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## **Mechanical Properties of Nanoclay Reinforced Polypropylene Composites at Cryogenic Temperature**

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### **Abstract**

Polymer–clay nanocomposites are a new class of materials which improve the properties at low levels of addition of nanoclay as compared with conventional filler composites. In this article, a comparative study on the mechanical behavior of nanoclay filled polypropylene composites at both room and cryogenic temperatures is presented. Nanoclays of 1–5% by weight are added to polypropylene matrix using maleic anhydride grafted polypropylene as compatibilizer. Polypropylene/clay nanocomposites are prepared by melt intercalation in a twin-screw extruder followed by injection molding. Composite properties such as tensile and impact at both room (25°C) and cryogenic (196°C) temperatures are calculated. The addition of clay to polypropylene matrix showed a remarkable enhancement in the mechanical properties at temperatures of 25°C and 196°C. Nearly 36.4% and 17.6% increase in the Young's modulus and about 45% and 62.5% increase in the impact strength are observed at both room and cryogenic temperatures, respectively. Scanning electron microscopy is used to assess the surface morphology of the fractured surfaces and dispersion of the nanoclay. The morphology of polypropylene/clay nanocomposites is also characterized by X-ray diffraction analysis and transmission electron microscopy.

**Keywords:** Polypropylene, Nanocomposites, Nanoclay, Cryogenic temperature, Tensile test, Impact test

## 1. Introduction

Polymer composite materials are widely used in diverse applications such as transportation vehicles, construction materials, electronics and sporting goods, and consumer products.<sup>1</sup> The properties of polymer composites are greatly affected by the dimensions and microstructure of the dispersed phase. Polymer nanocomposites are a new class of materials with a great deal of future promise for potential applications as high-performance materials. In this new class of materials, nanosized fillers (at least one dimension) are dispersed in the polymer matrix by which a tremendous improvement in the performance properties of the polymer can be observed.<sup>2</sup> Polymer–clay nanocomposites (PCNs) have recently attracted a significant attention due to their marked property enhancements at small contents of nanoparticles substantially less than conventional fillers. For instance, increased tensile strength and modulus along with improved permeation resistance, flame retardation, and heat distortion characteristics are reported for many common polymers with the addition of only a few weight percent of clay.<sup>3</sup> These remarkable property enhancements make PCNs superior candidates for material applications in the food packaging, electronics, and automotive industries.<sup>4</sup> Nanoparticles, such as carbon nanotubes (CNTs) and nanoclay, have attracted a great interest due to their nanoscale dimensions and remarkable prospects for improvement of mechanical, thermal, electrical, and chemical properties, when introduced as small quantities in polymer matrix composites.

Nanoclay particles, which are one of the most affordable materials that have shown promising results in polymers, are made from montmorillonite (MMT) mineral having a

platelet structure with an average dimension of 1 nm thick and 70–150 nm wide. Nanoclays are known to enhance properties of many polymers such as nylon 6, epoxy, polyethylene terephthalate, polyethylene, and polypropylene (PP). The addition of nanoclay in the PP matrix increases the thermal stability in air medium, increases physical properties (dimensional stability), improves flame retardant properties (increased thermal-oxidative stability and reduced heat release rate), and improves mechanical properties, fracture properties, and gas barrier properties. Several studies were conducted with various types of organoclays, clay concentrations, and compatibilizers.<sup>5,6</sup> Nanomer<sup>®</sup> and Cloisite<sup>®</sup> are the popular nanoclays available in the market. Cloisite additives are used in injection-molded parts, films, bottles, trays, blister packs, and wire and cable coatings.<sup>7–11</sup> PP is one of the most widely used polyolefins. PP is applied in a wide variety of applications including packaging industries, textiles, automobile, plastic parts and reusable containers of various types, furniture, laboratory equipment, and loudspeakers. It has good properties and is of relatively low cost.<sup>12,13</sup> Since the successful preparation of PP/ clay nanocomposites with maleic anhydride grafted PP (PP-g-MA) as a compatibilizer is possible, various studies on the preparation and characterization of PP/ clay nanocomposites are reported.<sup>14–17</sup>

In these articles, PP-g-MA was used to promote better dispersion of clay in a nonpolar PP matrix and to increase the adhesion between PP and clay particles. A few systematic studies are done on the preparation and characterization of PP composites. For instance, the addition of calcium carbonate (CaCO<sub>3</sub>) nanoparticles to a PP matrix resulted in some improvement in the modulus and impact strength and reduction in costs, but the tensile properties of PP was slightly reduced and also a poor dispersion of the CaCO<sub>3</sub> within the PP matrix was observed.<sup>18,19</sup>

The addition of silica powder to the PP matrix resulted in aggregated silica particles, but there was no outstanding improvement in the mechanical properties of PP/SiO<sub>2</sub> nanocomposites.<sup>20</sup> However, when polymethyl methacrylate grafted on nanosilica was added to the PP matrix, it improved the dispersion of the nanoparticles and interfacial adhesion and decreased the size of PP spherulites in the nanocomposites. Finally, it lead to an increase in the Young's modulus and toughness of PP/silica nanocomposites.<sup>21</sup> CNT particles are more rigid and interfacial adhesion of PP/CNTs is weak. Therefore, when PP-g-MA was added to the PP matrix, a better dispersion and good adhesion were attained between the nanotubes and the PP matrix as compared to the neat PP. This lead to an increase in the tensile and flexural moduli and Charpy impact strength.<sup>22</sup>

For engineering applications, the mechanical properties of PP composites are highly related to the changes in temperature. However, the results of the mechanical properties obtained from room temperature (RT) cannot be simply transferred to the cryogenic case. Thus, for cryogenic engineering applications, it is important and necessary to study the relative properties of PP/clay at cryogenic temperature (CT). During the recent years, a few studies on materials, particularly nanocomposites, were reported at CT.

For instance, CNTs are known as exceptional reinforcements for polymers. However, poor CNT/polymer interfacial bonding leads to the unexpected low reinforcing efficiency. The cryogenic mechanical properties are all enhanced at CT by the addition of multi-walled carbon nanotubes (MWCNTs) at appropriate contents. When the temperature decreases from RT to CT, a strong CNT/epoxy interfacial bonding was observed due to the thermal contraction of the epoxy matrix because of the large differences in the thermal expansion coefficients of epoxy and MWCNTs and resulted in a higher reinforcing efficiency.<sup>23</sup> Mechanical behavior of

MMT/epoxy nanocomposites at both RT and CT were investigated in terms of tensile and impact properties. The strength and modulus of the nanocomposites were significantly higher at CT than at RT and these observations were explained in terms of the interfacial adhesion and constituent properties at CT than RT.<sup>24</sup>

The effect of silica nanoparticles was studied on the cryogenic tensile and thermal properties of the epoxy nanocomposites. The results showed that the tensile strength at CT was significantly enhanced by the addition of a low content of silica nanoparticles, while the strength at RT was almost insensitive to the addition of silica nanoparticles. The modulus at both RT and CT was increased with the increase in silica content. The tensile strength and modulus at CT was much higher than at RT. The failure strain at CT was slightly enhanced by the addition of silica nanoparticles, yet the failure strain at RT was obviously improved by the addition of silica nanoparticles.<sup>25</sup>

Also, 30% chopped glass fibers reinforced polyether ether ketone (PEEK) composites were prepared by injection molding, and then, the tensile, flexural, and impact properties were tested at different temperatures. The modulus, strength, and specific elongation of glass fibers reinforced PEEK at 25°C, 196°C, and 253°C were compared. The results showed a dependence of mechanical properties of glass fibers reinforced PEEK composites on temperature. The coefficient of thermal expansion of unfilled PEEK and glass fibers reinforced PEEK were also investigated from 196°C to 25°C. The results indicated that the thermal expansion coefficient of PEEK matrix was nearly a constant in this temperature region, and it can be significantly decreased by adding glass fibers.<sup>26</sup>

By studying the works of other researchers, it was observed that no work or results were published regarding the PP polymer composites reinforced by clay nanoparticles at CT.

Therefore, in this article, PP/clay nanocomposites are prepared by melt mixing in a co-rotating intermeshing twin-screw extruder followed by injection molding with different weight percentages of clay content. PP-g-MA is added as a compatibilizer agent to the PP matrix. The effect of adding various amounts of nanoclay to the PP matrix is investigated.

Mechanical properties such as impact and tensile as a function of clay concentration are studied at both RT and CT. Impact and tensile tests are carried out according to ASTM standards. The surface morphology of the fractured surfaces of nanocomposite specimens and dispersion of the nanoclay in the PP matrix are investigated using a scanning electron microscopy (SEM) and the fractured surfaces are studied by the photographs obtained at a higher magnification.

## **2. Experimental**

### **2.1. Materials**

PP with a trade name of Jampilen HP500M from Jam Petrochemicals, Iran (melt flow rate at 230°C for 2.16 kg load = 9 g/10 min, density = 0.9 g/cm<sup>3</sup>) was used as the polymer matrix. Clay with the trade name of Cloisite® 15A (Pishgaman Fanavari Asia, Iran) was used. This is a natural MMT clay that was organically treated with a quaternary ammonium salt. The organic modifiers used were dimethyl, dehydrogenated tallow, quaternary ammonium salt. The *d*-spacing of Cloisite® 15A is 34.7 Å. Anti-peroxide Ciba-Irganox B215 from Jam Petrochemicals, Iran, was added to the PP matrix and PP-g-MA with a trade name of KARABOND® PCH (melt index at 190°C for 2.16 kg = 6 ± 0.5 g/10 min, melting point (1kg) = 165°C, and graft efficiency = 0.6–1%) from Karangin Company, Iran, was used as the compatibilizer.

## **2.2. Preparation of the PP nanocomposites**

Anti-peroxide (0.2 wt%), PP-g-MA (5 wt%), and different amounts of clay contents (1, 3, and 5 wt%) were added to the PP nanocomposites. The materials were melt-compounded in a brabender-type twin-screw extruder (plasticorder model pl2002, Germany) with a length/diameter ( $L/D$ ) ratio of 40. The barrel temperatures were set at 165-195°C (feed zone to die zone) and the velocity of the processing was fixed at 150 r/min. To achieve good mixing, the feeding rate was kept low at 2 kg/h. The resulting material is dried at a temperature of 80°C for 24 h. After that, the injection molding machine (Imen Machine Co., Tehran, Iran) was used for the preparation of impact and tensile specimens. Zone temperatures were 175-205°C and injection pressure 96 bar. The specimens were cut from the injection molded parts for impact test (7.2 mm thickness, 12.5 mm width, 63.5 mm length, and notch 3.50 mm) and for tensile test (3 mm thickness, 13 mm width, and 165 mm length) considering the recommendations of standard ASTM-D 256 and ASTM-D 638, respectively. Five specimens for each composite type were tested and the average values were reported.

## **2.3. Morphology characterization**

The structure of the nanocomposites was studied using X-ray diffraction (XRD) analysis and transmission electron microscopy (TEM) methods. The X-ray diffractometer (Philips Analytical, model XPert MPD with Co radiation from Japan) (0.175 nm wavelength at a generated voltage of 40 kV and current of 30 mA) of powder specimen was used. X-ray diffractograms were taken on Cloisite 15A clay particles and PP/clay nanocomposites. Microscopic investigation of selected nanocomposite specimens at the various weight

compositions were conducted using a TEM (Philips EM208, Germany) with an operation voltage of 100 kV.

## **2.4. Mechanical test**

The notched impact strength was measured for both RT and CT with a Charpy impact tester (Model ZBC1251, Santam Co., Iran) according to ASTM D-256. Impact testing at CT was performed with the specimens dipped in liquid nitrogen for 20 min and was completed in a couple of seconds after taking the specimens out from the liquid nitrogen tank. The tensile specimens were prepared according to the recommendation of ASTM D-638. The tensile properties of PP/clay nanocomposites at RT and CT were measured by a mechanical tester (Model Hounsfield, H25Ks, England) with a 25 kN load cell and with a crosshead speed of 25 mm/min at RT and 5mm/min at CT. The CT condition was achieved by dipping the specimens in liquid nitrogen for 20 min. The surface morphology of the fractured surfaces of the nanocomposite specimens and dispersion of the nanoclay in the PP matrix were investigated using an SEM (XL30 Philips, Dutch) and the fractured surface photographs were obtained by the high-magnified camera for detailed analysis.

## **3. Results and discussion**

### **3.1. Mechanical properties**

***Notched impact test.*** Notched impact tests of the PP/clay nanocomposites as well as the neat PP matrix were conducted at both RT and CT and the results are presented in Table 1. It can be seen that the addition of clay contents into the PP matrix can effectively enhance the impact strength of the PP matrix.

Table 1. Impact strength at RT and CT of the PP matrix and PP nanocomposites

| Property/sample                                                              | RT    |       |       |       | CT    |       |       |       |
|------------------------------------------------------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|
|                                                                              | P0RI  | PIRI  | P3RI  | P5RI  | P0CI  | P1CI  | P3CI  | P5CI  |
| Notched impact strength (kJ/m <sup>2</sup> )                                 | 15.63 | 18.48 | 19.32 | 22.68 | 12.34 | 13.88 | 16.97 | 20.06 |
| Density (g/cm <sup>3</sup> )                                                 | 0.91  | 0.915 | 0.92  | 0.925 | 0.91  | 0.915 | 0.92  | 0.925 |
| Specific notched impact strength ((kJ/m <sup>2</sup> )/(g/cm <sup>3</sup> )) | 17.17 | 20.19 | 21    | 24.51 | 13.56 | 15.16 | 18.44 | 21.68 |
| Specific notched impact strength increase in comparison to P0RI (%)          | –     | 17.58 | 22.30 | 42.74 | 21    | 11.7  | 7.39  | 26.26 |
| Specific notched impact strength increase in comparison to P0CI (%)          | 26.62 | 48.9  | 54.86 | 80.7  | –     | 11.8  | 35.98 | 59.88 |

Definition of the codes: P, polypropylene; 0–5, weight percentage of nanoclay; R and C, room and cryogenic temperatures, respectively, and I, impact test.

RT: room temperature; CT: cryogenic temperature; and PP: polypropylene.

Also, Figure 1 shows the plot of notched impact strength of the PP composites at RT and CT as a function of nanoclay content. When the clay content is 5 wt%, the impact strength is greatly enhanced and increased by 45% as compared to P0RI. This can be explained using the selective dispersion of clay in the brittle PP matrix. Also, it is possible that the clay layer orientation as well as molecular orientation contribute to the observed reinforcement effects.

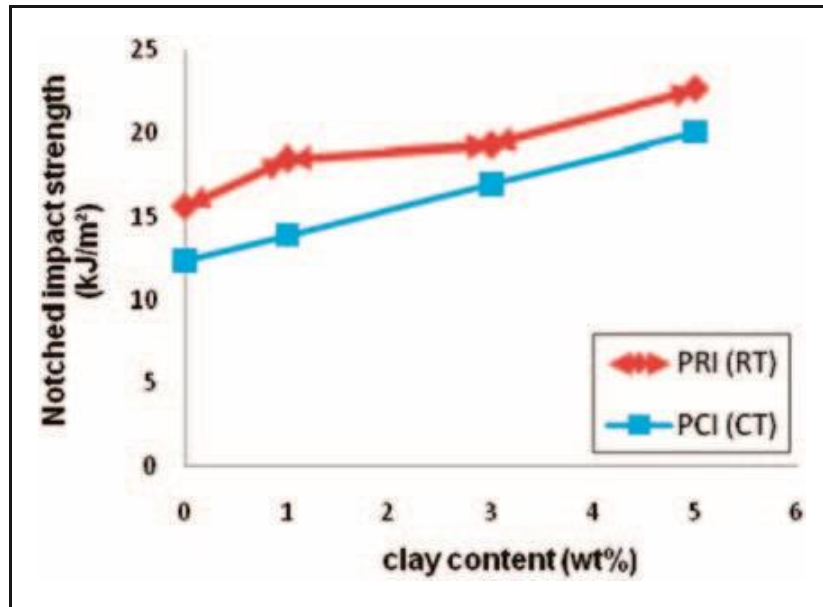


Figure 1. Notched impact strength of the PP composites at RT and CT.

(PP: polypropylene; RT: room temperature; and CT: cryogenic temperature)

Toughness improvement of nanocomposites can be attained when the clay in the PP matrix can withstand the propagation of cracks with increasing the clay content at RT, as indicated by the increase in notched impact strength. The micrographs of the fractured surfaces obtained by SEM are shown in Figure 2. The more rough surfaces of the nanocomposites in Figure 2(b) and (c) and better dispersion and distribution of clay particles in the PP matrix as compared to those of neat PP matrix in Figure 2(a) are indicative of the improvement in the toughness. The impact strength is higher at RT as compared to CT with the same composition. This is mainly because the molecular mobility of the PP matrix would be lowered when the temperature was down to CT from RT. When rapid impact loading was applied to the specimens, it would be difficult to have plastic deformation, and hence, relatively low impact energies were required to break the specimens at CT. Maximum and minimum notched impact strengths at CT related

to P5CI and P0CI are 20.06 and 12.34 kJ/m<sup>2</sup>, respectively, which shows an improvement of 62.5% as compared to P0CI.

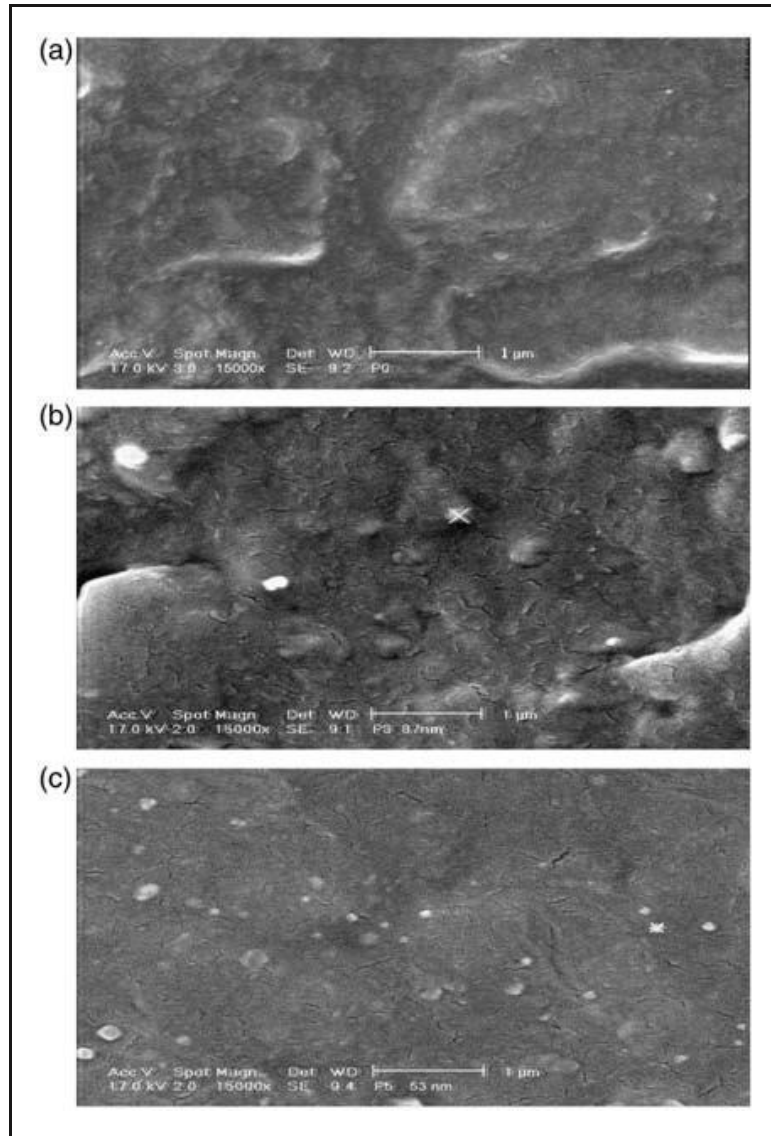


Figure 2. SEM micrographs of composite fracture surfaces for: (a) neat PP; (b) PP with 3 wt% nanoclay; and (c) PP with 5 wt% nanoclay.

(SEM: scanning electron microscopy; PP: polypropylene)

The improvement of PP nanocomposites by clay contents is higher at CT as compared to RT. It shows that the particles are more effective in stopping the crack propagation, besides the brittle behavior of the specimens at CT.

**Tensile test.** The PP/clay nanocomposites exhibit relatively ductile behaviors at RT compared to the results at CT and obviously shows brittle behaviors at CT for all compositions. Tensile properties such as ultimate tensile strength, yield strength, Young's modulus, displacement at break, and energy absorption (the area under stress-strain curve) for PP and PP/nanoclay composites at RT and CT as a function of weight percentage of nanoclay are presented in Table 2. It is seen that the yield strength of the PP/clay nanocomposites increased at RT by increasing the content of clay nanoparticles.

Table 2. Tensile properties at RT and CT of the PP and PP nanocomposites

| Property/sample                                                       | RT     |        |        |        | CT     |        |         |         |
|-----------------------------------------------------------------------|--------|--------|--------|--------|--------|--------|---------|---------|
|                                                                       | P0RT   | PIRT   | P3RT   | P5RT   | P0CT   | P1CT   | P3CT    | P5CT    |
| Displacement at break (%)                                             | 307.64 | 274.69 | 273.22 | 243.95 | 4.7    | 2.32   | 3.14    | 4.8     |
| Yield strength (MPa)                                                  | 24.4   | 23.16  | 23.76  | 24.12  | 43.69  | 40.53  | 46.19   | 48.07   |
| Energy absorption (J)                                                 | 156.68 | 151.89 | 131.99 | 118.23 | 5.04   | 1.6    | 2.94    | 5.34    |
| Ultimate tensile strength (MPa)                                       | 19.76  | 18.06  | 16.68  | 15.53  | 43.69  | 50.53  | 46.19   | 48.07   |
| Young's modulus (MPa)                                                 | 523.42 | 588.17 | 612.33 | 714.22 | 903.61 | 935.33 | 1003.9  | 1063.43 |
| Density (g/cm <sup>3</sup> )                                          | 0.91   | 0.915  | 0.92   | 0.925  | 0.91   | 0.915  | 0.92    | 0.925   |
| Specific ultimate strength (MPa/(g/cm <sup>3</sup> ))                 | 21.71  | 19.73  | 18.13  | 16.78  | 48.01  | 44.29  | 50.2    | 51.96   |
| Specific stiffness (MPa/(g/cm <sup>3</sup> ))                         | 575.18 | 642.8  | 665.57 | 772.12 | 992.97 | 1022.2 | 1091.19 | 1149.65 |
| Specific toughness                                                    | 172.17 | 166    | 143.46 | 127.81 | 5.53   | 1.74   | 3.19    | 5.77    |
| Energy absorption/P0RT energy absorption                              | –      | 0.96   | 0.84   | 0.75   | 0.032  | 0.01   | 0.02    | 0.034   |
| Energy absorption/P0CT energy absorption                              | 31.08  | 30.13  | 26.18  | 23.45  | –      | 0.31   | 0.58    | 1.05    |
| Specific ultimate tensile strength increase in comparison to P0RT (%) | –      | 9.12   | 16.5   | 22.7   | 121.14 | 104    | 131.22  | 139.33  |

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|                                                                       |       |       |       |       |       |       |       |       |
|-----------------------------------------------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| Specific ultimate tensile strength increase in comparison to P0CT (%) | 54.78 | 58.90 | 62.23 | 65.04 | –     | 7.74  | 4.56  | 8.22  |
| Specific stiffness increase in comparison to P0RT (%)                 | –     | 11.75 | 15.71 | 34.23 | 72.63 | 77.72 | 89.71 | 99.87 |
| Specific stiffness increase in comparison to P0CT (%)                 | 42.07 | 35.26 | 32.97 | 22.24 | –     | 2.94  | 9.89  | 15.77 |

Specimen codes: P, polypropylene; 0, 1, 3, and 5, weight percentage of nanoclay; R and C, room and cryogenic temperatures, respectively; and T, tensile test.

RT: room temperature; CT: cryogenic temperature; and PP: polypropylene.

The yield strength of PP composites at RT and CT as a function of nanoclay content is shown in Figure 3. It can be seen that the yield strength at CT is consistently higher than that at RT with the same composition. This is because of the nanolayers of clays which might be well separated and dispersed randomly in the polymer matrix. A significant enhancement in the cryogenic strength is observed by the addition of clay contents except for pure PP matrix samples. The results showed that with the addition of 5 wt% clay content, the yield strength of PP/clay nanocomposites at both RT and CT reached the maximum increase by 4% and 18.6% as compared to P1RT and P1CT, respectively. The value of ultimate tensile strength at RT for neat PP (P0RT) is 19.76 MPa, and at CT is 43.69 MPa, which shows an increase of 121% at CT, when compared to RT results. But, by adding 5 wt% nanoclay particles (P5RT), ultimate tensile strength reduces to 15.33, which shows a reduction of 21% as compared to P0RT. However, P5CT shows a strength of 48.07 MPa, which indicates an increase of 10% as compared to P0CT. This can be explained as when the temperature decreases from RT to CT, the chemical bond and the molecules of PP matrix get shrunk and the binding forces between the molecules become strong. Thus, a larger load is needed to break the PP matrix at CT, leading to a higher strength of the PP matrix at CT than at RT.

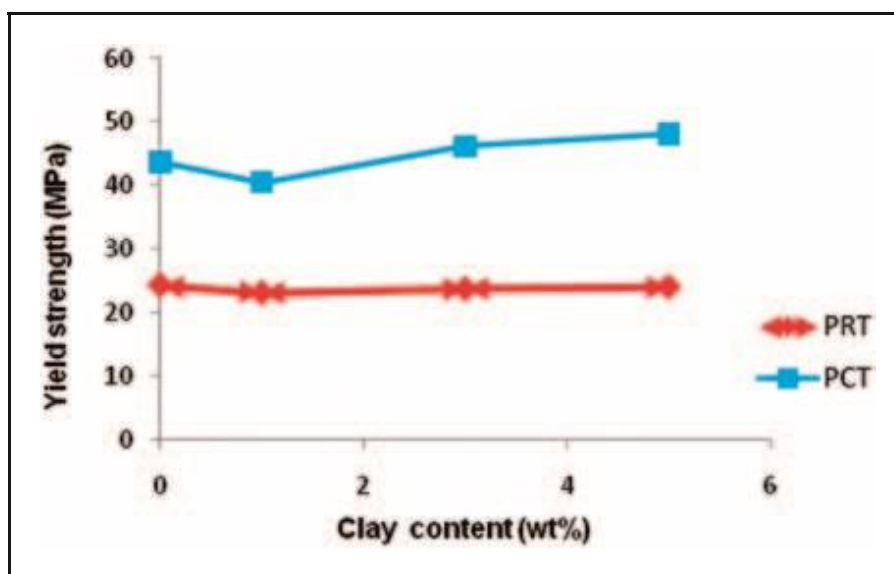


Figure 3. Yield strength of the PP composites at RT and CT.

(PP: polypropylene; RT: room temperature; and CT: cryogenic temperature)

The energy absorption at CT was enhanced by approximately 6% when 5 wt% of nanoparticles was added. Yet, energy absorption at RT was obviously reduced by 24.5% for P5RT as compared to P0RT. Maximum and minimum Young's moduli related to P5RT, P5CT and P0RT, P0CT are 714.22, 1063.43 and 523.42, 903.61 MPa, respectively. Young's moduli of P5RT and P5CT indicate an improvement of 36.4% and 17.6% as compared to P0RT and P0CT, respectively.

Also, Figures 4 and 5 show the variation of energy absorption and Young's modulus of the PP nanocomposites at RT and CT as a function of nanoclay content. The modulus at CT is much higher than that at RT; this is due to the tight arrangement of polymer molecules and also the good interfacial bonding. On the other hand, any material shows higher modulus at lower temperature.<sup>24</sup> So, the modulus of clay would also be higher at CT, contributing to the higher modulus of the composites at CT. The nanocomposites at CT are all brittle as compared to RT energy absorption. But, adding nanoparticles have different effects for CT and RT. The effect

of nanoparticles at RT is reduction of the energy absorption, but at CT, the materials show a slightly ductile behavior at higher wt% of nanoparticles.

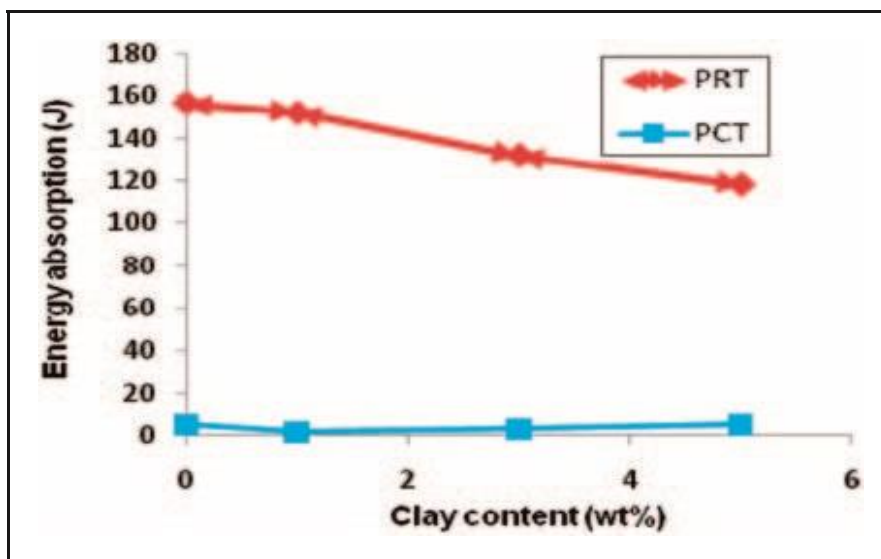


Figure 4. Energy absorption of the PP composites at RT and CT.

(PP: polypropylene; RT: room temperature; and CT: cryogenic temperature)

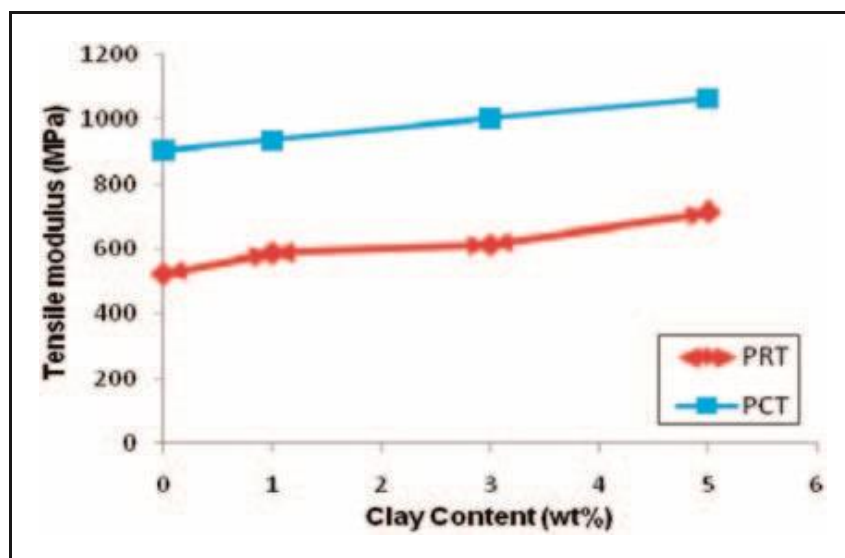


Figure 5. Young's modulus of the PP composites at RT and CT.

(PP: polypropylene; RT: room temperature; and CT: cryogenic temperature)

### 3.2. Scanning electron microscopy

The fractured surfaces of PP and its nanocomposites are observed using SEM. Figure 2(a) to (c) shows a transition from a ductile to brittle fracture of the notched impact specimens at RT. Matrix deformation is greatly reduced when the clay in PP matrix can withstand the propagation of cracks with increasing the clay content at RT as indicated by the increase in notched impact strength. Figure 2(b) shows the SEM micrograph of PP/clay (3 wt%) nanocomposite. This figure shows that the aggregates of nanoparticles are large as compared to PP/clay (5 wt%) nanocomposite. With the addition of nanoclay, the aggregates are small and this confirms the better dispersion of organoclay and PP matrix, which is shown in Figure 2(c). Also, this indicates higher impact properties and other more efficient mechanical properties.

Figure 2(a) shows that the surfaces of the neat PP specimens are smooth and featureless, representing brittle failure of homogenous materials. As shown in the SEM images for the PP/clay nanocomposite specimens with 3 wt% and 5 wt% nanoclay contents, the fractured surfaces obtained are significantly different from those of neat PP specimens. The fractured surfaces are broken into small and rough fracture pieces, contributing to the improvement of the toughness of the nanocomposites.

### 3.3. Structure and morphology of PP/clay nanocomposites

The structure and morphology of nanoclay filled PP composites was examined using XRD and TEM methods. Figure 6 shows the XRD patterns of PP/clay nanocomposites. The Cloisite 15A nanoclay shows a diffraction peak of  $2\theta$  at  $3.2^\circ$  and corresponds to an interlayer spacing of nanoclay ( $d$ -spacing) of  $34.79 \text{ \AA}$  (calculated from Bragg's diffraction law of  $2d\sin\theta = n\lambda$ ). From 1 to 5 wt% of nanoclay in the PP polymer, there is an absence of a diffraction peak, and

this suggests that the polymer matrix has entered into the interlayer spacing of the nanoclay and increased the original nanoclay spacing above or the nanolayers of clay could have randomly dispersed in the PP polymer. Hence, it can be concluded that the nanoclays in the PP polymer from 1 to 5 wt% formed an ordered exfoliated structure, or a randomly dispersed clay exfoliated structure. Additional information on the structure of the nanocomposites was obtained with the use of TEM, as shown in Figure 7. At the micrometer scale (scale bar 0.2  $\mu\text{m}$ ), the information obtained is similar to the SEM images in Figure 2, but is defined more clear. This is not only because of the higher resolution of TEM, but also due to the fact that the transmission images are not affected by the morphology of the fractured surface. In these TEM micrographs, some aggregations of clay are also detectable, which indicate the exfoliation degree in such PP/clay nanocomposites synthesized via melt mixing.

The bright region of the figure is the matrix phase and the dark region the particle phase. From 1 to 5 wt% nanoclay in the PP polymer matrix, the nanolayers of clay were well separated and dispersed randomly in the polymer matrix. This shows the formation of an exfoliated structure. The TEM images support the XRD results for the nanocomposite.

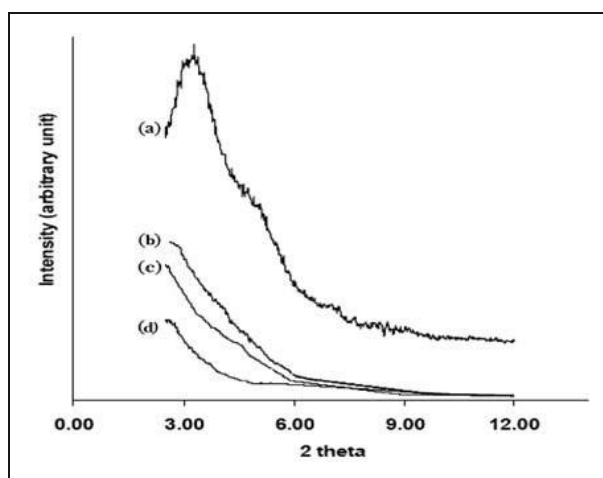


Figure 6. XRD patterns of: (a) Cloisite 15 A; (b) PP with 5 wt% clay; (c) PP with 3 wt% clay; and (d) PP with 1 wt% clay. (XRD: X-ray diffraction; PP: polypropylene)

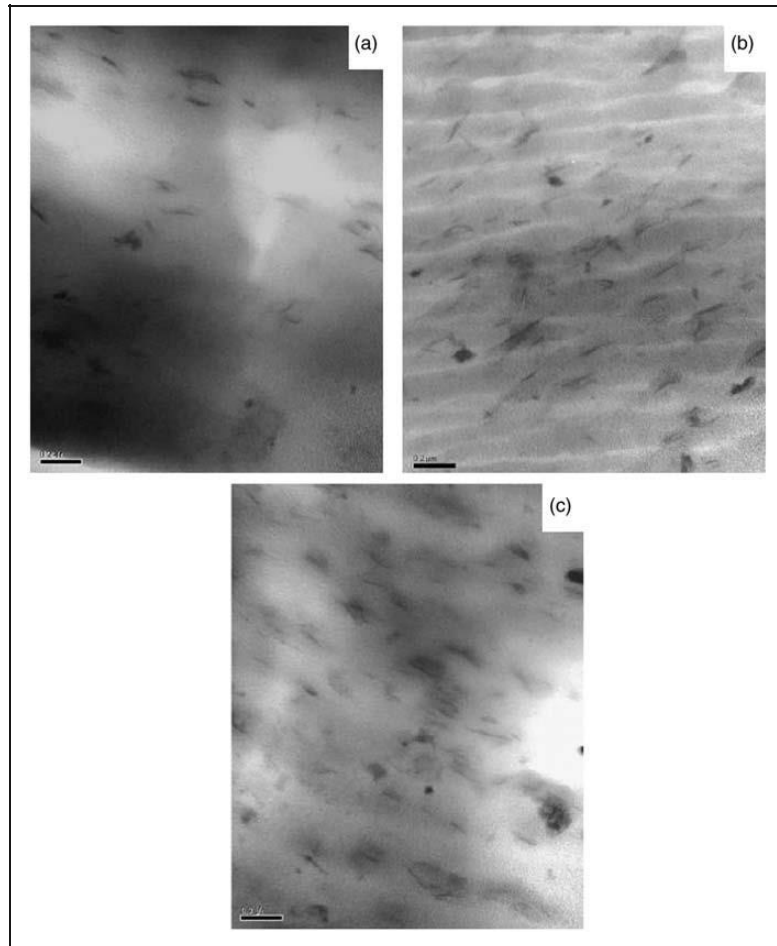


Figure 7. TEM image of PP/clay nanocomposites with different nanoclay contents: (a) 1 wt% clay; (b) 3 wt% clay; and (c) 5 wt% clay.

(TEM: transmission electron microscopy; PP: polypropylene)

### 3.4. Fractured surface analysis

Fractured surfaces of the specimens after tests are observed in this section. Figure 8 shows the fractured surfaces of P0CI, P5CI and P0RI, P5RI specimens after notched impact tests, respectively.

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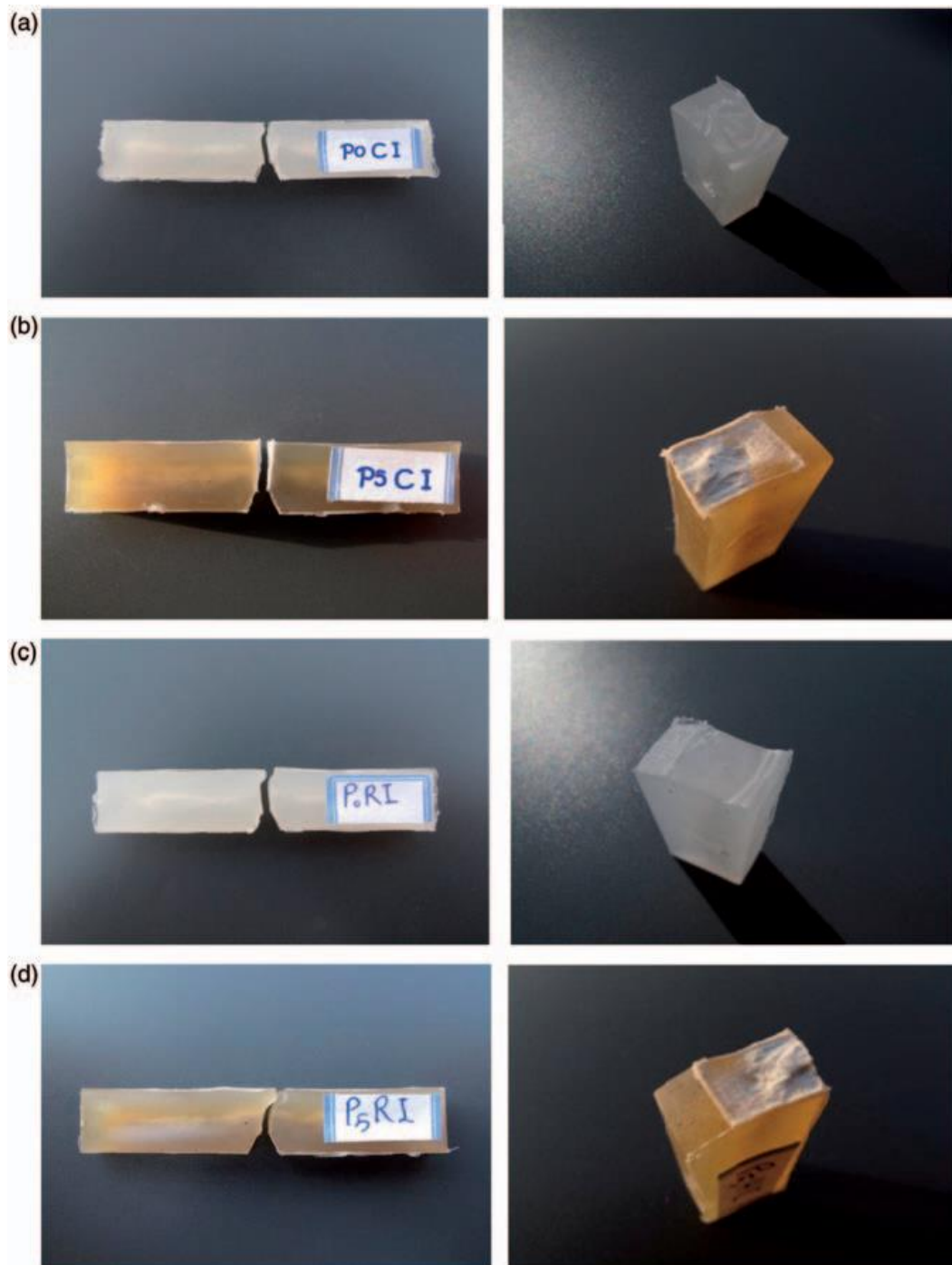


Figure 8. Charpy impact notched specimen after test: (a) P0CI; (b) P5CI; (c) P0RI; and (d) P5RI.

The specimens with nanoparticles indicate more dimple features than those without nanoparticles. This is due to the presence of nanoclay that indicates a strong interface between

PP matrix and nanoparticles, which shows higher notched impact strength. The specimens without nanoclay particles at both RT and CT show cleavage fracture, which is an indication of brittle materials.

Also, the fractured impact specimens at CT show more brittle behavior than the specimens at RT. Figure 9 shows the fractured surfaces of P0RT and P0CT specimens, respectively, after tensile tests. P0RT stretched more as compared to the P0CT, which indicates better elongation, but lower tensile properties at RT. The clay/PP nanocomposites specimens show a relatively ductile behavior at RT as compared to that at CT, and therefore, the PP matrix shows obviously brittle behavior at CT for all compositions of nanoparticles added to the PP matrix.

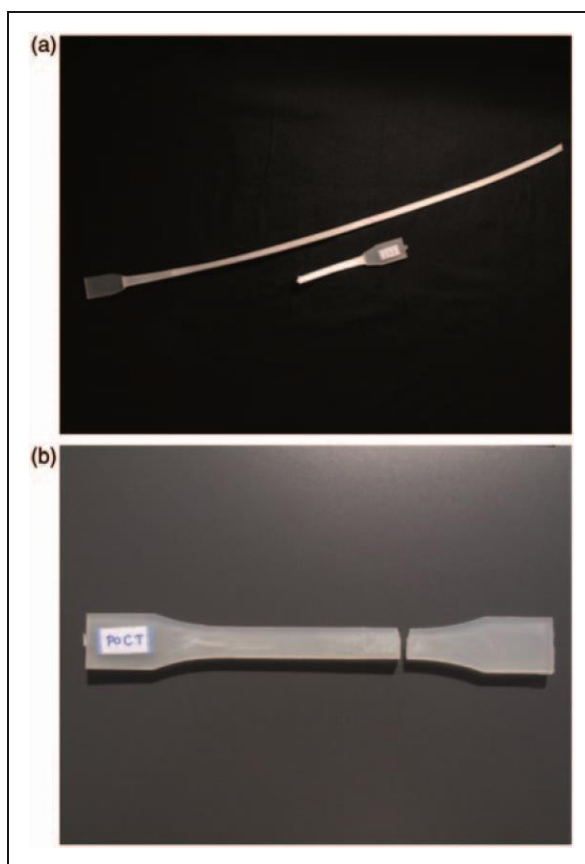


Figure 9. Tensile specimen after test: (a) P0RT and (b) P0CT.

#### **4. Conclusion**

The mechanical properties of PP/clay nanocomposites prepared by adding different clay contents to PP matrix are studied at both RT (25°C) and CT (196°C) and compared to the properties of the neat PP. The following conclusions are obtained.

1. Notched impact strengths are increased at both RT and CT by increasing the weight percentage of nanoclay, because of the reinforcement effect of the nanoclay particles. Notched impact strengths of PP matrix without nanoparticles and with 5 wt% nanoparticles are higher at RT.
2. The values of ultimate tensile strength at RT for PP matrix reinforced by 5 wt% nanoclay particles is reduced as compared to PP matrix without nanoparticles, but at CT, the same specimens show higher values.
3. Yield strength at CT for neat PP and specimen with 5 wt% nanoclay content are higher than neat PP and specimen with 5 wt% nanoclay content at RT.
4. The tensile modulus of nanocomposites at both RT and CT increased almost linearly with the increase of clay content. Moreover, both the tensile strength and tensile modulus at CT were much higher than those at RT.
5. The energy absorption at CT was slightly enhanced by the addition of clay nanoparticles. Yet, energy absorption at RT was obviously reduced by the addition of clay nanoparticles.

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