EFFECT OF KAOLIN ON THE MECHANICAL PROPERTIES OF POLYPROPYLENE/ POLYETHYLENE COMPOSITE MATERIAL

Samir Nassaf Mustafa
Technical Institute – Baquba
E-mail: m.eng_samir@yahoo.com
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ABSTRACT :- In this work, the mechanical and water absorption properties of polymer blend polypropylene (PP) and low density polyethylene (LDPE) at constant ratio (50/50) composites as a function of kaolin powder weight content in the grain size (100,120 µm) were investigated. Polymer composite was fabricated by mixing polymer blend (PP:LDPE) with (0, 1, 3, 5, 10, 14) wt.% of kaolin powder to obtain desirable properties. The parameters such as tensile strength, tensile modulus, elongation at break, yield stress, impact strength, shore D hardness and water absorption test were carried out on the prepared samples. The results showed that the addition of kaolin powder to the polymer leads to increase the tensile strength, modulus of elasticity, shore-D hardness and impact strength and it decreases the % elongation at break,. Water absorption of the composites behaves as function of time (days) has also been investigated, and it increases by increasing immersion time for the same filler content, while the absorbed amount of water increases, by increasing the wt% of kaolin at constant immersion time.

Keywords: mechanical properties. polypropylene, low density polyethylene, kaolin, blend, filler.

INTRODUCTION

Polypropylene was proved as one of the most interesting commodity thermoplastics in the last years. Since polypropylene (PP), are widely used in many scientific, industrial and daily applications since they are versatile and low cost materials [1].

Low-density polyethylene (LDPE) possesses good mechanical properties, good resistance to chemicals, and good process ability and has a wide range of applications, including the
insulation of wire and cable. However, it has some disadvantages, such as a low melting temperature, low thermal stability and high flammability [2].

Blending of polymers is often an important route for obtaining new and improved polymeric materials, which are difficult to obtain by direct polymerization process [3]. Blending is an attractive method of creating new materials with improvement and flexibility in performance, and better properties than existing polymers [4].

Fillers are used along with various commodity as well as engineering polymers to improve the properties and reduce the cost. Incorporating inorganic mineral fillers into plastic resin improves various physical properties of the materials such as mechanical strength, modulus etc. In general the mechanical properties of particulate filled polymer composites depend strongly on size, shape and distribution of filler particles in the polymer matrix and extend of interfacial adhesion between filler and matrix [1,5]. As fillers, mica, kaolin, calcium carbonate, and talc are the most often used to reduce both the production costs and to improve the properties of the thermoplastics, such as rigidity, strength, hardness, flexural modulus, dimensional stability, crystallinity, electrical and thermal conductivity. The fillers affect ultimate mechanical properties in two ways: (i) they act directly as harder particles with determined prop properties (shape, size, and modulus) and (ii) they affect crystallization processes in polymer matrix and ultimate super molecular structure of semi crystalline polymer [6–8].

A composite is a heterogeneous material that consists of two or more phases that are insoluble in each other. At least one phase is mostly inorganic as dispersed phase. Composites are designed to have mechanical properties and performances superior than constituent materials have independently. They may have unique advantages over monolithic materials such as high strength, high stiffness, low density, high corrosion, long fatigue life, high thermal stability, insulation and conduction. Besides that they have an important advantage that is reduction in life cycle cost [9]. Mixing of two or more polymers at different chemical composition is an effective way of combining performance and economic relationships using existing materials. Fundamental issues that affect the properties of blends include interfacial behavior and equilibrium phase, physical and chemical interactions between the components, phase morphology and rheology, all of which are related to the issues of compatibility. One of the most important way of polymer blending is the incorporation of an elastomeric phase in a rigid matrix to enhance mechanical toughness [10].

Although, in many respects, polypropylene is similar to polyethylene, since both are saturated hydrocarbon polymers, they differ in some significant properties. Isotactic
polypropylene is harder and has a higher softening point than polyethylene, so it is used where higher stiffness materials are required. Polypropylene is less resistant to degradation, particularly high-temperature oxidation, than polyethylene, but has better environmental stress cracking resistance [11]. Polypropylene PP and low density polyethylene (LDPE) blends produce immiscible form because the low interfacial adhesion [12]. Flexibility of LDPE similar to rubbery phase leading to improved the impact resistance of PP because of its ability to absorb impact energy [13].

Navin, and Vashishtha, studied development, structure and strength properties of PP/PMMA/FA blend. Addition of fly ash imparted dimensional and thermal stability, which has 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 increased mechanical interlocking of PP/PMMA chains inside the hollow structure of fly ash [14].

Da Silva A.L.N., et al, studied mechanical and rheological properties of composites based on polyolefin and mineral additives. The extensive range of fillers and reinforcing agents used nowadays indicates the major significance that these materials have attained. Although their original purpose was to lower the cost of the molding compounds; prime importance is now attached to selective modification of the properties of a specific plastic. In this paper results of rheological and mechanical analysis on polypropylene composites prepared with talc and CaCO₃ are reported. The previous results showed that the addition of fillers provoked an increase in modulus of elasticity, stress at break and yield stress. It was also verified that melt viscosity increases with the addition of reinforcements [5]. Unal H., studied morphology and mechanical properties of composites based on polyamide 6 and mineral additives. The results showed that the tensile strength and modulus of elasticity of polyamide 6 composite linearly increased with the increase in filler weight ratio while the impact strength and maximum elongation linearly decreased with the increase in filler weight ratio. The overall maximum improvement in mechanical properties was recorded with the addition of 10–20 wt% filler ratios [15]. This Y.S., et al, studied Toughening of isotactic polypropylene with CaCO₃ particles. Three types of particles with average diameters of 0.07, 0.7μm and 3.5μm were used at filler volume fraction from 0.05 to 0.30. The experiments included show tensile test, notched Izod impact test with varying notched depths and fracture resistance tests using double-cantilever-beam sample configurations. In slow tension addition of fillers increased the modulus and decreased the yield stress independently of filler type. The strain at break increased with initial incorporation of fillers but decreased at higher loading. The 0.7μm
diameter particles improved Izod impact energy up to four times that of the unfilled matrix. The other particles had either adverse or no the impact toughness. The toughening mechanisms at work were plastic deformation of inter particle ligaments following particle-matrix debonding with additional contribution coming from crack deflection toughening. The failure of the 0.07 and 3.5 µm diameter particles to toughen the matrix was attributed to poor dispersion[1]. Zebarjad S.M., et al, studied Influence of filler particles on deformation and fracture mechanism of isotactic polypropylene. The results of tensile tests show that the addition of CaCO₃ to PP decreases yield strength and elongation at break while causes to promote modulus. Optical microscopy taken from the polished surfaces of tensile test illustrates presence of the damage zones in all specimens. Looking at in more details via scanning electron microscope (SEM) show the damage zone included some crazes and some voids owing to poor adhesion between filler and matrix. Briefly the dominant mechanism of deformation in PP/CaCO₃, observed in this work, appears to be a craze type of damage, propagating via repeated cavitation. The results of three-point bending tests show that the fracture toughness of PP decreases with increasing of CaCO3 content. The results of four-point bending tests display that a wide spread damage zone in front of the pre-crack while SEM photographs appears that the damage zone including craze which is followed by repeated cavitation and debonding[16]. Akinci A., studied mechanical and morphological properties of basalt filled polymer matrix composites. In present study, it was found that, the content of basalt filler affected structural integrity and mechanical properties of composites. With increasing the amount of the basalt addition to the LDPE results in a decrease in elongation at break values[17]. Dr. Najat j. Saleh Development and study properties of PP/PMMA /Bentonite Blend Addition of filler increases the young modulus and tensile strength at break [18]. Salmah H., et al, studied compatibilisation of polypropylene/ethylene propylene diene terpolymer/kaolin composites: the effect of maleic anhydride-grafted-polypropylene. Results show that incorporation of MAPP increased the tensile strength and Young’s modulus, but reduced the elongation at break. Scanning electron microscopy (SEM) of the tensile fracture surfaces of composites indicates that the MAPP improved the interfacial interaction between kaolin and PP/EPDM matrix[19].

The aim of this work is to developed a new type of PP/PE blends of filled with local Iraqi Kaolin as filler and determining mechanical and physical properties of filled and unfilled PP/PE blends and also study the effect of particle size of filler on the mechanical of blend. In the present work, the mechanical and water absorption properties of PP/PE blends have been investigated. Also samples of different particle sizes (100 and 120) µm of filler (locally) were
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prepared. The effect of change in the percentage of kaolin on mechanical, and water absorption properties was quantitatively analyzed.

EXPERIMENTAL WORK:

Materials:
The PP and PE used for this study were supplied by Sabic Saudi Arabia. The Iraqi kaolin clay and the kaolin that was used in this work produce from Dawakhla location by Iraqi National Company for Geological Survey and Refinery and has a chemical composition and general properties shown in the table (3) and (4) respectively.

METHODS:

PP, PE and kaolin were dried in an air circulating oven at 60°C for 4 hr. Weighed amounts of materials were mechanically mixed, various compositions were prepared of (PP/PE) blend and kaolin table (5), and prepared. Also samples of two particle sizes (100, 120) µm of filler (kaolin). The samples of composite material then fed into 16 mm a L/D ratio of 25 single screw extruder. The barrel temperature was monitored and controlled by thermostat. The die temperature was also controlled by a thermostat and was adjusted, together with barrel temperature to yield uniform output. The temperature are measured at different zone across the extruder. Table (6) shows the temperature profiles of the extruder. The extruder produced 2 mm diameter monofilament which was cooled in air. The monofilament produced by using a screw speed of (35 r.p.m.) was uniform and opaque; it was cut in the form of granules of (3-4)mm length. The sheet was prepared by hot pressing the granules between hydraulic press at 180°C for PP/PE/Kaolin. A pressure of 20 kg/cm² was applied for 15 min. to allow the composite to melt and spread out between plates. Pressure was then increased to 200 kg/cm² for further 10 min. After the pressure was removed the mold sheet was quenched in water at constant temperature.

MATERIALS CHARACTERIZATION:

1. Tensile Testing:

Tensile test for composites was conducted according to ASTM D 638 - 99, with a Universal Testing Machine (Zwick Co.). Tensile test was carried out at crosshead speed of 50mm/min at room temperature.
2. Izod Impact Testing:

In order to measure the work of fracture (WOF), the Izod impact test was carried out. Impact bars were obtained by cutting specimens in rectangular shapes. These rectangular specimens are of thickness 3 mm, width 12 mm and length 62 mm according to ASTM D 256. The test was carried out with impact energy of 5 J and a span length of 60 mm at room temperature. The average value of notched Izod impact energy was obtained from each group of three specimens.

3. Hardness Testing:

Hardness properties were investigated by a Shore Durometer in Shore D scale at room temperature according to ASTM D 2240. Three specimens of each formulation were tested and the average values were reported.

4. Water Absorption:

A water absorption test was carried out according to ASTM standard D750-95. It involved the total immersion of three samples in distilled water at room temperature. All of the specimens were previously dried in an oven at 50°C for 24 h and stored in a desiccator. The water absorption was determined by weighing the samples at regular intervals. A Mettler balance type AJ150 was used with a precision of ±1 mg. The percentage of water absorption (Mt), was calculated by:

$$Mt = \frac{W_N - W_d}{W_d} \times 100\%$$

where $W_d$ and $W_N$ are the dry and wet weights after exposure, respectively. The average reading of three samples was taken.

RESULTS AND DISCUSSION:

1 Tensile strength:

Figure 4 shows the effect of filler contents, and particle sizes on the tensile strengths of unfilled, and filled polypropylene/Low density polyethylene. The tensile strength of polypropylene/Low density polyethylene composites was observed to increase with increases in kaolin powder content and decrease particle size. From Figure (4), it is clear that the smaller the particle size of kaolin powder, the higher the tensile strength of the polypropylene/Low density polyethylene composite any kaolin powder. The better dispersion
and filler-matrix interaction may be the main reasons or factors responsible for the observed trend. Similar observations have been reported by [22], and [23] for other filled systems.

2. Elongation at break:

Figure 5 shows that the elongation at break for kaolin powder polypropylene/Low density polyethylene composites decreases with increases in filler content at any given filler particle size considered. The elongation at break for kaolin powder polypropylene/Low density polyethylene composites high decrease with increases in kaolin powder content and the decreasing at (100 µm) particle size is higher than at (120 µm) particle size when the same amount of kaolin added. Fillers can be considered as structural elements embedded in the polymer matrix, and at the concentrations of the filler used (0 - 14 wt%), the contents might not be high enough to significantly restrain the polypropylene/Low density polyethylene molecules. Consequently, highly localized strains might have occurred at the concentrations investigated, causing dewetting between polypropylene/Low density polyethylene and the filler, and thus, leaving essentially a matrix that is not ductile. Such a reduction in elongation at break of a composite with increases in filler content, irrespective of filler particle size has been reported by [24].

3. Tensile elastic modulus:

As shown in figures 6 illustrates the effects of kaolin powder content, and particle size on the tensile elastic modulus of prepared polypropylene/Low density polyethylene composites. Like was observed on the effect of filler content and particle size on the tensile strength of filled polypropylene/Low density polyethylene, the tensile elastic modulus of the composites increased with the increases of filler content and decrease particle size. This observation highlights the fact that the incorporation of fillers into polymer matrix improves the stiffness of the composites [25].

4. Yield stress:

Yield stress, measured at large deformations, is much more dependent on interfacial adhesion with respect to Young's modulus, measured at small deformations. Tensile yield stress is proved to be an excellent property to correlate with interfacial interactions in heterogeneous polymer systems [26,27]. In figure 7, the dependence of yield stress of the polypropylene/Low density polyethylene / kaolin composites against kaolin content is
presented. The addition of different kaolin fillers in the polypropylene/Low density polyethylene matrix results in decreasing of the yield stress values in comparison to the neat polypropylene/Low density polyethylene matrix. The incorporation of kaolin fillers into the polypropylene/Low density polyethylene matrix results in higher yield stresses with kaolin powder (120 µm) particle size.

5. Impact Strength:

The impact strength of polypropylene/Low density polyethylene composites of kaolin powder at a particular filler particle size was observed to increase with increases in kaolin powder content as shown in figure 8. The increase in impact strength of the prepared composites was very remarkable for kaolin powder composites of polypropylene/Low density polyethylene. This remarkable performance indicates that kaolin powder was more effective in distributing the applied stress over a large volume at the base of the notch, and helped to prevent propagation of cracks by carrying large part of the load in the area under the crack. The increase in impact strength of a polymer composite with increase in filler content has been reported in the literature [22]. The impact strength of the prepared composites for particular filler and at a given filler content was observed to decrease with increase in filler particle size. Thus, increasing the particle size of kaolin powder at a given filler content probably increased the level of stress concentration in the composites with the resultant decrease in impact strength.

6. Shore-D Hardness:

At a given filler particle size, the Shore-D hardness of polypropylene/Low density polyethylene composites was observed to increase with increase in the amount of filler incorporated into polypropylene/Low density polyethylene figure 9. This result indicates enhancement of abrasion and impact strength of the composites. Generally, the hardness of the composites could be observed to decrease with increase in the particle size of the filler at a given filler content. Such a decrease in the hardness of polypropylene/Low density polyethylene composites with increases in filler particle size was reported by [28].
7. Water absorption:

As shown in figures 10 and 11, respectively. The water absorption of polypropylene/Low density polyethylene / kaolin composites increased continuously day by day and also showed a marked dose dependent effect with increasing water absorption at each time point with increasing kaolin in the composites. Normally, polypropylene and Low density polyethylene do not have good water absorption (< 0.02%). Kaolin was not considered as a hydrophilic material, but the significantly increased water absorption of polypropylene/Low density polyethylene / kaolin composites was likely to be attributed to the many pores and gaps in the kaolin structure. the formation of agglomerations increases due to the difficulties of achieving a homogeneous dispersion of filler at high filler content. The agglomeration of the filler in composites increases the water absorption of the composites. The composite filled with small size filler showed higher water absorption properties due to the presence of higher kaolin surface area. The figures also show that the ratio of water absorption by grain size (100 µm) was higher than for grain size (120 µm) for all fillers content, because of the surface area. The figure 12 show that the ratio of water absorption by grain size (100 µm) was higher than for grain size (120 µm) for 14% fillers content, because of the surface area.

CONCLUSIONS

The addition of kaolin filler has resulted in some improvement in the mechanical properties of polymer blend (polypropylene (PP) and low density polyethylene (LDPE)). The tensile strength, tensile modulus and shore-D hardness and impact strength of (PP/LDPE/kaolin powder) composite were slightly increase with the incorporation of the filler. Elongation at break decrease gradually with increasing filler content. Water absorption of the composites behaviors as function of time (days).

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Table (1): General properties of the Isotactic polypropylene.

<table>
<thead>
<tr>
<th>property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>0.908</td>
</tr>
<tr>
<td>Melt flow index (MFI) g/10 min</td>
<td>11</td>
</tr>
<tr>
<td>Tg °C</td>
<td>0 &lt;</td>
</tr>
<tr>
<td>Tm °C</td>
<td>170</td>
</tr>
<tr>
<td>Degree of crystalline %</td>
<td>50 - 70</td>
</tr>
</tbody>
</table>

Table (2): General properties of the Low-density polyethylene (LDPE).

<table>
<thead>
<tr>
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<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>0.9235</td>
</tr>
<tr>
<td>Melt flow index (MFI) g/10 min</td>
<td>0.18</td>
</tr>
<tr>
<td>Tg °C</td>
<td>~ -120 °C</td>
</tr>
<tr>
<td>Tm °C</td>
<td>108 - 115</td>
</tr>
<tr>
<td>Degree of crystalline %</td>
<td>40 - 50</td>
</tr>
</tbody>
</table>

Table (3): Chemical composition of kaolin clay.

<table>
<thead>
<tr>
<th>Sio₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>TiO₂</th>
<th>MgO</th>
<th>Na₂O</th>
<th>CaO</th>
<th>L.O.I</th>
</tr>
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<tbody>
<tr>
<td>52.48</td>
<td>31.31</td>
<td>2.094</td>
<td>1.43</td>
<td>0.33</td>
<td>0.28</td>
<td>0.462</td>
<td>10.93</td>
</tr>
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Table (4): General properties of Kaolin.

<table>
<thead>
<tr>
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<th>Quantity</th>
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</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>2.64</td>
</tr>
<tr>
<td>Powder color</td>
<td>white</td>
</tr>
<tr>
<td>Melting point °C</td>
<td>1755</td>
</tr>
<tr>
<td>Resistance fracture</td>
<td>high225</td>
</tr>
<tr>
<td>Thermal properties</td>
<td>Endothermic at 260 °C</td>
</tr>
<tr>
<td></td>
<td>Isothermic at 980 °C</td>
</tr>
</tbody>
</table>
Table (5): Samples prepared of composite material.

<table>
<thead>
<tr>
<th>No.</th>
<th>materials</th>
<th>% PP</th>
<th>% PE</th>
<th>% Kaolin</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>50</td>
<td>50</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>49.5</td>
<td>49.5</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>48.5</td>
<td>48.5</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>47.5</td>
<td>47.5</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>45</td>
<td>45</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>43</td>
<td>43</td>
<td>14</td>
<td></td>
</tr>
</tbody>
</table>

Table (6): Temperature profiles of the single screw extruder.

<table>
<thead>
<tr>
<th>Blend</th>
<th>Temperature ºC</th>
<th>Screw speed (r.p.m)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zone₁</td>
<td>Zone₂</td>
</tr>
<tr>
<td>PP:PE</td>
<td>145</td>
<td>165</td>
</tr>
<tr>
<td>PP:PE \ kaolin</td>
<td>145</td>
<td>165</td>
</tr>
</tbody>
</table>

\[
\text{CH}_3 \quad \text{CH}_2=\text{CH} \quad \text{CH}_3
\]

Fig. (1): Polymerization of polypropylene [20].

\[
\text{n CH}_2=\text{CH}_2 \quad 200^\circ\text{C} \quad \text{20,000 - 35,000 psi} \quad \text{200}^\circ\text{C} \quad \text{20,000 - 35,000 psi} \quad \text{organic peroxide present} \quad \text{organic peroxide present}
\]

Fig. (2): Polymerization of PE [20].

\[
\text{H}_2\text{O} \quad \text{H}_2\text{O}
\]

Fig. (3): Structure of Kaolin[21].
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Fig.(4): The relation between the tensile strength of PP/LDPE with weight percent of kaolin filler for different particles size.

Fig.(5): The relation between the % Elongation at break of PP/LDPE with weight percent of kaolin filler for different particles size.

Fig.(6): The relation between the tensile modulus of PP/LDPE with weight percent of kaolin filler for different particles size.
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Fig.(7): The relation between the yield stress of PP/LDPE with weight percent of kaolin filler for different particles size.

Fig.(8): The relation between the Impact strength of PP/LDPE with weight percent of kaolin filler for different particles size.

Fig.(9): The relation between the Shore D hardness of PP/LDPE with weight percent of kaolin filler for different particles size.
Fig. (10): shows the relation between the % water absorption of PP/LDPE with time at specific value of kaolin filler for 120 µm.

Fig. (11): shows the relation between the % water absorption of PP/LDPE with time at specific value of kaolin filler for 120 µm.

Fig. (12): shows the relation between the % water absorption of PP/LDPE with time at 14% specific value of kaolin filler for 100 µm and 120 µm.
تأثير الكاولين على الخواص الميكانيكية لمادة متراكبة لملحوظ بوليمر من البولي بروبليين والبولي اثيلين

سمير نصيف مصطفى
مدير مساعد
المعهد التقني - بعقوبة

الخلاص:
تم في هذا البحث دراسة الخواص الميكانيكية وامتصاصية الماء لملحوظ بوليمر يتكون من البولي بروبليين والبولي اثيلين الواطئ الكثافة ونسبة خلط (50/50) كدالة للنسبة الوزنية لمسحوق الكاولين. تم تحضير المتراكبات بزال المخلوط البوليمر مع نسب وزنية مختلفة (% wt. 14, 10, 5, 3, 1, 0) من مسحوق الكاولين للحصول على أفضل الخصائص. تم دراسة السلوك الميكانيكي من خلال الفحوصات الالتحافية (فحص الشد وفحص الضغط وفحص الصالحة وقياس معامل المرونة وقياس الاستطالة عند الكسر) أظهرت النتائج العملية أن اضافة مسحوق الكاولين يؤدي إلى زيادة قوة الشد والقوة الصدمة والصالحة وقلل اجهاد الخضوع وقلل نسبة الاستطالة عند الكسر، كما تم دراسة سلوك امتصاصية الماء كدالة للزمن للمتراكبات المحمضة وتبين بأن كمية الماء الممتصة تزداد بزيادة زمن الغمر بالماء عند ثبوت النسبة الوزنية للكاولين. وكذلك فإنها تزداد بزيادة النسبة الوزنية لمسحوق الكاولين مع ثبات الزمن.