A Study of the Effect of Iraqi Bentonite on Some Properties of Polymeric Blend

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Received on: 29/6/2011 & Accepted on: 7/6/2012

ABSTRACT

In this research, the specimens of composite materials were prepared from matrix (polypropylene and polymethylmethacrylate) (80 /20) wt% in different weight fractions (3, 6, 9, 12 & 15) wt% and different particle sizes (45, 60, 70 & 110) µm of Bentonite. The results have shown that after the reinforcement with different weight fraction of Bentonite powder most mechanical properties, such as hardness and modulus of Elasticity increase. Elongation decreases with an increase in weight fraction of (3, 6, 9, 12, 15) wt% and decrease with particle size decrease (45, 60, 70, 110) µm. Also the results have shown that the thermal stability of PP/PMMA increases with addition of Bentonite and the thermal stability of small particle size (45 µm) was higher than larger particle size (110 µm).

PP/PMMA/BN [(80/20/15) > (80/20/12) > (80/20/9) > (80/20/6) > 80/20/3)].

The results have shown the diffusion coefficient (D) was increased with temperatures (30, 45, 60) °C and decreased with filler content (3, 6, 9, 12, 15) wt% increase.

The activation energy (E) increase with filler content (BN) increase and (E) increases with particle size of BN filler decrease.

Also the results have shown the addition of BN particles of PP/PMMA blend increases the discontinuity and restricts the growth of crystal therefore crystalline peak intensity is reduced in filler blend composite. Addition of large particle size of BN adversely affects the crystallization of PP.

دراسة تأثير البنتونايت العراقي على مخلوط بوليمر مكون من بولي برووسلين/بولي ميثاكريلينيت

الخلاصة

المواد المترابكة تم تحضيرها من خلط البولي بروبلين بنسبة 80% والبولي ميثاكريلينيت بنسبة 20% مع مختلف النسب الوزنية من مسحوق البنتونايت وباحجام حبيبية مختلفة وبعد ذلك جرت دراسة بعض الخواص الميكانيكية والفيزياوية وحرارية وفحص البلورية وجميع المترابكات المحضرة.

النتائج بينت أنه بعد التحديم لمختلف النسب الوزنية (3-12) % والاهجام المختلفة (5, 10, 6, 7, 10-200) مايكرميتر من مادة البنتونايت فان أغلب الفحوصات الميكانيكية ازدادت مثل هذا.
INTRODUCTION

Composites are combination of two or more materials presented as separate phases and combined to form desired structures Navin and Vashishtha[1], used x-ray then they developed a new type of fly ash filled PP/PMMA blends system. Effect of incorporation of fly ash particulate on microstructure, strength and thermal stability has been determined and analyzed. Addition of fly ash imparted dimensional and thermal stability, which have been observed in scanning electron micrographs and in TGA plot. Increase in fly ash concentration increases the initial degradation temperature of PP/PMMA blend. The increase in thermal stability has been explained based on increases in mechanical interlocking of PP/PMMA chains inside the hollow structure of fly ash.

Abed-Alhakem[3], studied the effect of Iraqi raw ceramic (Kaoline and boxide) on the epoxy resin, he showed the tensile properties, elongation at break, Young’s modules, and stress break of composites filled with proportionally small particles all increase with increasing filler content and decreasing filler size.

BalaMuruaan et al[4], studied the development and spectral characterisation of polymethylmethacrylate/ Hydroxyapatite composite for biomedical application. Among the composite materials (ceramic/ metals, ceramic/ polymers and ceramic/ ceramics) available, ceramic/ polymer composite possesses significant advantages of high mechanical reliability (polymers) and excellent biocompatibility (ceramics) for applications in load bearing areas. The study is an attempt to develop a ceramic/ polymer composite made by grafting characterized using spectral techniques.

Suryasaralhi and Mahanwar[23], studied the effect of added (mica) with variable particle size on the mechanical, thermal, electrical, rheotical and morphological properties of filled Nylon [6]. They showed improved mechanical, thermal as well as electrical properties on addition of filler. It is also observed that mechanical properties, electrical properties as well as thermal properties increase with decrease in particle size.

Anshulshal[5], found that fillers such as carbon black and clay, which are currently used in polymer blends (PS/PMMA) improve the physical properties of the material, also can hinder the adhesion at polymer interfaces. Fillers can block polymer diffusion by interacting with the polymer molecules and pinning them, causing adhesive failure. However, he started demon that this effect can be overcome by the addition of colloidal silica particles, by where the surface interaction is screened.
Mustafa [6], prepared polymer blend of polypropylene (PP) and polymethylmethacrylate (PMMA) with Kaoline filler (5,10,15) w% in different particle size (40,60,80,100) mixing of PP, PMMA and Kaoline was carried out then the mixture was further mixed using mixing machine and single screw-extruder operated at a temperature ranging from (120-200°C). In order to determine the performance of polymer blend prepared, in this work several mechanical and physical tests were carried out before and after filler (Kaoline) added to PP/PMMA. Results obtained in this study indicate that most mechanical properties such as (tensile strength, impact, hardness and elasticity) except elongation at break percent increase after adding reinforcement with (5,10,15) w% of kaoline and the same properties also increase with decrease in particle size of filler. The absorption test was carried out in water, the results of absorption show that it obeys Fick’s law and after the addition of Kaoline the absorption decreases.

Mahmood [7], prepared polymer blend of polypropylene (PP) and polycarbonate (PC) with carbon black (1%). Mixing of PP, PC and carbon black was carried out then the mixture was further mixed using mixing machine and single-screw extruder operated at a temperature ranging from (120-200°C). In order to determine the performance of polymer blend prepared in this work several mechanical and physical tests were carried out before and after exposing the samples to weathering test. Results obtained in this study indicate that most mechanical properties such as (tensile strength, impact, hardness and elasticity) except percentage of elongation at break increase after reinforcement with (1%) of carbon black. The absorption test was carried out in oil (kerosene) H₂SO₄ (2N) and NaOH (2N), the results of absorption show that it obeys Fick’s law and after the addition of carbon black the absorption decreases.

Al-Allaq [9], prepared polymeric blends from epoxy resins, unsaturated polyester and polysulphed rubber with (0%-100%) ratio as binary blends. To study the mechanical properties and thermal properties and microscope structure, then she prepared two types of composite which consist of matrix of (pure polymer and polymer blends) and carbon fiber + glass fiber as filler. Al-Allaq Found that when comparing the mechanical properties of composite materials based on the pure polymer matrix and those of composite materials based on polymeric blend it can be seen that composite materials based on blends matrix have improvements in the tensile strength as well as in the thermal stability In this work development of a new type of PP/PMMA blend filled with local Iraqi Bentonite as filler using single screw extruder and molding compression. Determining the morphology, ultimate tensile strength, thermal properties, x-ray, diffusion, hardness, elastic modulus and elongation of filled and unfilled PP/PMMA blends.

**EXPERIMENTAL WORK**

**Matrix (polymer raw materials)**

Commercial grade polypropylene (pp) was supplied by Sabic – Saudi Arabia. The melt flow index and the density of this material were 11gm/10min and 0.98 (gm/cm³) respectively, melting temperature was (168°C). Commercial grade of polymethylmethacrylate (PMMA) was supplied by M/S Monika Plastic – India. The density of the material was 1.19 (gm/cm³). Melting temperature was (160°C) and the melt flow index was 1 gm/10 min [10].

**Reinforced (filler) materials**
Bentonite clay (BN) has particle size of 110µm and density (2.55) g/cm³. It has a good strength, stiffness, and it is able to absorb moisture and available at low cost. Bentonite was supplied by Iraqi National Company for Geological Survey and Mining. Mineralogy of Bentointe: ca-montmorillonite. Its chemical composition is shown in the following table: [11]

Table (1) Chemical composition of Bentonite [11].

<table>
<thead>
<tr>
<th>SiO₂%</th>
<th>Al₂O₃%</th>
<th>Fe₂O₃%</th>
<th>CaO%</th>
<th>MgO%</th>
<th>Na₂O%</th>
<th>K₂O%</th>
<th>P₂O₅%</th>
<th>SO₄%</th>
<th>CL%</th>
<th>LiO₃%</th>
</tr>
</thead>
<tbody>
<tr>
<td>56.77</td>
<td>15.67</td>
<td>5.12</td>
<td>4.48</td>
<td>3.42</td>
<td>1.11</td>
<td>0.60</td>
<td>0.65</td>
<td>0.59</td>
<td>0.57</td>
<td>9.49</td>
</tr>
</tbody>
</table>

PROCEDURE

Extrusion procedure

(PP, PMMA) granules and (Bentonite powder) were dried in an air circulating oven type (Hs.31A, Germany) at 70°C for 4 hours. Weighed amount of material were mechanically mixed, at various composition were prepared (PP/PMMA) (80/20) %wt blend and (0, 3, 6, 9, 12, 15) %wt concentration filler (Bentonite) of different particle size (45, 60, 70, 110) µm were prepared. This mixture was then fed into 25 mm single screw extruder. The barrel temperature was monitored and controlled by thermostats. The die temperature was also controlled by a thermostat and was adjusted together with barrel temperature yield uniform output. Feed, compression and metering zone temperature listed in table (3).

The extrudates produced in the form about of (1.5-2) mm diameter sheet were cooled in water. The composite sheet was cut and put in the mold.

Table (3) Extrusion Parameter [10].

<table>
<thead>
<tr>
<th>polymer</th>
<th>Temperature C°</th>
<th>Screw speed r.p.m</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zone 1 Zone 2 Zone 3 Zone 4</td>
<td></td>
</tr>
<tr>
<td>PP/PMMA</td>
<td>190°C 220°C 240°C 220°C</td>
<td>20</td>
</tr>
<tr>
<td>PP/PMMA/BN</td>
<td>190°C 220°C 240°C 220°C</td>
<td>20</td>
</tr>
</tbody>
</table>

Molding process

The mold was prepared, wrapped with glycerin solution. (Three to four mm) thick sheet was prepared by hot pressing the sheet between hydraulic press at 180°C for (PP/PMMA/Bentonite). A pressure of 20 kg/cm² was applied for 5 minutes to allow the composite to melt and spread out between plates. Pressure was then increased to 200 kg/cm² for further 5 minutes. The compression molding parameters are listed in table [4]. The pressure was removed and the mold sheet was quenched in water at room temperature.
Table (4) Compression Molding Parameters.

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>Upper Plate</th>
<th>Lower Plate</th>
<th>Pressure (kg/cm²)</th>
<th>Time (minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>180°C</td>
<td>180°C</td>
<td>20</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>180°C</td>
<td>180°C</td>
<td>200</td>
<td>5</td>
<td></td>
</tr>
</tbody>
</table>

PHYSICAL PROPERTIES
Diffusion and permeability of the prepared polymeric blends
The prepared samples (composite different weight and different particle size) were weighted by using digital balance (Sartorius – Germany). The weight gave the value m₁ all the specimens were immersed in acid (H₂SO₄ solution) with concentration of (1N), (28ml H₂SO₄/1L H₂O). after that the specimens were placed in a water bath (electro – MAGAS) Ykitelli O.S.B, Demircilersitesi (B7 BLOK NO: 153) (M96K) at different temperature (30°C, 45°C, 60°C).the samples were reweighed after the immersion. The samples in water bath and this gave the value m₂ the process was repeated for several weeks and the percentage of absorption was calculated by using the below equation:

Absorption % = \frac{M₂ - M₁}{M₂} \times 100 \quad \text{….. (1)}

Then from Fick’s second law, diffusion coefficient can be determined by using equation

\[ D = \frac{K \times b}{2} \quad \text{….. (2)} \]

Where K is the slope of the linear portion of the plot of Mt versus t1/2, Mt (change of weight with time). b is thickness of specimen [24]. E is the activation of diffusion process. E can be calculated from relationship between Ln D (Diffusion coefficient) with 1/temperatures. Slope of straight line of this relation equals E/R. R is the gas constant. Temp at (30, 45, and 60).

Morphology tests
Suited plates were chosen of microscope examination, they were put on a glass holder, were covered with a glass slide, and were watched at 100 fdd magnification.

MECHANICAL PROPERTIES
Hardness Tests
Shore a hardness was measured using ZWICK hardness machine available in National Company for Chemical and Plastic Industries. Tester type 7901, Test was carried out according to DIN 53505, ASTM (D1706 – 61) and (ISO DR 988)[12].

Tensile tests
Tensile properties were obtained using an Instron tensile tester machine (model 1445) on dumbbell – shaped die – cut sample according to ASTM (D638)[13].
Specimens were cut and their thickness was determined with a micrometer having resolution of 0.025 mm (0.001 inch). Available in National Company for Chemical and Plastic Industries.

**Elongation**

Elongation was measured by using ZWICK machine. The measurement was carried out according to the test specification of ASTM (D638) available in National Company for Chemical and Plastic Industries.

**X – Ray measurement**

X- Ray analysis of the samples was done on Phillips X- Ray diffractmeter (Model pw1840) Intensity value was recorded between $10^\circ$ – $70^\circ$, 2$\phi$ angle, scan speed 3 degree / minute. Wide – angle X- Ray Diffraction (XRD) was used to examine the extent of dispersion of clay (Bentonite) in filled sample. XRD patterns were obtained on the SCINTAG pad V theta - 2$\phi$ Goniometer with SI (LI) PELTIER detector, using CUKA radiation with wave length $\lambda = 1.54$ angstroms. Typical power setting is 20mA and 40 KV. The scan rage is 2 – 30 Degree with scan rate of 3$^\circ$/minute.

**Thermal properties**

**Thermal analysis**

Thermal analysis of samples before and after reinforcement was done on ZWICH thermal analyzer (Z405 ZWICK), (DIN 16927) specified polymer sheet duration of 60 minutes and 4 mm thickness

**RESULTS AND DISCUSSIONS**

**Physical properties:**

**Morphology:**

The microstructures of (PP/PMMA) (80/20) wt% blend are showed in Fig. (1). PMMA, which is a brittle polymer, was presented as a second phase in (PP) matrix. (PP) acts as a matrix and is of white color in the micrograph spread all over the surface as spherulite and (PMMA) was in discrete from a block portion. (PMMA) was reported to be in the form of small balls distributed evenly in the (PP) matrix. This system shows the morphology of incompatible blend. Both the phases are observed in the micrograph. Our result, is in good agreement with result obtained by Hashmi, Frank and Navin Chand.

Figure (2) and (3) show optical surface microstructure of 15% (75gm) of Bentonite at (70 and 45 µm particle size) filled (PP/PMMA) (80/20) wt%. (PP/PMMA) blend were an incompatible blend. PMMA was found evenly distributed in the form of small balls / globules in the PP matrix. Shape of PMMA globules were not uniform and the size in the range (2-30) µm.

It can be seen that globules of PMMA were poorly bonded with the matrix phase (PP) during fracture of blend. It has been observed the PMMA present in the form of globules which comes out and creates cavities and fracture in brittle manner. (PP/PMMA) blend fractured surface has river pattern type morphology. Due to a large difference in melting point of PP and PMMA and lack of co-crystallization owing to difference in the molecular structure, both the polymers showed incompatibility in the solid state. It was clear that, the filler (Bentonite, yellow portion) filled the cavities which were present in PP/PMMA blend. Also it shows that the filler with particular size 45µm (15% of Ben.) filled these cavities more than filler with particular size 70µm (15% of Ben.). Our results are in good agreement...
with the results obtained by NAVIN CHAND [1]. Other samples show the same behavior (for example 3%).

**Figure (1) Optical Surface Microstructure Of PP/PMMA (80/20) Wt % Blend.**

**Figure (2) Optical Surface Microstructure of 15% Ben. (70µm particle size).**

**Figure (3) Optical Surface Microstructure of 15% Ben. (45µm particle size).**

**Diffusion coefficient for H₂SO₄:**

Figurs. (4 to 10) show the relation between the weight gain with the square root of time of exposure for filled PP/PMMA unfilled with different amount of Bentonite (3-15) wt% at different particle size (45-110) µm, at three temperatures (30, 45 and 60) °C. It is clearly seen that the behavior of the tested samples fills obeys the Fickian law. The weight gain of these samples increases with the increase in time of exposure until it reaches a maximum value after which it will decrease. The diffusion coefficients for the above samples were calculated using equation (D= \(\pi(kb/4m)^{1/2}/60\), k was obtained from the slope of the straight lines in figs. (4 to 10). It is clearly shown that the calculated values of (D) decrease as the amount of filler increases Table (5).
Table (5) Diffusion coefficient calculated for PP/PMMA unfilled/filled with different filler content and particle size.

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<thead>
<tr>
<th>Filler content</th>
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<tr>
<td>45 µm particle size of BN</td>
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<td>3%</td>
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<td>PP/PMMA</td>
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<table>
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<th>110 µm particle size of BN</th>
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<th>110 µm particle size of BN</th>
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<tbody>
<tr>
<td>3%</td>
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<td>6%</td>
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<td>PP/PMMA</td>
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The diffusion coefficient value decreases, this is due to the fact that as the filler increases the local stress on the sample increases. Thus the permeability of acid inside the sample will be difficult [14]. In this work it is also clear that as the particle size of the filler increases the diffusion coefficient obtained increases. This may be due to the fact that as the particle size increases, the surface area increases, thus pore size and interspaces’ between pores will increase; therefore the absorption will be increased [15].
Figure (4) Relation between weight gain % and time$^{1/2}$ of PP/PMMA/unfilled/filled with different weight and particle size of BN at 110 µm at $t = 30^\circ$C.

Figure (5) Relation between weight gain % versus time$^{1/2}$ of PP/PMMA/unfilled/filled with different weights and particle sizes of BN at 110 µm at $t = 45^\circ$C.
Figure (6) Relation between weight gain % and time\(^{1/2}\) of PP/PMMA/unfilled/filled with different weights and particle sizes of BN at 110 µm at \(t = 60^\circ C\).

Figure (7) Relation between weight gain % and time\(^{1/2}\) of PP/PMMA/unfilled/filled with different weights and particle sizes of BN at 45 µm at \(t = 30^\circ C\).
Figure (8) Relation between weight gain % and time² of PP/PMMA/unfilled/filled with different weights and particle sizes of BN at 45 µm at t = 45°C.

Figure (9) Relation between weight gain % and time² of PP/PMMA/unfilled/filled with different weights and particle sizes of BN at 45 µm at t = 60°C.
A Study of the Effect of Iraqi Bentonite on Some Properties of Polymeric Blend

X – RAY MEASUREMENT

Figures (10) and (11) illustrate the x-ray diffraction pattern of (PP/PMMA) blend unfilled/filled with (45µm, 15% Bentonite). The characteristic intensity peaks of (PP) are recorded at (10.0, 13.4, 18.1 and 20.6) 2θ values and the calculated d-spacing of (8.799, 6.58, 4.88 and 4.28) angstrom. Diffraction patterns of (PP/PMMA) filled with (45µm, 15% Bentonite) exhibit all reflection of (PP/PMMA) blend/along with the addition of Bentonite peaks at (26.3, 36.3, 42.0 and 42.4) 2θ angle value and the calculated d-spacing of (3.38, 2.466, 2.144 and 2.126) angstrom as shown in Fig. (11). It can be observed from these results that the particles Bentonite modified the intensities of peaks of the crystalline PP polymer present in the matrix. Peak intensity for 2θ angle at (13.4, 18.1 and 20.6) reduced to 3647 from 4843, 2862 from 4745 and 3156 from 4705 respectively. This may be explained by the fact that Bentonite particles adversely affect the crystallization of PP by increasing the discontinuity and restrict the growth of crystal and therefore crystalline peak intensity reduced in the field blend composite, this decrease in crystalline of PP due to the presence of PMMA at the time of crystallization of PP reduces the strength of PP. The results agree with result obtained by Hashmi[2], NAVIN CHAND[3]. Figs. (11) and (12) illustrate the x-ray diffraction pattern of (PP/PMMA/ 45µm Bentonite) of (80/20/15) wt% and (PP/PMMA/70µm Bentonite) of (80/20/15) wt%. The characteristic intensity peaks of PP are recorded at (10.8, 11.7, 12.2, 13.9, 15.7, 16.4, 18.2, 19.6 and 21.2) 2θ values and the calculated d-spacing of (8.1, 7.5, 7.2, 6.3, 5.6, 4.8, 4.5 and 4.1) angstrom as shown in Fig. (12). The peak intensities for PP at (13.7, 16.4, 18.2 and 21.2)20 values are reduced to 3352 from 3647, 2725 from 3901, 2529 from 2862 and 3117 from 3588. This may be explained by the fact the addition of (70 micron of 15wt% Bentonite) adversely affects the crystallization of PP than (45 micron of 15wt% Bentonite) by increasing
the discontinuity face and restricting the growth of crystal in the blend composite. This result shows that large particle size of filler is better than small to reduce the crystallization of PP. this result agrees with results obtained by Navin$^{[1]}$, Hashm$^{[2]}$.

**MECHANICAL PROPERTIES**

**Hardness**

Figure (13) shows the effect of weight fraction and particle size before and after reinforced (Bentonite) addition on shore hardness. It was clearly seen that the increase weight fraction of the filler increase in hardness and the hardness increases with decreasing particle size. The addition of Bentonite filler increases the hardness of composite material due to increase in the resistance strength of polymer to plastic deformation$^{[6, 3]}$. This result agree well with results obtained by Abed Al-Hakem$^{[3]}$, Mustafah$^{[6]}$.
Tensile strength
Elastic modules

It is clearly seen that the modulus of elasticity increases with increasing weight fraction of filler and decreases with increase particle size of filler, this may be attributed to the fact the Young’s modulus of composite depends on the Young modulus of the weight fraction and particle size of filler. These results agree well with results obtained by Sumita[17], Mustafa[6], Mohammed[20]. They found that the Young’s modulus of composite increases with increases filler content and decreases with filler size. This variation in Young’s modulus in both cases indicates good dispersion of smaller particle size of the filler than of larger particle size at lower filler loading. The smaller particle size filler agglomerate hence reduces the strain value restriction of mobility of matrix chains. The mobility of matrix chains and agglomeration of smaller particle size filler are clearly indicated by the increase in Young’s modulus with higher rate than that of large particle size of filler[19]. The value of elastic modulus increases because the Bentonite material is a brittle materials (ceramic material) therefore, it decreases the strain rate and increases the value of the matrix, this leads to increase the elastic modulus according to Hooke’s Law. When the weight fraction increases in the polymer blend then that leads to agglomeration, consequently, the distribution of tensile stress will be irregular[21, 25]. The results agree well with results obtained by Ahmed[21], Abed Al-Hakem[3].

Elongation

It is clearly seen that the elongation increases with increasing of the applied stress before reinforcement. After reinforcement this depends on the type of matrix material, the elongation decreases; at the same applied force; when the weight fraction of filler increases and the elongation will increase when the particle size of filler increases because the elongation of polymeric composite depends on the elongation of filler, polymer, weight fraction and particle size[18]. These results agree well with the results.
Thermal properties

Thermal stability

Figur (18) and Fig. (19) show residual weight percent changes with respect to temperature for different contents of filler (0, 3, 6, 9, 12, and 15) wt% and different particle size of filler (45 and 110) µm. an increase in filler content leads to increase in thermal stability of the composite. It’s clear that the composite material of PP/PMMA/45µm Bentonite has higher thermal stability than that of composite material PP/PMMA/110µm Bentonite because smaller particle size increases interstitials volume or increases matrix filler surface contact. Also a significant increase in heat temperature was expected because inorganic filler have high thermal stability [23].

These results agree well with results obtained by Hashmi, he showed that increase in red mud content raises the thermal stability of PP/PC blend[2]. Also these results agree with results obtained by NAVIN CHAND [1]. Obtained by Hassan[22].

![Figure (15) Effect of addition of 110 µm of BN filler on thermal stability of PP/PMMA blend.](image-url)
CONCLUSIONS
At the end of this study about polymeric reinforced with mineral clay some conclusions are drawn:
The mineral clay (Bentonite) was found to improve the modulus of elasticity and hardness. The modulus of elasticity and hardness increase with increasing wt% and decrease in particle size for PP/PMMA blend after reinforcement with Bentonite filler.
The elongation of unfilled (PP/PMMA) blend is large. After addition of different amounts of Bentonite filler, the elongation is decreased with increase in filler content and decreases with particle size decrease.
Bentonite particles of PP/PMMA blend increase the discontinuity and restrict the growth of crystal and therefore crystalline peak intensity is reduced in filler blend composite. Also large particle sizes of Bentonite adversely affect the crystallization of PP. The thermal stability of PP/PMMA blend increases with the addition of Bentonite and the thermal stability of small particle size is higher with large particle size.
The diffusion coefficient (D) is increased with temperature and decreases with filler content increase, also activation energy (E) increases with filler content (BN) increase and (E) increases.
REFRENCES