

# Effects of Montmorillonite Clay on Mechanical and Morphological Properties of Papers Made with Cationic Starch and Neutral Sulfite Semichemical or Old Corrugated Container Pulps

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The impact of montmorillonite (nanoclay) addition on the mechanical and morphological properties of paper made from neutral sulfite semichemical (NSSC) and old corrugated container (OCC) pulps was evaluated in systems utilizing cationic starch for dry-strength. The nanoclay amount was considered at five levels, and cationic starch was applied at a 1% level. In OCC pulp, by increasing the nanoclay up to 4%, the tensile index and corrugating medium test (CMT) were increased; however, there was a decrease when the nanoclay was increased to 8%. By increasing the nanoclay to 2%, the tear and burst indexes and ring crush test (RCT) increased, but there was a reduction when the nanoclay was increased to 8%. Average air resistance tended to increase as the nanoclay was increased to 8%. In the case of NSSC pulp, by increasing the nanoclay amount from 0 to 8%, the average tensile, tear, and burst indexes decreased, while the air resistance increased. The CMT amount increased with increasing nanoclay content to 2%, but a reduction was seen for other levels. The RCT exhibited an increase with increasing nanoclay up to 4%, after which it decreased. Results suggest that the proportion of nanoclay added with cationic starch needs to be optimized, depending on furnish conditions.

*Keywords:* OCC pulp; NSSC pulp; Nanoclay; Mechanical properties; Morphological properties

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## INTRODUCTION

Mineral fillers used in the paper industry include calcium carbonate (both in crushed and chemically precipitated form), talc, titanium dioxide, clay fillers, and other materials. By adding fillers, the paper physical properties of appearance, brightness, opacity, whiteness, clarity, printability, and smoothness can be improved and the cost of production can be reduced, but the reduction in strength and abrasiveness are among some of the common disadvantages of adding fillers (Afra 2005). By filling paper's pores, fillers have the ability to increase the air resistance of paper (Nazeri 2007). Furthermore, these materials increase the paper opacity (Hagemeyer 1997; Laufmann 1998). However, as has been widely noted, adding fillers reduces the overall paper strength properties (Afra *et al.* 2014).

Nanotechnology is an emerging technology that has influenced various areas of human life in recent decades. Accordingly, using nanotechnology, the paper industry has

developed as a leading industry in the last two decades. Today, among the various nano-scale mineral additives, nanoclay particles (*i.e.* montmorillonite or “bentonite”) are common because these particles have the possibility of generating different structures (intercalated, intercalated-flocculated, and exfoliated structures), and the given structure will have an impact on the product’s properties (Tjong 2006).

Nanoclay particles are very small (less than 2  $\mu\text{m}$ ) and extremely thin when fully exfoliated (approximately 1 nm). The clay has a layered structure and its internal surfaces are not accessible. If its layered structure is effectively separated, then a higher surface area, smaller size, higher light scattering (dispersion), and higher optical properties can be achieved. As the clay particles become smaller and penetrate the micro-fibrils, making fewer pores, there is an increase in the resistance to passage of air, while maintaining other strength properties.

Mokhtari *et al.* (2013) examined the application of a cationic starch-nano-bentonite system on improving the OCC pulp strength properties. Their results shed light on the fact that the addition of nano-bentonite led to a dramatic drop in the tensile strength and burst index, but the addition increased the resistance to air passage and the ash content of the paper. In a study conducted on the incorporation of recycled OCC pulp and hardwood NSSC pulp, the results showed that the recycled OCC pulp had better strength characteristics than the NSSC pulp (Asadpour *et al.* 2008). The results of a study by Rasa *et al.* (2012) to improve the OCC pulp strength characteristics through refining and using cationic starch and imported long-fiber pulp showed that all treatments improved OCC pulp strength properties. Afra *et al.* (2014) made a comparative analysis of the physical, mechanical, and optical properties of papers containing nanoclay and homogenized nanoclay; their results revealed that the application of nanoclay with a homogenizer, compared with that of raw nanoclay in the presence of a preservative, increased the density and the resistance to air passing through the paper. In the meantime, the burst and tear strength values showed a slight reduction. Finally, the high levels of durability in the case of homogenized nanoclay, compared with raw nanoclay in the presence of the preservative represent an economical system with less wastewater pollution.

The objective of this study was therefore to evaluate the impact of nanoclay, OCC, and NSSC paper pulp on the physical, mechanical, and morphological characteristics of handsheets in systems where cationic starch was being used as a dry-strength additive.

## EXPERIMENTAL

### Materials

#### *Old corrugated container (OCC) pulp*

This study used 100% recycled old corrugated cardboard that was prepared at an Iran cardboard company. After the removal of physical contaminants and impurities, cartons (cardboard) were converted into small pieces by hand. To separate the fibers, the cardboard pieces were kept at a solids content of 10% in water for 4 h and were turned to pulp in a laboratory mixer with a concentration of 0.2% and 3000 rpm.

#### *Neutral sulfite semichemical (NSSC) pulp*

Neutral sulfite semichemical pulp was prepared from the NSSC pulp storage tower in the Mazandaran Wood and Paper industry and transferred to the laboratory. Then, using

a laboratory PFI mill, the pulp was prepared according to the TAPPI T248 sp-00 (2000) standard to a drainability level of 300 mL (CSF).

#### *Cationic starch*

The cationic starch with a degree of substitution (DS) of 0.035 was prepared from Glucosan® Company (Ghazvin, Iran) and was used at 1% of the paper pulp dry weight. Because some starches are insoluble in cold water, the cationic starch used in this study must be heated to dissolve it. To be soluble in water, a beaker with water and starch at a concentration of 5% was kept for 30 min at 90 °C, and when the starch dispersed and was absorbed on the fiber surface, pulp slurry with 0.03% dryness was added. 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)
Degree of substitution	0.035

#### *Nanoclay*

The nanoclay (K10) for the study was obtained from Sigma-Aldrich (Sigma-Aldrich Company Ltd., Saint Louis, MO 63103, USA 3050) and was used at 5 levels, *i.e.*, 0%, 2%, 4%, 6%, and 8%, proportional to the dry weight of the paper pulps. To do so, nanoclay particles were placed into distilled water for 24 h to swell. Then, the nanoclay-distilled water suspension with a concentration of 10 g/L was prepared and stirred continuously for 4 h using a magnetic stirrer. The nanoclay properties are presented in Table 2.

**Table 2.** Nanoclay Properties\*

Trade Name	K10
Base	Montmorillonite
Product Number	281522, CAS
Density	300-370 kg/m <sup>3</sup>
Anion	Chloride
Modifier Concentration	48 meq/100 g
Moisture	1%-2%
Weight Loss on Ignition	30%

\*Note: According to Producer Information

#### **Preparing Handsheets**

To prepare 127 g/m<sup>2</sup> handsheets, the fiber suspension was placed in a mixer, 1% cationic starch solution (based on dry mass) was combined with the pulp suspension, and various concentrations of nanoclay were added to the mixture for two different pulps (OCC and NSSC). At the end, eight handsheets were made for each treatment according to TAPPI T205 sp-02 (2002) standards for each of pulps.

### Determining the Pulp Characteristics

Mechanical tests such as tensile, burst, and tear indexes, as well as CMT and RCT state, were conducted on handsheets using TAPPI standards T494 om-01 (2001), T403 om-02 (2002), T414 om-04 (2004), T809 om-99 (1999), and T818 cm-97 (1997), respectively. To measure the air resistance, the Gurley Machinery was used, which is commonly referred to as a densometer test. In the given Gurley method, the values represent the air resistance.

### Scanning Electron Microscope (SEM)

To evaluate the presence of nanoclay particles on the surface fibers and paper, a scanning electron microscope of JXA-840 made by JEOL Company, Japan, was used.

### X-ray Diffraction Test

X-Ray diffraction tests were done using an X-ray diffraction device (XPert model) manufactured by the Philips company of The Netherlands. The test was done by applying radiation from a cobalt lamp with a wavelength of 1.79 Å, step 0.02 degrees, speed of 0.3 degrees per second and emission angle  $2\theta$  in the range of 1-9 degrees. Samples were prepared as plate forms with dimensions of  $1 \times 1 \times 10 \text{ mm}^3$ , and the device generating power adjustments were set to 30 mA and 40 KV.

### Statistical Analysis

The data were analyzed using SPSS statistical software (IBM Software, Armonk, New York; version 11.5) with a complete randomized factorial test and then the averages were compared and grouped using Duncan's test at 95 percent significance level ( $P=0.5$ ).

## RESULTS AND DISCUSSION

The effects of nanoclay levels on the tensile index, tear index, burst index, RCT, CMT, and air resistance were significant in NSSC pulp. In OCC pulp, there were not significant differences in tear index at different nanoclay levels. Also, nanoclay levels had significant differences on the tensile index, burst index, RCT, CMT, and air resistance in OCC pulp.

**Table 3.** Analysis of Variance (F-Value and Significance Level) of the Results of Pulp and Nanoclay Particles on Handsheets

Property	Tensile index (Nm/g)	Tear index (mN.m <sup>2</sup> /g)	Burst Index (kPa.m <sup>2</sup> /g)	Ring crush test (KN/m)	Corrugated medium test (N)	Air resistance (S)
OCC pulp	6.605*	2.460ns	5.167*	3.567*	10.476*	4.965*
NSSC pulp	6.041*	7.030*	6.054*	6.177*	9.654*	12.654*

\* 95% significance level    <sup>ns</sup> no significance

### Tensile Index

The tensile index changes for handsheets made from NSSC and OCC pulps at various nanoclay levels are shown in Fig. 1. The average tensile index of handsheets made from OCC pulp was far more than that of handsheets made from NSSC pulp. The tensile index is an indicator of the paper tensile potential durability caused by paper utilization level under tensile stress. The most important factor affecting the paper tensile index is the

quality and number of connections among fibers (Afra 2005). Increasing the fiber connections to each other, which is caused by increased refining or wet pressing, will increase the paper tensile index. However, paper tensile index will always be less than that of the fiber (Afra 2005). Tensile index in the machine direction is always greater than that in the cross direction because the fibers are aligned more in the longitudinal direction than in the cross direction. Considering the machine direction, two connection categories are being placed under tension, covalent bonding (O-C, C-C) within and between the existing glucose in cellulose chains, and hydrogen bonds between the fibers. Generally, there are higher covalent bonds in the machine direction and fewer covalent bonds are observed in the cross direction (Hamzeh and Rostampour 2008). Considering handsheets, the longitudinal and cross directions do not make any sense because the fibers are randomly oriented. For the tensile index, both the connection strength between the fiber and the fiber itself will experience tension. Therefore, the fibers length and stronger connections between the fibers improve the strength. When using recycled OCC pulp, the tensile index is higher than that of the pure NSSC pulp, which can be related to longer OCC fiber structure, more flexibility, the higher attraction to connections, and higher connection strength than the NSSC pulp.

Increasing the nanoclay levels may form a suitable binary system with cationic starch, providing an enhanced dry-strength system. As a result, by increasing the nanoclay level to 4%, first an increase was witnessed in the tensile index. However, as the treatment included more nanoclay, the tensile index faced a reduction. Such a decrease is suggestive of weak boundaries created within the paper structure. As the relative amount of nanoclay in the mixture is increased, it becomes increasingly likely that the clay particles will end up facing each other directly within the paper. In other words self-agglomeration of clay particles will become increasingly prominent. The weakness of the boundaries created by such self-agglomeration is already well known, since montmorillonite is known to exfoliate easily into thin platelets. In other words, when the ratio of nanoclay to cationic starch is high, there may be insufficient cationic starch available in order to cover the available surfaces of the nanoclay platelets, and lower strength results can be expected.

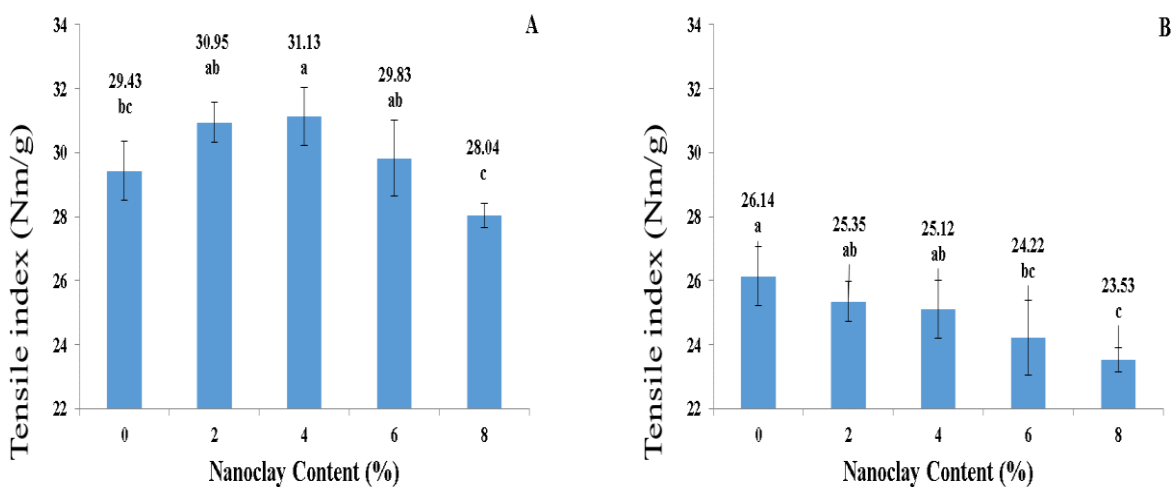
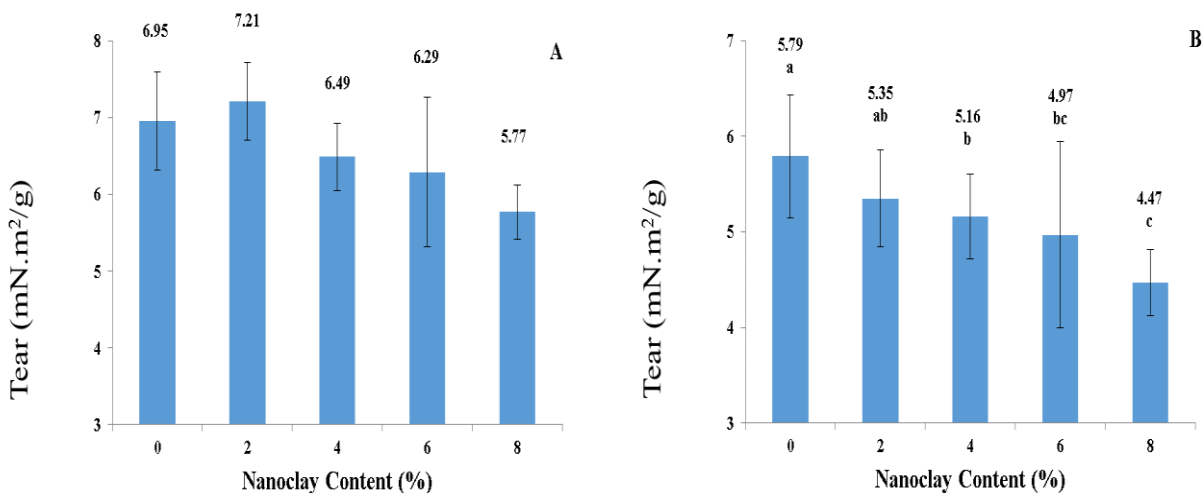


Fig. 1. The effect of nanoclay on tensile index, A) OCC pulp, B) NSSC pulp

## Tear Index

The tear index of handsheets made from NSSC and OCC pulp at various nanoclay levels is shown in Fig. 2. Handsheets made from OCC pulp showed far higher tear index than paper made from NSSC pulp. One of the parameters affecting the tear index is the fiber length and strength, but if these two factors are fixed, then the bonds between the fibers can affect the tear index. Maintaining an increase in the proportion of long fibers to short fibers increases the overall tear index. Therefore, considering the above characteristics and a high percentage of lignin and short NSSC pulp fiber, a lower level of strength seems logical. Tear index primarily depends on fiber length and the connection between the fibers. The OCC pulp has longer fibers than the NSSC pulp, but in contrast, the content of fine fibers in NSSC pulp is higher than in the OCC pulp (average fiber length of NSSC pulp will increase towards the recycled pulp); therefore, the OCC pulp can achieve a higher value of tear index because it has longer fibers and stronger connections between the fibers (Asadpour *et al.* 2008). In fact, the fiber length has a positive effect on most paper properties, especially paper tear index. Because nanoclay particles are so small, by increasing the nanoclay level, it seems likely that, however, the generated flocs form a better bond; on the other hand, they create a sheet with open structure leading to low strength areas. Because the nanoclay particles are placed among the fibers, which weakens fiber bonding, the strength between the fibers decreases, but the individual fiber strength does not change; as a result, the tear index loss is negligible, which is similar to results obtained by Lindström *et al.* (2008).



**Fig. 2.** The effect of nanoclay on tear index, A) OCC pulp, B) NSSC pulp

## Burst Index

The burst index of handsheets derived from NSSC and OCC pulps at various nanoclay levels is shown in Fig. 3. The handsheets made from OCC pulp showed higher burst index than those made of NSSC pulp. The burst index is based on the force required to deform paper with an expandable rubber membrane, determined by measuring the hydraulic pressure at the paper tearing point (bursting). Increasing the fiber length, enhancing the refining, and pressing would all increase the burst index, whereas increasing the fillers and poor formation will reduce the strength. The burst index depends on the fiber length and the bonds between the fibers, but is mostly affected by the bonds between the

fibers. The burst index is proportional to the square of the average fiber length (Asadpour *et al.* 2008; Akbarpour 2009). The NSSC pulp exhibited the smallest burst index, caused by shorter fibers, less flexibility, and a weak bond between the fibers. As the fibers become thinner and more flexible, the bonding between the fibers increases because of additional hydrogen bonding, which improves the burst index. By increasing the application of longer fibers, the burst index will increase, so that when recycled OCC pulp is used, the burst index rate increases.

At first, the application of nanoclay led to an increase in burst index, but by increasing the nanoclay level a decrease in burst index was observed. The results again were consistent with a mechanism in which the amount of nanoclay needs to be in a suitable range, relative to cationic starch, in order to provide a strengthening effect. Nanoclay particles, in the absence of cationic starch, would merely act as mineral fillers and weaken the capacity of fiber bonding. And at too high a proportion of nanoclay, there would be weak junctions within any agglomerates of nanoclay in the structure. The present results regarding the burst index of paper treated with clay particles are similar to results reported by Neimo (2000). Research also has shown that the addition of fillers reduces the strength and that clay, talc, and crushed calcium carbonate are highly responsible for mechanical strength loss (Afra *et al.* 2014).

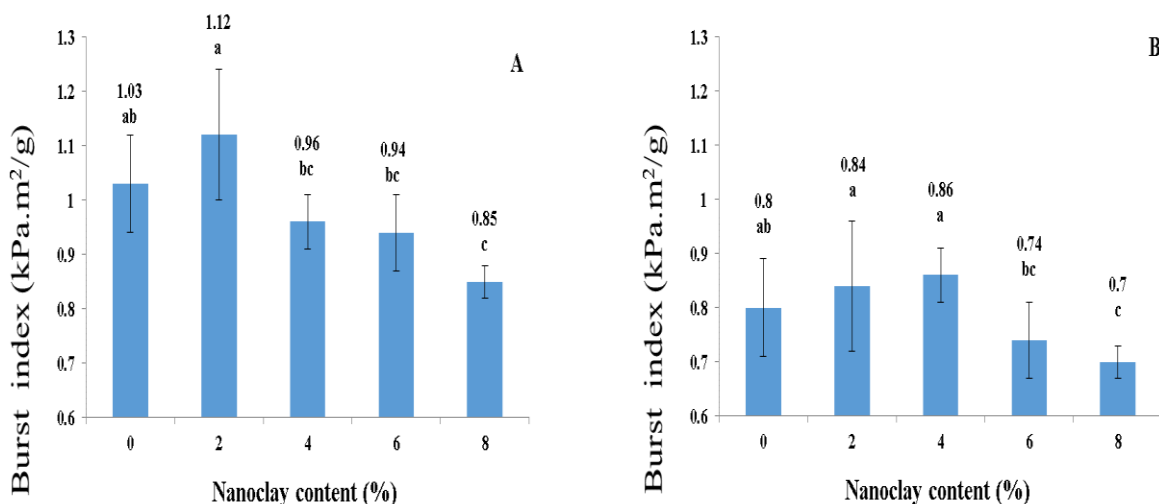
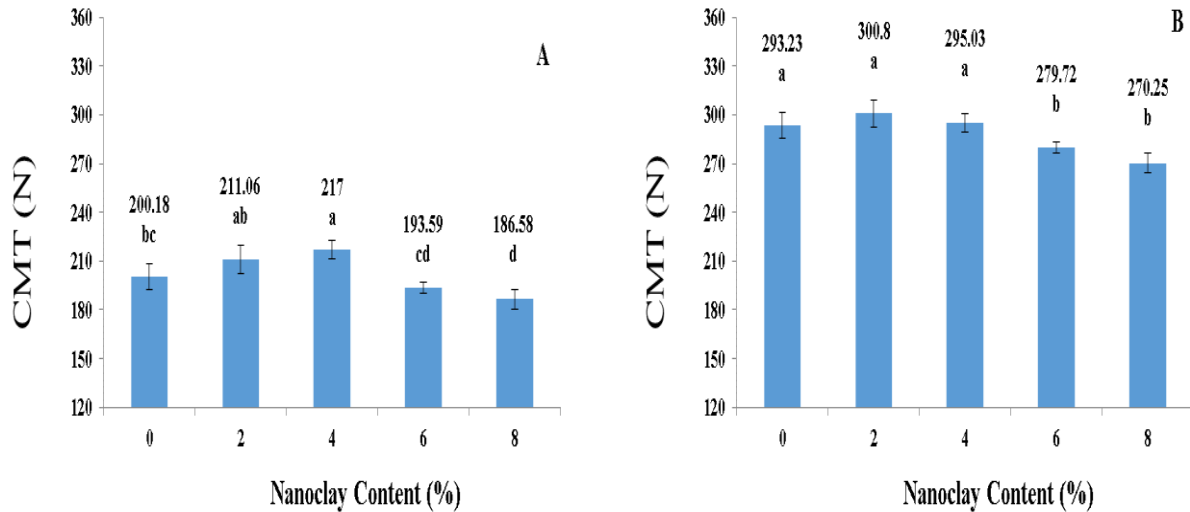


Fig. 3. The effect of nanoclay on burst index, A) OCC pulp, B) NSSC pulp

### Corrugating Medium Test

The crushing strength in the corrugated medium test (CMT) for handsheets derived from OCC and NSSC pulps at various nanoclay levels is shown in Fig. 4. The average CMT for handsheets made from NSSC pulp was much higher than that for handsheets made from OCC pulp. Because semichemical pulps have a good stiffness, they are the best types of pulps to produce corrugated paper; therefore, the highest CMT amount is related to the time when only 100% NSSC pulp is used. Because of the high lignin content, on the one hand, and the high percentage of hemi-cellulose, on the other hand, the NSSC pulps have a high stiffness. In fact, in primary NSSC pulp made from hardwood, both the diameter and wall thickness are high because the force is perpendicular to the fiber length; therefore, they have a high crushing strength in CMT.

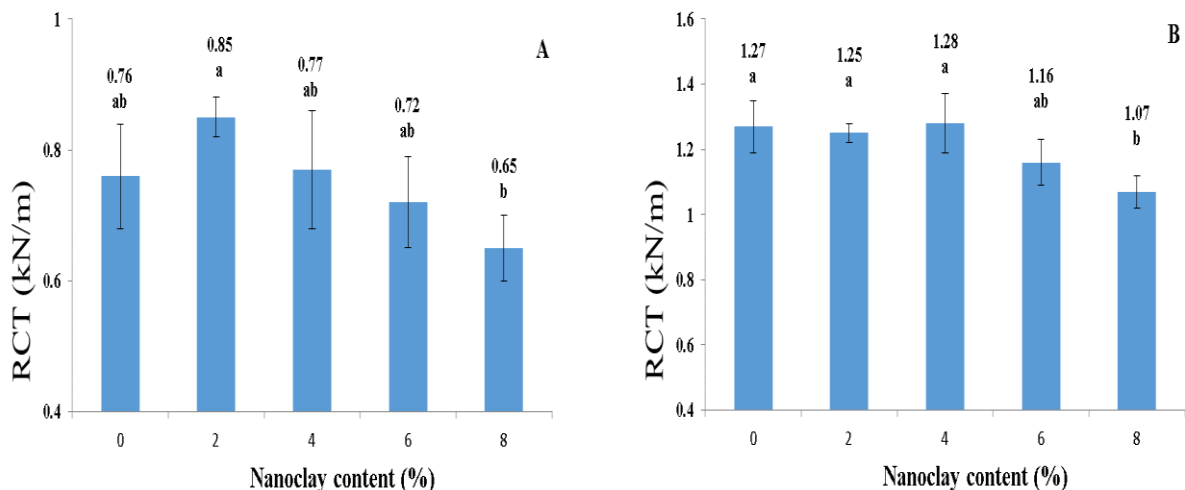


**Fig. 4.** The effect of nanoclay on corrugated medium test (CMT), A) OCC pulp, B) NSSC pulp

Since the CMT test measures the resistance of the beams of wavy layers and there can be significant forces perpendicular to the fiber length, a long fiber pulp that has thin cell walls may be crushed by the pressure, leading to a drop in the strength. When using OCC pulp, this strength will decrease. Using the nanoclay up to 4% results in increased crushing strength in CMT, but with higher levels of nanoclay, the crushing strength will decrease because of the same factors as mentioned earlier.

### Ring Crush Test

Results for the crushing strength in the ring crush test (RCT) for handsheets made from OCC and NSSC pulps at various nanoclay levels are presented in Fig. 5. The average crushing strength in RCT for handsheets derived from NSSC pulp was much higher than that of handsheets formed using OCC pulp.



**Fig. 5.** The effect of nanoclay on ring crush test (RCT), A) OCC pulp, B) NSSC pulp

The RCT results can be attributed to the pressure and force on the cardboard edge and to a large extent are proportional to the CMT. The force is applied to the cylinder axis. NSSC pulp exhibited the highest RCT. Because of the fiber diameter and thickness of the



wall (from the higher lignin content) on the one hand and the high percentage of hemicellulose, on the other, the primary NSSC pulp from hardwood enjoyed a satisfactory stiffness. Generally, semi-chemical pulps have an acceptable stiffness and are strong against crushing. That is, as the wall thickness is increased, the strength will also increase (Asadpour *et al.* 2008). The presence of long fibers with thin cell walls crushed by the pressure would lead to lower strength. As a result, such strength would be further reduced when using OCC pulp.

Because fines have good bonding properties, by adding nanoclay as a result of links between fibers, fines, and cationic starch, there was an increase of the RCT. But as the nanoclay level was increased, the crushing strength decreased. A high level of nanoclay addition would be expected to result in a lot of self-agglomeration of clay. These weak agglomerates would provide points of weakness in the dried structure.

### Air resistance

The air resistance of handsheets made from OCC and NSSC pulps at various nanoclay levels is presented in Fig. 6. The average air resistance of handsheets made from NSSC pulp was much higher than that of handsheets made from OCC pulp. Comparing the long fibers to those of hardwood NSSC pulp, OCC pulp has less air resistance because the short NSSC fibers are twisted together and are stronger than the OCC pulp. By increasing the nanoclay level, the additives and fiber durability in paper pulp increased partly because of the filling of pores and felt fibers with fibers, and cationic starch resulted in an increased state of air resistance. According to previous research, by increasing the amount of filler, the air resistance will also increase. This is due to the filling of felt fiber pores with filler, blocking the air passing through the paper (Nazeri 2007). The better dispersion of filler particles improves the air resistance (Hubbe and Gill 2004). Because of the plate structure of nanoclay particles, the felt fibers and pores will be filled, preventing air from passing.

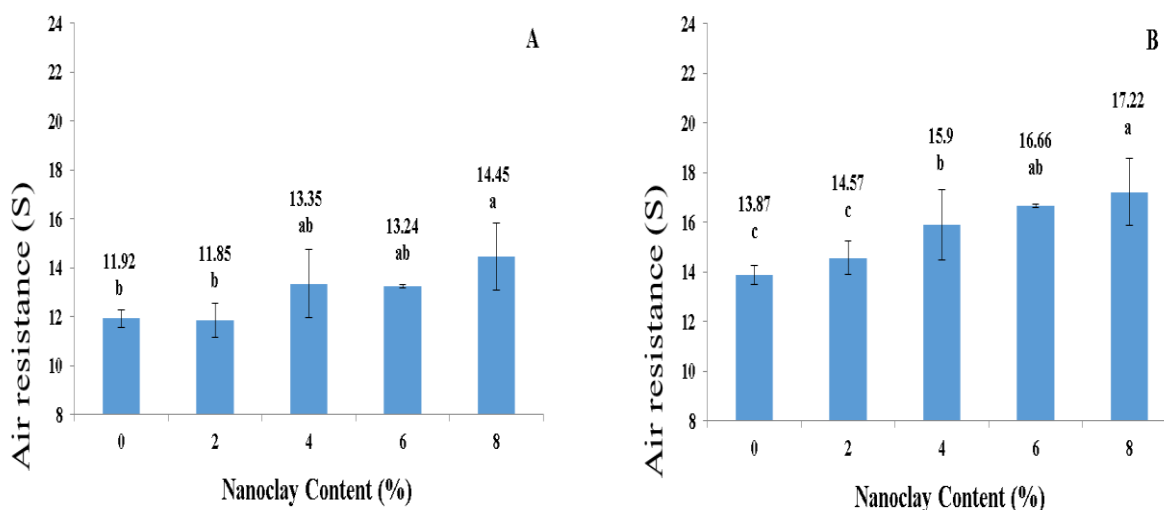


Fig. 6. The effect of nanoclay on air resistance, A) OCC pulp, B) NSSC pulp

### Scanning Electron Microscopy

SEM images of the surface of handsheets made from NSSC and OCC pulps at 0%, 4%, and 8% nanoclay are shown in Figs. 7 and 8. With increasing nanoclay level, the nanoclay particles partially filled the fiber pores, increasing the air resistance.

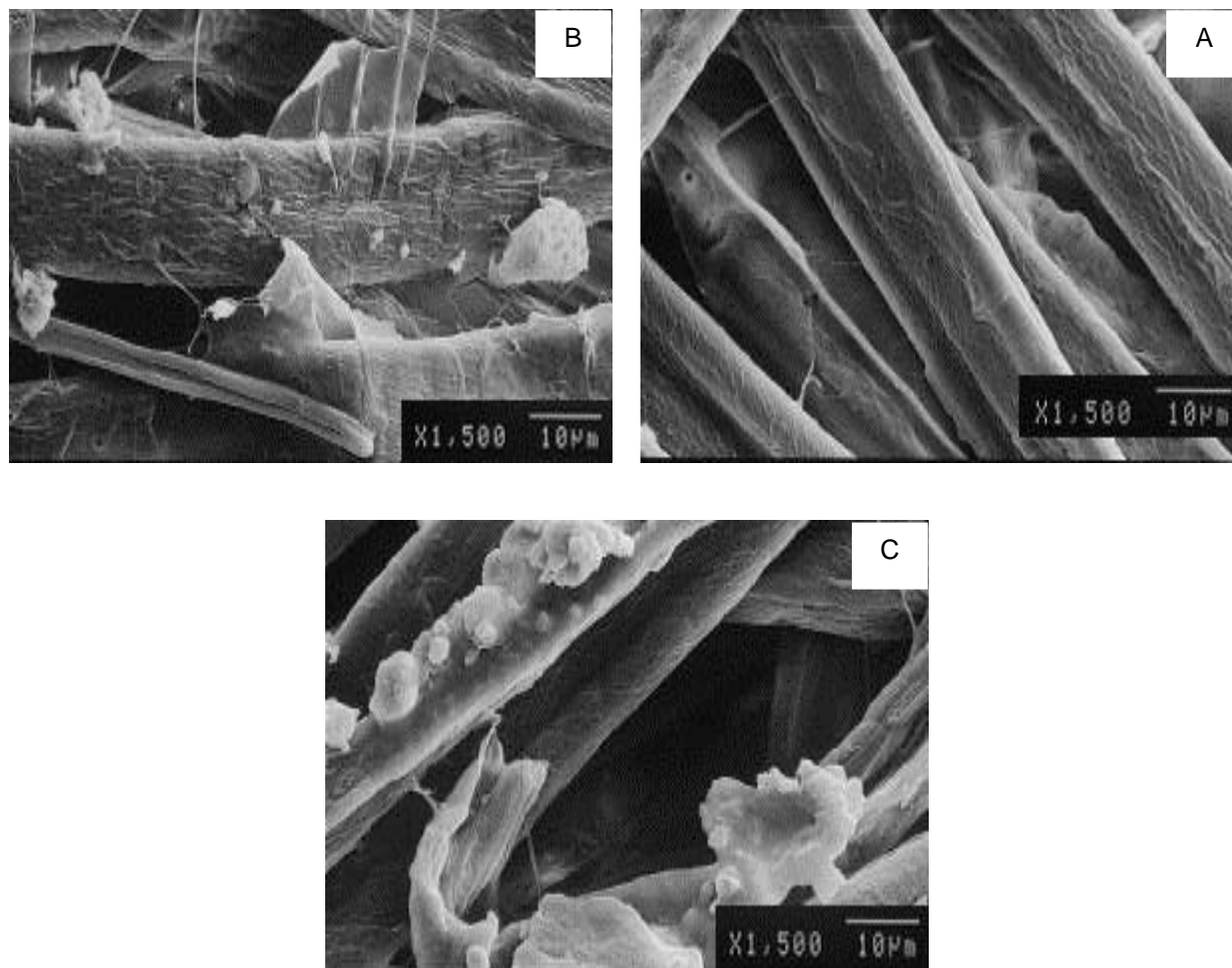


Fig. 7. SEM image prepared from OCC fibers: A) 0% nanoclay, B) 4% nanoclay, C) 8% nanoclay

### Structural Study by X-ray Diffraction Test (XRD)

By increasing the nanoclay content to 8% of the total weight in NSSC pulp, the peak angle would increase up to  $2\theta$ .

As it is witnessed in Fig. 9, the pure nanoclay XRD peak was at  $2\theta=6/9^\circ$  angle and the layer spacing was  $14/87 \text{ \AA}$ . By increasing the nanoclay to 4% of the total weight, the XRD peak was transferred to the bigger angle of  $2\theta=7/01^\circ$  with a layer spacing of  $d=14/63 \text{ \AA}$ . By increasing the nanoclay content to 8 percent of the weight, the XRD peak moved forward and reduced the silicate layers spacing ( $2\theta=7/01^\circ$  and  $d=14/56 \text{ \AA}$ ).

Considering the OCC pulp, when increasing the nanoclay content to 4% of the total weight, the XRD peak was transferred to a bigger angle ( $2\theta=8/14^\circ$  and  $d=12/61 \text{ \AA}$ ). The XRD peak moved forward and reduced the silicate layers spacing when the nanoclay content in OCC pulp was increased to 8 percent of the total weight ( $2\theta=8/24^\circ$  and  $d=12/46 \text{ \AA}$ ). As it is observed, the morphological structure is of interlayer type.

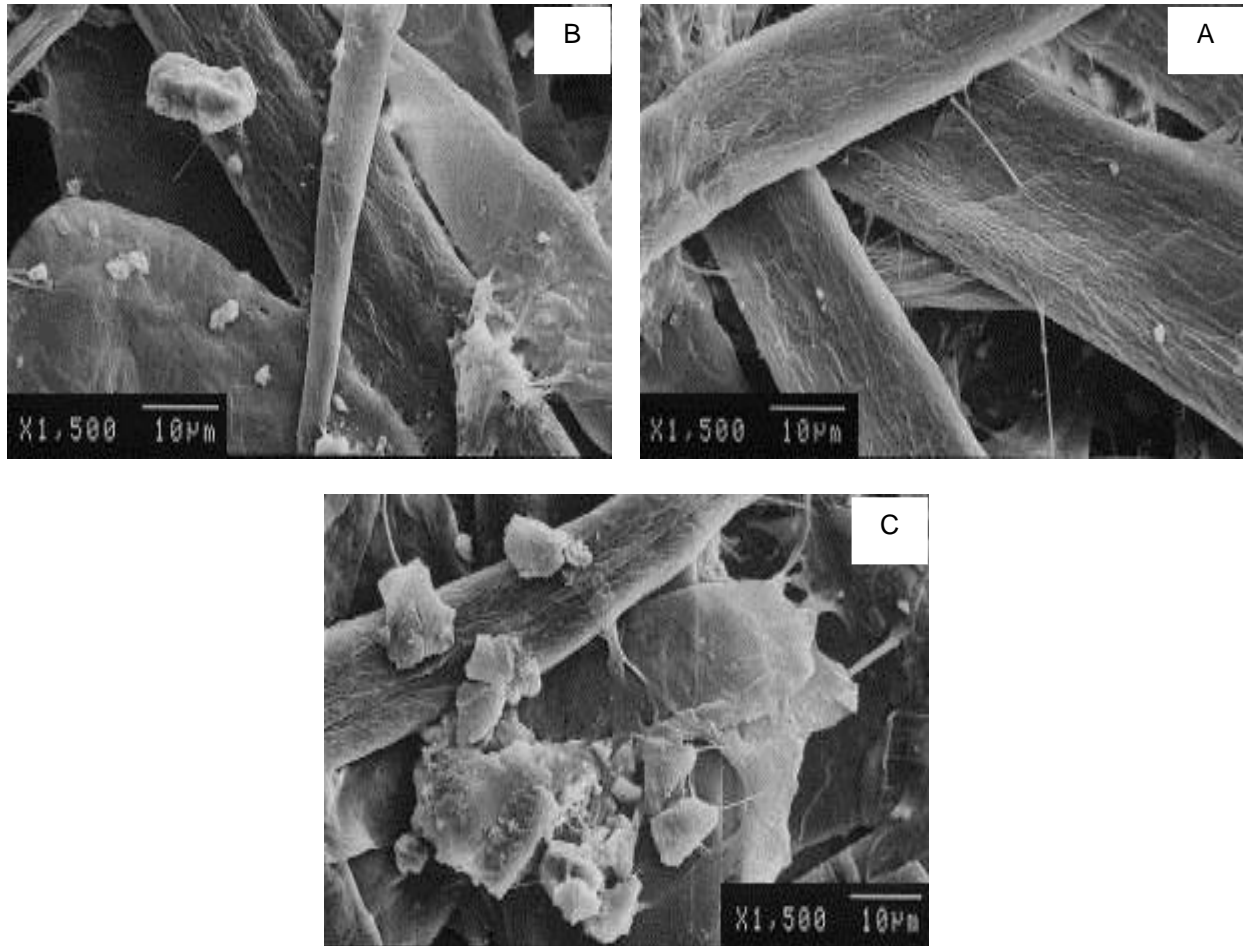


Fig. 8. SEM image prepared from NSSC fibers: A) 0% nanoclay, B) 4% nanoclay, C) 8% nanoclay

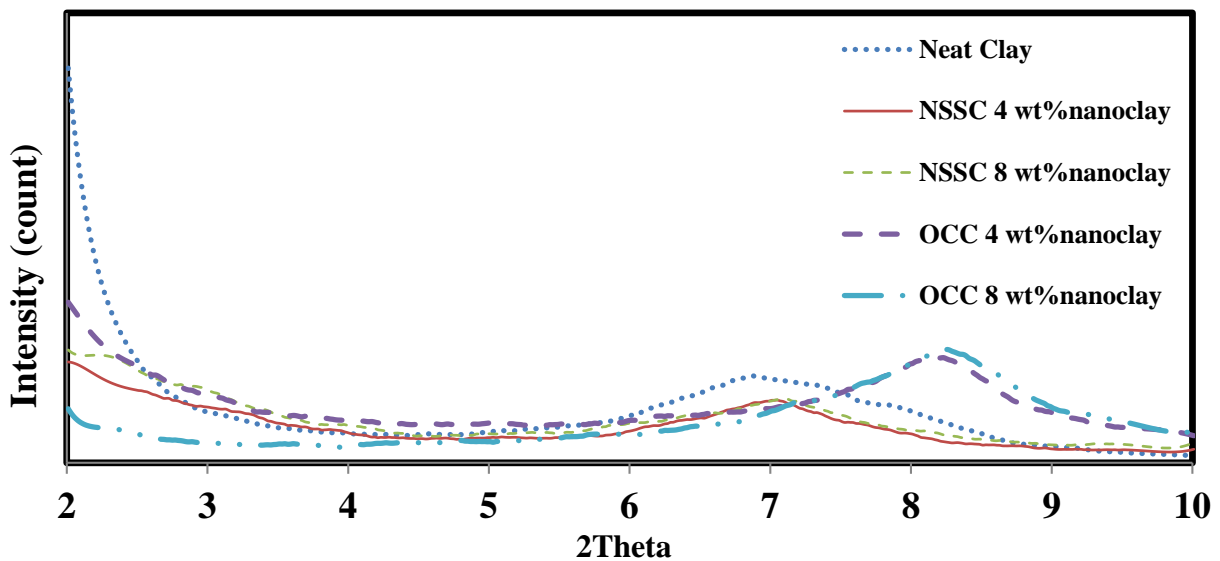


Fig. 9. The XRD ray range on papers made from OCC and NSSC pulp containing the 4% and 8% nanoclay

## CONCLUSIONS

1. For OCC pulp, by increasing the nanoclay amount to 4%, the tensile strength and CMT showed increases of 5.8% and 8.4%, respectively, while using 8% nanoclay resulted in decreases of 4.9% and 7.3%, respectively.
2. Regarding the NSSC pulp, by increasing the nanoclay level up to 8%, the tensile index, tear index, and burst decreased by 11.1%, 29.5%, and 14.3%, respectively, while the air resistance increased by 24.2%.
3. In OCC pulp, by increasing the nanoclay amount to 2%, the tear index, burst index, and RCT increased by 3.7%, 8.7%, and 11.8%, respectively, while using 8% nanoclay led to decreases of 20.5%, 21.2%, and 16.9%, respectively.
4. For OCC pulp, by increasing the nanoclay up to 8%, the air resistance increased by 21.2%.
5. In NSSC pulp, by increasing the nanoclay to 2%, CMT increased by 6.2%, while using 8% of nanoclay brought a decrease of 8.5%.
6. By increasing the nanoclay amount to 4% in NSSC pulp, the RCT increased by 0.8%, while using 8% nanoclay resulted in a decrease of 18.7%.
7. The SEM results showed that with increasing nanoclay amount, the fiber pores were partially filled by nanoclay.
8. The XRD test results showed that the peak pertaining to the clay crystalline was not lost and only moved forward and the  $2\theta$  got larger; therefore, this data suggested that the created morphological structures of interlayer type.

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