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FTIR Analysis and thermal behavior of microcomposite Pozzolan/ DGEBA

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Abstract. In the present study six blends of Diglycidyl ether of bisphenol-A and pozzoan filler were synthesized by physical mixing at 100°C. Interaction among epoxide group of DGEBA, composition of fillers and anhydride group of curing agent discussed through FT-IR analysis. The TGA studies revealed a increase in thermal stability and the glass transition temperature (Tg).
The integral procedure decomposition temperature increased from 571,3 °C to 617,9,3°C. The composites had a higher glass transition temperature than the neat epoxy resin. The coefficient of thermal expansion of the composites at the glassy and rubbery regions decreased with increasing filler content. Scanning electron microscopy (SEM) indicated the presence of two-phase morphology in the blends.

Keywords: Granulation, Pillared clay, Gluten, Malachite Green, Adsorption.

1 Introduction

Fillers or fillers are used in polymers for a variety of reasons, namely to reduce the cost, improve the treatment, control density, thermal conductivity, thermal expansion, electrical properties, magnetic properties, flame retardancy, and to improve the properties mechanical [1-3].
Each type of expense has different properties depending on the particle size, shape and surface chemistry [4-6]. The most used are thermosetting resins fillers calcium carbonate, kaolin, alumina hydrate and [7]. The other filler typically used include clay, carbon black, microspheres mica, silica, glass, and glass favorites [8].

2 Materials and methods

2.1 Epoxy resin.

In our work, we used the STR MEDAPOXY resin Granitex company, known for its high performance and good quality. **Fig.1**

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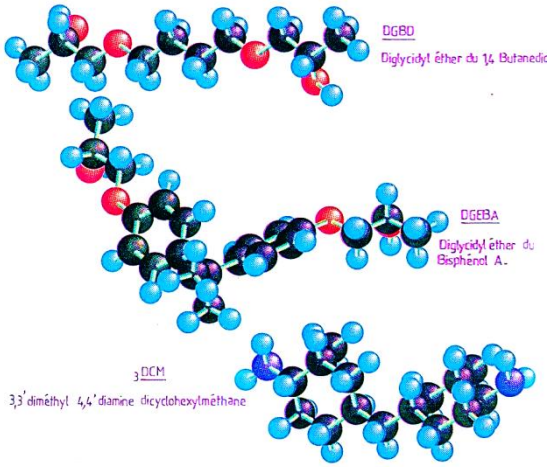

Fig. 1. 3D atom of carbon BADGE representation in black, hydrogen in blue and oxygen in red

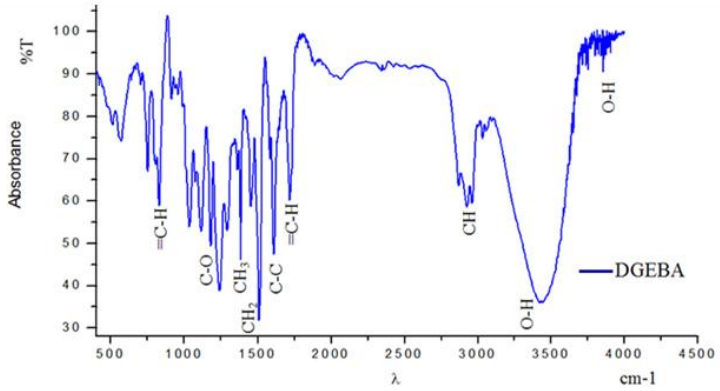

Fig. 2. IR Spectrum of DGEBA

This figure shows the infrared spectrum of the Fourier transform F DGEBT resin, the CF group absorption band appears at 1105 3 cm-1. The absorbent characteristic bands at 910 and 835 cm-1 indicate the presence of the epoxy Glycidyl.

2.2 Loads minerals

The hybrid nanocomposites prepared using matrix (diglycidyl ether of bisphenol A) mixed with nanofillers minerals, very high silica such as silica fume, pozzolana and the blast furnace slag. Six mass concentrations wt% (0, 20, 30, 40, 50 and 60%) and two particle sizes have been studied in the presence of a diluent which serves to reduce the viscosity of the mixture

2.2.1 Loads minerals characteristics

The filler used in this work is a natural pozzolan (PZ) recover in the deposit of Beni Saf, Algeria. The samples were mechanically grinded to obtain a very fine particle size (fig). Is an amorphous mineral filler, very rich in silica (wt% SiO_2 = 60,67%), (wt% CaO=8,98; MgO=3,45; Al_2O_3=15,10; Fe_2O_3=5,14) (**Fig.3**), and some alkali traces, with a bulk volume mass of 2.66 g/cm3. In **figure 4**, it is shown that a significant amorphous phase is present, and crystalline phases are visible.

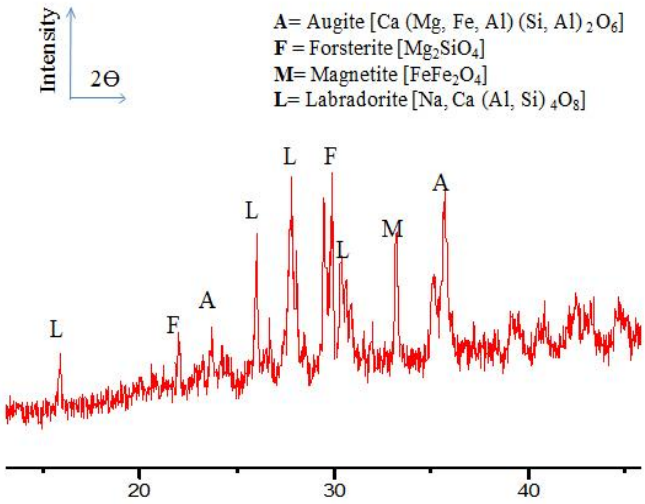

Fig.3 X-ray Diffraction (XRD) of Pozzolan

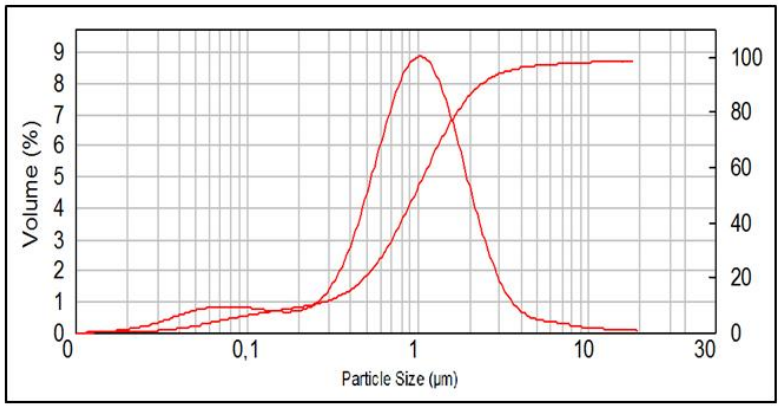

Fig. 4 Particles size distribution of pozzolan

The particle size distribution shown in Fig.4 shows that a large part of the particles has a diameter of less than 4µm with a medium of 0.98 µm.

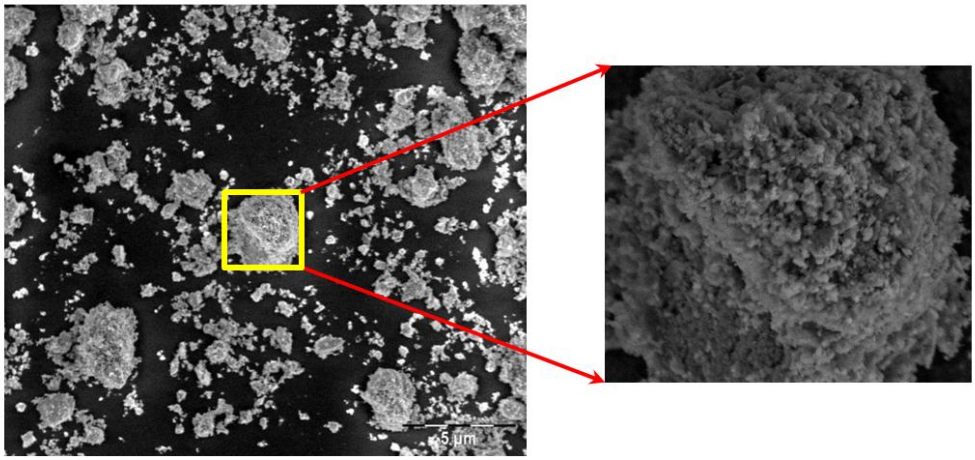

Fig. 5 SEM micrographs of pozzolan filler

The zooms realized shows that the particles have a very high porosity, which explains the high specific surface areas, determined using the BET method, value of 23,5 $m^2.g^{-1}$.

2.3.Characterization and measurements

The thermogravimetric analysis is a thermal analysis technique that measure the quantity and rate of change of mass of a sample as a function of temperature and time. It assesses loss of mass or phase changes when the material decomposes, becomes dehydrated or oxide. In our case, the ATG was used to evaluate the water content of cellulosic substrates (fiber component) and determining the area of degradation thereof. Found values of moisture content are 6% and 8% by weight respectively for virgin fibers PLD and those modified by oxidation with TEMPO. The device used is a thermobalance SETARAM TGA-9212. The test sample varies between about 10 and 15 mg of sample, the heating rate is 10 ° C / min under nitrogen atmosphere.

Results and discussion

The graph above gathers the results ATD / ATG of the epoxy resin (médapoxy STR) calcined at a temperature of 600 ° C.

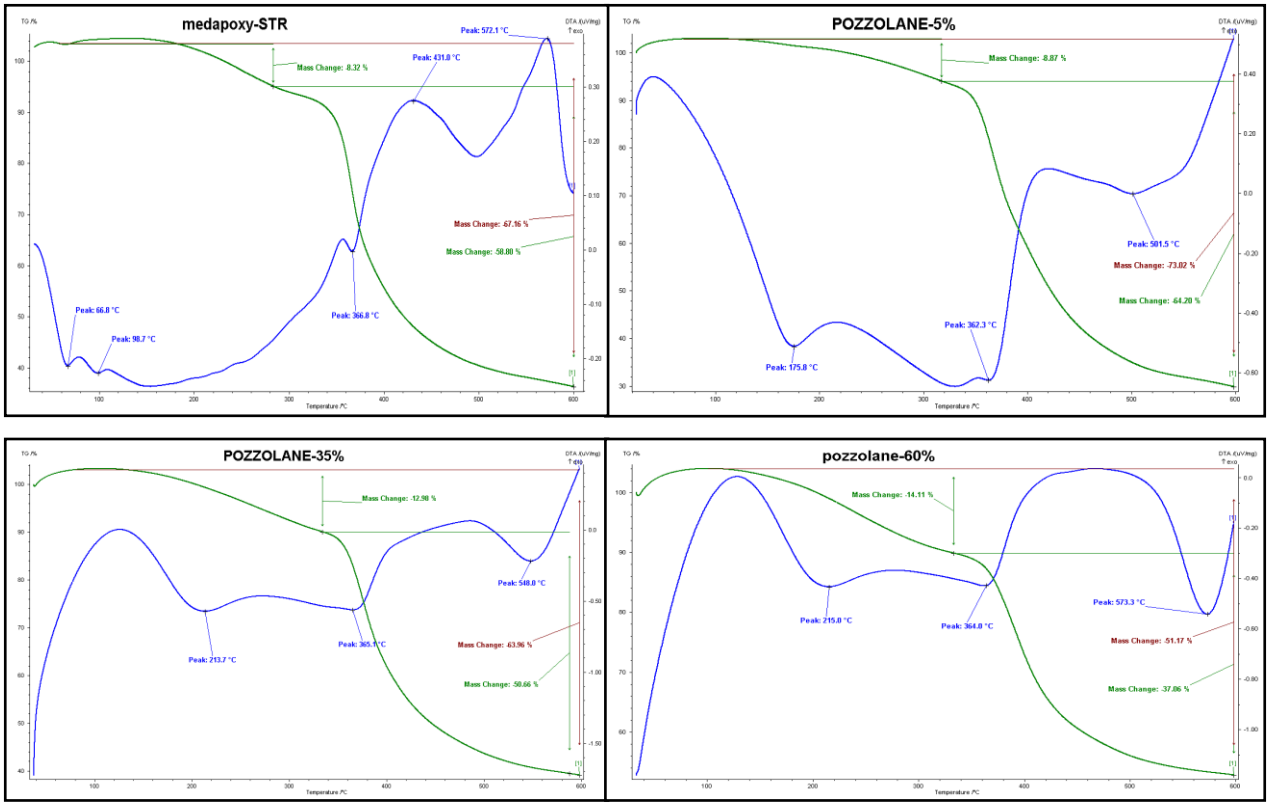

Fig 6. Results ATD / ATG of the composite epoxy resin /pozzolan

We observe three endothermic peaks located at 66.8 ° C, respectively, 98.7 ° C and 366.8 ° C due to considerable weight losses. This is associated with the loss of the various water present in the sample structure.

The first endothermic peak at 66, 8 ° C represents the elimination of free water filling macrospores between particles, involving mass loss estimated at 8.32% .The second endothermic peak is observed at a temperature of 98.7 °C, corresponding to the limitlessness of the molecules. The third endothermic peak is observed at a temperature of 366.8 ° C, corresponding to the limitlessness oligomers involving however a rather large mass loss estimated at 58.80%.

There is however the presence of two exothermic peaks at 431 ° C and 572.1 ° C.

Nous observons les pics endothermiques localisés vers 175.8°C, 362.3°C et 501.5°C pour la matrice avec 5% de charge ; 213.7°C, 365.1°C et 548°C pour celle à 35 % de pouzzolane et 215°C , 364°C et 573°C pour la résine a 60% de PZ ; dû à des pertes de masse considérables. Ceci est associé à la perte des différentes eaux présente dans la structure de l'échantillon.

Les premiers pics endothermiques situés à 175.8 °C, 213.7 °C, 215 °C respectivement pour les pourcentages de 5% ; 30% et 60% représente l'élimination des eaux libres remplissant les macrospores, impliquant une perte de masse évaluée respectivement à 8.87% , 12.98 % et 14.11 % .Les seconds pics endothermiques sont observés à 362.3°C, 365.1°C , 364°C correspondant à l'illimitation des molécules d'impuretés

Les troisièmes pics endothermiques localisés à 501.5°C pour la 5 % en PZ, 548 °C pour celle à 35%en PZ et 573°C pour la 60% en PZ ; correspondant à l'illimitation des oligomères impliquant toutefois des pertes de masse assez importante évaluée respectivement à 64.20% , 50.66% et 37.06 %.

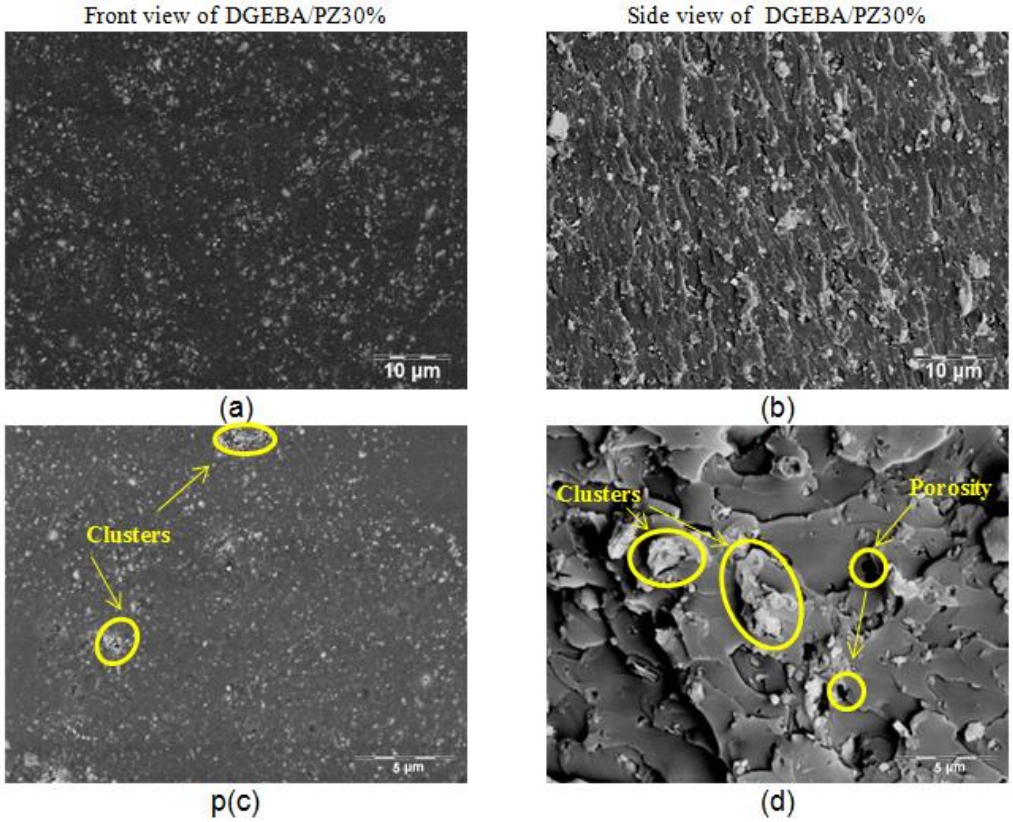

Fig. SEM micrographs of composite with 30wt% of pozzolan (DGEBA/PZ30%)

The pozzolan particles are finely dispersed in the material. Moreover, the finest dispersion can probably not be detected by SEM. In the micrographie (b), the particles of size 4 and 10 µm diameter formed large ordered clusters and macro and micropores are also observed. The morphology of the matrices remains without form and it can generate the appearance of major flaws in the layout. Internal porosity are more concentrated in the areas where the flow is disturbed.

Conclusion

The thermal properties of the organic-inorganic hybrid materials based on DGEBA epoxy resin and pozzolan particles were examined using a range of techniques [17-20].

The thermogravimetric analyzes of the different samples give us a comparison between the loaded and uncharged samples, to deduce the contribution of the mineral load on the thermal behavior of the resin and its influence on the thermal durability. The Tg of both composites was 116 C higher than that of the neat epoxy resin.

They show that the temperature of first degradation of the resin is similar to that of the loaded composites up to the temperature of 400 ° C where the degradation accelerates for the resin alone and slows down for the loaded samples. The degradation gradient increases as a function of the temperature until the total decarbonation of the samples

These analyzes showed that the degradation of the resin is the main reason for the decrease in the properties of all the compositions. The importance of these analyzes gives us a major understanding of the thermal durability of these composite materials.

These results suggest that the thermal stability of the epoxy resinwas improved by the addition of pozzolan particles. The charge is a thermal barrier and protects the heat flow matrix.

References

[1] R. M. Laine, J. Choi, I. Lee, "Organic-Inorganic Nanocomposites with Completely Defined Interfacial Interactions," Adv. Mater. 13, 800-3 (2001)

[2] Lichtenhan JD, Otonari YA, Carr MJ. "Linear hybrid polymer building-blocks". Macromolecules, 28, 8435-8437. (1995)

[3] Fong H, Dickens SH, Flaim GM. Evaluation of dental restorative composites containing polyhedral oligomeric silsesquioxane methacrylate. Dent Mater, 21, 520. (2005).

[4] Kannan RY, Salacinski HJ, Butler PE, Seifalian AM. Polyhedral oligomeric silsesquioxane nanocomposites: the next generation material for biomedical applications. Acc Chem Res. 38(11), 879-84, (2005).

[5] Nikolic, Lj.; Skala, D.; Nikolic, V; Stamenkovic, J.; Babic, D.; Ilic-Stojanovic, S. Methylmethacrylate and acrylamide crosslinked macroporous copolymers. J. Appl. Polym. Sci. 2004, 91,387-395.

[6] Nikolic, Lj.; Raskovic, Lj.; Stamenkovic, J.; Nikolic, V. Crosslinking of the thermoreactive polyacrylates in polymer coating, World Polym. (Serb.) 2000, 3, 6-10.

[7] Nikolic, Lj.; Nikolic, V; Stankovic, M.; Todorovic, Z.; Vukovic, Z. Porous poly(methylmetacrylate) and poly(methylmetacrylate-co-acrylamide). Chem. Ind. (Serb.) 2006,60, 327-332

[8] Du, B.X. ,Zhang, J.W. ; Gao,Y., Effects of TiO2 particles on surface charge of epoxy nanocomposites, Dielectrics and Electrical Insulation, IEEE Transactions on (2012) (Volume:19 , Issue: 3)

[9] S.B. Damman, J.A.H.M. Buijs, J. van Turnhout, Polymer 35 (1994) 2364–2371.

[10] P. Avakian, J.C. Coburn, M.S. Connolly, B.B. Sauer, Polymer 37 (1996) 3843– 3850. 548
[11] L.H. Sinh et al. / Reactive & Functional Polymers 72, 542–548. (2012)
[12] Irekti Amar, et al. FTIR Analysis and Rheological Behavior of Bisphenol: A Diglycidyl Ether Resin Filled Fume-Silica " Journal of Materials Science and Engineering A 4 (11) (2014) 340-347. https://doi:10.17265/2161-6213/2014.11.004
[13] A. Irekti and B. Bezzazi. Rheological Study of Composite Materials Based on Thermosetting Matrix and Fillers Mineral. Key Engineering Materials, 550 (2013) 79–84, doi.org/10.4028/www.scientific.net/KEM.550.79.
[14] A. Irekti, B. Bezzazi, A. Smith, C. Aribi. Experimental Study of Dielectric Properties of Composite Materials Pozzolan/DGEBA" Journal Polymer Composites, 38(2) (2017), https://doi.org/10.1002/pc.23589
[15] A. Irekti, B. Hami, C. Aribi, B. Bezzazi, I. Briki "Rheological behavior of the epoxy resin loaded with the pozzolan" Journal of Materials Science and Engineering (A&B), ISSN 2161-6213 & ISSN 2161-6221. (2012) pp.519-524. DOI:10.17265/2161-6213/2012.07.005
[16] Irekti Amar, Hami Brahim, Aribi Chouaib, Bezzazi Boudjema "Dielectric behavior study of thermosetting matrices loaded" European Scientific Journal Vol 8, No 27 (2012) http://dx.doi.org/10.19044/esj.2012.v8n23p%25p
[17] F. Zibouche, N Boudissa, A. Irekti, M.T. Abadlia, « Géopolymérisation d'aluminosilicates. Influence des rapports Silice/Alumine. » Journal of Materials, Processes and Environment ISSN: 2335-1020 Vol.1 (02) (2013). pp 147-156. http://www.urmpe.dz/journal/archives
[18] Hami B., Irekti A., Bezzazi B - Etude du comportement au choc d'un Matériau Composite renforcé par des Fibres de Verre. Journal of Materials, Processes and Environment ISSN: 2335-1020 Vol.1 (01) (2013) pp 57-59. http://www.urmpe.dz/journal/archives
[19] Irekti A., Hami B., Aribi C., Abadlia M.T." Static and cyclic behavior of concrete reinforced with materials composite". Journal of Materials, Processes and Environment ISSN: 2335-1020 Vol.1 (01) (2013). pp 67-72. http://www.urmpe.dz/journal/archives
[20] B. Hami, A. Irekti, C. Aribi, B. Bezzazi, A. Mir, "Experimental study of sandwich multilayer reinforced by glass fibre and agglomerated cork" Advanced composite letters. 23(5), 2014. pp121-125 https://doi.org/10.1177/096369351402300503