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Recycling of carbon fibers inserted in composite of DGEBA epoxy matrix by thermal degradation

Gleice Guevara Matielli Rodrigues, Jane Maria Faulstich de Paiva, Janaína Braga do Carmo, Vagner R. Botaro*

Universidade Federal de São Carlos, UFSCar Campus de Sorocaba, Rod. João Leme dos Santos, Km 110 – Bairro Itinga, Sorocaba, SP, CEP 18052-780, Brazil

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ABSTRACT

In this study, carbon fibers inserted into an epoxy resin derived from diglycidyl ether of bisphenol-A (DGEBA) were recovered based on the complete removal of the matrix by thermal treatment. The degradation of the composite was carried out at various experimental conditions and the ideal one was obtained at 450 °C for 2 h. Under this condition, all of the resin was removed without degrading the C fibers to any significant degree. The results of thermogravimetric analysis revealed several stages of thermal decomposition depending on the material analyzed. It was established by thermogravimetry analyses that the epoxy matrix starts to experience intense degradation at temperatures above 280 °C with maximum loss in the temperature range of 360–410 °C. In the case of composite samples above 800 °C a residue of approximately 50% by weight can be observed, which is related to carbon fibers. Confirmation of the removal of the resin from the carbon fiber was obtained by means of scanning electron microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The kinetic of release of CH_4, CO_2 and N_2O gases during degradation process was accompanied by gas chromatography. The concentrations of methane and carbon dioxide increased significantly during the first 10 min of the processing and after this, no released quantities were verified. On the other hand N_2O continues to be released even after 3 h of the process of burning the composite in air atmosphere. The treatment proposed in this study can be considered to be a low-cost one as well as being interesting for the recovery of C fibers present in DGEBA matrix which would normally be discarded after use.

1. Introduction

Polymers reinforced with carbon fiber are engineering materials of significant commercial interest, mainly in the automotive, aerospace and sporting goods sectors. The main factors responsible for such a wide range of applications are the low density coupled with the resistance to corrosion and the reduced cost [1,2]. The development of the technology of composites has been boosted by the constant need to reduce weight in aircraft and space structures [3]. In the literature, there are not many papers which describe the recovery of reinforcement agents present in composites of thermoset matrices.

According to Yang (2012) [4], the carbon fiber is already recovered by Boeing based on the pyrolysis process. This recovered fiber has been tested in non-structural aircraft components [5]. Another company that is also a part of AFRA (Aircraft Fleet Recycling Association) is Embraer (Empresa Brasileira de Aeronáutica S.A.) which became a member of the association in July 2011. In addition to AFRA there is also the PAMELA-LIFE (Process for Advanced Management of End of Life Aircraft) project which was created by Airbus in 2005 with the same aim of coming up with a sustainable end for aircraft which are no longer in use. Like Boeing, Airbus uses the pyrolysis system to extract the carbon fiber from the polymer matrix. However, the greatest challenge at present is to set up a market for the recycled material [4].

Therefore, the recycling of carbon fiber from polymer composites is a very topical theme [6]. A few studies related to this topic can be found in the literature: characterization of carbon fiber recycled using supercritical n-propanol [7,8], recycling of the fiber with water in supercritical conditions [9,10], recycling with various fluids under supercritical and subcritical conditions [11,12], recycling of carbon fiber using pyrolysis [1,13,14], use of the coupled thermogravimetric technique and mass

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* Corresponding author. Tel.: +55 1532296019; fax: +55 15 32296000.
E-mail addresses: gleice.guevara@gmail.com (G.G. Matielli Rodrigues), jane@ufscar.br (J.M. Faulstich de Paiva), jbcarmo@ufscar.br (J. Braga do Carmo), vagner@ufscar.br (V.R. Botaro).

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spectrometry (TG-MS) to investigate the oxidative decomposition of the carbon fiber composite containing epoxy resin [15], and recycling of carbon fiber for structural applications [16].

It is observed that the increase in the application of thermoset polymer reinforced with carbon fiber in the industrial segment is generating a marked growth in the quantity of waste material, both that which is removed during the manufacturing processes, as well as the component that remains at the end of the item's useful life [7].

In Brazil, according to the Resolution of the National Environment Commission (CONAMA n° 358/2005), it is the responsibility of the party that generates the waste material to administer it from its origin until its final disposal. The State Waste Policy (PNRS), State Law nº12,305/10 of August 2010 also regulates the polluter pays principle, under which the management of waste matter is the responsibility of those who generate it.

In the case of the composites containing thermoset polymer matrices, recycling is a problem due to the fact that these matrices, after the curing process, formed crossed links which prevent the polymer from being reshaped. As a result of this, the most common procedures in terms of the final disposal of the thermoset items are as follows: storage, co-processing, incineration and landfill. Storage is a form of disposal that may be limited by the amount of space that the generator has available for storage of the material. This procedure is generally only used as a provisional measure. Co-processing consists of the reutilization of waste matter in furnaces producing cement, and in Brazil this activity is licensed by CONAMA Resolution 264/1999. In this process, the waste matter can be used as a raw material provided that it exhibits a composition similar to that employed in producing cement. In addition to this, the waste matter can also be used as a substitute for fuel associated with reutilization purposes. The disadvantage of using this process as a final destination for the waste matter, is that the generator does not gain any profit from the process in so far as he has to pay for the removal of the material, for the cost of the co-processing and has to make arrangements with the state's environmental body for the transport document known as CADRI (Certificate of Movement of Waste Matter of Environmental Interest).

In Brazil it is permitted to dispose of carbon fiber in landfills provided that, in accordance with ABNT NBR 10004:2004, this material is classified as class II B waste, in other words, not dangerous and inert. However, when one opts for one of these procedures to dispose of the carbon fiber, the generator faces a double loss, to the extent that he pays to dispose of a material that has a high cost. Recycling is the way of disposing of waste material that produces the greatest benefits for the environment as it helps to reduce soil, air and water pollution, extends the useful life of the landfills, generates revenues by means of the sale of the recyclable items and encourages competition to the extent that recycled products are sold alongside those which are produced using virgin raw material. One of the objectives of national law number 12305/10, which refers to the National Solid Waste Policy, is to encourage the recycling industry, with a view to fostering the use of raw materials and inputs derived from recycled and recyclable materials. In addition to this, as a management guideline applicable to solid waste this law has the following order of priority: non-generation, reduction, reutilization, recycling, treatment of the solid waste and environmentally appropriate final disposal of the waste. In this study, carbon fibers inserted into an epoxy resin derived from diglycidyl ether of bisphenol A (DGEBA) were recovered based on the complete removal of the matrix by thermal treatment in atmosphere.

2. Experimental

2.1. Presentation and preparation of the raw material

2.1.1. Matrix

The matrix is an epoxy resin derived of Diglycidyl ether of bisphenol A; 2,2-Bis (4-glycidyloxyphenyl) propane commonly named DGEBA. These resins are thermosetting polymers and are used as adhesives, high performance coatings and potting and encapsulating materials. These resins have excellent electrical properties, low shrinkage, good adhesion to many metals and resistance to moisture, thermal stability and mechanical strength [15]. The epoxy resin employed in this study is a viscous liquid one and present a molar mass around 700 g mol^{-1}. The n parameter: relation between molar quantity of epichlorohydrin and bisphenol A is around 0.15.

2.1.2. Reinforced agent: carbon fibers

The properties of the carbon fiber (CF) used in the preparation of the composite are presented in Table 1. The first stage of the process used for recycling the carbon fiber consists of breaking up the piece in a grinder type mill with high speed blades.

Table 1
Properties of the carbon fiber (CF) studied for this research.

Characteristics	Unit	Typical values
Density	g/cm^3	1.76
Tensile strength	MPa	3950
Elastic modulus	GPa	238
Elongation at break	%	1.7
Specific heat capacity	J/kgK	710
Thermal conductivity	W/mK	17
Thermal expansion coefficient	10^{-6}/K	−0.1
Specific electrical resistance	Ωcm	1.6×10^{-3}

2.1.3. Fragmentation of composite

The starting material used in this study was obtained from cylindrical parts of continuous parallel carbon fiber composite produced by filamentary winding. A slicer mill with rotation 1000 rpm was used for composite fragmentation. Fig. 1(a and b) shows details of some composite fragments after mechanical degradation.

The fragments were classified by vibratory screening using a sequence of screens with the following openings in Tyler/Mesh: 5, 10, 16, 20, 50, 60 and 100. The screening time was one of 1 h.

2.2. Thermal treatments of the carbon fibers and composites

The thermal stabilities of carbon fibers of origin (control, CF) and comminuted composites of epoxy matrix were evaluated by treatment using various temperatures and times in air atmosphere. The CF samples were subjected to temperatures of 450, 500, 600 and 700 °C for 3 h. These samples were labeled FC450, FC500, FC600 and FC700 respectively. The initial and final mass were determined in order to calculate the percentage of volatiles released during the thermal treatment. The percent volatile (% V) was calculated by the equation (Eq. (1)):

$$\%V = \frac{\left(m_i - m_f\right)}{m_i} \times 100 \qquad (1)$$

Where: m_i is the initial mass of fibers; and m_f is the final mass after treatment.

The equipment used was a common laboratory muffle kiln adapted to include a gas collector at the equipment's upper vent.

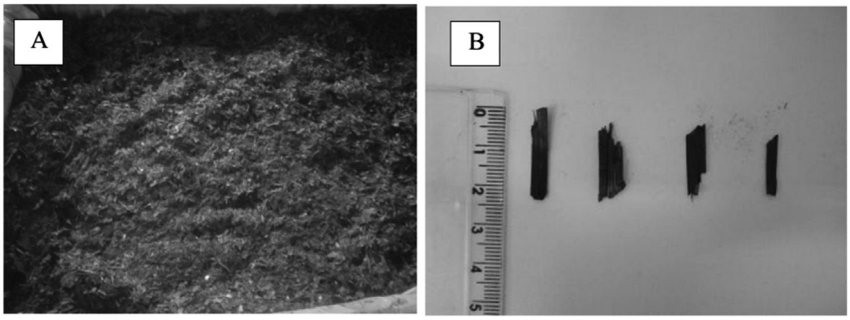


Fig. 1. Composite of carbon fiber (CF) and epoxy resin after decharacterization, where: a) Assembled fragments; b) Detailed photo of a few fragments.

The composites were subjected to thermal treatment for different durations and at different temperatures, in order to find the condition with the greatest possible removal of epoxy resin without causing any pronounced thermal degradation in the fibers. These tests were monitored by SEM and FTIR. In addition, important parameters such as shorter heating time with less expenditure of energy were also taken into account in order to select the ideal condition. The experimental conditions of the degradation of the composites are listed in Table 2. The C letters correspond to the composite epoxy resin denomination, while the intermediate numbers describe the temperature of the test and the last digits describe the degradation time applied.

2.3. *Kinetic monitoring of the release of gases during the thermal treatment of the composite*

The gases released during the thermal treatment of the composite were analyzed by gas chromatography. The gases were collected by upper vent of the muffle kiln with the help of a 5 mL plastic syringe with a system that allows for it to be opened in order to collect the gas and closed so that the gases do not escape from the syringe. During the thermal treatment which is carried out at a temperature of 450 °C, the gases collected were injected into a 25 mL glass flask with a septum seal. The samples were collected in triplicate. The gas chromatograph used is a Shimadzu 2014, with an ECD type detector for N_2O and an FID type one for CH_4 and CO_2. The carrier gas used for the chromatographic analyzes was Helium. The column operated at 80 °C, the ECD at 325 °C and the FID at 250 °C. The stationary phase of the column used was the polymer ethylvinylbenzene–divinylbenzene, the trade name of which is “Porapak Q”, which is supplied by Sigma–Aldrich (Beckett and Stenlake, 1988). Datailed methodology is found in Carmo et al. [17].

2.4. *Scanning electron microscopy (SEM)*

The surface of the sample analyzed by the scanning electron microscope was scanned using a beam of electrons. The reflected beam of electrons was collected and then, exhibited at the same scanning rate on a cathode ray tube. The image on the screen represents the characteristics of the surface of the sample. The SEM used for this task is an XL 30 model Phillips one. Typical parameter employed were Electron Microscope using a 1.2 kV accelerating voltage and 6.5 mm working distance.

Table 2
Experimental parameters of the thermally treated carbon fiber and epoxy resin composites.

Identification of the composite	Temperature (°C)	Time (h)
C200-2	200	2
C300-2	300	2
C400-2	400	2
C450-2	450	2
C700-1	700	1

2.5. *Thermogravimetric analysis (TGA)*

The thermogravimetric analysis of the composite was carried out on the Netzsch equipment, model STA/C7. The test was performed in a synthetic air atmosphere at 20 ml/min gas flow rate. The ratio of heating used for the tests was one of 10 °C/min and the heating interval ranged from 25 °C to 800 °C. About 2–3 mg sample was used.

2.6. *Fourier transform infrared spectroscopy (FTIR)*

The infrared spectroscopy analyses made it possible to observe the presence of residual polymer resin in the recycled carbon fiber. In addition to this, it helped with the observation of characteristic bands of the PU and epoxy resin samples. The samples were analyzed by ATR in the transmission mode between 400 and 4000 cm^{-1}, with the accumulation of 128 scans by means of a JASCO brand spectrometer.

3. Results and discussions

3.1. *Results of the granulometric classification of the comminuted composite*

The use of the C fibers inserted in the epoxy matrix composite was possible after the material had been comminuted in a specific mill. The comminuted material was classified by screening and the results are presented in Fig. 2.

The results of Fig. 2 show that the comminution process of the composite was efficient if we take into account the low emission of small particles, of less than 100 mesh. This fact is significant, as it shows that in general the degradation process preserved the C fibers, generating fibers with of lengths that are interesting from the point of view of reutilization.

3.2. *SEM analyses*

3.2.1. *Carbon fibers (CF)*

According to the images in Fig. 3 one can observe the existence of impregnated material between the carbon fibers of origin (CF). The small particles observed on the surfaces are due to the presence of the denominated “size” used to protect the fiber against external damage and to provide greater adhesion with the polymer matrices. It could be observed that the average diameter of the carbon fibers of origin was one of 7 μm.

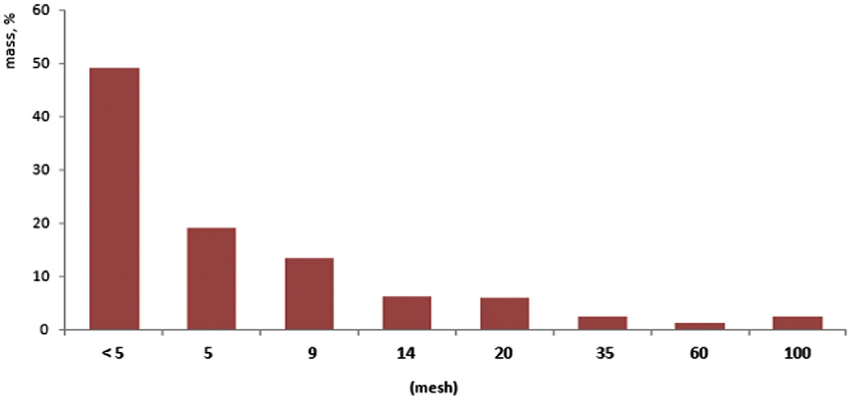


Fig. 2. Results of the classification process of the comminuted composite.

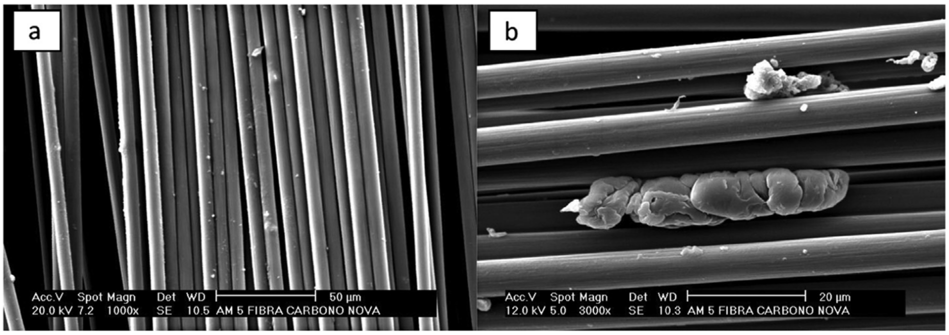


Fig. 3. Micrographs of SEM surface of the carbon fibers of origin amplified 1000× (a), 3000× (b).

The thermal treatment proposed for the composite should prevent degradation of the C fibers and promote the complete removal of the matrix. Scanning electron microscopy was an important technique for deciding what would be the best time and temperature to use for recovering the C fibers. Fig. 4(a), (b) and (c) show the SEM micrographs for the fibers heated for a period of 3 h at temperatures of 450, 500 and 600 °C respectively, amplified 6000×.

The treatments carried out at 450° C and 500 °C showed similar results with regard to the presence of additive on the surface of the fibers and in relation to the preservation of the integrity of the said fibers (Fig. 4a and b). The C fibers recovered after burning at these temperatures are completely preserved with regard to degradation. On the other hand, the burning of the fibers at 600 °C caused a significant surface degradation for a number of fibers, which can be seen from the detail highlighted in Fig. 4(c). In this sense, thermal treatment of the composite at temperatures exceeding 500 °C should be avoided. This is the temperature limit for structural preservation and consequently for the reutilization of the recycled C fiber by the proposed treatment.

3.2.2. *Comminuted composite without thermal treatment*

After the fragmentation or grinding, the composite is in the shape of small irregular pieces which can be visualized in detail by using the micrographs presented in Fig. 5. By means of the images it is possible to verify that the carbon fibers are arranged in a parallel manner and enveloped in epoxy resin. In addition to this, it is possible to observe in details (Fig. 5d) the appearance of the resin after it has been broken up during the fragmentation process.

3.2.3. *C fibers after burning of the composite*

The SEM analyses showed that 450 °C was the lowest temperature at which it was proven that a large part of the epoxy resin was eliminated in the form of gases (Fig. 6). Fig. 6a and b shows that the fibers are free of matrix after the burning. To confirm the data

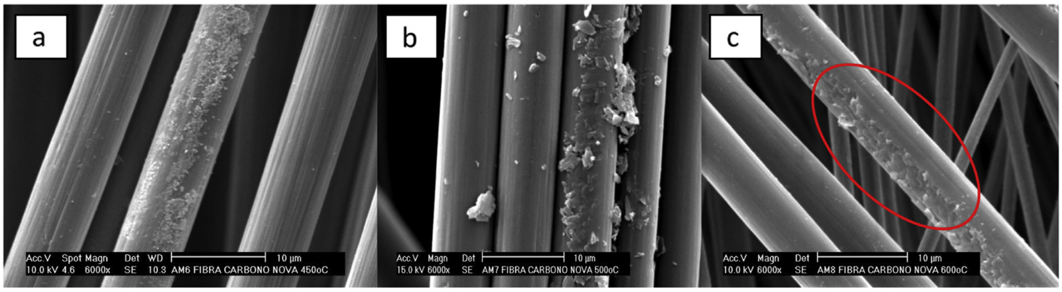


Fig. 4. Micrographs of SEM surface of the carbon fibers of origin heated for a period of 3 h at 450 (a), 500 (Bb) and 600 °C (c) amplified 6000×.

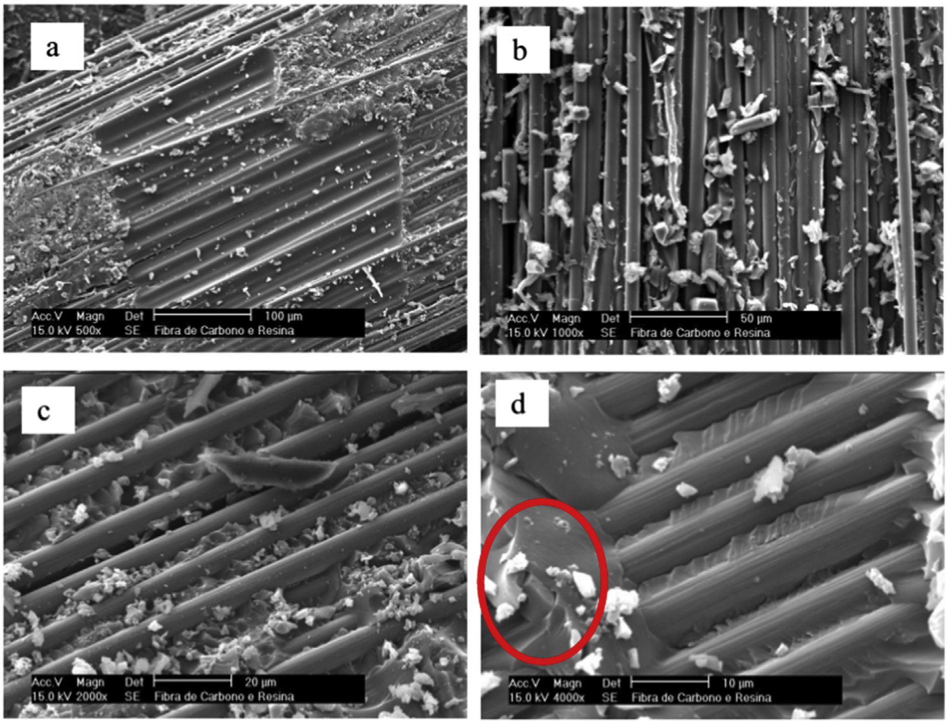


Fig. 5. Micrographs of SEM surface of the fragmented composite (a) amplified500×, (b) amplified 1000×, (c) amplified 2000× and (d) amplified 4000×.

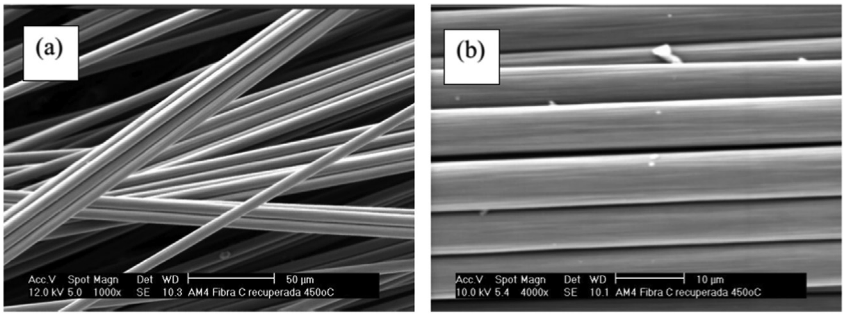


Fig. 6. Micrographs of SEM surface for the waste product generated after burning by the C450-2 sample amplified 1000× (a) and 4000× (b).

obtained by SEM, the mass difference before and after thermal treatment tests were performed. In these tests, samples of about 1.000 g of C-fibers were subjected to heaters 450, 500, 600 and 700 °C for 3 h in an oven. The percentage by volume was calculated according to Eq. (1). Results showed that the mass losses were 4, 29, 90 and 100%, respectively. This interesting result showed that the fibers treated at 450 °C small amount of mass lost after thermal treatment, only 4%, which may be related to evaporation of additives only. As a result, coupled with the fact that the temperature of 450 °C did not cause damage to the C fibers as pointed by SEM (Fig. 4a) this was the temperature chosen for recycling the fibers.

SEM analysis of the residue after burning generated by the C700-1 fiber showed that the residue was completely eliminated at this temperature. This very marked degradation in the matrix was already expected given that the resin was eliminated at even lower temperatures (Fig. 4 a). In addition to this, details of the micrograph of Fig. 7a show that the C fiber presented marked degradation after burning at 700 °C. A likely cause is that the carbon fiber of origin utilized in the composites is PAN based with carbonization treatment and is therefore more sensitive to thermal degradation than the graphitized fibers. This fact was confirmed in the analysis of volatile content by gas chromatography, which will be discussed later.

3.3. *Thermogravimetric analysis (TGA)*

3.3.1. *DGEBA resin*

The thermal stability of the DGEBA epoxy resin cured with methyl tetrahydrophthalic anhydride was analyzed by TGA. The TGA curves presented in Fig. 5 show that the degradation of epoxy resin exhibits two stages. The first, which occurs initially at 268 °C, is attributed to the breaking down of the non-reacted epoxy resin or other traces of impurities from the cured DGEBA. Above 410 °C, there is the stage of greater loss of mass which is due to the thermal degradation of the cross-links of the cured DGEBA [18]. The maximum points on the derived curve occur at approximately 343 °C and 513 °C for the respective events (Fig. 8).

The literature describes exactly the same behavior for thermal degradation of DGEBA resin. According to El Gouri [20] the pure DGEBA sample began to lose its weight at about 300 °C and degraded in the range of 300–450 °C with around 50% weight loss and little residue remained at above 600 °C.

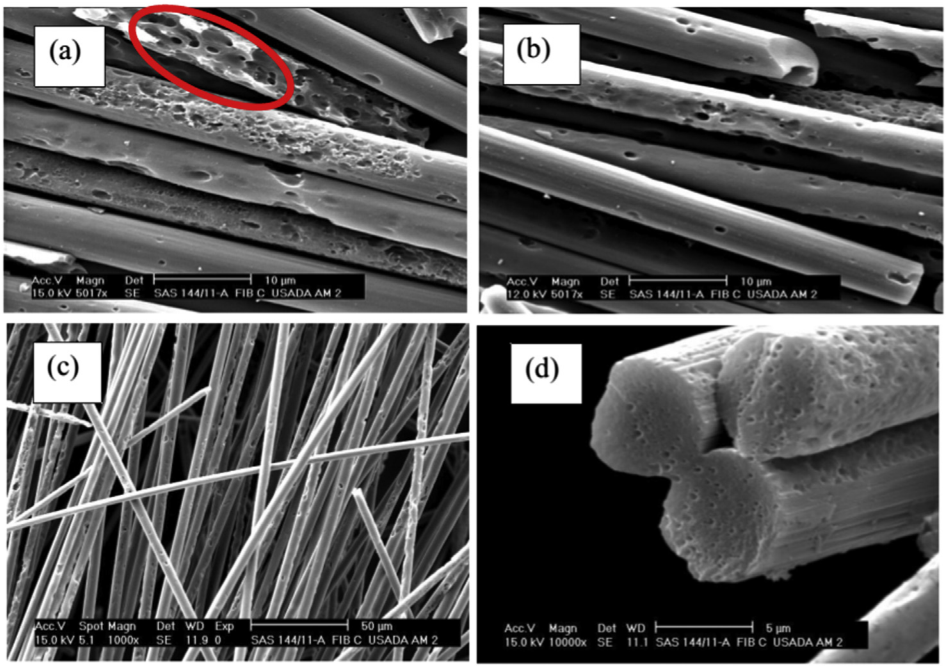


Fig. 7. Micrographs of SEM surface for the waste product generated after burning by the C700-1 composite sample amplified 5000× (a) and (b), amplified 1000× (c) and amplified 10000× (d).

3.3.2. Comminuted composite without thermal treatment

It is noted that the derivative of the composite's TGA curve (Fig. 9) presents two peaks, at 360 °C and 530 °C, which relate to the epoxy resin as was discussed earlier in the presentation of the result of the resin's TGA curve (Fig. 8). The third loss of mass which initially occurs at roughly 600 °C relates to the oxidization of the carbon fiber.

Therefore, it can be seen that the degradation of the composite presents three typical stages which begin at 280, 450 and 600 °C. In their paper, Dao et al. (2012) [19] described the four regions of the result of thermal analysis of epoxy resin composite reinforced with carbon fiber. The thermal behavior observed by the author is very close to the results obtained by means of this study. According to Dao et al. (2012) [19], the first phase which occurs at between 0 °C and approximately 280 °C consists of the thermal decomposition of the epoxy resin which is characterized by the segmentation of random chains and of terminal chains which result in the loss of hydrogen atoms and other organic groups having low molar mass. The second phase (from approximately 280 °C–450 °C) is attributed to the acceleration in the thermal decomposition of the epoxy resin during which there is the formation of carbon chain.

These by-products of the epoxy resin are broken down into gases and porous carbon chain. The third phase (between approximately 450 °C and 610 °C) consists of the second stage of thermal decomposition. During this phase the thermal decomposition of the remaining epoxy resin takes place. The fourth phase which gets underway at 610 °C corresponds to the oxidization of the carbon chain formed during the combustion of the epoxy resin and the decomposition of the carbon fiber. In addition to this, the TGA result for the composite shows that the inclusion of carbon fiber, which exhibits a greater resistance to thermal degradation than the matrix, increased the resin's thermal stability. According to the TGA curve shown in Fig. 10, the residual carbon after the burning was one of approximately 52% at 800 °C, while the residual pure epoxy resin was one of 0% from above roughly 600 °C, according to what is shown in Table 3.

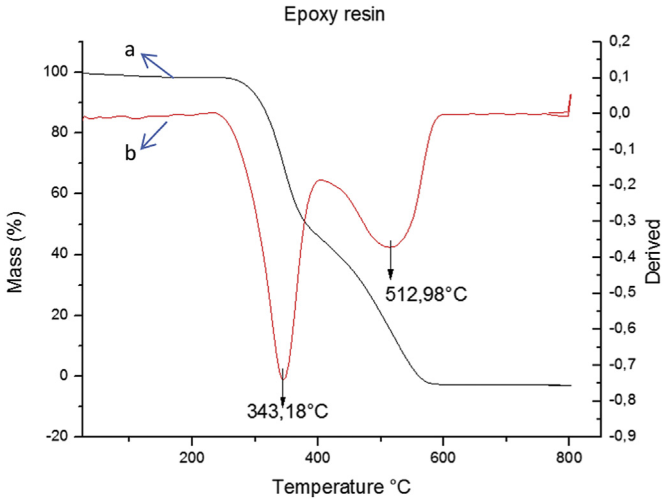


Fig. 8. TGA (a) curve for the sample of DGEBA resin cured with methyl tetrahydrophthalic anhydride and derived curve (b).

3.3.3. C fibers after burning of the C450-2 composite

The result of the thermogravimetric analysis for sample C450-2 can be observed in Fig. 10. The carbon fibers resist exposure to temperatures in excess of 400 °C better than the matrix and on account of this fact decomposition begins to occur at temperatures above roughly 530 °C. Even above 800 °C, a residue of approximately 45% can be observed, associated with the non-degraded C fiber part.

3.4. FTIR

3.4.1. Composite without thermal treatment

A partial molecular structure of the DGEBA is presented in Fig. 11 so that each functional group present in the formula can be observed [21].

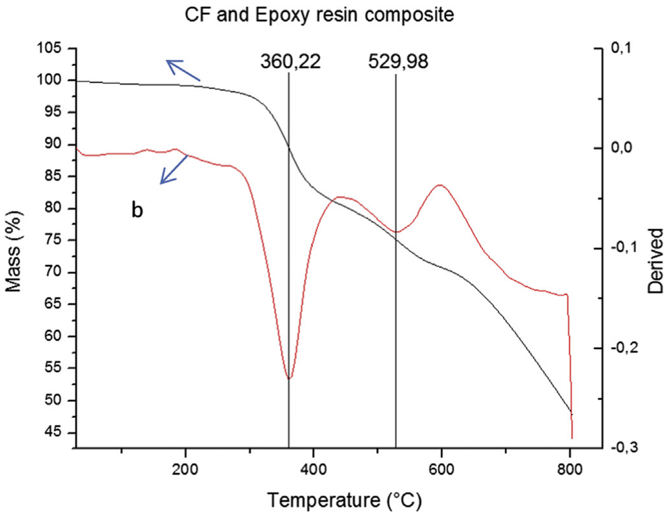


Fig. 9. TGA (a) curve for the epoxy resin and carbon fiber composite and derived curve (b).

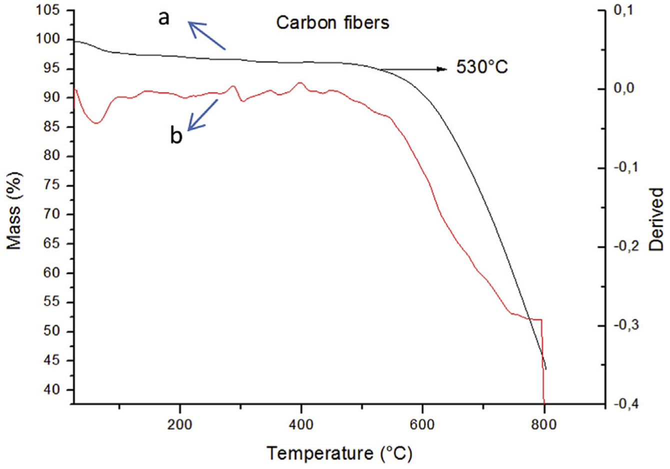


Fig. 10. TGA curve (a) for C fibers obtained after burning of the C450-2 sample and derived curve (b).

Table 3
Temperatures of loss of mass for the main events found for the DGEBA resin and composite.

Sample	Temperature of 15% loss of mass (°C)	Residual C at 400 °C (%)	Residual C at 600 °C (%)	Residual C at 800 °C (%)
Epoxy resin	323	55	0	0
Epoxy resin and carbon fiber	385	82	70	52

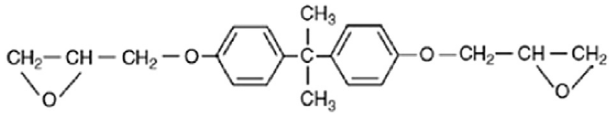


Fig. 11. Partial structure of the DGEBA resin used as matrix of the C fiber composites.

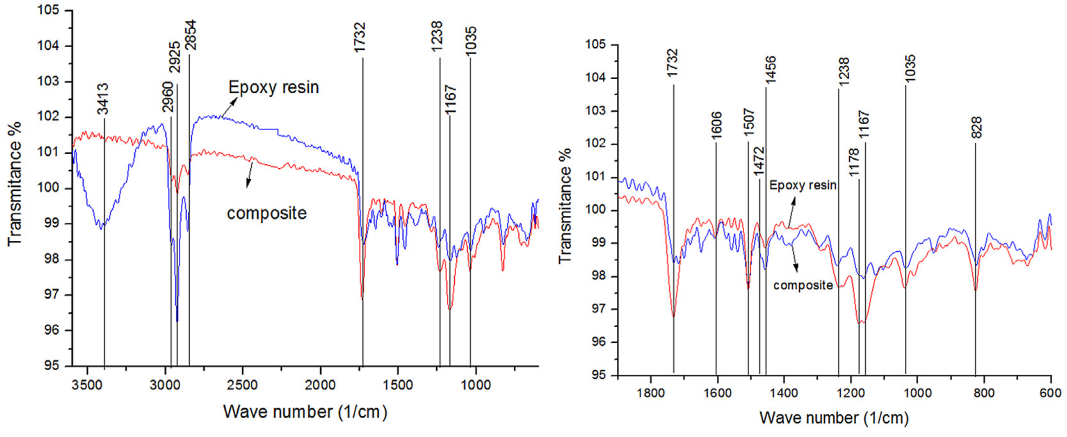
The FTIR spectrum of the carbon fiber and of the DGEBA epoxy resin composite is presented in Fig. 12. Since the matrix is cured it is not possible to observe the characteristic band of the axially asymmetric deformation of the DGEBA's C—O—C ring, in the region between 950 and 850 cm^{-1}. In this deformation, the C—C links spread out while the C—O links contract [22]. The bands in the regions of 2925 and 2854 cm^{-1}relate, respectively, to the methyl and to the methylene. They are axially asymmetric and symmetric deformations, respectively, for C—H of methyl and methylene groups. In the case of the axially asymmetric deformation, two C—H links spread out, while the third one contracts. In the case of the axially symmetric deformation the C—H links spread out and contract during the process [22,23]. The absorption band in 1732 cm^{-1} refers to the axial deformation of the C=O of the ester resulting from the reaction of the anhydride with the epoxidic group which can be observed in the amplified spectrum of Fig. 12(B).

According to Meure (2010) [21], the aromatic carbons are responsible for the band at approximately 830 cm^{-1} due to the vibration of angular deformation outside the level of the C—H links. The aromatic rings can be observed in the structure of the DGEBA epoxy resin, as in Fig. 11. The absorption band resulting from the angular deformation in the C—H level of the aromatic compounds is observed at roughly 1180 cm^{-1}and the absorption band in the region between 1230 and 1250 cm^{-1} refers to the aromatic ethers Aryl—O—CH_2 due to the asymmetric angular deformation of CH_2 [21]. It can be observed that in the absorption bands in the region between 1456 and 1507 cm^{-1} there is axial deformation of the ring's C=C links, covered by the symmetrical angular deformation in the CH_2 level at 1472 cm^{-1} [21,22]. At 3413 cm^{-1} a band is observed resulting from the axial deformation of O—H in intermolecular hydrogen link [22].

3.5. *Kinetic accompaniment of the release of gases by gas chromatography*

In the literature there are few studies reporting degradation mechanisms of epoxy resins. Koyama et al. [23] describes the optimum pyrolytic conditions for thermosetting epoxy resin samples. In conclusion, various phenols characteristic of the prepolymers and pyrolyzates characteristic of the reacted curing agents were observed on the pyrograms of the cured resin samples. The epoxy prepolymers and the curing agents used in certain cured resin systems could be readily identified from these characteristic pyrolyzates. Epoxy prepolymers and epoxy resin samples cured with and without curing agents, such as diamine and dicarboxylic acid anhydride, were studied using high resolution pyrolysis-gas chromatography. The main results for uncured resin show characteristic pyrolyzates, such as isopropenylphenol, bisphenol A and monoglycidyl ether of bisphenol-A that were observed in addition to the intense peak of the original monomer on the pyrogram of DGEBA. The low boiling pyrolyzates with earlier retention times involve ethylene oxide, acetaldehyde, acrolein, acetone and ally1 alcohol, which are expected to be formed mostly from the epoxide end groups. For DGEBA resins cured, low boiling pyrolyzates related to the epoxide end groups and various phenols, such as phenol, isopropenylphenol and bisphenol-A, which are characteristic of resin, and are commonly observed. Pyrolyzates such as m-xylene and m-methylbenzylamine, characteristic of the reacted curing agent (m-xylenediamine) are observed.

In this work, the release of CH_4, N_2O and CO_2 was accompanied by gas chromatography (Fig. 13). The main observation shows that the concentrations of methane and carbon dioxide increase until roughly 10 min after the start of the thermal degradation of the composite at 450 °C in air. Later on, there is a decrease in the intensity of release of these gases. The epoxy matrix starts to experience intense degradation at temperatures above 280 °C with maximum loss at approximately 360 °C, as can be seen from the



(A) (B)

Fig. 12. A: Comparison between absorption spectrums in the infrared region of the DGEBA epoxy resin (in blue) and the carbon fiber and DGEBA epoxy resin composite (in red). B: FTIR of the region amplified between 1850 and 600 cm^{-1}. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

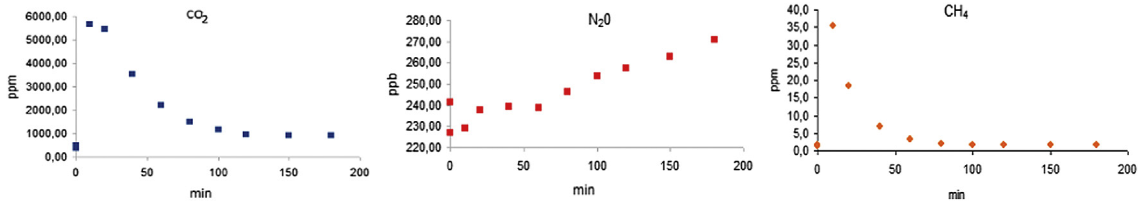


Fig. 13. Kinetic of the release of CH_4, N_2O and CO_2 gases during the burning process of the composite.

TGA curves (Fig. 9). In addition, the degradation of the epoxy matrix was also confirmed by SEM (Fig 7a and b). As described previously, the segmentation of random chains and of terminal chains of epoxy resin results in the loss of hydrogen atoms and other organic groups having low molar mass [19].

The increase in the nitrous oxide (Fig. 13) shows that the 3-h long thermal treatment was not enough to release all the nitrogen present in the sample in the form of oxide. This gas originates from the oxidization of the nitrogen present in the structure of the C fiber produced from the carbonized PAN based resin which is not graphitized [24].

4. Conclusions

According to the analyses that were carried out the most favorable condition for the recycling of the carbon fiber using the system proposed in the study was that of the sample treated at 450 °C for 2 h. Under these conditions, all the resin was removed without degrading the fiber to any appreciable degree. The treatment proposed in this study can be considered to be a low-cost one as well as being interesting for the recovery of C fibers present in DGEBA matrix which would normally be discarded after use.

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