

WASTE VALORIZATION TOWARDS CIRCULAR ECONOMY: OCEAN RECYCLED NYLON AND LIGNIN-DERIVED BIOCARBON IN ENGINEERING LIGHT-WEIGHT SUSTAINABLE BIOCOMPOSITES

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Abstract

Traditional non-renewable petroleum-derived plastics present increasing environmental concerns observed through rising plastic pollution, where currently ~10 million tons of plastic are entering oceans annually. The use of recycled and agro/forestry-waste products in developing automotive parts supports the movement towards a circular economy. This work focuses on engineering biocomposites using ocean recycled nylon (ORN) for high-performance automotive applications. Lignin (waste from pulp and paper industries) was carbonized at 600 and 900 °C and used as sustainable fillers for manufacturing biocomposites. The influence of carbonization temperature on the thermal, structural, surface morphology, and mechanical properties of ORN biocomposites was analyzed. A decrease in melt flow index was observed by adding the biocarbon (BioC) as a filler in the matrix. Compared to the non-functional 900 °C BioC; 600 °C BioC showed reduced MFI suggesting an improved interfacial adhesion of 600 °C BioC with ORN. Adding lignin BioC to ORN enhanced its flexural strength and modulus. The tensile modulus also increased in biocomposites with respect to the neat ORN. The effect of maleated compatibilizers and chain extender on mechanical properties were further studied. The developed biocomposites with more than 90% sustainable content have the potential to be used as light-weight automotive parts.

Background

Plastic plays an important role in our day-to-day lives. The lightweight and inexpensive nature of plastic results in it being preferred over other materials. Since 1950, more than 7.8 billion metric tons of plastic have been produced, and over 50% of this has been produced from 2004 to 2017,

with a compound annual growth rate of 8.4%.^{1,2} It is expected that by 2050 plastic production will emit over 56 billion metric tons of carbon-dioxide-equivalent in greenhouse gases.² Upon entering marine environments, plastic pollution presents harmful effects, including habitat destruction, transporting invasive species, entanglement and ingestion by aquatic animals, and depositing in sediments.³⁻⁵ Current estimates predict that approximately 100,000 tons of plastic are found in surface waters and by 2050, it is expected that the quantity of plastic in the ocean will outweigh fish.^{1,6} The growing concern of microplastic consumption also poses threats to humans as it is estimated that 39,000 to 52,000 microplastic particles are consumed annually from food and beverages.⁷

Nylon is a polyamide that consists of monomeric amide subunits (-CONH-), which was discovered in 1938 by W.H Carothers.⁸ A few of the commercial nylons used are nylon 6, nylon 6/6 and nylon 11. Nylon 6 is synthesized by the ring opening polymerization of ϵ -caprolactam, nylon 6/6 by polycondensation of hexamethylene diamine and adipic acid.⁸ Nylon 11 is synthesized by the polycondensation of 11-aminoundecanoic acid.⁸ Nylon is a widely studied engineering polymer due to its ability to perform under high temperatures and pressures.⁹ This allows for uses in high-performance applications such as under hood in the automotive industry.¹⁰

Fillers used in engineering plastic for fabricating automotive composites such as carbon fiber, nanotubes and graphene are of high density and expensive materials.⁹ Alternative low cost fillers such as talc, clay and glass fibers also have the disadvantage of high density.⁹ The density of commonly used fillers like glass and carbon fibers are ~ 2.55 and 1.77 g/cm^3 , respectively.¹⁰ Agro-waste (corn cobs, peanut hulls, wheat straw) can be fabricated to make sustainable, low cost fillers (biocarbon)¹¹. Biocarbon (BioC), also known as biochar, is a thermally stable carbon-rich material.⁹ BioC can be used as fillers in making composite material as an alternative to other commonly used materials such as glass fibers, carbon fibers, and mineral particulates.^{9,12} The use of BioC presents an inexpensive, lower density ($1.34\text{-}1.96 \text{ g/cm}^3$), and sustainable alternative to these other fillers.^{9,13}

Lignin is found in many natural sources, such as woody biomass and agricultural residues.¹⁴ In plant cells, lignin acts as a structural support providing plants with the required mechanical strength. It also acts as an adhesive agent between the cellulose fibers.¹⁵ Behind cellulose, lignin is the second most abundant renewable raw material.¹⁶ Lignin is a by-product of the pulp and paper industry, where about 50 million tons of lignin waste are generated each year.¹⁷ Most of this waste ($\sim 95\%$) is directly discharged into rivers or burned after the concentrating process.¹⁷ This method of disposal is both a waste of energy and a cause of environmental pollution.¹⁷

Due to the abundance of lignin waste that is produced, utilizing the material as a source of BioC can serve as a promising waste management option. The properties of lignin can be influenced by different pyrolysis conditions such as extraction method, carbonization temperature, heating rate, and residence time.¹⁸ Different pyrolysis temperatures play an important role in determining the functionality of the lignin BioC. The functionality of biocarbon can influence the interactions with the polymer matrix which can affect the thermal, mechanical, and electrical

properties of composites.¹⁸

This work aims to evaluate the effect of adding lignin biocarbon produced at different pyrolysis temperatures, maleated compatibilizers and a chain extender to ocean recycled nylon (ORN) composites. The composites were characterized to determine the mechanical, thermal, and structural properties.

Materials

The ocean recycled nylon (ORN) pellets used in this work were acquired from DSM, with the trade name of Akulon® (Made in India). The Softwood Kraft lignin with the tradename Indulin®AT used to produce the biocarbon was acquired from MeadWestvaco. The maleic anhydride grafted polypropylene (PP) compatibilizer with the trade name Fusabond P353 was supplied by Dupont. The maleic anhydride was purchased from Acros Organics (Hungary) and the free radical initiator, dicumyl peroxide (DCP), was purchased from Acros Organics. Joncyl (ADR-4368), an epoxy functional oligomeric agent, was purchased from BASF and was used as a chain extender.

Methods

Pyrolysis of lignin

The pyrolysis of 1.5 kg of lignin was conducted in a Carbolite Gero GLO (UK) batch type retort furnace in an inert atmosphere. The operating temperatures of 600 and 900 °C were achieved with a heating rate of 5 °C/min held for 90 minutes and 60 minutes respectively.

Preparation of maleated ocean recycled nylon

The maleated ORN compatibilizer was prepared from the extrusion of 200 g of ORN, 10 g of maleic anhydride and 2 g of DCP (5 and 1 phr respectively). The synthesis was carried out in Thermo Scientific Haake PolyLab™ at an operating temperature of 245 °C. First, ORN was added to the extruder and was mixed for 2 minutes. Next, DCP was added and mixed for an additional 2 minutes. Finally, the maleic anhydride was added and mixed for 6 minutes.

Preparation of biocomposites

The pellets were prepared using a Labtech Engineering Co. 26 mm twinscrew extruder with a temperature profile of 235-235-245-245-245-245-245-245 °C for the eight heating zones. The prepared compositions are outlined in Table 1. A single screw minijector (MPM model #55, US) was used to prepare the tensile, flexural, and impact samples. The three heating zones were set to 240, 243 and 245 °C with a packing pressure of 750 psi.

Table 1: Sample identification and ratio of the materials within the composites

ID (Ocean recycled nylon and its composites)	Ocean Recycled Nylon (ORN) (wt%)	Lignin Biocarbon 900 °C (900) (wt%)	Lignin Biocarbon 600 °C (600) (wt%)	Maleated grafted Ocean Recycled Nylon (MN) (wt%)	Maleated grafted polypropylene (PP) (wt%)	Joncryl (phr)
ORN	100	-	-	-	-	-
ORN/900	77	23	-	-	-	-
ORN/600	77	-	23	-	-	-
ORN/600/3MN	74	-	23	3	-	-
ORN/600/3MN/0.5J	74	-	23	3	-	0.5
ORN/600/3MN/2J	74	-	23	3	-	2
ORN/600/5MN	72	-	23	5	-	-
ORN/600/3PP	74	-	23	-	3	-
ORN/600/5PP	72	-	23	-	5	-
ORN/900/3PP	74	23	-	-	3	-

Characterization

Fourier transform infrared spectroscopy (FTIR) was performed using a Nicolet 6700 (Thermo Scientific, USA). All samples were scanned with 4 cm⁻¹ resolutions and 256 scans.

The density of the composites was determined using an electron MD-300S densimeter (Alfa Mirage, Japan).

The Melt Flow Index (MFI) was performed in accordance with ASTM 1283 using a melt flow indexer (2000A, Qualitest) at a temperature of 245 °C with a load of 5.00 kg.

The Heat Deflection Temperature (HDT) was measured using a Dynamic Mechanical Analyzer (DMA) Q800, TA Instruments (USA). The tests were run in accordance with ASTM D648 using a 0.455 MPa load, 2 °C/min heating rate and a three-point cantilever.

A scanning electron microscope was used to determine the surface morphology of the impact

samples at an acceleration voltage of 10 kV on an SEM Phenom ProX (Netherlands).

The notched impact samples were tested on a Zwick/Roell HP790 impact tester (Germany) with a 2.75 J capacity Izod hammer in accordance with ASTM D256.

The tensile strength was determined using an Instron 3382 Universal Test Machine (USA) according to the ASTM D638 standard with a test rate of 5 mm/min for type IV tensile samples.

The flexural properties of the composites were tested in accordance with the ASTM D790 standard with a test rate of 14 mm/min. The tests were performed on an Instron 3382 Universal Test Machine (USA).

Results and Discussion

Grafting: FTIR

The efficiency of maleation of the ORN can be determined with the use of FTIR (Figure 1). The characteristic peaks for nylon 6 can be observed in Figure 1, which include N-H stretching (3301 cm^{-1}), C=O stretching (1635 cm^{-1}) and N-H bending (1535 cm^{-1}). Comparing the spectra of ORN to the maleated ORN there is the presence of two additional peaks between 1650 and 1850 cm^{-1} which represent the formation of the maleic anhydride ring. The band at 1725 cm^{-1} typically represents the C=O stretching of the maleic anhydride ring, while the band in the range of $1705\text{-}1715\text{ cm}^{-1}$ could result from carboxylic acid C=O stretching associated with the ring opening hydrolysis of the maleic anhydride ring.¹⁹

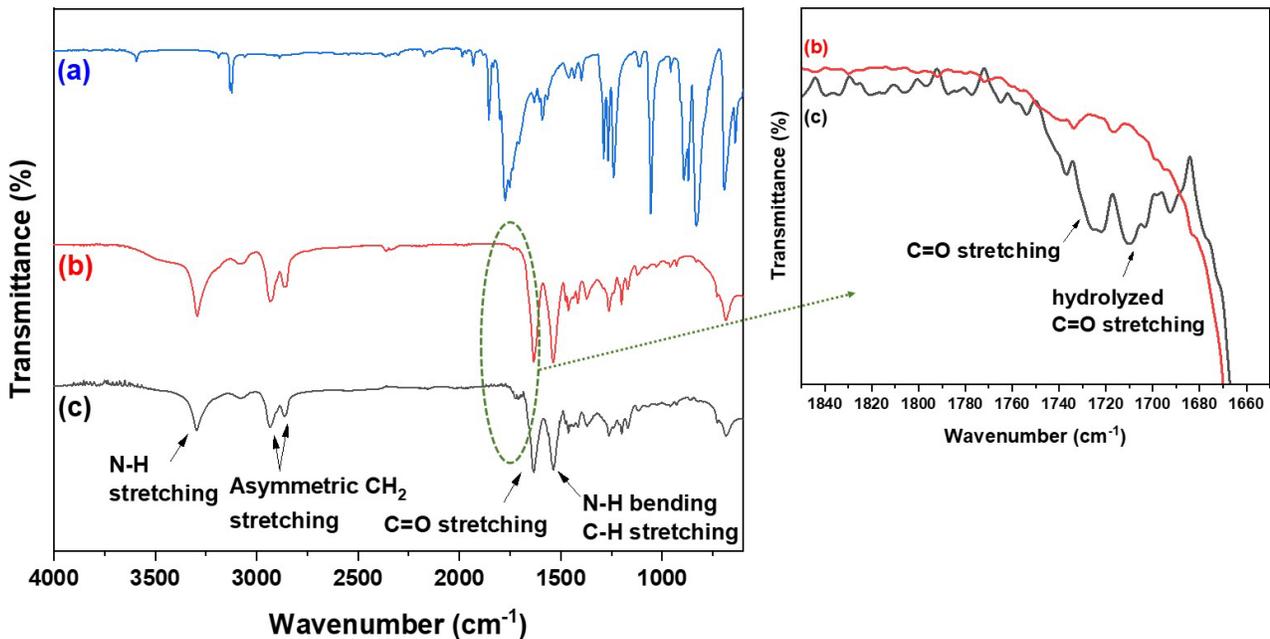


Figure 1. FTIR spectra of (a) maleic anhydride (b) ORC and (c) maleated ORN

Density, Melt flow and Heat deflection properties

The density measurements of the composites are outlined in Table 2. These results show that lignin BioC is a low density filler as the density only increased by a maximum value of 0.079 g/cm³ compared to the neat polymer.

The MFI of the prepared composites is outlined in Table 2. The MFI is a measure of the flowability of the composite material in the molten state. When BioC is added to the composites, the MFI reduces significantly when compared to neat ORN. The reduction in MFI is a result of the biocarbon acting as a barrier to the mobility of the polymer chain, which causes a reduced flow of the material.¹⁰ This trend continues with the addition of the PP and ORN compatibilizers. The composites containing 900 °C BioC have higher MFI values than the 600 °C BioC, which might be a result of the better interfacial adhesion of the functionalized 600 °C BioC with the polar ORN matrix, compared to the 900 °C BioC.

The HDT measurements of the samples are given in Table 2. With the addition of BioC to the ORN matrix, the HDT is increased. The addition of BioC to ORN results in restricted polymer chain mobility, which as the temperature rises under a constant load, prevents the composite from deforming; therefore, leading to a higher HDT.²⁰ When maleated grafted ORN compatibilizer was added to the composites, the HDT increased compared to the neat polymer. However, in the ORN/600/3PP and ORN/600/5PP composites the HDT decreases compared to the neat polymer, which may indicate poor interaction of the BioC with the polymer matrix leading to more mobile polymer chains, and therefore a lower HDT.

Table 2: Density, MFI, and HDT of samples

ID	Density (g/cm ³)	MFI (g/10 min)	HDT (°C)
ORN	1.107 ± 0.001	69.0	173.26
ORN/900	1.183 ± 0.003	40.0	185.94
ORN/600	1.175 ± 0.003	36.0	183.87
ORN/600/3MN	1.172 ± 0.003	32.4	178.06
ORN/600/3MN/0.5J	1.171 ± 0.005	32.6	175.76
ORN/600/3MN/2J	1.168 ± 0.005	31.4	173.38
ORN/600/5MN	1.174 ± 0.001	32.6	176.20
ORN/600/3PP	1.176 ± 0.002	32.1	169.63
ORN/600/5PP	1.168 ± 0.002	31.6	169.76
ORN/900/3PP	1.186 ± 0.002	39.0	180.22

Surface Morphology

The SEM images of the fracture surfaces of the impact samples are outlined in Figure 2. In the ORN/600 composites (Figure 2A), several empty macro pores could act as a source of crack initiation.²¹ Figure 2(A) further shows the pull-out of biocarbon at various locations (identified by red arrows) uniformly, representing a good distribution of the BioC within the ORN matrix. Pull out of biocarbon occurred more in ORN/600 (Figure 2(A)) with respect to Figure 2(B) ORN/600/3MN and Figure 2(C) ORN/600/3MN/0.5J. Further, the maleated matrix without and with 0.5 phr Joncryl showed a smoother surface than Figure 2(A) refers to the good interaction between the biocarbon and the matrix. However, adding 2 phr Joncryl made the surface less uniform, suggesting the interfacial adhesion between matrix and filler was reduced. In the ORN/600/3MN, ORN/600/3MN/0.5J, and ORN/600/3MN/2J composites, a more uniform surface was observed compared to ORN/600, which may result in better impact strength.

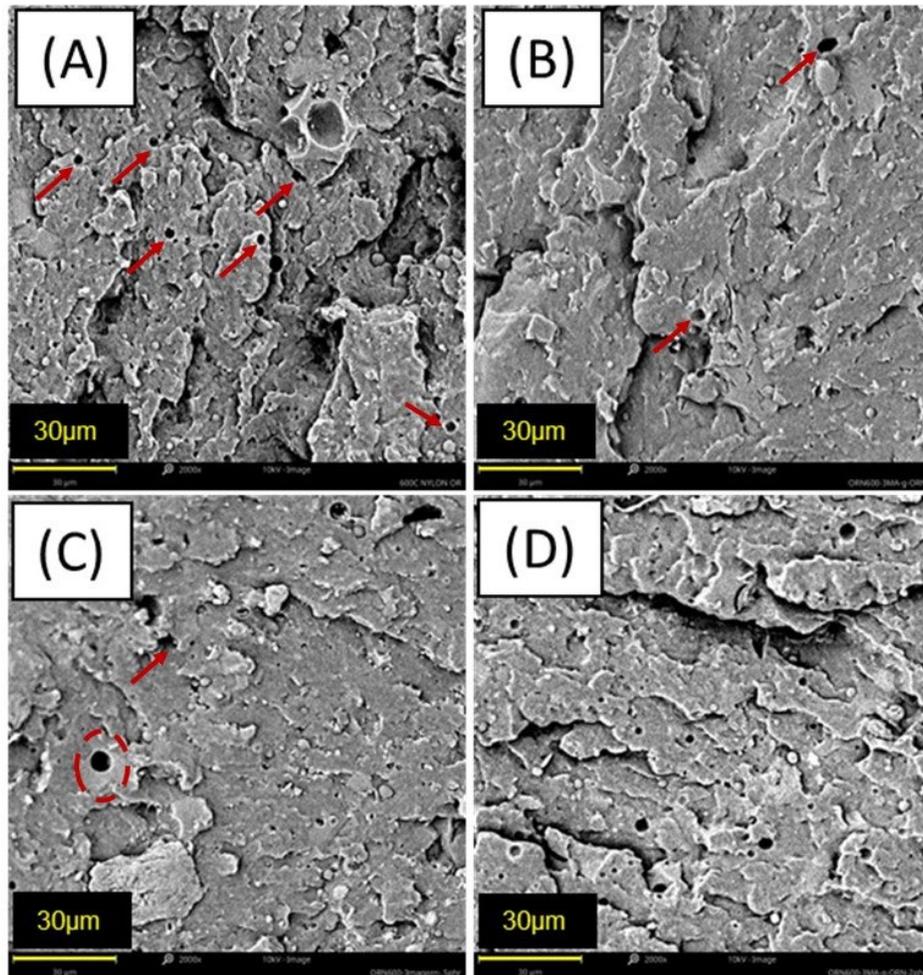


Figure 2. SEM images of (A) ORN/600, (B) ORN/600/3MN, (C) ORN/600/3MN/0.5J, (D) ORN/600/3MN/2J.

Mechanical Properties

The impact strength of the Izod notched samples all decreased with respect to the neat polymer (Figure 3). This reduction in impact strength is a result of the addition of BioC, a highly rigid filler, causing the composites to become more brittle due to reduced ductility.²² The reduction in ductility restricts the polymer chain which obstructs plastic deformation.²² The addition of BioC to the composites results in an approximate 50% reduction in impact strength compared to the neat polymer. With the addition of maleated grafted PP and ORN compatibilizers, there is an improvement in this property. An enhancement of 37% in impact strength is observed for the ORN_600_3MN composite when compared to ORN/900 and ORN/600. The addition of Joncylr, a chain extender, to the ORN/600/3MN composite results in a further increase in impact strength. The addition of a chain extender results in an increase in molecular weight from the formation of long, branched chains.²³ The enhancement in impact strength with the addition of Joncylr may be a result of the increased molecular weight and the morphology of the branched chains leading to a reduction in evolving cracks.²³

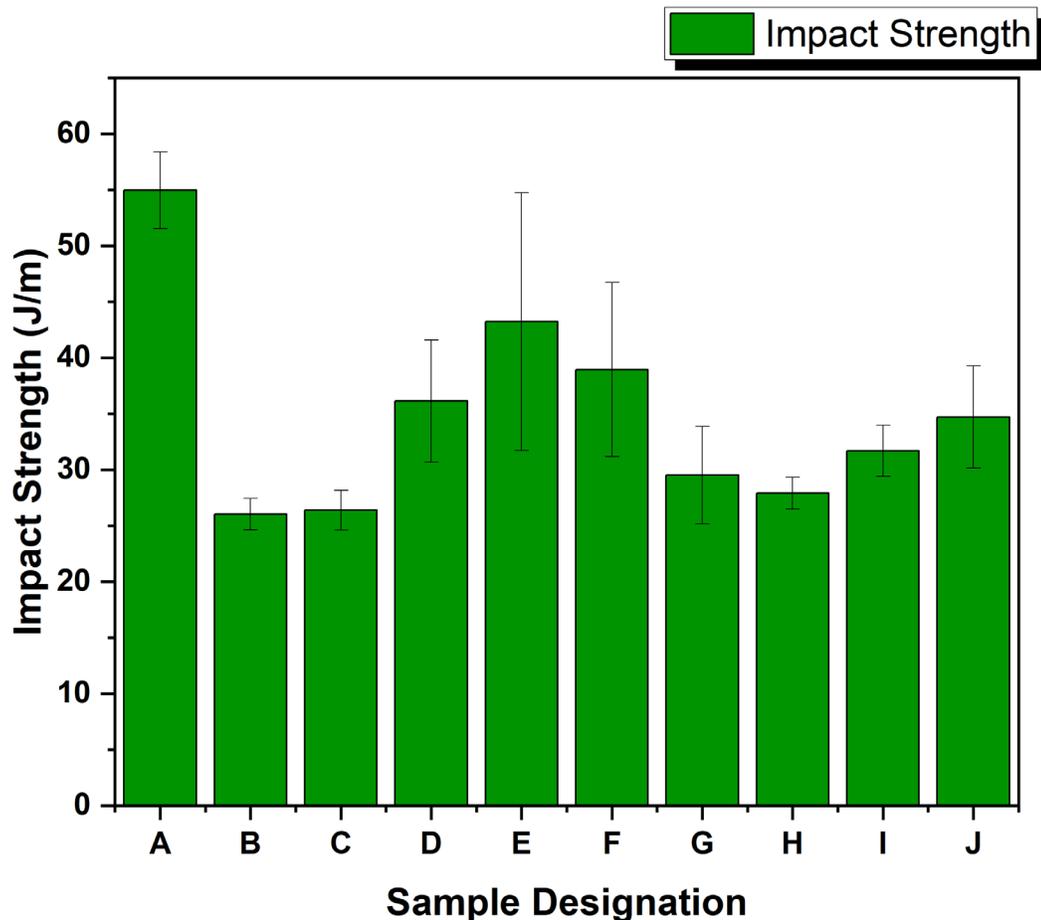


Figure 3. Impact strength of (A) ORN, (B) ORN/900, (C) ORN/600, and (D) ORN/600/3MN, (E) ORN/600/3MN/0.5J, (F) ORN/600/3MN/2J, (G) ORN/600/5MN, (H) ORN/600/3PP, (I) ORN/600/5P, (J) ORN/900/3PP

The flexural properties of the composites and neat ORN are outlined in Figure 4. The addition

of BioC, a high modulus filler, to the neat ORN matrix results in an increase in flexural strength and modulus. The increase in flexural strength, of over 50%, for ORN/600 and ORN/900 is comparable to the increase observed for the maleated grafted ORN composites. The flexural strength is reduced in the maleated grafted PP composites; however, this property is still enhanced over the neat polymer by up to 37%. The flexural modulus is enhanced in all composites compared to neat ORN (flexural modulus: 1774 ± 169 MPa). The greatest increase, of 84%, is observed for the ORN/900 composite (flexural modulus: 3258 ± 63 MPa), with comparable values observed for the ORN/900/3PP composite (flexural modulus: 3212 ± 163 MPa). The maleated grafted ORN composites showed reduced flexural modulus compared to ORN/600 and ORN/900 composites, however, this property was improved by 61% compared to the neat polymer. The addition 0.5 phr of Joncryl to the ORN/600/3MN composite resulted in the largest increase in flexural strength (110.68 ± 4.67 MPa), with a 56% increase compared to the neat polymer (70.87 ± 6.15 MPa). The flexural modulus was also enhanced compared to the ORN/600/3RN composite by 10%. The addition of 2 phr of Joncryl to this composite did not enhance the flexural strength or modulus of the ORN/600/3MN composite.

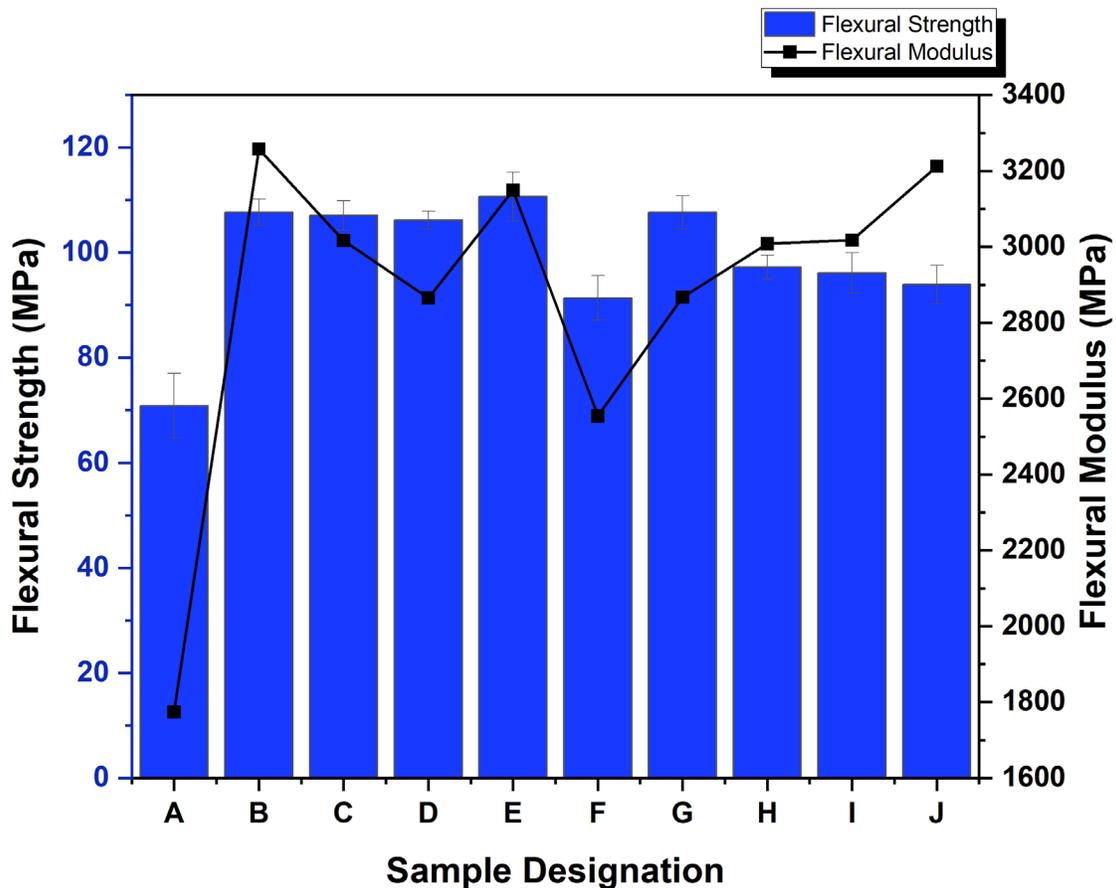


Figure 4. Flexural strength and modulus of (A) ORN, (B) ORN/900, (C) ORN/600, and (D) ORN/600/3MN, (E) ORN/600/3MN/0.5J, (F) ORN/600/3MN/2J, (G) ORN/600/5MN, (H) ORN/600/3PP, (I) ORN/600/5PP, (J) ORN/900/3PP

Figure 5 outlines the tensile properties of the composites and neat ORN. In comparison to the neat polymer, the tensile modulus increases in all composites. The greatest increase is observed for the ORN_600 (tensile modulus: 4050 ± 125 MPa) and ORN_900 (tensile modulus: 4130 ± 138 MPa) composites with an increase greater than 60% compared to neat ORN (tensile modulus: 2496 ± 125 MPa). The ORN and PP maleated grafted compatibilizers did not show a comparable enhancement in tensile modulus when compared to ORN_600 and ORN_900. The tensile strength was greatest for the ORN_600 composite at 74.7 MPa, a 34% increase compared to neat ORN. The maleated grafted ORN composites also showed increased tensile strength in comparison to the neat polymer, with a 22% enhancement. The PP maleated grafted composites did not show any enhanced tensile strength compared to the neat polymer. Upon the addition of 0.5 phr of Joncryl to the ORN/600/3MN composite, there was no change in tensile strength and modulus. The addition of 2 phr of Joncryl resulted in a decrease in both tensile strength and modulus compared to ORN/600/3MN.

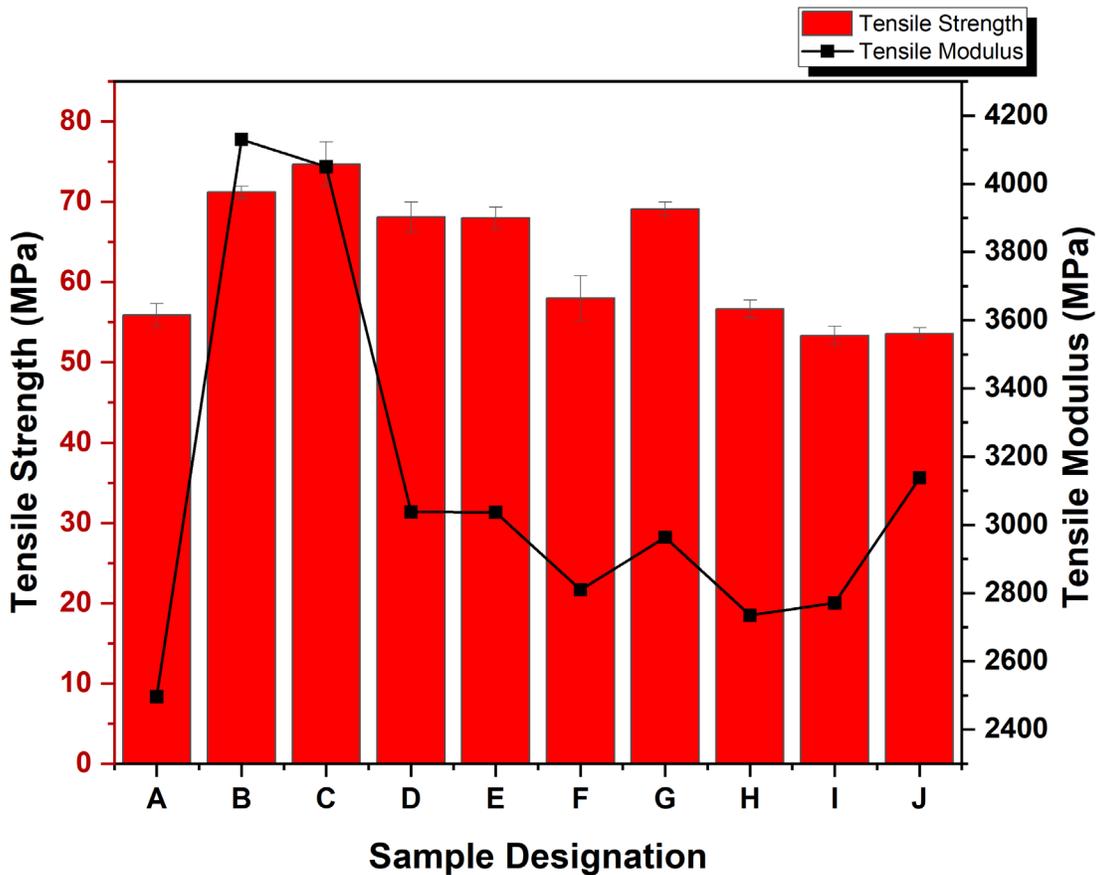


Figure 5. Tensile strength and modulus of (A) ORN, (B) ORN/900, (C) ORN/600, and (D) ORN/600/3MN, (E) ORN/600/3MN/0.5J, (F) ORN/600/3MN/2J, (G) ORN/600/5MN, (H) ORN/600/3PP, (I) ORN/600/5PP, (J) ORN/900/3PP

Summary and Next Steps

Lignin BioC produced from pyrolysis at 600 and 900 °C was used as a filler in the development of sustainable ORN biocomposites. The investigation examined the thermal, structural, surface morphology, and mechanical properties of the biocomposites with alterations in the carbonization temperature of the lignin. Additionally, ORN and PP maleated compatibilizers, and Joncyl were added to determine the effect on these properties. It was determined that the MFI reduced when BioC was added to the ORN matrix. A further decrease in MFI was observed for the 600 °C composites when compared to the 900 °C which could result from the better interfacial interaction of the functional 600 °C BioC with the polar ORN matrix. The impact strength was reduced with the addition of BioC to the matrix, however, with the addition of maleated ORN compatibilizer and Joncyl this property was enhanced. The SEM images of the fractured surface of the impact samples showed that these samples had fewer empty macro pores which reduce the number of sources of crack initiation, therefore, increasing impact strength. The flexural and tensile properties were enhanced in comparison to neat ORN. Due to these properties the developed biocomposites have the potential to be used as high-performance parts in automotive applications. The next steps for this research could include conducting flammability and electrical conductivity tests on the composites, which would determine suitable applications. Also, increasing the porosity of the composites, by altering pyrolysis temperature, could be used to reduce the density of the composites.

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