Prospects for the Preparation of Paper Money from Cotton Fibers and Bleached Softwood Kraft Pulp Fibers with Nanofibrillated Cellulose

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Paper money passes through various environments during its life span, causing its physical, chemical, and optical properties to change. More than 90% of paper money worldwide is composed of natural cotton fiber. The present study examined the properties of paper money made of bleached softwood kraft fibers or its blends with cotton fibers, where nanofibrilled cellulose was employed as a strengthening agent. Nano-cellulose was added at 4 levels: 0, 0.3, 0.6, and 0.9%. Handsheets with a basis weight of 90 $g{\cdot}m^{-1}$ were made by mixing the pulp furnish with nano-cellulose in the identified percentages, and the physical and mechanical properties of the handsheets were tested. By increasing the amount of nano-cellulose up to 0.9% in cotton pulp, the tensile strength, bursting resistance, tear resistance, and resistance to folding endurance were increased by 33, 33.5, 6.6, and 63.2%, respectively, compared with the control sample. The addition of nano-cellulose up to 0.9% in cotton pulp increased the surface smoothness by up to 13.5% compared with the control sample, and porosity and water absorbance decreased by 16.6 and 4%, respectively, in comparison with the control sample. By increasing nano-cellulose up to 0.9% in cotton pulp, the opacity, brightness, and whiteness were decreased by 0.1, 1, and 4%, respectively. The SEM results indicated that the increased nano-cellulose percentage led to decreased porosity.

Keywords: Nano-cellulose; Chemical pulp; Paper money; Cotton; Mechanical properties; Optical properties; SEM

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INTRODUCTION

In a typical process for preparation of base-stock for paper currency, cotton is transported to the fiber cutter after undergoing batting in a high-speed cleaner. After this stage, the pulp is dewatered and is pumped to the dyeing section. The bleaching operation of the cotton fibers is accomplished using peroxide hydrogen and sodium silicate. After bleaching, the fibers are dewatered again in a felted press nip to remove the soluble chemical substances from the cotton fibers. Cotton fibers have good strength compared with other natural fibers. With cotton fibers, a shorter fiber length coupled with a greater diameter is more favorable for production in terms of cleanness, fiber processing, refinement, bleaching, and consumption of chemical material (Abdi *et al*. 2015).

Nano-additives can be used to improve pulp and paper properties, especially those papers that are produced from poorly bonded fibers. It has been shown that nano-materials have the potential to improve various properties of paper (Ramsden 2004). The application of nanocellulose has been an emerging field because of its specific properties, including biodegradability. Nanotechnology has been used to boost paper strength properties (Luu *et al*. 2011; González *et al*. 2012).

Nanofibrillated cellulose (NFC) is formed by exposing cellulosic fibers to mechanical shearing (Lavoine *et al*. 2012). The term NFC can be applied when sufficient mechanical energy has been applied so that the original cellulose fibers have been separated into fibrillated materials, the individual strands of which are less than about 100 nm in diameter (Hubbe *et al*. 2017). Micrographs of typical NFC preparations show networks of fibrils, still joined together at multiple branch-points. A variety of shearing devices have been shown to be effective. Also, it has been found that various pretreatments of the fibers can greatly decrease the amounts of energy required. In particular, treatment of the pulp fibers with enzymes (Anderson *et al*. 2014) or TEMPO-mediated oxidation (Hirota *et al.* 2010) can make it easier to mechanically convert pulp fibers into NFC.

When added to paper, these NFC particles have been found to reduce its porosity but increase its strength. The development of more joints and the creation of more hydrogen bonds during paper drying could be the cause of these results (Rezayati Charani *et al*. 2013). Bio-based nano-materials, especially cellulose-based nano-materials, have specific importance because of their high inherent strength, their non-hazardous nature, and their biodegradability (Hadilam *et al*. 2013). Lacani and Afra (2013) considered the effect of mixing time on pulp and microfibrillated cellulose (MFC) and on paper properties. Their results indicated that increasing the mixing time increased the dewatering time and decreased the air permeability (Lacani and Afra 2013). Henriksson *et al*. (2008) used wood cellulosic nano-fibrils for producing porous cellulosic nano-papers. They succeeded in producing one type of nano-paper with a tensile strength of 214 MPa. Madani *et al*. (2011) studied the preparation of MFC from bleached chemical pulp of hardwoods, and its effects on paper tensile strength. The results showed that the addition of MFC particles without long fibers significantly increased the tensile strength of the produced paper relative to the control without MFC addition. Yousefi *et al*. (2011) considered the effect of cellulosic nano-fibrils on the mechanical resistance of a paper made from *Brassica napus* stem. The results showed that the mechanical properties of nano-paper were superior to those of the micro-paper. Moreover, the amount of hydrogen bonding and the involvement of fibrils with each other were increased (Yousefi *et al*. 2011). High-quality raw materials should be used for the production of durable papers. The most important raw material for production of such paper is cotton cellulosic fibers. Because of the high purity and crystallinity of cotton fibers compared with other natural fibers, they have a high intrinsic resistance and durability. Therefore, more than 90% of worldwide paper money has been composed of natural cotton fiber. In global trading, the high cost of cotton fiber in comparison with other cellulosic fibers has led to a higher cost for paper products that are made of cotton (Abdi *et al*. 2015).

The present work addresses the hypothesis that high folding endurance and a range of other properties suitable for paper currency can be achieved by use of nanofibrillated cellulose in combination with bleached softwood kraft fibers. Though paper currency is ordinarily comprised of cotton fibers, the idea is that the superior flexibility and strength of inter-fiber joints resulting from the addition of nanocellulose, in relatively low amounts, will be able to make the structure formed with the softwood fibers perform at a higher level.

In this research, cellulosic nano-fibers were added to cotton pulp at different levels to investigate the feasibility of substituting cheaper pulp (linter, brock, *etc*.) as a part of the consumed furnish stock, and also to determine the effects on the properties of durable papers made of cotton fiber.

EXPERIMENTAL

Materials

Pulp and paper

Cotton pulp was purchased from Ehpak Ind. Co, Mazandaran, Iran. It was refined using a PFI mill to 250 mL CSF prior to any treatment and making handsheets. Bleached softwood kraft pulp was purchased from U-ilimsk, Russia. It was refined by a PFI mill to 250 mL CSF prior to any treatment and making handsheets. The obtained pulp and paper was dewatered to a concentration of 10 to 15% and then put in plastic bag and kept in a refrigerator until consumption.

Nano-cellulosic fiber

In this examination the crude material was unadulterated mercentile cellulose strands of softwood, acquired from Nano Novin Polymer Co (Gorgan, Iran) at four different levels: 0, 0.3, 0.6, and 0.9% the dry weight of pulp and paper. Cellulose nanofibers were set up from long fiber α-cellulose mash by a super-pounding method. First, long fiber α-cellulose mash was cleansed with purified water three times; then, it was set in a 5% concentration of potassium hydroxide (KOH) solution for 1 h at 80 °C under mechanical mixing. After this basic treatment, 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 deliver cellulose nanofibers. The super-grinding disk machine was comprised of a static and a turning processor disc. The pounding stone was SiC, and its diameter was 6 inches. The time and speed of crushing were 40 g/hour and 1800 rpm, individually. The energy consumption of the processor was 25 KWh/Kg. The nano size fibers were thereby obtained in the form of a hydrogel.

Polyacrylamide

To prepare a solution, 0.01 g of cationic polyacrylamide with a molecular weight of 3500 kg/mol was poured into a 100 mL volumetric flask. Then 1.5 mL of ethanol was added and after 2 min, 50 mL of distilled water was added to the volumetric flask and shaken for 2 min. The contents of the volumetric flask were stirred with a magnet for 3 h. The volumetric flask containing the polymer was kept in the refrigerator for about 24 h. Then, the contents of the volumetric flask were distilled with 100 mL volumetric flasks, and then they were stirred for 10 min. The solution, which had a concentration of 0.01%, was used to maintain the nanofibers in pulp suspension for making handmade paper.

Methods

Preparation of handsheets

Four different sub-batches of pulp were prepared, as shown in Table 1. These consisted of 100% bleached softwood kraft (100LP), 100% cotton pulp (100CP), a mixture of 85% cotton and 15% bleached SW kraft (15LP+85CP), and 70% cotton and 30% bleached SW kraft (15LP+85CP). To prepare a handsheet, NFC was added to aliquots of pulp at the dry-mass levels of 0, 0.3, 0.6, and 0.9%. This mixture was done in the presence of polyacrylamide at the 0.1% (based on the amount of NFC + pulp). The obtained mixture was mixed by magnetic stirring for 30 min at room temperature. Finally, nine handsheets were prepared for each treatment based on the TAPPI T205 sp-02 (2002) standard.

Table 1. Combinations of Different Ratios of Pulp Type for Handsheet Making

Sample code	Chemical pulp (%)	Cotton pulp (%)
100LP	100	0
100CP	0	100
15LP+85CP	15	85
30LP+70CP	30	70

Determining pulp and paper properties

The physical properties (porosity, surface smoothness, and water absorbance) were determined according to the TAPPI standards T460 om-02 (2002), T555 om-04 (2004), and T441 om-09 (2009). Mechanical properties (tensile strength, bursting strength, tearing strength, and folding resistance) were determined according to the TAPPI standards T494 om-01 (2001), T403 om-02 (2002), and T414 om-04 (2004). Optical properties (brightness, opacity, and whiteness) were determined according to the TAPPI standards T452 om-98 (1998), respectively

Scanning electron microscopy (SEM)

A scanning electron microscope (JEOL, model JXA-840, Tokyo, Japan) at the Academic Jihad Laboratory of Sharif Industrial University was used.

Data analysis

Data were analyzed using a randomized statistical plan, a two-way analysis of variance, and the Duncan test was used for mean comparisons. Results were analyzed with SPSS software (version 11.5, IBM Software, Armonk, NY, USA)

RESULTS AND DISCUSSION

There was a significant direct effect of pulp type and nano-cellulose on porosity, surface smoothness, water absorbance, tensile strength, bursting strength, tearing strength, folding resistance, brightness, whiteness, and opacity at the 5% significance level. The F-value and significance levels are both shown in Table 2.

The direct effects of nano-cellulose on surface smoothness, water absorbance, tensile strength, bursting strength, folding resistance, brightness, and whiteness were significant at the 5% level, while there was no significant effect on opacity and tearing resistance at this level.

The interaction effects of pulp and paper and nano-cellulose on porosity, surface smoothness, water absorbance, tensile strength, bursting strength, folding resistance, and brightness were also significant at the 5% level, while the effects on whiteness, opacity, and tearing resistance were not significant at this level. Figures 1 and 2 show the effects of type of pulp and paper and nano-cellulose on the physical and mechanical properties of handsheets.

Table 2. Variance Analysis (F-value and Significance Level) of Paper Type and Nano-Cellulose

Property	Nano-cellulose	Pulp Type	Nano-cellulose × Pulp Type
Surface Smoothness (mL/min)	6.485*	15.630*	18.429*
Porosity (mL/min)	34.905*	102.180*	11.851*
Water Absorption (g.m^{-2})	11.553*	727.854*	549.687*
Tensile Strength (Nm)	12165.529*	32.553*	48.389*
Tearing Strength (mN)	1.355ns	14.393*	.944ns
Bursting Strength (kPa)	50.367*	80.832*	14.068*
Folding Resistance (N)	22.070*	58.100*	10.970*
Opacity (%)	0.929ns	2.282ns	1.137ns
Brightness (%)	9.579*	61.424*	.062*
Whiteness (%)	5.567*	59.252*	1.309ns

*Significance level of 95%
ns: Not significant

Figure 1 indicates that the highest porosity, 2017 mL/min, was associated with 100% chemical pulp without nano-cellulose, and the lowest amount, 517 mL/min, occurred in the case of the 70% cotton pulp along with 0.6% nano-cellulose. When the nano-cellulose was 0.9%, the porosity was reduced. Porosity is one of the most important properties affecting ink absorption, as ink capillaries permit ink infiltration when there is porosity and gaps among fibers. Coated paper provides more control over this penetration than non-coated paper. By coating paper with nano-cellulose, the porosity is reduced and the time required for passage of air increases (Hamzeh *et al*. 2013). According to graphs showing the paper porosity level, the greatest effect of nanocellulose was observed in the case of chemical pulp, for which there was a linear decline with increasing NFC.

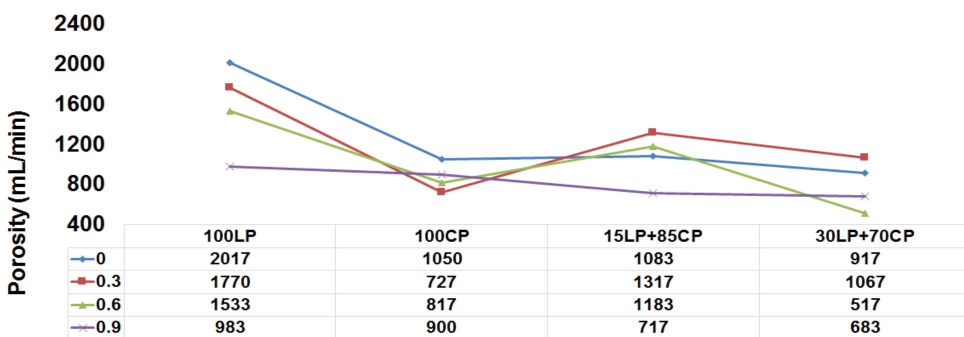


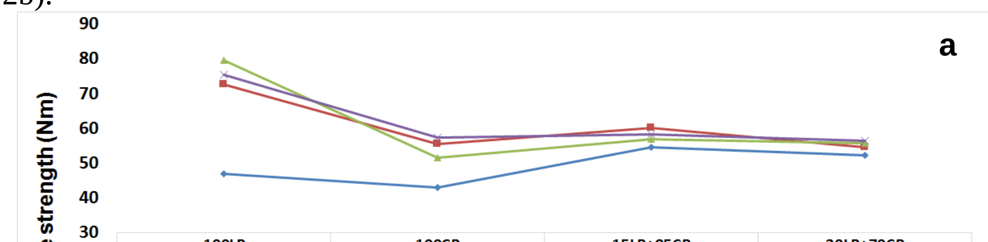
Fig. 1. The effect of nano-cellulose and pulp and paper type on porosity

Figure 2a shows that the highest tensile strength, 79.5 Nm, was associated with 100% chemical pulp and 0.6% nano-cellulose, while the lowest amount, 43 Nm, was associated with 100% cotton pulp without nano-cellulose.

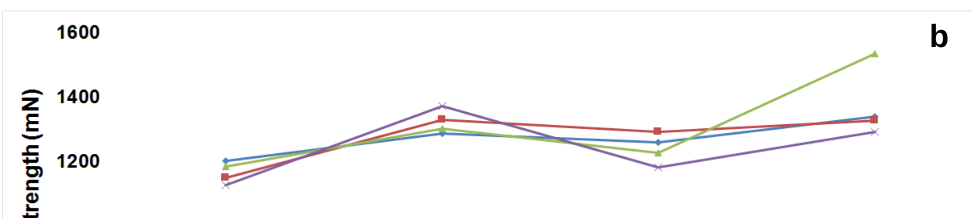
Tensile strength is the most suitable index for all inter-fiber joints, which is considered a combination of other resistances. Tensile strength is an index of the potential tensile durability of paper. The most important factors affecting the tensile strength of a paper are the number and quality of the fiber bonds to each other.

Enhancement of fiber joints to each other, which has been obtained under the effect of enhancement of refinement or humid press *etc*., will increase paper tensile strength. Meanwhile, the tensile strength of paper is always less than that of fiber (Petroudy *et al*.

2014). Tensile strength in the machine direction is higher than that in the transverse direction, because the fibers becomes aligned in the longitudinal direction more readily than in the transverse direction. In the machine direction, two different sets of bonds are stretched: the covalent (C-C, C-O) inter- and intra-glucose bonds in the cellulose chain, and the hydrogen bonds between fibers. In general, there are more covalent bonds in the machine direction and fewer covalent bonds in the transverse direction. As in handsheets, where fibers are located at various directions randomly, the longitudinal and transverse directions are not significantly different. Because the dimensions become smaller up to the nano-meter scale, the specific surface of the cellulose fibers increases. This means that there are more available hydroxyl groups at the nano-fiber level that are able to create hydrogen bonds with adjacent nano-fibers, thus forming a network of nano-fibers (Yousefi *et al*. 2011) that increases this strength. When nano-cellulose is added up to 0.9%, an increase in tensile strength can be observed. The effect of nano-cellulose is more significant on chemical pulp than on cotton pulp, but in paper made with composite pulp, increasing the proportion of chemical pulp has a positive linear effect on the tensile strength of paper. As a result, the effect of the addition of nano-cellulose to pulp and paper (either independently or in combination) was found to be an increase in the tensile strength index of paper, which is the most crucial mechanical factor of durable papers. When nano-cellulose was increased to the 0.9% level, a reduction in tearing resistance was observed (Fig. 2b).



	100LP	100CP	15LP+85CP	30LP+70CP
0	46.8	42.9	54.6	52.2
0.3	72.5	55.4	60.0	54.6
0.6	79.5	51.6	56.9	55.6
0.9	75.4	57.2	58.1	56.4



	100LP	100CP	15LP+85CP	30LP+70CP
0	1202.1	1287.1	1260.3	1338.7
0.3	1150.1	1328.8	1291.2	1326.4
0.6	1184.3	1301.9	1226.8	1533.3
0.9	1126.9	1372.3	1182.5	1291.9

Fig. 2. The effect of nano-cellulose and pulp and paper type on a) Tensile strength, b) Tearing strength, c) Bursting strength, and d) Folding strength

The results of the tests and analysis of variance for the folding resistance of the handsheets treated with nano-cellulose showed that the addition of nano-material up to 0.3% in combined pulp, especially chemical pulp, increased the folding resistance, but increases in the nano injection level decreased the folding resistance. Folding is affected by the intrinsic flexibility of fibers, joint surfaces, and the bonds created among them. The independent effect of nano-material on the folding of chemical pulp was significant (because of fiber entity), and this feature can be more important in the enhancement of chemical pulp consumption than on basic pulp. Folding strength is an important index in durable paper, especially paper money, so that, recycled and weaker fiber can be used in primary stock furnish by increasing this index.

Figures 3 through 6 show the surface fibers of treated handsheets containing pulp and paper and nano-cellulose. As it is observable in these figures, the pulp and paper type did not affect the fiber surface, but the enhancement of nano-cellulose could cover the fiber surface in a way that when the nano-cellulose was 0.9%, little porosity could be observed. The SEM studies revealed that the type of pulp and paper does not have any significant effect on the structure of fiber surface, but by increasing the consumption level of the nano-cellulose, the fiber surface is covered and the porosity reduced. Based on this picture and on considerations of air permeability graphs, it appears that treatment with nano-cellulose can fill the vacant gaps between fibers. The papers that were made by combining pulp and paper in a ratio of 30:70 had the lowest porosity at 0.6% nano-cellulose.

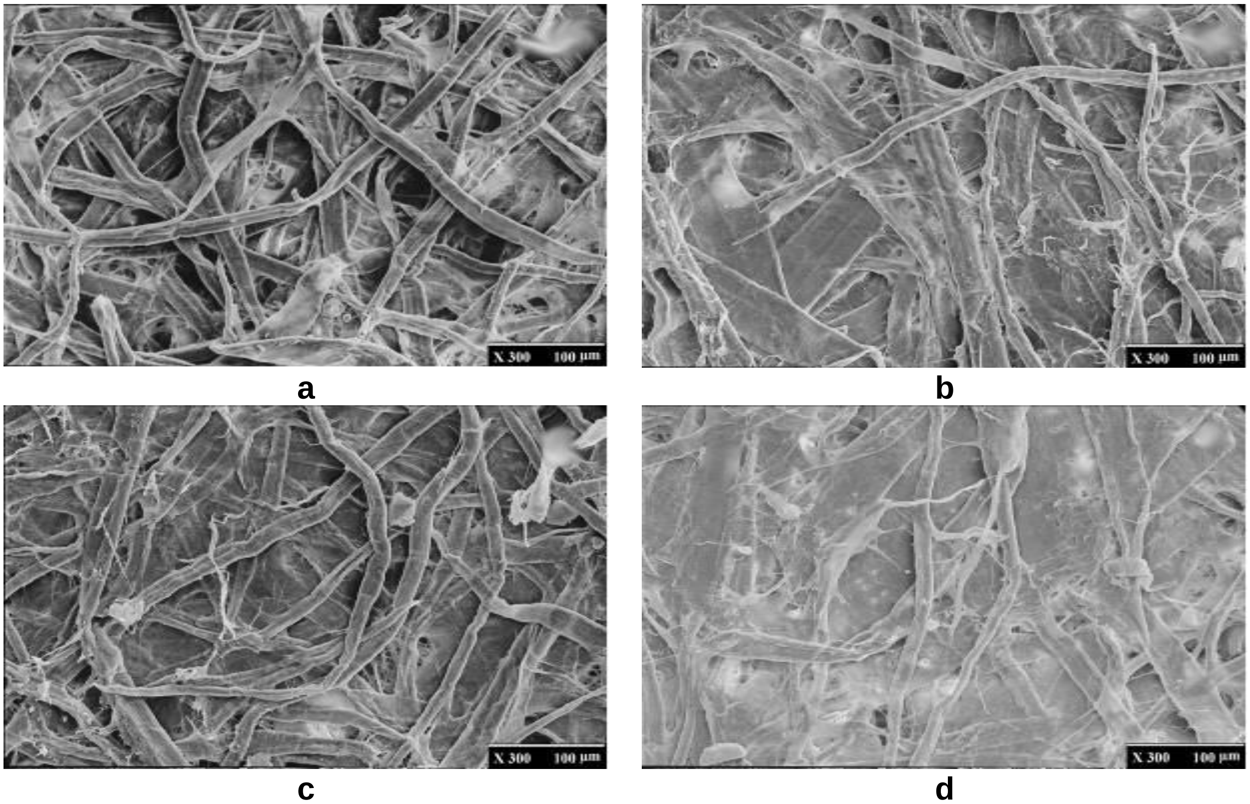


Fig. 3. The surfaces of paper made from chemical pulp and a) 0% nano-cellulose, b) 0.3% nano-cellulose, c) 0.6% nano-cellulose, and d) 0.9% nano-cellulose

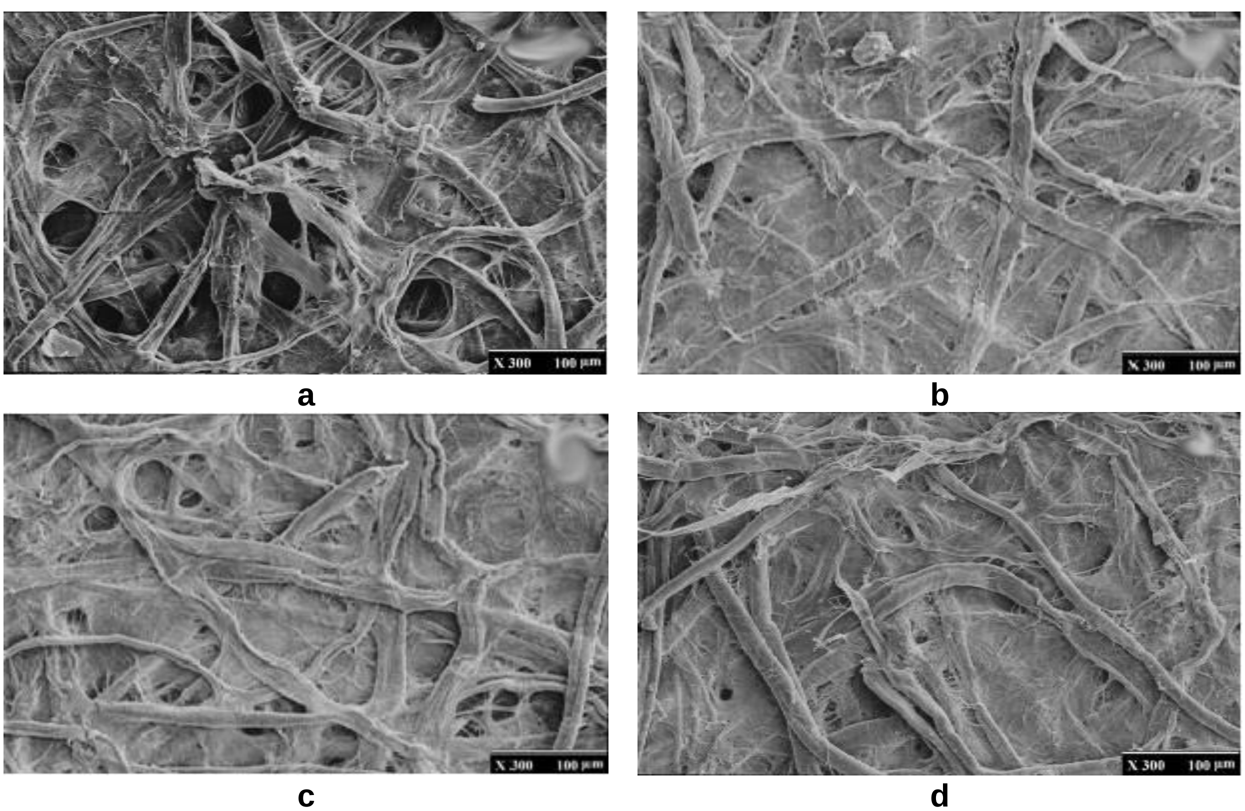


Fig. 4. The surfaces of paper made from cotton pulp and a) 0% nano-cellulose, b) 0.3% nano-cellulose, c) 0.6% nano-cellulose, and d) 0.9% nano-cellulose

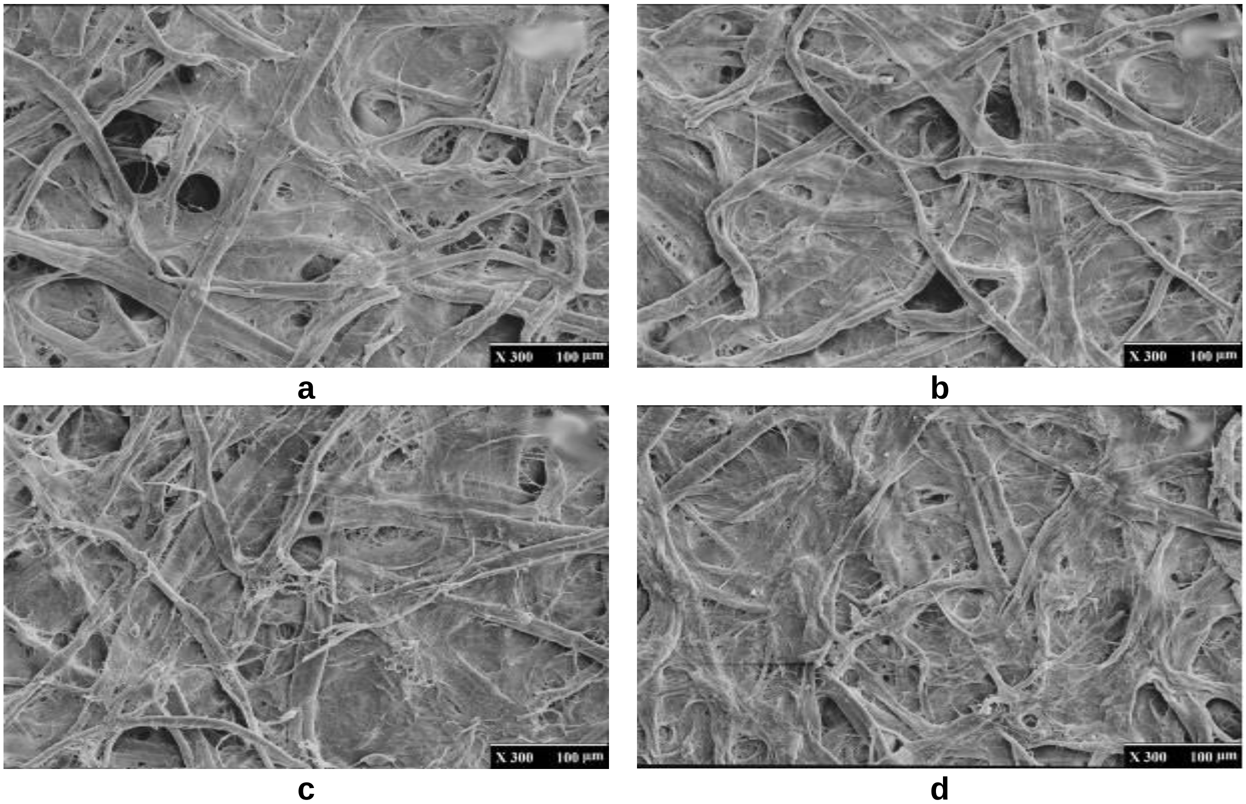


Fig. 5. The surfaces of papers made from 15% chemical pulp and 85% cotton pulp and a) 0% nano-cellulose, b) 0.3% nano-cellulose, c) 0.6% nano-cellulose, and d) 0.9% nano-cellulose

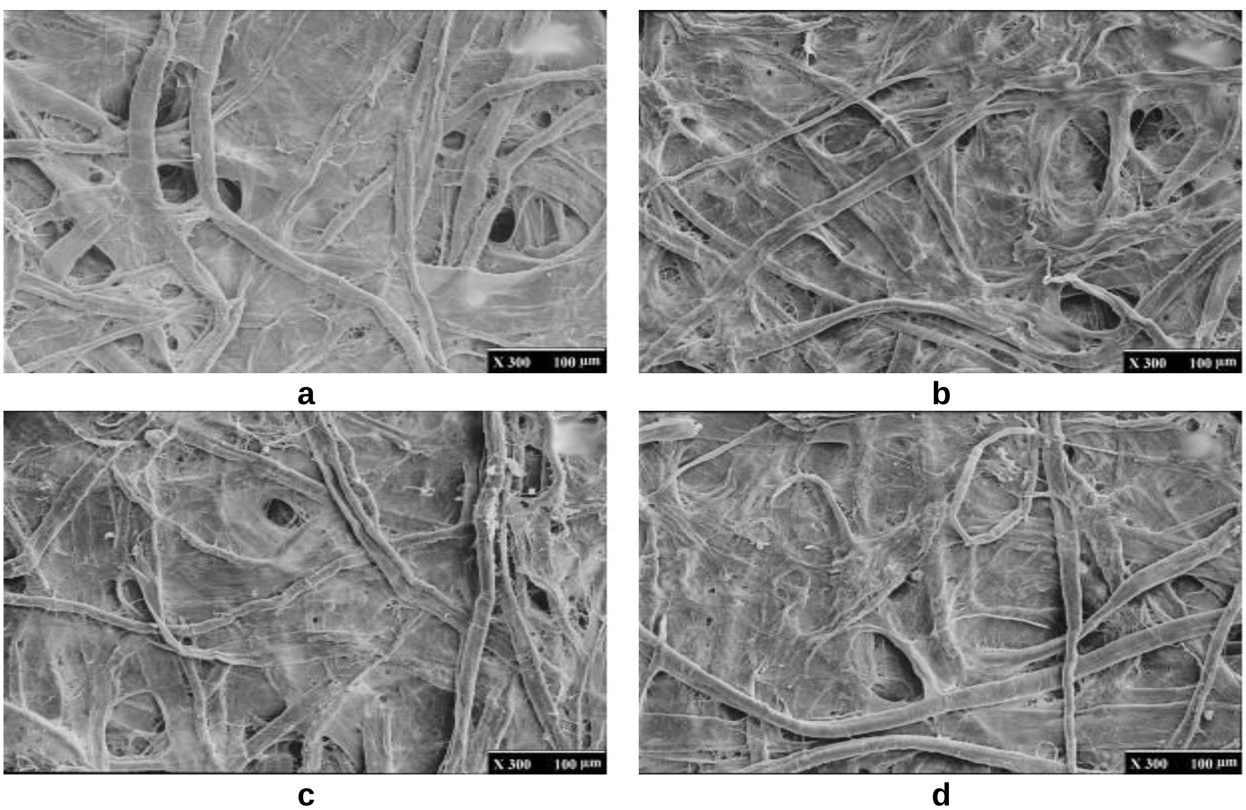
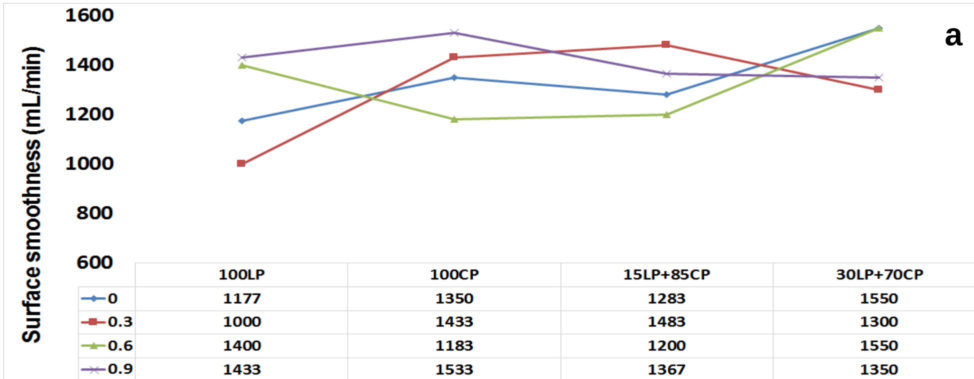
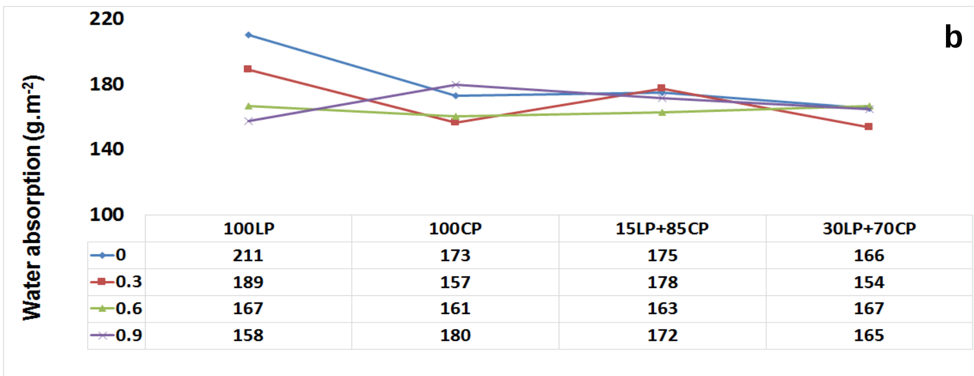


Fig. 6. The surfaces of papers made from 30% chemical pulp and 70% cotton pulp and a) 0% nano-cellulose, b) 0.3% nano-cellulose, c) 0.6% nano-cellulose, and d) 0.9% nano-cellulose



	100LP	100CP	15LP+85CP	30LP+70CP
0	1177	1350	1283	1550
0.3	1000	1433	1483	1300
0.6	1400	1183	1200	1550
0.9	1433	1533	1367	1350



	100LP	100CP	15LP+85CP	30LP+70CP
0	211	173	175	166
0.3	189	157	178	154
0.6	167	161	163	167
0.9	158	180	172	165

Fig. 7. The effect of nano-cellulose and pulp and paper type on a) Surface smoothness, and b) Water absorption

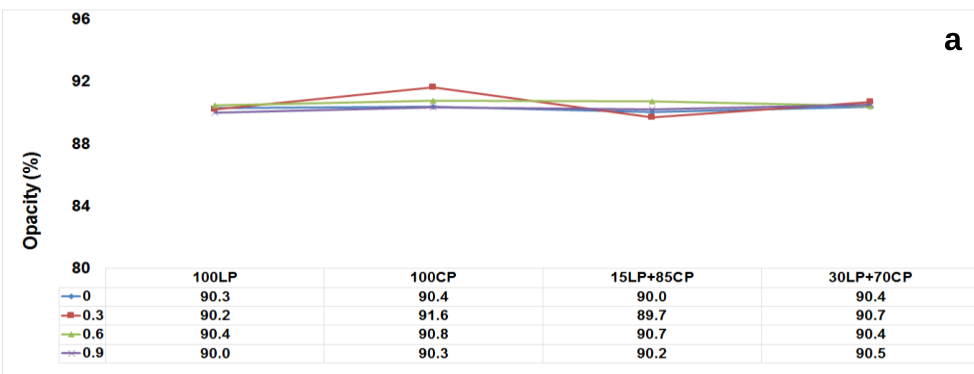
Figure 7 shows the effects of type of pulp and paper and nano-cellulose on the physical, mechanical, and optical properties of handsheets. Figure 7a shows that the lowest surface smoothness, 1000 mL/min, was associated with 100% chemical pulp and 0.3% nano-cellulose, and the highest surface smoothness, 1550 mL/min, was associated with the use of 70% cotton pulp without nano-cellulose. When nano-cellulose is added at 0.9%, the increase in surface smoothness should be observable in the obtained papers made by independent pulp. But in combined pulp and paper, the greater the ratio of chemical pulp, the more surface smoothness could be achieved during the consumption of higher amounts of nano-cellulose. Thus, in the furnish ratio of 15:85, the most efficient amount of nano-cellulose was the 0.3% level, while in the furnish ratio of 30:70 the efficient consumption was at 0.6%, as seen in the obtained graphs.

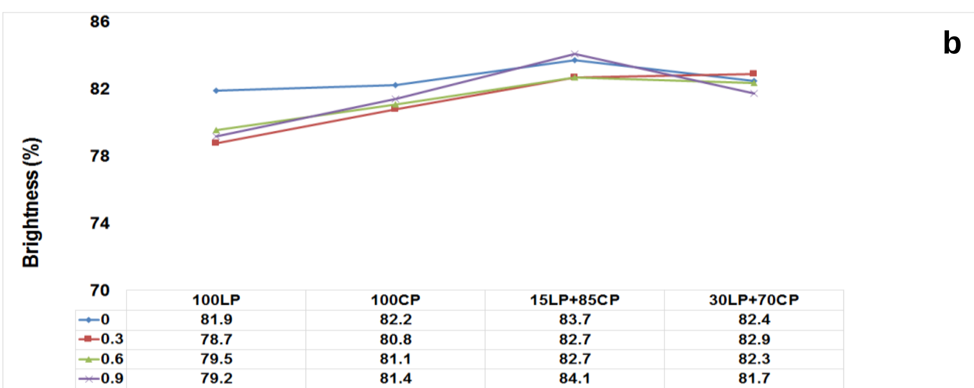
Figure 7b shows that the lowest amount of water absorption, 154 gr/m^2, was associated with 70% cotton pulp and 0.3% nano-cellulose, while the highest amount of water absorption, 211 gr/m^2, was associated with 100% chemical pulp along with 0% nano-cellulose. One of the most important properties for the natural durability of paper in environmental conditions is its ability to control liquid absorption (Ashori and Raverty 2007). The results of measurements have shown that the nano-cellulose reduces the water absorption of paper; as nano-cellulose has more molecular mass and more viscosity, the rate of water permeation is reduced, and this reduction of water absorption can improve paper printability (Ashori *et al*. 2005). Duncan tests on handsheets showed that the use of nano-material in separated pulp causes reverse effects on paper water absorption. Meanwhile, in combined pulp and paper, the higher the ratio of chemical pulp to paper, the lower the rate of water absorption will be. Increasing the amount of nano-cellulose (especially at the 0.3% level) has a significant effect on reducing the water absorption, and thus this level can be identified as the most efficient level of nano-cellulose.

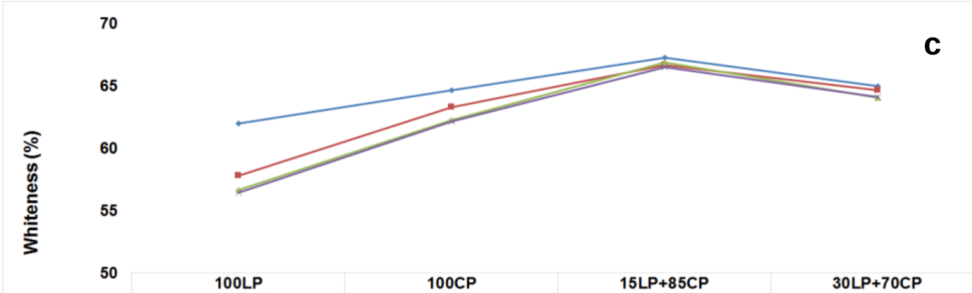
Figure 8a indicates that the lowest opacity, 89.7%, was associated with 85% cotton pulp and 0.3% nano-cellulose, and the highest opacity, 91.6%, was associated with 100% of cotton pulp along with 0.3% nano-cellulose. In fact, opacity can be achieved through light absorption of paper. Various tests have been carried out on the opacity factor and have shown that paper composed of 100% cotton fibers and 0.3% nano-cellulose had the highest level of opacity. However, in general, especially in paper composed of both pulp and paper, enhancement of the nano-cellulose level did not have a significant effect on the factor.

Figure 8b shows that the lowest brightness, 78.7%, was associated with 100% cotton pulp and 0.3% nano-cellulose, and the highest brightness, 84.1%, was associated with 85% cotton pulp with 0.9% nano-cellulose. When nano-cellulose was consumed at 0.9%, a reduction in brightness could be observed. The obtained graphs showed that the addition of nano-cellulose to chemical pulp caused an extreme reduction in brightness, especially at the 0.3% level. Despite the fact that combining pulp in the ratio of 15:85 and injection of 0.9% nano-cellulose has resulted in the highest brightness, the purpose of present study is enhancement of consumption of chemical pulp in the production of durable papers. Thus, according to the graphs, combining pulp in the ratio of 30:70 and injection of 0.3% nano-cellulose was identified as the most efficient level for achieving the best level of paper brightness.

Figure 8c shows that the highest whiteness, 67.2%, was associated with 85% cotton pulp and 0% nano-cellulose, and the lowest whiteness, 56.4%, was associated with 100% chemical pulp and 0.9% nano-cellulose.







	100LP	100CP	15LP+85CP	30LP+70CP
0	62.0	64.6	67.2	65.0
0.3	57.8	63.3	66.7	64.7
0.6	56.7	62.3	66.9	64.1
0.9	56.4	62.1	66.5	64.1

Fig. 8. The effect of nano-cellulose and pulp and paper type on a) Opacity, b) Brightness, and c) Whiteness

CONCLUSIONS

1. The paper made using bleached softwood kraft pulp and 0.3% or more of nanofibrillated cellulose (NFC) had higher tensile strength, bursting strength, tearing strength, and folding strength than papers made from other pulps.
2. By adding nano-cellulose up to a level of 0.9% in cotton pulp, the tensile strength, bursting strength, and tearing resistance were increased by 33, 33.5, and 6.6%, respectively, compared to the control sample.
3. By increasing nano-cellulose up to 0.9% in cotton pulp, the surface smoothness increased by 13.5% compared to the control sample, and the porosity and water absorption were reduced by 16.6 and 4%, respectively, compared to the control sample.
4. The achieved results represent important contributions to the production of durable papers. Based on the purpose of the present study, the treatment of paper with 0.3% nano-cellulose is suggested to improve most of the parameters of durable paper.
5. SEM results showed that increase in the percentage of nano-cellulose reduced porosity, which increases the printability of durable papers.

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