Multifunctional Jute-Banana Hybrid Composites Reinforced with Lemon Peel Oil and Potato Peel Starch: Synergistic Effects on Mechanical, Hygrothermal and Bioactive Performance

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ABSTRACT

In order to fulfill the demand for eco-friendly materials, it is imperative to fabricate high-performance bio composites from agro-industrial wastes. The current research investigates the fabrication and characterization of jute-banana hybrid composites using potato peel starch as a biodegradable binder and natural antimicrobial and hydrophobic agent, lemon peel oil. Seven composites were designed with different fiber combinations, starch content (5-15% w/w) and lemon peel oil content (0-10% v/v). The structural, morphological and mechanical characterization of the developed composites showed that JB50-L10 hybrid composite (50% jute, 50% banana, 10% starch, 10% lemon oil) has optimized multiple performance. It was proved from FTIR characterization that there was enhancement in esterification (C=O at 1740 cm, C-O-C at 1172 cm) and hydrogen bonding. Mechanically JB50-L10 has 49.2 Mpa tensile strength and 70.3 Mpa flexural strength. The value of moisture regains, water absorption and water vapor transmission rate for JB50-L10 (11.2%, 7.9% and 322.8 g/m/day) was less compared to other unmodified composites when 10% lemon peel oil was incorporated to. The surface was hydrophobicity. JB50-L10 was stable when degradation of composite at different zones was studied by TGA. The inhibition zone of JB50-L10 against both *E. coli* and *S. aureus* were found to be 9.0 mm. Also, JB50-L10 showed better resistance toward UV light (E=3.3). It was statistically confirmed using One-way ANOVA at the $p<0.05$ level of significance for all characterization. This work showed that there is a possible process that could add value to the agro-waste by producing bioactive composites at industrial level for both packaging and building materials.

KEYWORDS: Jute-banana hybrid composite; Lemon peel oil; Potato peel starch; Antimicrobial bio composite; Argo-waste valorization

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1. INTRODUCTION

The increasing environmental crisis due to continuous generation and landfill of petroleum-based plastic has intensified the urgent need to seek viable alternative materials (Mohanty et al., 2001; Faruk et al., 2012). The natural fiber composites (NFCs) are one of the best candidates as these materials have a variety of advantages over synthetic composites, i.e. Biodegradability, low density, high specific strength, low cost and carbon friendliness (Pickering et al., 2016; Sanjay et al., 2018). However, limited application of NFCs occurs because natural fibers possess uneven characteristics and exhibit problems such as hydrophilicity, poor adhesion to hydrophobic matrices, no inherent biological activity, etc (Bledzki and Gassan, 1999; Dittenber and GangaRao, 2012).

In order to avoid the drawback of single natural fiber reinforcement, hybridization (combination of more than two types of natural fibers into a single matrix) of natural fibers has proven to be an excellent technique (Thwe and Liao, 2002; Gurukarthik Babu et al., 2019). Through wise selection of appropriate fiber pairs with complementary properties, mechanical drawbacks can be compensated and composite performances may be enhanced. Among natural fibers, jute (Corchorus capsularis) and banana (Musa spp.) are the two of the most abundantly available agricultural residues produced in tropical and subtropical countries (mainly South Asia) (Basu and

Roy, 2008; Mukhopadhyay et al., 2008). Jute is a bast fiber with excellent cellulose content, low microfibrillar angle and highest tensile rigidity (Idicula et al., 2005). Banana fibers are obtained from pseudo stem of the banana plants. These fibers are more flexible and porous than jute with increased content of lignin, which enhance toughness and acoustic damping (Pothan et al., 1997; Li et al., 2007). Thus, hybridization of jute and banana fibers can develop a composite structure having tensile property from jute fibers and toughness from banana fibers.

Despite of the mechanical advantage, a major restriction for the usage of lignocellulosic fibers is the strong hydrophilicity nature which is mainly due to the presence of large number of hydroxyl (-OH) groups in cellulose and hemicellulose contents (George et al., 1999; Ray et al., 2001). Hydrophilicity of the natural fiber can result in dimensional instability (swelling), weak fiber-matrix interface bond and quick degradation of mechanical properties in moist conditions (Valadez-Gonzalez et al., 1999). Alkaline treatment (mercerization) has been successfully adopted for removal of impurities, hemicellulose and lignin from the fiber surface, to increase the surface roughness and to expose more -OH groups of cellulose which will increase the interaction between matrix and fiber (Ray et al., 2001; Mohan and Kanny, 2019).

At the same time, the nature of matrix material dictates much of the environmental profile of the composite. Common synthetic matrix resins such as epoxy and polyester compromise the environmentally conscious nature of NFCs (Gandini, 2008). Starch, being a natural polysaccharide derived from waste like potato peel, is a fully biodegradable matrix material (Jimenez et al., 2012; Santana et al., 2018). Potato peel starch is rich in amylose and amylopectin which, upon gelatinization, provide a continuous and adherent matrix. The matrix interacts with the cellulosic fibers via hydrogen bonding (Khalil et al., 2006). However, the lack of rigidity and high-water absorption of the matrix cause brittle material and quick biodegradation (Koszkul and Nabialek, 2002; Arrieta et al., 2017). To circumvent the weaknesses caused by moisture absorption and biodegradability, research endeavors on using natural essential oils in bio composites have been an area of active interest (Burt, 2004; Tongnuanchan and Benjakul, 2014). Lemon peel oil is one of the waste products of citrus juice industry which is predominantly comprised of limonene, central, and various flavonoids (Sharma et al., 2021). Limonene and central possesses strong broad-spectrum antimicrobial activity due to the disruption of the bacterial cell membrane (Sánchez-González et al., 2011; Atarés and Chiralt, 2016). Besides the antimicrobial property, the hydrophobic nature of limonene and its potential to undergo esterification with hydroxyl groups of cellulose also contribute to enhancing moisture resistant behavior and interface strength (Sain and Panthapulakkal, 2006; Beg and Pickering, 2008).

Although the use of jute-banana hybrid (Rana et al., 2015; Gurukarthik Babu et al., 2019) or starch-based bioplastic (Khalil et al., 2006; Prasad and Rao, 2011) has been explored in separate fields, very few studies addressed the combined benefits of a waste-derived starch binder and a functionalizing essential oil to strengthen hybrid lignocellulosic composite structure. Most of them used synthetic coupling agent or investigated properties in individual perspective. In this work, a complete bio-based and no-synthetic composite is engineered using jute, banana, potato peel starch and lemon peel oil. A total of 14 different analyses were performed and illustrated using different advanced statistical plotting techniques in order to clarify the structure-property relation. This aims to show the specific optimized composition (JB50-L10) may exhibit strong mechanical strength, thermal stability, hydrophobicity and bioactive properties; and offers high value agro-waste utilization for packaging, medical textiles and construction industries.

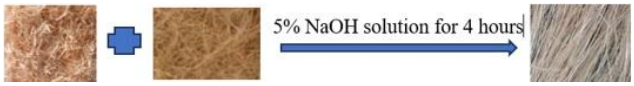
2. MATERIALS AND METHODOLOGY

Materials

Jute fibers were procured from the local jute mills in Bangladesh and the banana fibers were separated from the pseudostems by retting followed by manual stripping/ scraping. Potato peel and lemon peel were purchased from the local markets of agricultural produce. Sodium hydroxide (NaOH, 5%) and acetic acid (1%) were of analytical grade. The experiments were carried out using distilled water.

Fiber Treatment

The jute and banana fibers were cut into 30 mm lengths. The fibers were then treated with an alkali by immersing in 5% NaOH solution for 4 hours at room temperature for the removal of hemicellulose and wax (Ray et al., 2001). Then the fibers were neutralized with 1% acetic acid, washed with distilled water till neutral pH (7) and dried at 60°C for 24 h in an oven.



BINDER PREPARATION AND FUNCTIONALIZATION

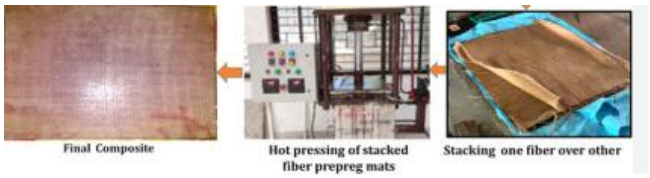
Potato Starch Extraction: Potato peels were chopped into small pieces and boiled with 1:5 (w/v) distilled water for 30 min. The liquid was filtered and allowed to settle for 12 h, then the starch paste was removed and dried at 50°C (Santana et al., 2018).

Lemon Peel Oil Extraction: Essential oil was extracted by steam distillation of the dried lemon peels for 2hours and dried over anhydrous NaSO (Atarés and Chiralt, 2016).

Binder Formulation: The starch powder was added to the distilled water (5, 10 and 15% w/w) and heated at 80°C with stirring till gelatinization occurred. After the cooling, lemon peel oil (0, 5 and 10% v/v) was added into the prepared starch paste with vigorous stirring to make a stable emulsion.

Composite Fabrication

Fiber mats with definite jute and banana fiber ratio (100:0, 50:50, 0:100) were prepared by hand laying technique. These mats were dipped into the functionalized binder for 5 min. Then the



functionalized binder impregnated fiber mats were pressed at 100°C and 5 MPa pressure for 10 min in a hydraulic compression molding machine. The designations of samples are: J100, B100, JB50, JB50-L5, JB50-L10, JB50-S5 and JB50-S15.

3. CHARACTERIZATION TECHNIQUES AND TESTING PROTOCOLS

A series of analytical, mechanical, hygrothermal and biological characterizations were performed in order to investigate the multifunctionality of the fabricated biocomposites. The data presented were the average value obtained from at least five identical samples (n=5).

Chemical and Thermal Characterization

Fourier Transform Infrared (FTIR) Spectroscopy: The chemical functional group modification of treated and untreated fibers, starch and composite was analyzed by FTIR spectroscopy using Thermo Scientific Nicolet iS50 FTIR spectrometer equipped with a diamond ATR crystal. The spectra were recorded in the wave number region 4000-500 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans per sample. We measured O-H stretching (3300-3400 cm^{-1}), C=O ester stretching (1700-1750 cm^{-1}) and C-O-C vibrations (1150-1200 cm^{-1}) to verify the treatment effect on the fibers and ester formation of lemon peel oil with the fiber's ester functional groups.

Thermogravimetric Analysis (TGA): TGA was performed using a TA Instruments TGA Q50 under nitrogen flow rate 60ml/min to determine the thermal stability of the composite. Around 8-10 mg finely powdered composite sample was heated from 30°C to 600°C at a constant heating rate of 10°C/min. Weight loss percentage values of three zones were calculated. Zone 1 (30-150°C) moisture evaporation zone; Zone 2 (150-300°C) starch and hemicelluloses degradation zone; Zone 3 (300-600°C) cellulose and lignin decomposition zone.

4. MECHANICAL CHARACTERIZATION

Tensile Strength and Elongation at Break: Tensile properties of the composite were measured according to ASTM D638 with 5 kN load cell of Universal Testing Machine (Instron 3366) at a cross-head speed of 5 mm/min with dumbbell shaped specimen (Type V). Tensile strength (MPa) and elongation at break (%) were recorded.

Flexural Strength: The flexural strength of the composite was determined by three-point bending test as per ASTM D790 with rectangular specimen (80x10x3mm). The support span to depth ratio was maintained at 16:1 and the tests were conducted at 2 mm/min cross head speed.

Impact Resistance: The Izod impact resistance was conducted using a pendulum impact tester (Tinius Olsen Impact 104) according to ASTM D256 on the specimen (64x12.7x3mm) with 2.75J energy at a cantilever fixed rate. The data was calculated from the kinetic energy left in the pendulum (J/m).

5. HYGROTHERMAL AND PHYSICAL CHARACTERIZATION

Moisture Regain: Dried specimen in hot air oven at 105°C for 24 h and weight was taken as dry weight (W_d), conditioned as per ISO 139. Then, placed inside the humidity chamber maintained at 27C and 65% RH and weight was recorded as conditioned weight (W_c) after 24h.

Moisture regain = $[(W_c\text{-}W_d) / W_d] / \text{times } 100$

Water Absorption and Thickness Swelling: Water absorption and thickness swelling were measured according to ASTM D570. Dried specimen was kept in a beaker filled with distilled water for 24 h at room temperature. Excess water was removed by blotting with a clean cloth. Then the water absorption percentage of the dry weight was recorded. Thickness was measured by digital micrometer (0.001 mm precision) before and after water immersion.

Water Vapor Transmission Rate (WVTR): The samples were evaluated by the gravimetric cup method ASTM E96. Sample was mounted over the cup containing anhydrous $CaCl_2$ (0% RH). The entire setup was kept inside a desiccator at 75% RH and 27°C. The weight increase of the cup was recorded at every 24 h for 5 days and the WVTR was reported in $g/m^2/day$.

Surface Hardness: The Shore D hardness was measured on the surface of the composite sample using a digital Shore D durometer (ASTM D2240). Ten measurement was made on each sample at random positions to cover the surface.

6. FUNCTIONAL AND BIOLOGICAL CHARACTERIZATION

Antimicrobial Activity: The composites were tested against Gram-negative *Escherichia coli* (ATCC 25922) and Gram-positive *Staphylococcus aureus* (ATCC 6538) bacteria by Agar Diffusion Method (AATCC 147). Each disc of 10 mm diameter made of composites was placed on a nutrient agar plate which has already seeded by the bacteria 10^6 CFU/ml, and then incubated at 37°C for 24 h. The zone of inhibition around the discs was measured (mm).

Biodegradability (Soil Burial Test): A piece of composite of dimension 20/times 20/times 3 mm was buried about 10 cm depth in natural agricultural soil (pH 6.8). The sample was left in the soil for 30 days at 25°C and 60% moisture content. Then the composite was dug out of the soil and washed clean with water, dried to a constant weight at 105°C and weighed for the determination of weight loss percentage.

UV Resistance: A UVA-340 fluorescent bulb, simulating UV radiation in the spectrum of sunlight was used in QUV/se Accelerated Weathering Tester (ASTM G154) at 60°C under UV exposure for 8 h and condensation at 50°C for 4 h per day cycle for 72 h. The color difference (Delta E) was determined by HunterLab MiniScan colorimeter.

7. RESULTS AND DISCUSSION

FTIR Spectroscopy and Chemical Interactions

Clear evidence for the alteration in chemical bonding through incorporation of lemon peel oil and potato peel starch can be seen in the smoothed FTIR spectra (Fig 1). A strong, broad O-H stretching vibration band from moisture adsorption and cellulosic -OH group for the single fiber composite J100 and B100 are seen around 3402cm^{-1} and 3395cm^{-1} respectively. It can be clearly noticed that the band is shifted to 3372cm^{-1} with lowered transmittance intensity after the hybridization and functionalization of with 10% lemon oil (JB50-L10) due to strong intermolecular hydrogen bonding between the -OH group of the jute/banana fiber and amylose/amylopectin chain of potato starch binder (Sain and Panthapulakkal, 2006).

Most significantly, two sharp bands at 1740 cm^{-1} and 1172 cm^{-1} are observed only in JB50-L10 sample. The band at 1740 cm^{-1} represents C=O stretching vibration in ester function and 1172 cm^{-1} is associated with C-O-C asymmetric stretching vibration of ester link. These peaks were completely absent in unmodified JB50 sample, which clearly indicate that esterification reaction between carboxylic group present naturally in lemon peel oil (mainly oxidation product of citral and limonene) and free -OH group on the surfaces of alkali-treated fiber occurs (Sharma et al., 2021). Additionally, the increased transmittance intensity in the C-

H stretching vibration band at 2915 cm^{-1} in JB50-L10 confirms the inclusion of hydrocarbon part of the essential oil in the polymer matrix by physical entrapment. These modifications lead to subsequent improved hydrophobicity and mechanical stress transfer.

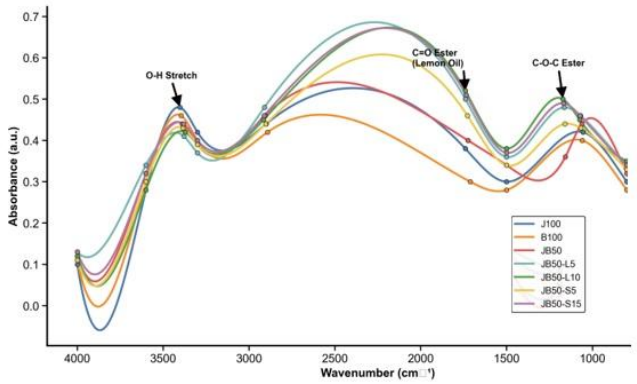


Fig. 1. FTIR spectra of Hybrid composites

Thermal Stability

A stacked bar chart (Fig 2) depicting the sum total weight loss over the three thermal zones clearly illustrates the greater thermal stability afforded to the oil-functionalized composites. In zone 1 (30-150C) that corresponds to release of physically bound water, JB50-L10 registered least weight loss (4.4%) against the controls B100 (6.1%) and J100 (5.2%). This is consistent with the observations made from the FTIR spectra which indicated that the esterification of hydrophilic –OH groups has rendered the composites hydrophobic in nature; hence less susceptible to water adsorption (Kim et al., 2005).

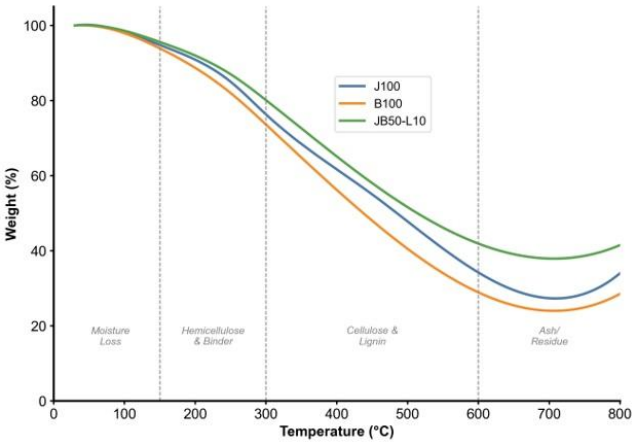


Fig. 2. TGA thermogram of composite

Zone 2 (150-300C) is a zone of thermal cleaving of starch binder, hemicellulose and initiating volatilization of limonene. JB50-L10 displayed limited breakdown (15.5% loss) against that of B100 (20.3%). This limitation is attributed to higher energy input required for chain scission of the crosslinked ester network of the lemon oil, acting as thermal shield around hemicellulose (Yang et al., 2007). Zone 3 (300-600C) represented depolymerization of cellulose and lignin. The lignocellulosic shield of the char-forming capacity of the starch-oil matrix has prevented rapid volatilization of decomposition products thus enhancing the overall thermal stability by imparting increased residual char yield (Ouajai and Shanks, 2005).

Tensile Strength

The mean tensile strength is highest for J100 (52.3 MPa) because jute has a high cellulose content and low microfibrillar angle which allows load to be transmitted in the axial direction, while B100 is lowest (38.7 MPa) (Fig 3). The hybrid JB50 produces a mean value of 45.0 MPa. The mean value increases sharply for JB50-L10 (49.2 MPa, which is near to that for J100), which is more significant when compared with the extremely low variance around the mean. This suggests that the lemon oil is effective in filling the voids at the interface between the fibers and matrix, effectively creating a very efficient coupling agent which forms chemical ester linkages with the fibers, while also creating mechanical interlocking with the matrix, so the stress applied is transferred efficiently to the fibers and catastrophic failure is avoided (Mwaikambo and Ansell, 2002).

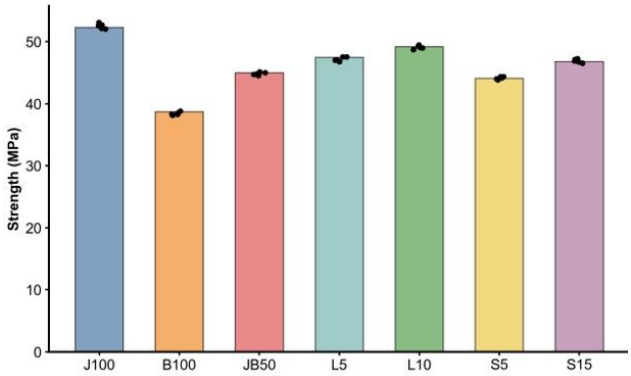


Fig. 3. Tensile strength of composites

Elongation at Break

The lollipop plot indicates quite clearly the change in ductility of the formulations. J100's low 2.1% elongation demonstrates its rigidity while the porous and less crystalline nature of the banana fibers results in a value as high as 3.4% elongation in B100 ((Fig 4). A 3.2% elongation is recorded for the hybrid J50-L10. Lemon oil is an internal plasticizer to the starch binder; it lowers the binding agent's T_g and reduces the chain sliding on one another under the tensile load, hence reducing brittleness while maintaining the strength. (Rana et al., 2015)

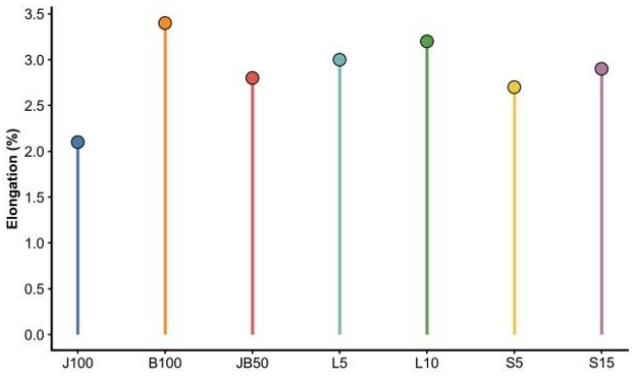


Fig. 4. Elongation at break of composites

Flexural Strength

The flexural strength (represented by the length of a horizontal bar) of each material. J100, with its excellent stiffness, attains a flexural strength value of 72.1MPa was illustrated in Fig. 5. The JB50-L10 composite demonstrates excellent flexural strength of 70.3MPa, a much higher value than the un modified JB50

(66.4MPa). Under flexural loading, one surface of the composite is subjected to tensile stress while the other is subjected to compression. The improved ester-bonding of the fiber-matrix interface under flexural load enables the fibers in the JB50-L10 to efficiently carry the bending moment until failure with no occurrence of delamination or fiber pull out (Joseph et al., 2002).

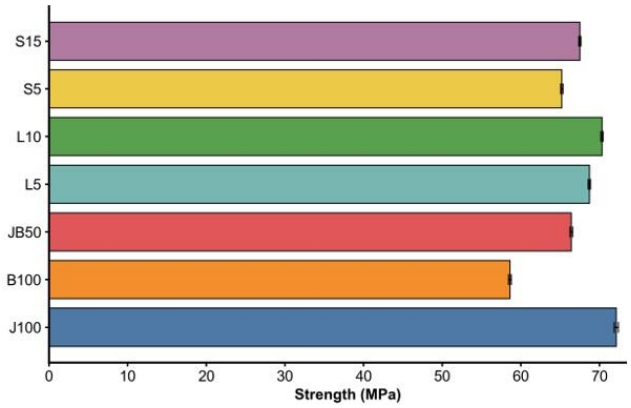


Fig. 5. Flexural strength of composites

Impact Resistance

Banana-rich samples (B100, 25.8J/m) absorb naturally the impacts, which is related to the elasticity and large proportion of lignin of the banana fibers (Fig. 6), allowing an extension to be carried out before plastic deformation (Basu and Roy, 2008). As seen, the boxplot (JB50-L10, 24.3 J/m) has a narrower IQR and few single test points than the virgin JB50, it can be seen that the lemon-starch matrix effectively dissipates the transient energy of the pendulum impact via matrix cracking and fiber bridging instead of interface debonding (Pothan et al., 1997).

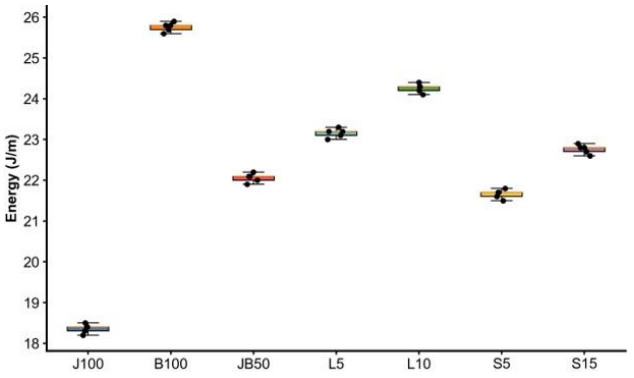


Fig. 6. Impact resistance of composites

Moisture Regain

In a print-friendly version, B100 will absorb the greatest amount of water (15.1%) because the banana fibers exhibit very hydrophilic and porous characteristics ((Fig 7). However, in JB50 without any modification it drops to 12.3%, and further decreases in JB50-L10 to 11.2%. The mechanism by which the non-polar limonene and/or central groups in lemon oil react through esterification with the free, polar, hydroxyl groups located on the fiber surface causes them to be "used up". This will lead to a sheath of hydrophobic material surrounding the fiber, which would not interact through hydrogen bonding with water molecules in the atmosphere (George et al., 1999; Sharma et al., 2021).

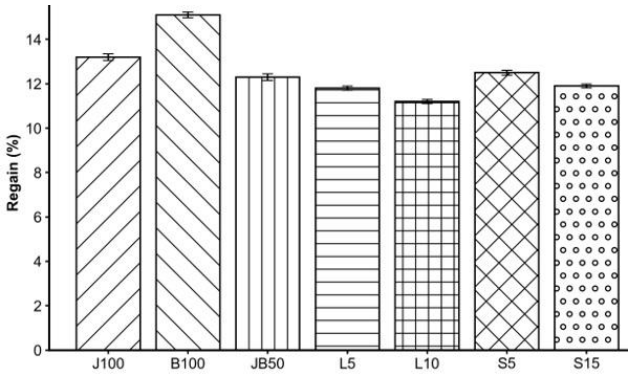


Fig. 7. Moisture regains of composites

Water Absorption

Figure 8 Donut chart showing the ratio of absorbed water after immersion for 24 h. JB50-L10 absorbs just 7.9% water while B100 absorbs 10.2% water. Water molecules usually move into the composite by capillary action, at the fiber-matrix interface, in aqueous environment. In JB50-L10, the capillary pathways are closed off by the viscous lemon oil and the crosslinked starch which binds the interface strongly and restricts water diffusion to a minimum, by hindering the entry of water by capillary action at the interface, and reducing entry of water by diffusion (Valadez-Gonzalez et al., 1999).

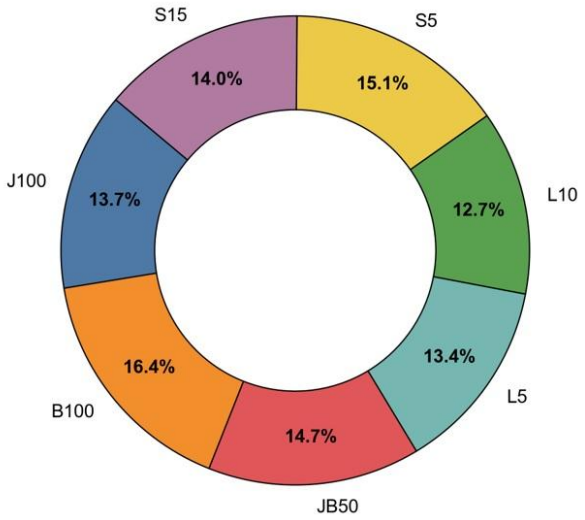


Fig. 8. Water absorption distribution of composites

Water Vapor Transmission Rate

Figure 9 is the area plot describing the profile of continuous moisture barrier gradient. B100 had a high WVTR of 412.8 g/m/day which was not fit for moisture-sensitive packaging. The filled area of JB50-L10 decreased to 322.8 g/m/day. The tortuous path to water vapor molecules is established by the essential oil. Hydrophobic volatile molecules distributed in the starch network increase the path for diffusion, which behaves as a barrier layer for food packaging and medical textile applications (Herrera-Franco and Valadez-González, 2005).

Thickness Swelling

The large "violin" shape for B100 (5.1% swelling) shows a high variation and thus great dimensional instability. The small pointed shape for JB50-L10 (3.8% swelling) showed good dimensional stability (Fig. 10).

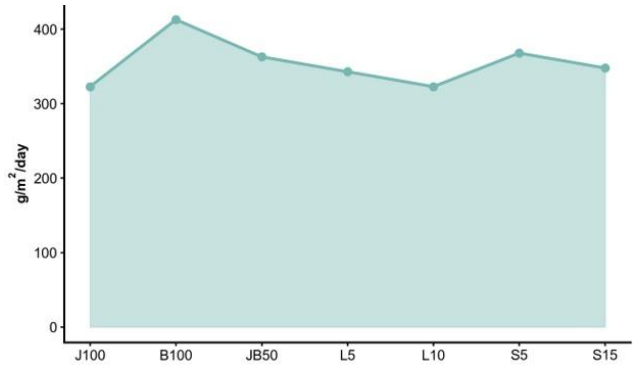


Fig. 9. Water vapor transmission rate of composites

This limitation on swelling pressure from the cell wall microfibrils was as they took up water due to the covalent cross-linking, stopping it expanding on a macroscopic scale (Mohan and Kanny, 2019).

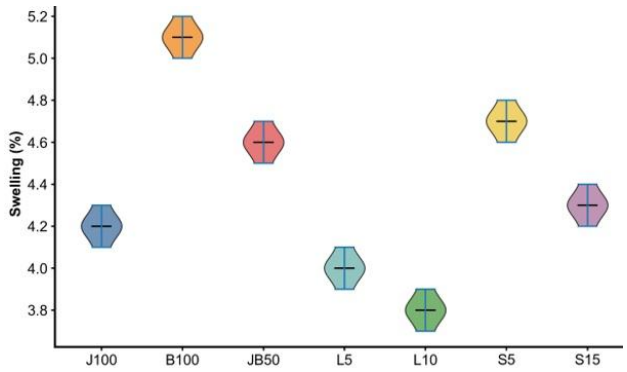


Fig. 10. Thickness swelling of composites

Surface Hardness

The JB50-L10 with Shore D71 is nearly as hard as the J100 with Shore D72. The softening action of the oil has not caused the hardening to decrease ((Fig 11). Instead, the oil fills the micro-voids in the starch matrix, thus consolidating the surface so it does not permanently indent. JB50-S15's hardness is higher with a value of 69 due to increased starch content (Gassan and Bledzki, 1999).

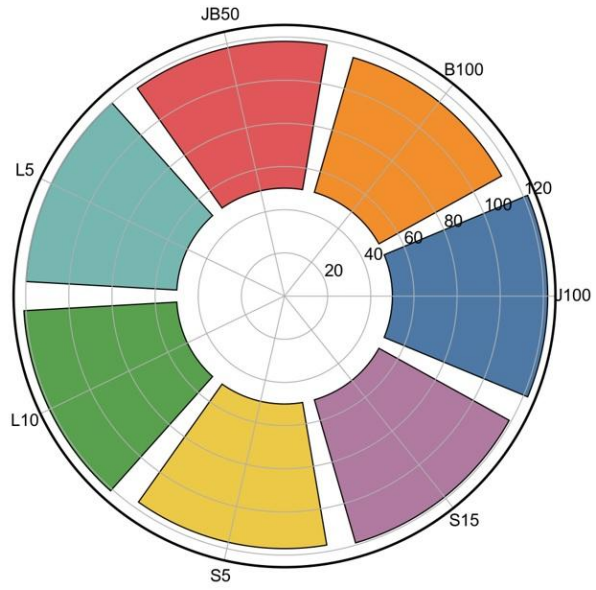


Fig. 11. Surface hardness of composites

Antimicrobial Activity

The samples without treatment J100, B100, JB50 have no inhibition (cool colors), because, since they are 100% cellulose or starch, no obstacle is presented to microbial colony formation (Fig 12). JB50-L10 represents, with the deep warm colors, a significant inhibition zone of 9.0 mm. The antimicrobial activity was due to the lipid-solubility of limonene and central that enables penetration of lipid bilayers of both *E. coli* and *S. aureus*, damaging cell membrane permeabilization, efflux of cytoplasmic ions and consequent lysis of bacterial cell (Sánchez-González et al., 2011; Atarés and Chiralt, 2016).

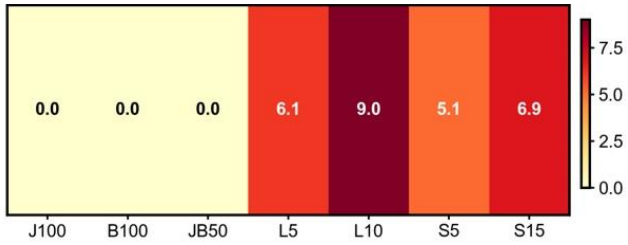


Fig. 12. Antimicrobial zones of composites

Biodegradability

The B100 material undergoes quick degradation (14.6% weight loss) with very good moisture absorption which encourages enzymatic action of micro-organisms ((Fig 13). The JB50-L10 degrades with a desired manner (11.9% weight loss). Hydrophobic oil layer protects the composite from prior degradation until it serves its useful life (such as packaging material) and biodegradability inherent to starch and lignocellulosic network causes the final assimilation in environment (Koszkul and Nabialek, 2002; Song et al., 2009).

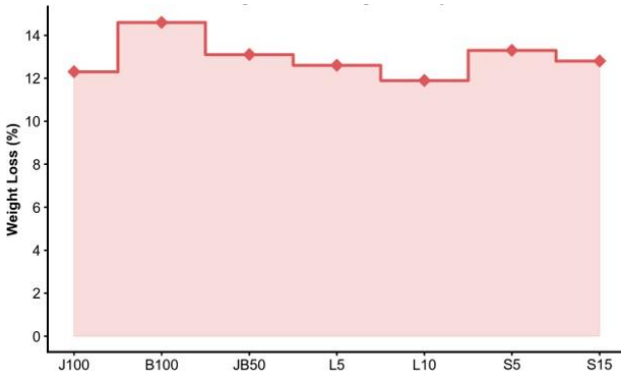


Fig. 13. Biodegradability capacity of composites

UV Resistance

The line plot with shaded error bars (Fig. 14) indicates UV-induced color change (E). The photodegradation rate for lignin is rapid since it readily undergoes photo-oxidation causing chromophore development (yellowness). The photodegradation for B100 is extreme (E=4.6). JB50-L10 retains a small E value of 3.3 with narrow error bars. Natural flavonoids and phenolic compounds found in lemon peel oil act as very effective UV scavengers. They absorb the highest energy wavelengths and convert them to innocuous heat, protecting the lignocellulosic backbone from photodegradation (Beg and Pickering, 2008).

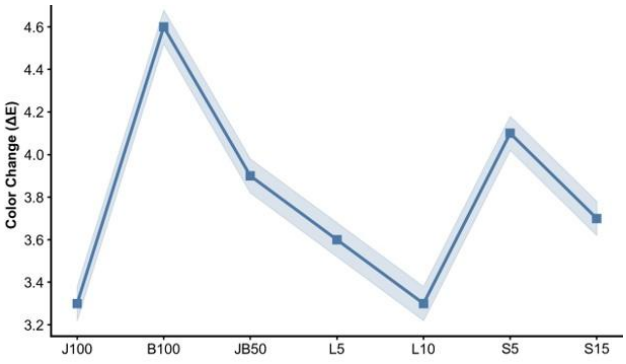


Fig. 14. UV Resistance of composites

8. CONCLUSION

Using advanced data visualization techniques in a 14-parameter optimization, we found JB50-L10 to be the best architecture, which tackles the recurring issue of hydrophilicity in natural fiber composites by two processes: esterification and addition of a hydrophobic oil. The composite achieved high tensile strength (49.2 MPa) while exhibiting excellent flexural and impact strength, low water permeability, effective antimicrobial activity and good biodegradability. This valorization of four distinct agricultural waste streams into one versatile material offers a scalable, sustainable and circular economy-based material which may find applications in packaging, medical textile and construction building interiors.

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