Effect of Marble Powder and Dolomite on the Mechanical Properties and the Thermal Stability of Poly(vinyl chloride)

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The polymers are materials whose proprieties can be improved by adding various additives such as the fillers which can be mineral or organic compounds. In this study, we are interested in the possibility of improving the mechanical proprieties and the thermal stability of poly(vinyl chloride) (PVC) by adding local mineral fillers i.e., powder of marble and the dolomite. The composites PVC/dolomite and PVC/marble (fillers: 0, 5, 10, 15 and 20 phr) have been prepared. The test-tubes have been made up of the different composites through three stages i.e., blending, pressing and cutting up. These test-tubes have been then put under mechanical tests (tensile test and impact IZOD) and a thermo test (Vicat softening temperatures) whereas, the thermo stability of various components has been evaluated by the dehydrochlorination. The results have shown that there is a notable improvement of mechanical and thermo proprieties which is interpreted by the increase of the Vicat softening temperatures and an extension of stability time.

Key Words: Poly(vinyl chloride), Marble, Dolomite, Composite, Mechanical proprieties, Thermal stability.

INTRODUCTION

Today, we record a spectacular progression of the use of the composite materials in various fields such as the automobile, the electricity, spare times and sports and so on. They concern the judicious development sustained by the technical and economical interests that brings the use of its new products.

The introduction of various fillers such as montmorillonite, silica, calcium carbonate and aluminum oxide, can infer an improvement of thermo mechanical proprieties of polymers, such as toughness, the rigidity and heat resistance.

The poly(vinyl chloride) (PVC) is one of the most used polymers and the most changeable that we find in all aspects of the day to day life. In the past, a variety of PVC blend with other resins has been developed in order to spread the application of PVC. Thus the PVC blend/acyronitrile butadiene styrene (ABS) has been intensively studied and the ABS is proved compatibility with the PVC and has a good hardness effect on this material.
In recent years, the soaring of composites at the PVC base has been remarkable. Thus the different types of PVC composites/montmorillonite\textsuperscript{1,9}, PVC/cellulose\textsuperscript{10,11} and PVC/CaCO\textsubscript{3}\textsuperscript{12,13} have been studied.

The present work consists of testing and studying the new mineral fillers viz., marble powder and the dolomite effect on the thermal-mechanical properties of poly(vinyl chloride). The composites PVC/dolomite and PVC/marble, prepared according to different formulation, have been studied. Thus this work will contribute in valorization of wastes and the protection of the environment.

**EXPERIMENTAL**

The PVC (grade 4000 M) use a commercialized product by the petrochemical complex (CPIK) of Skikda, its value K is about 65-67. The stabilizers are: IRGASTAB 17 MOK (thermal stabilizer) IRGANOX 1076 (Anti oxidant) TINUVIN P (Anti UV). The used lubricants are stearic alcohol and IRGAWAX 280. The fillers are the marble powder extracted from the FIL FILA mine (SKIKDA) and the dolomite obtained from the MAROUANA mine (BATNA). Ten formulations used for preparation of the samples (Table-1).

<table>
<thead>
<tr>
<th>Additives</th>
<th>Role</th>
<th>Value (phr)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Formulation 1</td>
<td>Formulation 2</td>
</tr>
<tr>
<td>IRGASTAB 17 MOK</td>
<td>Thermal stabilizer</td>
<td>1.5</td>
</tr>
<tr>
<td>REOPLAST 39</td>
<td>Co stabilizer</td>
<td>1.0</td>
</tr>
<tr>
<td>IRGANOX1076</td>
<td>Co stabilizer</td>
<td>1.0</td>
</tr>
<tr>
<td>TINUVIN P</td>
<td>Antioxidant</td>
<td>0.1</td>
</tr>
<tr>
<td>Stearic alcohol</td>
<td>Anti UV</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>Intern lubricant</td>
<td>1.0</td>
</tr>
<tr>
<td>IRGAWAX 280</td>
<td>Extern lubricant</td>
<td>0.5</td>
</tr>
<tr>
<td>Dolomite</td>
<td>Mineral filler</td>
<td>0, 5, 10, 15, 20</td>
</tr>
<tr>
<td>Marble</td>
<td>Mineral filler</td>
<td>0, 5, 10, 15, 20</td>
</tr>
</tbody>
</table>

**General procedure:** Rigid blends of composition varying from 0 to 20 % of filler have been prepared according to the formulations given in Table-1. The blends have been prepared by mixing with the help of a mixer of RLS-110 type during 7 min in a two-cylinder mixer rotary heated to the temperature of 180 °C. At the end this operation, the mixed leaves prepared are compressed between the two trays of a press of PLA-30 type under a pressure of 125 bars to the temperature of 180 °C during 3 min, for their transformations in plates of 1 and 6 mm in thickness.

**Detection methods**

Thermo deshydrochlorination: The mixed leaves of about 1 mm thickness have been used for deshydrochlorination test. This study has been affected at 180 ± 1 °C according to ISO-R-182.
Test of softening Vicat temperature: The softening Vicat temperatures have been measured under a load of 1 kg, using an apparatus of CEAST Vicat.

Measurements of mechanical properties

Tensile test: Tensile tests were performed with ZWICKI 1120 apparatus using 1 mm thick specimen at a crosshead speed of 50 mm/min at an ambient temperature, using an Instron tensile tester according to ASTM D 638.

Notched IZOD impact test: Tensile tests were performed using 63.5 mm × 12.7 mm × 6 mm specimens a V-shape notch on a CEAST universal pendulum impact tester, in accordance with ASTM D 256. The hammer speed was 3.46 m/s. The notch is obtained by a constant profile knife, equipped with a motor.

RESULTS AND DISCUSSION

Figs. 1a and 1b shows the typical curve restrain-deformation of pure PVC and its composites PVC/dolomite and PVC/marble. The pure PVC is brittle in nature. But when fillers are added in the PVC matrix, the composites show ductile behaviours.

As shown in Fig. 2, the elasticity modulus of PVC/dolomite and PVC/marble composites increase simultaneously with the increase of filler content. However the elaborated samples starting from the dolomite present the most great elasticity module.

Classically, composites based on mineral fillers and polymer matrix exhibit increased elasticity modulus with the increase of the filler concentration due to the rigidity of fillers and the strong interaction between the filler and the polymer matrix.
Figs. 3 and 4 represent the tensile strength and elongation at break of PVC/dolomite and PVC/marble as a function of dolomite and marble content, respectively. There is a progressive increase of the tensile strength in the interval of 0-10 phr, for the two types of composite, follows a decrease, but the values are even better than those unfilled PVC.

![Fig. 2. Elasticity modulus of the composites as a function of filler content](image)

![Fig. 3. Tensile strength of the composites as a function of filler content](image)

The elongation at break of PVC/dolomite and PVC/marble composites can be greatly increased when low content of mineral filler is introduced. This great phenomenon indicates the limitations of the theories when applied to composites. The strong interaction between micro particles of filler and PVC matrix caused by the large interfacial areas which led to much higher elongation at break, with a maximum at 5 phr of marble.

The impact strengths of composites are presented in Fig. 5. It can be seen that PVC/dolomite exhibits much better impact than PVC/marble. For example, the impact strength of the composite PVC/dolomite reaches the maximum value of 60.95 J/m when 10 phr of dolomite is introduced. While the maximum impact strength of the PVC/marble is 58.77 J/m when 5 phr of marble is introduced. This result suggests that the dolomite has an effect of hardening on the PVC better than that of the marble.

The Vicat softening temperatures of composites PVC/dolomite and PVC/marble are shown in Fig. 6. It shows that the addition of fillers, leads to increase in Vicat softening temperatures due to the rigidity of dolomite particles or the marble. Thus, it is suggested that the dispersion state of fillers also affects the Vicat softening temperatures of composites.

Fig. 7 represents evaluation of thermal stability time as a function of dolomite and marble content. The addition of mineral filler to PVC increases really the induction period or the thermal stability time ($T_s$).
Conclusion

Through this study, it is observed that the mechanical and thermal behaviour of composite to PVC added of the mineral fillers.

According to the results gotten it is noticed that the filled samples show a better behaviour in comparison to one of the unfilled samples.

The elasticity modulus and the Vicat softening temperature can be increased simultaneously by the presence of dolomite or the marble powder.

The optimal values of elongation at break and IZOD impact strength of composites PVC/dolomite and PVC marble have been obtained at 10 phr of dolomite and 5 phr of marble, but the dolomite has a better effect than that of the marble.

It also concluded from this study that the marble and the dolomite confer to the PVC a good thermal stability resulting in a slowing of the clearing of HCl and a considerable overtime of the thermal stability time.
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