

SUSTAINABLE RECYCLED POLYCARBONATE (PC) BASED BLEND AND THEIR BIOCOMPOSITES FOR AUTOMOTIVE APPLICATIONS

Neelima Tripathi^{1,2,§}, Amar K. Mohanty^{1,2,}, Arturo U. Rodriguez^{1,2}, Alper Kiziltas³, Deborah F. Mielewski³, and Manjusri Misra^{1,2}*

¹*Bioproducts Discovery and Development Centre, Department of Plant Agriculture, Crop Science Building, University of Guelph, Guelph, N1G 2W1 Ontario, Canada*

²*School of Engineering, University of Guelph, Guelph, N1G 2W1 Ontario, Canada*

³*Ford Research and Innovation Center, Ford Motor Company, Dearborn, Michigan 48124, U.S.A.*

[§]Presenting author: ntripath@uoguelph.ca

^{*}Corresponding author: mohanty@uoguelph.ca (A. Mohanty)

Abstract

This work focuses on developing thermoplastic blends and bio-composites by utilizing a combination of bio-sourced, recycled and waste resources. The sustainable blends and bio-composites, prepared by an upcycling approach, will be beneficial for reducing the cost and CO₂ emissions of the automotive parts while improving vehicle performance and preserving limited resources. This work focuses on utilizing recycled PC (rPC) as a matrix for one such material. The bio-based content of the matrix was enhanced by incorporating renewable poly(lactic acid) PLA. A small amount of hydrolytic stabilizer (BioAdimide) in PC:PLA blend has proved to make the products durable and weather resistant for automotive applications. The addition of PLA resulted in improved melt flow properties by 34% in the rPC/PLA compatibilized blends. The mechanical strength of the compatibilized blends of rPC and PLA were tested. In addition, lignin (waste from paper and biofuel industries) was pyrolyzed at 500 °C to prepare a bio-carbon (having functionality) to include as a reinforcing agent. From these materials, lightweight, sustainable bio-composites were developed using biocarbon, recycled carbon fibers (rCF) and recycled PC/PLA blends. The final, lightweight bio-composites prepared by injection molding showed enhanced properties. This novel work developed biocomposites with more than 90% sustainable content (that includes recycled, bio-source and waste resources) supporting a groundbreaking circularity.

Background

It is estimated that by 2050 the world population will speedily escalate from 7 billion to 9 billion. The rise in population will lead to an increase in plastic consumption to 185.7%. The shift from a linear economy (throwaway culture) to a circular economy (reuse of waste plastic efficiently) is the call of the hour.(1)

The linear economy (i.e., products that are produced, used, and discarded) depends extensively on non-renewable petroleum. More options for developing sustainable products are

yet to be explored so that the currently present waste plastic can be reused to reduce or eliminate the waste from the environment. Furthermore, a bio-based economy is the greener alternative (derived from renewable biological sources) to fossil-derived products. The bio-based economy utilizes the waste from the agriculture and food processing industries.

Polycarbonate (PC) and its blends are commonly used petroleum-based plastics used for automotive parts in applications such as lighting, dashboards, panoramic roofs, door handles and instrument panels. PC possesses high impact strength and thermal stability compared to industrial compostable polymers such as polylactic acid (PLA). Bisphenol A moiety in PC (consisting of aromatic rings) imparts stiffness.(2) PC has gained popularity due to its clarity and remarkable impact strength. The black color of the rPC limits its use where transparency is desired. However, the rPC significantly contributes toward developing a sustainable environment with enhanced mechanical properties and can be used in under-the-hood applications.(3)

It has been stated that durable polymers should be preferable as it is superior to biodegradable or industrial compostable polymers based on the following reasons:

- Biodegradable polymers tend to be dumped into the environment more conveniently and hence foster dumping waste.
- Dependence on climatic conditions for biodegradation, i.e., survival of biodegradable plastics for decades in the absence of desirable conditions in the environment.
- Landfill of biodegradable plastics will result in the evolution of methane (greenhouse gas) during anaerobic biodegradation.
- Biodegradation leads to the production of micron size bioplastics that is transported by wind and water. The micro-bioplastic result in breeding grounds for bacteria and micro-organisms, and hence result in polluting the environment.(1)

Thus, the utilization of industrial compostable and biobased polymer such as polylactic acid (PLA) for preparing sustainable automotive parts can be explored. The utilization of PLA in car parts will contribute toward energy security and a low carbon footprint.(2) PLA has a great potential for developing durable automotive products. Mohanty et al.(4) developed durable PC:PLA blends and compositions. The work utilizes hydrolytic stabilizers (consisting of carbodiimide groups such as BioAdimide) to improve the durability and weather resistance of PLA in PC:PLA blends. Furthermore, it has been observed that PLA will enhance the melt flow index that will increase the scope of adding more filler into the composites.

The researchers are exploring various fillers to make the automotive parts sustainable, lightweight and low-cost without compromising mechanical properties. It has been estimated that 1 kg of weight reduction can result in reduction in 20 kg of vehicle carbon emission.(5) By 2025, standards demand an average fuel economy of 54.5 mpg, which can be achieved by reducing the weight of automotive components. Carbon fibers (CF) have a great potential in developing

automotive advanced strong and stiff composites because of their lightweight. However, its high cost (€ 30-40/kg) limits its use in many applications. This work focuses on exploring recycled CF (rCF) and lignin biocarbon as fillers. It has been estimated that rCF has the potential to reduce 40% of the cost of fiber (€ 10-20/kg). In addition, the global warming potential of rCF is <10% with respect to virgin CF. The steps involved in recycling CF are: (a) metal removal and cutting of the bigger composite parts to smaller; (b) shredding of laminates; and (c) pre-impregnated composite (prepreg) were sent to the modified pyrolysis process where the matrix is burnt-off. It was determined that the pyrolysis process retains 90% of the fibers' tensile strength and does not affect modulus. The achieved CF after pyrolysis were milled.(6,7)

Another filler that can be used in making composites are biocarbon obtained after pyrolysis (a process of converting biomass to biocarbon by thermochemical conversion under a limited oxygen atmosphere) of biomass such as lignin. Lignin is the second most abundantly available natural polymer/biomass, a by-product of the pulp and paper industry. It is estimated that the production of lignin from the pulp preparation process is ~70 million tons/year.(8) Lignin is a 3-dimensional macromolecule that imparts strength and rigidity to the cell walls of plants. It is composed of guaiacyl, p-hydroxyphenyl and syringyl units.(9) The density of biocarbon ($1.34-1.96 \text{ g cm}^{-3}$)(5) compared to conventional fillers [carbon fiber (1.8 g cm^{-3}), glass fibers (2.5 g cm^{-3})(10) and mineral (2.8 g cm^{-3})] is significantly lower.(5)

The compatibilization between matrix-matrix and filler-matrix is also a challenging task that needs to be addressed to achieve the desirable properties for automotive applications. The blends and composites require good interfacial adhesion. Good interfacial adhesion can be achieved by compatibilization. Mohanty et al.(11) described the following strategies (Table 1) to enhance compatibilization between polymer-polymer or polymer-matrix.

The present work focuses on developing a sustainable product having recycled polycarbonate (rPC) (a post-industrial recycled material).(3) The potential of rPC for automotive applications has been explored. The rPC, rPC:PLA blend, rPC:PLA:Lignin biocarbon composite and recycled carbon fibers (rCF)-based (rPC:PLA:rCF) composites were injection molded. The injection-molded samples were analyzed by surface morphology, rheology, thermal and mechanical tests. The schematic representation of the aim, objectives and process of this work is shown in Figure 1.

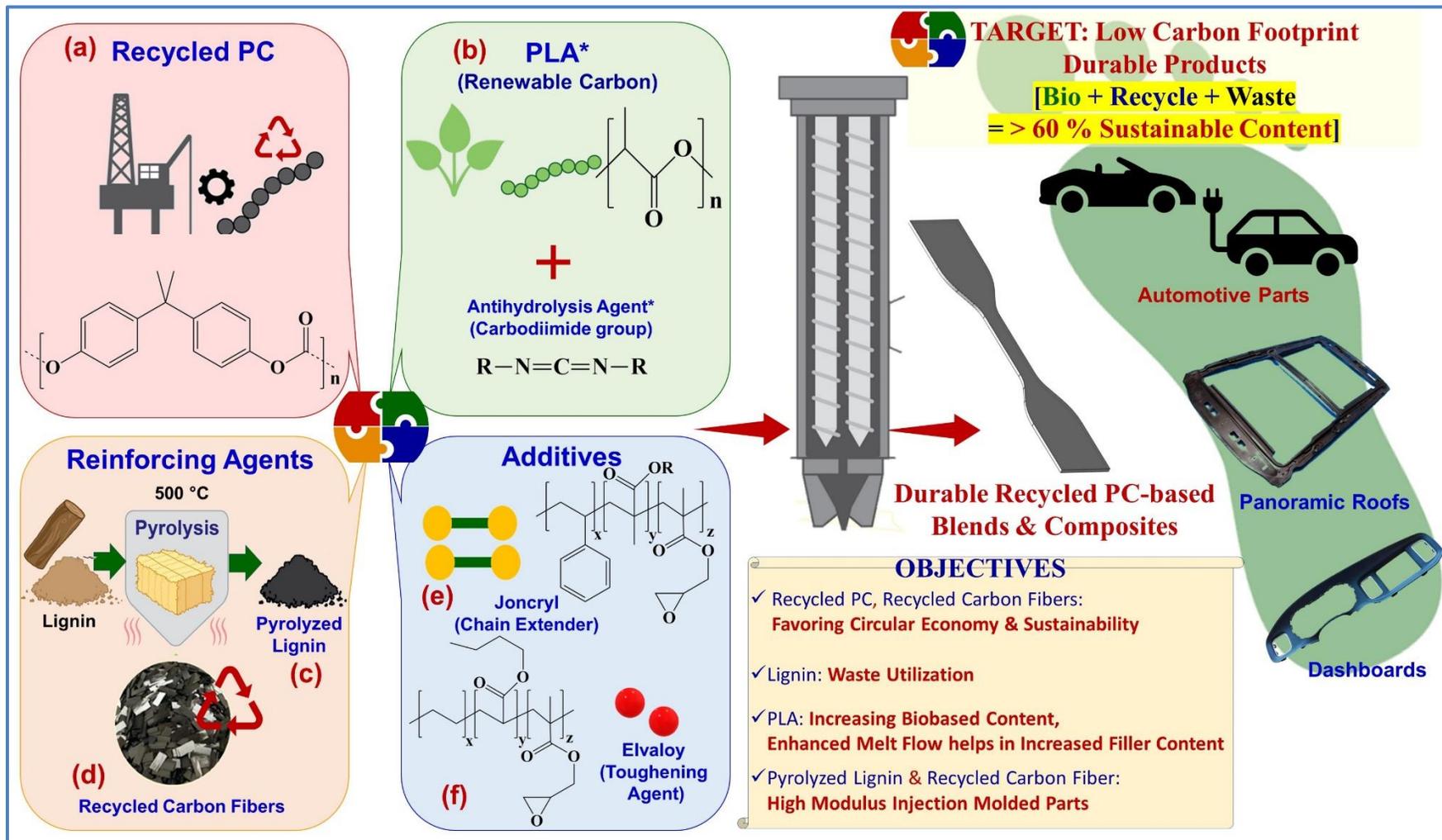


Figure 1. Schematic representation of the aim, objectives, and process to develop sustainable recycled polycarbonate (rPC) based blends & their biocomposites.
 (* small amount of hydrolytic stabilizer (BioAdimide(4)) added with PLA has proved to enhance the durability of the automotive parts)

Table 1. Compatibilization strategies for making blends and composites with enhanced properties.(11)

Compatibilization Strategies		
Matrix & Matrix	Physical Compatibilization	Adhesion mechanism by trapped entanglements
	Physical & Chemical Compatibilization	Branching & Crosslinking
Matrix & Fiber	Functionalized Matrix	
	Functionalized Fibers	
Hybrid synergistic assembly in composites	1. Compatibilization between polar polymer and apolar polymer can be enhanced using a multifunctional (having apolar & polar parts) compatibilizer.	a. The apolar-apolar components between matrix-matrix or matrix-filler will result in physical interaction.
	2. The polar (functionalized) filler, such as functionalized fibers, and apolar (non-functionalized) filler, such as biocarbon (pyrolyzed at > 800 °C), can be incorporated into the hybrid polar-apolar matrix to check the compatibilization effect.	b. The polar-polar components between matrix-matrix or matrix-filler will result in chemical interaction.

Materials

The rPC (OPTICARB 1614) was purchased from The Materials Group, North America. PLA (Ingeo 3251 D; Mw: 55 000; polydispersity index: 1.62) was procured from Nature Works LLC (USA). Recycled carbon fiber (rCF) (milled) [CARBISO MF80] was purchased from ELG Carbon Fibre Ltd (UK). Poly(ethylene n-butylene acrylate glycidyl methacrylate) (EBA-GMA) or Elvaloy PTW (trade name) was purchased from DuPont. Elvaloy PTW is a terpolymer consisting of ethylene (66.75 wt%), butyl acrylate (28 wt%), and glycidyl methacrylate (5.25 wt%). The glass transition temperature (T_g) and melting point were 55 and 72 °C, respectively. According to ASTM D1238, MFI was 12g (10 min)⁻¹.(12) Joncryl ADR 4368 (a multi-functional reactive chain extender, T_g: 54 °C, epoxy equivalent weight: 285 g mol⁻¹) was acquired from BASF. It was preferred to diminish thermal degradation between polymers. Lignin, a by-product from the paper industry (Protobind 2400 was purchased from ALM Private Limited, India), was used to prepare lignin biocarbon.

Methods

Pyrolysis of Lignin

Lignin (Protobind 2400) was pyrolyzed in a GLO pyrolyzer in an inert atmosphere at 500 °C. The ramp rate to reach the set temperature was set at 7.5 °C min⁻¹. The 500 °C was selected so the functional groups present in lignin can be retained. The moisture content of lignin before pyrolysis was ~3 %. The percent yield of lignin biocarbon after pyrolysis was 38.6%. The ash

content of biocarbon was 3.17%. After the pyrolysis, the lignin biocarbon was ball milled for 1 h at 200 rpm. The ball milling duration was selected only for 1 h to limit the energy consumption and processing cost of the lignin biocarbon. The lignin was stored at 105 °C and was used for making composites.

Blend and Composites Preparation

Dried rPC and PLA (moisture content <1%) were taken for preparing blends and composites. Injection molding machine co-rotating twin-screw extruder (microcompounder 15 cm³, DSM Xplore 15, Netherlands). The screw speed was set at 100 rpm. The processing temperature for neat rPC and rPC:PLA (68:32 wt%) blend was 260 °C. The composites were processed at 270 °C. Subsequent injection molding was conducted at room temperature. The injection pressure and holding time were set at 16 bars and 8 sec. The holding time for rPC was set for 1 min, and blends and composites were set for 2 min holding time. The melt flow index (MFI) of rPC, rPC:PLA:Lignin biocarbon composite and rPC:PLA:rCF composite were 21 ± 0.6, 26.2 ± 2.55 and 33.6 ± 0.28 g (10 min)⁻¹. The processed samples formulation ratio is provided in Table 2.

Table 2: Sample designation and the ratio of components taken for making blends and composites formulations.

Sample Designation						
Sample Code	Recycled Polycarbonate (rPC) (wt%)	Poly-lactic acid (PLA) (wt%)	Elvaloy PTW (E) (wt%)	Joncryl (J) (phr)	Recycled Carbon Fiber (rCF) (wt%)	Pyrolyzed (500 °C) Lignin Biocarbon (wt%)
rPC	100	-	-	-	-	-
rPC:PLA blend	68	32	-	-	-	-
rPC:PLA:Lignin biocarbon composite	62	32	6	0.3	-	7
rPC:PLA:rCF composite	62	32	6	0.3	7	-

Measurements

FTIR spectrometer (Nicolet 6700, Thermo Scientific, USA) was used to determine functional groups in the lignin and lignin biocarbon. The analysis was conducted for 64 scans at 4cm^{-1} resolution.

Heat distortion/deflection temperature (HDT) of the rPC, rPC:PLA blend, rPC:PLA:Lignin biocarbon composite and rCF composite were tested using Q800, TA instruments. ASTM D 648 was followed for the 3-point bending measurements. Under 0.455 MPa load, the temperature was increased at a ramp rate of $2\text{ }^{\circ}\text{C min}^{-1}$.

Density of the samples was measured following ASTM D 792. Densimeter (MD 300, Japan) was used to determine the density.

The morphological study of rPC, rPC:PLA blend and composites was conducted on the impact fracture surface. The study was conducted using a scanning electron microscope (SEM) (Phenom ProX, Netherlands), having a back scattering electron (BSE) detector. The impact samples were gold coated in argon atmosphere for 6 sec using TED PELLA, Inc. sputter coater.

The rheological analysis was conducted with plate-plate configuration. A gap distance of 1 mm was set. Measurements were performed under a nitrogen atmosphere at $260\text{ }^{\circ}\text{C}$. Angular frequency was set in the range of $100 - 0.1\text{ rad s}^{-1}$ and strain amplitude to 1%.

Tensile and flexural analyses were measured using the Universal Testing Machine (Instron). The tensile test was performed at a 5 mm min^{-1} rate based on ASTM D 638. The flexural tests were carried out at 14 mm min^{-1} crosshead speed based on ASTM D790. The impact strength analysis of the samples was performed based on ASTM D256. The samples were notched 48 h before measuring Izod impact strength by notching cutter. Six samples for each mechanical test were conducted, and the average value with standard deviation has been reported.

Results and Discussion

Chemical Structure Determination

Figure 2 depicts the comparison between the functionality of the lignin and lignin biocarbon pyrolyzed at $500\text{ }^{\circ}\text{C}$. The spectra of biocarbon shows that after pyrolysis of lignin at $500\text{ }^{\circ}\text{C}$, the functional groups are retained in the biocarbon. Table 3 shows that the C-H bend of aromatic compounds, C=C (stretch) of aromatic ring is retained. After pyrolysis, the peak at 2918.94 cm^{-1} and band $3700\text{-}3000\text{ cm}^{-1}$ diminishes that represent C-H stretch of $-\text{CH}_2$, $-\text{CH}_3$ and O-H stretch, respectively.

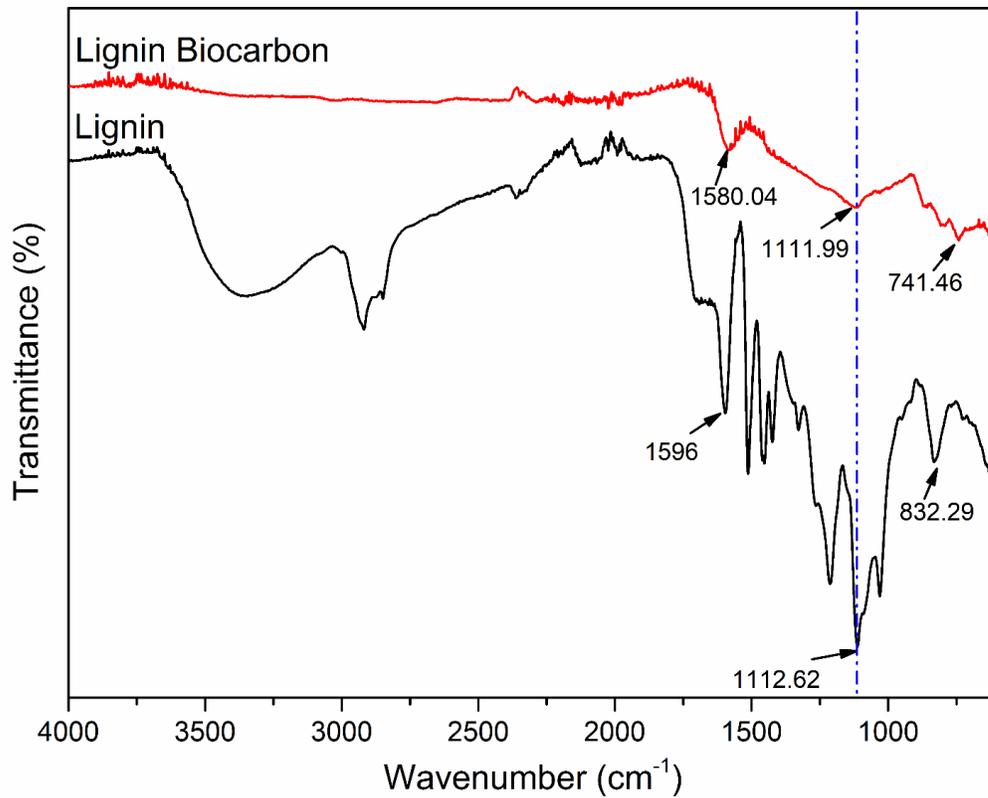


Figure 2: FTIR spectra of lignin (before pyrolysis) and lignin biocarbon (pyrolyzed at 500 °C).

Table 3: FTIR absorbance peak assignment for lignin (before pyrolysis) and lignin biocarbon (pyrolyzed at 500 °C).

Assignment of FTIR Absorbance Peaks		
Wavenumber (cm ⁻¹)	Group	Reference
850-750	C-H bend of aromatic compounds	Asadieraghi et al.(13)
1510-1650	C=C (stretch) of aromatic ring	Asadieraghi et al.(13)
1127	Ether (C-O-C)	Li et al.(14)
3000–2800	C-H stretch of -CH ₂ , -CH ₃	Asadieraghi et al.(13)
3700-3000	O-H stretch of phenolic, alcoholic, carboxylic groups	Asadieraghi et al.(13)

Heat distortion temperature (HDT) and Density Measurements

Similar HDT of the rPC and the composites were observed. However, rPC:PLA blend showed lower heat distortion temperature (Table 4). The increased standard deviation in case of the non-compatible rPC:PLA blend was also observed. The density is also not much affected after incorporation of the fillers. (Table 4)

Table 4: HDT and density measurements of samples.

HDT & Density Measurements		
Sample Code	HDT (°C) (at 0.455 MPa)	Density (g cc ⁻¹)
rPC	136.02 (± 0.39)	1.16 (± 0.003)
rPC:PLA blend	134.35 (± 3.56)	1.19 (± 0.002)
rPC:PLA:Lignin biocarbon composite	135.07 (± 0.47)	1.17 (± 0.002)
rPC:PLA:rCF composite	136.4 (± 0.47)	1.19 (± 0.002)

Surface Morphology

Figure 3 shows the surface morphology of the rPC, rPC:PLA blend, rPC:PLA:Lignin biocarbon composite and rPC:PLA:rCF composite. The analysis was conducted on the fracture surface of the impact bars. Figure 3 (a) shows the smooth, uniform surface of the rPC surface that was impacted during the Izod impact test. rPC:PLA blend clearly exhibits incompatible systems with poor interfacial adhesion. Figure 3 (b) shows the flaky surface showing incompatibility between the rPC and PLA.

Figure 3 (c) shows the surface morphology of rPC:PLA:Lignin biocarbon composite. Enhanced compatibility between rPC and PLA in composites (Figure 3 (c)) were observed. Cohesion between the matrix components (rPC and PLA) was also observed in the composite. Further, in the micrograph of rPC:PLA:Lignin biocarbon composite it is clearly visible that even the bigger lignin biocarbon particles are embedded in the rPC:PLA matrix (encircled). The pyrolysis temperature of lignin (500 °C) was selected to ensure the functionalized lignin biocarbon can affect the interaction between the functionalized lignin biocarbon with the matrix. Since only 1 h ball milling was selected, coarser lignin biocarbon was observed in the matrix. rCF are uniformly distributed in the matrix offering homogeneity in the rPC:PLA composites. In

rPC:PLA:rCF composite, the smaller size rCF are embedded in the matrix (Figure 3 d). At few of the places, pull out of carbon fiber took place due to impact fracture. AFM topographic image of the rCF composites is illustrated in Figure 4.

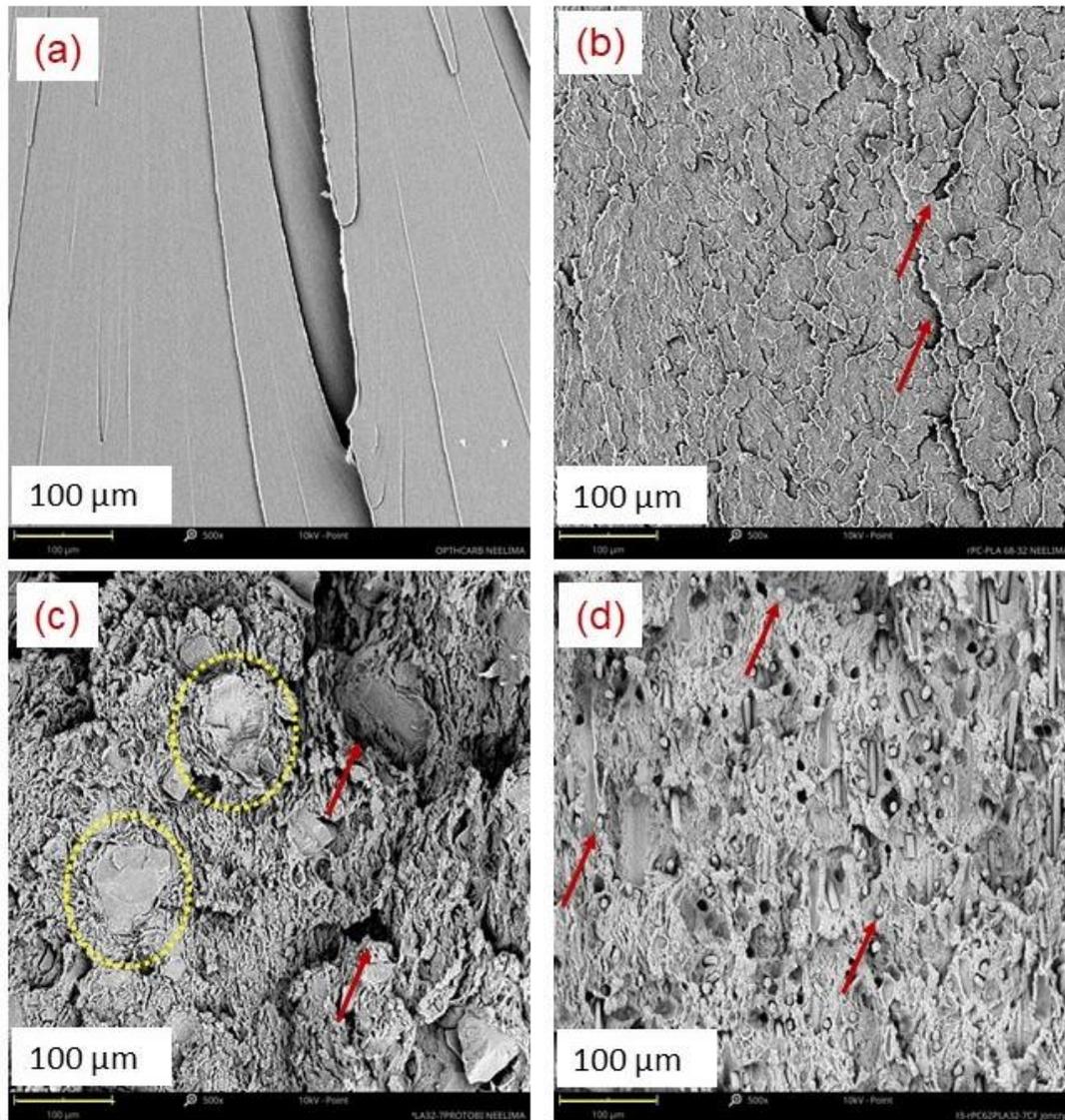


Figure 3: SEM micrographs of a) rPC; (b) rPC:PLA blend; (c) rPC:PLA:Lignin biocarbon composite; (d) rPC:PLA:rCF composite.

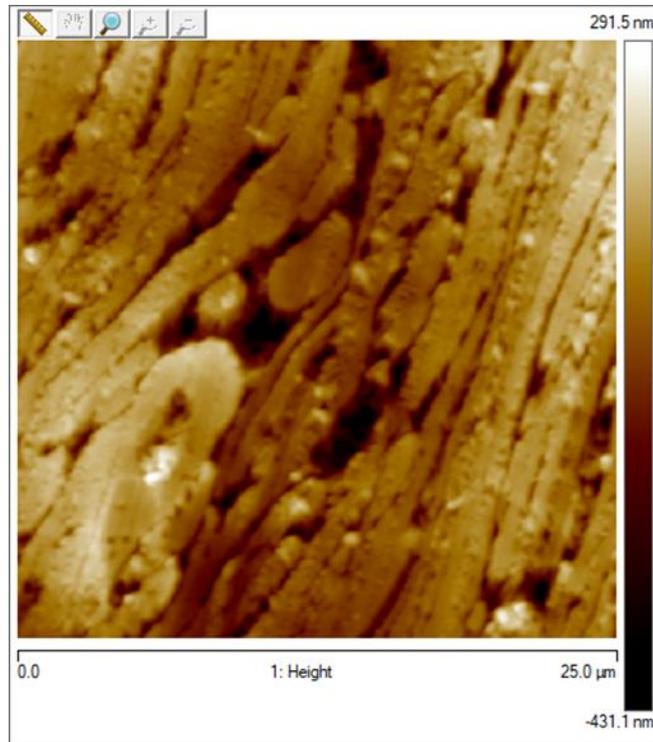


Figure 4. AFM topographic image of rPC:PLA:rCF composite.

Rheological Properties

The melt rheology of rPC, rPC:PLA blend, rPC:PLA:Lignin biocarbon composite and rPC:PLA:rCF composite were tested (Figure 5). The results give an idea of the viscoelastic behavior of the samples.

Typically, storage (G') and loss modulus (G'') of a polymer increase with increasing angular frequency (ω). This increase in modulus with angular frequency (ω) shows that the sample follows linear viscoelasticity. Furthermore, with an increase in ω , complex viscosity (η^*) reduces, suggesting shear thinning (polymer chains disentanglement at the higher shear rate) behavior of the samples. Due to the shear, chain entanglement density reduces.

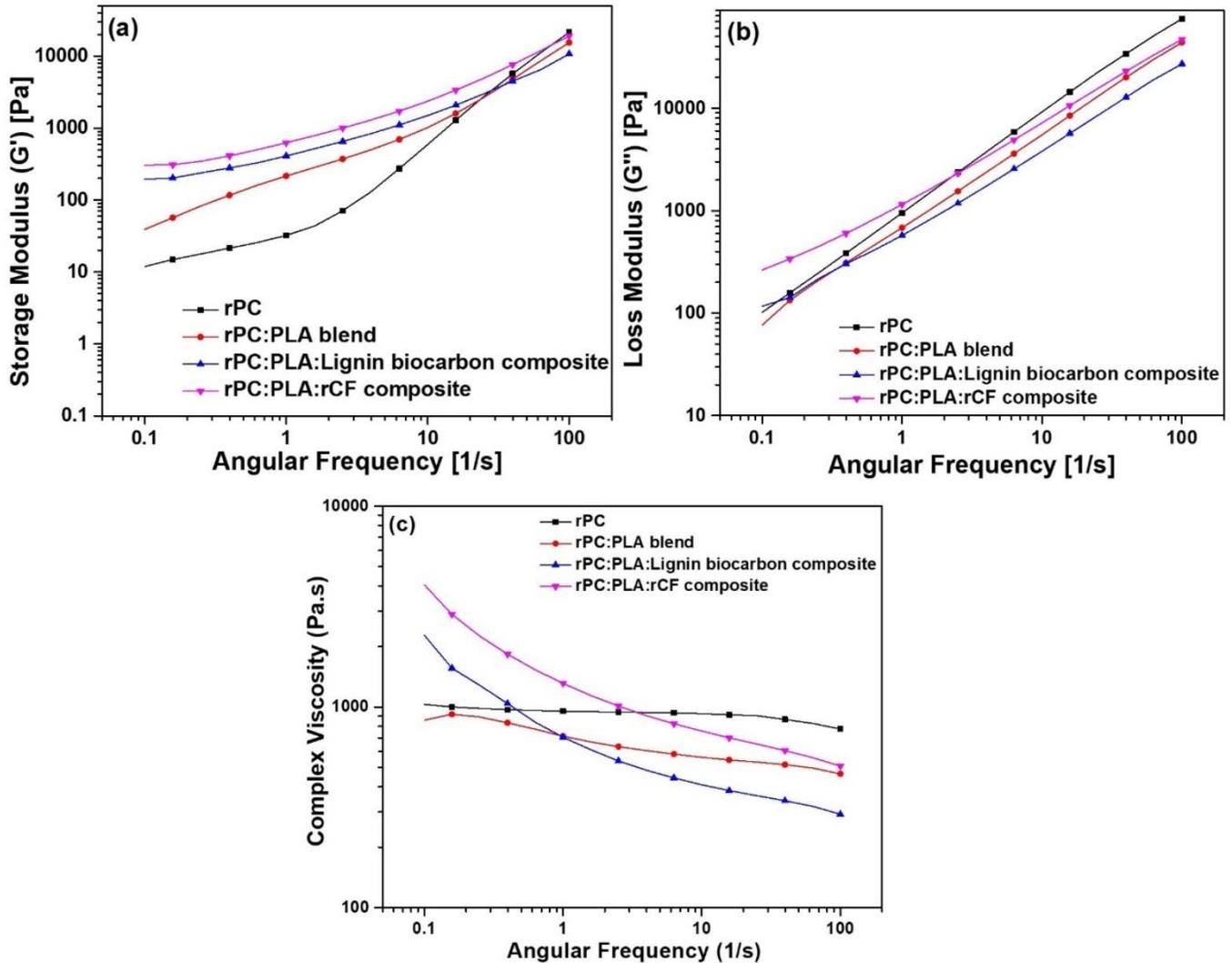


Figure 5. Rheology study of the injection molded samples (a) storage modulus, (b) loss modulus, (c) complex viscosity.

Mechanical Properties

The primary factors that affect the mechanical properties are polymer-polymer interaction, particle size, filler loading, filler and matrix interaction.

Elongation at break of rPC:PLA blend resulted in the 21.5% enhanced elongation with respect to rPC (Figure 6). No significant change was observed in rPC:PLA:rCF composite with respect to the rPC. Lignin biocarbon showed an 81% reduction in elongation at the break due to the coarser lignin biocarbon particles. A significant decrease in elongation at break in rPC:PLA:Lignin

biocarbon composite can be ascribed to the particle size. As the particle size of lignin biocarbon (ball milled for only 1 h) is relatively larger than the rCF. The smaller particle size of lignin biocarbon would have resulted in better mechanical properties for rPC:PLA:Lignin biocarbon composite.

The Izod notched impact test (Figure 7) was conducted for the rPC, rPC:PLA blend, rPC:PLA:Lignin biocarbon and rPC:PLA:rCF composites. The maximum strength was achieved by the rPC. A significant decrease in the impact strength was observed for the non-compatible rPC:PLA blend. The rPC:PLA blend was compatibilized by the addition of Joncryl (chain extender). The impact strength was increased by 67.7 and 105.8 % for compatibilized rPC:PLA:Lignin biocarbon and rPC:PLA:rCF composites, respectively with respect to the incompatible rPC:PLA blend. rPC showed the highest impact strength 695.9 ± 14.7 . Substantial reduction in impact strength was observed for the rPC:PLA blend and composites.

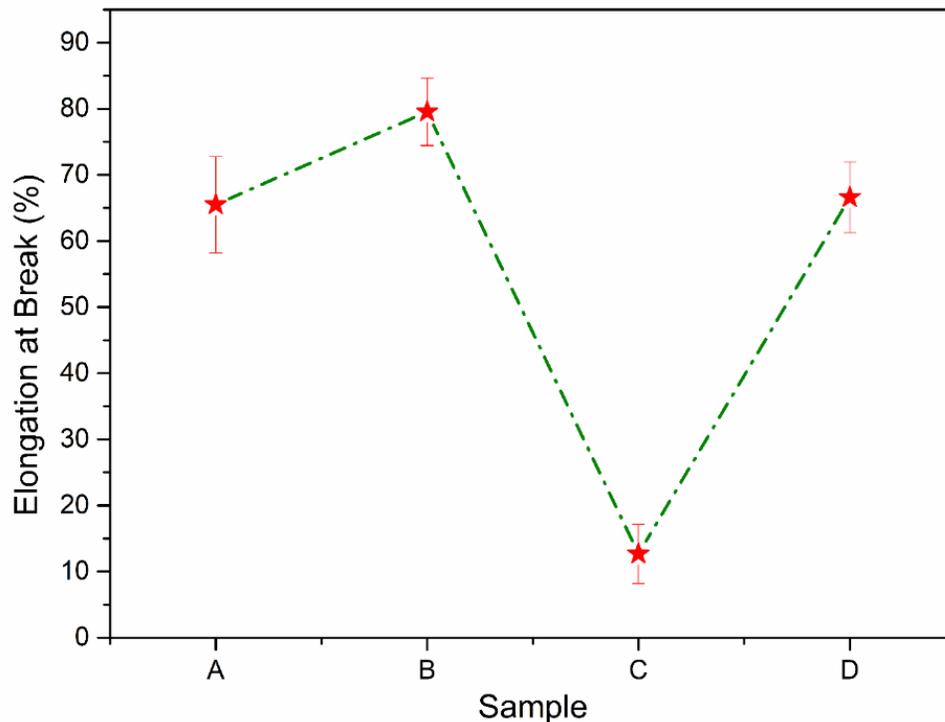


Figure 6. Percent elongation at break of (a) rPC; (b) rPC:PLA blend; (c) rPC:PLA:Lignin biocarbon composite; (d) rPC:PLA:rCF composite.

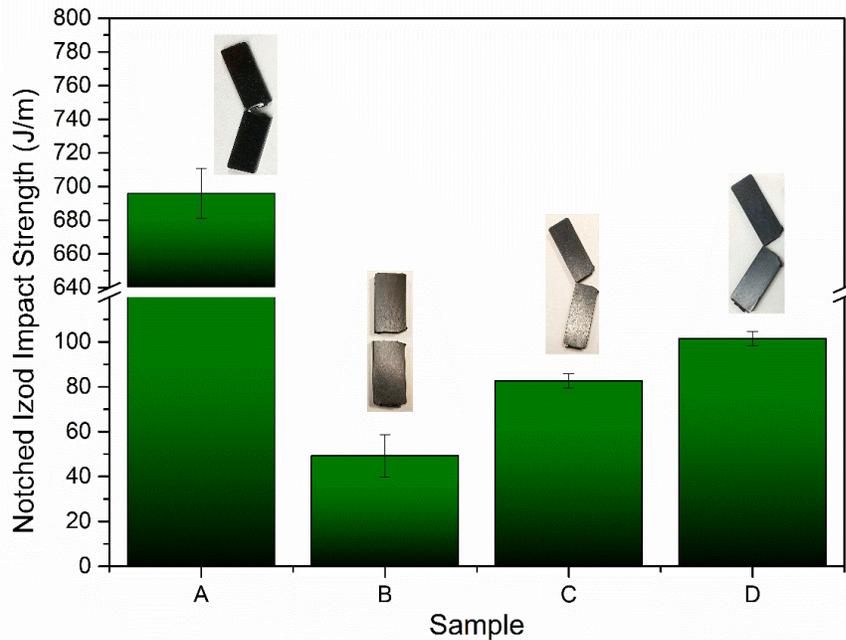


Figure 7: Impact strength measurements of (a) rPC; (b) rPC:PLA blend; (c) rPC:PLA:Lignin biocarbon composite; (d) rPC:PLA:rCF composite.

The flexural strength of rPC and rPC:PLA:Lignin biocarbon composite shows a similar flexural strength (Figure 8). However, the flexural strength of rPC:PLA:rCF composite shows a 3% enhancement with respect to rPC. The tensile strength (Figure 9) for rPC:PLA:Lignin biocarbon composite was reduced to 10.75 % and for rPC:PLA:rCF composite reduced to 7.3%, with respect to the rPC. However, the addition of PLA in rPC resulted in a 4.3% enhancement in the tensile strength. The tensile and flexural modulus of rPC:PLA:rCF increased to 78.88 and 42.81 % with respect to the rPC.

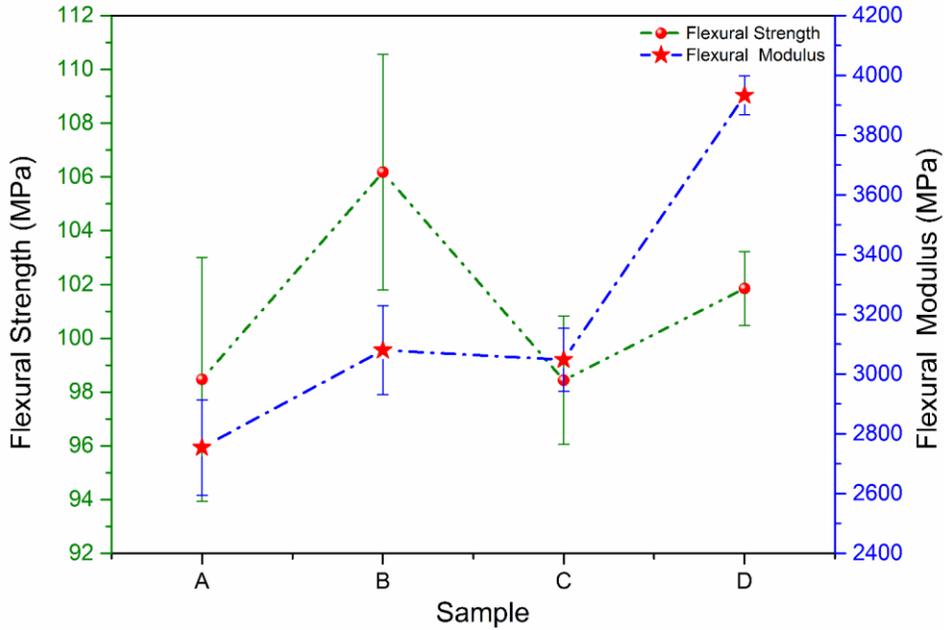


Figure 8: Flexural strength and modulus measurements of (a) rPC; (b) rPC:PLA blend; (c) rPC:PLA:Lignin biocarbon composite; (d) rPC:PLA:rCF composite.

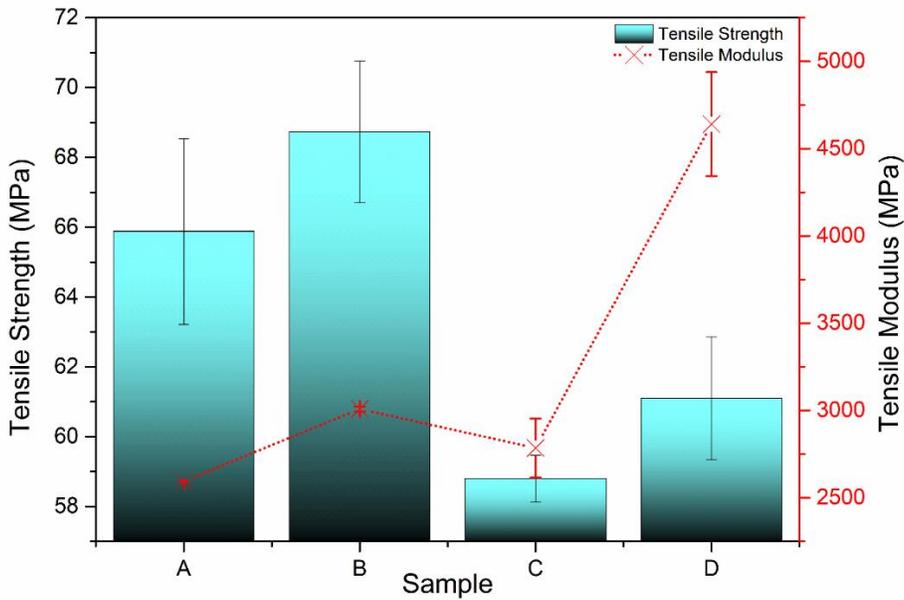


Figure 9: Tensile strength and modulus measurements of (a) rPC; (b) rPC:PLA blend; (c) rPC:PLA:Lignin biocarbon composite; (d) rPC:PLA:rCF composite.

Summary and Next Steps

The sustainable composites were prepared using agro-waste (lignin), recycled (rPC and rCF) and bio-based (PLA) components for automotive applications. The injection-molded samples were prepared for rPC; (b) rPC:PLA blend; (c) lignin biocarbon composites; (d) rPC:PLA:rCF composites. Lignin was pyrolyzed and utilized in the form of biocarbon in fabricating composites. The functional groups present in lignin biocarbon were compared with lignin by FTIR technique. Heat distortion temperature, density, surface morphology, viscoelastic and mechanical analysis were conducted and analyzed.

- The optimization of the processing cost and mechanical properties of lignin biocarbon should be conducted. The uniformly dispersed smaller lignin biocarbon (achieved by longer ball milling duration) may show significantly enhanced mechanical properties of the composites.
- Effect of various carbonization temperatures on lignin in rPC:PLA matrix can be studied.
- Utilization of hydrolytic stabilizers to prepare rPC:PLA automotive products will be studied that will considerably enhance the durability and weather resistance of the automotive parts.
- The functionalization of the recycled commercial fillers with reactive compatibilizers may further enhance the mechanical strength of the composites.
- The incorporation of functionalized (polar) biocarbon and non-functionalized recycled carbon fiber with compatibilizers in the hybrid matrix (consisting of the polar and apolar matrix) can be explored.

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