DOI: 10.2478/sspjce-2024-0002

Viscoelastic and Thermal Characterization of a Composite Material Based on Unsaturated Polyester Resin Reinforced with Perlite

Ouided Dehas[1*], Yasmina Biskri[1], Mohammed Benzerara[2], Laidi Babouri[1], Acheref Cherifi[3]

[1]Higher Normal School of Technological Education, Azzaba, Skikda, Algeria.
[2]Materials, Geomaterials and Environment Laboratory (LMGE), Department of Civil Engineering, Badji Mokhtar- Annaba university, P. O. Box 12, 23000 Annaba, Algeria.
[3]Research center in industrial technologies, additive manufacturing research unit, Industry zone A15, Setif 19000, Algeria.
[*]e-mail: dehasouided@gmail.com

Abstract
In this work, it was proposed to replace the conventional reinforcement of the unsaturated polyester resin by a mineral, from a siliceous volcanic rock of volcanic nature, perlite. UPR/perlite composites with different proportions of phase components (from 1% to 5% of powder mass part). We used unsaturated polyester resin (UPR) as well as the hardener cobalt octoate and treated and untreated perlite of different dimensions (greater than 60µm, and less than 60µm). The composites were prepared by the contact molding process. The composite plates are hardened for 24 hours at room temperature then placed in an oven for 15 hours at 50°C to undergo post-curing. The composites obtained were subjected to different characterization techniques, namely rheological tests (dynamic mechanical analysis (DMA)), thermal tests (differential calorimetric analysis (DSC)) and Thermogravimetric analysis (ATG) and structural characterization by Fourier transform infrared (FTIR). The DMA measurements showed that the UPR/perlite composites with untreated filler presented conservation modules higher than that of the resin without perlite for the rates of 3% and 4%, while for the composites with treated filler, that at 3% of perlite shown the highest modulus along the glassy zone. Also, the glass transition temperature of the UPR resin was not affected by the addition of perlite. The decrease in intensity at mid-height of the tan δ peaks allowed deducing the existence of a fairly strong UPR/perlite interface. DSC thermograms showed that the exothermic peak is shifted to higher temperatures, due to a delay in the curing reaction caused by the presence of the perlite particles. This study concluded that the perlite enhances the properties of composites.

Keywords: unsaturated polyester resin, perlite, composites, DMA, Tg.

1 Introduction

In recent years, polymers have been the subject of increasing interest from manufacturers. The aim was to achieve the production and marketing of new materials with improved thermomechanical properties. Currently, polymer materials offer ease of processing and are therefore widely used in all industrial sectors. Their intrinsic properties being, however,

relatively weak, it is necessary to improve those using adjuvants (coloring, fillers, reinforcements, unfolding, etc.) The evolution of polymer materials has involved the development of composites with an organic matrix reinforced by small particles (e.g. talc, fiberglass, wood chips, calcium carbonate, etc.), also called filler. The introduction of filler makes it possible to improve the mechanical and physical properties of the matrix (polymer) [1].

Polymer composite materials are multiphase materials used for their physicochemical, mechanical and electrical properties, etc. However, when designing the object, we often realize that the properties of the polymer alone are insufficient. Indeed, modern technology requires materials that combine rigidity, mechanical resistance, high toughness and great lightness. No simple material can combine all these physical characteristics at the same time; this is why, for a number of years, we have been seeking to obtain materials combining different properties, "composite materials".

Composites have often played a role as a substitute for metallic and ceramic materials, due to their lightness, ease of execution and good properties. In fact, the respective qualities of the associated constituents complement each other to form a material with improved mechanical, thermal, electrical and/or physicochemical performances. The progress of composite materials having one or more of these particular properties generally rejoins to a specified need. It is thus possible to create heterogeneous materials allowing, for example, to reduce the mass of a part, while improving its mechanical properties, thanks to the combination of a fibrous reinforcement and an organic resin.

The adaptability of this material makes it an incontestable asset which explains its increasingly widespread use, particularly in the transport industry (air, maritime and rail), sports and leisure, and the construction industry [2].

Recently, nanocomposites with UPR matrix and particle reinforcements have been an exciting area of research due to the significant improvements in all the properties among these nanomaterials we can cite epoxy resin/perlite [3-9], These composites have been widely Used in industrial applications such as marine, automotive, pipeline systems and construction industrial due to their low cost, high chemical resistance and good workability.

Several studies in which fibrous reinforcements have been incorporated into thermosets and particularly into polyester resin can be found in the literature [10-35] Indeed, among fiber-reinforced polyester materials, glass fiber composites are the most general due to their favorable mechanical and economic characteristics [10]. However, a disadvantage constituted by the lack of affinity between the matrix and the reinforcement generates a transition to fragile mechanical behavior and can inflict limitations on many applications.

For this reason, research on fiber-reinforced thermosetting polymers generally focus on characterizing interface behavior, adaptation of the interface [11-15] as well as on the effects of the fiber-matrix interface and on the mechanical properties of composite materials [12] Organosilanes are the most widely used coupling agents for improving the interfacial adhesion of fiber glass reinforced materials. Their efficacy depends, among other things, on the nature and pretreatment of the substrate, the type of silane used and the process [19-21]. In addition to glass fibers, thermosets have also been reinforced with other types of fibers. A study of the influence of interfacial shear strength on various mechanical strength properties of a carbon fiber-reinforced epoxy composite showed that the strengths with notable shear stresses could be significantly improved by a more effective interface [22].

Currently, there is great interest in incorporating plant fibers into polymers in order to develop low-density biocomposites. Similarly, several natural fibers such as sisal, jute and the Alfa have been studied as reinforcement in UPR composites [25-29]. Recently, nanocomposites with an unsaturated polyester matrix and mineral filler have been a new area of research due to the acceptable performance on all properties among these nanomaterials we can cite polyester resin / montmorillonite (MMT) [36-38], UPR /clay [39].
In this work, we proposed to replace the classic reinforcement of unsaturated polyester resin with an ore, coming from a siliceous rock of volcanic nature, perlite. This filler has the particular characteristic, relative to ores already incorporated into polymers, of containing a very large proportion of pores which can allow it to exfoliate under certain conditions, and to show a large specific surface area. Access of the liquid resin to the pores of the filler could make it possible to develop sufficient interactions to obtain new composites with acceptable performance, which constitutes the essential objective of our study.

2 Experimental

2.1 Materials

2.1.1 Unsaturated polyester resin

Isophthalic unsaturated polyester resin provided by the company Maghreb Pipe of M'sila (Algeria) specialized in the manufacture of polyester resin laminate and fiber glass was used in this study. It is a product marketed by the company Exon Mobil Chemical in the form of a low viscosity mixture of 373 mPa.S at 25°C. The mechanical and physical characteristics of this resin are given in Table 1.

Table 1: Mechanical and Physical characteristics of resin

Characteristics	Unit	Value
Tensile strength	MPa	40-60
Barcol hardness	-	40-45
Flexural strength	MPa	80-100
Application method	-	Manual application
Density	kg/dm^3	1.09 -1.11
Curing time	min	45-60

2.1.2 Hardener

It is a substance which increases the speed of a chemical reaction without being consumed and without modifying the final position of the thermodynamic equilibrium of this reaction. The dosage to be respected for this catalyst is 1.5-2% by mass. Raw molecular formula of catalyst ‘’ methyl ethyl ketone peroxide (MEKP)”: $C_8H_{18}O_6$

Table 2: Characteristics of MEKP catalyst

Chemical formula	$C_8H_{18}O_6$
Molecular weight	210.22 g/mol
Density at 20°C	1.14 g/cm^3

Active oxygen	9.0-9.2 (% mass)
Storage temperature	25°C max
Color	Clear
Storage	6 months

2.1.3 Perlite

In the initial state, perlite looks like sand containing water. It is a natural siliceous volcanic rock. It is expanded industrially by a heat treatment which expands it by 4 to 20 times its volume and forms small porous grains showing tiny cavities. It is very light and friable; it is a natural and ecological mineral insulator, non-polluting and non-toxic for humans. The perlite used in this work was supplied to us in this expanded form and comes from Tlemcen-Maghnia, Algeria. The general characteristics of perlite are grouped in Table 3.

Table 3: Physical characteristics of perlite

Characteristics	Unit	Value
Color	-	White
Shine	%	84
Refractive index	-	1.47
Specific gravity	g/cm^3	2.34
Density	Kg/m^3	70

2.2 Preparation of UPR/Perlite composites

2.2.1 Sifting perlite

Before preparing the composites, the perlite was sifted using sieves with different mesh sizes, in order to separate two fractions of ores with the following grain sizes: between 80 µm and 60 µm (Perlite >60 µm) and less than 60µm (Perlite <60 µm).

2.2.2 Silane treatment

In the literature, chemical treatment with (3-aminopropyl) triethoxysilane, also known as silane (Figure 1) is often used on fibers of mineral and plant origin.

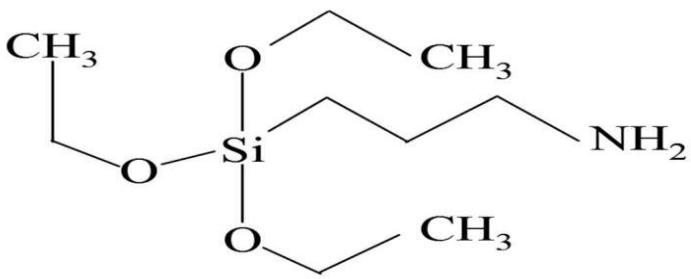

Figure 1: Chemical formula of (3-aminopropyl) triethoxysilane

Silinization is one of the chemical treatments which aim to facilitate adhesion between the fillers and the polymer matrix. The procedure adopted for chemical treatments with silane is as follows: firstly, the charge submits to gasification. Thus, an ethanol/water solution (60/40, v/v) was prepared; the pH was adjusted between 4 and 4.5 by adding a drop of glacial acetic acid. Then, silane (3% by mass) was added. Hydrolysis of the prepared solution was carried out for 2 h at room temperature. Finally, perlite was added, and the suspension was kept for 3 h at 70°C. Then, before extrusion, the load is pre-dried at 105°C for 24 hours in order to eliminate the absorbed humidity and ethanol, and thus avoid the formation of agglomerates. A single concentration of silane was chosen and fixed [40]. The manufacturing process of composite materials influences the properties of the finished product; so, this is a very important step. Indeed, depending on the desired performance of the composite, the conditions and implementation parameters were determined. The production process used in this work is contact molding, frequently used for shaping thermosetting matrix mixtures. The accelerated unsaturated polyester resin is mixed randomly and gently in a container. Perlite, of both particle sizes, untreated and surface treated and steamed at 60°C for 24 hours, is added at the following rates 1, 2, 3, 4 and 5%. Finally, to have good curing of the polyester resin a concentration of about 1.5 % of methyl ethyl ketone peroxide (MEKP) is added. The different mixtures, thus prepared, are poured onto a Teflon sheet on which the wooden mold is placed. The plates of the three types of composites are hardened for 24 hours at room temperature then placed in an oven for 15 hours at 50°C to undergo post-curing (Figure 2).

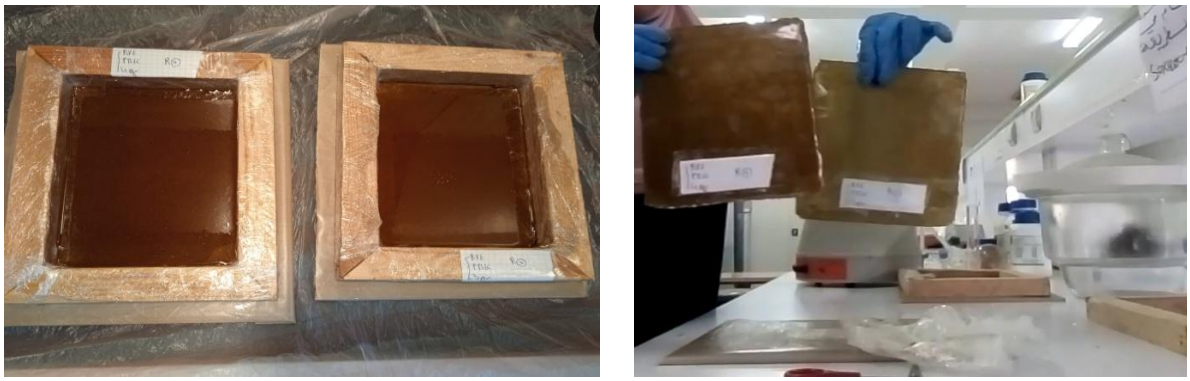

Figure 2: UPR/perlite composites preparation steps

3 Experimental techniques

3.1.1 Structural analysis by FTIR spectroscopy

The structural study by infrared spectrophotometry aims to highlight the existence of interactions between the matrix and the incorporated reinforcements. The perlite ore was analyzed in the form of pellets prepared according to the following compositions: 3% perlite and 97% potassium bromide (KBr) (scan number: 10). The FTIR spectra of the polyester resin and composites were obtained on very thin films (scan number: 5). The infrared spectra were recorded using a Perkin Elmer type spectrophotometer over a wave number range spanning from 4000 to 500 cm^{-1}, using a resolution equal to 2 cm^{-1}.

3.1.2 Dynamic Mechanical Thermal Analysis

The viscoelastic properties of pure UPR resin and UPR/perlite composite materials were studied in dynamical mechanical thermal analyzer DMAQ-800TA (TA instruments, New Castle, DE). The tests were carried out at a frequency of 1 Hz, amplitude of 10 µm, a heating rate of 5°C/min and over a temperature range ranging from -50 to 180°C. The tests were performed using rectangular samples with dimensions of (35x10x4) mm^3. All experiments were carried out under nitrogen atmosphere (flow: 50 mL/min).

3.1.3 Differential Scanning Calorimetry (DSC)

The DSC study was carried out using a TA Q-100 apparatus (TA instruments, New Castle, DE). Samples weighing between 5 and 10 mg was placed in a sealed aluminum pan, then placed in the oven where they submit to the following three-step program: heating from 25 to 250°C at a heating rate of 10°C / min, then they were maintained at 250°C for 2 min followed by cooling down to 25°C. Afterwards, a second DSC heating run was carried out from 25 up to 25°C at a heating rate of 10°C /min. All experiments were carried out under nitrogen atmosphere (flow rate: 50 mL/min).

3.1.4 Thermal Gravimetric Analysis (TGA)

Thermal Gravimetric Analysis (TGA) is a thermal analysis technique for measuring the quantity and rate of change in mass of a sample as a function of temperature and time. It makes it possible to evaluate the loss of mass or phase variations when the material decomposes, dehydrates or oxidizes. The TGA of the composites was carried out in Q-500 TA instrument (TA Instruments, New Castle, DE). An inert gas sweep is provided in the balance compartment with a flow rate of 100 mL/min, in order to protect the mechanism carrying out the weighing from any risk of oxidation. In this study, the measurements were carried out using a temperature sweep ranging from ambient to 600°C, at a heating rate of 10°C/min.

4 Results and discussion

4.1.1 Characterization of UPR, perlite and UPR/perlite composites by IRTF

The FTIR spectrum of the unsaturated polyester resin, represented by Figure 3, shows a large mass characteristic of the elongation vibrations of the hydroxyl groups between 3600 and 3200 cm^{-1}. The elongation vibrations of aromatic CH are observed between 3090 and 3000 cm^{-1}, while those of aliphatic CH are detected between 2990 and 2850 cm^{-1}, approximately. On the other hand, a strong band is observed around 1720 cm^{-1}. This is a characteristic of the carbonyl group of polyester while the vibration bands of the aromatic cycle are identified around 1600, 1504 and 1470 cm^{-1}. The region between 1400 and 1000 cm^{-1} is particularly complex because it is rich in bands and contains the deformation vibration of the C-O bond of polyester [41].

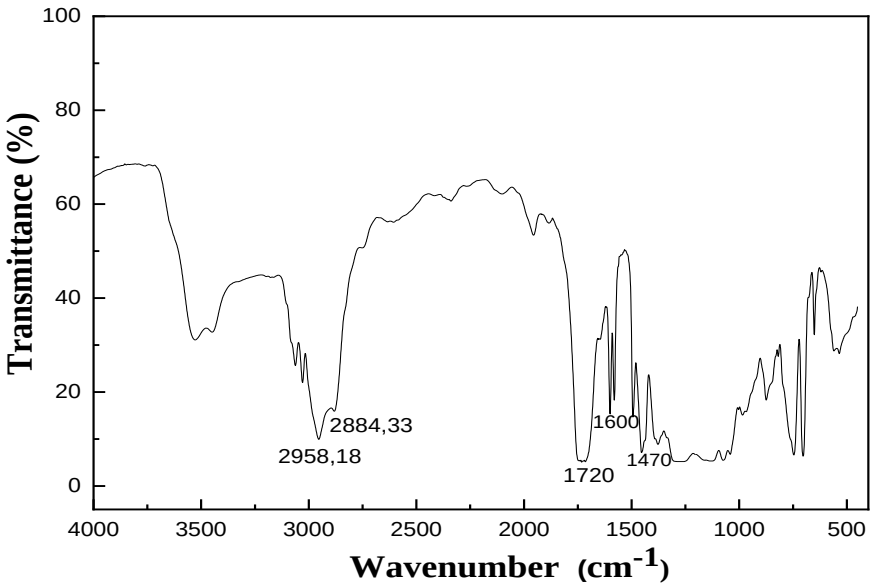

Figure 3: FTIR spectra of unsaturated polyester resin (UPR)

The FTIR spectra given in (Figure 4) characterizes untreated and treated perlite by silane. The spectrum of untreated perlite shows a broad absorption band, situated between 3600 and 3000

cm^{-1} and attributed to the valence vibrations of hydroxyl groups. Furthermore, the band observed at 1637 cm^{-1} is characteristic of the vibration of the H-O-H bond of water adsorbed on the surface of the ore. The intense band detected between 1400 and 800 cm^{-1} and centered around 1046 cm^{-1} is attributed to the vibration of the Si-O bond of the Si-O-Si group, while the one located at 797 cm^{-1} corresponds to the vibrations of deformation of the Si-O bond of the Si-O-Al group [42-43].

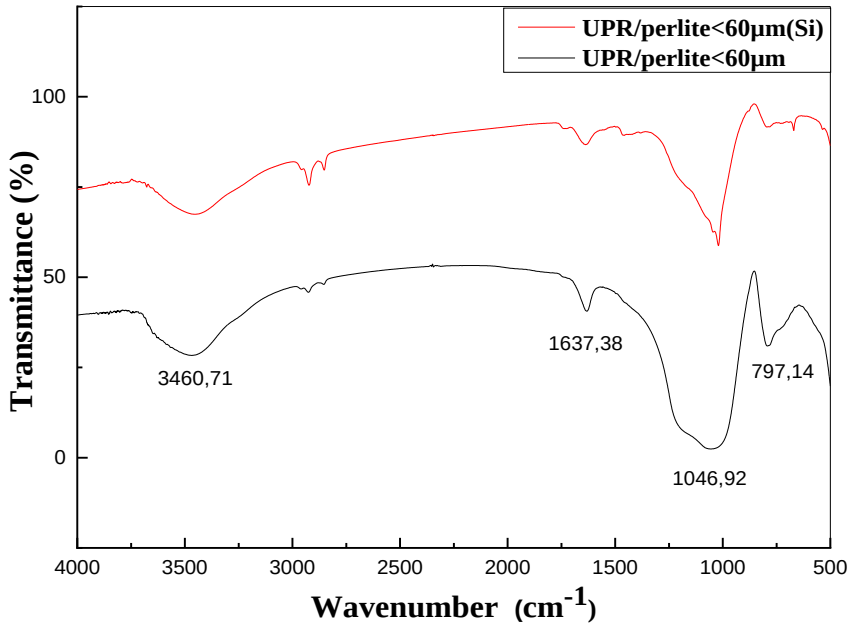

Figure 4: FTIR spectra of entreated perlite and perlite treated by silane

After treatment, we notice that the spectrum of the perlite treated with the silane coupling agent is partially different from that of the untreated perlite and this is explained by:
The surface treatment should lead to the appearance of a new bond relating the silicon atoms of the treating agent to the hydroxyls of the filler, which results in the formation of a Si-O bridge between the two components. But, if the vibration frequency of this last bond coincides with that of the Si-O bond already existing in the structure of the perlite, it becomes difficult to comment on the result of the treatment.
Furthermore, the FTIR spectrum of the UPR/perlite composite >60µm at 3pcr, given in (Figure 5), is perfectly similar to that of UPR. We note, however, a rather remarkable intensification of the band characteristic of the valence vibrations of the hydroxyl groups, which appears in this spectrum in the form of a fairly broad mass, denoting a greater concentration of the OH groups in the composite, following the incorporation of the load.

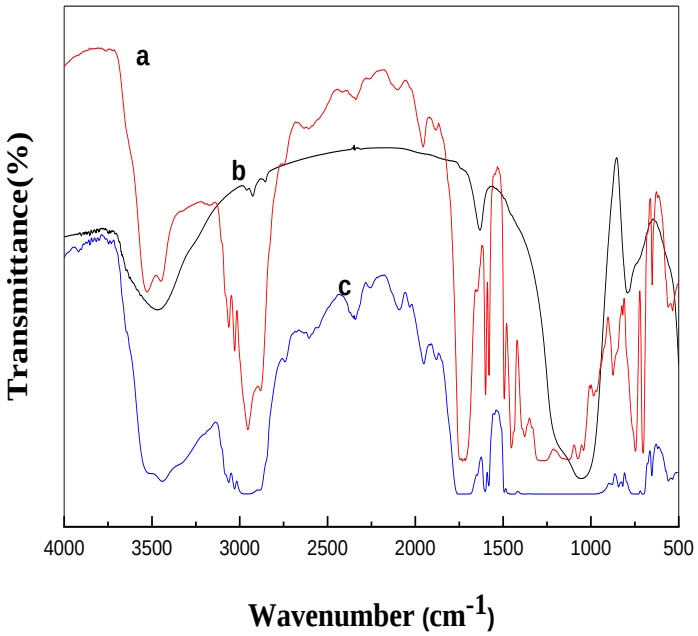

Figure 5: FTIR spectra of: (a) UPR, (b) untreated pearlite and (c) UPR/perlite composite >60µm at 3%

4.1.2 Viscoelastic properties of UPR/perlite composites

4.1.2.1 Effect of rate of filler

The variations in the conservation module E' of the UPR resin and the UPR/perlite composites<60µm formulated from the untreated mineral charge are given in (Figure 6). The thermograms show a similar appearance including, firstly, the glassy domain extending until the occurrence of the glass transition, identified thanks to the strong reduction in the modulus, then the region characteristic of rubber behavior where the materials present constant modules. In the glassy domain where the storage modulus does not vary with increasing temperature, we found that after the addition of perlite, the modulus increases with the loading rate. This increase is very evident for the loading rates of 3 and 4% which, apparently, contribute to a notable increase in the stiffness of the composites. So, more the rate increases, more the modulus is higher. Approaching the glass transition, the conservation module of composites drops sharply due to the increase in flexibility of composites. Beyond the glass transition zone, the modulus E' of the composites reaches very low values because of their rubbery behavior and does not vary with the increase in temperature and perlite content. The result concluded from this study funds the fact that the addition of 3-4% perlite improves the properties of UPR resin.

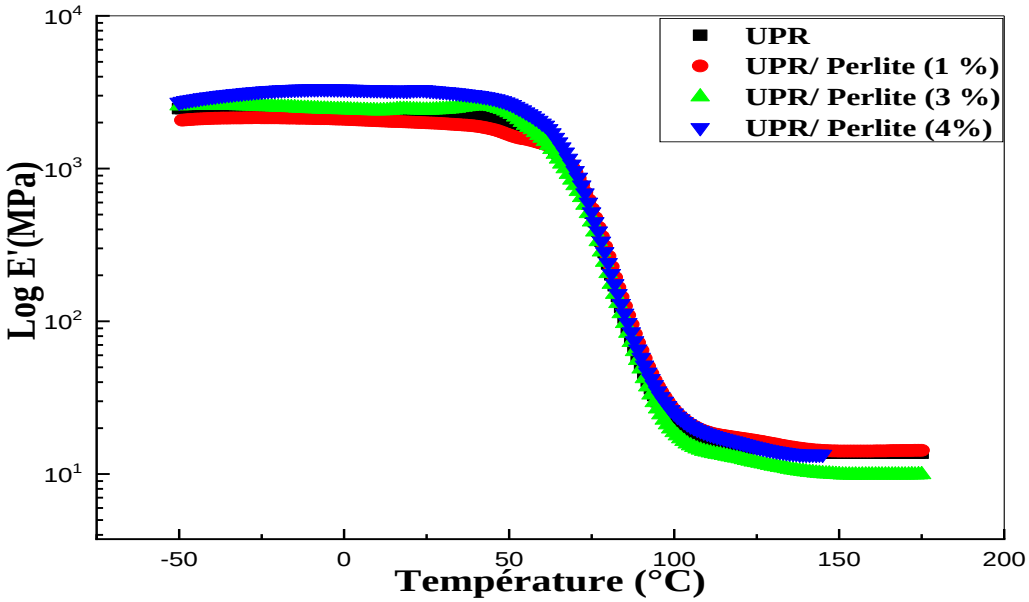

Figure 6: Variations in the conservation module E' of UPR resin and UPR/perlite composites <60µm as a function of temperature and the rate of untreated perlite

The study of the variations in the glass transition temperature of the resin after the incorporation of the perlite is carried out by monitoring the variations of the loss factor tan δ as a function of the temperature and the loading rate as expressed in the (Figure 7**)**. The variations in tan δ show, essentially, a main structural relaxation due to the UPR resin whose Tg, being around 91°C, does not seem to be affected by the addition of perlite. A minor structural relaxation is also detected around 60°C and is also observed during variations in the storage modulus E', for perlite rates of 3 and 4%. By analogy with the UPR/PET fiber composites [44], the demonstration of such a transition suggests the formation of an interface resulting from the favorable interactions between the perlite and the UPR resin. However, due to the lack of affinity between the mineral perlite and the organic resin, it is quite possible that the interactions are of a mechanical nature, resulting from the platelet or flake shape of the perlite, which allows it to anchor in the matrix.

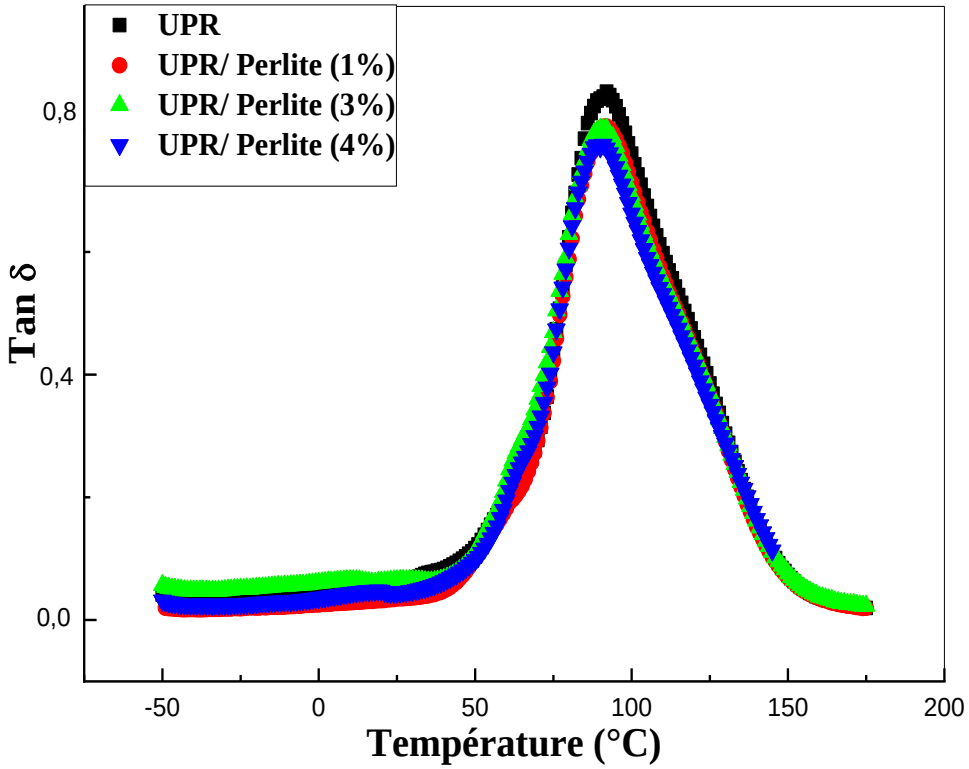

Figure 7: Variations in the factor of loss tan δ of UPR resin and UPR/perlite composites <60µm as a function of temperature and the rate of untreated perlite

Morote-Martinez et al. [39], Pothan et al. [45] et Aziz et al. [46], proposed that the interactions between a reinforcement and a matrix can be evaluated from the values of the maximum of tan δ. Similarly, Martınez-Hernandez et al. [47] et Keusch et al. [48] stated that the intensity of the tan δ peak is inversely proportional to the interface resistance. Furthermore, the width at half maximum of the tan δ peak indicates the volume of the interface [46]. According to the values shown in Table 4, we found that compared to the UPR resin, the values of the tan δ peak maxima of the composites decrease slightly with the addition of perlite. This supports the existence of interactions between these two components and/or the increase in the degree of crosslinking of the UPR resin in the presence of perlite. Furthermore, the increase in the value of the width at half height of the tan δ peak when the concentration of the filler is 4% is equal with the development of a wider interface between the perlite and the UPR resin. This suggests that at this rate, optimal dispersion of the load is achieved and thus supports the results of the mechanical tests.

Table 4 : Tg values, from height to maximum and the width at half maximum of the tan δ peak of UPR and untreated UPR/perlite <60μm composites

Composites	Tg (°C)	Maximum height	Width at half height (cm)
UPR	91	0.86	1.4
UPR/ Perlite (1%)	90	0.80	1.4
UPR/Perlite (3%)	90	0.80	1.4
UPR/Perlite (4%)	91	0.77	1.6

4.1.2.2 Effect of perlite surface treatment

The effect of the surface treatment of perlite with silane on variations in the conservation module E' of the UPR/perlite <60μm composites is shown in (Figure 8). In the vitreous domain, the modulus E' of the composites with the rates of filler of 1 and 3% is not affected at all by the silane treatment, but shows an improvement, relative to the composites with untreated filler after the Tg of the UPR resin. Furthermore, the modulus E' decreases notably for the composite with 4% perlite, and this along the vitreous and rubbery domains. This seems to suggest that the best dispersion is obtained for low concentrations of perlite whose composites show a higher modulus in the vitreous domain. The decrease in modulus in the case of the 4% composite could probably be due to the agglomeration of the filler which distortions the effect of the surface treatment. As for untreated perlite composites, variations in E' show a second minor structural relaxation occurring around 55°C and which affirms to the formation of an intermediate structure which has its own viscoelastic characteristics, different from those of the matrix.

The results of E' are supported by (Figure 9) giving the variations of tan δ as a function of the temperature and the rate of perlite treated and the values of the parameters reported in Table 5. Indeed, we notice that the Tg of the resin UPR is not affected by the treatment and the maximum height of the tan δ peak decreases slightly with the increase in the rate of filler, which confirms the existence of interactions between the UPR resin and the perlite. Furthermore, the volume of the interface decreases with the increase in the treated perlite rate up to 4%, which confirms the formation of charge aggregates when the perlite rate ranges this concentration.

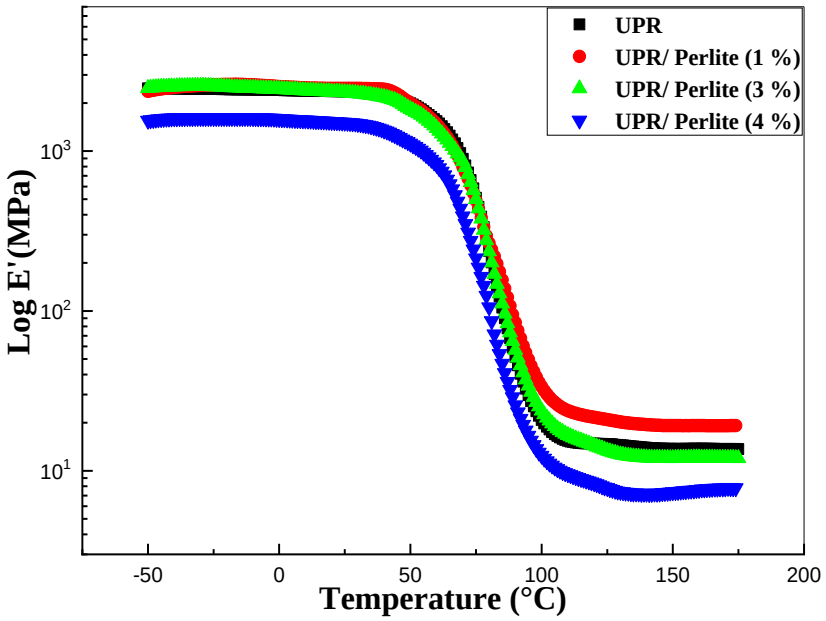

Figure 8: Variations in the conservation module E' of UPR resin and UPR/perlite composites <60µm treated with silane as a function of temperature

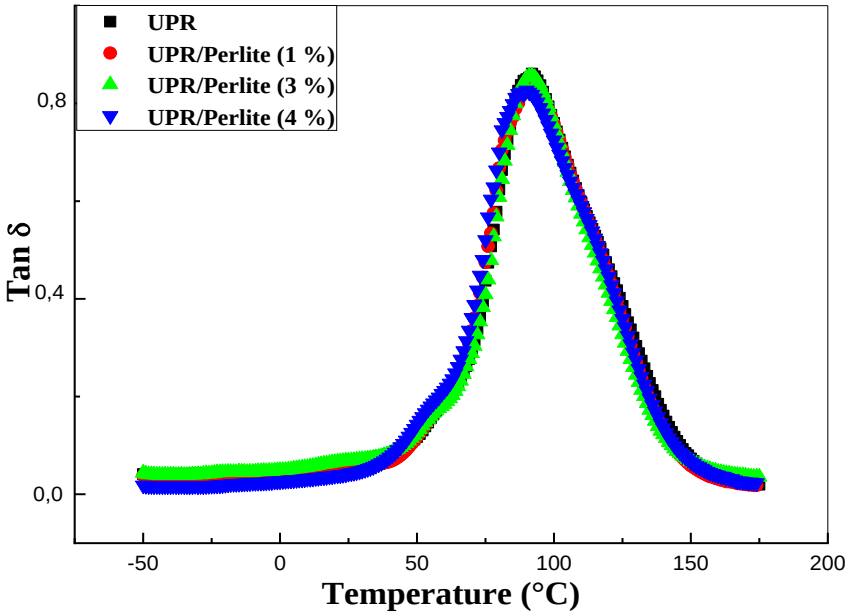

Figure 9: Variations in the factor of loss tan δ of UPR resin and UPR/perlite composites <60µm treated with silane as a function of temperature

Table 5: Tg values, from height to maximum and the width at half maximum of the tan δ peak of UPR and silane-treated UPR/perlite <60µm composites

Composites	Tg (°C)	Maximum height	Width at half height (cm)
UPR	91	0.86	1.4
UPR/Perlite (1%)	91	0.83	1.4
UPR/Perlite (3%)	91	0.85	1.4
UPR/Perlite (4%)	91	0.82	1.3

4.1.3 Thermal properties of UPR/perlite composites

4.1.3.1 Study of post-curing and glass transition processes

The DSC analysis was carried out in order to highlight the influence of the perlite ore particles on the curing behavior of the polyester resin, and to evaluate the evolution of the thermal characteristics of the UPR/perlite composites as a function of the rate of filler and surface treatment. The thermograms representing the first heating cycles of the UPR/perlite composites are given in (Figure 10). They present, first, the glass transition temperature of the UPR matrix, then an exothermic peak corresponding to the post-curing process whose temperature maximum and area closely depend on the rate of perlite contained in the composite. Furthermore, the thermograms of the second heating cycle reveal only the Tg of the UPR resin, without any exothermic peak, which suggests the total hardening of the matrix, as illustrated in (Figure 11).

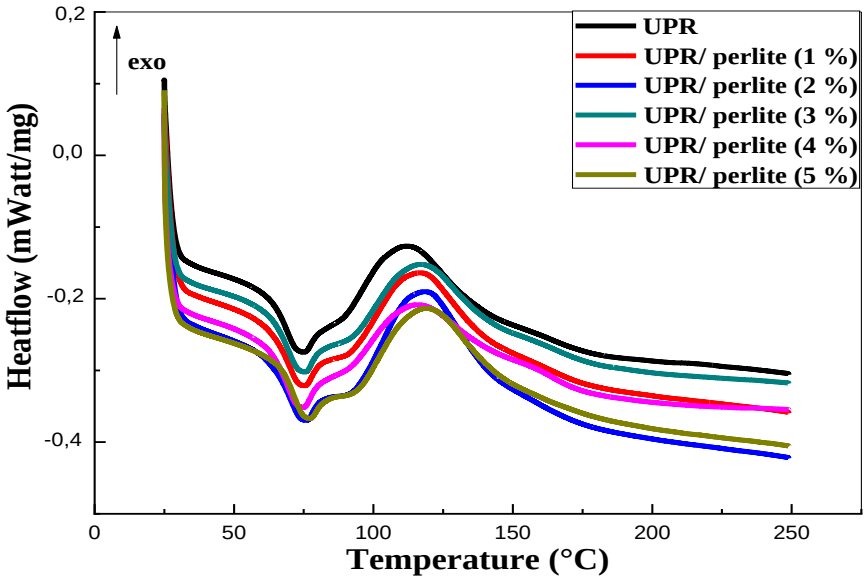

Figure 10: DSC thermograms of UPR resin and UPR/perlite composites <60µm treated with silane (first heating cycles)

Table 6 brings together the values of the maximum temperature of the exothermic peaks and the enthalpy of the post-curing reaction for the pure resin and the UPR/perlite composites. Relative to pure resin, the addition of perlite resulted in a slight delay in the matrix curing

reaction, shifting the exothermic peak to higher temperatures (3 and 4% specifically), as is shown in (Figure 8). Furthermore, the enthalpy of the reaction decreased for concentrations of 3, 4 and 5%, but increased for rates of 1.2% perlite. Same results were noticed by Chieruzzi et al [37] who noted that the addition of clay nanoparticles causes a delay in the curing process and affects the final crosslinking rate of the resin. For 1 and 2% composites, the increase in post-curing temperature and enthalpy reflects slow hardening due to low reactivity of the UPR resin following the addition of perlite. Furthermore, the composites reinforced with 3 and 4% perlite exhibit a reduction in the enthalpy of the reaction and an increase in the temperature at the maximum of the exothermic peak which expresses a great ability of the resin to harden because of the good dispersion of perlite within the matrix. For the composite containing a concentration of 5% perlite, the increase in the reaction enthalpy and the slight decrease in the exothermic peak temperature, compared to the previous composites, reflect a low ability of the UPR resin to harden.

Table 6: Post-curing temperature and enthalpy values evaluated from the thermograms of the first heating cycle of the UPR resin and the UPR/perlite <60μm composites treated with the silane

Composites	$T_{post\text{-}Curing}$ (°C)	$\Delta H_{post\text{-}curing}$ (J/g)
UPR	112	21
UPR/Perlite (1%)	118	26
UPR/Perlite (2%)	118	25
UPR/Perlite (3%)	120	14
UPR/Perlite (4%)	119	11
UPR/Perlite (5%)	117	19

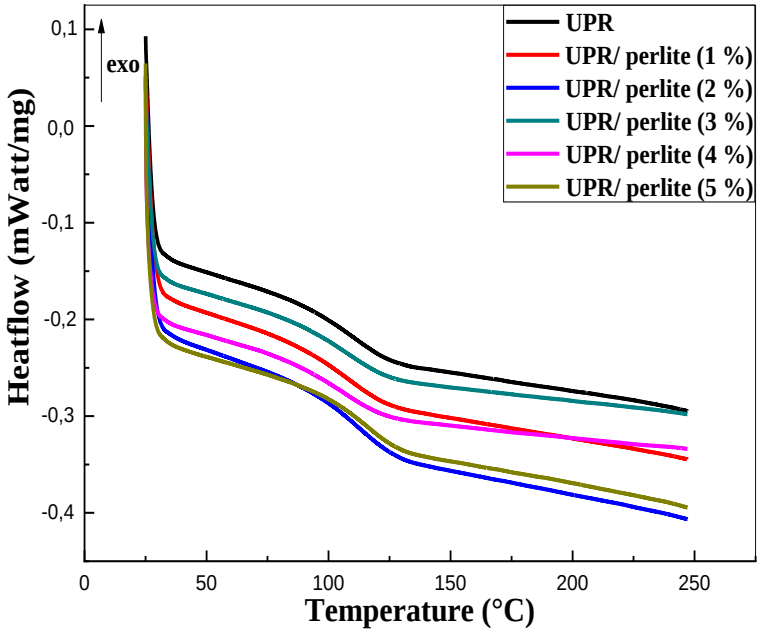

Figure 11: DSC thermograms of UPR resin and UPR/perlite composites <60µm treated with silane (second heating cycles)

The Tg values of the UPR resin in the composites with perlite filler are determined from the thermograms of the second heating cycle and reported in Table 7. Due to the absence of affinity between perlite and UPR, small variations are observed on the Tg of the UPR matrix. This means that in the presence of perlite, the kinetics of the crosslinking reaction of the UPR matrix is considerably disturbed, as was concluded from the thermograms of the first heating cycles; however the total rate of the reaction or the degree crosslinking is only slightly influenced by the presence of the filler and its concentration. Thus, the variations noted in the Tg values can only be attributed to variations in the degrees of crosslinking of the UPR resin in the presence of perlite.

Table 7: Tg values of pure UPR resin and UPR/perlite <60µm composites treated with silane, evaluated from the thermograms of the second heating cycle

Composites	T_g (°C)
UPR	91
UPR/Perlite (1%)	91
UPR/Perlite (2%)	92
UPR/Perlite (3%)	91
UPR/Perlite (4%)	91
UPR/Perlite (5%)	92

4.1.4 Study of the thermal stability of UPR/perlite composites

Figures 12 and 13 represent, respectively, the TG and DTG curves of the matrix and the UPR/perlite composites. Similar to UPR resin, the composites show single-step decomposition.

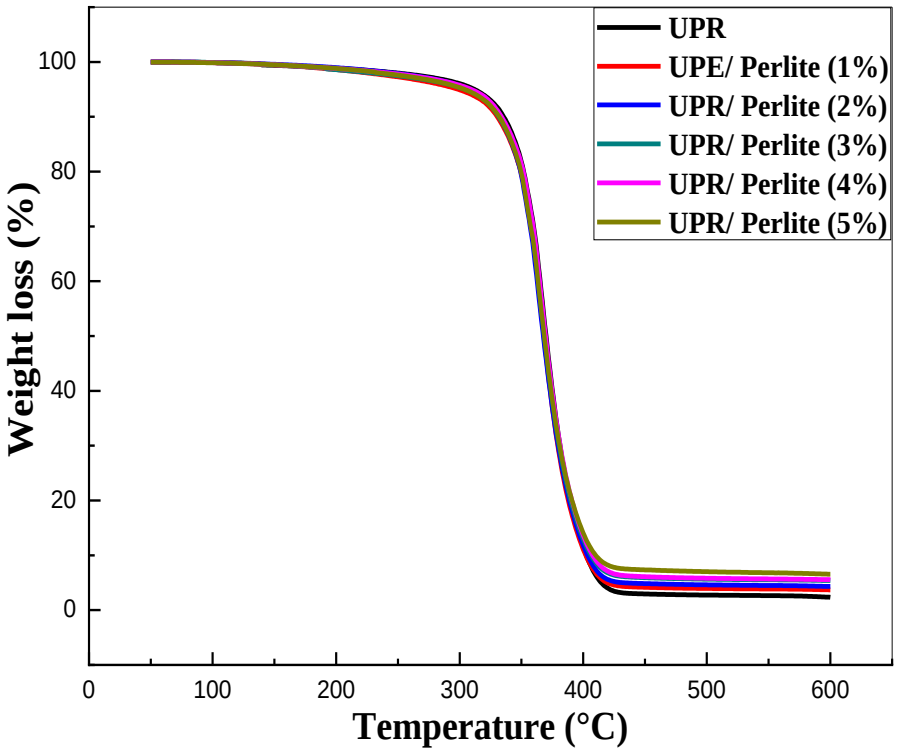

Figure 12: TG thermograms of UPR resin and UPR/perlite composites<60 μm treated with silane

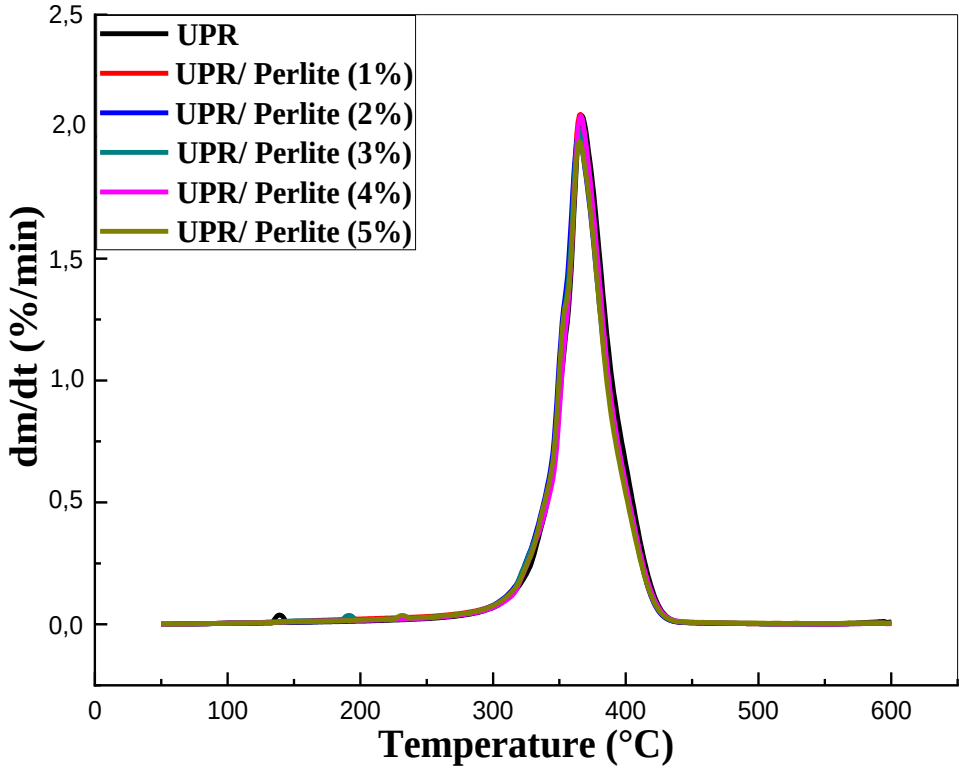

Figure 13: DTG thermograms of UPR resin and UPR/perlite composites<60 µm treated with silane

UPR/perlite composites have thermal stability close to that of pure resin. The values of the degradation parameters, estimated from the TG and DTG curves, are assembled in Table 8. The start of decomposition temperature increases significantly after the addition of perlite, unlike the maximum and end of decomposition temperatures which vary slightly, as shown in Figures 14 and 15.

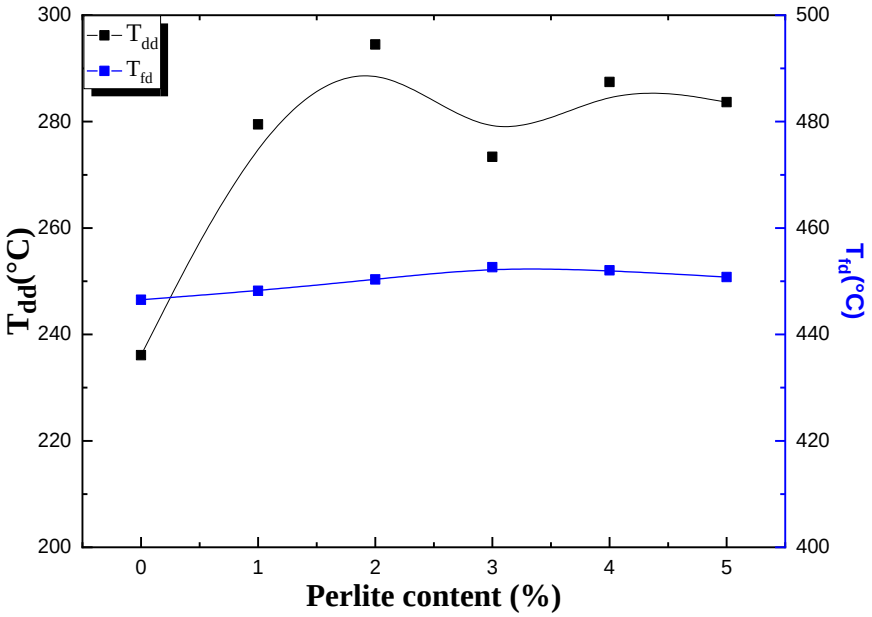

Figure 14: Variations in the start and end temperatures of decomposition of the UPR resin in the UPR/perlite composites <60 µm treated with silane as a function of the rate of charge

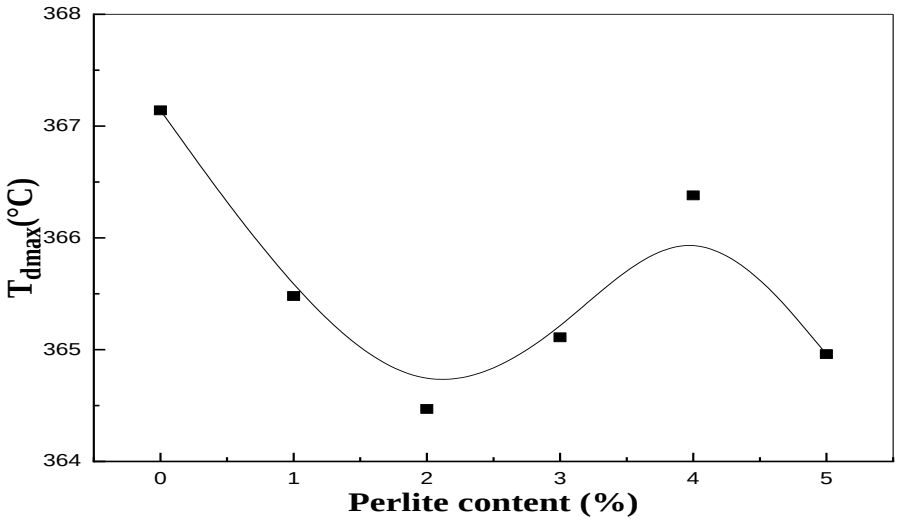

Figure 15: Variations in the maximum decomposition temperature of the UPR resin in the UPR/perlite composites <60 µm treated with the silane as a function of the rate of charge

Table 8: Values of decomposition parameters of UPR resin in UPR/perlite composites <60 µm treated with silane

Rate of filler (%) / Parameter	0	1	2	3	4	5
T_{dd} (°C)	236	279	295	273	287	284
T_{fd} (°C)	447	448	450	453	452	451
T_{dmax} (°C)	367	365	364	365	366	365
V_d (% min)	2,10	2,09	1,99	2,02	2,08	1,98
Loss of mass at T_{dmax} (%)	56	54	55	53	57	60
m_{res} (%)	3	3	4	6	6	7

5 Conclusion

The characterization of the UPR/perlite composites as a function of the rate of filler, the particle size and their surface treatment by the silane coupling agent made it possible to attract certain conclusions. Thus, the infrared analysis did not make it possible to highlight interactions between the filler and the resin, even after the surface treatment with silane, but a slightly remarkable intensification of the band characteristic of the vibrations of the hydroxyl groups was observed.

The filler (perlite) used in this study has the particular characteristic, relatively to minerals already incorporated in the polymers, contain very important pores that can allow it to exfoliate under certain conditions, and show a high specific surface. The access of the liquid resin at the pores of the filler might help develop sufficient interaction to obtain new composites with acceptable performance, which is the main objective of our work.

DMA measurements showed that the filler platelets are well inserted into the matrix and contribute to the mechanical strength of the composites, and this is because "the conservation moduli are higher than that of the resin for rates of 3 and 4% " and we also have " the decrease in the intensity at mid-height of the tan δ peaks made it possible to deduce the existence of a fairly strong UPR/perlite interface" which improves the properties of these composites. Another thermal analysis confirmed that the incorporation of this mineral does not affect the thermal stability of the polyester resin. DSC thermograms showed that the exothermic peak is removed to higher temperatures, due to a delay in the curing reaction caused by the presence of the perlite particles. so we can say that the performances of this material are improved and we have a resistant and at the same time light composite material and also the quality/price ratio increases (economic side). The substitution of traditional fibers such as glass, carbon fibers, etc. Due to the mineral fillers, perlite is interesting from an economic and environmental point of view. We were interested in valorizing this local mineral filler, perlite. Several aspects of this work deserve to be developed subsequently.

These include preparing composite materials with particle sizes less than 60μm; Use other chemical treatment of this filler to increase adhesion and specific surface area, Production of hybrid composites with PET mats and particle reinforcement. Perlite-reinforced unsaturated polyester resin (UPR) composites have been widely used in industrial applications such as marine, automotive, pipeline systems and construction industries due to their low cost, high chemical resistance and good workability.

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