

# EFFECTS OF THE HIGH FREQUENCY ELECTROMAGNETIC FIELD ON THE EPOXY RESIN PROPERTIES

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**ABSTRACT:** In the paper were performed comparative studies on properties and kinetics of physical and mechanical hardening of epoxy resins subjected to the action of the high frequency and conventional electromagnetic field. The epoxy resins tested for this study are P722 and P722M. The Diaminodiphenyl sulfone was used as a hardener in the preparation of both systems undergoing experiments. These experiments were performed in isothermal and non-isothermal conditions. In both cases there was a significant increase in the fractional conversion of the microwave treated samples compared to the conventionally treated samples.

**KEY WORDS** epoxy resin, microwaves

## 1. INTRODUCTION

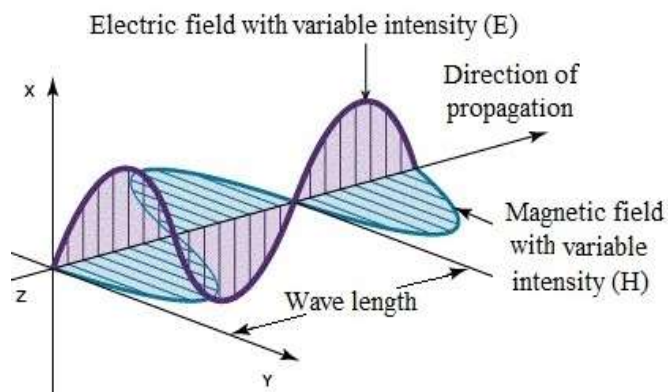
Epoxy resins are polymers with a three-dimensional molecular structure. The network structure generates high rigidity, high strength, heat resistance and solvent resistance. Due to these excellent properties, they are widely used in the manufacture of electrical insulators, structural applications, aerospace structures, high quality flooring, etc. One of the major disadvantage of epoxy resins is their low impact strength and crack initiation. Accordingly, in order to improve breaking strength and impact resistance, while maintaining the desired properties, are added polymeric materials such as rubbers and thermoplastics and reinforcing them using high strength fibers.

Microwave heating uses the ability of liquids and solids to convert electromagnetic energy into heat, which is based on the principle that a material can be heated by applying energy in the form of high-frequency electromagnetic waves, an electromagnetic radiation that consists of an electric field with plane perpendicular on the magnetic field.

The microwave heating effect comes from the interaction of the microwave electric field component with the particles loaded into the materials. A current will be induced by the material if the charged particles are able to move through the electric field. If the particles can not move because are stucked to the material, they will simply be re-arranged in phase with the electric field, through the mechanism called dielectric polarization. [1].

In practice there are two mechanisms through which materials respond to microwave processing.

Depending on the material, the response mechanism can be either dipole rotation, ionic conduction, or a combination of both mechanisms:



**Figure 1.** Electromagnetic wave propagation

- The Rotation Dipole operates on electrically neutral polar molecules, with positive and negative electrical charges, separated. The amplitude of the microwave field increases from zero in one direction, reaches a maximum, decreases to zero, and then increases and reaches a maximum in the opposite direction. The molecules in the field, respond by rotating their respective polar heads in the direction of increasing amplitude. The resulting molecular friction generates heat instantly and uniformly across the compound. In microwaves, at the 2.45 GHz frequency, the oscillation of the electric field of radiation takes place at  $4.9 \times 10^9$  times per second, which is identical to the response time (relaxation time) of the permanent dipoles present in most organic and inorganic molecules. This is a basic feature for an efficient interaction between the electromagnetic field of the microwaves and the chemical system; [2];

- Ionic conduction is the second mechanism used for microwave heating. It generates heat through an induction phenomenon. The microwave electric field attracts the free ions of the compound. Are produced collisions with non-ionised molecules, which gives kinetic energy in the form of heat. The phenomenon does not depend much on the temperature or frequency of the microwaves. [2]

Materials presents differ reaction to the high frequency electromagnetic field. The metals for exemple tend to reflect microwaves, a feature that allows them to be used for wave guides and containers to hold, direct, and apply microwave energy. Some other materials, that are mostly non-polar compounds are transparent to microwaves and will not heat up and reflect the microwave energy. These materials are often used as processing containers, especially for liquids. Materials that respond and can be processed through microwave energy are composed of polar, ionized or conductive compounds. [3]

Unlike thermal heating that involves heat transfer with it thermal delays associated, microwave heating generates heat directly into the sample, and for this reason it can provide the potential for higher efficiency, a faster production rate, lower costs, uniform treatment to which the material is subjected, and is observed an improvement in the physical and mechanical properties to thermal heating. [4]

Microwave heating provided is more selective, more volumetric, faster and more controllable than thermal heating. [5] This higher control of the process eliminates the temperature losses that occur during the hardening of the samples undergoing the experiments.

## 2. EXPERIMENTS AND RESULTS

The analysed epoxy resins and subjected to experiments were selected for their industrial importance and because their dielectric properties predispose them to efficient microwave field treatment [6]. Experiments have shown that the cure speed depends more on the way the microwave pulses are applied than on the total applied power. Some of the results obtained from experiments indicates that the cure rate is not directly related to the sample temperature [7].

In the experiments, in order to achieve the crosslink, the application of the epoxy resin, the deposition and cure of the epoxy resin, was used a mono-mode cylindrical cavity with a radius of 64.7mm and a length of 328mm, the frequency of 2.45GHz, TEM<sub>122</sub>.

The chemical structures of the epoxy resins P722 and P722M used in these experiments are confirmed, and the molecular weights of the resins are also confirmed. In order to determine the thermal behaviour, the resins have been characterized.

It is important to determine the chemical composition of the resins, because this play a crucial role in the properties of the final product. Also, since the microwave energy is transferred to the material by dipolar polarization, the interaction between the high frequency electromagnetic field and the material is affected by the chemical composition. The reaction can also be controlled in order to determine its optimum properties.

The molecular weight distribution curve for the resin is shown in Figure 2. The results show that the average molecular weight ( $M_n$ ) of P722 resin was 471.460 g mol<sup>-1</sup>, the average molecular weight ( $M_w$ ) was 942.500 g mol<sup>-1</sup>, and for P722M the average molecular weight ( $M_n$ ) was 475.842 g mol<sup>-1</sup>, the average molecular weight ( $M_w$ ) was 986.73 g mol<sup>-1</sup>.

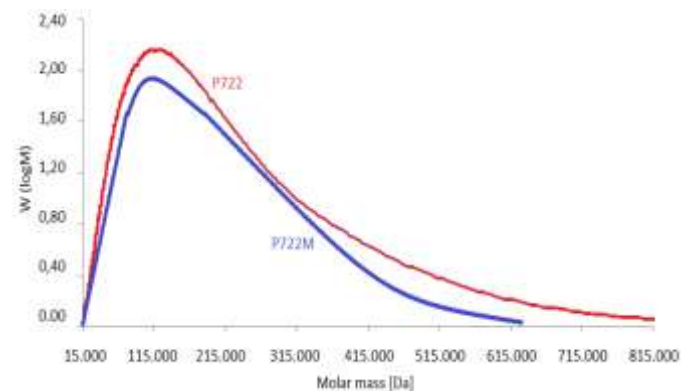


Figure 2. The Molecular Weight Distribution Curve

Table 1 - Comparison of manufacturer's density with experimental density values for P722 and P722M

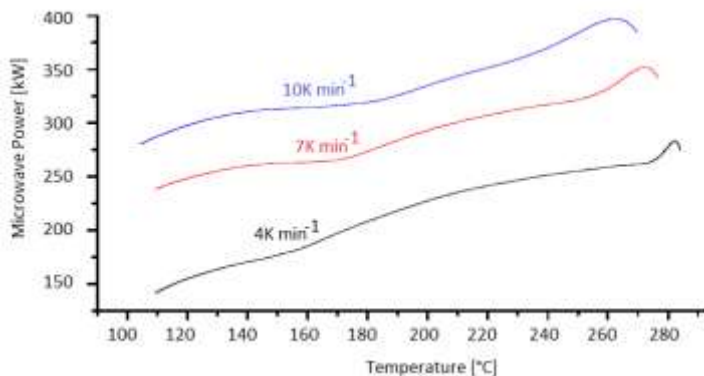
Material	Density - Producer value [g / cm <sup>3</sup> ]	Density - experimental value [g / cm <sup>3</sup> ]
P722	1,20	1,23 ± 3
P722M	1,24	1,20 ± 2

Thermal furnace measurements where the experiments were performed by monitoring the microwave power level according to sample temperature during controlled heating programs [13]

Table 2 –The peak value of the exothermic temperature  $T_p$ ,  $\alpha_p$  with an amine / epoxy ratio of 0.93 sprayed at different heating temperatures using microwave heating

Heating rate [Kmin <sup>-1</sup> ]	$T_p$ [°C]	$\alpha_p$
2	156	0,52
4	169	0,48
6	184	0,50
8	203	0,50
10	224	0,47

Measurements of the exothermic reactions for the P722 and P722M epoxy systems with an amine / epoxy ratio of 0.93 pulverized at different heating rates are shown in Figure 3. The peak exothermic temperature and the fractional conversion value at the exothermic maximum temperature are presented in Table 2.



**Figure 3.** Epoxy system with an amine / epoxy ratio of 0.93 obtained from microwave heating at different heating rates

The properties of epoxy resins subjected to the high frequency field have been modified and we can say that they have grown compared to dried conventional epoxy resins. The main properties regarding the experimental point of view, are mechanical properties, chemical resistance, thermal resistance, electrical properties, flame resistance and weathering resistance, data being shown in Table 3. Epoxy resins have been shown to have excellent mechanical properties compared to other materials. They present high tensile strength, bending and high impact resistance [14].

**Thermal resistance:** Heat resistance is directly related to resin composition, molecular size and crosslinking frequency have direct effects on heat resistance and high stiffness. Cross-character enhances the resistance to softening and deformation at high temperatures. The structural transitions of these crosslinked networks are characterized by DSC or by measuring the temperature of distortion for the heat. These studies give the transition temperature at which the rigid crystalline network and amorphous limits turn into a state of flexibility. The reinforcements improve thermal properties.

**Flame resistance:** Flame resistance is defined as a slow burning rate while is in contact with a heat source or the ability to extinguish itself when the fire source is removed. Fire resistance is generally achieved by incorporating chlorine, phosphorus, bromine or fluorine in the form of compounds that remain stable at both indoor and outdoor exposure. In the epoxy resin, flame retardation is achieved by advancing the liquid DGEBA resin (wpe 180-200) upto tetrabromobisphenol A (wpe 450-500). Flame

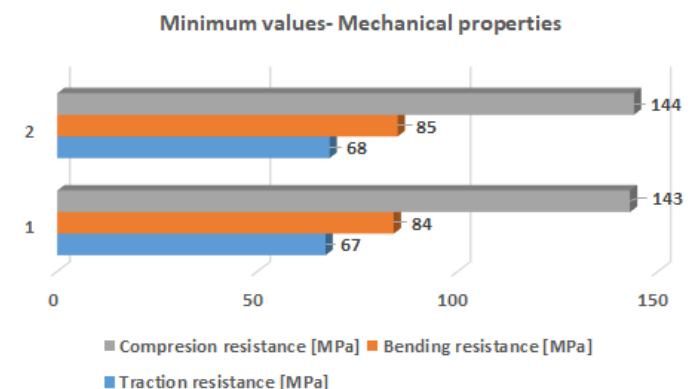
retardation can be improved by incorporating dialkyl (or aryl) phosphate into the epoxy resin [8]. Although halogen-free, it has been found that mixtures of epoxy resins and unsaturated polyester resins have improved flame retardation [9].

**Heating:** The studied epoxy resins are extensively used for outdoor applications such as boats, tanks, car bodies, signs and structural panels. Warming occurs through chemical exposure or other environmental conditions. Damages that occurs is discoloration, erosion and exposure to fibres.

Major disadvantages of unmodified epoxy resins include high viscosity, high cost, and too high rigidity for specific applications. Therefore, these resins must be modified by the incorporation of diluents, fillers, flexibilizers and other additives. Diluents reduce viscosity of resin and simplify handling. Xylene and dibutyl fbrate are used as non-reactive diluents, while phenylglycidyl ether, butyl glycidyl ether and octane oxide are used as reactive diluents in epoxy resins. Because diluents affect the physical properties of the resin and the treatment delay they are used in low doses [10].

The unmodified DGEBA resins show low brittleness and elongation after use. Flexibilers and plasticizers are added to improve the flexibility and hence the hardness of the resin. The aliphatic dioxides increase flexibility by providing more free rotation of the chain segments between cross-links. Polyamides have been used as fexibilers in the epoxy resins [11]. Low molecular weight polyamides are not only flexible but also act as amine-hardening agents. Liquid polysulfide polymers with terminal mercaptan functionality improve the impact properties when used with hardening agents. Flexibility is accompanied by a reduction in rezistance of the elastic modulus and hardness. Some flexibilers of the amine are realised through cyanoethylation of the amines hardener [12].

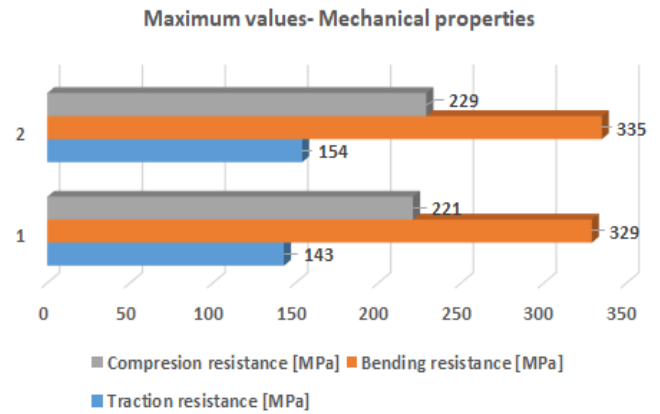
Figure 4, 5, 6 and 7 will point out the characteristic properties of P7222 and P722M resins.



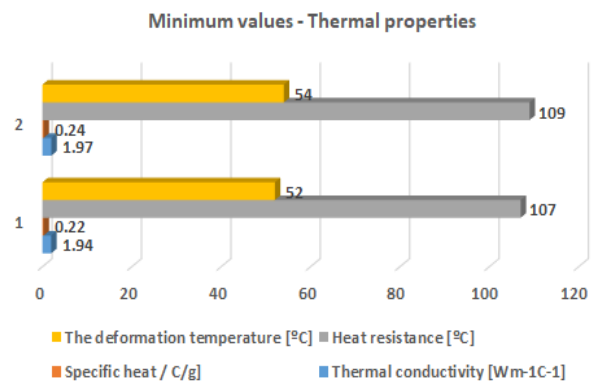
**Figure 4.** Minimum values of the mechanical properties

**Table 3 - Casting Properties of P722 and P722M Epoxy Resins in Microwave Field**

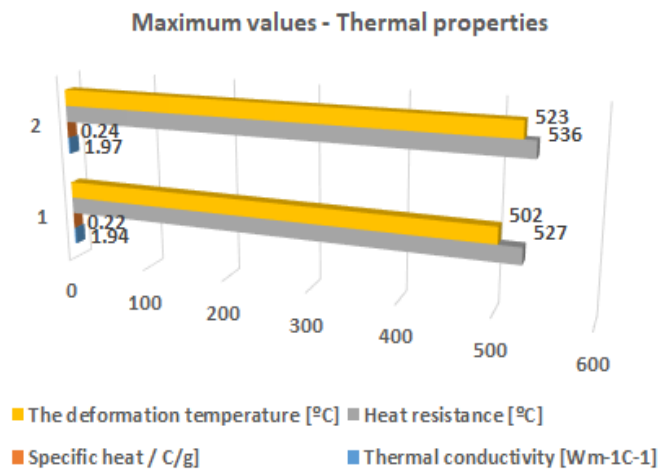
Properties	Cast resin		Tested sample
	Rigidity	Flexibility	
<i>Processing</i>			
1. Compression casting temperature [°C]	---	---	107 ÷ 162
2. Maleability of compression at casting [MPa]	0,001÷0,005	0,001÷0,003	2,15 ÷ 3,52 0,003÷0,007
3. Contraction rate	1,14 ÷ 1,5	1,03 ÷ 1,38	1,4 ÷ 2,2
4. Specific gravity			
<i>Mechanical</i>			
5. Traction resistant [MPa]	25 ÷ 92	16 ÷ 75	67 ÷ 143
6. Elongation at break [%]	2,8 ÷ 6,4	18 ÷ 84	5
7. Bending resistance [MPa]	58 ÷ 143	---	84 ÷ 329
8. Compression strength [MPa]	95 ÷ 154	6 ÷ 92	143 ÷ 221
9. Hardene Rockwell	M80 – 110	---	M100 – 110
<i>Thermal</i>			
10. Thermal conductivity [Wm <sup>-1</sup> C <sup>-1</sup> ]	0,22 / 20,15 / 0,32	---	1,94 / 0,22 / 107 ÷ 527
11. Specific heat [cal / C/g]	130 ÷ 507	27 ÷ 102	52 ÷ 502
12. Heat resistance [°C]	- 149 ÷ -		
13. Deformation temperature [°C]	123		
<i>P722M</i>			
<i>Processing</i>			
1. Compression casting temperature [°C]	---	---	109 ÷ 175
2. Maleability of compression at casting [MPa]	0,002÷0,007	0,003÷0,005	2,23 ÷ 3,84 0,005÷0,009
3. Contraction rate	1,15 ÷ 1,7	1,04 ÷ 1,52	1,5 ÷ 2,7
4. Specific gravity			
<i>Mechanical</i>			
5. Traction resistant [MPa]	28 ÷ 97	18 ÷ 79	68 ÷ 154
6. Elongation at break [%]	2,8 ÷ 6,8	19 ÷ 87	7
7. Bending resistance [MPa]	60 ÷ 154	---	85 ÷ 335
8. Compression strength [MPa]	97 ÷ 171	7 ÷ 95	144 ÷ 229
9. Hardene Rockwell	M80 – 110	---	M100 – 110
<i>Thermal</i>			
10. Thermal conductivity [Wm <sup>-1</sup> C <sup>-1</sup> ]	0,22 / 20,15 / 0,32	---	1,97 / 0,24 / 109 ÷ 536
11. Specific heat [cal / C/g]	130 ÷ 507	27 ÷ 102	54 ÷ 523
12. Heat resistance [°C]	- 149 ÷ -		
13. Deformation temperature [°C]	123		



**Figure 5. Maximum values of mechanical properties**



**Figure 6. Minimum values of mechanical properties**



**Figure 7. Maximum values of thermal properties**

### 3. CONCLUSIONS

The objectives of these experiments were to compare the hardening kinetics, the reaction paths, the physical and mechanical properties of the two epoxy systems prepared by microwave heating.

- The P722M synthesized epoxy resin treated in the high frequency field generally has properties comparable to industrial epoxy resins;
- Utilizarea înlocuitorilor de fenol sau dipenol în sinteza rășinii epoxidice este importantă deoarece poate fi obținută o rășină mai flexibilă, structura

materialului fiind modificată atunci când interacționează cu microundele;

- Incorporation of bisphenol A in addition to the P722M epoxy resin and its treatment in the microwave field, results in a harder resin that has better strength and a higher elongation;
- Among the copolymers used as modifiers, it is found that phenol mixtures are the most effective choice for increasing the resistance in the epoxy resin P722, its resistance increasing significantly after exposure to the high frequency electromagnetic field;
- DGEBA hybrid polymer networks show a significant improvement in hardness and water resistance properties;
- The epoxy resin blended with epoxidized neolacquer resin and exposed to the high frequency electromagnetic field presents better aging resistance compared to the untreated resin in the microwave field;

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