Lignocellulose Nanofiber and Starch for Surface Application on Recycled Linerboard

Ghazal Hasanpour,[a] Amir Khosravani,[b,*] Mehdi Tajvidi,[c] and Pejman Rezayati Charani [d]

In a paper production line, starch is widely used for surface treatment and strengthening of linerboard at a size press. Also, the application of lignocellulose nanofibers (LCNFs) is growing because of its relatively low production energy demand, cost, and less hydrophilic nature in comparison to lignin-free nanofibers. Therefore, the addition of LCNFs to starch for paper surface treatment to reinforce the starch film and improve certain physical and mechanical properties of recycled linerboard was investigated in this work. Various LCNF/starch ratios were homogenized and then applied on the paperboard surface. The results revealed that a low mixing ratio of LCNF (5%) with starch improved the tensile index of the recycled paperboard, and at 50% LCNF content in the surface-treating material, film forming on the linerboard was observed in field emission-scanning electron microscopy images. In the case of 95% LCNF addition to starch, bending stiffness was significantly increased. Additionally, the viscosity of the sizing suspension was studied as a crucial parameter in the process and was found to increase significantly following the addition of LCNF.

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*Contact information: a: MSc graduate, Department of Wood and Paper Science and Technology, Faculty of Natural Resources, Tarbiat Modares University, 4641776489 Mazandaran, Noor, Iran; b: Department of Wood and Paper Science and Technology, Faculty of Natural Resources, Tarbiat Modares University, 4641776489 Mazandaran, Noor, Iran; c: School of Forest Resources, Advanced Structures and Composites Center and Forest Bioproducts Research Institute, University of Maine, Orono, ME, USA; d: Department of Wood and Paper Science and Technology, Faculty of Natural Resources, Behbahan Khatam Alanbia University of Technology, Iran; *Corresponding author: khosravani@modares.ac.ir*

INTRODUCTION

Various chemicals are used in paper production lines to improve the product quality or process parameters. These chemicals can be added into the furnish prior to the paper sheet forming process or through paper surface treatment (Maurer 2009; Shen *et al.* 2011).

As a common group of surface treatment chemicals, surface sizing materials are generally applied to inhibit water absorption to the paper, while filling the paper surface pores and voids and improving the strength properties (Maurer 2009; Moutinho *et al.* 2009). Various starch derivatives, some hydrophobic materials, and synthetic polymers are common sizing chemical ingredients for such purposes (Koskela and Hormi 2003; Laleg 2004; Lertsutthiwong *et al.* 2004; Mesic *et al.* 2004; Moutinho *et al.* 2007).

For surface treatment, a variety of starches, such as oxidized, native, cationic, and anionic starches, are used. Oxidized starch has excellent film-forming properties for surface treatment (Maurer 2009). In contrast, the use of cationic starch is increasing, as it results in oxygen demand reduction in the effluent by interacting with fibers and anionic fillers, leading to a significant reduction of suspended solids and chemical oxygen demand (COD) in the wastewater of the paper mill (Maurer 2009).

In contrast, the use of nanocellulose for packaging and surface treatment of paper has been widely studied (Olsson *et al.* 2011; Lavoine *et al.* 2012; Paunonen 2013; Brodin *et al.* 2014; Cowie *et al.* 2014; Bradet and Bras 2015; Li *et al.* 2015; Hubbe *et al.* 2017; Vaezi *et al.* 2019; Sharma *et al.* 2020). Shatkin *et al.* (2014) stated that nanocellulose has great potential for use in paper and packaging applications. Meanwhile, in recent years, the demand for paper and board packaging materials has increased as an alternative to plastic materials, which have an extremely long biodegradation period (Olsson *et al.* 2011; Paunonen 2013).

Yang *et al.* (2014) reviewed the surface sizing using cationic starch and nanocrystalline cellulose. The results showed an increase in mechanical properties, including tensile index, tear index, bending stiffness, and burst. At a low coating weight, CNF coatings improve the strength properties. Further, paperboard with CNF coating shows excellent barrier properties to air, grease, and oil, and the water vapor transmission rate (WVTR) is significantly improved (Kumar *et al.* 2016).

Some researchers have proposed a mix of nanofiber / nanocrystalline cellulose with cationic starch in a coating formulation can work as enhancer agents for mechanical, air-resistance, and optical properties of paper (Cheng *et al.* 2017; Veazi *et al.* 2019; Sharma *et al.* 2020; Fidan *et al.* 2021).

Among cellulose nanomaterials, recently extensive research has been conducted on the production and some applications of lignocellulose nanofibers (LCNFs) (Gu *et al.* 2019; Yousefhashemi *et al.* 2019; Amini *et al.* 2020; Solala *et al.* 2020; Tayeb *et al.* 2020). The LCNFs are delicate fibrillar lignocellulose structures, generally with nano-size diameter and a couple of micrometers length. In comparison to cellulose nano-fibers (CNFs) that are produced from bleached pulp, LCNFs can be isolated from lignin-containing fibers such as thermo-mechanical pulp (TMP), unbleached chemical pulp, or recycled old corrugated container (OCC) pulp (Gu *et al.* 2019; Yousefhashemi *et al.* 2019; Tayeb *et al.* 2020). Following successful isolation and application of lignocellulose nanofibrils from such source materials for various end uses, there is no need for complete delignification, purification, and yield reduction (Bardet and Bras 2014; Delgado-Aguilar *et al.* 2016). Amini *et al.* (2020) reviewed physical and mechanical properties of LCNF and CNF films and proposed that the use of LCNF could provide an opportunity to reduce costs.

Tayeb *et al.* (2020) used an LCNF layer for coating the packaging paper. The results showed the ability of LCNF layer against penetration of cooking oils for 5 months and improvement in tensile strength.

In LCNFs, the lignin inherently possesses a hydrophobic nature. LCNFs can also be used to improve the mechanical properties and can be used to improve the quality of starch film in surface treatment (sizing/coating) process on a brown recycled linerboard. The LCNF and the recycled linerboard are of similar brown color. Therefore, in this research, the effect of LCNFs (isolated from recycled OCC fibers) in a mixture with starch on surface properties and enhancement of certain physical and mechanical properties of the produced recycled linerboard was investigated.

EXPERIMENTAL

Materials

In this study, as a base paper, a recycled linerboard roll was provided by a local paper mill (Mazandaran, Iran). In the production process of the mentioned paper roll, no additive or sizing material or calendaring process had been applied. Cationic quaternary tapioca starch (degree of substitution: 0.018 mol/mol) was provided from Siam Modified Starch Co. (Thailand).

Lignocellulose nanofibers (LCNFs) were produced from recycled linerboard by an ultra-fine grinding method (MKCA6-2; Masuko Co., Japan) by three passes, at 1800 rpm, while disk distance was less than 600 µm. The fibril diameter range of the isolated LCNFs were 10 to 80 nm (mostly 30 to 50 nm), and the appearance was in the form of brown LCNF gel (Nano Novin Polymer Co., Mazandaran, Iran) that was kept in refrigerator at 4 °C. More details about the isolated LCNF from the recycled linerboard were described in Yousefhashemi *et al.* (2019).

Methods

Preparation and application of surface treating material

To prepare cationic starch (CS), the starch suspension (different concentrations: 1 to 8% (w/w)) was kept stirring and gently heated up to 90 °C for 30 min. Then, it was kept at this temperature for 30 min. Finally, the solution was cooled to ambient temperature and used for the experiments.

If required, LCNF (4%, w/w gel) was added to the starch (4%, w/w) solution. The addition of LCNF to CS of high concentrations (more than 4%) resulted in high viscosities that made the evenly distributed application of sizing impossible. Therefore, LCNF gel was only applied in mixture with CS: 4% (Table 1). The mixing ratios of LCNF in total solid material of sizing were 5, 50, and 95%, as presented in Table 1.

Table 1. Ingredients of Various Sizing Material Mixtures

No.	Sizing Material Mixtures	Code
1.	Control (untreated base paper)	Control
2.	Cationic starch (CS) with concentration of 1% (w/w)	CS 1%
3.	CS with concentration of 2% (w/w)	CS 2%
4.	CS with concentration of 4% (w/w)	CS 4%
5.	CS with concentration of 6% (w/w)	CS 6%
6.	CS with concentration of 8% (w/w)	CS 8%
7.	Mixing ratio: 5% LCNF with 95% CS (based on solid content). Concentrations of CS and LCNF were both 4% (w/w)	CS 4+LCNF 5%
8.	Mixing ratio: 50% LCNF with 50% CS (based on solid content). Concentrations of CS and LCNF were both 4% (w/w)	CS 4+LCNF 50%
9.	Mixing ratio: 95% LCNF with 5% CS (based on solid content). Concentrations of CS and LCNF were both 4% (w/w)	CS 4+LCNF 95%

As the fibrillated cellulose materials show shear-thinning behavior, all the resulting mixtures were homogenized with a homogenizer for 1 min to have the same pre-shear

condition. Then, the suspensions were tested for viscosity with a Wiggen Hauser, D-500 viscometer (Germany). The homogenized mixtures were spread uniformly and similar to a size press. The paperboard with the applied film was placed under constant linear pressure of about 500 g/cm by rolling between two metal surfaces. Then, the treated paperboard samples were fixed for drying in TAPPI drying rings (according to TAPPI T205 sp-02 2002) for 24 h at 40 °C in an oven equipped with ventilation system.

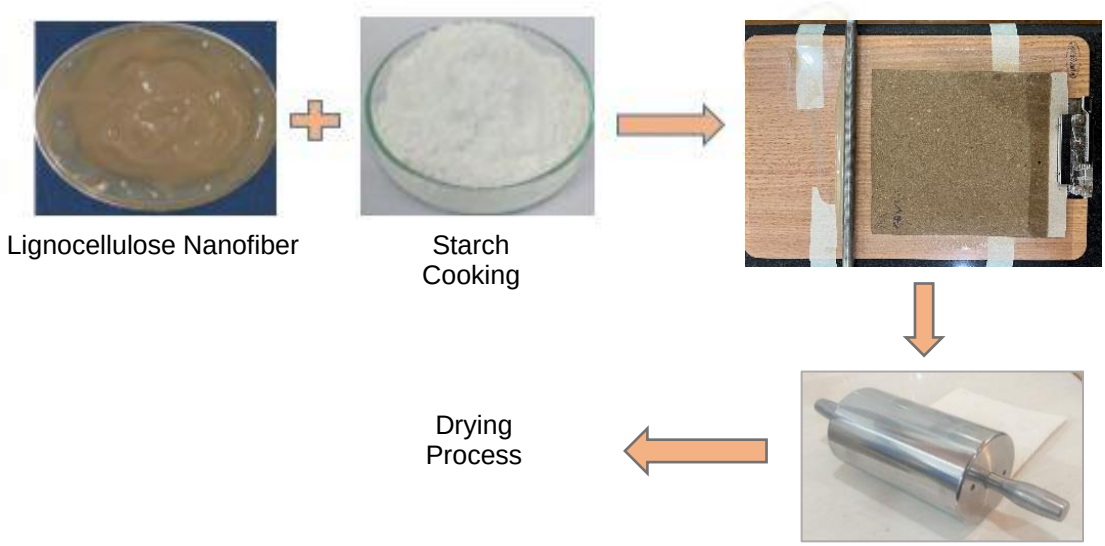

Fig. 1. Surface application of LCNF and starch on recycled OCC paperboard

Evaluation of the paper samples

Field emission scanning electron microscope (FE-SEM) images were taken on a ZEISS FE-SEM microscope, with voltage of 5 kV, at various magnifications. For this purpose, prior to FE-SEM imaging, the paper specimens were coated with a gold sputtering technique.

Tensile, tear, bending indices, and ring crush values were obtained according to TAPPI T494 om-01 (2001), TAPPI T414 om-04 (2004), SCAN-P 29:95 (1995), and TAPPI T818 cm-97 (1997), respectively.

Contact angle of water droplet on the sample surface was evaluated using a PG-X Goniometer (FIBRO System AB, Stockholm, Sweden) with a droplet volume of 30 µL, at 23 °C and 50% relative humidity (RH). Additionally, Cobb test results were collected according to TAPPI T441 om-04 (2004).

Statistical analysis

To identify the statistically significant variances, the analysis of variance (ANOVA) method was used (SPSS software, SPSS Inc., Version 16.0, Chicago, IL, USA). The data were presented as mean ± standard error, and if required, the mean comparisons were done using Duncan's multiple range test at 95% confidence level. Small alphabet letters in the charts indicate the results of the comparisons. The same letters show there is no statistically significant difference between the means and those that are in the same group.

RESULTS AND DISCUSSION

FE-SEM micrographs

The FE-SEM images of test specimens following various surface treatments are shown in Fig. 2. The micrographs of the surface of paper samples showed that the sizing materials were successful to cover the pores and voids on the surface, especially in the case of CS containing 50% LCNF (CS 4+LCNF 50%), while surface treatment by CS 4% resulted in no significant film forming on the surface (Fig. 2). This observation can be attributed to the rheological behavior of the sizing mixture, as the addition of LCNF to CS increased the viscosity significantly (Fig. 3). Further, this shows that LCNF addition to the sizing mixture improved film-forming properties of sizing material on the paperboard. Figure 2 demonstrates that the addition of 50% LCNF (CS 4 + % 50 LCNF) helped form a rather complete film on the paperboard surface.

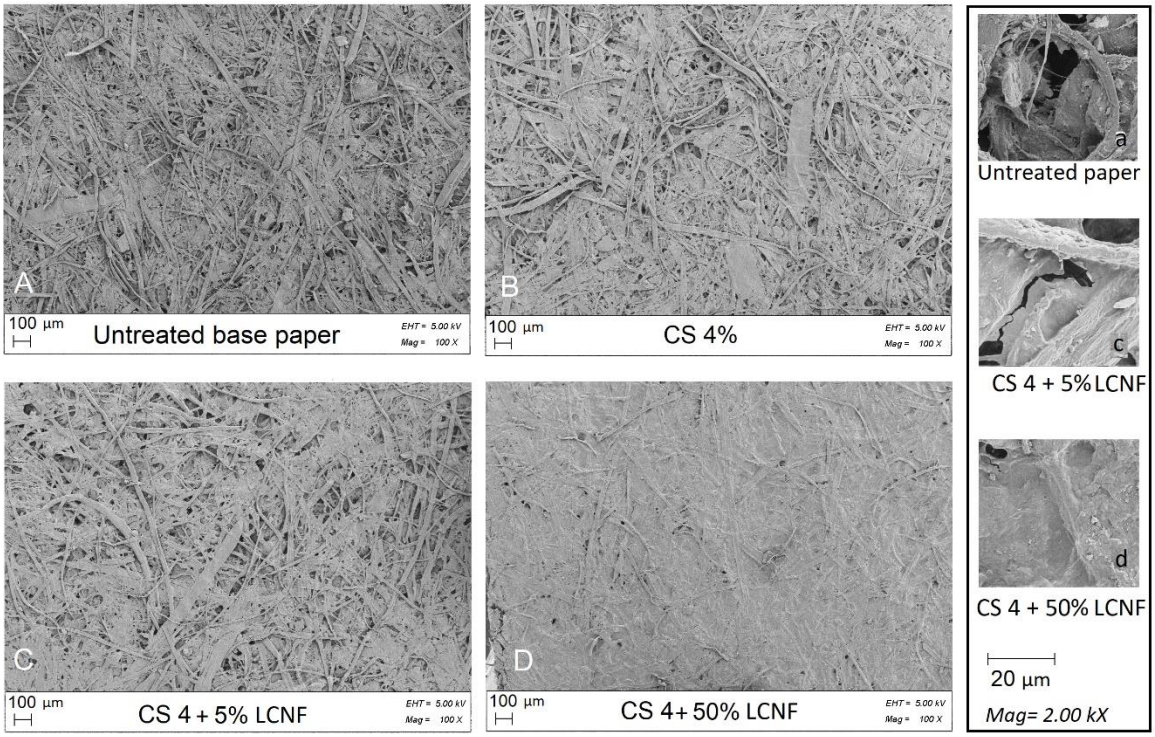

Fig. 2. The FE-SEM images from the surface of recycled linerboard following application of various sizing mixtures (A: untreated base paper; B: surface treated by CS 4%; C: surface treated by CS 4 + LCNF 5%; D: surface treated by CS 4 + LCNF 50%; a, b and d are the same surface as A, B and D, respectively, but at 2 kX magnification)

Viscosity

The viscosity of the sizing solution/colloid material can affect its penetration or surface covering process during the surface treatment of paperboard. Therefore, this property was evaluated and analyzed.

It is generally believed that starches with low viscosity values have more potential to penetrate the paper structure. In contrast, at higher viscosity values, it is more probable that a starch solution will form a thin layer film (coating) on the paper structure due to the fact that it cannot readily penetrate into the pores of the paperboard surface.

As shown in Fig. 3, by increasing the starch concentration, the viscosity increased up to 1200 mPa.s. Moreover, following addition of LCNFs to the starch, the viscosity was greatly increased so that in the 4+LCNF 50%, it reached very high value of 36000 mPa.s. However, it is noteworthy that for 4+LCNF 95%, too much LCNF share in the mixture resulted in a decrease in viscosity (Fig. 3).

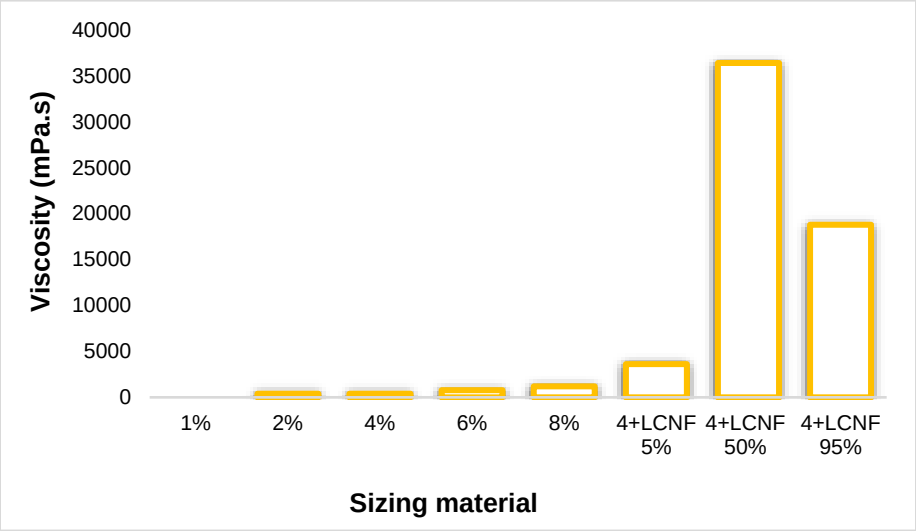

Fig. 3. Viscosity of prepared sizing material following homogenizing

Tensile index

Treated papers were evaluated for tensile index. Various shares of starch and LCNF were applied on paper, and the average tensile indices were compared with untreated (control) paper (Fig. 4). According to Fig. 4, the tensile index of the treated papers increased from 29 up to 45 Nm/g following the surface treatment with starch. It is well-known that tensile index is influenced by bonding strength and bonded area in the sheet, fiber strength, length, orientation, shape, and distribution (Taipale *et al.* 2010; Brodin *et al.* 2014). Because most of the recently noted effective parameters (related to fiber as length, distribution, *etc.*) were set to be constant in this research, the remaining effective item was bonding strength and bonded area. Ekhtera *et al.* (2008), reported increase of tensile index by surface treatment of paper with starch. Starch can penetrate the paperboard structure and so, it is proposed to enhance bonding strength or to develop bonded area (Biricik *et al.* 2011; Tutus *et al.* 2017).

Figure 4 also indicates that grammage of paperboard increased when starch at high concentrations were applied. This shows that more starch was absorbed to the paper, although the increase in tensile index was not significant for 4 to 8% starch concentrations. The result can be attributed to the starch penetration and absorption to the paper porous structure. This reveals that starch is not able to improve tensile index in cases where it cannot penetrate the paper structure.

Furthermore, with 5% LCNF addition, the tensile index showed another significant increase and reached to 53 Nm/g. The observation should be due to LCNF role in the starch film. LCNF is a network fibrillar structure and this structure helps strengthening of paper structure or the starch film. Higher amounts of LCNF (more than 5%) could not improve tensile index, which can be attributed to higher viscosities of the sizing mixture. High

viscosity value of sizing mixture makes penetration into the paper structure difficult; therefore, this decreases sizing mixture contribution to the tensile index improvement.

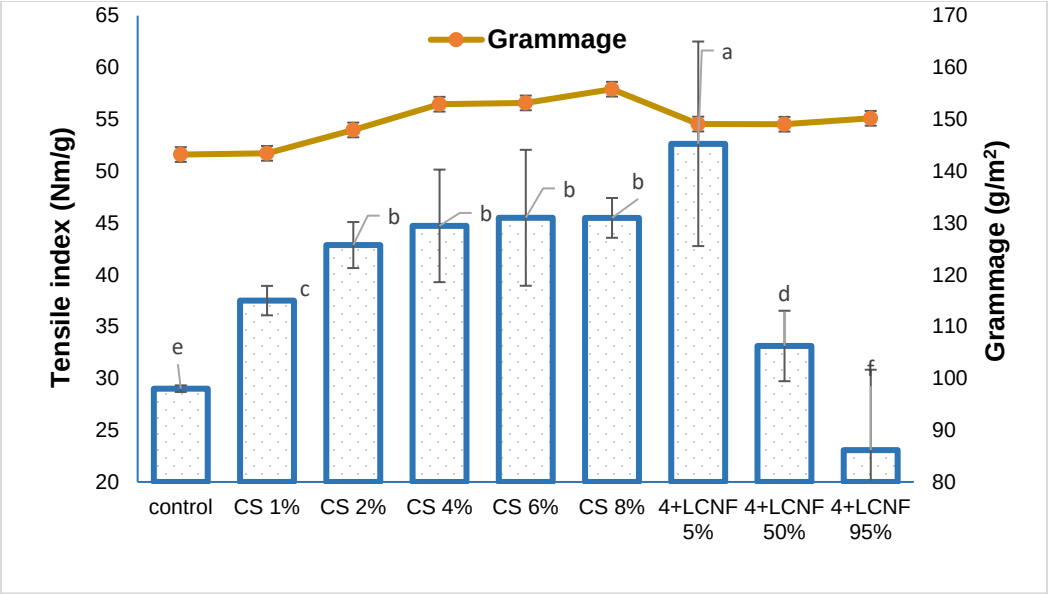

Fig. 4. The effect of surface treatment of additives on tensile strength index

Tear index

According to Fig. 5, the tear resistance index increased with the addition of 1% and 2% CS; a similar result has been reported by Kassem *et al.* (2009). Tear resistance changes mostly when average fiber length, individual fiber strength, or bonding are affected. Therefore, where fiber length and strength remained unchanged, the improvement of bonding in the sheet structure can be the most probable reason for tear index enhancement.

However, this resistance index no longer showed a significant increase with increasing starch concentration (Fig. 5).

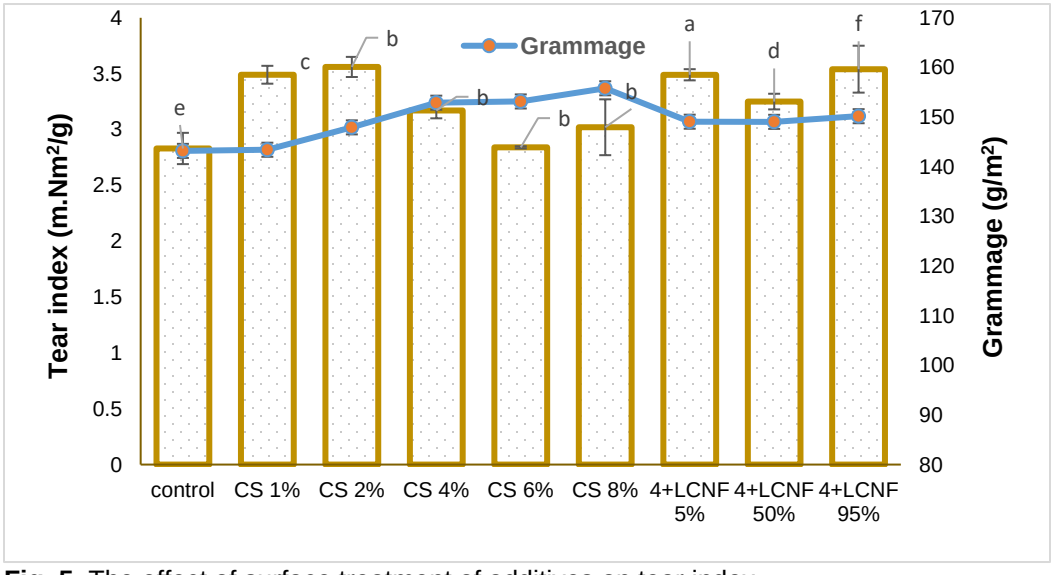

Fig. 5. The effect of surface treatment of additives on tear index

This observation can be due to less penetration of starch with high concentrations while this starch increases the grammage of the sheet (Fig. 5). Because the tear index is the result of dividing the resistance by grammage, where the resistance does not increase as much as the sheet grammage, therefore, the resulted index cannot show an increasing trend.

Bending resistance index

The paperboard bending resistance index was another measured strength parameter, which was statistically analyzed due to its importance for packaging. As shown in Fig. 6, the bending resistance index showed a significant increase following the surface treatment of paperboard with cationic starch or any other CS - LCNF mixture used. However, no statistically significant difference was observed due to the starch concentration. But the amount of this resistance increased by the addition of LCNFs so that with 4+LCNF 95% surface treatment, it reached 15.0 ($N.m^6/kg^3$), almost 1.7 times that of the control sample (SCAN-P 29:95 1995).

In contrast, bending resistance is highly affected by thickness of the paperboard (Yusefhashemi *et al.* 2019). The average thicknesses of the paperboard specimens are reported in Fig. 6. Therefore, eventually the results in Fig. 6 are the consequence of changes in thickness, and modulus of elasticity of surface treated paperboards.

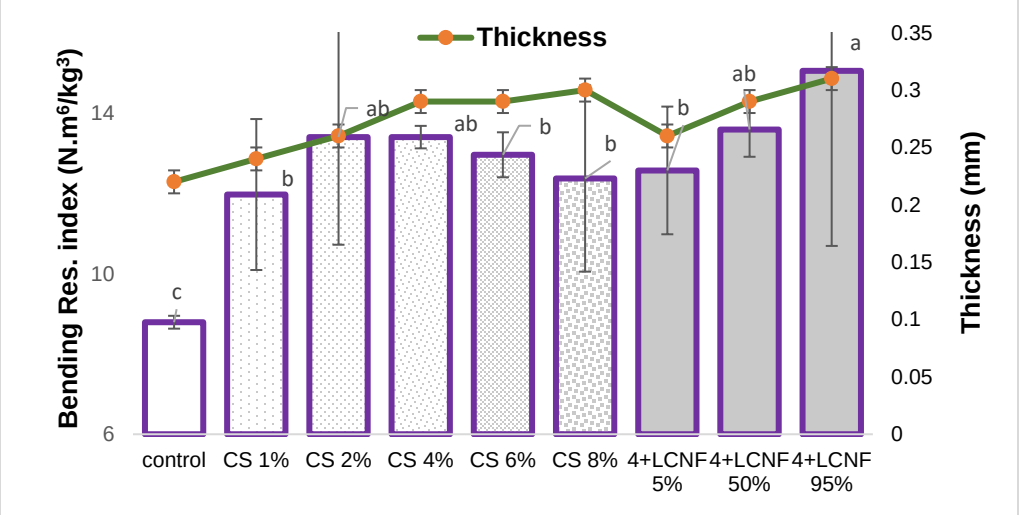

Fig. 6. The effect of surface treatment of additives on bending resistance index

Ring crush test (RCT)

According to Fig. 7, the surface application of cationic starch with optimum concentration (CS 4%) increased the ring crush test (RCT) values. The data showed that higher concentrations of CS were too viscous to be homogenously applied on the paperboard sheet and therefore resulted in less RCT values. The RCT of 4+LCNF 5% treated paper was not significantly different from the CS 4% treated sample, although it was 1.74 times as much as the control paperboard. Moreover, with more LCNF in the CS-LCNF mixture, the RCT was reduced again probably due to increasing the viscosity of sizing material mixture (Fig. 3), which inevitably deteriorates the homogenous application.

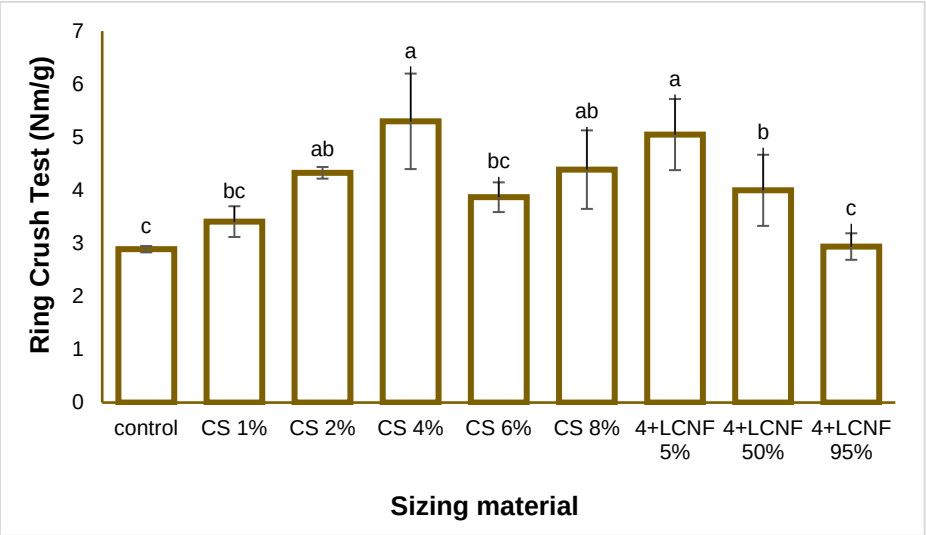

Fig. 7. The effect of surface treatment of additives on ring crush test

Table 2. The Effect of Surface Sizing Treatment on the CA of Water Droplet

No.	Sizing Material Mixtures	Code	CA (initial)	Time to Zero-Degree (s)
1.	Control (untreated base paper)	control	70 to 80	3 to 4
2.	Cationic starch (CS) with concentration of 1% (w/w)	CS 1%	60 to 65	3 to 4
3.	Cationic starch (CS) with concentration of 2% (w/w)	CS 2%	60 to 65	5 to 10
4.	Cationic starch (CS) with concentration of 4% (w/w)	CS 4%	55 to 60	> 20
5.	Cationic starch (CS) with concentration of 6% (w/w)	CS 6%	55 to 60	> 20
6.	Mixing ratio: 5% LCNF with 95% cationic starch (CS)	CS 4+LCNF 5%	60 to 70	> 20
7.	Mixing ratio: 50% LCNF with 50% cationic starch (CS)	CS 4+LCNF 50%	70 to 80	> 20
8.	Mixing ratio: 95% LCNF with 5% cationic starch (CS)	CS 4+LCNF 95%	70 to 80	5 to 10

Contact angle and Cobb value

The initial contact angle (CA) of a water droplet (at collision moment) is governed to the affinity of the surface toward water, which shows the effect of surface treatment on wettability. Other factors, such as porous structure of paper and surface quality also affect CA (TAPPI T458 cm-04 2004). Additionally, changes in CA during the time reveal interaction of water-based solutions with paper (Khosravani *et al.* 2016).

Control (untreated paperboard) samples showed relatively higher CA than the treated samples for which starch/LCNF were applied on the surface. In contrast, the time taken for CA to reach zero degree (absorbed completely by the paperboard) increased following surface application of these materials. Therefore, it can be concluded that although LCNF/starch cannot change the hydrophilic nature of the paperboard surface, they cover the pores and voids on the paperboard surface and increase the water droplet absorption time (more time is needed to reach zero degree).

Cobb test results can confirm the above discussed idea (Fig. 8). Starch is well-known as an inherently hydrophilic material because of the presence of hydroxyl groups; but Fig. 8 shows covering the paperboard surface by applying starch or starch/LCNF mixture reduced the water uptake by the linerboard structure. Because the thin film of starch or starch/LCNF mixture fill in the pores and voids or cover them, less water uptake resulted in the Cobb test, despite the hydrophilic nature of starch.

When an even and continuous film covers the pores and voids on the paperboard surface, it is expected that little water will be allowed to penetrate the paperboard structure. Indeed, lower Cobb values were observed (Fig. 8). Therefore, the reduction of Cobb values due to surface application of starch/ LCNF mixtures can be attributed to the film forming features or filling the pores and voids on paper surface.

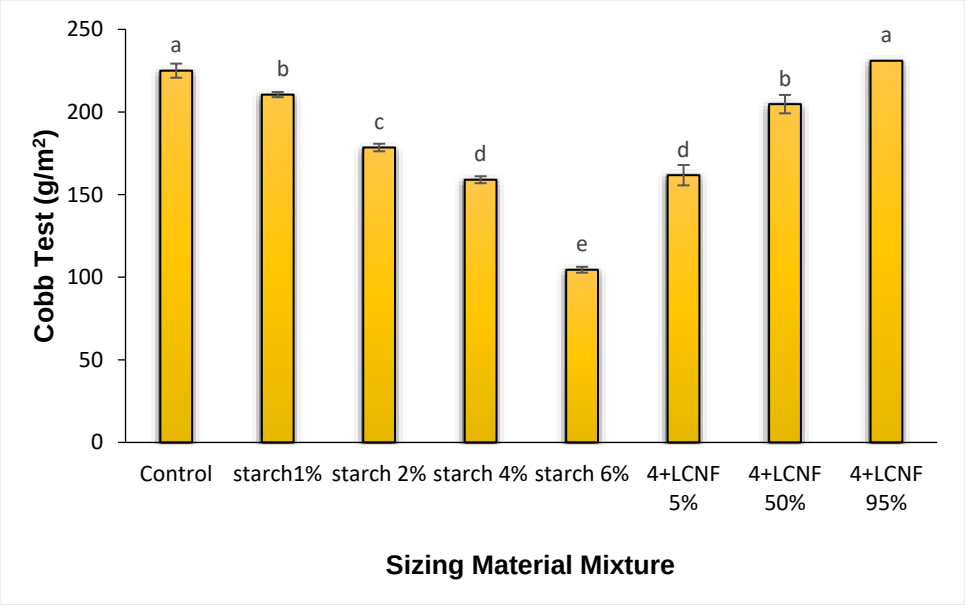

Fig. 8. Cobb test results of treated and untreated paper samples

CONCLUSIONS

1. The field emission scanning electron microscopy (FE-SEM) images showed that high viscosity surface treatment materials, such as sizing mixture containing starch with 50% lignocellulose nanofibers (LCNFs) (CS 4+LCNF 50%), resulted in better paper surface coverage of pores and voids.
2. The addition of a small amount of LCNFs (5%) to the starch in the sizing material mixture resulted in a significant increase in the tensile index of the paperboard.
3. The Cobb values and contact angle data indicated that high concentrations of starch can fill the pores and voids on the paperboard sheet, therefore, water droplet needed much more time for penetration and absorption to the paper structure.

4. The LCNF addition to the cationic starch, can highly increase the viscosity of the sizing liquid mixture. Therefore, because viscosity of the surface treating (sizing/coating) material is an important parameter, the issue needs much more comprehensive investigations. As a promising suggestion, this can be used for adjustment of the viscosity of surface treating material.

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