HYBRIDIZATION OF BAST NATURAL FIBERS AND SYNTHETIC FIBERS

IN THERMOPLASTIC COMPOSITES

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Hybridization of bast natural fibers and synthetic fibers in thermoplastic composites

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ABSTRACT

In recent years, the use of natural fiber-reinforced composites in more advanced applications has grown substantially. Applications of high strength require high mechanical properties. An effective method for increasing the field of application and mechanical properties is the hybridization of natural fibers with synthetic fibers. In this research, the effects of recycled carbon fiber hybridizing flax (Linum ussitatissimum L.) and hemp (Cannabis sativa L.) fibers were investigated to identify trends in mechanical properties resulting from varied weight fractions. A new high-performance composite was demonstrated for injection molding applications by hybridizing bast natural fibers and recycled carbon fibers in a polyolefin thermoplastic. After reinforcing recycled carbon fiber with flax and hemp fibers, this study showed a 10-15% increase in tensile strength. After reinforcing recycled carbon fiber with hemp fiber, a 30-35% increase in flexure strength was observed. Impact strength for hemp fiber also increased by 60% compared to recycled carbon fiber.

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DEDICATION

To my parents and my brother,

for their endless love, support and encouragement.

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1. INTRODUCTION

1.1. Natural Fibers

Emergence of polymers in the beginning of the 19th century ushered a new era of research with a new option of using the natural fibers in more diversified industries. At the same time interest in synthetic fibers, because of its superior dimensional and other properties gained popularity slowly replaced the natural fibers in different applications (Mitra, 2014). Changes in raw materials and the production of synthetic composites, however, require a large amount of energy and environmental pollution during the production and recycling of these synthetic materials (Holbery & Houston, 2006).

This has once again drawn the attention towards natural fibers due to their distinct advantages. Thus, the renewed interest in the natural fibers has resulted in numerous modifications to make it parallel and even better than synthetic fibers. Because of such enormous changes in the quality of natural fibers, they quickly emerge as a composite reinforcing material (John & Thomas, 2008).

Given the high-performance standard of composite materials in terms of durability, maintenance, and cost effectiveness, the use of natural fiber-reinforced composites as building material has enormous potential and is critical to achieving sustainability (Taj & Munawar, 2014). Though hydrophilic character of natural fibers leads to composites with weak interface, pretreatments of natural fibers is aimed at improving the adhesion between fibers and matrix. In pre-treatments, either hydroxyl groups get activated or new moieties are added that can effectively interlock with the matrix (Sanjay et al. 2016).

Natural fibers can be classified according to their origin and grouped into leaf fibers: abaca *(Musa texilis* L.), date palm (*Phoenix dactylifera* L.), pineapple (*Ananus comosus* L.), sisal, and banana; seed fibers: cotton (*Gossypium hirsutum* L.); bast fibers: flax, hemp, and jute (*Corchorus olitorius* L.), fruit fibers: coir and, oil palm (*Elaeis guineensis* Jacq.); grass fibers: sugarcane bagasse (*Saccharum officinarum* L.) and bamboo (*Bambusa vulgaris* L.), and stalk fibers: straw (cereal). The bast and leaf (hard fibers) types are the most commonly used in composite applications (John & Thomas, 2008). Commonly used plant fibers are cotton, jute, hemp, flax, and sisal. Natural plant fibers are constituted from cellulose fibers, consisting of helically wound cellulose micro fibrils, bound together by an amorphous lignin matrix. Lignin keeps the water in fibers, acts as a protection against biological attack and as a stiffener to give stem its resistance against gravity forces and wind (Swolfs, et al. 2014) Hemicellulose found in the natural fibers is believed to be a compatibilizer between cellulose and lignin (Swolfs, et al. 2014). The cell wall in a fiber is not a homogenous structure as shown in Figure 1.

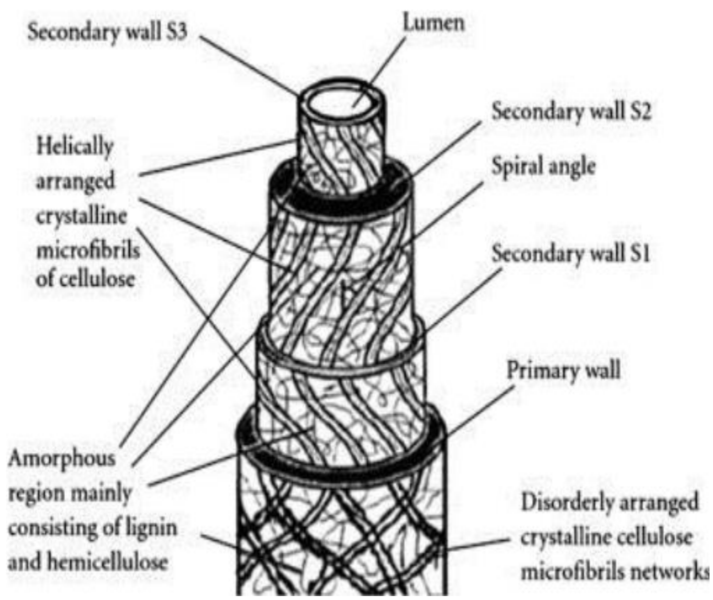

Figure 1: Structure of bio-fiber (John & Thomas, 2008).

Each fiber has a complex, layered structure consisting of a thin primary wall that is the first layer deposited around a secondary wall during cell growth. The secondary wall consists three layers and the mechanical properties of the fiber are determined by the thick middle layer. The middle layer consists of a series of helically-bound cellular micro fibrils formed from long-linear chains of cellulose (John & Thomas, 2008). The angle between the fiber axis and the micro fibrils is called the micro fibrillary angle. The characteristic value of micro fibrillary angle varies from one fiber to another. These micro fibrils typically have a diameter of about 10 to 30 nm and consist of 30–100 cellulose molecules in extended chain conformation and provide mechanical fiber resistance (John & Thomas, 2008).

Several factors such as variety, retting degree, climate, and harvest affect the properties of natural fibers. It becomes necessary to know the mechanical, physical, and chemical properties of natural fibers to understand the properties of natural fiber-reinforced materials. Compared to other natural fibers, flax fibers are relatively strong. The tensile strength of the elementary fibers is 1500 MPa and 800 MPa is observed for technical fibers at a clamp length of 3 mm (Baley et al. 2006). Intense retting process separates the bast fibers into elementary fibers which are several centimeters long. Technical fibers refer to the fibers which are used for industrial applications such as automotive sector rather than household applications such as carpets (Andersons, et al. 2011).

Lamy and Baley (2000) investigated the modulus of flax fibers. Young's modulus is the measure of elasticity which is equal to the ratio of stress acting on a substance to the strain produced. The elementary fiber modulus depends on the fiber diameter and ranges from 39 GPa for approximately 35 μm diameter fibers to 78 GPa for 5 μm diameter fibers. This variation is related to the variation between fibers with different diameters in relative lumen size. After numerous traction tests on single flax fibers, an average Young's module of 54 GPa was observed and the results are within the range of modules measured on technical fibers (Lamy & Baley, 2000). The mechanical, chemical and physical properties of plant fibers are strongly dependent on climate, location, weather conditions and soil characteristics. These properties are also affected during the processing of the fiber such as retting, bleaching and spinning (Bharath & Basavarajappa, 2016). Natural fibers have relatively high strength, high stiffness, and low density (Nunna et al. 2012).

The characteristic value for soft wood Kraft fibers and flax has been found close to the value of E-glass fibers. E-glass is alumino-borosilicate glass with less than 1% alkaline oxides, which is used primarily for glass-reinforced plastics. Different mechanical properties can be incorporated in natural fibers during processing. The fiber properties and structure are influenced by several conditions and varies with area of growth, its climate and age of the plant (Arbelaiz et al. 2005). The elastic modulus of the bulk natural fibers such as wood is about 10 GPa. Cellulose fibers with moduli up to 40 GPa can be separated from wood by chemical pulping process. Such fibers can be further subdivided into micro fibrils within elastic modulus of 70 GPa. Theoretical calculations of elastic moduli of a single cellulose chain have been given values up to 250 GPa. However, no technology is available to separate these from micro fibrils (Arbelaiz et al. 2005).

Natural fiber's tensile strength depends on the specimen's test length, which is of major importance in terms of enhancing efficiency. Tensile strength of flax fiber has been reported to be significantly more dependent on the fiber's length-to-diameter ratio than other natural fibers. The tensile strength of pineapple fiber is less dependent on the length compared to this, while the variation of the measured values for both fibers is within the standard deviation range. The properties of flax fiber are controlled by the molecular fine structure of the fiber which is affected by growing conditions and the post-harvest fiber processing techniques (Monteiro et al. 2010).

In the automotive and packaging industries (e.g. egg boxes) there is an emergence in the use of natural fibers as reinforcements in technical applications. For years, textile waste has been used in the automotive industry to strengthen plastics used in cars. Mercedes Benz Company ®, planned a K-car-series where the' K' stands for' herb' in German. For these cars, renewable fibers such as flax and hemp have been used. Because of their specific properties (Nunna et al., 2012) and components such as door panels (molded wood, natural fiber moldings and laminated panels) and automotive roofs (composites made of natural fiber-fleece-flax-with epoxy resins or polyurethane), Ramie (Bharath & Basavarajappa, 2016) fibers were also examined (Boehmeria level L.). Another example of industrial applications of natural fibers is the use of flax fibers in car disk breaks to replace asbestos fiber, a mineral fiber (Kalia et al., 2009).

1.2. Recycled-Carbon Fibers

Carbon fiber and its enhanced polymers are key elements in the manufacture of advanced composite materials for aerospace, automotive, plastic and cement applications. Carbon fiber remains, for many industries, a premium and expensive material and out of reach. Recycling and mixing the virgin fibers with thermoplastic therefore offers an affordable alternative. Virgin fiber is a cellulose fiber directly derived from newly pulped or unused trees and other plants. Approximately 90% of the carbon fibers produced are made of polyacrylonitrile (PAN). The remaining 10% is made of pitch radius or petroleum. All these materials are organic polymers, characterized by long strings of carbon atoms binding together molecules. Recycled carbon fiber composites in several industries have become increasingly popular. In everything from aircraft to wind turbines and cars, super light and strong material is used. Recycled carbon fibers are used as sheet molding compounds and as recycled materials in load-bearing shell structures for smaller, non-load-bearing components (Pickering, 2004). It can be observed that the use of recycled carbon fiber has the potential to reduce fiber reinforcement costs by about 40% (Pimenta & Pinho, 2011). Moreover, recycled carbon fiber has less than 10% of the virgin carbon fiber's global warming potential. Carbon fiber reclamation initially involves metal removal and cutting to sizes suitable for down-steam processing of large composite structures. Figure 2 shows laminate shredding and prepregs with shredder capable of producing 4 ton/h (Holmes, 2018). Prepreg refers to a fibrous material pre-impregnated with a synthetic resin, used in the production of reinforced plastics. Fiber recovery is achieved by a modified pyrolysis process in which the resin is burnt off as shown in Figure 3 and Figure 4. Pyrolysis is the thermochemical conversion process which heats the materials to high temperature, typically 400-600℃, in the absence of oxygen.

Figure 2: Carbon fiber reclamation by shredding (Holmes, 2018).

Figure 3: Fiber recovery through pyrolysis process (Holmes, 2018).

Figure 4: Feeding reclaimed carbon fiber to the furnace (Holmes, 2018)

Carbon fiber recovery has grown from below 200 tons in 2012 to almost 1100 tons in 2015 worldwide (Holmes, 2018). Feedstock is primarily unused prepreg, but it is also suitable for cured waste production and end of life materials. To convert recycled carbon fiber, a carding line is used as shown in Figure 5 (Holmes, 2018) which is capable of providing up to 250 ton/year of carbon fiber.

Figure 5: Carbon fiber carding line (Holmes, 2018).

Further, the fiber is chopped at the milling center with the length depending upon the application. Possible savings with recycled carbon fiber is significant as virgin fiber products bare typically between $40-55/kg while recycled materials are $15-25/kg. It can be observed that using recycled carbon fiber has the potential to reduce the cost of the fiber reinforcement by around 40% (Pimenta & Pinho, 2011). Further advantage is that recycled carbon fiber has lower global warming potential of virgin carbon fiber as 10%. According to ELG Carbon Fiber, the pyrolysis process of a recycled material maintains 90% of the carbon fibers tensile strength, without change in modulus. The following Table 1 shows the typical properties of a recycled carbon fiber reinforced PA66 polymer (base polymer and no coupling agents) (Holmes, 2018).

Table 1: Properties of recycled carbon fiber composite†

Carbon content	TS	TM	FS	FM	IS
	MPa	GPa	MPa	GPa	kJ/m^2
30%	177.9	23.7	288.8	23.1	5.4
40%	217.1	26.5	348.0	25.9	5.9

† TS: Tensile strength, TM: Tensile modulus, FS: Flexure strength, FM: Flexure modulus, IS: Impact strength

1.3. Factors Affecting Composite Properties

Several factors like manufacturing defects, bonding properties between fiber and matrix and the durability of natural fiber reinforced composites (NFRC) exposed to different harsh environments, contribute to the low performance of the bio composites. Hence it is essential to study these factors and further investigate the root cause of low or high mechanical trends. These factors also affect the strength and stiffness of the composite and hence need to be studied thoroughly. In this study factors like orientation of fiber, interfacial bonding, treatment of fibers and effect of environmental conditions, have been studied and discussed.

1.3.1. Orientation of fiber

Good interfacial bonding between the fiber and matrix is required for optimum performance in composite materials. Most of the NFRCs are made of short fibers, and therefore fiber orientation is necessary in order to control their mechanical properties (Kin-tak Lau et al. 2018). Fiber orientation is highly affected by the resin flowing pattern, viscosity of resin, and shape of products, (molding cavity design) as shown in Figure 6 (Kin tak Lau et al. 2018).

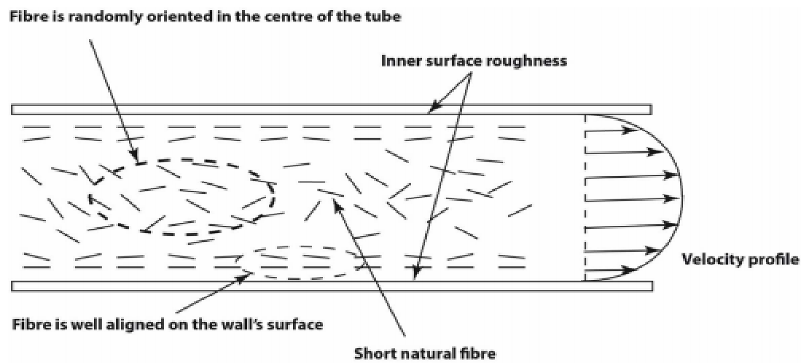

Figure 6: Influence of flow on short fiber orientation inside a barrel (Kin tak Lau et al., 2018).

The surface roughness of an inner wall of a barrel is a crucial factor as the resin flow velocity would increase from the wall towards the center. It has been claimed that aspect ratio (L/D), where L is the length of fiber and D is the diameter of the fiber is an appropriate parameter to determine the properties of composites (Kushwaha & Kumar, 2009). It reflects the total surface area used to bond with the matrix, and thus enhance the interfacial bonding strength. The failure of the NFRC composites is highly sensitive to the size of fiber and the cross section of a fiber. A natural fiber is not uniform along its fiber length and may cause inaccuracy of measurement (Kin tak Lau et al. 2018).

The effect of weaving patterns and random orientation on the mechanical properties of banana, kenaf, and banana/ kenaf fiber-reinforced hybrid polyester composites were found (Alavudeen et al. 2015). They prepared composites using hand lay-up method with two different weaving patterns, namely plain and twill type. Twill is a type of textile weaved with a pattern of diagonal parallel ribs. The plain weaving pattern showed higher strength compared with the twill type, and random orientation composites. This proved that the higher strength is due to the uniform load transferring ability and better inter-locking capability between the constituents in plain-type composites (Alavudeen et al. 2015).

1.3.2. Fiber volume or weight fraction

Fiber volume or weight fraction is the measure of volume or weight proportion of fibers in a hybrid composite. When polymer composites are manufactured, fibers are impregnated with resin. The amount of resin to fiber ratio is calculated by the geometric organization of the fibers, which affects the amount of resin that can enter the composite (Tse et al, 2018). The impregnation around the fibers depends heavily on the fiber’s orientation and the fiber’s architecture. The composite's geometric analysis can be seen in the composite's cross-section. Vacuums are often formed throughout the manufacturing process in a composite structure and are calculated into the composite's total fiber volume fraction. The fiber reinforcement fraction is very important in determining a composite's overall mechanical properties. A higher fraction of the fiber volume typically results in better composite mechanical properties than lower fractions of weight (Nunna et al. 2012).

Many researchers studied the behavior of hybrid composites by varying the fiber volume or weight fraction of fibers and because of this variation observed positive and negative effects on mechanical properties. Researchers conducted an investigative study on hybrid composites made from oil palm and reinforced polyester glass fibers to obtain mechanical and physical properties at different fiber content ratios. The properties of the tensile are increased up to 45% wt. Of the loading of fiber. If the fiber content is further increased due to agglomerate formation, which acts as barriers to stress transfer, a declining trend has been observed. The tensile properties are enhanced with the fraction of the glass fiber volume, but a reverse trend has been observed by increasing the content of oil palm fibers due to poor matrix-fiber bonding. Flexure and impact properties have been observed to increase up to 35 percent wt. Of the loading of fiber. In addition, there was a decline in the trend due to poor fiber dispersion in the matrix phase (Khalil et al. 2007).

A recent study studied the tensile and flexural properties of polyester-reinforced hybrid composites with glass and jute fibers. It was concluded that as the volume fraction of the glass fibers increased, the tensile strength of the hybrid composites increased. For the hybrid composite with a larger fraction of jute fiber volume, the Young's module was higher than the fraction of glass fiber volume (Vara Prasad, 2011).

1.3.3. Interfacial bonding

The most essential parameter for optimum performance is the bonding between the fiber and polymer matrix in the composite. Pendant hydroxyl and polar fiber groups result in extremely high fiber absorption of moisture resulting in weak interfacial bonding between the fiber polymers and the hydrophobic matrix. The bonding between the matrix and the fibers becomes extremely important when a single fiber fractures before the composite ultimately fails (Mohanty et al. 2005).

The strength of the bond determines how micro cracks propagate at the ends of the fiber. The cracks do not propagate along the length of the fiber when there is a strong bond between the fibers and the matrix. This means that there is still the transfer of load from the matrix and fiber and the fiber remains a viable reinforcement. A strong bond also leads to higher transverse strengths and better environmental performance such as water resistance (Mohanty et al. 2005).

Poor interfacial bonding between matrix and fiber is a common problem when we work with natural fibers. Natural fibers being hydrophilic in nature often suffer from high moisture absorption while many polymers are hydrophobic in nature which creates a high degree of incompatibility between fiber and resin (Kalia et al. 2009; Agarwal et al. 2017). In addition to dissimilarities in polarity between matrix and fiber, the surface quality of natural fibers also presents adhesion issues. A lot of research work is being carried out to improve the bond strength. Many researchers have chosen different techniques to improve the bond strength, which involve the altering of the fiber's surface to provide more bonding sites for the polymer through chemical methods (Kalia et al. 2009).

1.3.4. Effect of environmental conditions

The various environmental conditions affect the mechanical properties of hybrid composites considerably. This fact has led many researchers to study the effects on the behavior of hybrid composites of different environmental conditions. In a recent study, the behavior of polypropylene hybrid composites enhanced by hemp and glass fiber was studied. The hybrid composites were exposed to water at 40°C, 60°C and 80°C for certain periods by different weight fractions of glass fibers (Panthapulakkal & Sain, 2007).

It was observed that the saturation points of absorption of moisture were reached prematurely with an increase in volume fraction of glass fibers. Due to the leaching of the materials from the natural fibers at high temperatures, the maximum moisture content was observed at 80°C. The composite's tensile behavior was studied before and after water exposure. It was observed that due to the loss of interfacial bonding between fibers and matrix, the tensile properties of the composite material decreased. The dried samples were better than the wet ones (Nunna et al. 2012).

A study showed variations in the flexural and compressive properties of unsaturated polyester hybrid composites reinforced by jute and glass fiber. These hybrid composites have been exposed for three weeks to three aqueous environmental conditions such as distilled water, sea water and acidic water at 6.8 pH. For each specimen, the diffusion coefficient was calculated and higher for distilled water followed by acidic and marine water. With increasing immersion time, the strength of the composites decreased. Furthermore, the hybrid composite's flexural and compressive strengths were greater than the natural fiber composites. This was due to the increased strength of hydrophobic glass fibers in nature (Zamri et al. 2012).

1.3.5. Treatment of fibers

The properties in (NFRC) depend mainly on the combination of the selected polymer matrix and their adhesion. The adhesion of the fiber to the resin depends on the nature and composition of the fiber (lignin, cellulose, and hemi-cellulose). By treating the fiber with certain appropriate coupling agents, the bonding between the fiber and the resin can be enhanced. Poor component adhesion and interaction causes uneven distribution of stress in the composite. Therefore, it is necessary to treat the fiber with chemical agents to obtain desirable properties (Nunna et al. 2012). Hydroxyl group, which is present in the natural fiber, makes the fibers as hydrophilic. Therefore, the combination of resin with hydrophobic nature should be necessary to obtain a material, with desired characteristics, which in turn will enhance the interfacial bond between the fiber and the matrix (Pimenta & Pinho, 2011).

The presence of lignin, cellulose and hemicellulose in the plant fiber will increase the rate of water absorption, which in turn will degrade the composite properties. Fiber surface modification is a must to avoid fiber wettability and to increase the strength of the bond between other composites.

1.4. Overview of Hybridized Composites

Composite materials are gaining more preference as a choice of material in several industrial applications. They help in achieving the desired mechanical properties by combining different materials in a skillful way. In general, they possess high strength-to-weight and high stiffness-to-weight ratios which makes them useful in aerospace and automotive applications (Mallik, 2007).

Using natural fibers as reinforcing agents, composite materials offer significant performance gains. Due to a growing demand for bio-sustainability and a reduction in our current carbon footprint, the research and development of renewable materials has increased (Dittenber & Gangarao, 2012). Bio-sustainability is the quality of not being harmful to the environment or depleting natural resources, thereby supporting long term ecological balance (Ben-eli, et al. 2012).

Natural fiber composites are found to have higher strengths than composites of wood and a few plastics, which will improve their ability to compete in future as materials of choice. Natural fibers lack thermal stability, strength degradation, water absorption and poor impact properties despite their suitable properties. In order to overcome these properties, researchers focused on studying the effect of mechanical properties due to hybridization of natural fibers with synthetic fibers (Chandramohan & Marimuthu, 2011).

Hybrid composites consist of an amalgamation of two or more fibers in a polymer matrix. Hybrid composites have comprehensive applications in the engineering field due to low cost, high strength to weight ratio and ease of manufacturing (Safri et al. 2018). A different blend of mechanical properties such as stiffness, ductility, and strength can be achieved through hybridization which cannot be achieved by single fiber reinforced composites. A natural fiber reinforced hybrid composite possess a good strength and stiffness value near to glass fiber reinforced composites (Pickering et al. 2016).

Prasad et al. (2016) examined the effect of coir (*Cocus nucifera* L) fiber addition along with banana (*Musa spp*.)-fiber in low-density polyethylene (LDPE) to develop cost-effective and high-performance composite material. Through their research they were able to analyze the effect of hybridization through the mechanical properties (tensile, flexural, and impact), thermal stability, morphological behavior, and water absorption behavior. Additionally, scanning electron microscopy (SEM) studies were also carried out on the tensile fractured surface for all composite samples to examine the fracture behavior of the composite samples. The results showed that the incorporation of coir fiber into the banana-fiber composites of up to 50% by weight led to enhancement of the mechanical properties and thermal stability and to reduction of the water absorption capacity of the banana fiber/LDPE composites (Prasad et al. 2016).

Meenakshi et al. (2013) examined natural fibers like flax (*Linum ussitatissimum* L.), sisal (*Agave sisalana Perrine ex. Engelm)*, kenaf (*Hibiscus cannabinus* L.), and aloe vera (*Aloe barbadensis*, Mill.) which were combined with glass fiber in an epoxy matrix to form a hybrid-fiber-reinforced composite laminate. Their mechanical properties such as tensile, flexural and impact strength were compared. Kenaf/glass hybrid samples had mechanical strength values 40 to 50% higher than that of other types of samples tested. Tse et al. (2018) examined the flexure behavior of short recycled carbon fibers (rCF) which were mixed with flax fiber in the PLA matrix. Experimental data showed that the flexural properties increased with higher rCF content, with the maximum being a flexural modulus of approximately 14 GPa and flexural strength of 203 MPa. Furthermore, rCF showed poor matrix adhesion with PLA, which indicate a poor interfacial bonding between the matrix and fibers. Bachmann et al. (2018) examined monolithic nonwoven laminates containing flax fibers and recycled carbon fibers produced with a fiber volume fraction of 30 percent compared to pure flax and rCF reinforcement references. By combining rCF and flax fiber in a hybrid nonwoven, three-point bending tests showed a potential increase in flexural mechanical properties.

In the aerospace and automotive industries, carbon fiber has already gained substantial acceptance. Carbon fiber is an expensive material and out of reach for most mainstream vehicle manufacturers, but recycling and mixing carbon fibers with thermoplastic is an affordable alternative. It is environmentally and socially desirable to reduce waste and reuse materials with high-embedded energy and recycle them in an energy-efficient manner. Although recycled carbon fiber has been reclaimed from waste, it retains many of its inherent advanced properties. Moreover, the price of carbon fiber recycled is at least half the price of pure carbon fiber (Azwa et al. 2013).

There are many different possible fiber combinations for high stiffness applications. Bachmann et al. (2018) examined the hybrid effect for flax and rCF with thermoset resin. There is lack of research for the impact of hybrid effect on thermoplastic composites which use rCF blended with natural fibers. In this study, a combination of flax and hemp (*Cannabis sativa* L.) fibers were hybridized with recycled carbon fiber keeping the polypropylene matrix constant. As an initial investigation this study only considered a few combinations to observe potential trends. The hybridization of flax, hemp and recycled carbon fiber offers a good potential for developing high stiffness composites for various automotive applications while simultaneously incorporating bio-based materials into a product.

2. OBJECTIVES

In this study, flax, hemp, and recycled-carbon fibers were used to reinforce a polypropylene resin matrix to produce a series of hybrid fiber-reinforced composite materials. Using a hybrid natural/synthetic fiber system, it is possible to achieve significant savings in terms of weight and cost while improving mechanical properties for specific applications such as aerospace and automotive industries.

Through hybridizing fibers, the scope of this research intends to accomplish the following:

- Combine flax and hemp fibers each with recycled carbon fibers at varying fiber weight fractions to determine mechanical trends.
- To prove reproducibility of mechanical property results through hybridization.
- To perform different mechanical tests on the bio-composites to obtain different mechanical properties such as strength and modulus which are further used in the design of composite structure.
- To study the surface morphology of the hybrid composites using Scanning Electron Microscopy (SEM) to investigate failure mechanism to ensure reliability of the product.
- To study the void formation using Micro-CT and to investigate the quality of the composite structure which has a strong effect on the mechanical properties.
- To boost the use of recycled carbon fiber reinforced composites, focusing on light weighting and improved sustainability for various applications.

3. MATERIALS AND METHODS

The materials used in this study for developing hybridized composites were maleic anhydride, recycled-carbon, flax, and hemp fibers in a polypropylene matrix. The materials were processed into a composite by using a Leistriz co-rotating twin-screw extruder Type Mic 18/GI-40D and pelletized with a Scheer Bay pelletizer model BT25. Once the materials were processed they were injected using a Technoplas hydraulic injection molder (Technoplas Inc, SIM 5050A, USA). After injection, a wide range of mechanical testing was performed to determine the mechanical abilities of the hybrid composites. Further analysis of void formation was also done using SEM and Micro-CT.

3.1. Fibers Description

The recycled carbon fibers used in this study were manufactured by ELG Carbon Fiber Ltd. The recycled carbon fibers came in already chopped and were 6.3-mm in length and 20-μm in diameter. A published fiber density of 1.40 g/cm^3 was used (Sanjay et al., 2016). The glass transition temperature was 151℃.

Flax and hemp fibers used in this study were manufactured and provided by Sunstrand LLC. Both fibers came in already chopped and were 6.35-mm in length and 20-μm in diameter. A published fiber density of 1.50 g/cm^3 and 1.48 g/cm^3 for flax and hemp, respectively was used (Sanjay et al., 2016). Flax fibers have a tensile modulus of 50 GPa and a tensile strength of 500 MPa (Kin tak Lau et al., 2018). Hemp fibers have a tensile modulus of 30 GPa and a tensile strength of 300 MPa (Kin tak Lau et al., 2018).

3.2. Polymers Used

The polymer used throughout this study was polypropylene PP78151E and it was supplied by ExxonMobil Chemical. The density of polypropylene used was 0.90 g/cm^3. The flexure modulus and tensile strength was 2.068 GPa and 34.40 MPa, respectively. Polybond 3200 was used as the maleic anhydride compatibilizer. The density was 0.91 g/cm^3 and melt flow rate was 115 g/10 min.

3.3. Materials Processing

Flax, hemp and recycled-carbon fibers were compounded with polypropylene (PP) and a coupling agent, maleic anhydride grafted PP (MA-g-PP/MAPP), using a co-rotating twin screw extruder and then subsequently pelletized with a pelletizer. Maleic anhydride surface treatments were tested at 2% weight concentrations. The mechanical properties of the materials were analyzed in accordance with ASTM standards. The PP was dried for 24 h at 80°C. The moisture content of the PP was tested using a MAX 4000XL moisture analyzer and showed 0.001% moisture content prior to processing operations. Flax, hemp and recycled carbon fibers were dried at 80°C for 12 h and moisture content was below 0.1%. After drying, the materials were mixed according to the material weight fractions with formulations used shown in Table 2 and Table 3.

Table 2: Test matrix for flax and hemp fiber reinforced composites†.

Sample	Flax or hemp fiber	rCF-chopped fiber	Matrix-PP	Treatment-MAPP
	------------------------------	%wt.	------------------------------	
1	20	10	70	None
2	10	20	70	None
3	20	10	68	2
4	10	20	68	2

†rCF: Recycled carbon fiber, PP: Polypropylene, MAPP: Maleic anhydride

Table 3: Samples with constant fiber % wt. and matrix PP†.

Sample	Fiber	Matrix-PP
	--------------%wt.---------------	
1	Flax -30	70
2	Hemp-30	70
3	rCF-30	70

†rCF: Recycled carbon fiber; PP: Polypropylene

The materials were compounded using a Leistriz co-rotating twin-screw extruder Type Mic18/Gl-40D as shown in Figure 7 and pelletized with a SCHEER BAY pelletizer model BT25 (Bay Plastics Machinery, Michigan, USA). The compounding of materials was carried out at 250 rpm screw speed with a barrel profile temperature from nozzle to feed throat of 149, 171, 177, 182, 188, 188, and 188°C for each section respectively.

Figure 7: Twin screw extruder.

Test samples were dried again and then injected into a mold using a 50-ton Technoplas hydraulic injection molder model SIM-5080 as shown in Figure 8. The injection molding processing conditions for samples were an injection pressure of 300 psi with a temperature profile from nozzle to feed throat of 193, 199, 199, 186, and 188°C, for each section respectively. A time of 25 s was given for the specimens to cool under pressure before opening the mold.

Figure 8: Injection molder.

3.4. Tensile Test

The tensile properties of the samples, strength and modulus were measured using an Instron universal tester model 5567 as shown in Figure 9. Tensile testing was performed according to American Society for Testing and Materials (ASTM D638), using an MTS Extensometer model 632.11B-20. Five specimens were tested for each test sample. The specimens were tested in tension at a crosshead speed of 5 mm/min with a 2 kN load cell. They were 3.92-mm in thickness and 9.96-mm in width.

Figure 9: Tensile testing setup.

3.5. Flexure Test

Flexural properties of the samples, strength and modulus were tested using a three-point bend testing, as specified in ASTM D790, using an Instron 5567 load frame as shown in Figure 10. The speed of the crosshead was calculated according to the guidelines specified in the standard, which resulted in 1.36 mm/min. Five specimens were tested for each test sample. A load cell of 2kN was used in the test setup. The support span used for the testing was 51-mm in length. The specimens were 3.2-mm in thickness and 12.7-mm in width.

Figure 10: Flexure test set up.

3.6. Impact Test

The composite materials were tested for their impact properties using a Tinius Olsen Model Impact 104 Izod impact tester as shown in Figure 11 with a pendulum energy of 2.7548 J. The impact testing was carried out in accordance with ASTM standard D256. A minimum of five specimens were tested for each sample. The samples were 10.93-mm in width and 3.18-mm in depth.

Figure 11: Izod impact tester**.**

3.7. Scanning Electron Microscope (SEM)

Fractured-test specimens were mounted on cylindrical aluminum mounts with colloidal silver paste (Structure Probe Inc., West Chester PA, USA) for view of the fractured surface and then coated with a conductive layer of gold using a Cressington 108 auto sputter coater (Ted Pella Inc., Redding CA, USA). Figure 12 shows the images that were obtained at an accelerating voltage of 15 kV using a JEOL JSM-6490LV scanning electron microscope (JEOL USA, Peabody MA, USA).

Figure 12: Scanning electron microscope **(**JEOL JSM-6490LV).

3.8. Micro-CT

The General Electric (GE) v|tome|x s micro CT as shown in Figure 13 enables nondestructive evaluation of internal structure. Micro-CT refers to micro computed tomography. A 240kV micro focus X-ray computed tomography system with an additional 180kV submicron X-ray tube. A high-contrast digital flat panel detector was used for the greatest possible versatility. Void content and fiber concentrations of the fractured surface was observed using the micro-CT setup.

Figure 13: GE v|tome|x s micro-CT set up.

3.9. Statistical Analysis

An analysis of variance (ANOVA) was carried out to study the effect of interaction between treatment and fiber effects. The experimental design was a completely randomized design with four replicates and two runs. Mean separation test was used which was *F*-protected Least Significant Difference (LSD) at 95% confidence. SAS Version 9.4 was used to perform the tests.

4. RESULTS AND DISCUSSION

4.1. Tensile Test Results

The (ANOVA) carried out for fibers and treatments and the interaction between them was significant ($P \leq 0.05$) for the main effects and the interaction. Recycled-carbon fiber has significant difference when added more than 10% in flax and hemp fiber composites. Table 4, Figure 14, and Figure 15 shows the comparison and interaction between the mean values of tensile strengths of all fibers and treatments. The reinforcement of recycled-carbon fiber in flax fiber improved the tensile strength by 10-15%. A significant 25-30% increase in the tensile strength was observed after the addition of the compatibilizer (Table 4, Figure 15). The reinforcement of recycled carbon fiber in hemp fiber improved the tensile strength by 5-15%. A significant 30-35% increase in the tensile strength of hemp fiber was observed after the addition of the compatibilizer (Table 4, Figure 15). Recycled carbon fiber shows the highest tensile strength than both flax and hemp fibers when blended with the resin. Arbelaiz et al. (2005) conducted some study where different fibers such as flax, jute and glass were compounded with polypropylene at different weight fractions. Similarly, to our results, tensile strength decreased as the amount of flax fiber bundles was increased, this reduction being more drastic when MAPP modifier in the matrix was not used. MAPP-modified composites showed better mechanical properties than the unmodified ones since MAPP maleic anhydride group can bond with both flax and glass fibers, resulting in improved interfacial adhesion between matrix and both types of fibers. Wong et al., (2012) showed a study where recycled carbon fiber composites were studied and tested. The study shows similar test results when compared with the current study conducted. The reinforcement of recycled-carbon fiber in the polypropylene matrix has increased the tensile and flexure properties significantly.

Table 4: Mean tensile strength for flax and hemp fiber reinforced composite†

Sample	Flax	Hemp	rCF
	----------------------------MPa-----------------------------		
1	29.97 ± 0.52	30.24 ± 0.50	-
2	32.93 ± 0.38	32.13 ± 0.48	-
3	41.02 ± 0.29	41.04 ± 0.30	-
4	45.22 ± 0.66	47.50 ± 0.33	-
5	29.50 ± 0.51	29.93 ± 0.96	44.62 ± 0.66
LSD (0.05)		0.647	

† a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP 70%.

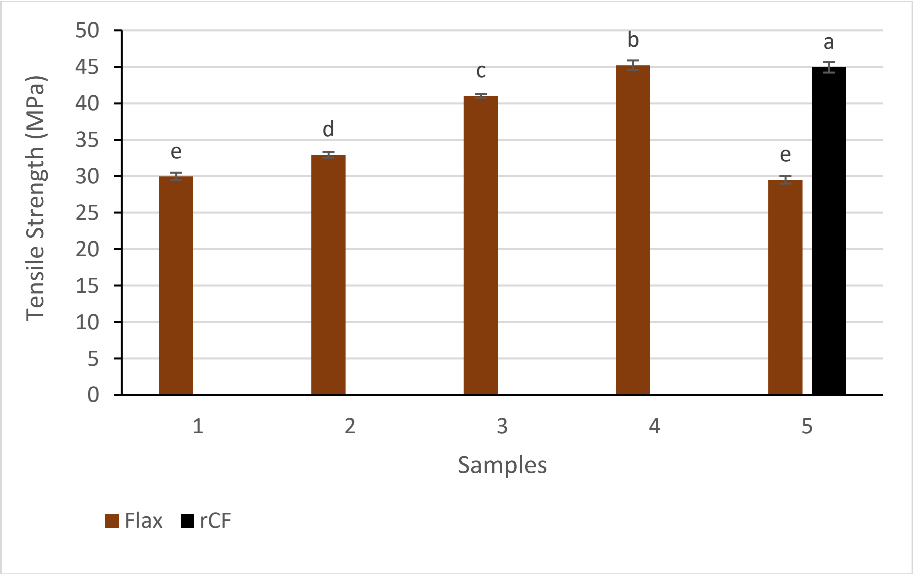

Figure 14: Comparison of the tensile strength for flax and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP 70%.

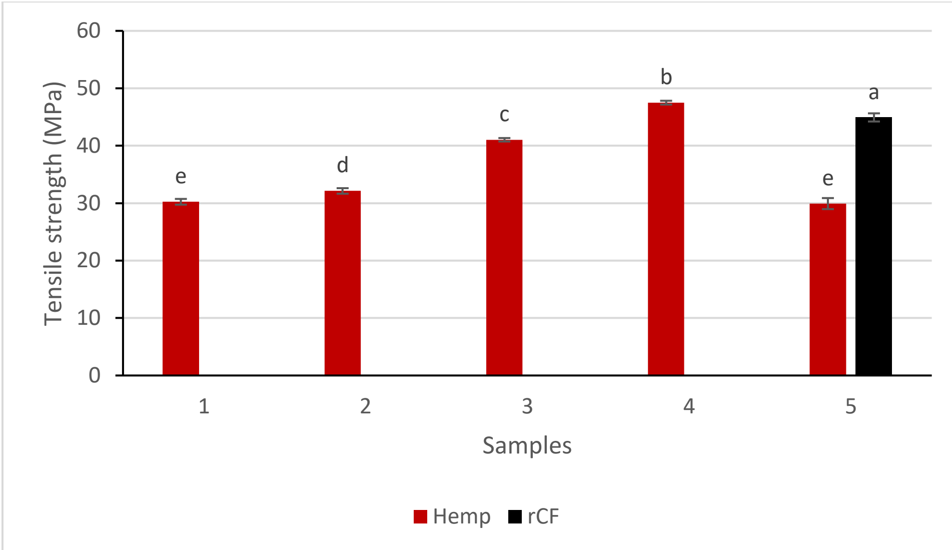

Figure 15: Comparison of the tensile strength for hemp and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, (e) Sample 5-fiber 30% + PP 70%.

For tensile modulus, the ANOVA indicated a significant effect of fiber, treatment and their interaction ($P \leq 0.05$). Recycled-carbon fiber when hybridized with flax and hemp fiber composites don't show significant difference when treated with compatibilizer. Table 5, Figure 16, and Figure 17 shows the comparison and interaction between the mean values of tensile modulus of all the fibers. The reinforcement of recycled carbon fiber in flax fiber has improved the tensile modulus by 10-15%. A significant 30-35% increase in the tensile modulus can be observed after the addition of the compatibilizer (Table 5, Figure 16). A significant 10-15% increase in the tensile modulus of hemp fiber can be observed after the addition of the compatibilizer (Table 5, Figure 17). Recycled carbon fiber shows the highest tensile modulus than both flax and hemp fibers. The modulus observed a decreased trend when compared to the recycled carbon fiber. The current study also indicated similar trends (Etaati, et al., 2014). MAPP-modified hybrid composites showed higher modulus than unmodified hybrid composite ones (Arbelaiz et al., 2005).

Table 5: Mean tensile modulus for flax and hemp fiber reinforced composite†

Samples	Flax	Hemp	rCF
	………………GPa………………		
1	4.97 ± 0.59	5.19 ± 0.32	-
2	6.80 ± 0.27	6.88 ± 0.37	-
3	5.6 ± 0.20	5.27 ± 0.05	-
4	6.93 ± 0.35	8.02 ± 0.27	-
5	6.71 ± 1.42	9.85 ± 0.25	13.81 ± 1.02
LSD (0.05)		0.533	

† a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP 70%.

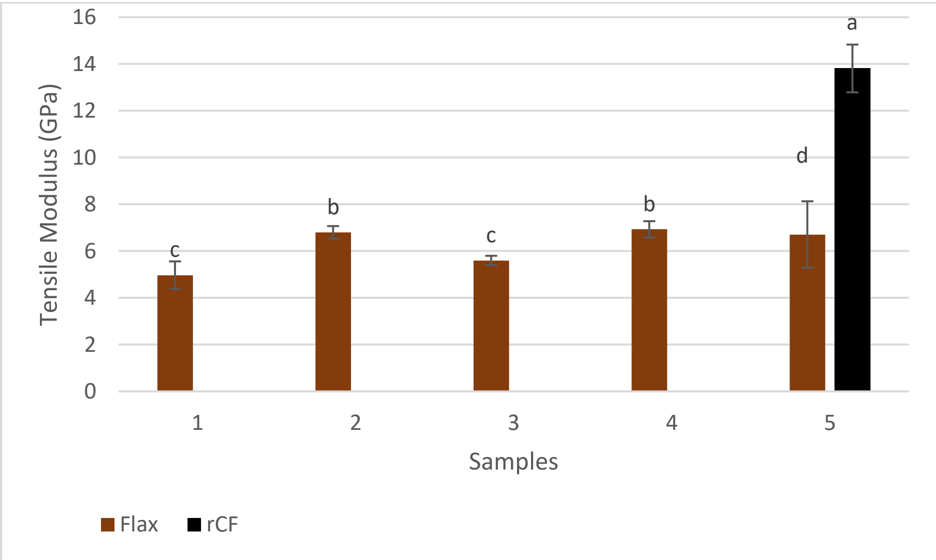

Figure 16: Comparison of the tensile modulus for flax and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5-fiber 30% + PP 70%.

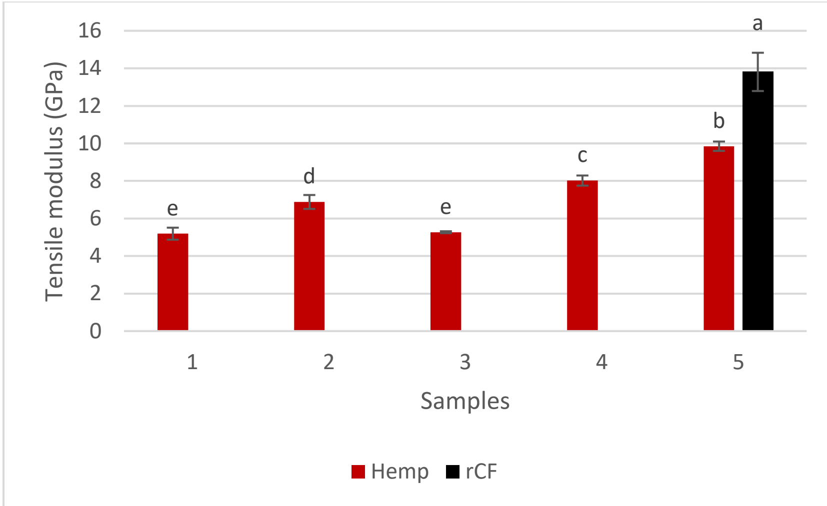

Figure 17: Comparison of the tensile modulus for hemp and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5-fiber 30% + PP 70%.

To support these results a SEM study was conducted. The following images portray the fiber polymer bonding between different fibers which help to study the characteristics and nature of the fibers. Figure 18 shows the comparison of SEM for all the fibers and composites. It can be observed that there is poor adhesion between the interface of flax fiber and polypropylene resin in Figure 18a. It can also be observed that polymer particles are stuck around some parts of the fiber, which creates a stress concentration leading to low tensile modulus. Tensile strength and modulus values for flax fiber composite are lower as compared with hemp and recycled-carbon fiber. Some voids can be observed in the image (Fig. 18a). Though fiber pullout can be spotted in Figure 18b there still exists a strong adhesion between the interface of recycled-carbon fiber and the polypropylene resin. This tends to improve the tensile strength for the composites. This combination has no voids which supports the fact of high mechanical properties. A strong adhesion between the fiber and the resin can be observed in the top left corner of Figure 18c, which is an evidence why the bond is strong. It can be observed that there are no polypropylene particles stuck on the hemp fibers which reduces the stress concentration giving it high strength.

The tensile strength is high and after the addition of the compatibilizer it can be observed that there is a remarkable increase in the tensile properties. Wong et al. (2012) studied SEM for flax fiber blended with polypropylene resin. The results showed a clean fiber surface indicating poor wettability and lack of adhesion, which agreed with the tensile results. It was also observed that fibers were unevenly distributed throughout the matrix due to differences in characteristics between flax fiber and the matrix.

This current study also shows similar results and agrees with the strength and modulus values of the composites (Wong et al., 2012). By comparing with the specimen without the coupling agent, a significant difference in fracture morphology was observed (Wong et al., 2012). At high magnification, the fiber surfaces appeared rough and surrounded by a layer of polymer. All these indicated that a good adhesion was achieved. Degree of polymer coverage on the pulled-out fibers and their adhesion with the host matrix depends on the coupling agent type and loading (Wong et al., 2012). Plastic deformation of the host matrix was also very different from that of the sample without coupling agent and again the appearance varied with the type of coupling agent. Each coupling agent exhibited a somewhat different improvement in strength (Li et al., 2007).

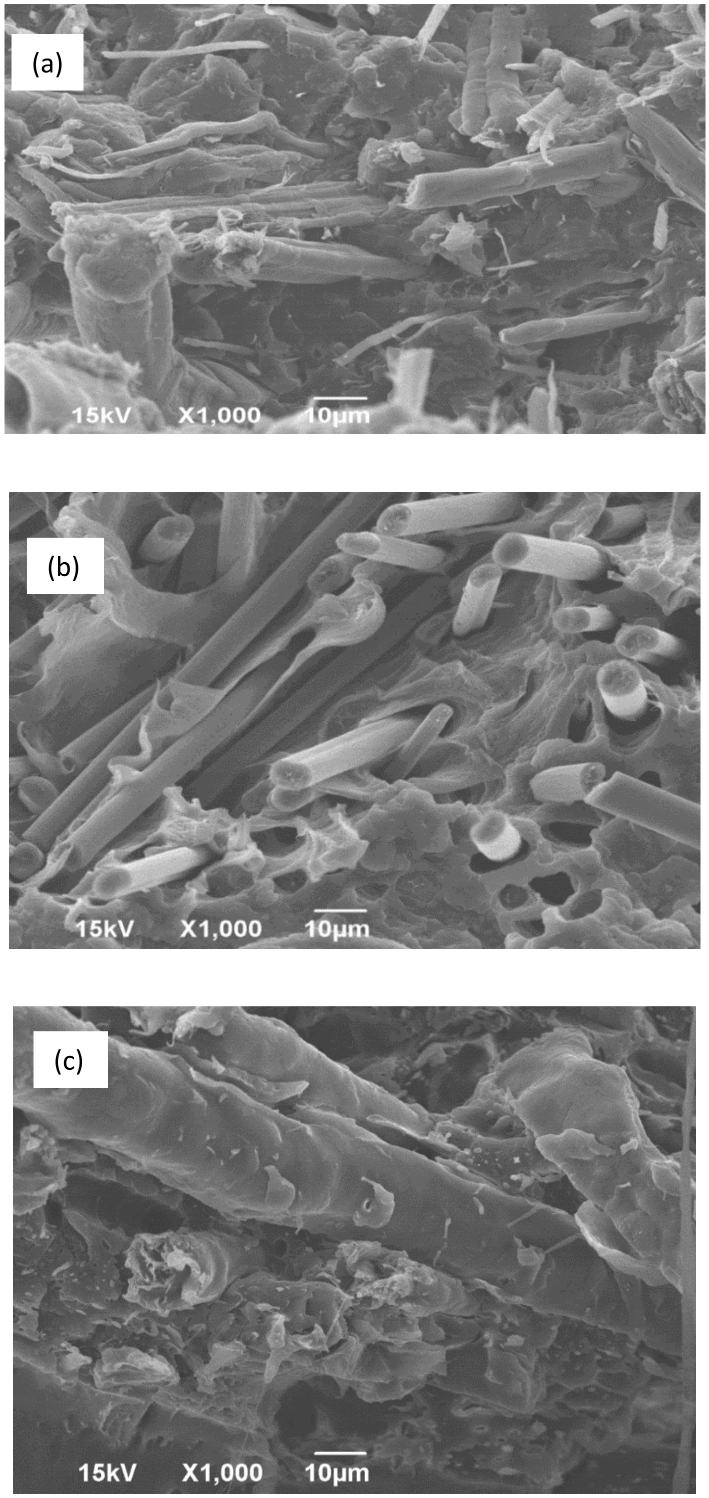

Figure 18: Comparison of SEM for (a) flax-30% PP-70%, (b) rCF-30% PP-70%, and (c) hemp-30% PP-70%.

4.2. Flexure Test Results

The ANOVA indicated a significant effect of fibers and treatments, and the interaction between them ($P \leq 0.05$). Recycled-carbon fiber when hybridized with flax fiber composite doesn't show a significant difference in the interactions between samples one and two. Interactions between treatments three and four neither have significant difference. Whereas hemp fiber reinforced composite materials have significant difference between interactions. Table 6, Figure 19 and Figure 20 show the comparison of flexure strength for flax and hemp fiber reinforced composites. The reinforcement of recycled carbon fiber in flax fiber improved the flexure strength by 4-5%. A significant 25-30% increase in the flexure strength of flax fiber composite was observed after the addition of the compatibilizer. The reinforcement of recycled-carbon fiber in hemp fiber improved the flexure strength by 30-33%. A significant 20% increase in the flexure strength of hemp fiber composite was observed after the addition of the compatibilizer. Recycled carbon fiber shows high flexure strength values when compared with flax and hemp fibers, respectively. Shahzad et al. (2013) investigated various mechanical properties of hemp and flax fibers. The results showed that when compared to PP, adding flax and hemp fibers to the PP matrix increased the flexure strengths. MAPP treatment significantly increased the values of flexure strength. After adding the fibers in a polymer matrix, a marked increase was also seen. A significant increase in flexural strength was associated with the presence of polypropylene treated with maleic anhydride in the composite matrix, while paradoxically a decrease in the flexural modulus was also associated. Elastic deformation occurs in the compression stage and not in the tensile stage that supports the fact that the modulus is low over strength (Adalberto et al., 2007). Pimenta & Pinho (2011) investigated the current outlook for recycled carbon fiber composites applications in the industry. Several mechanical properties were investigated, and the results had similar trends to our current study. Recycled-carbon fiber when reinforced with PP matrix has high flexure strength than flax and hemp fibers respectively. Holmes et al. (2017) investigated different methods to recycle carbon fiber and use it in different industrial applications. The study also showed a very strong bonding between rCF and PP resin which lead to high flexure properties.

Table 6: Mean flexure strength for flax and hemp fiber reinforced composite†

Sample	Flax	Hemp	rCF
	--------------------------------MPa------------------------------		
1	45.15 ± 0.86	30.24 ± 0.50	-
2	46.41 ± 0.39	32.13 ± 0.48	-
3	64.66 ± 0.75	41.04 ± 0.30	-
4	63.76 ± 0.54	47.50 ± 0.33	-
5	47.86 ± 1.83	46.39 ± 1.56	55.46 ± 1.13
LSD (0.05)		1.694	

† a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP-70%.

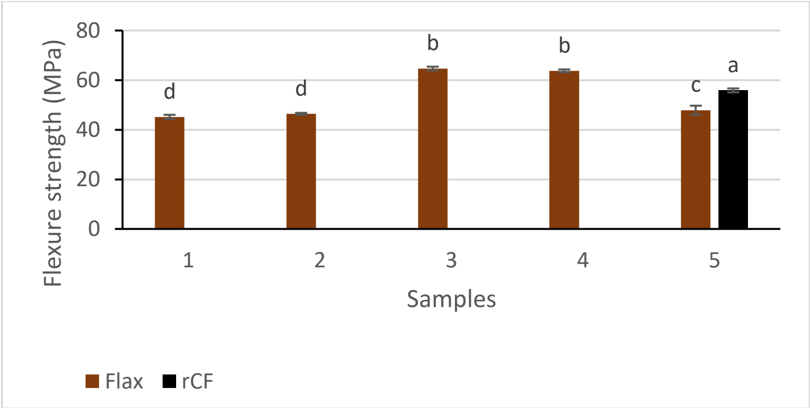

Figure 19: Comparison of flexure strength for flax and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP 70%.

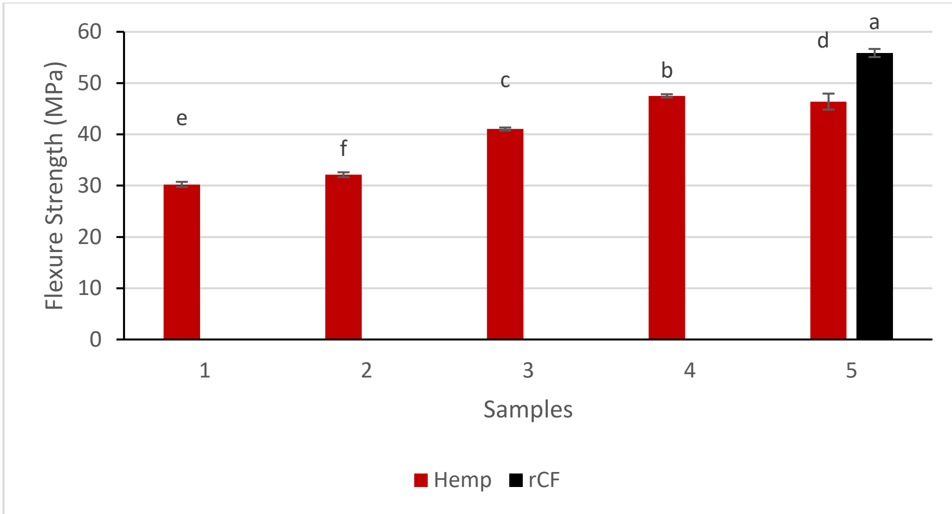

Figure 20: Comparison of the flexure strength for hemp and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber30% + PP 70%.

The ANOVA results indicated fibers, treatments, and the interaction between them was significant ($P \leq 0.05$). Recycled-carbon fiber does not show any significant difference in interactions between samples one and two as well as three and four in flax fiber composites. Recycled carbon fiber does not show significant difference between treatments one and three as well as treatments two and four for hemp fiber composites.

Table **7**, Figure 21 and Figure 22 , show the comparison of flexure modulus for flax and hemp fiber reinforced composites. The reinforcement of recycled-carbon fiber in flax fibers improved the flexure modulus by 25-30%. A significant 27% increase in the flexure modulus of flax-fiber composite can be observed after the addition of the compatibilizer. The reinforcement of recycled-carbon fiber in hemp fiber improved the flexure modulus by 40-45%. A 10-15 % increase in the flexure modulus of flax and hemp fibers can be observed after the addition of the compatibilizer. Recycled-carbon fiber shows the highest flexure modulus than both flax and hemp fibers. Arbelaiz et al. (2005) investigated the mechanical properties of flax fiber bundles and the influence of several factors such as fiber/matrix modification, on mechanical properties. Comparing flexural modulus results, the stiffness of unmodified composites was reduced as the amount of flax fiber bundle reinforcement was increased. Oppositely, MAPP-modified flax-fiber composites exhibited slightly lower modulus than those of glass fiber-PP ones Elkhaoulani et al., (2013)

Table 7: Mean flexure modulus for flax and hemp fiber reinforced composite†.

Sample	Flax	Hemp	rCF
	------------------------------GPa------------------------------		
1	2.22 ± 0.10	2.33 ± 0.08	-
2	3.58 ± 0.33	3.47 ± 0.24	-
3	3.07 ± 0.21	2.61 ± 0.28	-
4	3.68 ± 0.20	3.51 ± 0.19	-
5	2.55 ± 0.23	1.91 ± 0.16	5.60 ± 0.38
LSD (0.05)		0.255	

† a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP-70%.

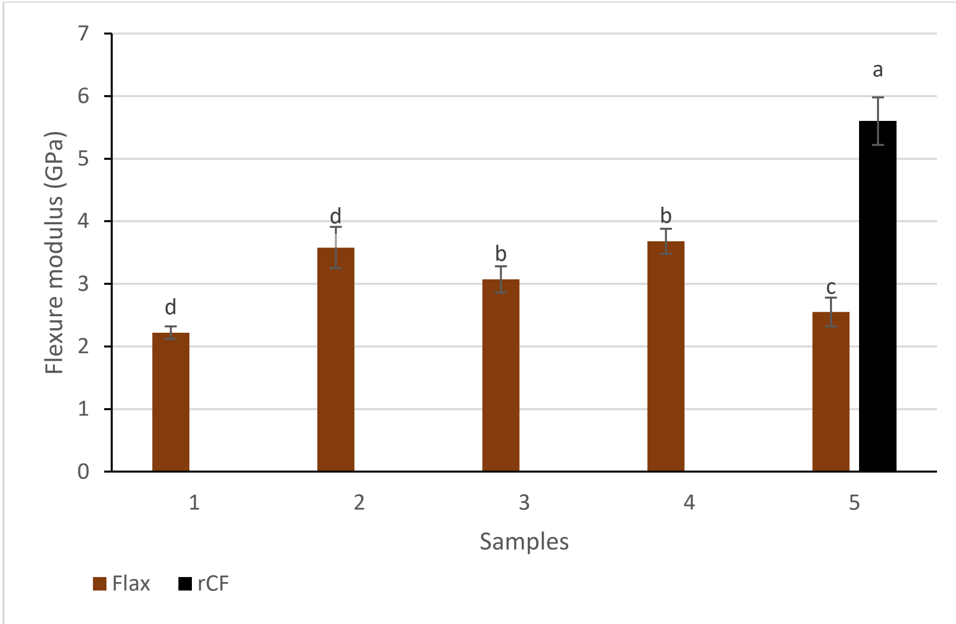

Figure 21: Comparison of the flexure modulus for flax and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5-fiber 30% + PP 70%.

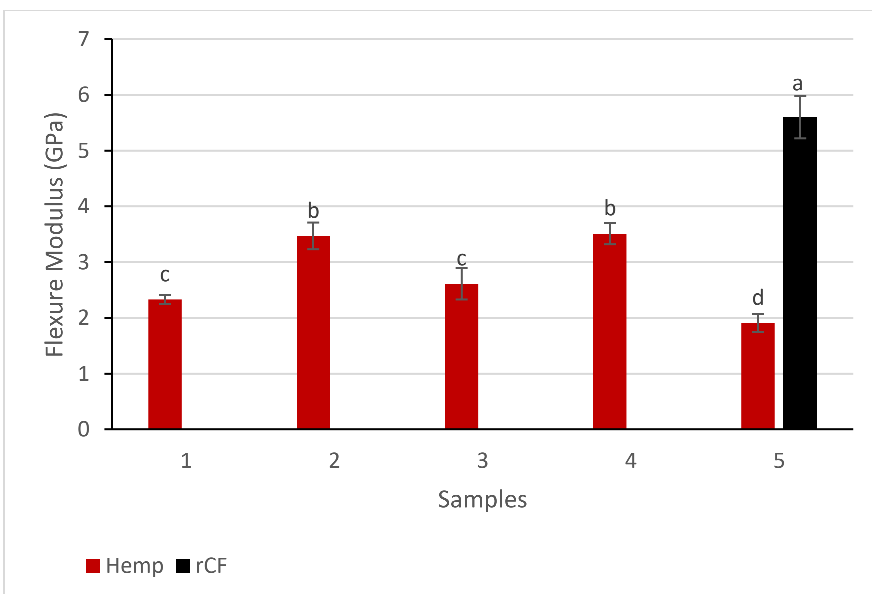

Figure 22: Comparison of the flexure modulus for hemp and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5-fiber 30% + PP 70%.

To support these flexure results, Micro-CT was carried out for the flexure samples to investigate void formation. Figure 23 and Figure 24 portrays the comparison between different fibers. It is observed in Figure 23a, flax fiber used as a reinforcement in polypropylene matrix is clean and pristine. No void formation can be seen in the composite and the fiber distribution is also random. This is also supportable by the fact that no appreciable differences between the density of fibers were mentioned. Some shive can be observed in the upper right corner of the composite which might be due to stress concentration. Figure 23b shows the micro-CT study for fractured surface of recycled carbon blended with PP. The study shows clean and pristine surface with no void formation resulting high flexure properties. Figure 23 (c), (d), (e), (f) show the micro-CT for fractured surface of flax fiber reinforced composite treated with and without the compatibilizer. The images show clean surface and no voids which indicate high mechanical property trends. Figure 24 (g), (h), (j) shows the micro-CT for fractured surface of hemp fiber composites. The composite shows clean and pristine surface with no void formation which leads to high impact properties. No void formation also leads to high mechanical property trends. Figure 24 (i), (k), shows the micro-CT for the fractured surface of the hemp fiber composites treated with MAPP. The composite shows random orientation of fiber which is also the reason for lower flexure properties of flax and hemp fibers compared with recycled carbon fiber (Etaati et al., 2014). A study was conducted wherein glass fibers were reinforced with PP and the failure strain analysis was investigated. Micro-CT scans were also conducted which reveal a strong heterogeneity of the microstructure, in terms of fiber orientation. In fact, short-glass fibers followed in-plane distributions of orientation around a preferential orientation in shell and core layers of the injected composite plates, with preferential orientation parallel to the injection flow direction (IFD) in shell layers and highly angled with respect to IFD in core layer (Notta-Cuvier et al., 2016).

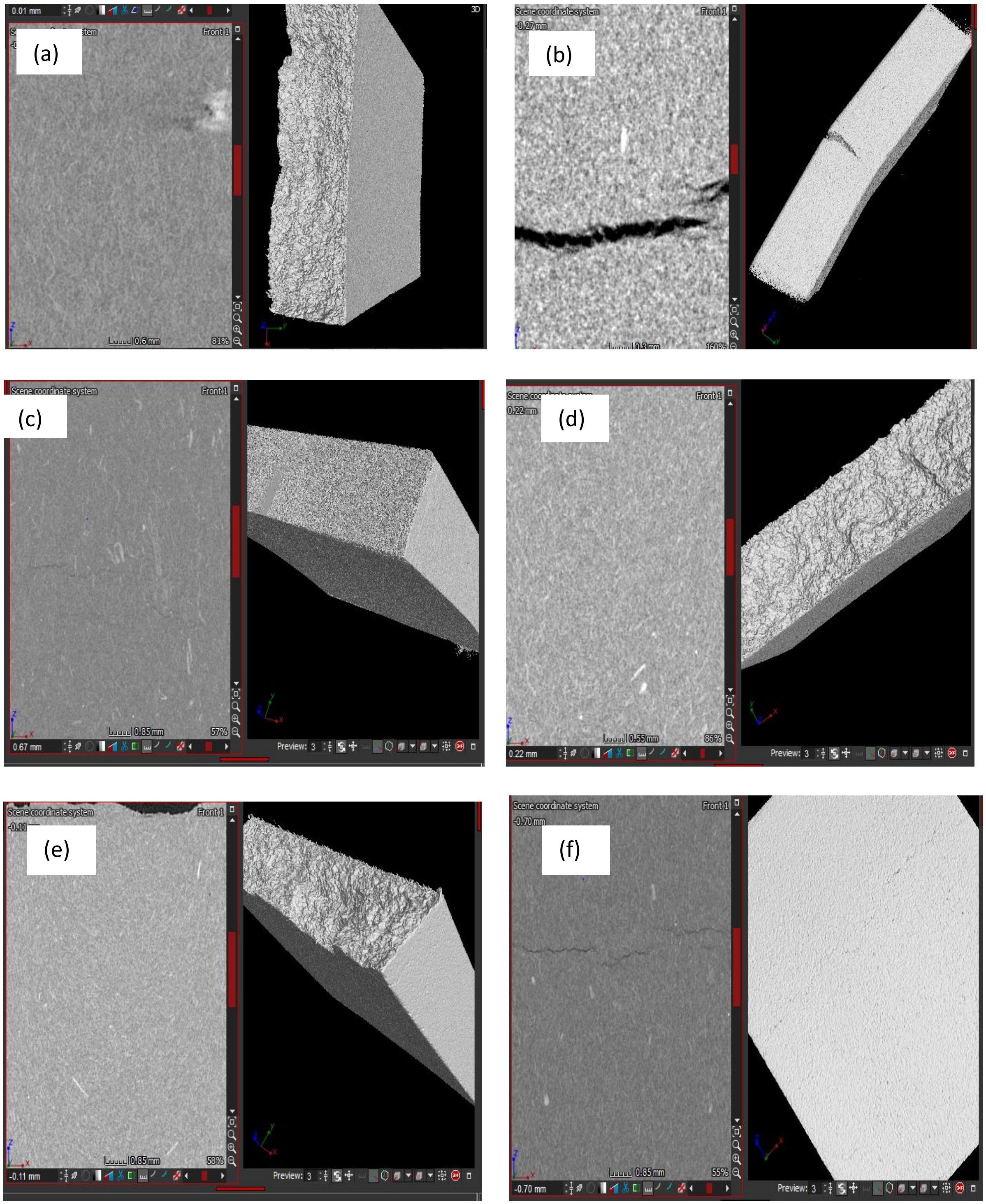

Figure 23: Comparison of micro-CT between (a) flax-30% PP-70%, (b) rCF-30% PP-70%, (c) flax-10% PP-70% rCF-20%, (d) flax-10% PP-68% MA-2% rCF-20%, (e) flax-20% PP-70% rCF-10%, (f) flax-10% PP-68% MA-2% rCF-20%.

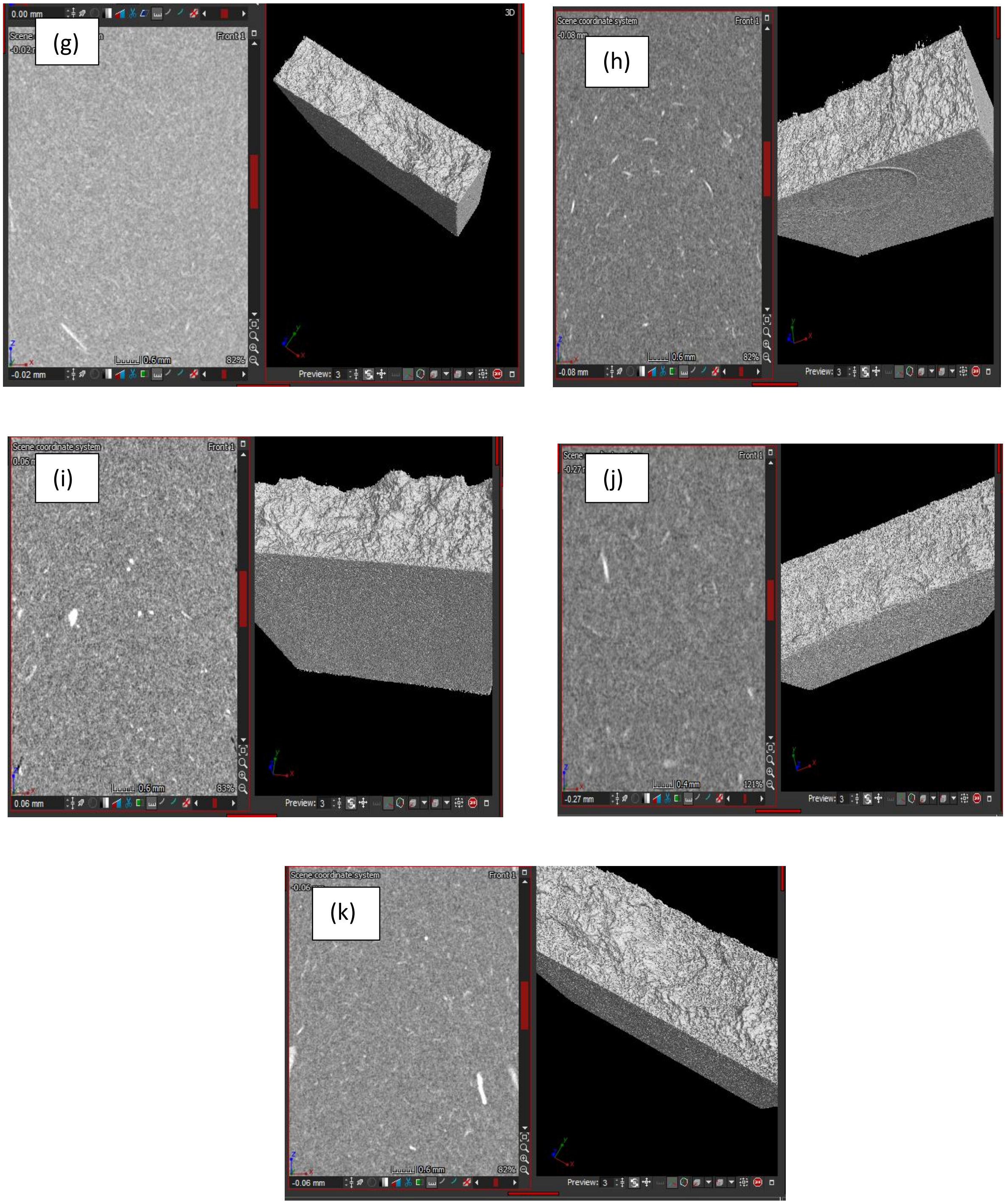

Figure 24: Comparison of micro-CT between (g) hemp-30 % PP-70%, (h) hemp-10% PP-70% rCF-20%, (i) hemp-10% PP-68% MA-2% rCF-20%, (j) hemp-20% PP-70% rCF-10%, and (k) hemp-20% PP-68% MA-2% rCF-10%.

4.3. Impact Test Results

The ANOVA results indicated a significant effect of fibers and treatments, and the interaction between them ($P \leq 0.05$). Recycled-carbon fiber does not show significant difference between interactions in samples two and three as well as one and five for flax and hemp fiber composites. Table 8, Figure 25 and Figure 26 shows the comparison and interaction between the mean values of impact strengths of all the fibers. The reinforcement of recycled-carbon fiber in flax fiber improved the impact strength by 5-10%. A 15-20% increase was observed in the impact strength after the treatment of compatibilizer. A 30-35% increase was observed in the impact strength of hemp fiber after the maleic anhydride treatment. The impact strength for recycled-carbon fiber is the lowest as compared with that of flax and hemp fibers. Hemp fibers show the highest impact strength. Hemp fiber has very high shear strength compared with that of flax and recycled-carbon fiber making it difficult to break. (Višnjić et al., (2014) investigated the effect of recycled-carbon fiber composites on different mechanical properties. The results showed that the dispersion of the reinforcements/fillers has a strong influence on mechanical and morphological properties of composites. The impact strength decreased as the percentage of recycled-carbon fiber increased which agrees with the results of the current study. Elkhaoulani, et al. (2013) investigated the effect of reinforcement of hemp fiber composites on mechanical properties. The results showed that the bonding between the hemp fiber and the resin was very strong which was attributed to the good adhesion between fibers and the matrix. It was worth noticing that the interactions between the PP matrix and the hydroxyl groups on cellulosic fibers were enhanced as the fiber weight percentage increased. Safri et al. (2018) investigated the impact properties for different composite materials. Higher impact strength values for many natural fibers such as flax, hemp and sisal when compared with other synthetic fibers such as carbon fiber as weight fraction increased.

Table 8: Mean impact strength for flax and hemp fiber reinforced composite†.

Sample	Flax	Hemp	rCF
	-----------------------------	kJ/m^2	----------------------------
1	3.51 ± 0.33	4.48 ± 0.54	-
2	3.80 ± 0.51	4.38 ± 0.22	-
3	4.40 ± 0.56	7.03 ± 0.47	-
4	4.56 ± 0.67	6.00 ± 0.71	-
5	3.11 ± 0.17	5.52 ± 0.38	2.23 ± 0.05
LSD (0.05)		0.658	

†a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber 30% + PP 70%.

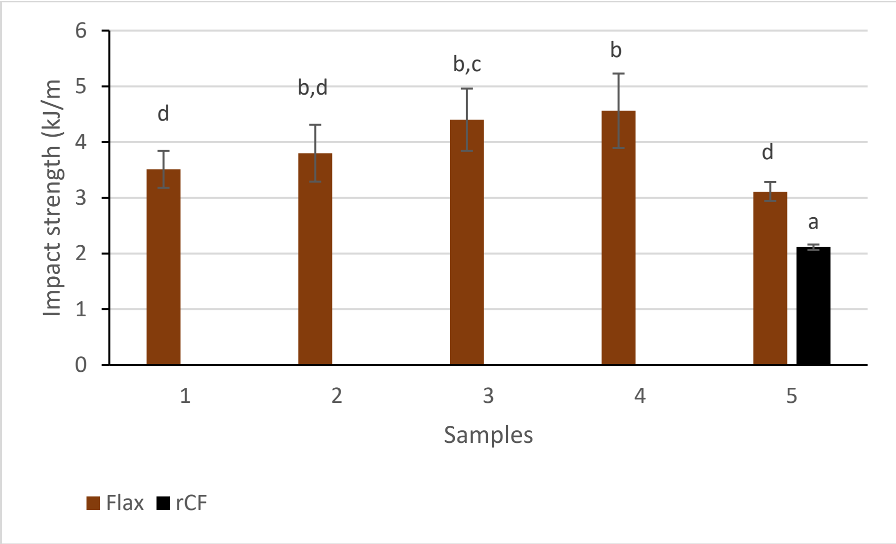

Figure 25: Comparison of impact strength for flax and rCF composites between a) Sample 1- fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3- fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber-30% + PP 70%.

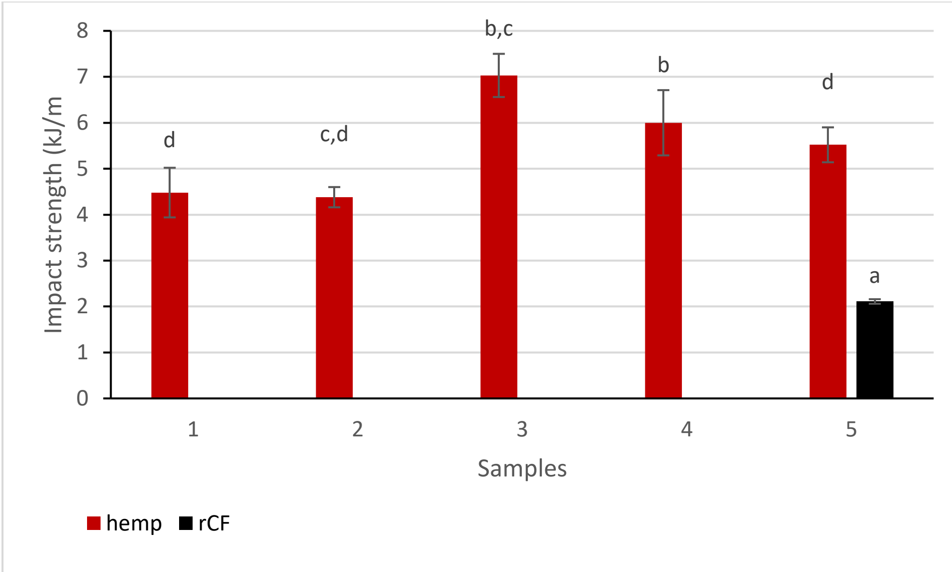

Figure 26: Comparison of impact strength for hemp and rCF composites between a) Sample 1-fiber 20% + rCF 10% + PP 70%, b) Sample 2- fiber 10% + rCF 20% + PP 70%, c) Sample 3-fiber 20% + rCF 10% + PP 68% MA 2%, d) Sample 4- fiber 10% + rCF 20% + PP 68% MA 2% and, e) Sample 5- fiber-30% + PP 70%.

To support these results SEM study was conducted. The following images portray the fiber polymer bonding between different fibers and help to study the characteristics and nature of the fibers. Figure 27 shows the SEM comparison between flax, hemp and recycled carbon fiber composites. It can be observed in Figure 27 (a) that there is poor adhesion between the interface of flax fiber and polypropylene resin which is responsible for the low impact strength observed as compared with hemp fiber. Impact strength for flax fiber was higher as compared with recycled-carbon fiber. The high shear strength of flax fiber is the reason for high impact strength observed. It is very difficult to break flax fiber due its high stiffness. Some negligible voids can be seen in the study. It can be observed in Figure 27 (b) that the polymer-fiber bonding is relatively weaker resulting in low impact properties. The fibrilization process takes place in the specimen, which results in poor strength of the impact test. The impact strength is decreased due to the non-uniform size of filler material (recycled-carbon fiber) and its distribution over the surface occupying the over edges of the matrix. Fiber pullout can be easily spotted. Voids can be observed which supports the fact of low impact strength. Recycled carbon fiber has low shear strength and hence is brittle in nature. Therefore, it can be observed that the impact strength of recycled-carbon fiber is lower than both flax and hemp fibers. It can be observed from the Figure 27 (c) that the polymer fiber bonding for hemp fiber is relatively stronger than both flax and recycled carbon fiber resulting in high impact properties. Strong adhesion between the interface of fiber and matrix can be observed in the image. The SEM image clearly portrays no occurrences of fiber pulled out in the specimen, leading to high impact properties. High impact strength plays a key role in aerospace and automotive applications. Figure 28 (a) and (b) shows the comparison of SEM study for hemp fiber reinforced-rCF composites with and without the treatment of the compatibilizer. It can be observed from Figure 28 (a) that the polymer fiber bonding is relatively stronger than the untreated fibers resulting in high impact properties. Although fiber pullout can be spotted, it can be observed that after the maleic anhydride treatment the covalent bond between the fiber and polymer was strong. Hybridization of recycled fiber with hemp fiber has improved the impact strength.

Yan et al. (2014) investigated the properties for flax fibers and its composites. The results showed a gap at the interface between fiber and PP matrix. Hence, fibers had no coating of PP around their surfaces, which indicates poor interactions between flax fiber bundle surface and PP matrix. The current study also showed similar results. Park, et al. (2006) investigated different properties of hemp fibers and its composites. The results were like the current study which proves that hemp fiber has high shear strength and high impact properties. Both the natural fibers are hydrophilic in nature and contain a lot of fibrils which tend to bond and entangle with the hydrogen bonds in the matrix. Mallik, et al. (2007) investigated different fiber reinforced composites and the results showed low impact properties for recycled carbon fiber with increasing weight fractions.

The results were also in agreement with the current study which proves the fact that rCF has low impact properties when compared with other with increasing natural fibers content.

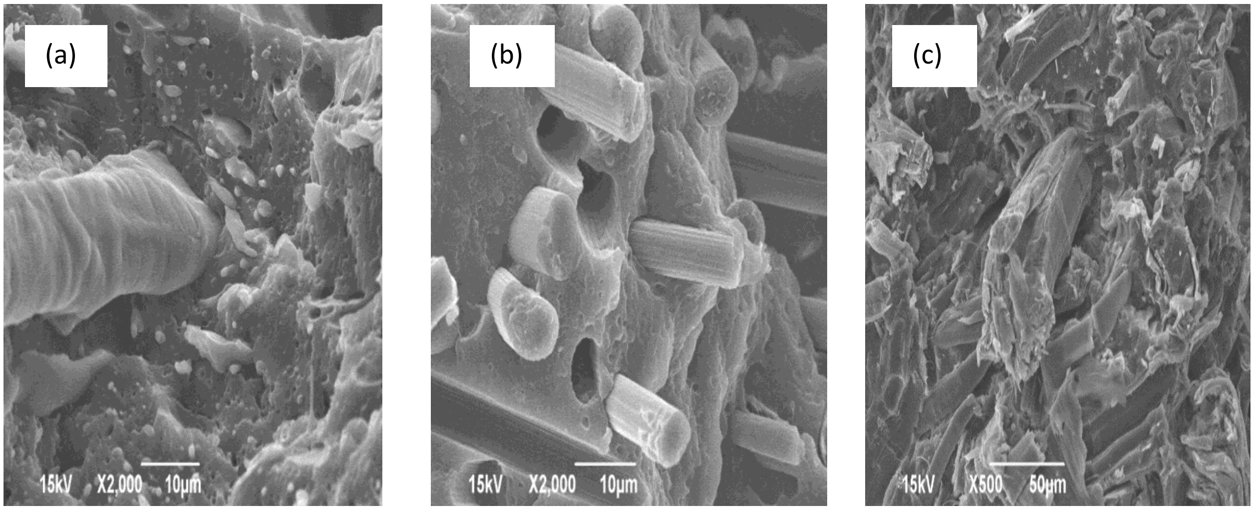

Figure 27: Comparison of SEM between (a) flax-30% PP-70%, (b) rCF-30% PP-70% and (c) hemp-30% PP-70%.

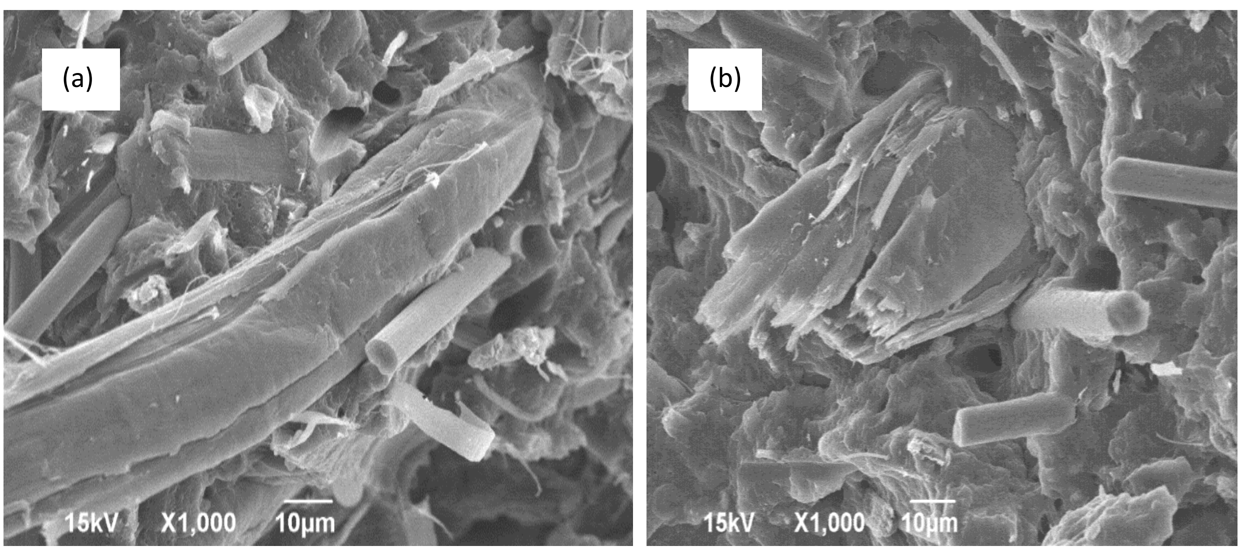

Figure 28: Comparison of SEM between (a) SEM study for hemp-20% rCF-10% PP-70% and (b) hemp-20% rCF-10% PP-68% MA-2%.

5. CONCLUSIONS

Hybridizing synthetic fibers with natural fibers has been found to be an effective method for improving the mechanical properties. In addition to the gains in mechanical properties, which resulted from hybridizing synthetic fibers with natural fibers, it was also concluded that the use of recycled carbon fiber as a reinforcement improved flexure and tensile properties significantly. The reinforcement of recycled carbon fiber reduced the mechanical variability which is evident in the micro-CT images. Micro-CT showed improvement in the orientation of the fibers which helped to reduce mechanical variability having a very strong effect on the mechanical properties. The treatment of natural fibers with maleic anhydride played a major role in improving the interfacial adhesion between fibers and matrix thus enhancing the mechanical properties. An increase of 35-40% was observed in the tensile strength of the composites, whereas an increase of 30% was observed in the flexure properties of the composites after the treatment. Impact properties showed an increase of 10-15% after the treatment of the compatibilizer. Improvement in the mechanical trend of properties was supported by statistical analysis. From a composite designer's point of view both the natural fiber composites can be used for applications with reasonably high tensile and flexure strengths, whereas hemp fiber reinforced composites would be the best choice for applications which require high impact strengths. Hemp fiber is coarse in nature due to the arrangement of its constituents (pectin, lignin and hemi-cellulose) which promote higher impact strength over flax fiber.

The end goal of this research was to provide a means for natural fibers to expand into various light weighting and sustainable design applications. Through hybridizing bast natural fibers and recycled carbon fibers in a polyolefin thermoplastic, a new class of high-performance composites can be demonstrated for a cluster of different applications.

6. RECOMMENDATIONS FOR FUTURE WORK

To further expand and extend the scope of this study, additional and future work could be done based on the results of this project. The next phase of testing may involve composites that are treated with a compatibilizer with different volume fractions. In the hybridization study, various natural fibers such as kenaf and coconut could also be used. It is possible to investigate dynamic and static mechanical properties for comparison between them. Another area to be examined would be natural fiber surface treatments to achieve a better representation of actual mechanical data. Researchers have found improved mechanical properties by incorporating surface treatments that make fiber and matrix more bonding. The main cause of failure was natural fiber debonding when looking at the hybrid impact specimen's failure modes. This debonding could potentially be reduced through surface treatment, resulting in improved impact strengths. There could be improved mechanical properties across the board through surface treatments and a more real sense of what is happening could be determined.

Another area that could help engineers use hybrid composites better would be to investigate the effects on mechanical properties of the environment. This would involve submitting composites for a period to a weathering chamber after mechanical testing. This could range from water absorption, thermal aging, and how hybrids work at different temperatures. The development of theoretical modeling should be considered to predict the properties of hybrid fiber composites based on an understanding of fiber and matrix and their interrelationship. Collaborating with various companies to develop new demonstrations of technology would also help researchers to grow and develop.

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APPENDIX. STATISTICAL ANALYSIS OF DATA

Table A1: Mean square values of tensile strength, tensile modulus, flexure strength, flexure modulus, and impact strength†.

SOV	df	TS	TM	FS	FM	IS
Fiber	1	2.04*	24.06*	155.97*	0.58*	32.38*
Sample	4	550.3*	10.0*	875.30*	4.60*	5.97*
Sample **x** Fiber	4	3.20*	21.33*	37.39*	0.23*	1.98*
Error	44	0.25	0.17	1.76	0.04	0.26

† * is significant at 0.001. TS: Tensile strength, TM: Tensile modulus, FS: Flexure strength, FM: Flexure modulus, IS: Impact strength, df: degrees of freedom, SOV: sources of variation.