, with the biochar additive contributing to our composite's inconsistencies. The authors were able to determine good PEG dispersion throughout the PLA matrix [42]. Similarly, Li et al. (2018) explored PEG-plasticized PLA composites with wood flour, and found a significantly rough surface, possibly due to the fibrous structure [21].

In general, consistent BC dispersion has been attributed (at low concentrations) to improved mechanical properties. These improved properties are thought to be caused by the biochar porosity, as melted polymer deposits within the pores, creating an interlocking effect [11].

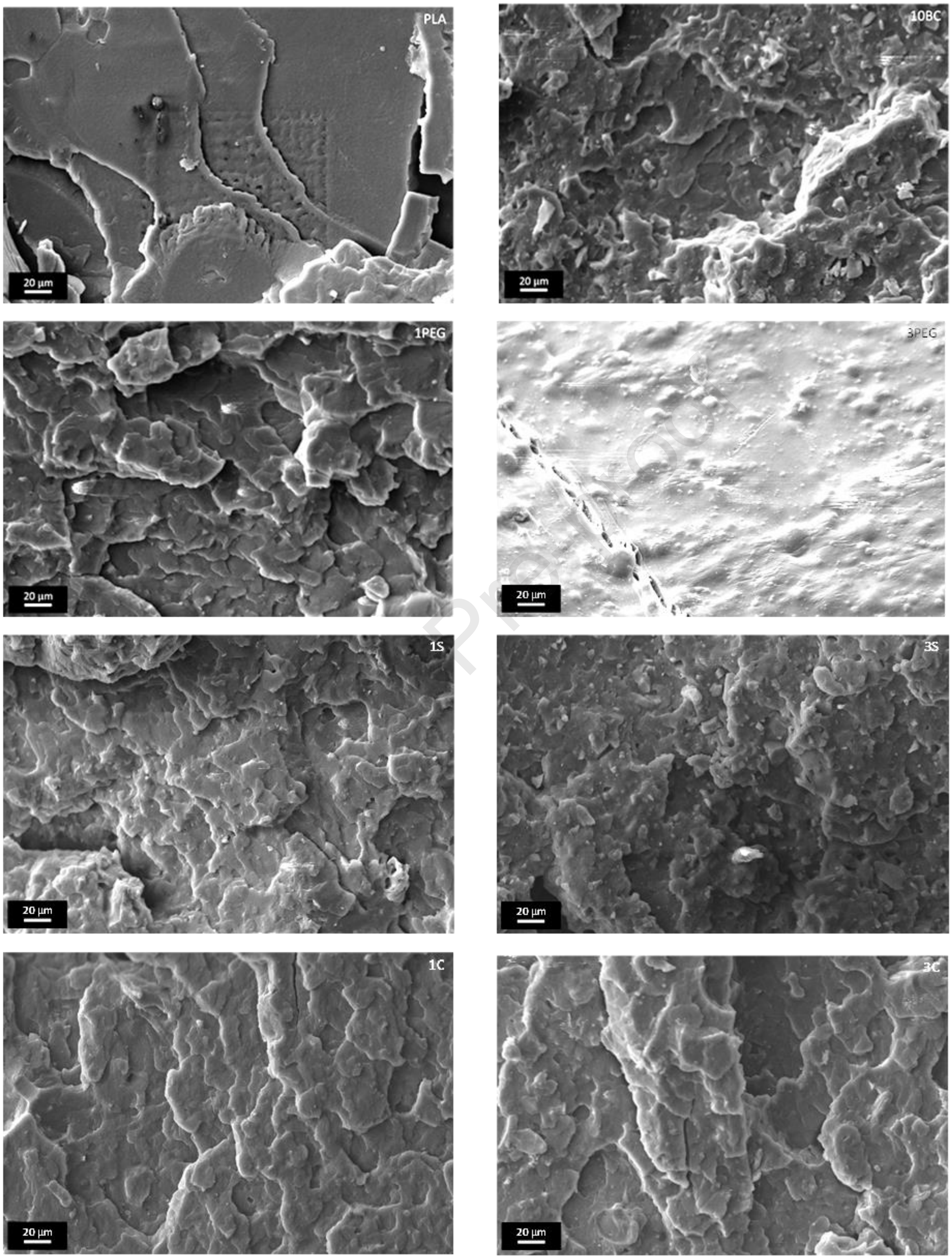


Figure 5. SEM images (1000x magnification) taken of pristine PLA and biochar composites.

A study by Hernandez-Charpak et al. (2022) found that biochar effects within polymer matrices were attributed to the morphology rather than chemical reactions [35]. This emphasizes the importance of analyzing the biochar morphology and dispersion in the matrix. Upon observation of the biochar composites, it appears that the biochar aggregation is visually more prominent in the 10BC, 1PEG, 3PEG, 1S, and 3S composites, while both 1C and 3C experience more consistently smooth surfaces. This may be a similar effect to what the biochar-polymer interaction experiences, in which the melted polymer deposits within the porous cork. Though cork morphology was unable to be determined, it can be estimated according to other literature. Cork being a porous material, it may have the potential for similar interactions that the porous biochar experiences, based on images taken by Anjos et al. (2014) [47]. However, it has been discussed that injection molding has damaging effects on the cork particle structure [36]. This could potentially change the interaction of the cork particles in the polymer composites by destroying the porous network within the particles themselves.

3.4 Thermal Properties

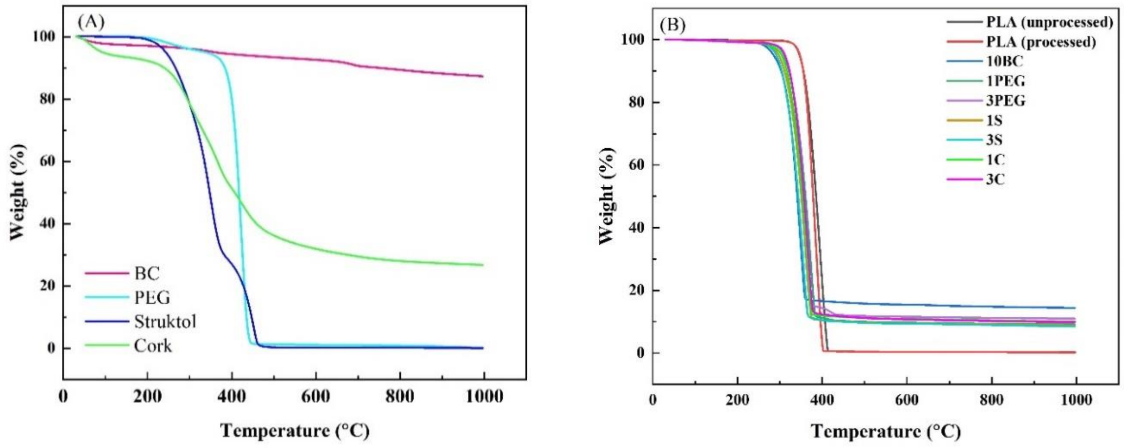


Figure 6. Thermal gravimetric analysis of the composite feedstocks (A) and composites themselves (B).

Figures 6A and 6B show the results of TGA data taken of all feedstocks and composites, respectively. It should be noted PLA unprocessed refers to the material before thermal compounding while PLA processed refers to the material after compounding. For the feedstock degradation, only cork experiences an initial decrease in weight, which may be a result of moisture loss. Daver et al. (2018) determined that pristine cork had a 5% weight loss ~251°C [48], corresponding closely with the onset of significant degradation seen in this study. Biochar, PEG, and STRUKTOL® are able to maintain better stability until after 200°C. However, both plasticizers degrade before biochar and cork. PEG feedstock was found to vary across literature. In the instance of PEG with a molecular weight of 400 g mol^{-1}, the major decomposition began at ~150°C, while a weight of 2000 g mol^{-1} showed major decomposition beginning beyond ~350°C [20, 21]. It has been proposed that the molecular weight of PEG can affect what temperature the PLA/PEG composite begins degradation [21]. The PEG for this study had a high molecular weight of 6000 g mol^{-1}, which may explain why it maintains a high thermal stability until after 300°C. Data for neat STRUKTOL® TR451 was unavailable for comparison.

Figure 6B compares each of the composites and neat PLA materials up to 1000°C. It is important to note that there was little difference in the unprocessed and processed PLA trends seen in the TGA data. This can indicate little changes in thermal stability due to the thermal compounding process. In comparison to literature, PLA has been found to have a significant weight loss starting around 300-350°C [12, 13, 36, 48]. For this work, PLA begins significant degradation between 350-375°C, slightly higher than reported literature. The earliest degradation onset began for 10BC but was very close to the weight loss trends seen in the additive-based composites. For PLA/BC, the addition of 10 wt.% BC showed degradation onset temperatures that varied depending on the study [13, 33, 37]. This work determined that the biochar was able to decrease the onset degradation temperature for all BC composites.

Similar to the trends seen for pristine PEG, the molecular weight of this plasticizer influences degradation temperature. In comparison, Li et al. (2018) found that PLA with 10 wt.% PEG of 4000 g mol^{-1} began degradation ~300°C, where pristine PLA began degradation just after 300°C [21]. Mohapatra et al. (2014) were able to incorporate 20 wt.% of 6000 g mol^{-1} PEG into a PLA matrix, with TGA data showing an onset degradation just before 300°C [42]. Both studies support this work in which PLA/BC/PEG composites began degradation ~300°C. For the STRUKTOL®, our lab previously found that PEG/lignin/STRUKTOL® composites had an onset of significant degradation ~350°C. However, low concentrations of the STRUKTOL® plasticizer cause little change between the PLA/lignin composites with and without plasticizer [19]. This trend is seen in this work as well, with the plasticizers and cork particles having little impact on degradation temperature changes due to their low concentrations (1 wt.% and 3 wt.%). They remain consistent with the PLA/BC composite. Daver et al. (2018) found that degradation for the 5 wt.% cork/PLA composite began at ~339°C [48]. Andrzejewski et al. (2019) examined the TGA data of 10 wt.% cork in a PLA matrix, resulting in an onset degradation temperature slightly less than 350°C. It is further stated that cork particles have a high thermal stability, attributed to lower hemicellulose. Meanwhile, the suberin content in the cork has a higher degradation temperature of 360°C [36].

Overall, all materials experienced similar degradation patterns and all significant degradation patterns began after the processing range (170-175°C for thermal compounding and 185-190°C for injection molding). This means that the material was unlikely to have any significant degradation occurring during processing.

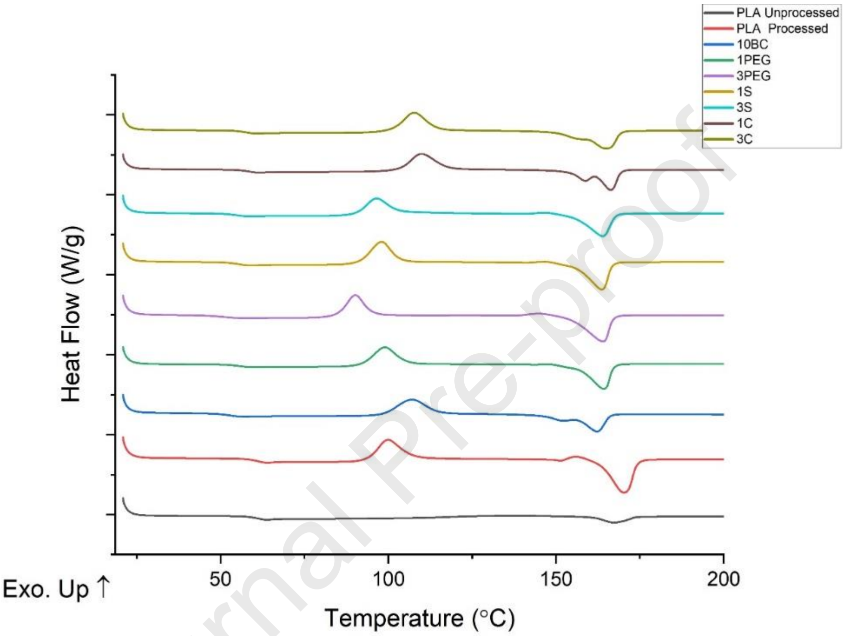


Figure 7. Differential scanning calorimetry for all specimens, including unprocessed PLA.

Table 4. Resulting temperatures as determined by DSC.

		PLA unprocessed	**PLA processed**	**10BC**	**1PEG**	**3PEG**	**1S**	**3S**	**1C**	**3C**
Temperature (°C)	T_g	61.4^{a}	61.3^{a}	53.1^{d}	56.0^{c}	50.6^{e}	55.9^{c}	54.2^{d}	58.9^{b}	59.2^{b}
	T_{cc}	-	98.9^{cd}	107.0^{b}	99.4^{c}	90.5^{e}	99.3^{c}	96.9^{d}	110.0^{a}	108.7^{ab}
	T_m	167.9^{a}	169.4^{b}	162.1^{e}	163.8^{d}	163.1^{de}	164.1^{d}	162.7^{de}	165.9^{c}	166.4^{c}

*Note: each Least Significant Difference test was conducted with each row independent from the next.

The results of DSC are shown in Figure 7, with the values calculated in Table 4 for clarity and statistical analysis. As it can be seen, all three temperatures (glass transition, cold crystallization, and melting) experienced some shift throughout the composite-making process. For glass transition (T_g), biochar itself significantly lowered this temperature, though pristine PLA is consistent with previous works [12, 13, 20, 21, 27, 42]. Huang et al. (2023) attributed the lower glass transition temperature to polymer degradation in composites with biochar incorporated [13]. Both plasticizers contributed to the low glass transition temperatures, while the cork particles were able to increase the value from that of the 10BC sample. Furthermore, Mazur et al. (2022) found a decrease of 1.1-1.8°C between the PLA and the PLA/cork composites, consistent with the findings of this study even with the biochar filler [17].

This study found variations increasing by 9.8-11.1°C for T_{cc} and decreasing by 3.0-3.5°C for T_m when the PLA/BC/cork composites were compared to the neat PLA. Daver et al. (2018) found that T_{cc} (116°C) and T_m (166°C) were maintained in PLA/cork (5 wt.%) composites in contrast to this study's results [48]. These findings suggest, it is possible that the biochar is influencing these phase transitions more so than the cork particles. The T_{cc} experienced a similar trend in which the two plasticizers significantly lowered the temperature from that of the 10BC sample. This has been attributed to plasticizers' improvement of molecular chain movement for both T_g and T_{cc} [21]. Overall, a lower cold crystallization temperature indicates crystal nucleation improvement [13]. It appears that the plasticizers improved nucleation, while cork particles limited nucleation.

For the melt temperature, all composites were lower than that of the neat PLA samples, though 10BC and 3S were the lowest overall. In cases of PLA/BC composites, literature has found that melting temperatures typically decrease slightly as biochar concentrations increase [12, 13, 27]. This is cited as being possibly due to the polymer matrix decomposition upon the introduction of biochar [13]. The composite matrix may have slightly degraded to due to the filler and additives, though TGA results (Figure 6) showed no significant degradation throughout the processing temperatures.

4. Conclusions

Within this work, pristine PLA and biochar composites were produced and tested. The biochar composites included 1 wt.% and 3 wt.% additives of either PEG, STRUKTOL®, or cork particles. A high concentration of carbon (81.12%) and fixed carbon (74.83%) was calculated in the biochar, indicating significant potential for carbon sequestration. Additionally, nitrogen and sulfur were detected, both of which hold potential for improving soil and increasing plant growth. Furthermore, testing revealed that 1C and 3C showed promise in their tensile strength as they were consistent with the 10BC composite despite including an additive (ranging between 51.5-54.6 MPa). Impact strength was found to be consistent across almost all composites. Of the SEM images taken, the biochar showed aggregation in the BC, PEG, and STRUKTOL®-based composites. The cork-based composites had a slightly smoother surface than the other additive-based composites. The composite feedstocks were found to degrade at higher temperatures, indicating no significant degradation during processing. However, the decrease in glass transition and melting temperatures indicated slight degradation had occurred due to filler and additive additions. Cold crystallization temperature data indicated that the plasticizers improved nucleation effects, while the cork reduced nucleation. Through this analysis, it is hoped that the biochar composites hold potential for biodegradable plant containers.

CRediT authorship contribution statement

Rachel Day: Conceptualization, Investigation, Methodology, Writing-original draft, Writing-review & editing. **Sushil Adhikari:** Conceptualization, Funding Acquisition, Resources, Supervision, Writing-review & editing. **Yucheng Peng:** Resources, Writing-review & editing

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Properties of polylactic acid and biochar-based composites for environment-friendly plant containers

- High biochar carbon content for better carbon sequestration
- Cork composites produced comparable tensile strength to 10 wt.% biochar composites
- Smoother tensile breakage for additive-based composites compared to biochar-based
- No significant degradation seen within processing temperatures for all composites
- Biochar and cork additives discouraged crystal nucleation

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The author is an Editorial Board Member/Editor-in-Chief/Associate Editor/Guest Editor for *[Journal name]* and was not involved in the editorial review or the decision to publish this article.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: