

Effects of Montmorillonite Nanoclay on the Properties of Chemimechanical Pulping Paper

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The effects of nanoclay were studied relative to the physical, mechanical, optical, and morphological properties of chemimechanical pulping papers. Nanoclay was incorporated at 0%, 2%, 4%, 6%, 8%, or 10%. To increase the retention, 1% cationic starch was used in all test papers. Handsheets (60 g/m² in weight) were tested to determine their physical, mechanical, optical, and morphological properties. Up to 2% nanoclay increased the tensile strength; at values greater than 2%, the tensile strength decreased. The addition of up to 4% nanoclay increased roughness; between 4% and 10% nanoclay, roughness decreased. With 10% nanoclay, the tear strength, burst strength, and brightness decreased, but the air resistance, opacity, and yellowness increased. Scanning electron microscopy showed that the nanoclay filled the pore spaces between fibers, thus increasing air resistance. X-ray diffraction patterns indicated an intercalated structure.

Keywords: CMP; Nanoclay; Mechanical properties; Optical properties; SEM

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INTRODUCTION

Paper and paper products are prominent in human life and have many uses. New paper products are being marketed every day, necessitating improved product quality, new product development, and the use of new technology and materials. The chemimechanical pulping process has been used since 1970 to produce newspaper pulp. Chemimechanical pulp (CMP) has substitutability with sulfite pulp, without any negative effect on the quality and passing ability of paper from the printing machine. There are various types of mineral fillers, such as titanium dioxide, talc, clay fillers, and calcium carbonate (in both types of deposited and ground). Paper additives can improve the paper's appearance, opacity, brightness, whiteness, printability, and roughness, and they are helpful in saving other material costs. The addition of fillers also can cause some problems, such as the reduction of paper strength and increases in abrasiveness (Afra 2005).

Chemical-mechanical pulp can be produced by a variety of chemicals and mechanical treatments. Different types of chemicals are used during the chemical treatment phase. Sodium sulfite and sodium hydroxide are two of the most important chemicals used in chemical-mechanical pulping. In chemical-mechanical pulping, one main stage of chemical treatment and one stage of mechanical treatment, such as refining with disk refiners, are used for fiber separation. The yield of these pulps is 80% to 95%. The properties of these pulp are between those of high-yield chemical pulps and mechanical pulp. By filling pores in paper, fillers have the ability to increase the air resistance of the paper (Nazeri 2007). In addition, these materials increase the opacity of the paper (Hagemeyer 1997). However, as widely noted, the addition of fillers reduces the overall

strength properties of the paper (Afra *et al.* 2013). Nanotechnology is an emerging technology that has affected various areas of human life in recent decades. Nanotechnology improvements in the domains of fiber science, minerals, and different added substances will give papermakers the way to place request and construction into the plans of a sheet (Mohieldin *et al.* 2011). Accordingly, using nanotechnology, the paper industry has developed as a leading industry in the last two decades. Today, among various nanoscale mineral additives, nanoparticles (*e.g.*, montmorillonite (MMT) or "bentonite") are common because these particles can produce different structures (intercalated, intercalated-flocculated, exfoliated). The given structure will affect the product characteristics (Tjong 2006). Clay particles, for example MMT, is likewise preferred in paper filler and coatings to further improve barrier resistance (Samyn *et al.* 2018). The particles are very small (less than 2 micrometers) and very thin (approximately 1 nanometer). The clay has a layered structure, and its internal surfaces are inaccessible. Greater specific surface area, smaller dimensions, greater light scattering, and improved optical properties can be obtained if the layer structure is separated. It is predicted that clay particles can increase the air resistance of microfibers. Mokhtari *et al.* (2013) considered the use of a cationic starch-nano-bentonite system to improve the strength specifications of old corrugated container (OCC) papers. Their results showed that adding nano-bentonite decreased the tensile strength and burst strength indices and increased air resistance and paper ash content. Maloney *et al.* (2005) suggested that higher consumption of fillers improves the optical and volumetric properties of paper. Afra *et al.* (2013) compared the physical, mechanical, and optical properties of papers that were coated by nanoclay and homogeneous nanoclay. Different types of natural or synthetic nanoclays, such as MMT, have been investigated to reinforce the starch matrix for coatings and films (Kiaei *et al.* 2016). They found that using homogeneous nanoclay led to higher density and air resistance compared with raw nanoclay along with retention materials, while burst strength and tear strength were reduced slightly. Eventually, higher durability of the homogenized nanoclay, in comparison with the raw nanoclay, indicated a lower contamination load of water wastes and cost effectiveness of the system. Therefore, the purpose of this study was to consider the effects of nanoclay on the physical, mechanical, optical, and morphological properties of CMP papers.

EXPERIMENTAL

Materials

CMP

Chemimechanical pulp was prepared from the CMP storage tower of the Mazandaran Wood and Paper Company (Sari, Mazandaran, Iran) and was transferred to the laboratory. The CMP was refined to 300 mL of Canadian standard freeness (CSF) using a laboratory PFI mill (Model 277, Labtech Instruments Inc., Laval, Canada) according to the TAPPI T 248 sp-00 (2000) standard.

Cationic starch

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% dosage based on the dry pulp weight. The cationic starch had a pH of 6, a DS of approximately 0.035 mol/mol, a protein level of 1.5%, N of 0.25%, and a moisture content of 11% based on wet weight. To prepare

a starch solution with a concentration of 0.005 g/cm³ (*i.e.*, 0.5 g of pure starch per 100 cm³ of starch and water solution), the required gross amount of starch was determined by considering moisture percentage. The determined gross amount was poured into an Erlenmeyer flask, and its volume was increased to 100 cm³ by adding distilled water. During mixing, the temperature inside the Erlenmeyer flask was monitored with a thermometer, and foil was put on top of the flask as a lid to prevent evaporation. The flask was put on a heater for 30 min, and the temperature was increased to 90 °C slowly, remaining at 90 °C for 30 min. The resulting starch lotion was produced each day to prevent viscosity changes and other concentration changes due to environmental effects. The cationic starch properties are presented in Table 1.

Table 1. Cationic Starch Properties

Property	Value
Moisture	14%
Gelation temperature	70 °C
Cooking temperature	90 °C
pH	6
Viscosity	75.7 cP
Degree of substitution (DS)	0.035

Nanoclay

The nanoclay (K10) was obtained from Sigma-Aldrich (St. Louis, MO, USA) and was used at 6 levels: 0%, 2%, 4%, 6%, 8%, and 10%, proportional to the paper pulp dry weight. The 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
Product Number	281522
CAS Number	1318-93-0
Surface Area	220-270 m ² /g
Base	Sodium Montmorillonite
Density	300 kg/m ³ to 370 kg/m ³
Anion	Chloride
Modifier Concentration	48 meq/100 g
Moisture	1% to 2%
Weight Loss on Ignition	30%

*As reported by the manufacturer

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 nanoclay were added to the mixture of the CMP pulp. In total, eight handsheets were made for each treatment according to the TAPPI standard T 205 sp-02 (2002). The different ratios of CMP, nanoclay, and cationic starch used for handsheet making are shown in Table 3.

Table 3. Combinations of Different Ratios of CMP Hardwood Pulp, Nanoclay, and Cationic Starch for Newsprint

Sample Code	CMP Hardwood Pulps (%)	Nanoclay (%)	Cationic Starch (%)
A	99	0	1
B	97	2	1
C	95	4	1
D	93	6	1
E	91	8	1
F	89	10	1

Methods

Determining CMP specifications

Mechanical properties (tensile strength, burst strength, and tear strength indices), roughness, and optical properties (brightness, opacity, and yellowness) were tested on handsheets according to TAPPI T 494 om-01 (2001), TAPPI T 403 om-02 (2002), TAPPI T 414 om-04 (2004), TAPPI T 538 om-01 (2001), and TAPPI T 452 om-98 (1998), respectively.

X-ray diffraction test

The X-ray diffractometer (X'Pert model) that was used was manufactured by Philips (Amsterdam, The Netherlands). Radiation was emitted from a cobalt lamp with a wavelength of 1.79 Å, a step of 0.02°, a speed of 0.3°/sec, and an emission angle 2θ in the range of 1° to 9°. The samples were prepared as plate forms with dimensions of 1 mm × 1 mm × 10 mm.

Scanning electron microscopy

To observe the presence of nanoclay on the sheet surface and the fibers, a scanning electron microscope (AIS 2100, Seron Technologies, Uiwang, South Korea) was used.

Statistical analysis

Data analysis was performed using SPSS statistical software (Version 11.5, IBM, Armonk, NY, USA) with a complete randomized factorial test. Mean comparisons and classification were performed using Duncan's test at a 95% significance level. The relations between nanoclay percentage and the mechanical specifications of the CMP paper were determined by linear regression.

RESULTS AND DISCUSSION

In this study, nanoclay was used at six levels (0, 2, 4, 6, 8, and 10 percentage). The F-values and p-values are shown in Table 4. The independent effects of nanoclay on air resistance, roughness, tensile strength index, burst strength index, brightness, opacity, and yellowness were significant at the 95% significance level, while effect on the tear strength index was not significant. Paper strength is an important property, because most papers are used under stressful conditions. There are various kinds of strength tests for papers, but the most important tests are for burst strength, tensile strength, and tear strength. None of these properties are fundamental, but the combinations of them, such as flexibility, the strength

of inter-fiber joints, and inherent fiber strength, are fundamental. All the paper strengths depend on fiber type, length, thickness, flexibility, network, number of joints, paper weight, and density (Afra 2005). Strength properties of CMP paper rely on the types of primary materials and the production process.

Table 4. Variance Analysis (F-values and significance levels) of the Effects of Nanoclay on Paper Properties

Nanoclay (%)	Tensile Strength Index (N.mg ⁻¹)	Tear Strength Index (m.N.m ² .g ⁻¹)	Burst Strength Index (KPa.m ² .g ⁻¹)	Roughness (μm)	Air Resistance (s)	Opacity (%)	Brightness (%)	Yellowness (%)
F-Value	34.217*	1.152 ^{ns}	7.067*	6.817*	49.406*	76.406*	588.293*	448.138*
0	34.59 ^d (0.31)	4.92 (0.56)	1.43 ^d (0.04)	8.39 ^{bc} (0.11)	6.61 ^a (0.11)	85.24 ^a (0.28)	63.74 ^f (0.17)	13.46 ^a (0.19)
2	34.93 ^d (0.59)	4.89 (0.25)	1.35 ^{cd} (0.02)	8.08 ^b (0.43)	7.13 ^b (0.09)	86.57 ^b (0.65)	62.32 ^e (0.30)	14.87 ^b (0.16)
4	33.15 ^c (0.25)	4.82 (0.45)	1.27 ^{abc} (0.07)	8.75 ^c (0.14)	7.44 ^{bc} (0.42)	87.54 ^c (0.49)	60.81 ^d (0.22)	15.74 ^c (0.07)
6	31.73 ^b (1.01)	4.53 (0.19)	1.29 ^{bc} (0.04)	7.96 ^b (0.59)	7.79 ^c (0.08)	89.55 ^d (0.12)	59.10 ^c (0.21)	17.35 ^d (0.21)
8	31.85 ^b (0.17)	4.54 (0.35)	1.18 ^{ab} (0.04)	7.39 ^a (0.11)	8.29 ^d (0.15)	89.05 ^d (0.41)	57.82 ^b (0.17)	18.28 ^e (0.35)
10	30.12 ^a (0.48)	4.41 (0.03)	1.15 ^a (0.14)	7.85 ^{ab} (0.06)	8.92 ^e (0.16)	90.90 ^e (0.33)	55.08 ^a (0.25)	20.28 ^f (0.11)
R ²	0.913	0.917	0.933	0.41	0.986	0.939	0.986	0.989
*95% significance level; ns no significance (small letters indicate the Duncan ranking of the averages at a 95% confidence interval.) Values in parentheses are standard deviation. R ² : coefficient of determination								

Tensile Strength Index

The tensile strength index changes for handsheets made from CMP at various nanoclay levels are shown in Table 4. The highest average tensile strength index was observed in the sample with 2% nanoclay, with a value of 34.93 Nm/g, and the lowest tensile strength index was observed at 10% nanoclay, with a value of 30.12 N·m/g. Moreover, there was a negative correlation between the percentage of nanoclay and the paper tensile strength ($R^2 = 0.913$). The tensile strength was an indicator of the paper's potential durability under tensile stress. The most important factors affecting paper tensile strength are the quality and number of fiber-to-fiber bonds. Increasing the fiber-to-fiber bonding by increased refining or wet pressing increases the paper tensile strength. However, paper tensile strength is always less than that of the fiber (Afra 2005). The addition of up to 2% nanoclay increased the tensile strength slightly, but adding more nanoclay led to a distribution of flocs and creation of coarser flocs. This phenomenon was attributed to the location of fillers among fibers and the reduction of bonds between fibers, because common fillers cannot create strong bonds with cellulose fibers.

Tear Strength Index

Tear strength changes in the CMP handsheets made with nanoclay are shown in Table 4. The highest average tear strength index was observed in the control sample ($4.92 \text{ mN}\cdot\text{m}^2\text{g}^{-1}$), and the lowest was observed with 10% nanoclay ($4.41 \text{ mN}\cdot\text{m}^2\text{g}^{-1}$). Moreover, there was a negative correlation between the percentage of nanoclay and the paper tear strength ($R^2 = 0.917$). One parameter affecting tear strength was the fiber length. The tear strength is proportional to the average fiber length to the power of three halves. The nanoclay particles were very small. Increased nanoclay additions led to a distribution of floc, creation of coarser floc, and reduction of tear strength. However, this reduction was not significant because tear strength depends on inter- and intra-fiber strengths. Because nanoparticles are placed between fibers, which weakens the fiber bond, the strength between the fibers decreases, but the strength of the individual fiber does not change. As a result, the tear index decline is negligible, which is similar to the results obtained by Lindström *et al.* (2008).

Burst Strength Index

Burst strength changes in the CMP handsheets with the addition of different levels of nanoclay are shown in Table 4. The highest average burst strength index was observed in the control sample with 0% nanoclay, with a value of $1.43 \text{ kPa}\cdot\text{m}^2/\text{g}$, and the lowest was observed in the sample with 10% nanoclay, with a value of $1.15 \text{ kPa}\cdot\text{m}^2/\text{g}$. Moreover, there was a negative correlation between the percentage of nanoclay and the paper burst strength ($R^2 = 0.933$). Burst strength depends on fiber length and the fiber bonding, but it is affected more by the connections between the fibers (Asadpour *et al.* 2008; Akbarpour and Resalati 2011). Nanoclay particles, as mineral fillers, weaken inter-fiber bonds. These findings were confirmed the results of Afra *et al.* (2013). In addition, previous studies have shown that adding fillers reduces strength, and clay soil, talc, and calcium carbonate are more effective in reducing mechanical strength. Moreover, the addition of nanoclay distributed more floc and created coarser floc, so it can reduce the strength.

Roughness

Changes in roughness resulting from the addition of nanoclay are shown in Table 4. The highest average roughness was observed in the sample with 4% nanoclay ($8.75 \mu\text{m}$), and the lowest was observed at 8% nanoclay ($7.39 \mu\text{m}$). There was a negative correlation between the percentage of nanoclay and paper roughness ($R^2 = 0.41$).

Air Resistance

The air resistances of the CMP handsheets made with nanoclay are shown in Table 4. The lowest average air resistance was observed in the control sample with 0% nanoclay (6.61 s), and the highest was observed at 10% nanoclay (8.92 s). There was a positive correlation between the percentage of nanoclay and the paper air resistance ($R^2 = 0.986$). Silicate plates of nanoclay are impermeable to air, and they make the air resistance pathways long and sinuous. This feature is considered superior for printing and writing papers. Air resistance increases by enhancing fillers and fines because the fiber pores are filled with the fillers (Nazeri 2007). In addition, better dispersion of filler particles improves air resistance (Hubbe 2004). Increasing the nanoclay level improved the durability of the fibers and additives in the CMP because the fiber pores were filled by cationic starch and fines.

Opacity

The opacities of the CMP handsheets made with different levels of nanoclay are shown in Table 4. The lowest average opacity was observed in the control sample with 0% nanoclay (85.2%), and the highest was observed at 10% nanoclay (90.9%). There was a positive correlation between the percentage of nanoclay and paper opacity ($R^2 = 0.939$). Paper opacity is a property of paper that prevents light from passing through the paper. Base weight, absorption coefficient and light scattering coefficient of paper are the factors affecting the opacity of the paper and the degree of opacity is directly related to these three factors. According to the results, with the addition of nanoclay, a significant difference has occurred in the amount of paper opacity. These results were similar to those of Hagemeyer (1997). When light mineral particles refract light, greater opacity and opaque fillers are observed (Nazeri 2007). Filler particles increase light scattering due to their empty spaces and affect the opacity (Hubbe 2004). The addition of fillers improves paper opacity (Song *et al.* 2009).

Brightness

Brightness changes in the CMP papers with the addition of different levels of nanoclay are shown in Table 4. The lowest average brightness was observed in the control sample with 0% nanoclay, with a value of 63.74%, and the highest was observed at 10% nanoclay, with a value of 55.08%. Moreover, there was a negative correlation between the percentage of nanoclay and the paper brightness ($R^2 = 0.986$). Increasing the filler percentage decreased the paper's primary brightness.

Yellowness

Yellowness changes in the CMP handsheets with the addition of different levels of nanoclay are shown in Table 4. The lowest average yellowness was observed in the control sample with 0% nanoclay (13.46%), and the highest was observed at 10% nanoclay (20.28%). There was a positive correlation between the percentage of nanoclay and paper yellowness ($R^2 = 0.989$).

Morphology

Scanning electron microscopy of the paper structure

Figure 1 shows fibers in the handsheets with 0%, 2%, 4%, 6%, 8%, and 10% nanoclay. Increasing the nanoclay level up to 10% substantially covered the surfaces of the fibers; the paper surface was fully filled, with very few visible pores. Thus, air resistance increased compared with the control sample. In addition, low consumption of nanoclay increased strengths, but with a further increase in nano-clay due to excessive negative ionic charge, the formation of coarse floc, which causes a decrease in resistance.

Structural study by X-ray diffraction

The X-ray diffraction spectra of the handsheets with nanoclay at different addition levels were obtained at angles of 2° to 11° (Fig. 2). The whole morphological structure was of interlayer type. As the nanoclay values ranged from neat nanoclay (100 wt%), 2 wt% to 10 wt%, the peak angle at 2θ first decreased and then increased. As shown in Fig. 2, the X-ray diffraction peak of the neat nanoclay was created at an angle of $2\theta = 7.03^\circ$, with a distance between layers (d) of 14.58 Å.

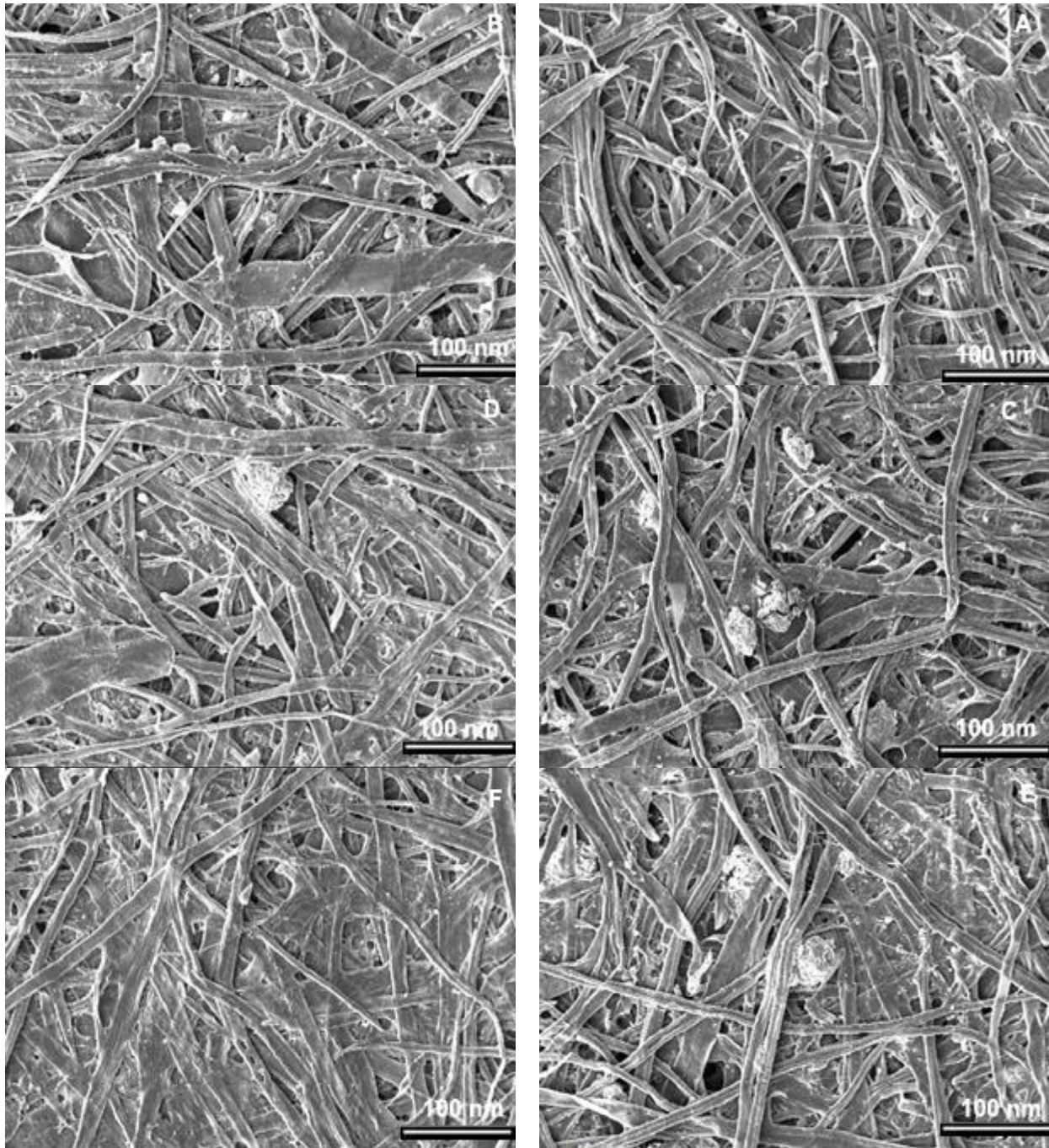


Fig. 1. CMP paper surfaces: (a) 0%, (b) 2%, (c) 4%, (d) 6%, (e) 8%, and (f) 10% of nanoclay

Increasing the nanoclay to 2 wt%, the X-ray diffraction peak shifted to a smaller angle ($2\theta = 6.89^\circ$). At this angle, the distance between the layers was $d = 14.89 \text{ \AA}$. As the nanoclay increased to 4 wt%, the X-ray peak moved forward again and the distance between the silicate layers increased ($2\theta = 7.11^\circ$ and $d = 14.43 \text{ \AA}$). Increasing nanoclay to 6 wt% changed the X-ray diffraction peak ($2\theta = 7.28^\circ$), and at this angle, the inter-layer distance was $d = 14.09 \text{ \AA}$. Increasing the nanoclay up to 8 wt% moved the X-ray peak moved forward, and the distance between silicate layers decreased ($2\theta = 7.53^\circ$ and $d = 13.96 \text{ \AA}$). By increasing the amount of nanoclay to 10 wt%, the 2θ peak angle increased to

7.48°, and the distance between layers was 13.71 Å. As the peak angle 2θ first decreases and then increases, the distance between the clay plates (according to the Bragg's equation) increases and then decreases when using 2 wt% nanoclay. Therefore, this information shows that the morphological structures formed have an interlayer morphological structure.

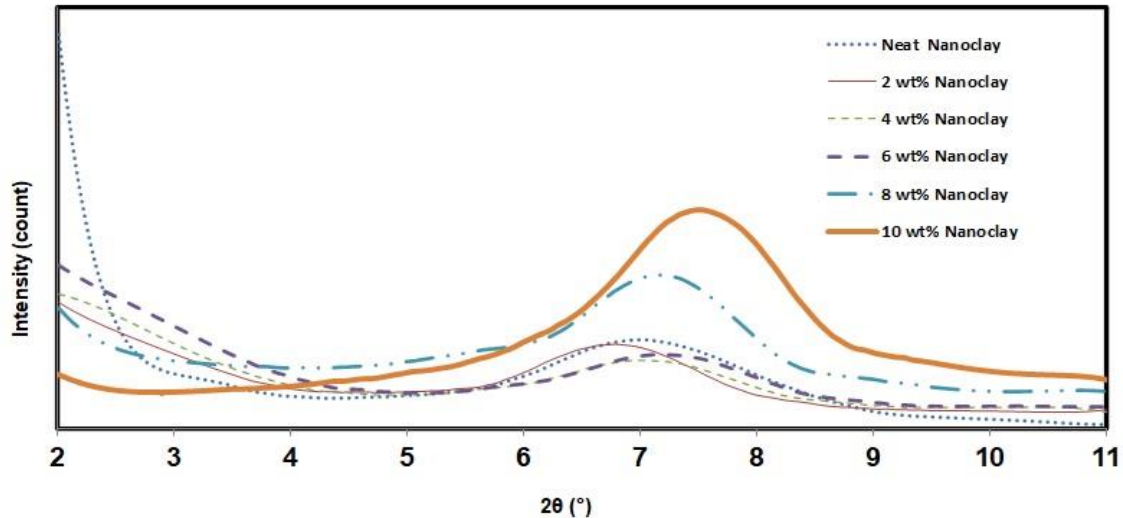


Fig. 2. X-ray spectra neat nanoclay (100 wt%), 2 wt%, 4 wt%, 6 wt%, 8 wt%, and 10 wt% of nanoclay

CONCLUSIONS

1. At first, some of the properties, such as tensile strength, roughness, and air resistance, increased with the increasing addition of montmorillonite. However, further increasing nanoclay levels increased floc distribution, created coarse floc, and ultimately reduced the strength.
2. Because the nanoclay particles were very small, they reduced the tensile strength, tear strength, burst strength, and roughness. They also decreased brightness.
3. Increasing nanoclay levels increased the air resistance, opacity, and yellowness.
4. Increasing the nanoclay up to 10% increased the air resistance, opacity, and yellowness by 34.9%, 6.6%, and 50.7%, respectively.
5. Increasing nanoclay up to 10% decreased the tensile strength, tear strength, burst strength, roughness, and brightness by 14.8%, 11.6%, 24.3%, 6.9%, and 15.7%, respectively.

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