

COMPARATIVE STUDY OF THE USE OF NON-IONIZING AND IONIZING RADIATION IN THE CURE OF EPOXY RESIN: MICROWAVE VERSUS ELECTRON ELECTRON

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ABSTRACT

Several processes for curing epoxy resins were developed over the years. Two methods are discussed in this paper, in order to present the main advantages and disadvantages of using microwave radiation (non-ionizing radiation) and electron beam radiation (ionizing radiation). The microwave radiation is a non-ionizing radiation, with great power of penetration and transfer of heat in microwave absorbing materials, or materials with microwave absorbing fillers. The frequency usually used in research and development is 2.45 GHz, the same available in commercial equipments. The "microwave effect" provides increase on the collision velocity between the reactant which, combined with energy absorbed by the reaction system, accelerates the curing reaction. None modifications in the epoxy system are required to use microwave heating for the curing process. On the other hand, the electron beam is a form of ionizing radiation in which the high energy electrons have the ability to interact with the irradiated material and produce ions, free radicals, and molecules in excited state, which can be used to initiate and propagate a polymerization. Specific initiators are necessary for an effective cure of the resin. In this study, a DGEBA epoxy resin with initiators based on anhydride and amine was used under the same conditions indicated by the manufacturer. The curing of the catalyzed system was performed in a domestic microwave oven adapted for laboratory use. The degradation and glass transition temperatures were evaluated by thermal analysis techniques. For comparative purposes, it was used data available in the literature for electron beam irradiation.

1. INTRODUCTION

Nowadays, some composite systems using epoxy matrices cured by the traditional process, in ovens and kilns, require a long time to cure and an established logistics. On systems using epoxy resins and anhydride based hardeners, such as filament winding process, for example, a longer working time (pot life) is required and a cycle of curing that can get to 16 hours and

reach a high temperature, typically between 100°C and 200°C. In the process of manufacture of polymeric structural composite, raw materials, molds, the consumption of electricity and manpower are important factors in the cost of the production. This fact has led to the application of new technologies in the production process to ensure the competitiveness of produced materials [1]. Searching new solutions for process optimization without loss of quality as well as the energy saving, several unconventional processes of curing epoxy resin were developed over the years. Two methods are discussed in this paper, in order to present the main advantages and disadvantages of using microwave radiation (non-ionizing radiation) and electron beam radiation (ionizing radiation).

The use of microwave irradiation began after II World War, with the invention of RADAR. The microwave radiation is a non-ionizing radiation, with great power of penetration and great transfer of heat in microwave absorbing materials, or materials with microwave absorbing fillers. The frequency usually used in research and development is 2.45 GHz, the same available in commercial equipment. For curing epoxy resins are not necessary changes in the system reactive with specific initiators sensitive to microwaves. The "microwave effect" provides increase on the collision velocity between the reactants, which combined with energy absorbed by the reaction system, accelerates the reaction curing, allowing the resin to cure long pot-life (times greater than 24 hours) in minutes [2].

In contrast, the first application of electron beam for curing epoxy resin began in 1979 with the use for curing composites by the european company Aeroespatale, for application in rocket engines. The electron beam is a form of ionizing radiation in which the high energy electrons have the ability to interact with the irradiated material and produce ions, free radicals, and molecules in excited state, which can be used to initiate and propagate a polymerization. Specific initiators are necessary for an effective cure of the resin. The infrastructure required for electron beam irradiation is considerable, and the dimensions of the parts to be irradiated are also important. For thick parts and/or with higher density, higher energies are involved for complete cure of the resin, and consequently high power is a requirement for the irradiator [1].

In this study, a DGEBA epoxy resin with initiators based on anhydride and amine was used under the same conditions indicated by the manufacturer. The curing of the catalyzed system was performed in a domestic microwave oven adapted for laboratory use. The degradation and glass transition temperatures were evaluated by thermal analysis techniques. For comparative purposes, it was used data available in the literature for electron beam irradiation.

1.1. Curing Process by Electron Beam Irradiation

The study of the curing process of polymer composites by electron beam comprises the steps of selecting the matrix polymer and cationic initiator, study of process parameters such as dose rate and total dose, making manner appropriate to the process and the study of the interface between the fiber and matrix. The high-energy electrons to interact with the material irradiated generate ions, free radicals and molecules in the excited state able to initiate and propagate a polymerization. Depending on the type of resin, the polymerization may occur by free radical mechanism or ionic. For the epoxy acrylic resins with curing reaction induced by electron beam, polymerization occurs via free radicals, whereas for epoxy resins with cationic

initiator (diaryliodonium and triariliodônio) occurs through cation. Some works indicates that an epoxy resin of the diglycidyl ether of bisphenol A (DGEBA) type and diaryliodonium hexafluor antimonate as the cationic initiator presents a cure cycle time for the electron beam curing polymeric composite of 40 minutes to complete an approximated total dose of 200kGy. For this same system, the glass transition temperature, determined by a dynamic mechanical analyzer (DMTA), was 167°C, and the degradation temperature, determined by a themogravimetric analyser (TGA), was 360°C [1].

In the polymerization by free radicals, the double bond-containing oligomer doesn't need initiators when irradiated by electron beam. The formation of free radicals occur in acrylates or methacrylates contained in the epoxy oligomers. These oligomers are widely used in the composition of coatings and adhesives, however, they have some negative aspects, such as low glass transition temperure, low fracture resistance, and contraction up to 8% when cured in thick layers. These factors prevent the manufacture of structural composites, for example [3].

In cationic polymerization, commercial epoxy resins based on DGBA, DGBF, Novolacs, cicloaliphatics can be cured by electron beams and provide materials with higher glass transition temperatures and mechanical properties similar to or slightly lower than the same resin cured by thermal process. This curing process requires the addition of 1% to 3% of a cationic initiator with respect to the weight of resin to be irradiated with high energy supplied at controlled rates, in order to initiate polymerization and crosslinking. The electron beam curing is performed at ambient temperature, however, the heat of the curing reaction can raise the temperature of the matrix until it reaches approximately 90 °C [4].

The infrastructure for installation of a electron accelerator, for epoxy curing process, with electron beams can be considered relatively complex. Fig. 1 is presents an image with internal parts of a irradiator by electron beams installed at IPEN (Nuclear and Energetic Research Institute) in São Paulo.

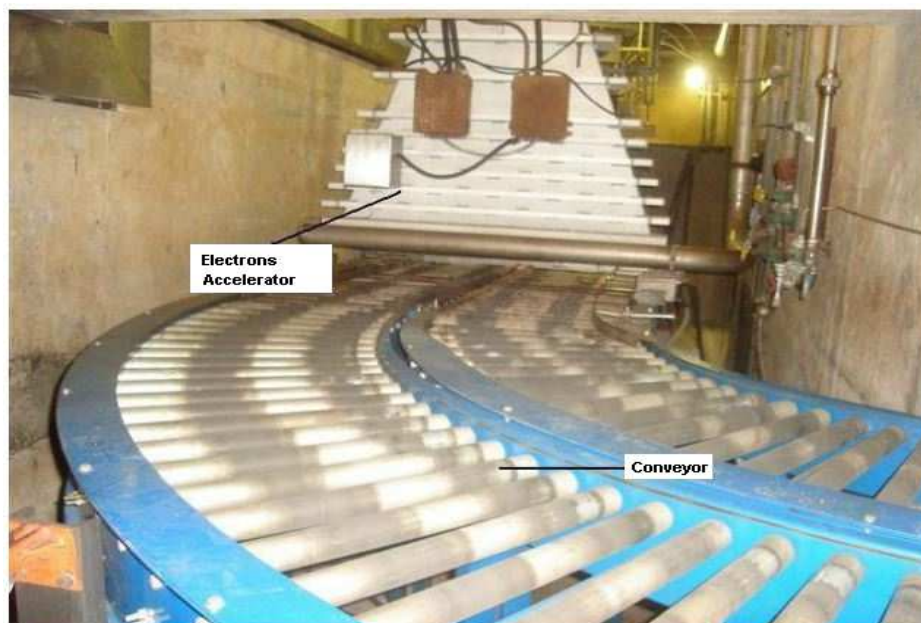


Figure 1: View of internal parts of an irradiator of electron beam (IPEN) [5].

The parameters of the equipment operating at IPEN are presented on Table 1. The electron accelerators are the sources for electron beams for curing of epoxy resins. In all types of accelerators, electrons are produced in a cathode heated and maintained in a region of highest potential. These electrons are accelerated by a potential difference applied between the cathode and anode. The electrons acquire enough energy to get through the exit window which consists of a thin sheet of titanium with a thickness between 20µm and 40µm.

Table 1: Irradiation Parameters for the Equipment Install at IPEN [5].

Parameters	Values or Range (Unit)
Electrons Energy	0,5 – 1,5 MeV
Potency	37,5 kW
Current	0,01 – 25mA
Scanning Width	60 – 112 cm
Dosage by Step	0,5 kGy
Irradiation Area	0,6735 cm ² (near by 10cm of passage)

The entire region of production and acceleration of electrons must be maintained in high vacuum to allow focus and accelerate the beam toward the window appropriately. The energy of the electron beam is calculated as a function of the current in high voltage divider accelerator, taking into account the thickness and density of the target material (material to be irradiated). The energy is calculated for electrons to move across the entire target material, so that the doses at the point of input and output are equal [1].

Curing by electron beam has some advantages over thermal curing, such as [6]:

- The reduction of the contraction of the epoxy matrix from 4% to 6%, to 2% to 4%;
- The composites, metallic, and ceramic substrates can be glued with adhesives that cure by electron beam, allowing manufacture large parts or structures;
- The manufacture of large composite that can not be cured in ovens or autoclaves, because of the limitations of the dimensions of these equipment;
- The increase in glass transition temperature using the same resin;
- The reduction of internal stresses, since this process is held at room temperature;
- The reduction of the generation of volatiles is not used as hardeners and accelerators in its formulation;
- The reduction in costs for the manufacture of molds, since it is not necessary to use materials resistant to high temperature;
- Reducing the consumption of electrical energy due to reduced time of the cure cycle; and
- The working time or pot life of the formulation is long since protected from ultraviolet radiation. The formulation may be stored at room temperature for a period of up to two years.

1.2. Curing Process by Microwave Irradiation – Microwave Heating

The mechanism of heating via microwave is independent of the thermal conductivity of the irradiated material and offers a good solution to operation with materials which do not have a good thermal conductivity, such as polymers. This relatively new form of energy transfer offers several distinct advantages of heating with kilns or with conventional ovens:

- It's highly selective;

- It's fast (almost instantaneous), and
- It's controllable.

The energy transmitted via microwave has a large wavelength compared to other radiations, and offers a greater penetrating power than other radiations, such as ultraviolet or electron beam. Despite this advantage, the frequency of microwave energy is not enough to provide the breaking of chemical bonds, promoting only dissipative heating rate. This leads to a quick distribution of energy through the volume of the material [1].

The various mechanisms of energy transfer in heating ovens also result in several new challenges for materials processing. As energy is transferred by electromagnetic field, any non-uniformities in the electromagnetic field will result in uneven heating. Furthermore, to the extent that different types of materials are processed, physical and structural changes occur that affect their dielectric properties. Pronounced changes in the capacity of microwave absorption by the material can cause difficulties with the process modeling and control. Understanding the interaction of materials with microwave is an important step in the processing. The processes of energy transfer between molecules, in absorbing materials, can be of two kinds: rotation of dipole, and/or ionic conduction. For high frequency radiation (such as ultraviolet, for example), the dipoles cannot follow the changes of the electric field, and for very low frequencies (such as radio waves, for example) the molecular reorientation is much faster than the switching fields. For both cases, the heating of the material will not occur. For frequencies between these two limiting cases, the electric dipoles follow the changes of the electric field and part of the electromagnetic energy is stored, turning into heat due to friction with the dipoles of neighboring molecules [8].

The heat from the rotation of dipoles relates to the alignment of the molecules (which has permanent or induced dipoles) with the applied electric field. When the field is removed the molecules return to a disordered state, and the energy that was absorbed to this guidance in these dipoles is dissipated as heat [2].

For the ionic conduction, the heating is generated by friction that occurs through the migration of ions when dissolved under the action of the electromagnetic field. This generation depends on the size, charge, and conductivity of dissolved ions, and interaction of the latter with the solvent. Unlike the rotation mechanism of the dipole, the temperature rise during the heating of the material also results in increased mobility of ions and, consequently, the efficiency of heating by the ionic conduction mechanism [8].

Since the beginning of the first research on the use of irradiation of microwaves for chemical synthesis, substantial differences were noted in the types of products produced, quantity, and reaction rate of the reactants. These differences led to speculation about the occurrence of effects "specific" and "non-thermal" heating by microwaves. As an example of these effects, in addition to faster reaction and higher efficiency, there are reactions which do not normally occur with conventional heating but with irradiation with microwaves starts to occur. There were also observed changes in selectivity in reactions when the microwaves are employed [2].

Wittig Reactions (Wittig-Emmons-Horner), in organic synthesis, have been conducted in domestic microwave oven in the absence of solvents. A remarkable fact observed in this case is that the reaction of a steroid derivative has not occurred using the traditional procedure

(reaction solution under reflux and conventional heating.) Another reaction where no products are obtained by conventional heating is the saponification of 2,4,6-trimethyl benzoic acid octyl. But with the use of microwave, the reaction was developed with the formation of the corresponding acid with in good efficiency [2].

Currently it has been known that in most cases the reactions assisted by microwave are influenced essentially by thermal and kinetic. In other words, the responses obtained via irradiation of microwaves are a result of high temperatures reached very quickly. There is still controversy about the existence of "specific effect", which is related to reactions to results different from those obtained by conventional routes, and effects "non-thermal", caused by direct interaction of the electric field and its influence on the chemical transformations. The major problem of the study of these three effects, thermal, specific and non-thermal, is that two or three of them occur at the same time being responsible for the observed phenomenon, making a more clear and objective extremely complex [9].

The cure reaction by irradiation with microwave, in general, follow the same steps observed in other healing processes, considered the proper proportions for epoxy polymer systems. From a certain energy level, the initial reactants collide with each other in a particular orientation that favors the formation of an activated complex, higher energy. The difference between the initial level and the level achieved by the activated complex is called the activation energy, which is the energy that the system must absorb the reaction medium for chemical reaction to occur. After the time that the state of higher energy level is reached, the reactants are converted into products quickly, reducing the energy level to a new state. The existing differential with respect to healing via microwave irradiation is that the microwaves do not alter the activation energy, but increase efficiency in energy transfer. Thus, an energy transfer with most intense and localized, it is more easily established the time for the reagents to reach the energy barrier required for the reaction, increasing reaction rate compared to conventional heating [11].

The microwave irradiation transfers energy in 10^{-9} s for each cycle of electromagnetic energy used. The kinetics of relaxation to the molecules involved, so that the energy level is approximately 10^{-5} s. This means that energy is transferred more rapidly (almost immediately) than the molecules can relax, resulting in an out of balance condition and high temperatures, thus affecting the speed of the system as a whole. The lifetime of the activated complexes is approximately 10^{-13} s, which is shorter than the rate of energy transfer microwave.

However, some activated complexes are stabilized by resonance and their life times become larger, around 10^{-9} s, which is the length of each energy transfer cycle. In these reactions, the more stable activated complexes are decomposed and formed at the same time, further enhancing the effect "microwave". Many of the activated complexes have a more polar character and other ionic, making them excellent candidates for the transfer of microwave energy [11].

Fig. 2 presents the two main kind of equipment used for P&D.



Figure 2: Kind of microwave irradiator, a domestic adapted model [10] and an industrial model [11].

Considering the equation of Arrhenius, the rate of a reaction depends on two factors: the number of effective collisions between molecules, and the fraction of molecules that reach the minimum energy to surpass the activation energy barrier. The incidence of microwave increases the likelihood of effective shock, its influence will be clearly noticed. Some authors believe that the microwaves affect the orientation of the molecules to the collision and the effective activation energy of the process, but there are still no concrete evidence. However, it is known that the microwaves affect the reaction temperature [12].

Curing by microwave process has some similarities with thermal curing:

- The process with microwaves uses the same epoxy system used in thermal curing, without changes in the quantities of hardeners or accelerators;
- The kinetics of reactions involved in microwave curing and thermal curing are similar; and
- The infrastructure used for microwave curing is similar to that used in thermal process, allowing the utilization of both processes at the same time, in complementary ways.

On the other hand, the curing process by irradiation of microwaves presents some advantages over thermal curing, such as [13-15]:

- The increase in the glass transition temperature and the degradation temperature using the same resin with microwave process. For the same resin, an increase between 50 and 100 degrees are observed;
- The mechanical performance of the microwave-cured epoxy under tension, compression, and flexure is in close comparison (and sometimes even better) to the thermally cured epoxy; and
- Reducing the consumption of electrical energy due to reduced time of cure cycle (thirty times faster and twenty times more energy-efficient, in some cases).

2. EXPERIMENTAL

2.1. Materials

For the present work, one type of epoxy-anhydride-amine system, normally used for curing on kilns, or ovens, was chosen: bifunctional epoxy – diglycidyl ether of bisphenol A (DGEBA), methyltetrahydrophthalic anhydride (MTHPA), and benzyldimethyl amine (BDMA). It's a epoxy system with a long pot-life, generally used for the production of composites materials by filament winding and manual lamination (hand lay-up, for example).

The resin, hardener, and accelerator were provided by Araltec Ltda., a Huntsman distributor. The commercial codes for the resin (DGEBA), hardener (MTHPA), and accelerator (BDMA) are MY750, HY917, and DY062, respectively. The proportion used in this study was 100:85:0,5 (by weight) for resin, hardener, and accelerator, according to the suppliers. The chemical formulae of the epoxy formulation is presented in Fig. 3.

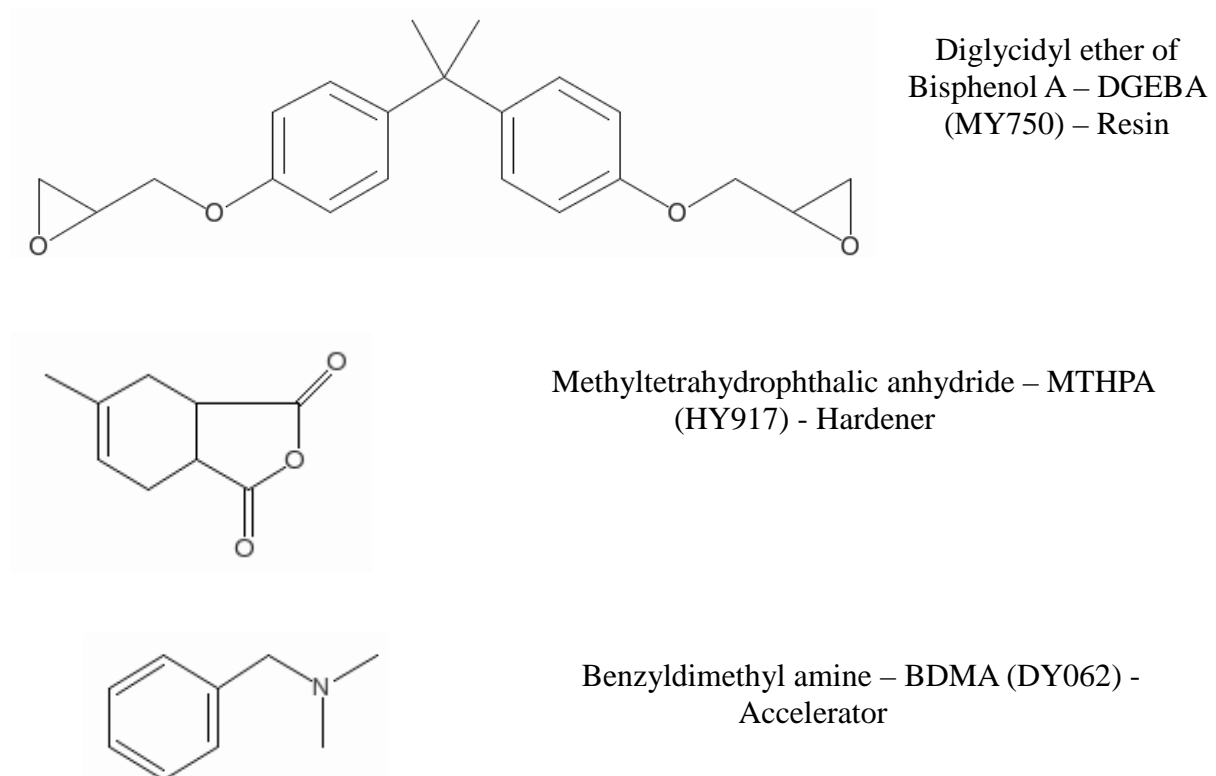


Figure 3: Chemical formulae of components of the epoxy system (MY750/HY917/DY062).

2.2. Methods

2.2.1. Sample preparation

After good mixing, air bubbles were released from the samples before they were poured into a mold. The absorbers were added after the preparation of the epoxy system, in the quantities above indicated. A circular polypropilene mold (6,5cm diameter X 0,5 cm depth) was used for microwave curing.

Microwave curing was performed in a Continental MOCT022SD2A1BR commercial microwave oven at a frequency of 2.45 GHz. This microwave oven was calibrated to facilitate the processing. It consisted of 10 power levels, with the maximum theoretical level equal to 800 W. In this work, the applied power was based on the physical performance of the cured samples.

No air bubbles and no burning were the criteria for good specimens. The microwave cure schedules were optimized using different power–time combinations, taking utmost care to minimize the appearance of localized hotspots in the samples.

2.2.2. Calibration of power levels of the microwave

The calibration procedure used was that described in Pecoraro [16 e 17]. The power output of the magnetron can be determined indirectly by measuring the temperature increase, in degree Celsius, for 1 liter of water heated for 2 minutes. Through Equation 1, the values of output power can be determined:

$$P = C_p K \Delta T m / t \quad (1)$$

where, P is the power absorbed by the water (Watts); K is the conversion factor from Calories to Watts; C_p is the heat capacity (cal. °C); m is the sample mass (g); and t is the time (s). The accuracy of measurements depends on locating the sample in the same place inside the cavity and the use of the same container.

2.2.3. Determination of glass transition temperature (T_g)

The glass transition temperature (T_g) of the microwave-cured samples was determined using a differential scanning calorimeter, model DSC 822, by Mettler-Toledo. A common heating rate of 20°C/min was employed for evaluating the T_g values, from 25 to 300°C. In order to prevent risks to the DSC equipment, a previous test in a thermogravimetric analyzer, model TGA/SDTA 851, by Mettler-Toledo, was performed. The degradation temperature of the materials was determined with a heating rate of 10°C/min, from 25 to 800°C.

3. RESULTS AND DISCUSSION

3.1. Calibration of power levels of the microwave

The calibration results are present on the Fig.4. From the graph, the differences between the theoretical value and the measured value of power can be observed. The measured value will indicate the amount of energy being employed to the cure epoxy system with different types and amounts of absorbers. For the theoretical maximum of 800W, the value of 516W was obtained, indicating a loss of the order of 35%.

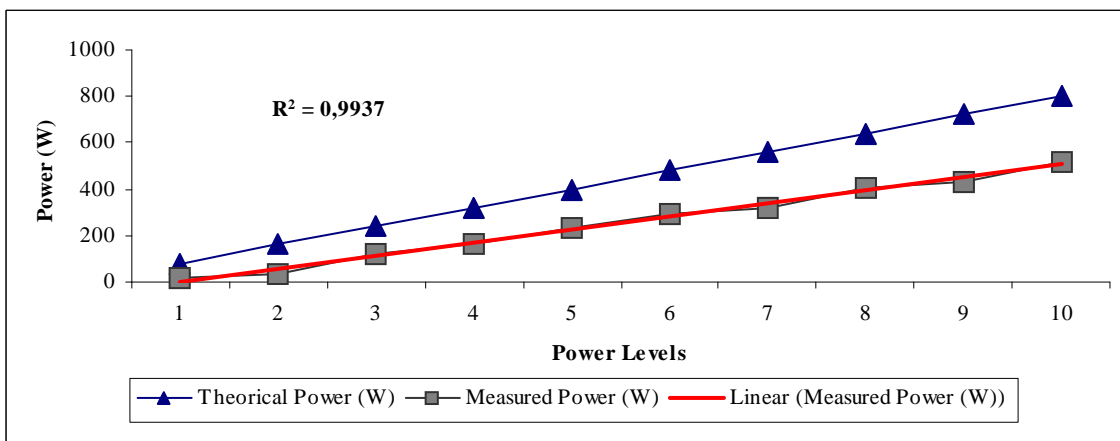


Figure 4: Graphic obtained for calibration of microwave oven.

This value is consistent with other studies that have also made use of domestic microwave adapted for use in the laboratory [16].

3.2. Determination of glass transition temperature (T_g)

The resin processed by irradiation with microwaves was evaluated in a TGA/SDTA in order to determinate the temperature of degradation. This information is an important reference to safe operation in DSC and an indicator to level of crosslinking, observed by the T_g attained by the resin. For some types of epoxy resins, higher values on temperature of degradation suggest a high level of cross-linking, and indirectly, higher values of T_g. [18]. The results obtained follows in the Fig. 5.

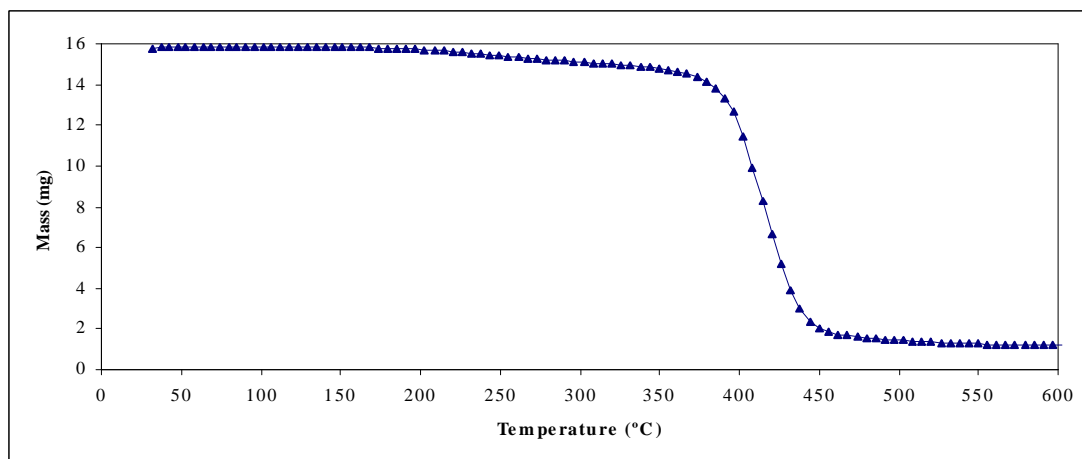


Figure 5: Thermogravimetric Analysis (TGA) of a sample resin irradiated with power level 3.

Most of the samples analysed presents similar results, with few differences in temperature of degradation and final residues. The sample starts to loose weight near 200°C, but only at 350°C the main loss of weight happens. The partial loss of weight between 200 and 350°C is 5,74%. The main loss of weight (85%) occurs between 330-480°C, with 6-8% of final residue. Comparing with the curing process made on ovens and kilns for bisphenol A based resins [18], the curing process using microwaves offers a better thermal resistance. In the

curing process by oven and kilns, the main loss of weight occurs at 200°C, approximately, losing 85% or more on one step.

The determination of the glass transition temperature started analysing the conditions of resin irradiated in four different conditions (power levels 1 to 4). Power levels above 4 were not used because the epoxy resin system studied starts to evaporate, reaching temperatures above 180°C.

The same results, despite of high temperatures observed during the curing process (above 100°C), were observed for the other samples. These results are shown in Fig. 6.

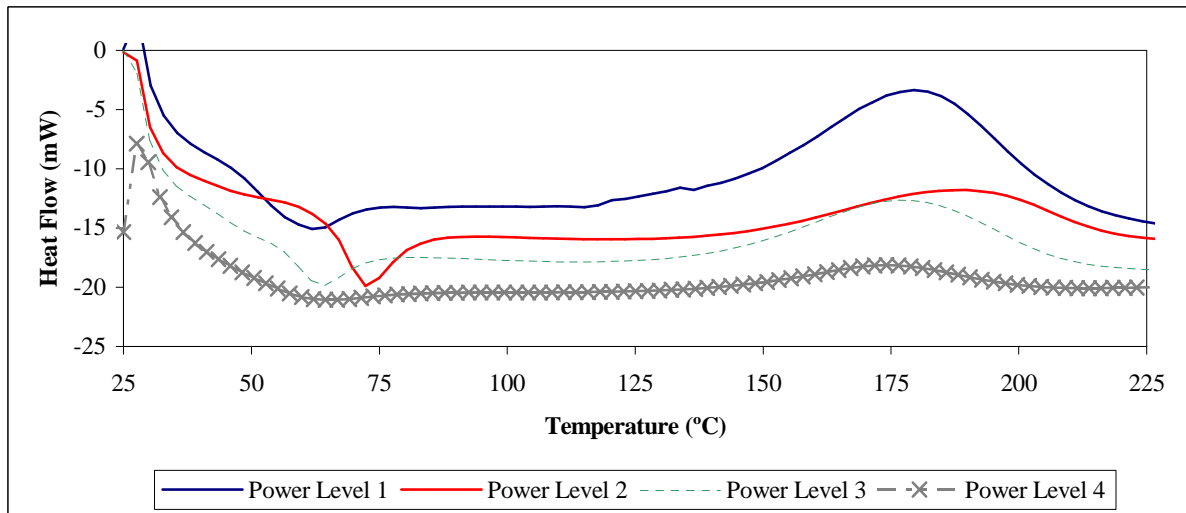


Figure 6: Differential Scanning Calorimeter (DSC) of samples for evaluation of curing process.

In order to provide a better curing degree, the samples were subjected to a post-curing process, on an oven, for 60 minutes at 200°C [18]. The results obtained after the post-curing process are presented in Fig. 7.

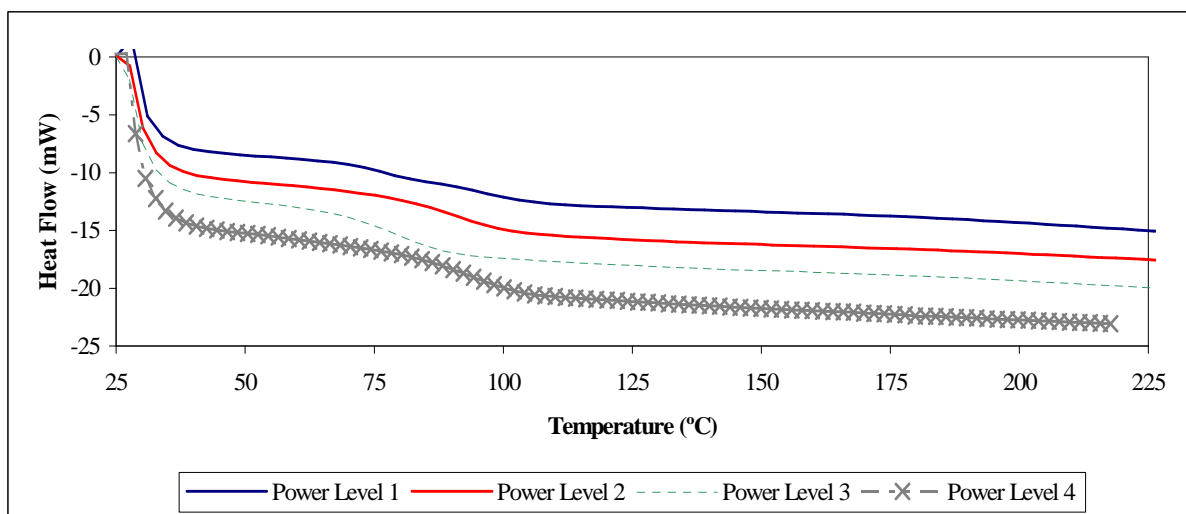


Figure 7: Differential Scanning Calorimeter (DSC) of samples for evaluation of curing process, after post-curing procedure.

After the pos-curing process, none heat was presented on DSC tests for the samples, indicating that the curing process occurred completely. In Fig. 10 it can be seen that, after pos-curing procedure, two Tg occurs for the samples, a first Tg between 60-80°C, and a second Tg between 170-190°C. Comparing this results with the Tg obtained through traditional curing processes, for bisphenol A based resins, it is possible to observe a gain of at least 50°C in Tg value when using microwaves [18].

3. CONCLUSIONS

The process for curing with electron beam is efficient and fast, resulting in a good alternative for curing epoxy resins. The thermal properties achieved by electron beam process are significantly better when compared with traditional methods of curing. Some mechanical properties are comparable with traditional methods, allowing the use of this method without regrets. On the other hand, the use of electron beam curing requires further to studies adjust the formulation and obtain the required properties observed in thermal curing. The electron beam curing also requires high investment for initial setup, and this can be a problem when to decide to migrate to this technology.

The microwave curing process, in the similar way as the electron beam process, is also efficient and fast for curing epoxy resins. Thermal properties, as observed in this study, are similar or better than the properties obtained through traditional methods using ovens or kilns. The mechanical properties are dependent of the power and/or time chosen for the curing process with microwaves, sometimes requiring the use of a pos-curing method (as observed in this work), similar to traditional process, or simultaneously, with a thermal curing process, in a hybrid equipment (microwave and thermal heating). Differently of the electron beam process, the change from thermal curing (with ovens or kilns) to microwave curing is a low cost change, and remarkably energy efficient. To use the microwave process, no alterations are required in the epoxy resin system, and the same formulations used on thermal curing can be used on microwave curing.

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