Effect of Nano-Cellulose on the Improvement of the Properties of Paper Newspaper Produced from Chemi-mechanical Pulping

Jafar Ebrahimpour Kasmani [a,]* and Ahmad Samariha [b]

The effect of nanofibrillated cellulose (NFC) was investigated relative to the strength of chemi-mechanical pulp (CMP) and paper. The NFC was added at five levels: 0%, 2%, 4%, 6%, and 8%. Handsheets with a basis weight of 60 g/m^2 were prepared, and the physical properties (air resistance and surface roughness), the mechanical properties (tensile strength, burst strength, and tear strength), and the optical properties (brightness, opacity, and yellowness) were measured according to TAPPI standards. By increasing the NFC content, the tensile strength, burst strength, air resistance, brightness, and whiteness increased by 10.9%, 12.5%, 23.6%, 0.6%, 3.5%, and 6.8%, respectively, compared to the control (0% NFC) samples. By increasing the NFC content, the tear strength, roughness, and opacity decreased by 10.4%, 11.1%, and 0.6% compared to the control samples.

Keywords: Nanocellulose; Mechanical properties; Mechanical and optical properties

*Contact information: a: Young Researchers and Elites Club, Savadkooh Branch, Islamic Azad University, Savadkooh, Iran; b: Young Researchers and Elites Club, Science and Research Branch, Islamic Azad University, Tehran, Iran; *Corresponding author: Jafar_kasmani@yahoo.com*

INTRODUCTION

Paper has always been considered a worthy issue that plays an important role in economic, cultural, and historical issues. Newsprint paper has been accounted as one of the most crucial paper goods at the global scale, and its consumption level is governed by such factors as population growth, city development, and enhancement of society's literacy level. Mazandaran wood and paper company is the only producer unit in Iran that uses chemical-mechanical pulp (CMP) and forest species in producing newspaper. CMP process has been considered an efficient pulp production method.

In recent years, using nanocellulose fibers as a new additive for improving paper strength has been drawing lots of attention (Hassan *et al*. 2011; Hasanjanzadeh *et al*. 2014; Petroudy *et al*. 2014). Recent reports have highlighted the role of nanocellulose in improvement of paper strength (Hassan *et al*. 2011; González *et al*. 2012; Hii *et al*. 2012; Hadilam *et al*. 2013). Some of the effects of adding nanocellulose can be reduction of pores and enhancement of inner joints and strength against air passage (Hii *et al*. 2012; Charani *et al*. 2013; Sehaqui *et al*. 2013).

Mechanical processes, such as refining, or chemical methods, such as alkaline treatments, can be used to enhance fiber quality and enable the production of higher quality paper that creates added value (Hadilam *et al*. 2013). One of the main goals of cellulose nanofiber applications in paper production is the improvement of its preventative properties. The addition of nanomaterials can improve the properties of pulp and paper products that are produced from weaker fibers.

The principle of using nanomaterials to increase the functionality of paper and board covers all the ranges of functional paper properties, such as optical properties, to resistance properties against the climate, and mechanical properties (Madani *et al*. 2011). The addition of nanofibers to typical paper and board products has made the production of layered products and covering papers with nanostructured materials possible. Nanobio-based materials, especially nanocellulosic-based materials, have gained attention due to their specific resistance properties and their biodegradability (Hadilam *et al*. 2013).

In most of the studies on application of nanocellulose fibers for paper making, a cationic polymer has been employed as a retention aid (Charani *et al*. 2013; Petroudy *et al*. 2014). Nanocellulose fibers alone, because of their similar anionic surface charge as cellulose, cannot become attached effectively and distributed uniformly within the paper network. Therefore, to increase their efficiency in paper making, either the fiber should be rendered cationic by modifying and changing the surface charge or it should be applied together with a cationic polymer as a retention aid (Habibi 2014). Cationic starch is commonly absorbed by cellulosic fiber in the wet section of a paper machine; therefore, the probability of creation hydrogen bonds is strengthened. In addition, Yoon and Deng (2006) reported that if starch has been used, improvement of paper strength in dry format can be attributed to enhancement of cutting strength per joint surface.

There are various studies that have considered the order of addition of anionic and cationic agents to paper pulp, and each one has its own advantages regarding the conditions (Hadilam *et al*. 2013). In the present project, nanocellulose fibers were added as the anionic component with a starch product serving as the cationic component. Obviously, in the case of addition of cation polymer as the first step, some sections of fibers will have been joined together by polymer; therefore, some portion of the added starch product will have been used for reinforcement of fiber joints. In this way, by addition of nanofiber at second stage, some of the nanofibers adhere to loaded parts of polymer and the rest will remain in the system as a suspension, whereby it can adversely affect drainage. It seems that in the case of addition of nanofiber at the beginning, much of it will remain in suspension and highly able to interact with cationic starch. The cationic starch can serve as a retention agent, helping to hold the nanocellulose efficiently on the surfaces of fibers in the suspension. Consequently, one can expect less of it to be carried away from the pulp suspension and the wet web during the drainage process. Therefore, in the present study, to preserve the maximum level of nanofiber in paper, this material has been added at the beginning.

Henriksson *et al.* (2008) used wood cellulosic nanofibrils to produce porous cellulosic nanosheets with high hardness. They succeeded in making a nanosheet with a tensile strength of 214 MPa, which is higher than the tensile strength of cast iron and is comparable to the strength of steel. Madani *et al*. (2011) considered the separation and elimination of nanofibrillated cellulosic (NFC) particles and their effects on the tensile strength of produced papers from the bleached chemical pulp of broad-leaved trees. The results of their experiments showed that the addition of NFC particles without long fibrils can increase the tensile strength of the produced sheets in comparison with the addition of particles without NFC treatment (Madani *et al*. 2011). Although the use of unnatural reinforcements can improve the strength properties of paper, they also increase the costs and create environmental problems. Lakani and Afra (2013) considered the effect of the pulp mixing duration and the use of NFC on sheet properties.

The results indicated that increasing the mixing time to 1 h can increase the dewatering time and decrease the air permeability. The main purpose of this study was to consider the effect of NFC addition on the properties of newsprint sheets produced from chemi-mechanical pulp (CMP).

EXPERIMENTAL

Materials

CMP pulp

The CMP was obtained from the CMP pulp storage tower in the Mazandaran Wood and Paper Industry (Mazandaran, Iran) and transferred to the laboratory. The pulp was refined according to the TAPPI standard T248 sp-00 (2000) using a laboratory PFI mill (Labtech, Québec, Canada). Refining was continued to a freeness level of 350 mL (CSF).

Cationic starch

The cationic starch with a degree of substitution (DS) of 0.035 was procured from the Glucosan Company (Ghazvin, Iran) and was used at a 1% dose based on the dry pulp weight. The cationic starch of present study had pH=6, degree of substitution (D.S) about 0.035 mol/mol, protein level 1.5%, N 0.25% and moisture 11% based on wet weight. To prepare starch lotion with concentration of 0.005 g/cm^3 (*i.e*. 0.5 g of pure starch per 100 cm^3 of starch and water solution), the required gross starch has been determined by considering moisture percentage. The determined gross amount was poured into an Erlenmeyer flask, and its volume was increased to 100 cm^3 by addition of distilled water. During mixing, the temperature of inside the Erlenmeyer was monitored by a thermometer, and a foil paper was put on the top of the Erlenmeyer as a lid to prevent evaporation. After heating is done, sufficient water has been added to restore the total volume to its calculated amount. The Erlenmeyer was put on heater for 30 min, and the temperature was brought to 90°C slowly and kept at this temperature for 30 min. The resulted starch solution was prepared freshly each day to avoid viscosity changes and other concentration changes as a result of environmental effects. The cationic starch properties are presented in Table 1.

Table 1. Cationic Starch Properties

Properties	Value
Moisture	14%
Gelation Temperature	70 °C
Cooking Temperature	90 °C
pH	6
Viscosity	75.7 (cp)
DS	0.035

Nanocellulosic gel

In this study the starting cellulose material was unadulterated commercial cellulose strands of softwood, acquired from Nano Novin Polymer Co (Gorgan, Iran) at four different levels: 0, 2, 4, 6, and 8% the dry weight of pulp and paper. Cellulose nanofibers were provided from long fiber α-cellulose material obtained through a super-pounding method. First, long fiber α-cellulose material was rinsed with distilled water three times;

then, it was treated in a 5% concentration of potassium hydroxide (KOH) lotion for 1 h at 80 °C under mechanical mixing. After this primary stage, an α-cellulose suspension with a 1% consistency was prepared and passed multiple times through the super-grinding disk machine (MKCA6-3; Masuko Sangyo Co., Ltd., Kawaguchi, Japan) to obtain cellulose nanofibers. The super-grinding disk machine consisted of a static and a turning processor disc. The pounding stone was SiC, and its diameter was 6 inches. The time and speed of grinding were 40 g/hour and 1800 rpm, respectively. The energy consumption of the processor was 25 KWh/Kg. The nano size fibers were thereby obtained in the form of a hydrogel.

Methods

Preparation of handsheets

To prepare the 60 g/m^2 handsheets, the fiber suspension was placed in a mixer (TestLab, Warsaw, Poland), 1% cationic starch was combined with the pulp suspension, and various concentrations of the NFC were added to the mixture of the CMP pulp. In total, eight handsheets were made for each treatment according to the TAPPI standard T205 sp-02 (2002). The different ratios of CMP, NFC, cationic starch, and imported long fiber pulp used for handsheet making are shown in Table 2.

Table 2. Combinations of Different Ratios of CMP Hardwood Pulp, NFC, Cationic starch, and Imported Long Fiber Pulp for Newsprint

Sample Code	CMP Hardwood Pulps (%)	NFC (%)	Cationic Starch (%)	Imported Long Fiber Pulp (%)
A (Control)	80	0	1	19
B	99	0	1	0
C	97	2	1	0
D	95	4	1	0
E	93	6	1	0
F	91	8	1	0

Measurement of the paper properties

The physical properties (the air resistance and the surface roughness), the mechanical properties (tensile strength, bursting strength, and tearing resistance indexes), and the optical characteristics (brightness, opacity, and yellowness) were measured on the handsheets according to the TAPPI standards T555 om-99 (1999), T460 om-02 (2002), T494 om-01 (2001), T403 om-02 (2002), T414 om-04 (2004), and T452 om-98 (1998).

Scanning electron microscopy (SEM)

To observe the presence of NFC on the sheet surface and fibers, a scanning electron microscope (AIS2100; Seron Technologies, Gyeonggi-do, South Korea) was used.

Statistical analysis

The data were analyzed using the SPSS statistical software (Version 11.5; IBM, Armonk, USA) with a complete randomized factorial test. The averages were compared and grouped using the Duncan's test at the 95% significance level (*p* equals 0.5).

RESULTS AND DISCUSSION

The NFC was dosed at levels of 0%, 2%, 4%, 6%, and 8%. The effect of the NFC was significant for the roughness, air resistance, tensile strength, tear strength, burst resistance, brightness, and yellowness at the 5% level. However, the effect of NFC was not significant on the opacity at this level.

Figures 1 through 8 illustrate the effect of NFC on the mechanical, physical, and optical properties of the handsheets. Duncan's test classified various levels of roughness means in the five groups (Fig. 1). As shown in Fig. 1, the incremental NFC levels up to 8% reduced the roughness so that the lowest roughness was related to 8% NFC addition (6.8 µm) and the highest roughness was seen in the control sample (8.1 µm).

Roughness of paper surface is an important structural feature and has significant importance in some applications that need better printability (Elyasi *et al*. 2016). This feature is highly affected by the mean of fiber length and presence of cellulosic fins and their stability in paper network and the quality of its formation. Therefore, in a specific combination; it can determine the function of additives to pulp suspension (Hamzeh *et al.* 2008). The addition of NFC at the 8% level reduced the roughness by 11.1% compared to the 0% NFC sample.

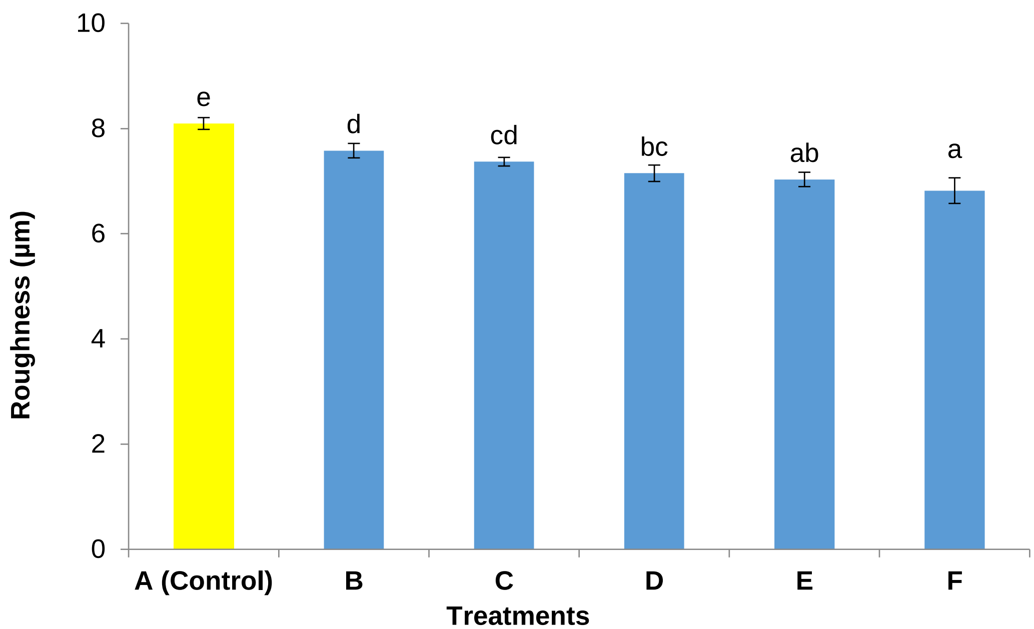

Fig. 1. The effect of NFC on the roughness (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Duncan's test classified various mean levels of the air resistance in four groups (Fig. 2). By increasing the NFC addition level up to 8%, the air resistance increased so that the highest air resistance was seen in the 8% NFC sample (9.8 mL/min) and the lowest air resistance was seen in the control sample (7.6 mL/min).

Porosity indirectly shows inner structure of paper that is under the effect of paper formation quality and the manner of fiber distribution, fines, and fillers. It can indicate the function of assistant materials in their distribution. Mostly, porosity level has been reduced by improvement of paper formation quality (Asadpour *et al*. 2015).

The air permeability of paper can be important for paper's strength, visual appearance properties, and requirements of final usage such as in different packaging papers. Because of effective absorption of cationic starch in fiber network, air permeability was increased. The effect can be attributed to the addition of starch, which has a strong tendency to be absorbed on negative surface of fibers, thus improving inter-fiber joints. Moreover, as nanofiber dimensions get smaller, specific surface of cellulosic fibers is increased. This implies more accessibility of hydroxyl groups at nanofibers surfaces. These groups can make hydrogen bonds with adjacent nanofibers, and eventually they create networks of nanofibers (Yousefi *et al*. 2011b). The result is increased paper strength and resistance to air permeability. The addition of NFC at the 8% level increased the air resistance by up to 23.6% compared to the 0% NFC sample.

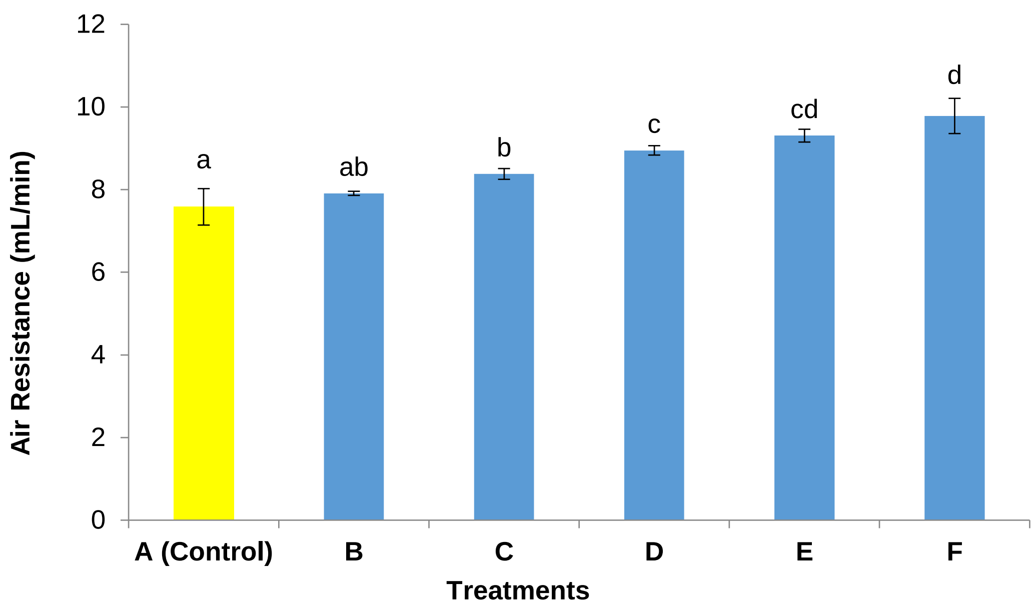

Fig. 2. The effect of NFC on the air resistance (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Duncan's test was used to classify the various mean levels of the tensile strength in four groups (Fig. 3). By increasing the NFC addition level to 8%, the tensile strength increased accordingly. The highest tensile strength was observed with the 8% NFC addition (33.6 Nm/g), and the lowest tensile strength was observed with the 0% NFC treatment (30.3 Nm/g).

The tensile index is an indicator of the paper tensile potential durability caused by the paper utilization level under tensile stress. The most important factor that affects the paper tensile index is the quality and number of connections (González *et al*. 2012). Increasing the fiber connections to each other, which is caused by increased refining or wet pressing, will increase the paper tensile index. However, the paper tensile index will always be less than that of the fiber (González *et al*. 2012; Petroudy *et al*. 2014).

However, the tensile strength of sheets is always less than the tensile strength of the fibers (Ashori *et al*. 2005). The machine direction (MD) tensile strength is always more than the cross-direction (CD) tensile strength because fibers align more in the longitudinal direction than in the transverse direction. In fact, two sets of joints are stretched in line with the MD: inter- and intraglucose covalent bonds (C-C, O-C) that are present in cellulose chains and hydrogen bonds between fibers. In total, there are more covalent bonds in the MD than in the CD (Hamzeh *et al*. 2013).

In handsheets, the categories of longitudinal and cross directions do not apply because the fibers are randomly oriented during the formation process. The most important factor on the tensile strength of a handsheet is the quantity and quality of the fiber bonds (Ashori *et al*. 2005). In tensile strength tests, both the interfiber joints and the fibers themselves are stretched. Therefore, longer fiber lengths and stronger interfiber joints help achieve this strength. As nanofiber dimensions get smaller, the specific surface of the cellulosic fibers increases. Therefore, more hydroxyl groups are accessible on the surface of the nanofibers that can create hydrogen bonds with their adjacent nanofibers and create a nanofiber network (Yousefi *et al*. 2011a), which will increase the strength.

When the sizes of lignocellulosic nanofibers get smaller, the specific surface of cellulosic fibers is increased. This means that more hydroxyl groups are available in nanofibers surface that are able to create hydrogen bonds with adjacent nanofibers and finally form a network of nanofibers (Yousefi *et al*. 2011b), this phenomenon increase the strength of resulted paper. Charani *et al*. (2013) have used cellulosic nanofibers in paper making and reported that the use of about 6% cellulosic nanofibers can improve the tensile strength equal to the use of 20% long length fiber in hardwood kraft pulp.

The addition of NFC at 8% increased the tensile strength compared to 0% NFC (10.9%).

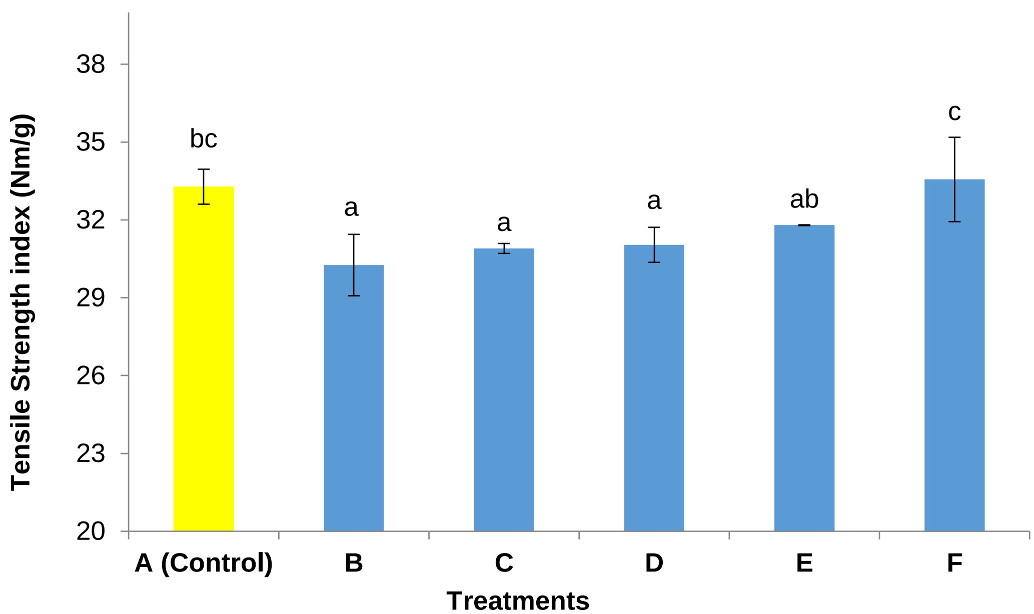

Fig. 3. The effect of NFC on the tensile strength index (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Duncan's test was used to classify various mean levels of the tearing strength for four groups (Fig. 4). Increasing the NFC levels up to 8% reduced the tearing strength. The highest tearing strength was observed in the control treatment (9.8 mN.m^2/g), and the lowest tearing strength was observed in the 8% NFC treatment (8.2 mN.m^2/g).

Parameters affecting the tear strength were the fiber length and strength. However, if these two factors are fixed, the bonds between the fibers can affect the tear strength. An increment in the ratio of long fibers to short fibers will increase the tear strength. Increasing the NFC dosage decreased the tear strength. A similar procedure has been reported about the reduction of the tear strength in NFC reinforced CMP papers (Hadilam *et al*. 2013). In this report, the changes in the two factors of the intrinsic strength of the fibers and the level of hydrogen bonds were identified as the reason for the changes and the severity of these changes in tear strength.

Tear strength is affected by various factors, and research results showed that tear strength is mostly affected by the mean of fibers' length, the level of hydrogen bonding, and fibers' inherent strength. Enhancement of lignocellulosic nanofibers increases the level of hydrogen bonding and decreases the mean of fiber length; therefore, tear strength is fluctuating. Totally, increasing nano lignocellulose can lead to reduction of tear strength because of enhancement of the level of hydrogen bonding (Hassan *et al*. 2011; Tajik *et al*. 2016). Hadilam *et al*. (2013) reported that addition of cellulosic nanofiber can reduce tear strength, so that the least level of tear strength has been observed in nano paper.

In all usage levels of cellulosic nanofibers, tear strength index was lower than the control treatment; therefore, the presence of nanofibers and fiber lobes can be followed with reduction of diameter mean and fiber length; this means that paper tear strength index has been reduced.

The addition of NFC at the 8% level resulted in a 10.4% reduction in the tear strength compared to the 0% NFC treatment.

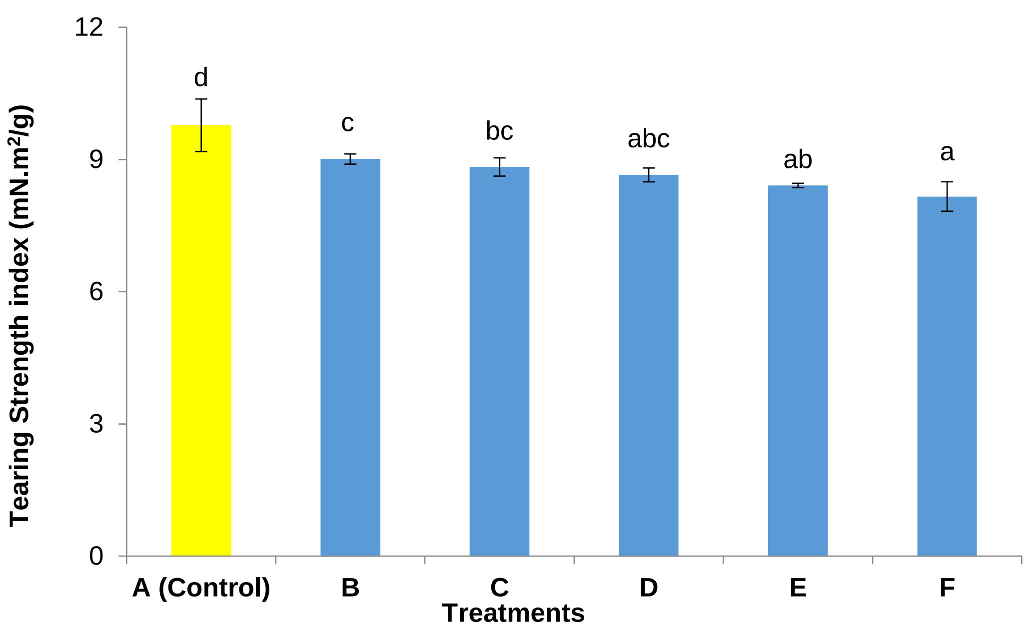

Fig. 4. The effect of NFC on the tear strength index (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Duncan's test also classified various mean levels of the burst strength for three groups (Fig. 5). Figure 5 illustrates that increasing the NFC level up to 8% increased the bursting strength. The highest bursting strength was observed in the control treatment (1.4 kPa/g), and the lowest bursting strength was observed in the 0% NFC treatment (1.2 kPa/g).

The burst strength depends on the fiber length and the bond between the fibers, but it is mostly affected by the bonds between the fibers. The burst index is proportional to the square of the average fiber length (Ashori and Raverty 2007).

Generally, the results indicated that cellulosic nanofiber along with cationic starch remain in paper structure and enhance the bursting strength through high slimming coefficient and physical involvement between nanofibers. As nanofibers have high specific surface and form physical involvement between nanofibers and bagasse fibers, the number of hydrogen bonds increases and the gap between fibers decreases. All of the aforementioned factors increase the inner fibers bonds and enhance the effectiveness of the fiber network; enhancement of bonding prevents fibers from sliding and leads to more stability of fiber network (Tajik *et al*. 2016). The addition of NFC at the 8% level increased the burst strength to 12.5% compared to the 0% NFC sample.

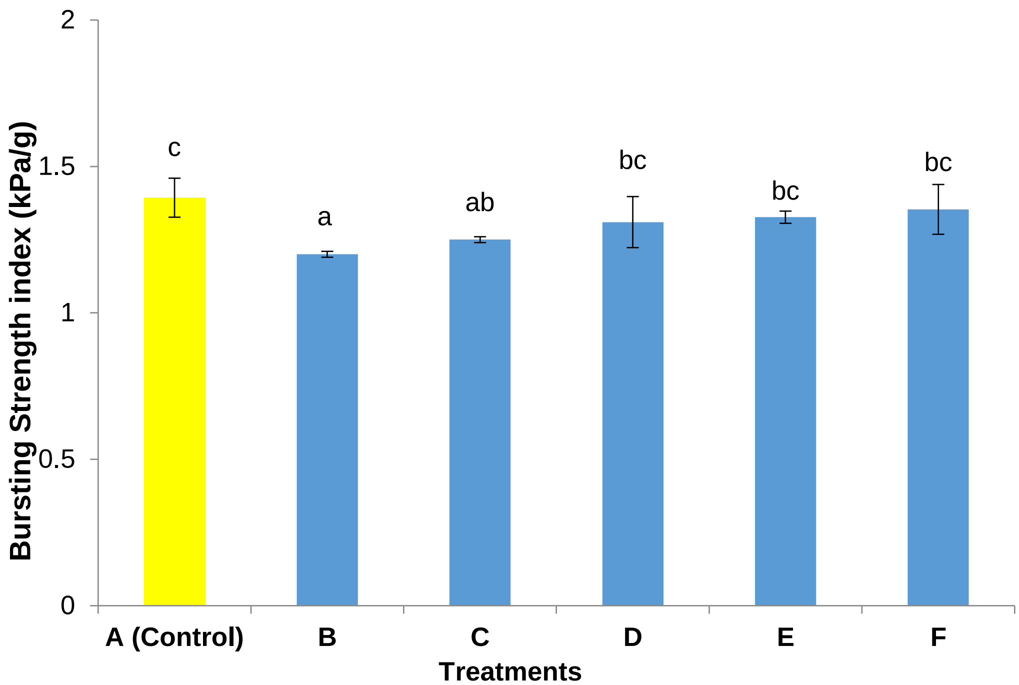

Fig. 5. Effect of NFC on the bursting strength index (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Duncan’s test was used to classify various mean levels of the brightness for five groups (Fig. 6). Increasing the NFC levels up to 8% increased the brightness. The highest brightness was observed in the control treatment (65.5%), and the lowest brightness was observed in the 0% NFC treatment (61.3%). Duncan’s test also was used to classify various mean levels of the opacity in one group (Fig. 7). Increasing the NFC level up to 8% decreased the opacity. The highest opacity was observed in the 4% NFC treatment (83.1%), and the lowest opacity was observed in the 8% NFC treatment (82%).

Lastly, Duncan’s was used to classify various mean levels of the whiteness of three groups (Fig. 8). Increasing the NFC level up to 8% increased the whiteness. The highest whiteness value was observed in the control treatment (33.8%), and the lowest whiteness value was observed in the 0% NFC treatment (31%).

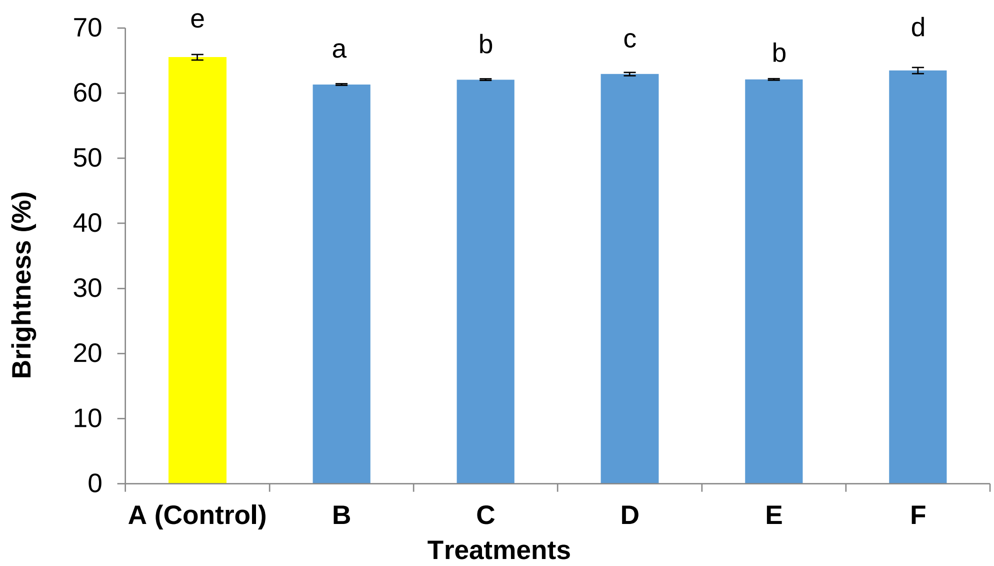

Fig. 6. Effect of NFC on brightness (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

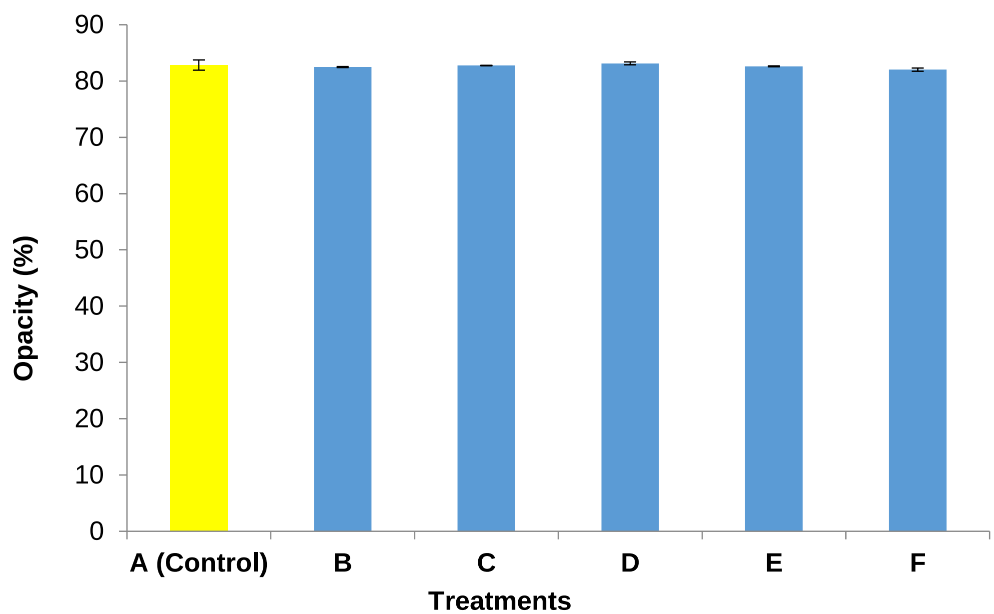

Fig. 7. Effect of NFC on opacity (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

Brightness and opacity degrees of papers are functions of optical characteristics of paper pulp and the amount and manner of filler distribution through the paper. The procedures of brightness, whiteness, and opacity changes are vague; so that sometimes the procedures reduced and sometimes increased. Cellulosic nanofibers fill the inner fibers gaps by increasing the link ability.

Addition of nanocellulose to pulps increased brightness degree. The presence of nanometer component of cellulose in paper structure caused extreme development of bonding surface, reduction of pores and bumps; all of these factors caused less light breakdown and more brightness (Nogi *et al*. 2009; Yousefi *et al*. 2011a).

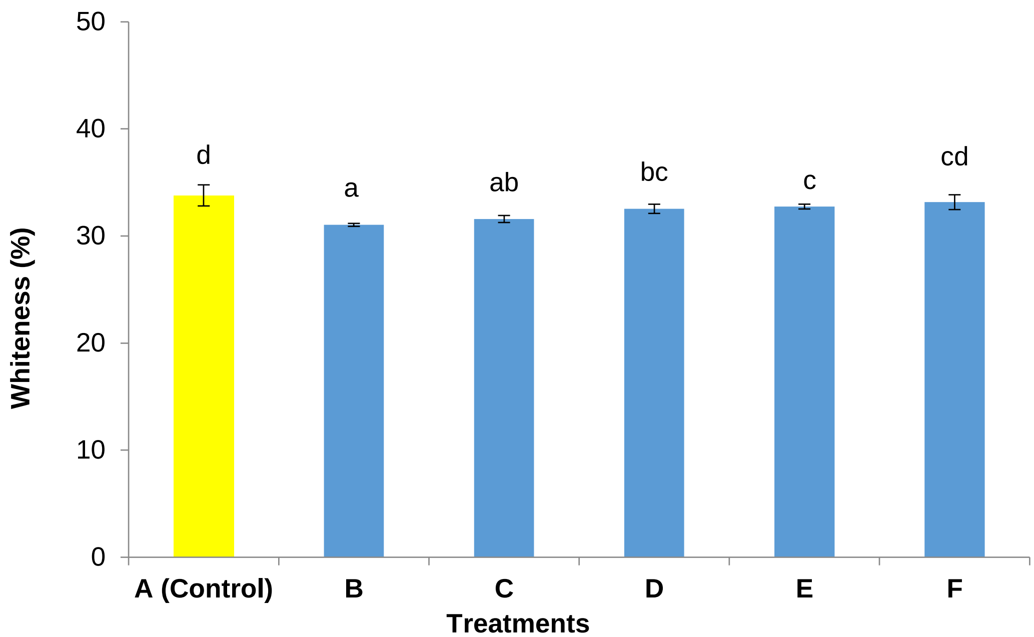

Fig. 8. The effect of NFC on the whiteness (B= 0% NFC, C= 2% NFC, D= 4% NFC, E= 6% NFC, F= 8% NFC)

The addition of NFC at the 8% level increased the brightness by up to 3.5% compared to the 0% NFC sample. The addition of NFC at the 8% level decreased the opacity by 0.6% compared to the 0% NFC sample. The addition of NFC at the 8% level decreased the yellowness by 6.8% compared to the 0% NFC sample.

Morphology

Microscopic studies of the paper structure

The SEM images of the surface of the CMP handsheets at 0%, 4%, and 8% NFC treatments are shown in Figs. 9 through 11.

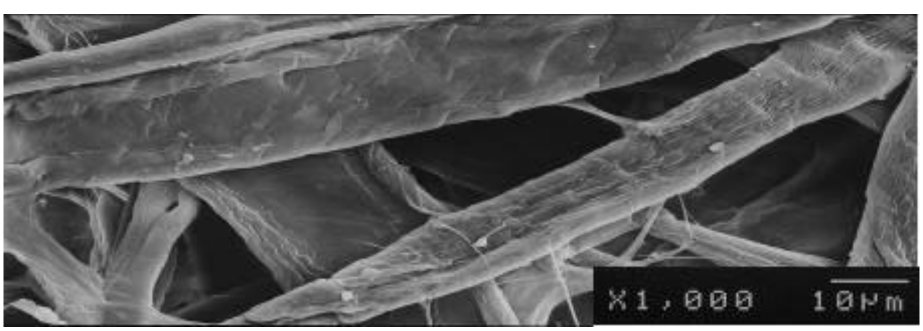

Fig. 9. The paper surface without NFC

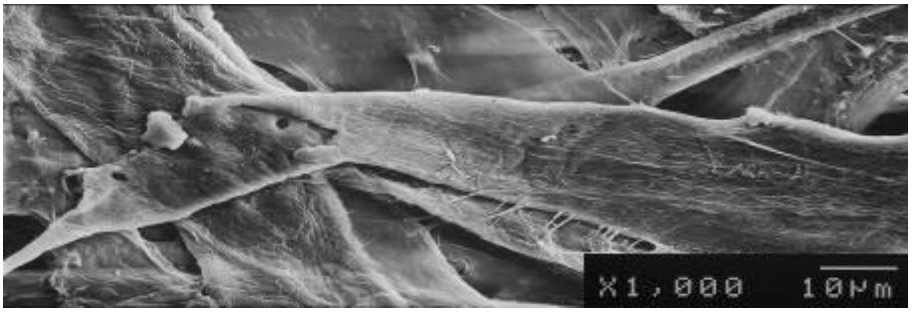

Fig. 10. The paper surface with 4% NFC addition

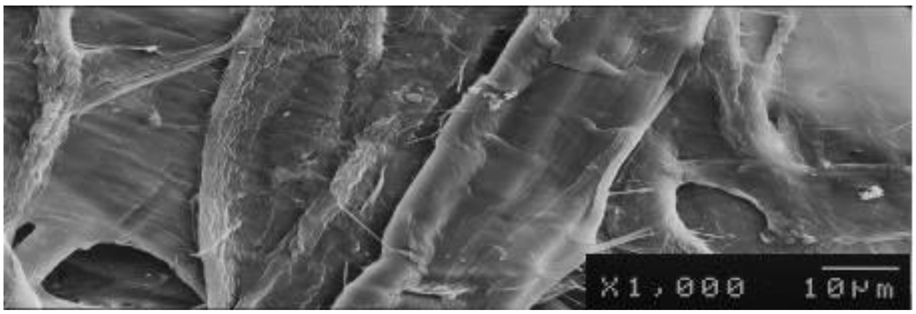

Fig. 11. The paper surface with 8% NFC addition

The SEM pictures indicate that the addition of cellulosic nanofiber and cationic starch has increased link ability, fibers concentration, filling the gaps between fibers and fines, and durability. Hii *et al*. (2012) have shown that cellulosic nanofiber aided to join filler and micro particles of fibers and reduced paper pores and also increased air passage strength, all of which led to better inner paper joints (Hii *et al*. 2012). By increasing the NFC level, the nanocellulose partially filled the fiber pores and increased the air resistance.

CONCLUSIONS

In the present study the effect of addition of nanocellulose to CMP pulp was considered, and mechanical and morphological strengths of resulted newspaper paper were studied. The following results were obtained:

1. The nanofibrillated cellulose (NFC) treatment at up to 8% addition increased the tensile strength and burst strength by 10.9%, and 12.5%, respectively, compared to the 0% NFC treatment.
2. The NFC treatment of up to 8% increased the air resistance, brightness, and whiteness by 23.6%, 3.5%, and 6.8%, respectively compared to the 0% NFC treatment.
3. The NFC treatment of up to 8% decreased the tear strength, roughness, and opacity by 10.4%, 11.1%, and 0.6%, respectively compared to the 0% NFC addition.

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