

# Thermal and Mechanical Properties of Polypropylene Nanocomposite Materials Reinforced with Cellulose Nano Whiskers

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**ABSTRACT:** We use polypropylene (PP, an apolar polymer) and cellulose nano whiskers (CNW, a polar material) to produce nano polymer composites with enhanced mechanical and thermal properties. To improve compatibility, maleic anhydride grafted PP has also been used as a coupling agent. To enhance the uniform distribution of CNW in the composite, the matrix polymer is dissolved in toluene, and sonification and magnetic stirring are applied. Good film transparency indicates uniform CNW dispersion, but CNW domains in the composite film observed under an scanning electron microscope may indicate slight agglomeration of CNW in the composite film. The tensile

strength of the composite compared with neat PP improves by 70–80% with the addition of CNW. The crystallinity has also been improved by about 50% in the CNW reinforced samples. As the content of CNW increases, the composite exhibits higher thermal degradation temperature, higher hydrophilicity, and higher thermal conductivity. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 2882–2889, 2012

**Key words:** polypropylene; cellulose nano whiskers; maleic anhydride; nano composite; mechanical properties

## INTRODUCTION

Developing new polymer composites and hence applications of such materials have been an active field of research in recent years. When comparing with composite materials containing micron or larger sized reinforcing materials, nano-sized reinforcements may provide better physical and mechanical properties, and thus, less material is needed for a given application at low cost. In addition to the plastics industry, nanocomposite polymeric materials can also be used as technical textiles in aerospace, automotive, building, defense, and other industries. They can be used directly or indirectly as load-bearing structural materials.

Nano materials such as silica, clay, carbon black, and carbon nanotube have been widely studied and

used to make nano polymer composite materials. Although some studies on cellulose nano whiskers (CNW) have been carried out in the last two decades, detailed investigations on the use of CNW in composite materials are quite recent. Cellulose, one of the most abundant natural polymers, has many advantages: renewability, biodegradability, nontoxicity, high specific strength and stiffness, low cost, good thermal stability, rigidity, high  $L/D$  (length/ diameter) ratio, among others. However, there are several obstacles for the use of CNW, that is, lack of commercial availability, low yield production, agglomeration in processing, and difficulty of using it in systems which are not water based and polar, because of its strong hydrogen bonding and its polarity.

Efforts have been made to disperse CNW in apolar solvents and to incorporate it into new polymeric matrix, such as by chemical modification of cellulose,<sup>1</sup> use of surfactants,<sup>2</sup> etc. However, there are some limitations for these applications. For example, it is necessary to use a large amount of surfactant, and chemical modification of cellulose often lowers the mechanical performance of the composites.<sup>3</sup> There are several studies involving transferring whiskers from water to other solvent, which is

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suitable for polymer matrix.<sup>4</sup> Favier et al.<sup>5</sup> mixed cellulose whiskers with polymer lattices resulting from the copolymerization of styrene, butylacrylate, and a small amount of acrylic acid. They examined the effect of CNW (from 1 to 14 wt %) on the mechanical properties of polymer matrix. It was pointed out that CNW improved the mechanical properties of nanocomposite films, which were heated above the glass transition temperature of the polymer. Petersson et al.<sup>4</sup> used poly(lactic acid) (PLA) as a polymer matrix and CNW as a nano reinforcement filler. The nanocomposites were prepared by solution casting using chloroform as a solvent. They pointed out that there was no degradation in composites within the temperature range where PLA was processed (25–220°C). Schroers et al.<sup>6</sup> tried to improve the mechanical properties of LiClO<sub>4</sub>-doped ethylene oxide-epichlorohydrin with cellulose whiskers with a dosage rate of 1–15 wt %. They pointed out that the Young's module of the nanocomposites was increased together with a small reduction in the electrical conductivities. Samir et al.<sup>3</sup> prepared nano composites with stable suspension of CNW in *N,N*-dimethylformamide (DMF). They found that the wettability associated with a high dielectric constant of DMF is sufficient to disperse CNW in DMF. They pointed out that CNW did not affect the thermal properties of the matrix. Marcovich et al.<sup>7</sup> used CNW to reinforce polyurethane. They showed that the tan  $\delta$  peak was shifted toward higher temperature, indicating an increase in  $T_g$  by  $\sim 7$ –8°C. Dufresne et al.<sup>8</sup> used an elastomeric medium-chain-length poly(hydroxyalkanoate) latex as a polymer matrix and cellulose whiskers as reinforcing filler. They pointed out that the effect of CNW on the glass transition temperature ( $T_g$ ) of the polymer composites was not strong. However, above  $T_g$ , a greater increase in composite modulus was observed with an increase of CNW. Goetz et al.<sup>9</sup> produced a cellulose nanocomposites, wherein the CNW were crosslinked with poly( $\alpha$ -methyl vinyl ether-*co*-maleic acid) and poly(ethylene glycol). The mass content of cellulose whiskers in the composite films included 0, 25, 50, 75, and 100 wt %. It was shown that, at equilibrium, the 25 wt % CNW film absorbed higher amount of water compared with 50 wt % CNW film and 75 wt % CNW film. They also concluded that the nanocomposite system could swell in water to form a stable gel.

Ljungberg et al.<sup>10</sup> produced nanocomposites made of an apolar matrix and CNW. Atactic polypropylene (PP) was used as the apolar polymer matrix, whereas different treatments were applied to CNW, such as CNW treated with surfactant, CNW treated with maleic anhydride (MA) grafted PP (MAPP), and CNW without surface modification. It was shown that all the CNW types studied resulted in improved mechanical properties for both tensile testing and

dynamic mechanical analysis (DMA) testing, compared with neat PP. In another study, Ljungberg et al.<sup>11</sup> used isotactic PP as apolar polymer matrix and three different CNW such as CNW treated with surfactant, CNW treated with MAPP, and CNW without surface modification. They concluded that at large deformation, the mechanical properties depended strongly on the dispersion quality of the whiskers.

From the survey, only a very limited number of studies were reported in the literature on PP reinforced with CNW.<sup>5,10,11</sup> PP is an apolar polymer and the solvent used to dissolve this polymer is also apolar. Studies to bring this polymer and CNW, which is polar, together are very limited. PPs have wide applications in the plastics, electric/electronic, automotive, and technical textiles industries. Examples include filler fibers in concrete, filters, automotive parts (nonwoven fabric used in kick panels, seat construction, cabin air filters, etc.), carpeting, ropes, automotive battery cases, fuel lines, packaging, bottles, pipes, tanks, etc. Thus, improvement of thermal stability and other mechanical properties of this polymer are very important.

In this study, apolar PP, together with polar CNW has been studied using apolar solvent toluene. Most studies on CNW composites focus on thermal and mechanical properties such as strength and elastic modulus at various temperatures. In this study, the scope of investigation is expanded to include hydrophilicity (contact angle), thermal conductivity, and other properties. In addition, the effects of MA concentration, acid hydrolysis time, and ultrasonic treatment time on the tensile strength of composites are also examined.

## EXPERIMENTAL

### Materials

The matrix polymer used was PP (from Capilen, 25 MFI). MAPP (from Westlake Chemical Corp., Epolen E 43-Wax, softening point 158°C, 8 wt % MA, acid No:45, molecular weight  $M_w$ : 15,789;  $M_n$ : 7105) was used to improve the adhesion between CNW and PP. Toluene was used to dissolve PP and MAPP. Microcrystalline cellulose (MCC) (Avicel type GP1030, from FMC Biopolymer) was used to produce CNW to be used as reinforcement. Sulfuric acid 95–97% and sodium hydroxide were also used during the production of CNW.

### Preparation of CNW

CNW were obtained by acid hydrolysis of MCC (from FMC Biopolymer). The method was similar to that used by Marcovich et al.<sup>7</sup> MCC (5 gr) was hydrolyzed in 43,75 mL of sulfuric acid at 45°C for 130 min. After this process, 170 mL of deionized

**TABLE I**  
**Samples Used for Experimental Studies**

Sample (Code)	Maleic anhydride (wt % in PP)	CNW content (wt % in matrix)	Hydrolyzing time (min)	Sonification time (min)
PP reference	0	0	–	–
08 ma reference	0.8	0	–	–
08 ma 5 cnw	0.8	5	130	120
08 ma 10 cnw	0.8	10	130	120
08 ma 15 cnw	0.8	15	130	120
08 ma 15 cnw 30 son	0.8	15	130	30
08 ma 15 cnw 60 hyd	0.8	15	60	120
2 ma reference	2.0	0	–	–
2 ma 5 cnw	2.0	5	130	120
2 ma 10 cnw	2.0	10	130	120
2 ma 15 cnw	2.0	15	130	120

water was added and the solution was kept for between 17 and 24 h. The sulfuric acid was removed by repeated cycles of centrifugation (9 cycles), each cycle for 40 min at 8000 rpm (Nüve NF 800 R centrifuge). The supernatant was removed from the sediment and was replaced by deionized water. The centrifugation continued until the supernatant became turbid. To decrease acidity of the solution, NaOH was added into the solution in the 8th cycle. The final suspension had a pH value of 9.

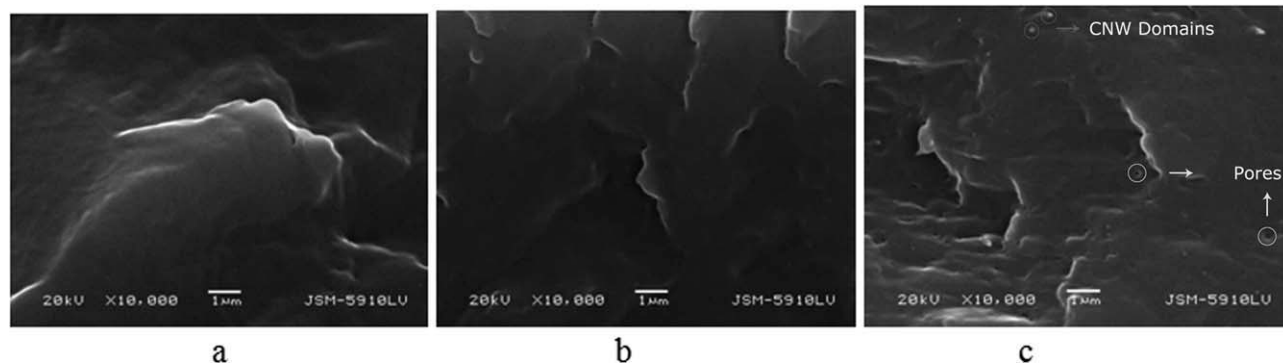
#### Preparation of composite polymer films

MAPP was added to PP such that the weight ratio of MA was 0.8 wt % in the polymer matrix. For the tensile breaking strength test, samples with 2 wt % MA were also produced. MAPP and PP were dissolved in toluene separately at around 105–110°C. The CNW loading varied from 0 to 15 wt %. CNW was added into the dissolved MAPP, and then they were exposed to sonification (Bandelin Sonoplus, ultrasonic homogenizator) for 2 h to improve the dispersion of the CNW in MAPP. MAPP solution with CNW was added into the dissolved PP. Magnetic stirring (Wise Tis HG 15d) was applied for 15 min (1500 rpm, 100°C) to allow some evaporation of Toluene while mixing PP and MAPP with CNW. Then, the solution was cast in Petri dishes and left for

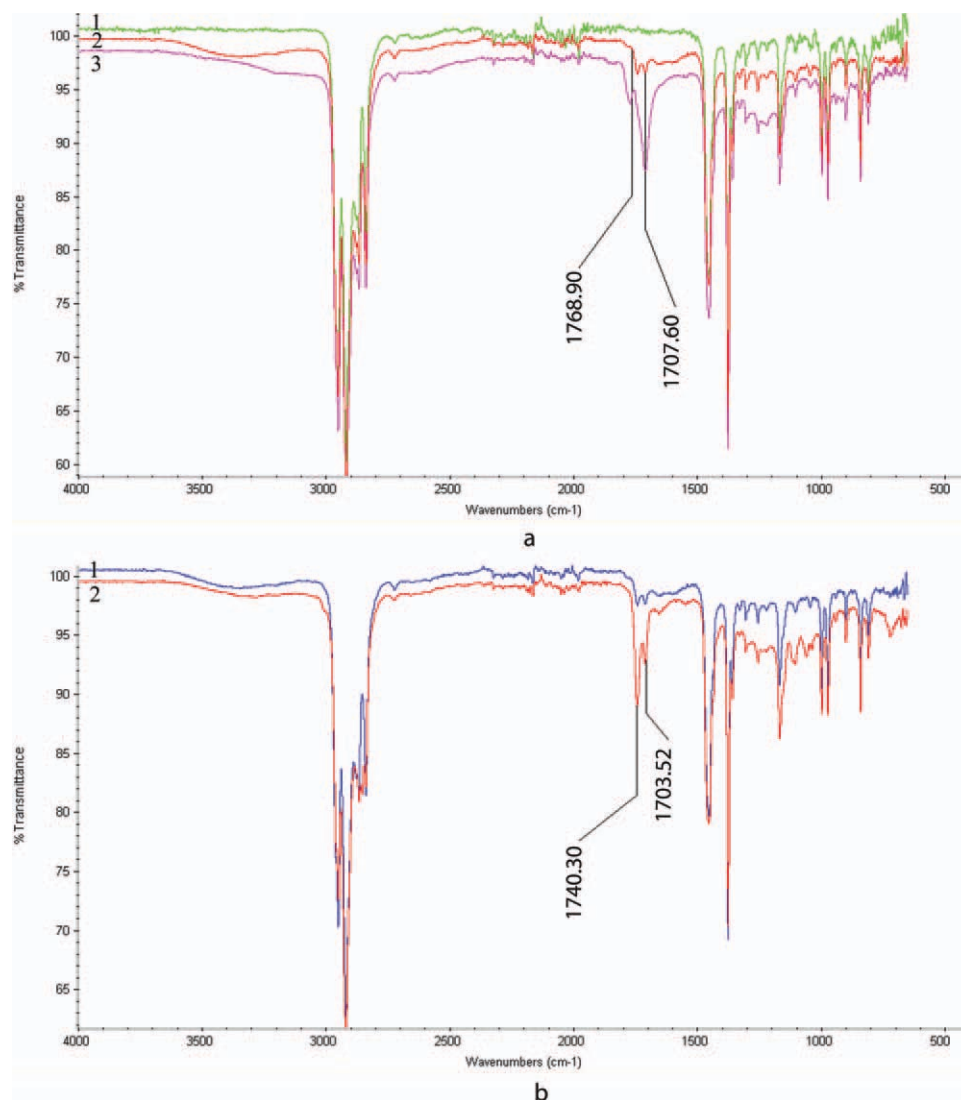
evaporation in a drying oven at 90°C for 1 day. The resulting composite materials were placed between two Teflon sheets and pressed at 135°C for 2 min with a pressure of 60 bars (6.08 MPa). 100% PP samples without MAPP and CNW were called pure polymer matrix [PP reference]. MAPP was added into the pure polymer matrix (PP) in such a way that the final polymer matrix (MAPP and PP) contained 0.8 wt % MA. Some samples were produced without CNW to evaluate the effect of MAPP on the properties of polymer matrix composed of MAPP and PP. These samples were referred to as [ma-reference]. Composite samples were fabricated with 5, 10, and 15 wt % CNW. For the tensile strength test, additional samples were produced which contained 2 wt % MA. Samples were also produced for the tensile strength test with different hydrolyzing times (60 and 130 min) and different sonification time (30 and 120 min). Test samples and their corresponding codes are summarized in Table I.

#### Appearance of samples

Surface appearance (transparency) of film was observed by naked eyes. After the tensile test, the surface of rupture was examined under a scanning electron microscope (SEM, JEOL Model JSM-5910LV, 20 kV accelerating voltage).



**Figure 1** SEM micrographs: (a) [PP reference], (b) [2 ma reference], and (c) [2 ma 15 cnw].



**Figure 2** FTIR spectra: (a) FTIR spectra of (1) [PP reference], (2) [08 ma reference], and (3) [2 ma reference], and (b) FTIR spectra of (1) [08 ma reference] and (2) [08 ma 15 cnw]. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

### Fourier transform infrared spectroscopy

Thermo Scientific Nicolet IS10 was used for the Fourier transform infrared spectroscopy (FTIR) analysis.

### Tensile testing

Experiments were done on an Instron 3345 Tensile tester with a 100 N load cell at a cross head speed of 10 mm/min. The length and width of the specimens were 35 mm and 5 mm, respectively. The gage length, distance between jaws, was 15 mm, and at least 10 specimens were tested for each sample.

### Differential scanning calorimetry

Differential scanning calorimetry (DSC, TA Q Series DSC Q10) was performed with a heating rate of 10°C/min, from  $-80^{\circ}\text{C}$  to  $250^{\circ}\text{C}$ .

### Thermogravimetric analysis

Thermogravimetric analysis (TGA, TA Q Series TGA Q50) was used to measure the temperatures of 5% and 50% mass loss and the residue amount. The samples were heated from  $30^{\circ}\text{C}$  up to  $800^{\circ}\text{C}$  with a heating rate of  $20^{\circ}\text{C}/\text{min}$ . Nitrogen flow rate was 90 mL/min.

### Dynamic mechanical analysis

Specimens with dimensions 40 mm by 10 mm was tested on a DMA (Perkin Elmer, Diamond DMA) at 1 Hz, with 0.05% strain magnitude and 20-mm gage length. The temperature was increased from  $-20^{\circ}\text{C}$  to  $175^{\circ}\text{C}$  at a rate of  $3^{\circ}\text{C}/\text{min}$ .

### Hydrophilicity (contact angle)

Attension theta optic contact angle test device was used. A droplet of 5  $\mu\text{L}$  of water was placed on the

**TABLE II**  
**Results of Tensile Strength Test**

Sample	Breaking strength (MPa)	Breaking strain (%)	Modulus (MPa)
PP reference	25.7	24.9	1258.5
08 ma reference	41.6	5.2	1534.0
08 ma 5 cnw	39.5	5.5	1498.2
08 ma 10 cnw	41.1	9.76	990.0
08 ma 15 cnw	44.4	7.38	1258.4
08 ma 15 cnw 30 son	40.9	5.61	1129.7
08 ma 15 cnw 60 hyd	46.6	7.4	1295.6
2 ma reference	33.5	2.17	1777.4
2 ma 5 cnw	32.8	4.3	1417.5
2 ma 10 cnw	36.4	5.4	1013.5
2 ma 15 cnw	39.0	4.8	1336.1

surface of the specimen with a syringe. Ten photographs were taken over a 1 min period. The results were the average of at least three measurements of each sample.

#### Thermal conductivity

Jenway 7700 Heat Conduction Apparatus was used. 2.2 W of heat flow was applied. Thermal conductivity ( $k$ ) was calculated according to Fourier law; that is,

$$Q = -kA\delta T/h \quad (1)$$

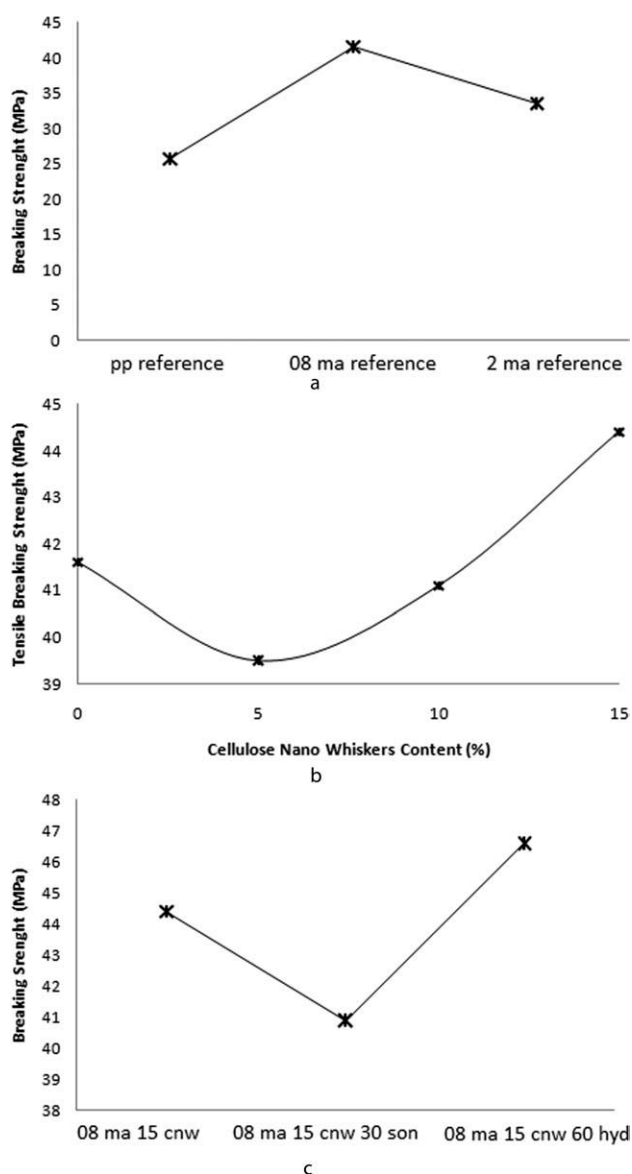
where  $Q$  is 2.2 W,  $A$  is  $4.9 \times 10^{-4} \text{ m}^2$  (area of film sample),  $\delta T$  is temperature difference, and  $h$  is thickness of film sample, respectively.

## RESULTS AND DISCUSSION

Surface appearance (transparency) of film was observed by naked eyes. It has been seen that all samples have good transparency with no marked opacity, indicating that there are no noticeable agglomeration. From SEM micrographs of the film samples (Fig. 1), some micropores (voids) and white dots representing CNW domains can be observed on the [2 ma 15 cnw] sample, which are generally smaller than 200 nm. According to literature reports,<sup>7</sup> the dimensions of most types of CNW are in the range of 5–20 nm in diameter and a few hundred nm to a few microns in length. Therefore slight agglomeration may have occurred, because the dimensions of some of the CNW domains and micropores (voids) are around 200 nm in dimensions.

From FTIR graphs [Fig. 2(a)], characteristics band of stretching of C=O in the zone of  $1700\text{--}1850 \text{ cm}^{-1}$  is observed from MA and characteristic band of stretching of —OH in the zone of  $3200\text{--}3550 \text{ cm}^{-1}$  is also observed from absorbed moisture by the MA groups.<sup>12,13</sup> These peaks confirm the presence of MA

in the structure. As seen from Figure 2(b), there are similar peaks in the zones of  $1700\text{--}1850 \text{ cm}^{-1}$  and  $3200\text{--}3550 \text{ cm}^{-1}$  for both samples [08 ma reference] and [08 ma 15 cnw], because MA and cellulose both exhibit the same band range. It is noticed that the peak intensity is higher in the zone of  $1700\text{--}1850 \text{ cm}^{-1}$ , possibly resulted from the interaction between CNW and the PP matrix containing MAPP. There are two possible interactions between CNW and the PP matrix. The first one involves the formation of covalent bonds between the anhydride groups and the OH groups of CNW to form ester and carboxylic acid groups. The second interaction is due to



**Figure 3** Tensile strength of the samples: (a) Tensile strength of [PP reference], [08 ma reference], and [2 ma reference]; (b) Tensile strength of [08 ma reference], [08 ma 5 cnw], [08 ma 10 cnw], and [08 ma 15 cnw]; and (c) Tensile strength of [08 ma 15 cnw], [08 ma 15 cnw 30 son], and [08 ma 15 cnw 60 hyd].

**TABLE III**  
Results of DSC Thermal Analysis

Sample	$T_m$ (°C)	$\Delta H_m$ (J/g)	$T_c$ (°C)	$\Delta H_c$ (J/g)	$X_c$ (%)
PP reference	162	106.7	115	115	51.05263
08 ma reference	161	93.5	113	94	44.73684
08 ma 5 cnw	161	98.3	114	96.7	49.50894
08 ma 10 cnw	160	98.9	114	95.9	52.57842
08 ma 15 cnw	161	135.7	114	120.8	76.38615

$T_m$ , melting temperature;  $\Delta H_c$ , heat of crystallinity;  $T_c$ , crystallinity temperature;  $X_c$  (%), crystallinity degree;  $\Delta H_m$ , heat of fusion.

hydrogen bonding between the OH groups of CNW and the formed ester and carboxylic acid groups as well as the unreacted MA groups in the matrix.

As seen from Table II and Figure 3(a), MA improves the elastic modulus and strength of the composites because of increased adhesion in the structure. However, there appears to be a critical amount of MA beyond which the strength of the composite decreases due to reduced molecular mobility in the structure, which leads to an increase in rigidity and fragility of the structure. Thus, when 0.8 wt % MA results in an increase in both rigidity and strength, 2 wt % MA results in an increase in rigidity but a decrease in strength.

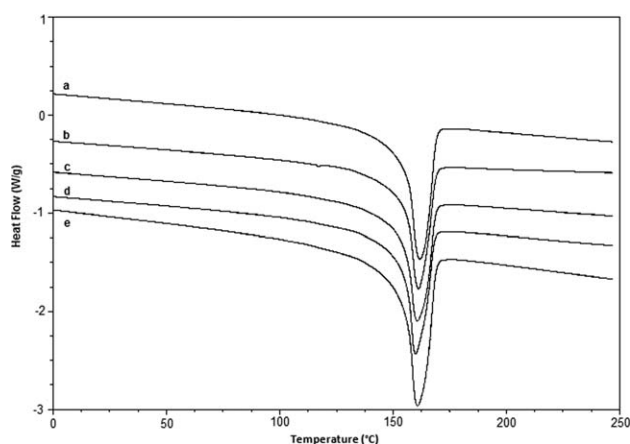
It is known that the effect of nano fillers on the mechanical behavior of composites is some different than that with fiber filler. Fiber fillers can carry the load resulting in an increase of the strength of composites; however, nano fillers, due to their large surface area, have more intense interaction with polymer matrix. Nano fillers interfere with polymer chain movement (blockage effect) that decreases the molecular mobility leading to an increase in the composite strength. Some nano fillers can also carry

the load and can transfer the stress away from the polymer matrix.<sup>14</sup>

As seen from Table II, Figure 3(b), the tensile breaking strength of the composite films increases with CNW content within each group of samples. When comparing the strength of [PP reference] (25.7 MPa) with those of [08 ma 15 cnw] (44.4 MPa) and [08 ma 15 cnw 60 hyd] (46.6 MPa), a 70–80% increase in strength is observed with 15 wt % CNW reinforcement. The contribution of CNW to the strength of composite material is more pronounced for the sample with 2 wt % MA than with the sample containing 0.8 wt % MA. This indicates that, within the range studied, an increase in MA content leads to better compatibility and adhesion between CNW and the polymer matrix.<sup>15</sup>

With respect to the effect of CNW content on tensile modulus and failure strain, the results suggest the existence of a critical amount for the nano filler in the composite. The breaking strain increases while the modulus decreases as the CNW content is increased up to 10%, beyond which the trends reverse. The likely causes of this phenomenon are the two competing effects when CNW filler is added, that is, increased micropores (voids) and the reinforcing mechanisms associated with reducing the molecular mobility of the matrix polymer. At lower CNW content, the effect of micropores is more pronounced whereas beyond the critical value, which appears to be around 10 wt %, the reinforcing effect dominates the deformation behavior.

It can also be seen that a shorter sonification time results in a decrease in the breaking strength



**Figure 4** DSC thermograms during melting process: (a) [PP reference], (b) [08 ma reference], (c) [08 ma 5 cnw], (d) [08 ma 10 cnw], and (e) [08 ma 15 cnw].

**TABLE IV**  
TGA Test Results Table

Sample	5% loss ( $T_{05}$ ) (°C)	50% loss ( $T_{50}$ ) (°C)	Residue (mg)
PP reference	396	453	1
08 ma reference	378	457	0
08 ma 5 cnw	385	465	0
08 ma 10 cnw	396	464	0
08 ma 15 cnw	409	471	1

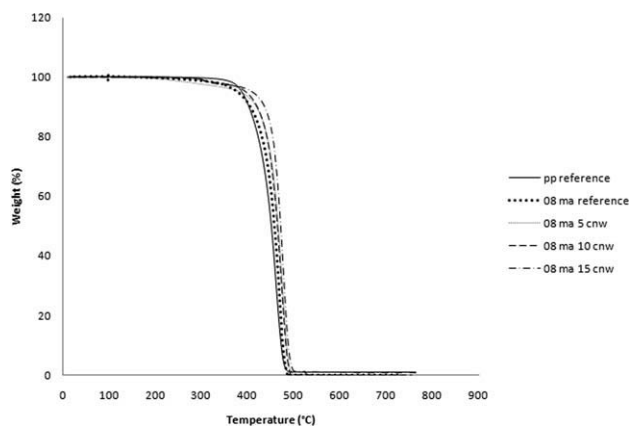


Figure 5 TGA test curves.

because of insufficient separation of CNW during sonification. However, a shorter hydrolyzing time results in slight increase in higher breaking strength of the composite film [Fig. 3(c)]. This may be resulted from the larger dimensions of CNW produced with shorter hydrolyzing times.<sup>16</sup>

As seen from Table III and Figure 4, an increase in CNW content results in an increase in the degree of crystallinity ( $X_c$ , %) of the polymer matrix, which is calculated from:

$$X_c = \Delta H_m / \Delta H_m^0 w \quad (2)$$

where  $\Delta H_m$  is the heat of fusion of the sample measured by DSC,  $\Delta H_m^0$  is the heat of fusion of perfect PP crystals (209 J/g<sup>17</sup>), and  $w$  is the weight fraction of PP.<sup>3</sup>

No distinctive changes in the melting and crystallinity temperatures are observed. [08 ma 15 cnw] exhibits higher crystallinity (by about 50%) and heat of fusion than the [PP reference] sample. The observed increase in crystallinity of the matrix polymer due to CNW filler is consistent with reported

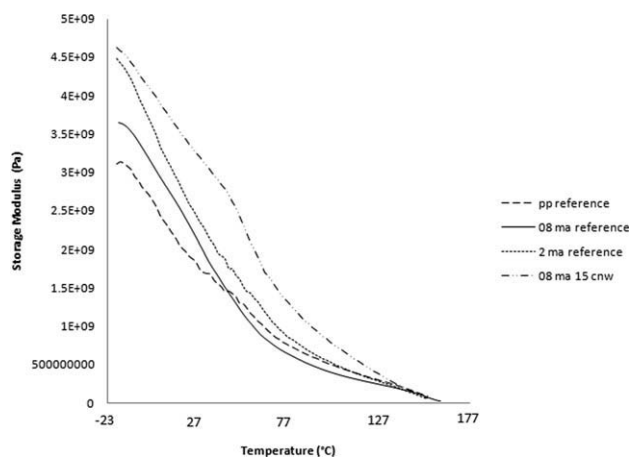


Figure 6 DMA test results.

TABLE V  
Contact Angle of Samples

Sample	Contact angle
PP reference	101.8
08 ma reference	99.4
08 ma 5 cnw	94.8
08 ma 10 cnw	90
08 ma 15 cnw	88.6

results in the literature.<sup>18,19</sup> A primary reason is that nano-sized fillers may act as a nucleating agent for the polymeric matrix to promote crystallization. Furthermore, nano fillers in the forms of polymer crystals may be more effective in promoting crystallization.<sup>20</sup> CNW as a nano-sized crystalline structure, therefore, is expected to act as an effective nucleating agent for the polymer matrix.

As seen from Table IV and Figure 5, an increase in CNW content leads to an increase in  $T_{05}$  which is the temperature at which the sample starts to decompose (5% weight loss). Thus, it can be said that an increase in CNW results in higher thermal resistance. The [08 ma 15 cnw] sample exhibits a higher  $T_{05}$  than [PP reference] by  $\sim 13^\circ\text{C}$ .  $T_{05}$  is around  $380\text{--}410^\circ\text{C}$  for all the samples.  $T_{50}$  is the temperature at which a sample loses 50% of its weight, and it is around  $450\text{--}470^\circ\text{C}$  for all the samples.

As seen from the DMA results (Fig. 6), the storage modulus of [08 ma 15 cnw] is higher than those of both [PP reference] and [08 ma reference]. The storage modulus increases with the content of CNW by more than 50% over a wide range of temperatures. An increase in storage modulus also corresponds to higher thermal stability. An increase in MA content also results in an increase in storage modulus; the storage modulus of [2 ma reference] is higher than that of [08 ma reference].

As seen from Table V, the presence of both MA and CNW leads to a decrease in the contact angle and, therefore, an increase in hydrophilicity of the samples.

As seen from Table VI, the presence of both MA and CNW leads to an increase in thermal

TABLE VI  
Thermal Conductivity ( $k$ )

Sample	Thermal conductivity (W/mK)
PP reference	0.2675
08 ma reference	0.3395
08 ma 5 cnw	0.3084
08 ma 10 cnw	0.3321
08 ma 15 cnw	0.4194

conductivity of the samples. This is due to the high thermal conductivity of CNW.<sup>21</sup>

### CONCLUSIONS

The benefits of polymer composites reinforced with nano materials have been widely reported. Derived from a renewable natural polymer, CNW offers great potential as an effective property enhancer for thermoplastic polymers. In this experimental investigation, the processing characteristics, mechanical and physical properties of CNW reinforced PP are studied as they are affected by CNW content, amount of MA grafted PP in the matrix polymer, and processing conditions. To enhance the uniform distribution of CNW in the composite, the matrix polymer is dissolved in toluene, and sonification and magnetic stirring are applied. Although good film transparency indicates uniform CNW dispersion, CNW domains in the composite film observed under an SEM may indicate slight agglomeration of CNW in the composite film. The tensile strength of the composite compared with neat PP is found to be improved by 70–80% with the addition of CNW. The crystallinity has also been improved by around 50% in the CNW reinforced samples. An increase of CNW content in the composite results in higher thermal degradation temperatures, higher hydrophilicity, and higher thermal conductivity.

Further investigation of finding effective surfactants and processing conditions could lead to better CNW distribution and mechanical/physical properties.

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