

Newly Developed Biocarbon to Increase Conductivity in Sustainable Polyamide 12 Composites

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Abstract

Sustainable manufacture caused shift in automotive manufacturing practices. Polymer-based composites make up almost 15% mass of the entire vehicle, most importantly the fuel system of the vehicle. Poor electrical conductivity of the polymer composites leads to electrostatic deposition which can lead to further issues. Carbon based synthetic fillers like carbon fiber and carbon nanotubes are attractive options to develop electrically conductive composites, owing to their excellent electrical and mechanical properties. However, the production process of these reinforcements is highly time and energy intensive making it quite expensive and not quite sustainable. Lignocellulosic feedstock can be carbonized at a high treatment temperature of $\geq 1000^{\circ}\text{C}$ to produce electrically conductive biocarbon filler. In this study biocarbon fibers developed using Douglas fir pulp, were incorporated into polyamide 12 matrix to develop composites. The composites were fabricated using hot compression mounting. At a filler loading of 7.5 wt. % the composites reported resistivity of $7.13 \times 10^3 \Omega \text{ cm}$ and at 35 wt. % filler loading rate the composite resistivity was $2.13 \Omega \text{ cm}$. The transverse volume resistivity value of resistivity of $5.04 \times 10^8 \Omega \text{ cm}$ for polyamide 6 composites filled with 20 wt.% carbon fiber and longitudinal volume resistivity of $10.08 \Omega \text{ cm}$ were reported in literature reviewed. The resistivity values for both 20 wt.% and 40wt.% carbon fibers is significantly higher compared to biochar filled composites at 25 wt.% and 35 wt.% biochar filler loading. This indicates the effectiveness of biochar filler as a conductive filler, in developing electrically conductive, sustainable composites.

1. Introduction:

Automobiles are a major contributor to greenhouse gas emissions. As per Environmental Protection Agency(EPA) on an average 8,887 grams of CO₂ is emitted from 1 gallon of gasoline and 10,180 grams of CO₂ is emitted from 1 gallon of diesel [1]. Annually 4.6 metric tons of CO₂ is released from a typical passenger vehicle [1]. These emissions have a highly detrimental effect on the global climate scenario. Increased fuel efficiency of vehicles can lead to lower fossil fuel consumption, in turn lower greenhouse gas emissions. To achieve this feat, the automobile manufacturers have resorted to lightweighting of the vehicles. As per the EPA 2020 Automotive trends report, the heaviest vehicles produced in model year 2020 generate half the amount of CO₂ compared to what was generated in model year 1978 [2]. The CO₂ generated for lighter vehicles in 2020 is two thirds of what was generated in 1978, all owing to the massive design changes and advancements undertaken by the automakers [2]. Lightweighting can be done by making smaller cars or by material substitution without compromising on the capacity and size[3]. Traditional materials are being replaced by light weight metals and largely by polymer composites. These polymer composites are generally filled with fillers like talc, glass fibers, calcium carbonate (CaCO₃), clay nanoparticles etc., [4], [5]. These polymer composites are lighter than the conventional metals, but the inorganic fillers have a high density making the finished products bulky. Increased use of plastic in lightweighting of vehicles might also led to increase in issues due to electrostatic deposition. Having electrically conductive automobile parts by incorporating electrically conductive polymer composites is a solution to this issue. Electrically conductive polymer composites (ECPCs) are polymer composites developed by incorporation of conductive filler material into a non-conductive matrix [7]–[10]. Electrically conductive composites can be developed by incorporation of various electrically conductive fillers in the matrix. Carbon based fillers like carbon nanotubes, carbon fibers, carbon black etc., have been reported to be used as filler and the composites have shown good conductivity values [11]–[13]. Electrically conductive polymer composites (ECPCs) have a wide range of application in sensors, electromagnetic shielding, capacitors etc. ECPCs are now being used in automobile industry to produce light weight electrically conductive parts to achieve fuel efficiency and cut down emissions. However, synthetic carbon fillers are highly energy and time intensive making procurement expensive and the whole process unsustainable. A cost effective and sustainable solution to develop lightweight electrically conductive composite is utilization of biochar as conductive filler.

Biochar is the carbon rich solid material that is left after the thermochemical conversion of biomass in an oxygen deficit environment [14]. Carbonization is a process in which the progressive conversion of a three dimensional organic macromolecular system like wood, into a macro-atomic network of carbon atoms [15]. In the process of carbonization, the biomass is heated to a certain temperature usually at a slow heating rate in an inert atmosphere using gasses like nitrogen, argon etc. As the thermal decomposition of the biomass takes place slowly the yield of solid carbon in carbonization is higher compared to other processes like pyrolysis. Biochar has been largely implemented in soil amendment and soil quality enhancement. Biochar has been reported to have improved the soil nutrient quality, has a positive effect on the soil microorganisms, owing to its high surface area and nutrient content it does a great job in soil quality improvement [16]–[18]. Biochar has been reported to be a great filler that is used for improvement of mechanical and thermal properties of polymer matrices. Das et al. 2016 [19] have reported improved tensile modulus and improved thermal stability in polypropylene filled with biochar developed from pine wood recovered from landfills. Similarly, Behazin, Mohanty, and Misra 2017 [21] incorporated ball milled switch grass (*miscanthus*) based biochar into a toughened polypropylene matrix and observed better mechanical properties in the composites. Nan et al. 2016 [22] developed electrically conductive Poly Vinyl Alcohol (PVA) composites by incorporating wood biochar developed from 3 different wood species, carbonized at different temperatures and reported electrical conductivity comparable to composites filled with carbon nanotubes in the polymer composites along with improved of mechanical properties. On a

similar note, Giorelli and Bartoli 2019 [23] developed biochar from coffee grounds by carbonizing the biomass at different temperatures and incorporated this biochar into epoxy matrix and reported electrical conductivity in composites filled with biochar carbonized at 1000°C. Biochar has been successfully implemented as a filler in many different polymer matrices to improve mechanical and thermal properties, however, not many studies have reported the incorporation of biochar as an electrically conductive filler to make non-conductive matrices electrically conductive creating a gap that needs to be addressed.

Polyamide is a ubiquitous polymer and has widespread application in daily life and in industrial manufacturing processes. Polyamide 12 is a semi-crystalline polymer. Polyamide 12 is a polymer with acid amide groups in a straight chain structure and 12 carbon atoms in its monomer [24]. Polyamide 12 has melting point range of 178 to 180°C, modulus of elasticity of 170,000 psi and specific resistivity of 1×10^{14} ohm.cm, it has excellent mechanical properties, chemical resistance and low water absorption compared to the other polyamide polymers [24]. The exceptional properties of Polyamide 12 have attributed to its wide use in the plastic industry and the automotive industry. Recycled Polyamide 12 recovered from Selective Laser Sintering (SLS) process is now receiving a lot of traction as a potential matrix material for composites. The recycled polymer is as good as pure polyamide 12. Feng, Wang, & Wei, 2019 [25] used polyamide 12 recovered from SLS to make filaments to be used in fused deposition modelling, they reported that the mechanical properties of recycled PA 12 is only slightly impaired compared to fresh polyamide 12. Incorporation of recycled polymer in the composite along with biochar makes it a closed loop process and enhances sustainable content in the finished product. Recovery and reuse of polyamide 12 from SLS can pioneer the use of polymers recovered from recycled automobile parts for instance.

2. Material and Methods

2.1. Biochar and polyamide

The biochar used in this study was produced in the lab in Michigan Technological University. The Douglas fir pulp feedstock was procured from Domtar paper org. The pulp was received in the form of compressed boards and was deagglomerated prior to carbonization. The pulp was dispersed in water to deagglomerate the fibers followed by washing with alcohol and then dried overnight at 90 °C. The dried fibers were deagglomerated in a coffee grinder prior to carbonization, the blades of the coffee grinder were masked using duct tape to minimize the effect of blade edges on the aspect ratio of fibers. The fibers were then carbonized in three heating zone tube furnace (Model 23-891, Lindberg, Watertown, WI, USA). The samples were loaded into a quartz tube and the ends were sealed. The fibers were heated to 1000 °C at a heating rate of 10 °C. A steady flow of nitrogen was maintained at 1000 cc/min throughout the process of carbonization. The samples were maintained at 1000°C for 60 minutes. The samples were weighed before and after carbonization to determine biocarbon retention. The biocarbon retention after carbonization at 1000 °C was about 14 – 16 wt. %.

Recycled polyamide 12 recovered from selective laser sintering (SLS) method was used as matrix. It was provided to us by Ford Motor company. The melting point of polyamide 12 was 178 °C and the density was 1.01 g/cm³. The polyamide was sieved before use to remove impurities. 20 wt. % Carbon fiber filled polyamide 6 extruded pellets were provided to us by BASF.

2.2.Characterization of biochar

2.2.1. Elemental Analysis of Biochar

The biochar fibers were characterized for carbon (C) and Nitrogen (N) using elemental analysis. Dried, ground, and homogenized samples were weighed into tin capsules and analyzed for carbon and nitrogen content on an Elemental Combustion System (Costech 4010, Costech Analytical Technologies, Inc., Valencia, CA) in the LEAF core facility at Michigan Technological University. The instrument was calibrated with atropine. Stability was checked with NIST 1547 every 12 samples with a relative standard deviation of 0.03 for N and 0.19 for carbon.

2.2.2. XRD Analysis of Biochar

The crystallinity of wood fiber and biochar samples were analyzed using X-Ray Diffraction. The analysis was done in XDS 2000 (Scintag Inc., USA) at a scattering angle 2θ , scanned from 5° to 60° (at 1.540562 Å wavelength, continuous scanning). The data was analyzed in Microsoft excel.

2.3.Composite formation

The composite samples are developed with different biochar concentrations. Biochar and nylon were weighed and dried overnight at 80°C overnight to remove moisture. The biochar fibers were dispersed in the nylon matrix in a coffee grinder with modified blades. The blades of the coffee grinder were wrapped with duct tape to mask the edges of the grinding blades. This is done to ensure minimal damage to the biochar fiber aspect ratio. The carbon fiber filled polyamide 6 extruded pellets were also conditioned and fabricated into composite discs. The composites were fabricated using hot compression mounting method. The samples were placed in the die and heated to 300°C while 95 psi pressure is applied on to the set up. The assembly was held at the final temperature and pressure for 30 minutes. Figure 1 shows the equipment used to fabricate the composite samples. The cured composites were then measured for diameter and thickness and characterized. The samples were lightly polished using a polishing unit (Leco Spectrum System 1000) at 100-150 rpms. An 8-inch P500 grit Alumina based polishing paper was used with water as polishing media. Each side was polished for about 3-5 minutes to achieve uniform surface texture. Figure 2 shows the entire process of fabrication of biochar filled polyamide composites. The diameter and thickness of the samples was measured.

Loading rate (%)	Biochar (g)	Polyamide (g)
7.5	0.1875	2.3125
9	0.225	2.275

15	0.375	2.125
25	0.625	1.875
35	0.875	1.625

Table I: Composite composition with different filler loading rates.



Figure 1. Heat compression mounting equipment used for fabrication of composites.

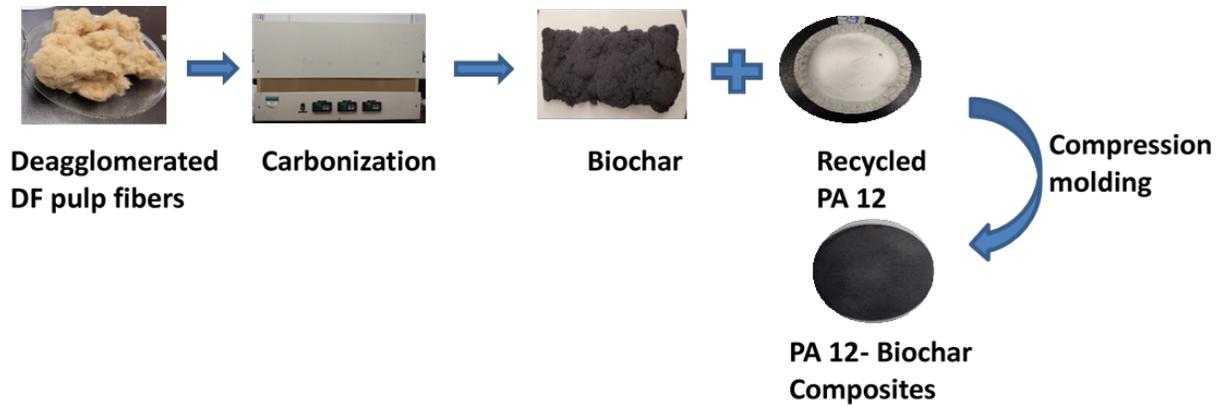


Figure 2. Composite fabrication process for development of biochar filled PA 12 composites.

2.4. Electrical conductivity measurement

The composite samples were characterized for electrical conductivity using a measuring device designed in the lab in Michigan Technological University (Figure 3). The samples are placed between the electrodes and current and voltage is applied to the samples. DC current is applied using a power source (Powerbes DC power supply SPS W1203, having output of 120V and 3A), the current and voltage readings are taken using (Sigilent SDM3065X 6 ½ digit Digital Multimeter) for current and (Sigilent Technologies SDM3055 5.5 Digit Digital Multimeter) for voltage. The current and voltage input values were regulated to keep them at minimum to prevent overheating of sample while obtaining accurate values for measurement. 8 measurements were taken for each sample and 5 replicates were characterized for each loading rate. Average and standard deviation was calculated for each set of samples.

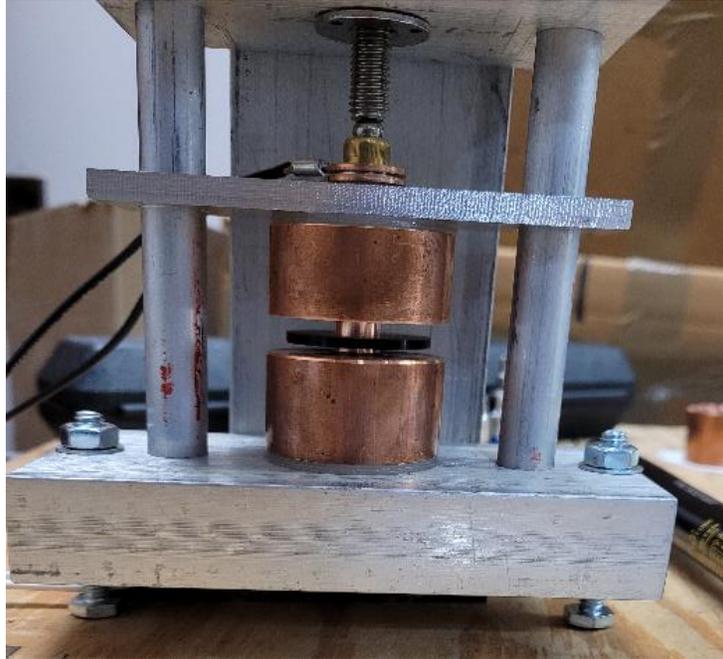


Figure 3. The setup used to characterize the composites for electrical conductivity.

The resistivity of the samples was calculated using the formula described in equation (1).

$$\rho = \frac{RA}{l} \quad \#(1)$$

ρ = Resistivity of sample in Ω cm.

R = Resistance of sample in Ω .

A = Area of the electrodes in contact with the sample in m^2 .

l = Thickness of the sample in m.

2.5. Dynamic Mechanical Analysis

The DMA analysis of pure polymer and biochar filled composites was done in tensile mode for 20mm long 4mm wide 1mm thick samples. The experiment was carried out by heating the samples from ambient temperature at 25 °C to 150 °C, at a strain amplitude of 0.01% and frequency of 1 Hz. The glass transition temperature (T_g) of the samples was calculated from the peak of tan delta peaks.

2.6. Thermal Properties

Thermogravimetric Analysis (TGA) of the composites was performed to determine the effect of biochar addition on the thermal properties of the composites. The analysis was performed

using a TA Q500 TGA instrument. 10 mg of sample was procured from different spots of a composite sample and was placed in platinum sample pans and heated from room temperature 25 °C to 500 °C at a heating rate of 10 °C/min in a nitrogen atmosphere. The samples were maintained at isothermal at 500 °C for 30 minutes. The thermal behavior of pure polyamide 12 and the composites were compared to determine the effect on biochar on the thermal stability of the composites.

2.7.Scanning Electron Microscopy Analysis

Scanning Electron Microscopy was used to analyze the morphology of the composites. The composite discs were placed in liquid nitrogen for 5 minutes and then broken using pliers. The pieces were coated with platinum/palladium using sputter coating to make the surface conductive. The SEM was done using Hitachi S-4700 Field Emission (FE) SEM instrument. The imaging was done at 100 kV, 30 μm magnification.

3. Results and Discussion

3.1.Elemental Analysis

The elemental analysis of Douglas fir biochar fibers carbonized at 1000 °C showed 88%

carbon content in the biochar and 0.7% nitrogen content. The carbon content in the biochar is influenced by carbonization temperature and it is believed with higher carbonization temperature a higher carbon content can be achieved.

3.2.XRD Analysis

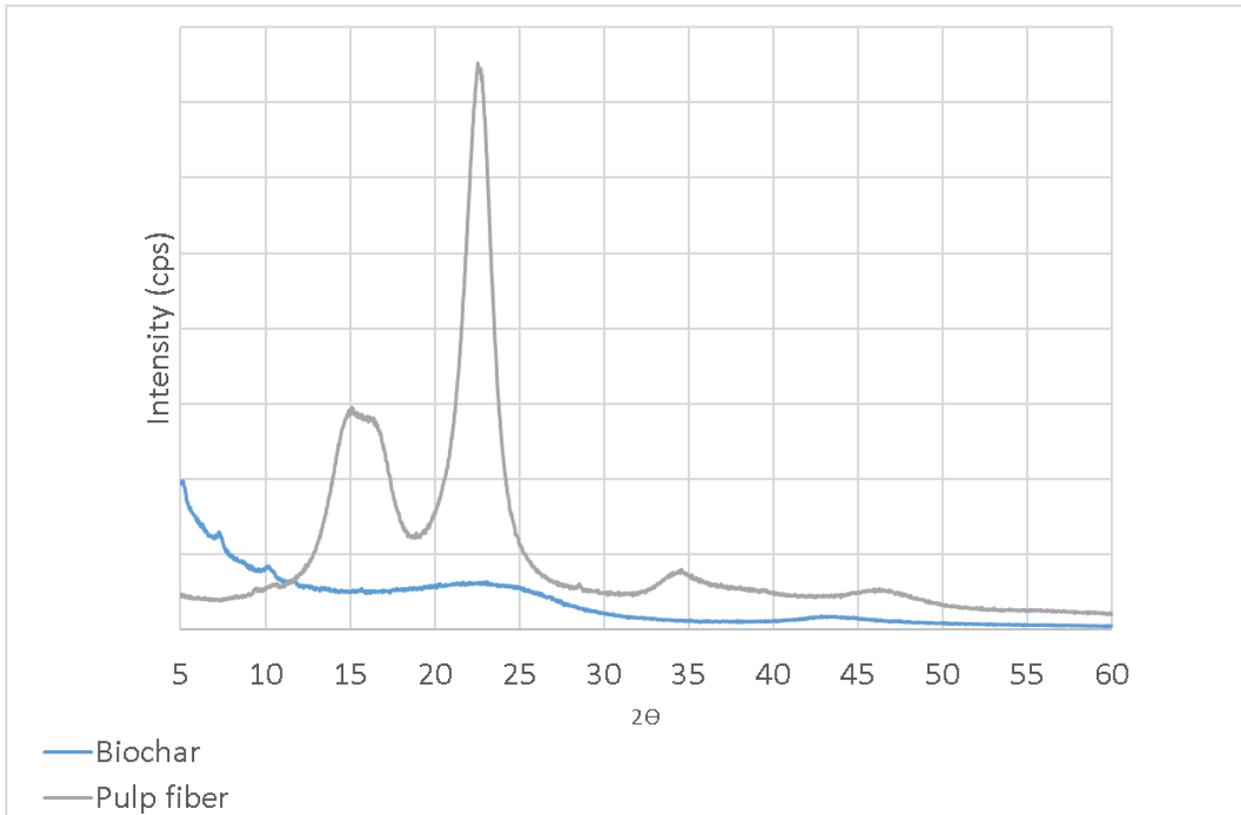


Figure 4. XRD pattern of pulp fiber vs biochar.

The Figure 4 here shows the comparison between the XRD pattern of Douglas fir pulp fiber and biochar fibers. The broad peak between 15°– 20° and the sharp peak at 20° for Douglas fir pulp fiber are believed to be cellulosic peaks. These peak patterns indicate the presence of a high cellulose content in the feed stock. On the contrary, in biochar these peaks are absent. The lack of significant peaks for biochar are indicative of the amorphous nature of biochar. The short and broad peak obtained at 20°– 25° for biochar could be due to the presence of turbostratic structure obtained by carbonization at high temperature like 1000 °C [26]–[28]. The transition from crystalline to amorphous could be a result of high temperature carbonization.

3.3.Electrical conductivity of composites:

Improved electrical properties were observed in the composites compared to pure polyamide 12 that has a volume resistivity between 10^{12} - 10^{14} Ω cm. At low biochar loading level of 7.5wt % the resistivity of the composite samples was reported to be 7.13×10^3 Ω cm indicating drastic drop in electrical resistivity compared to pure polymer. The electrical resistivity was reported to have further dropped with increasing filler concentration in the composites. At 35% filler concentration the resistivity value reported by the composites was 2.13Ω cm . Clingerman 2001 [29] reported transverse volume resistivity value of resistivity of 5.04×10^8 Ω cm for polyamide 6 composites filled with 20 wt % carbon fiber and longitudinal volume

resistivity of 10.08 Ω cm for 40% carbon fiber filled polyamide 6 composites. The resistivity values for composites filled with both 20 wt.% and 40 wt. % carbon fiber is much higher than biochar composites having a relative biochar fiber filler concentration with respect to carbon fiber concentration. This is an indicator of the effectiveness of biochar as a conductive filler. Several factors affect the electrical properties of biochar which in turn affects the overall composite properties. A major factor contributing to the electrical conductivity of biochar is the carbonization temperature. Several studies have reported good electrical conductivity in biochar carbonized at $\geq 1000^\circ\text{C}$ [22], [30]–[32]. The carbonization of feedstock at a high temperature results in a turbostratic structure in biochar which is composed of discontinuous highly ordered crystalline phases [15], [33]. The turbostratic structure of biochar is responsible for the conductivity of biochar. The loading rate is another factor, as it can be observed in the study that with increasing filler concentration results in improved composite electrical properties, similar trend is also observed in other studies conducted using biochar as an electrically conductive filler [22], [32]. Morphology of filler is also a contributing factor to the electrical conductivity of composite [34]. The fibrous morphology of the biochar fibers incorporated in the polyamide12 matrix in the study has contributed to the improved electrical properties even at low filler concentration. The electrical resistivity of biochar filled composites was compared to 20 wt.% carbon fiber filled polyamide 6 composites that were received from BASF. The 20-wt. %carbon fiber filled composites, reported a resistivity of 27.85 Ω cm that lies between the resistivity value of biochar composites filled with 25 wt.% biochar having a resistivity of 11.7332 Ω cm and 15 wt. % biochar filled composites having resistivity of 84.95 Ω cm. The carbon fiber composites at 20 wt.% have resistivity higher compared to 25 wt.% biochar filled composites. This indicates the good electrical conductivity of biochar fibers, that is reflected in the electrical properties of the composites. The electrical resistivity of biochar filled composites is presented in Table II.

Table II: Electrical resistivity of biochar filled composites.

Filler loading (%)	Resistivity (Ωcm)	COV (%)
7.5	$4.98 \times 10^6 \pm 1.11 \times 10^7$	222.93

9	$1.43 \times 10^6 \pm 3.05 \times 10^6$	161.86
15	84.953 ± 30.6	36.01
25	11.73 ± 1.74	14.8
35	2.06 ± 0.5	23.9

3.4. Dynamic Mechanical Analysis

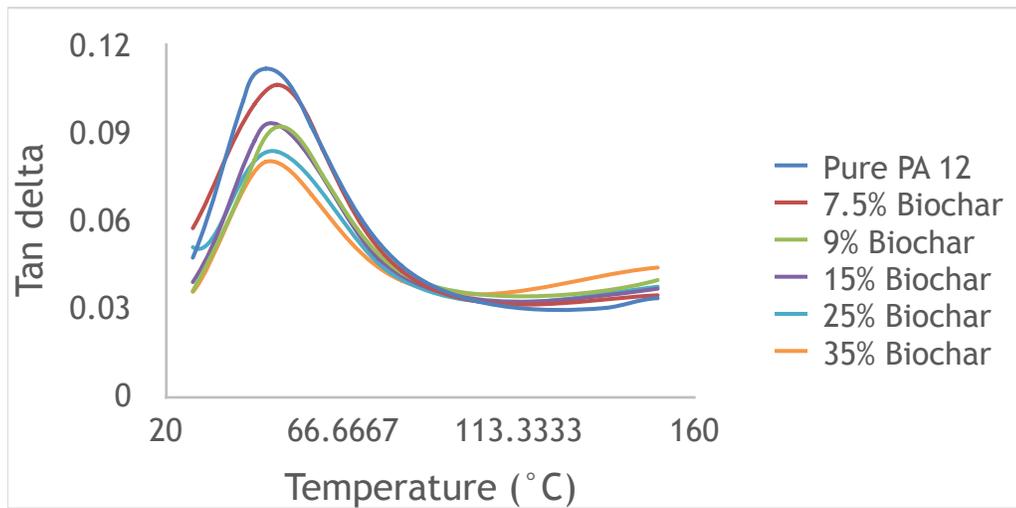


Figure 5 (a). Tan delta peaks for pure PA 12 and biochar filled polymer composites at 1 Hz.

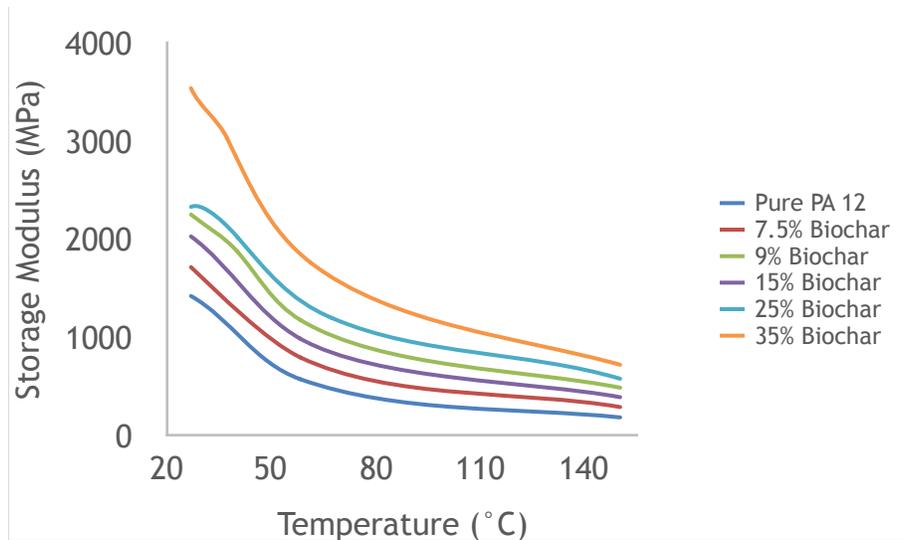


Figure 5 (b). Storage modulus trend for pure PA 12 and biochar filled composites.

The dynamic mechanical analysis of pure PA 12 and biochar filled composites show the glass transition temperature T_g of PA 12 at 46.27 °C and for the composites at 47 °C indicating no significance of biochar fillers on the T_g of composites as the value is very close to T_g value of pure PA12. The tan delta peaks (figure 5 (a)) show declining magnitude with increase in filler concentration, suggesting an increase in composite stiffness compared to pure PA 12 which reflects a decrease in energy absorption capacity of the composite. On the other hand, the storage modulus values (figure 5 (b)) show steady increase with increasing filler content, this improvement was expected with incorporation of filler.

3.5. Thermal properties

The TGA weight loss curves (figure 6 (a)) indicated a shift in the degradation temperature of the composites compared to pure polyamide 12 polymer. The onset of thermal degradation takes place at 372°C for polyamide 12 while for the composites lower loading rates 7.5% and 9% have temperatures close to the thermal degradation temperature of polyamide around 367°C. There is a significant shift in higher loading rate towards higher degradation temperatures. The temperatures for 25% and 35% filler loading composites are above 400 °C. This indicates enhanced thermal stability in composites introduced by incorporation of thermally stable biochar. A negligible amount ~1% of residue was obtained after the entire degradation cycle of polyamide 12, while in case of composites significant amount of residue was obtained on completion of the analysis. The amount of biochar residue increased with filler concentration of the composites. This is an indicator of improved thermal properties of composites as well. The residue is also indicative of the material loss during composite processing. The higher the filler retention the lower the material loss throughout the processing phase in the sample specimen(s). The amount of residue for each loading rate indicates very less material loss during the processing of composites.

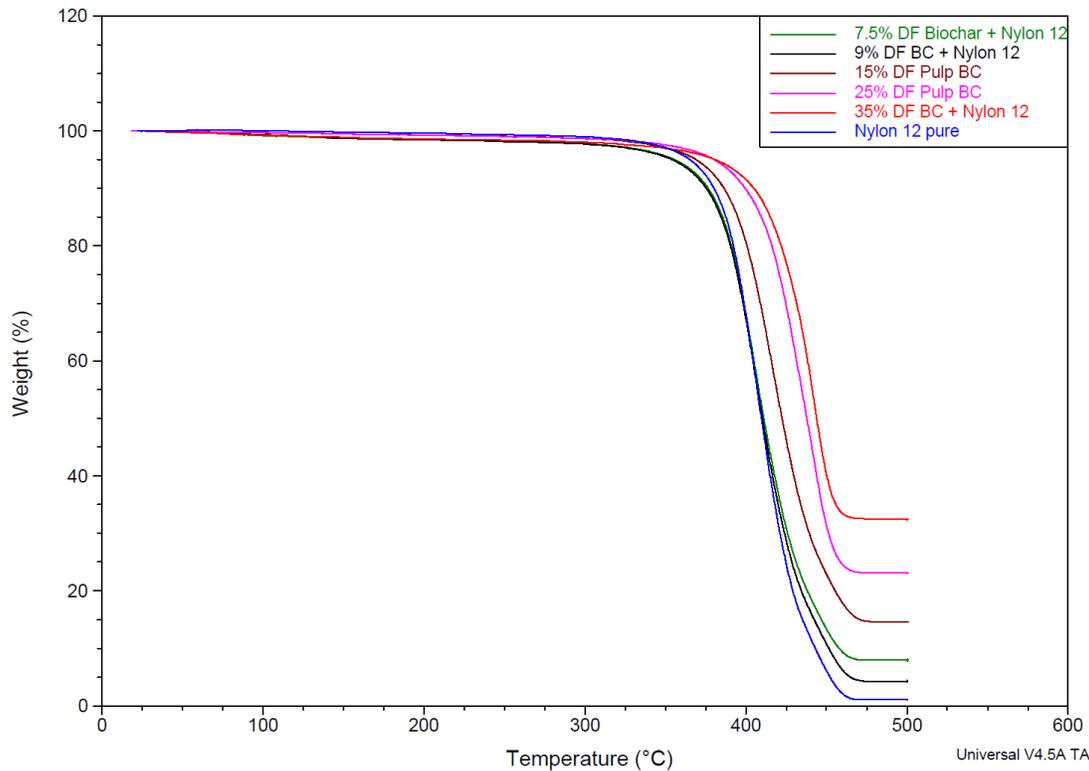


Figure6(a). TGA mass loss curves for biochar filled composites compared to mass loss curve for pure polyamide 12.

The DTG curves (figure 6 (b)) shows, the thermal decomposition of polyamide was initiated at temperature 400°C while in the case of composites, for lower loading rates 7.5% and 9% the maximum degradation temperature is similar to polyamide 12 however a shift towards right is observed with increasing filler loading rates. 15% biochar filled composites mark the beginning of improved thermal properties and the improvement is significant in 25% and 35% samples as a significant increase in maximum degradation temperature is observed. This shift shows the improved thermal properties in the composites on addition of biochar filler to the polymer matrix.

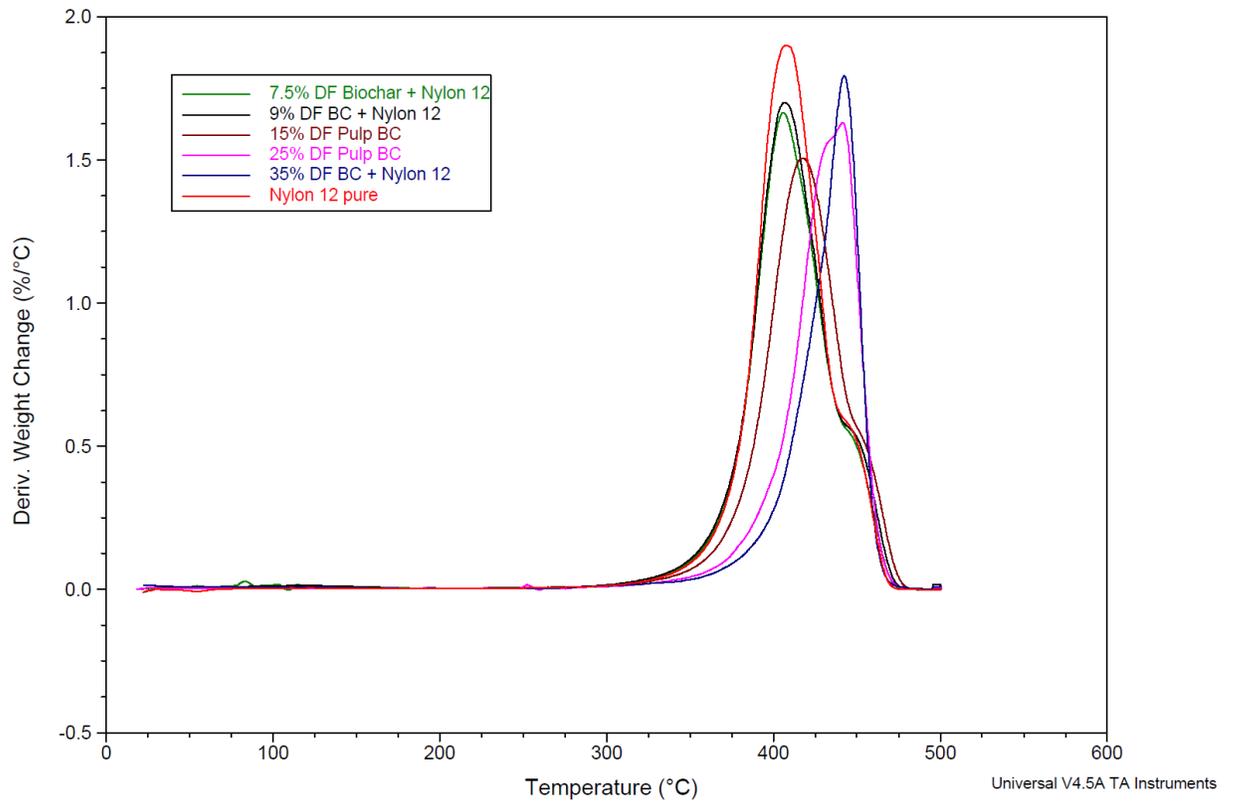


Figure 6 (b). DTG curves for biochar filled composites show significant improvement in maximum degradation temperature in composites in comparison with pure polyamide 12 polymer.

3.6.SEM Analysis

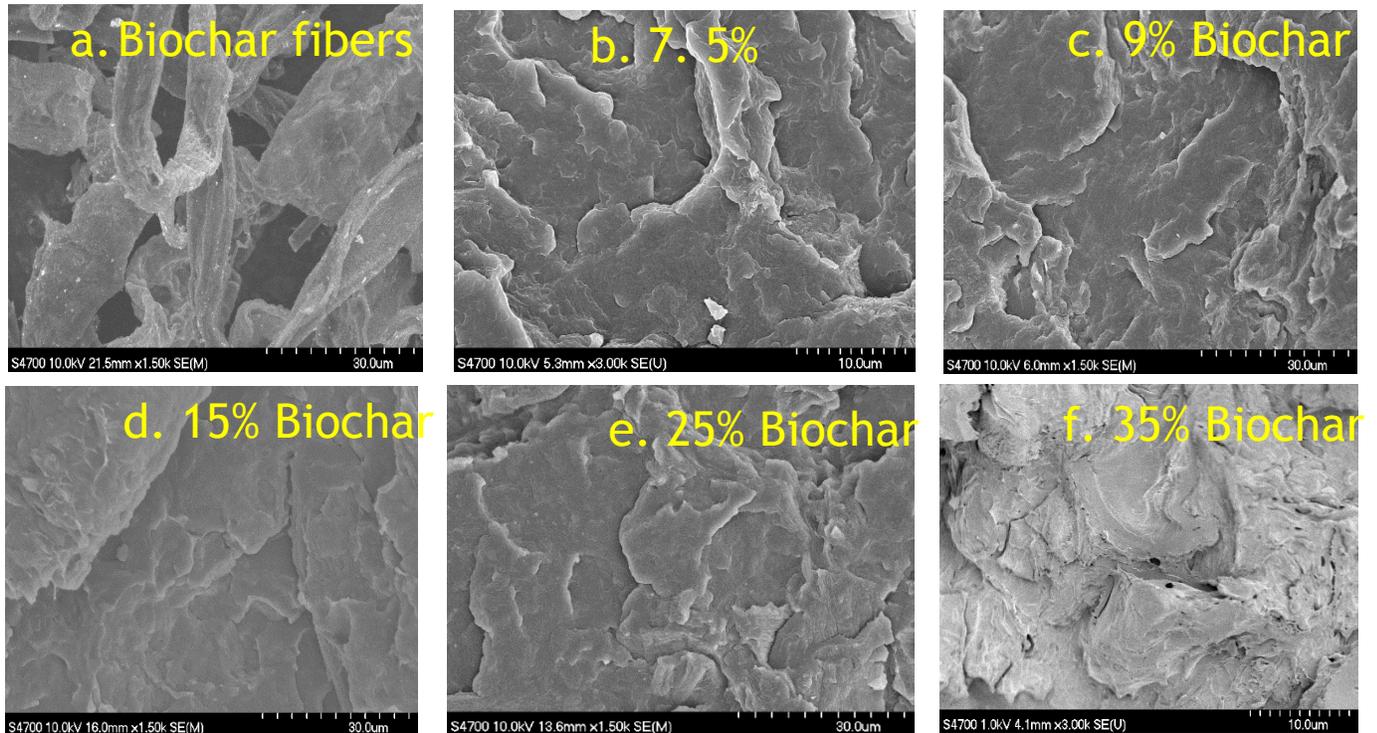


Figure 7. SEM images of biochar fibers and biochar-polyamide 12 composites, (a) Biochar Fibers, (b) 7.5% biochar filled composites, (c) 9% Biochar filled composites, (d) 15% Biochar filled composites, (e) 25% biochar filled composites (f) 35% biochar filled composites.

The Figure 7 (a) showing SEM image of biochar fibers does not show the presence of pores on the biochar surface. This is contrary to expectation since biochar is characterized by the presence of these honeycomb shaped pits. One of the reasons for the absence of these pits could be the pulping process to produce pulp from raw material that could have damaged the anatomical features of wood largely. Another reason could be the carbonization at such high temperatures that could have caused a certain amount of damage to the structure of biochar. The fibrous structure of Douglas Fir pulp fibers is quite maintained in the biochar fibers even after carbonization. In the SEM images of composites, interestingly no distinction was observed between the filler and the matrix. This could be due to the fibrous morphology of filler that makes it difficult to identify in the composites. The SEM results indicate further deeper investigation of the morphological features of composites to figure out the unique properties attributed to the incorporation of biochar fibers instead of biochar particles.

4. Conclusion

Incorporation of biochar as an electrically conductive filler has shown promising results. The resistivity value has shown a significant drop even at low filler concentrations. The carbonization temperature and morphology of the filler are two important factors in enhancing the electrical conductivity of biochar and hence improving the electrical properties of the composites. The thermal properties have also shown improvement by indicating higher thermal stability on incorporation of biochar to pure polyamide12 polymer matrix. These positive results definitely

indicate the potential of biochar as an electrically conductive filler to be used in manufacture of automobile parts. The use of biochar fibers is pretty unique since all the studies done previously have utilized biochar particles and/or powder. Utilization of recycled PA 12 from SLS waste definitely enhances the sustainability of the process and paves way for utilization of similar waste products maybe from automotive waste. Post end of life cycle automotive parts can be recycled providing raw material for further application as raw material. The recycling and reutilization of waste will make the process circular eliminating waste generation and improving the carbon footprint in the entire lifecycle of the vehicle. The utilization of biochar fiber filler can provide the manufacturers with a cost effective sustainable conductive filler alternative improving the economic returns while reducing the environmental cost for both the manufacturer and the consumer.

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