

Rice straw-based sustainable food packaging material with improved strength and barrier properties: Development and characterization

MAKDUD ISLAM, AKHOURI SANJAY KUMAR SINHA, AND KAMLESH PRASAD

ABSTRACT: Sustainable food packaging paper with high barrier and strength properties was developed with rice straw nanocellulose materials. Pulping and bleaching of rice straw were performed using an organosolv pulping and DED (D: chlorine dioxide bleaching; E: sodium hydroxide extraction) bleaching sequence. Bleached rice straw pulp was refined to 90°SR using a laboratory Valley beater. The laboratory handsheets were prepared using pulp slurry at 40°SR and 90°SR. The handsheets of cellulose nanofibrils (CNFs) made of highly refined pulp (90°SR) were surface sized using alkyl ketene dimer (AKD) wax to increase the barrier properties of paper for selective food packaging applications.

The paper samples were tested for mechanical, optical, surface, and barrier properties, including tensile index, burst index, tearing index, bending stiffness, elongation, porosity, apparent density, opacity, Cobb value, water vapor transmission rate (WVTR), oil and grease resistance, and contact angle. The refined pulp (90°SR) was analyzed using field-emission scanning electron microscopy (FE-SEM), and it was observed that the morphology of the developed fibers changes to the nanoscale (<100 nm) for at least one dimension. The particle size distribution of the refined pulp using DLS analyzer also confirmed the cellulose fibers to near nanoscale.

It was concluded that nanofibers were formed by a high degree of the mechanical pulp refining process and found to be much more economical than alternative processes in this direction. The sample handsheets of CNFs showed good strength and barrier properties. The barrier properties further increased when surface sizing was done using low-cost, nontoxic, and biodegradable AKD wax.

Application: The handsheets of CNFs after surface sizing with AKD wax can be used as an alternative to synthetic polymer-based food packaging materials for various foods and allied materials.

Packaging plays a crucial role in the food supply chain because it safeguards food at various stages until its final usage. Food packaging currently comprises roughly 85% of the packaging industry [1]. Since non-sustainable petroleum-derived polymers are widely used in food, pharmaceutical, electronics, and other industries, developing sustainable and recyclable barrier materials for packaging is a crucial challenge brought on by urgent environmental concerns. The petroleum-based polymers used for packaging such as polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) are popular due to their durability, high barrier properties, light weight, and low cost [2]. Despite these advantageous characteristics, most petroleum-based packaging is non-biodegradable and cannot be recycled.

On the other hand, cellulose is renewable, biodegradable, and abundant, and it can be derived from any natural resource. The linear chain of glucose units packed with β -1, 4 glycosidic linkages and consolidated by hydrogen bonds and Van der Waals forces gives cellulose its rigid and stiff

molecular structure [3]. Even though cellulose is naturally hydrophilic, it has an unmatched advantage over non-renewable polymers in the packaging and coating industries due to its abundance, biodegradability, and light weight, and surface hydrophobicity can be developed by adding suitable internal and external surface sizing chemicals [4]. When cellulose fiber is refined with mechanical impact, nanocellulose fibers with low diameter and high aspect ratio are produced, and the three-fold reduction in fiber diameter from micron to nanometer scale comes at a considerable energy cost. Nanocellulose from rice straw biomass has remarkable market potential as a sustainable food packaging material that is highly recyclable and sustainable, with excellent barrier properties [5]. Rice straw is an abundant source of nanocellulosic materials due to its massive cultivation in Asian countries (677 million metric tons) and around the world (765 million metric tons) [6].

In case of high barrier properties, nanocellulose is another viable option. Nanocellulose has a huge specific surface area and the capacity to create inter- and intra-molec-

ular hydrogen bonds, which enable the nanomaterial to build a dense and robust network that is exceedingly difficult for different molecules (such as oxygen and water) to pass through [7]. The resulting nanocellulose can be processed to create films and handsheet papers with excellent mechanical, thermal, moisture, and gas barrier qualities. Thus, nanocellulose is a possible material substitute for the synthetic polymers currently used as barrier materials in packaging applications [8]. However, the barrier properties of the cellulose nanofibrils (CNFs) handsheets are not superior or at par as compared to petroleum-based polymers. So, to increase the barrier properties of CNFs handsheets, the surface sizing process can be used to improve various aspects crucial for packaging paper, such as the bonding capacities with pulp fibers (oxygen, grease, antimicrobial properties, etc.) and surface hydrophobicity. It has been found that surface sizing using chitosan leads to antimicrobial activity [9]. However, alkyl ketene dimer (AKD) is the most popular sizing agent used globally to give paper and paperboard products high barrier properties and long life [10]. A few documents have been published on the application of AKD wax as a coating material for handsheets using CNFs [11,12].

Our present study aims to prepare handsheets of CNFs from highly refined (90°SR) rice straw bleached pulp to improve the barrier (moisture, oil, and grease resistance) and surface (smoothness, porosity, etc.) properties by surface sizing with AKD wax for the preparation of food packaging materials.

MATERIALS AND METHODS

Materials

For this study, rice straw was used as a raw material and was obtained from the local agricultural fields in Sangrur, PB, India. For further processing and analysis, the rice straw was cleaned with water, air dried, and then chopped into small pieces measuring between 0.5 and 1 cm. The AKD wax was procured from PS Polychem (Hari Nagar, DL, India), and n-heptane (Sigma-Aldrich; Bengaluru, KA, India) was used as the solvent for AKD wax.

Pulping and bleaching

A previously optimized oraganosolv (acetic acid) treatment was carried out for rice straw (RS) delignification with slight modification [13]. It was then followed by a 3-stage DED (D: chlorine dioxide [ClO₂]; E: sodium hydroxide [NaOH] extraction) bleaching sequence with ClO₂ = 2.5%, NaOH = 3%; and ClO₂ = 1.5%, respectively. This bleaching sequence was performed with sodium chlorite (NaClO₂) and NaOH at a pulp consistency of 10% (w/v) at a temperature of 70°C for 1 h for each bleaching stage to increase the brightness of unbleached pulp (UBP). Then, RS bleached pulp was dried for further application or further analysis.

Refining of bleached pulp

The RS bleached pulp (BP) was diluted to 4% (w/v) consistency and further refined in a Valley beater (Universal Engineering Corp.; Saharanpur, UP, India) at a 2 kg load condition and ambient temperature to obtain pulp of 40°SR and 90°SR, respectively. Finally, the refined pulp of 40°SR and 90°SR were stored at 4°C in a sealed container to avoid contamination and until further application.

Morphology and particle size distribution

The morphology of different refined pulp samples was analyzed using S-3400 N Type II field emission-scanning electron microscope (FE-SEM) from Hitachi (Tokyo, Japan), and a Litesizer 500 dynamic light scattering (DLS) analyzer from Anton Paar (Graz, Austria) was used to measure the particle size distribution. The Brownian motion that is produced among the particles due to the continuous flow of molecules was utilized here. Laser light is used to illuminate these particles' motions, and intensity fluctuates at a rate based on the particle size.

Handsheet manufacturing and required tests

Laboratory handsheets (100 g/m² grammage) were prepared according to TAPPI Standard Test Method T 275 sp-02 "Screening of pulp (Somerville-type equipment)" using different grades of refined pulp. However, the handsheets of CNFs were made using vacuum filtration methods, where highly refined pulp at 0.5% consistency was placed in a filter paper (Watman grade 41) over a Buckner funnel. The sheets of CNFs were dried for 2.5 h at 100°C and subsequently kept for 6 h at 55°C. All handsheets were incubated at the temperature of 23 ± 1°C and 50 ± 2% relative humidity as per TAPPI T 402 sp-08 "Standard conditioning and testing atmospheres for paper, board, pulp handsheets, and related products."

The surface properties of handsheets were measured as follows:

- Smoothness and porosity: Smoothness and porosity were measured by Gurley S-P-S (smoothness, porosity, and softness) testers using TAPPI T 460 om-11 "Air resistance of paper (Gurley method)."
- Brightness and opacity: Opacity was measured according to TAPPI T 415 om-11 "Opacity of paper," and brightness was measured by brightness tester (temperature: 28°C; relative humidity: 40%) as per TAPPI T 452 om-18 "Brightness of pulp, paper, and paperboard (directional reflectance at 457 nm)."

The mechanical properties of handsheets were measured as follows:

- Tensile index: TAPPI T 494 om-01 "Tensile properties of paper and paperboard (using constant rate of elongation apparatus)."
- Tear index: TAPPI T 414 om-4 "Internal tearing resistance of paper (Elmendorf-type method)."

- Tensile energy absorption (TEA) index and elongation: TAPPI T 494 om-01 via Universal testing machine (Universal; Saharanpur, UP, India).
- Burst index: TAPPI T 403 om-02 “Bursting strength of paper,” using burst tester from Universal at temperature of 25°C and relative humidity of 40%.
- Bending stiffness: TAPPI T 489 om-08 “Bending resistance (stiffness) of paper and paperboard (Taber-typed tester in basic configuration),” using Taber-type tester from Universal.
- Barrier properties (water absorptiveness): ISO Standard 535 “Paper and board — Determination of water absorptiveness — Cobb method.”
- Oil and grease resistance: TAPPI T 559 cm-12 “Grease resistance test for paper and paperboard,” using the kit test.
- WVTR: ASTM E96/E96M-05 “Standard test methods for water vapor transmission of materials,” using anhydrous calcium chloride (CaCl₂).
- Contact angle: via sessile drop method.

Surface sizing of CNFs handsheets

In this study, AKD wax was dissolved in n-heptane at 95°C to prepare the AKD solution at 5% (w/v), stirred for 10 min before use, and diluted to 1% using deionized water. The AKD wax solution was applied in handsheets of CNFs with a dipping method followed by a previous study [14,15]. The AKD wax solution was applied based on the dry weight of the sample sheets. Based on a previous study by [16], 0% and 1% AKD wax solution was used to absorb the sample handsheets. After dipping in the AKD wax solution, the sheets were dried for about 24 h at room temperature before re-weighting. Then, AKD wax-treated handsheets were dried in a rotary dryer at 105°C for 5 min. Finally, the AKD wax-treated handsheets were incubated for 24 h as per TAPPI T 402 sp-08 to allow handsheets to reach the equilibrium moisture conditions. A rotating dryer was employed (drum speed 15, with automatically controlled temperature), with a contact time of approximately 5 min.

RESULTS AND DISCUSSION

Chemical composition of bleached and unbleached pulp

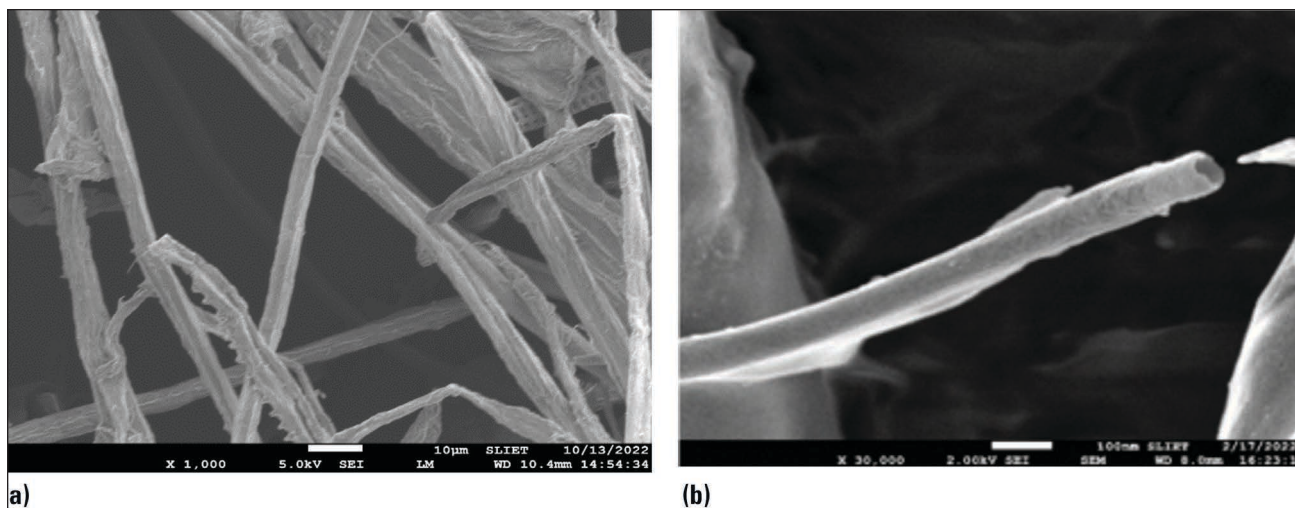
Table I shows the chemical composition of rice straw, rice straw bleached pulp, and rice straw unbleached pulp. There seems to be a significant increase in holocellulose and alpha cellulose by 57.65% and 90.50%, respectively. In contrast, there is a drop in Kalson lignin from 17.50% to 1.9% for bleached rice straw pulp. Silica content was also reduced from 13.9% to 6.5%. As seen from the table, most lignin was removed from the rice straw, whereas holocellulose and alpha cellulose increased significantly after the chemical treatment of rice straw pulp.

Rice straw fiber morphology

The FE-SEM image of bleached rice straw fiber with different refining levels (40°SR and 90°SR pulp) is shown in **Fig. 1**. The refining of pulp at 40°SR shows less fibrillation compared to the highly refined pulp. The average diameter of the fiber is about 4–6 μm, whereas highly refined fiber shows a fiber diameter of less than 100 nm. As per the standard definition of nanomaterials, if one dimension of a particle is less than 100 nm, it is termed a nanomaterial. So, these fibers can be termed nanofibers. The paper handsheets made from these nanofibers can be addressed as handsheets of cellulose nanofibrils. Refining of pulp in a Valley beater causes significant changes in fiber morphology due to external and internal fibrillation, along with shortening of fibers. The increase in the degree of refining of pulp also causes a decrease in freeness due to the absorption of water in the cell wall. External and internal fibrillation causes branching of fibers and swelling, respectively. So, highly refined pulp up to 90°SR results in significant disintegration of fibers and production of nanosized fibers. This finding is supported by some similar trends shown by another research group for bagasse, hardwood, and softwood pulps [17].

Analysis	Rice Straw, %	Rice Straw Unbleached Pulp, %	Rice Straw Bleached Pulp, %	TAPPI Standard Test Methods
Alpha cellulose	38.5	60.5	73.5	TAPPIT 429 cm-10 “Alpha-cellulose in paper”
Holocellulose	55.5	76.8	87.5	Browning method
Kalson lignin	17.5	4.2	1.9	TAPPIT 222 om-98 “Acid insoluble lignin in wood and pulp”
Silica	13.9	9.5	6.5	TAPPIT 211 om-02 “Ash in wood, pulp, paper, and paperboard: Combustion at 525°C”

I. The chemical analysis of bleached and unbleached pulp.



1. Rice straw fiber morphology at (a) 40°SR and (b) 90°SR.

Particle size analysis

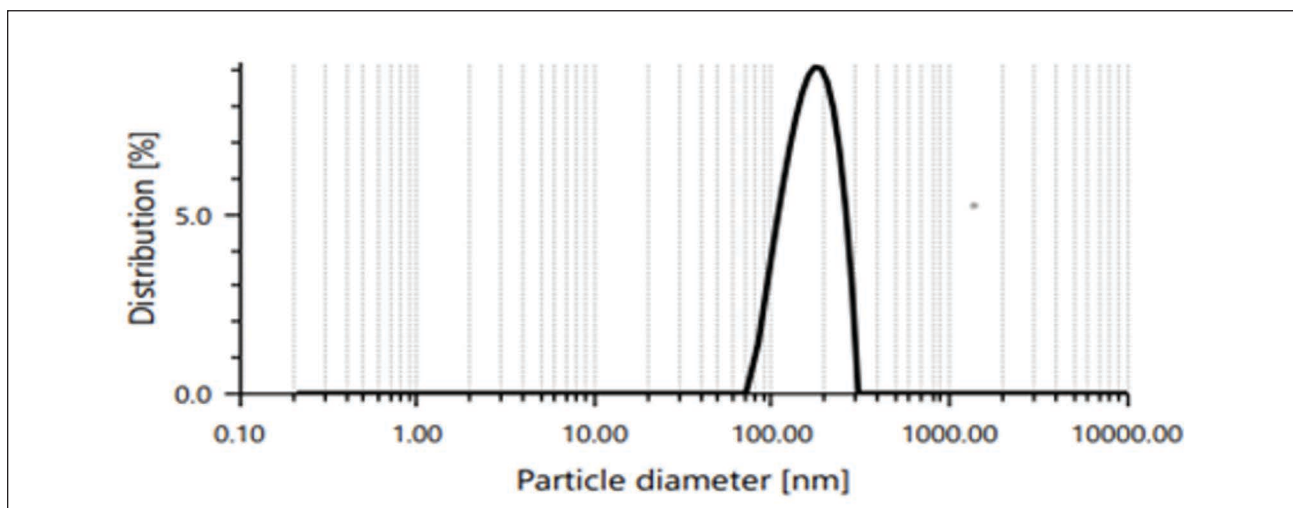
The particle size distribution of the highly refined pulp was measured using a DLS analyzer observing the Brownian motion of particles. **Figure 2** reflects that nearly 20% of particles (by volume) are less than 100 nm particle size, with the average particle size in the range of 150–160 nm. The FE-SEM analysis further confirms that at least one dimension of the developed fiber is in nano range. A similar kind of trend was observed when the bagasse, softwood, and hardwood pulps were refined with a laboratory PFI mill up to 90°SR [17].

Surface and optical properties of handsheets

Table II shows the surface and optical properties of handsheets made from bleached RS pulp at different refining conditions of 40°SR, 90°SR, and 90°SR pulp with AKD wax added, respectively. The optical properties, such as scattering coefficient and opacity of the refined (90°SR) CNFs handsheets decreased drastically. As the extent of refining

is increased, the transmittance percentage also increases, and as a result, the opacity of the refined pulp is decreased by 2.17 times. This could be due to the reduction of fiber diameter from microscale to nanoscale, which is below the visible range of 400–700 nm. However, comparatively less refined RS fiber (40°SR), with diameter in micron scale, can scatter some or all visible light when it passes through the pulp. The decrease in the specific scattering coefficient of the CNFs sheets is due to the reduction of surface scattering caused by the significant increase in the number of fiber-fiber bonds. A similar result was observed for the handsheets of CNFs obtained from bagasse pulp [18].

When CNFs handsheets were surface sized with AKD wax, the opacity and scattering coefficient increased, as AKD wax made an extra layer on the sample handsheets, enhancing the opacity and scattering coefficient. This highly refined pulp also observed a significant increment of smoothness and porosity reduction. This is mainly due to an increase in the apparent density of the CNFs sheet. The



2. Particle size distribution of refined fibers (90°SR) by dynamic light scattering (DLS) analyzer.

Treatments	Apparent Density, kg/m ²	Scattering Coefficient, m ² /kg	Opacity, %	Smoothness, s/100 mL	Porosity, s/100 mL
40°SR	360	26.5	98.3	19.0	8.78
90°SR	570	3.0	45.3	60.4	Infinity
90°SR + AKD wax	500	4.5	46.5	62.4	Infinity

II. Surface and optical properties of different cellulosic paper.

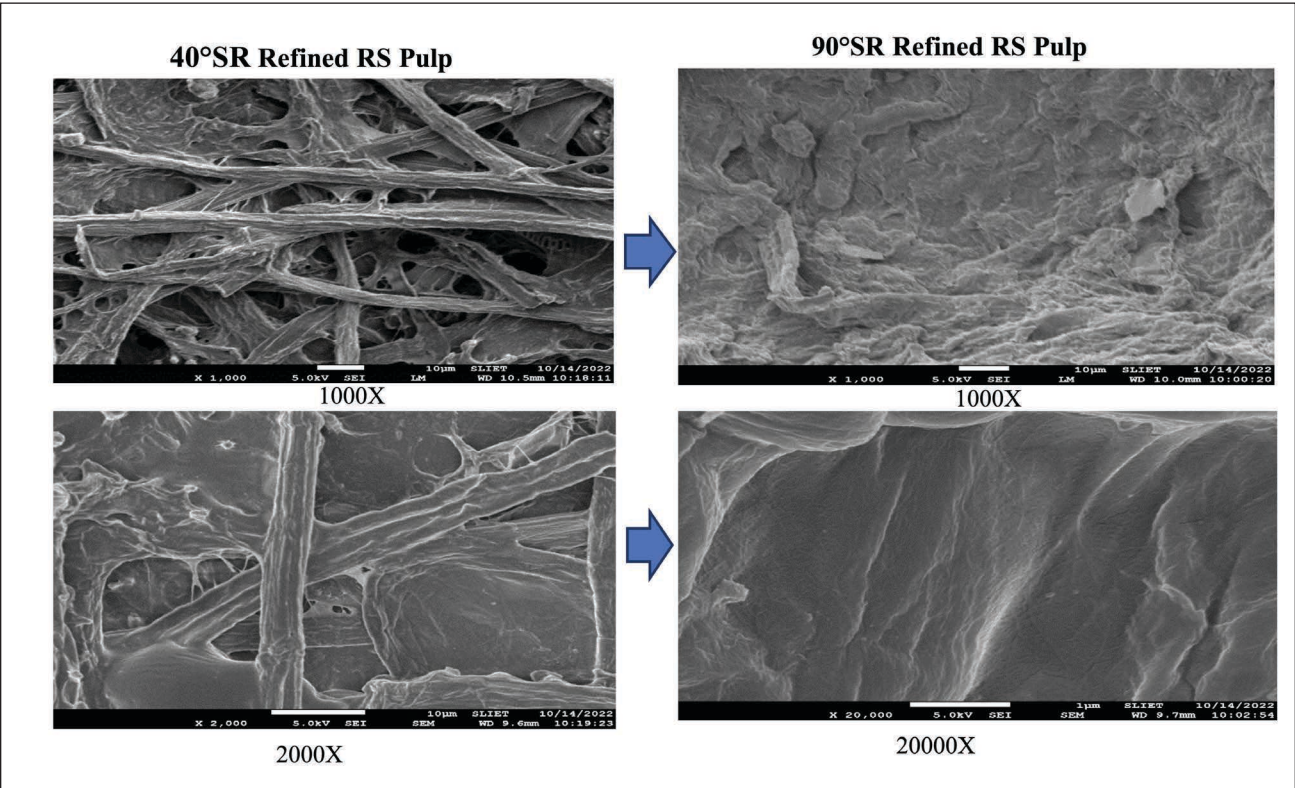
permeance of the sheet was measured by the Gurley porosity test, and porosity was found to be infinity for the handsheets of CNFs. Results show that CNFs handsheets made from rice straw were practically impervious to the air passage. The FE-SEM images in **Fig. 3** of CNFs handsheets shows a very dense, compact structure and has less air passage compared to the paper made from 40°SR pulp. Also, the smoothness was slightly increased, and porosity decreased when CNFs handsheets were treated with an AKD wax solution. Another research group showed a significant improvement in surface smoothness and a decrease in porosity when handsheets were surface sized with cationic starch [19].

Mechanical properties of handsheets

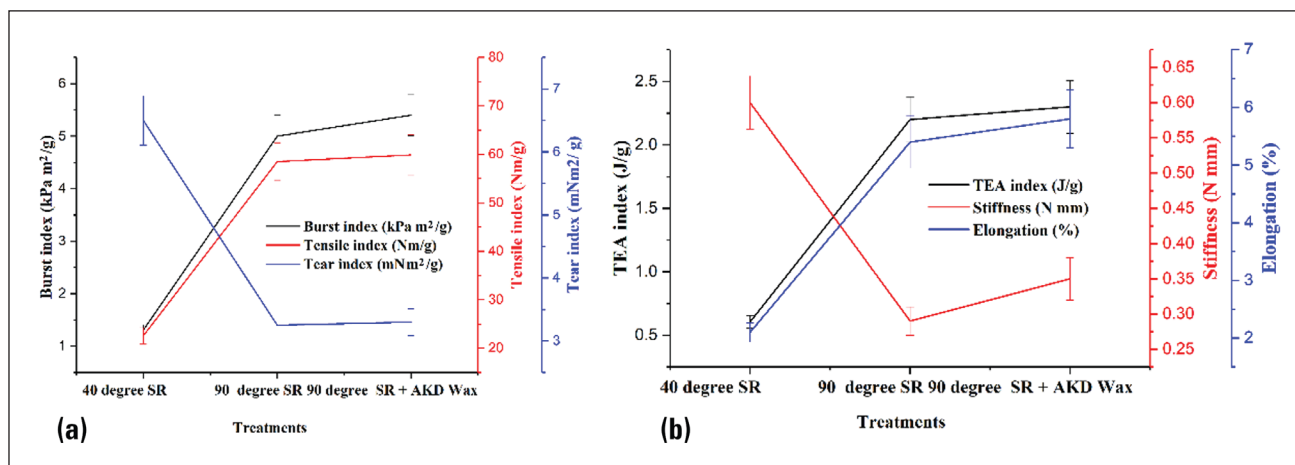
The mechanical properties such as burst index, tensile index, tear index, TEA index, bending stiffness, and elongation were evaluated for the cellulosic handsheets (40°SR),

CNFs handsheets (90°SR), and CNFs handsheets surface sized with AKD wax (90°SR + AKD wax). The refining process significantly affects the fibers through internal and external fibrillation, and as a result, there is a decrease in fiber width and an increase in overall surface area. When the pulp refining reached up to 90°SR, tensile and burst indexes increased by 2.6 and 3.8 times, respectively (**Fig. 4**) due to an increase in the specific surface area of fibers that occurs during the refining process, and this is caused by swelling and fibrillation of fibers. This increase in the surface area per unit mass of fibers provides better overlapping/interaction of fibers and results in a higher number of intra-inter hydrogen bonding in CNFs handsheets [20].

However, the tear index was decreased for the highly refined pulp (90°SR) due to the shortening of fibers, and the TEA index increased as fiber-fiber bond strength increased. The stiffness of the nanopaper also decreased sig-



3. Surface morphology of handsheets from rice straw (RS) pulp at 40°SR and 90°SR.



4. Mechanical properties of different degrees of refined cellulosic paper (AKD: alkyl ketene dimer).

nificantly. This is because CNFs handsheets show less bending stiffness or more flexibility compared to the handsheet beaten up to 40°SR, and stiffness is inversely proportional to the cube of paper density. However, the elongation decreased as the degree of refining increased up to 90°SR, but CNFs handsheets surface sized with AKD wax showed no significant effect on the elongation property.

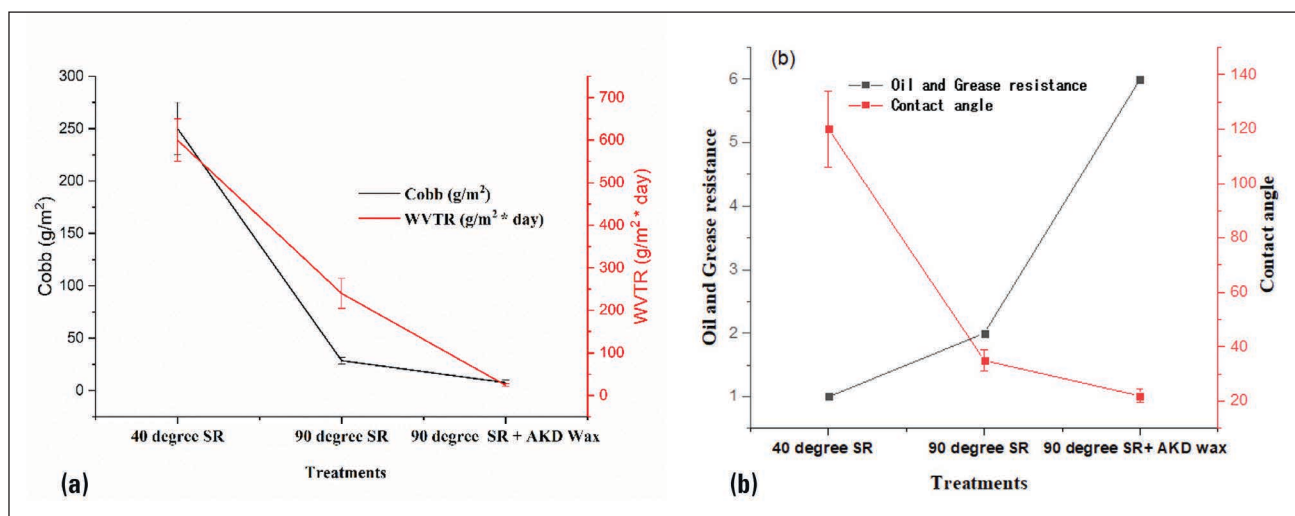
A similar result was identified by [21], who found that beached eucalyptus kraft handsheets of CNFs increase their structural properties compared to less refined pulp (40°SR). Furthermore, the handsheets of CNFs surface sized with the AKD wax also increased the burst index, as AKD wax filled the pores of the paper and simultaneously formed a coating on the surface.

Barrier properties of handsheets

Excellent barrier properties are crucial for good packaging material. The barrier properties of the different cellulosic paper (40°SR, 90°SR, and 90°SR + AKD wax) were evaluated through different tests, as described earlier. These tests

include a water absorption test (ISO Standard 535 Cobb30 test), WVTR (ASTM E96/E96M-05), oil and grease resistance test (TAPPI T 559 cm-12), and contact angle (sessile drop method). The Cobb value served as a gauge for resistance to water penetration.

As shown in **Fig. 5a**, with increased refining of RS pulp, the Cobb value and WVTR of the paper decreased significantly. The handsheets of CNFs (90°SR) show less air permeance due to the denser structure of nanofabricated fibers, as seen in a magnified SEM image at 20,000X in comparison to the porous structure found at 2,000X for 40°SR cellulosic paper (Fig. 3). When the CNFs handsheets were further surface sized with the AKD wax solution, the water absorption capacity (Cobb value) and WVTR dramatically decreased. These findings imply that even in situations where there have been few opportunities for chemical reactions involving the diketene group, the hydrophobicity of the AKD material and its capacity to form a film may be sufficient to impart hydrophobicity to paper. A similar kind of result was reported by [22], where they



5. Barrier properties of different degrees of refined cellulosic paper (AKD: alkyl ketene dimer; WVTR: water vapor transmission rate).

used both wet-end additives and coatings of AKD wax and cationic starch. They found that both methods show a significant improvement in barrier properties of cellulose paper made from secondary fiber material, such as corrugated boxes and handsheets of corrugated boxes.

The barrier properties of CNFs handsheets and surface-sized handsheets were also evaluated with oil and grease resistance tests and contact angle. As seen from **Fig. 5b**, surface sizing with AKD wax improved the water resistance of treated papers, which was measured by the contact angle test. There was clear evidence of increasing hydrophobicity with increasing °SR of cellulosic paper. Maximum hydrophobicity is due to the associated minimum contact angle for nanocellulose paper treated with AKD wax. Higher oil and grease resistance was associated with treating with AKD wax solution. These findings are supported by the earlier observation where the cellulosic paper was coated with an AKD wax composite mixture [22].

CONCLUSION

The present study explores the delignification of rice straw and refining up to 90°SR to prepare the CNFs handsheets and surface sizing with 1% AKD wax solution to improve the barrier properties, surface properties, and hydrophobicity. The CNFs handsheets (90°SR) made from rice straw bleached pulp improve the mechanical (tensile index, burst index, TEA index, bending stiffness) and surface and barrier properties, while tear index and opacity decreased significantly. However, barrier properties and surface hydrophobicity of CNFs handsheets were further significantly improved by surface sizing with 1% AKD wax solution,

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which was confirmed by different tests, including WVTR, oil and grease resistance test, contact angle, and Cobb test. However, the mechanical property was not significantly improved after treatment with the AKD wax solution. The improved barrier property of developed RS CNFs handsheets treated with AKD wax solution can thus be used as an alternative to traditional petroleum-based packaging materials. **TJ**

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ABOUT THE AUTHORS

This topic is about the utilization of agroresidues that cause a serious problem due to the stubble burning in India and other Asian countries. In this research, rice straw pulping and bleaching was performed using the previously optimized organosolv pulping method.

The most difficult part of this paper was the uniform surface sizing of cellulose nanopaper with AKD wax. It was resolved through several trials using a bar-type surface coater at laboratory scale.

We found that nanocellulose can be produced from bleached rice straw fibers through high refining in a Valley beater. It was interesting to find that cellulose nanopaper surface sized with AKD wax showed significantly improved barrier properties.

Because the barrier properties of paper materials prepared with AKD surface sizing were so improved, this research work is very interesting and significant for the pulp and paper industry, as well as food industries throughout the world. This cellulosic nanopaper surface sized with AKD wax can be used as an



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alternative to synthetic polymer-based food packaging materials for various foods and allied materials.

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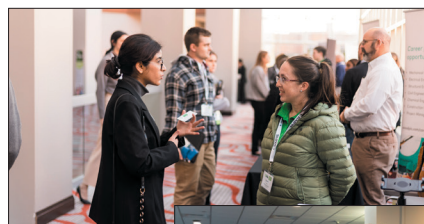
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