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The Study of Some Mechanical Properties of Some Composite Materials with Different Types of Matrices and Reinforcement from Chromat-type Isophthalic Resin Granules NPG

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Abstract. *Using the experimental determinations obtained on the basis of compressive stress, some mechanical properties were studied for composite materials with the matrix of three types of resin, epoxy, unsaturated polyester and hybrid based on Dammar natural resin, which was reinforced with isophthalic resin granules NPG (Neopentyl Glycol) Chromat Kayan / Javari / Payette type. The stress-strain diagrams, compressive yield strength, compressive strength and modulus of elasticity in uniaxial compression were obtained. With the EDS analysis, the graphical distribution of the atomic spectra of the elements identified in the hybrid resin was determined and the image of the fracture surface of a hybrid resin specimen was presented based on the stereomicroscopic analysis (SEM).*

Keywords*: composite materials, compressive yield strength, compressive strength, modulus of elasticity in uniaxial compression, atomic spectrum of elements*

1. Introduction

In the applications where the isophthalic entrapment granules NPG (Neopentyl Glycol) were to be used, several processes for their production were developed. An example of such a process can be found in [1], where a process was developed for the preparation of aqueous suspensions of vesiculated cross-linked polyester resin granules in which the granules have a maximum drying shrinkage of 5% of their diameter. This process consists in the polymerization of an unsaturated carboxylate polyester with acid values between 5-50 mg KOH/g and an unsaturated ethylenic monomer, through a double emulsion process and in the presence of a base such as oxides, hydroxides or metal salts. Another process for the production of isophthalic resin granules NPG is described in [2] and consists in the manufacture of polyester in several stages, including an immersion stage in a swelling medium that allows the kinetics of the post-condensation stage to be increased in solid phase.

Some properties of isophthalic resin granules NPG have been investigated both in separate studies and in association with other components. For example, in [3] the properties of a surface covered with a composite material imitating granite were studied. The respective composite was made from a matrix based on polyester or acrylic resin (with the related reinforcements), and resin granules with thermoplastic properties were used as reinforcement. The thickness of the surface coating was uniform and the appearance obtained was of granite.

Various properties of unsaturated polyesters (resistance to thermal shocks, some elastic properties and water absorption) as well as applications of their use for the manufacture of kitchen sinks and countertops were studied in the paper [4].

In the paper [5], an efficient strategy for the chemical recycling of unsaturated polyester resin (UPR) was developed through the selective cleavage of C-O bonds.

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More and more fire safety regulations have led to greater demands being placed on structural thermosets such as epoxy and unsaturated polyester resins. As a result of these requests, flame retardant thermosetting polymers of high ecological performance were developed, using fire retardant strategies with monomers [6].

Another method of polymer fireproofing is the use of aluminium trihydroxide (ATH), which is an ecological and economical additive, but which has a low thermal stability. This issue is critical in determining the fire protection and thermal stability of composites. In the study [7] some results were obtained by which the thermal stability of aluminium trihydroxide was improved by combining it with nano-porous silica known as silica air gel (SA), as a hybrid filler in unsaturated polyester resin.

In the same direction of obtaining mechanical and fire performances of glass fiber reinforced composites, in the work [8] the effects of matrices from co-reinforced mixtures of an unsaturated polyester (UP) with inherently fire-retardant phenolic solutions (PH) were investigated and which forms carbon.

The study of some mechanical properties for composite materials with the orthophthalic unsaturated polyester resin matrix reinforced with isophthalic resin granules NPG of the Chromat Buzzi/Perdido type were also investigated in the paper [9].

Data on some mechanical and chemical properties of Chromat-type isophthalic resin granules NPG (Neopentyl Glycol) can be accessed from the manufacturer's website [10, 11].

In papers [12, 13], some chemical and mechanical properties of hybrid resin with volume proportions between 50 and 80% Dammar natural resin were studied. More specifically, the tensile strength, percentage elongation at break and modulus of elasticity were determined based on the characteristic curves. A variation of these characteristics was recorded as follows: it had values between 19.5-20.9 MPa; had values between 1.89-2.05%; had values between 1630-1810 MPa.

In this paper, a hybrid resin with a volume proportion of 60% natural Dammar resin and 40% epoxy resin was made. Based on the EDS analysis, the graphic distribution of the atomic spectra of the chemical elements identified in the hybrid resin was established, and some mechanical properties of the same resin were studied based on the compression stress.

Also, on the basis of compression stress, by comparison, the mechanical properties of some composite materials were investigated, in which three types of isophthalic NPG resin granules of the Chromat Kayan / Javari / Payette type were used as reinforcement, and separately three types of resins used as matrices, more precisely epoxy resin, unsaturated polyester resin and hybrid resin with 60% Dammar.

As applications, it is recommended to use these composite materials in the field of construction, or to make sanitary objects.

2. Materials and methods

2.1. Preparation of samples

Casting of the bars from which the samples were cut for the compression stress was carried out at a controlled temperature between 21 – 23^{o}C.

To make samples from composite materials, Chromat type NPG isophthalic resin granules were used as reinforcement, and three types of resins were used as matrix:

- Resoltech 1050 epoxy resin with Resoltech 1058S related hardener [14];
- NORSODYNE S 20202-A unsaturated polyester resin [15];
- a hybrid resin based on Dammar natural resin (with a volume proportion of 60% Dammar, and 40% Resoltech 1050 epoxy resin with Resoltech 1058S its related hardener).

All the samples made were submitted to the compression test. The type of materials from which the specimens were made, their weight and abbreviation are presented in Table 1.

20 specimens from each set were made and the dimensions of the specimens required for compression were 14.7 mm x 14.7 mm x 14.7 mm.

Table 1. Types of samples made and their characteristics

No.	Materials for making the samples	Test weight [g]	Abbreviation
1.	hybrid resin based on Dammar, in which the volume proportion of natural resin is 60%	3.4	D xx
2.	epoxy resin Resoltech 1050 with the related hardener Resoltech 1058S	3.8	E xx
3.	unsaturated polyester resin - orthophthalic NORSODYNE S 20202 A	3.9	P xx
4.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Kayan type	4.0	DK xx
5.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Javari type	4.1	DJ xx
6.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Payette type	4.2	DP xx
7.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Kayan type	4.5	EK xx
8.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Javari type	4.6	EJ xx
9.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Payette type	4.6	EP xx
10.	composite materials with the matrix of unsaturated - orthophthalic polyester resin and the reinforcement of isophthalic resin granules NPG of the Chromat Kayan type	4.4	PK xx
11.	composite materials with the matrix of unsaturated polyester resin - orthophthalic and the reinforcement of isophthalic resin granules NPG of the Chromat Javari type	4.5	PJ xx
12.	composite materials with the matrix of unsaturated polyester resin-orthophthalic and the reinforcement of isophthalic resin granules NPG of the Chromat Payette type	4.6	PP xx

Figure 1 shows samples of specimens for compression stress, made with a matrix based on Dammar and reinforcement from isophthalic resin granules NPG of the Chromat Kayan / Javari / Payette type.

Figure 1. Specimens of specimens for compression stress with matrix based on Dammar

2.2. Technical characteristics of the equipment

The chemical composition of the hybrid resin was determined based on EDS analysis. This analysis was performed with the scanning electron microscope QUANTA INSPECT F50 [16] equipped with field emission electron gun – FEG (field emission gun), with a resolution of 1.2 mm and energy dispersive X-ray spectrometer (EDS), with a resolution at MnK of 133 eV; EDAX chemical micro-composition analyser and its related software for performing local micro-composition analyses.

Since the SEM analysis required a much higher magnification than the EDS microscope, the electron microscope – Hitachi model S3400N/type II [17] was used for this analysis, with the following characteristics: SE resolution: minimum 3.0 nm at 30kV (x100,000, WD = 5mm, high vacuum mode); minimum 10 nm at 3kV (x30,000, WD = 5mm, high vacuum mode); BSE image resolution: minimum 4.0 nm at 30kV (x60,000, WD = 5 mm, low vacuum mode); magnification range between 5x and 300,000x; acceleration voltage: 0.3 kV – 30 kV; electron gun with voltage: in stages with self-field and fixed field with continuously adjustable voltage; beam alignment: two-stage electromagnetic deflector; objective opening: 4 holes with click-stop system with diameter of 30, 50, 80 and 150 microns; image displacement: minimum ± 50 microns at WD = 10 mm.

The LLOYD Instruments Lrx PLU mechanical testing machine [18] with the following characteristics was used for compressive testing: maximum value of the applied force: 2.5 kN; stroke

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size was between 1 and 735 mm; application speed between 0.1 and 500 mm/min; NEXYGEN analysis software.

3. Results and discussions

Since the mechanical and chemical properties of the two synthetic resins and of the NPG isophthalic resin granules of the Chromat type, have been carefully studied by the manufacturers, a similar study is necessary for the hybrid resin used.

In the first part of the experimental research, the chemical composition was determined for the hybrid resin based on Dammar natural resin (with a volume proportion of 60% Dammar).

In the second part of these experimental determinations, the samples made from the composite materials described in section 2.2. were subjected to compression stress, the stress-strain diagrams being obtained. In order to be able to compare the behaviour of the composite materials made, the diagrams were compared according to the reinforcement used.

3.1. Chemical composition of hybrid resins

In order to establish the graphical distribution of the atomic spectra of the chemical elements in the hybrid resin, it was necessary to take a sample. The scanning electron microscope cannot record micrographs on this sample because it does not exhibit conductivity. This disadvantage was overcome by preparing the sample before it was subjected to EDS analysis, namely it was inserted for 60 seconds into a metallizer provided with a gold target in which a vacuum was created and a very large amount of little gold The final result of the chemical composition is not influenced by this process because it is only a gold coating to be able to do the analysis (at most, gold is also identified as an element in the EDS analysis).

The sample was analysed with the EDAX detector which gave the graphical representation of the distribution of the atomic spectra of the identified elements and the numerical values of the chemical composition of the hybrid resin.

The investigation of the morphological characteristics related to the sample was carried out both on the surface and in the section.

In Figure 2 this graphical distribution data of the atomic spectra of the elements identified in the hybrid resin. The main area of the graphical distribution of the spectra has been magnified 3.5 times within the figure in order to be correctly seen.

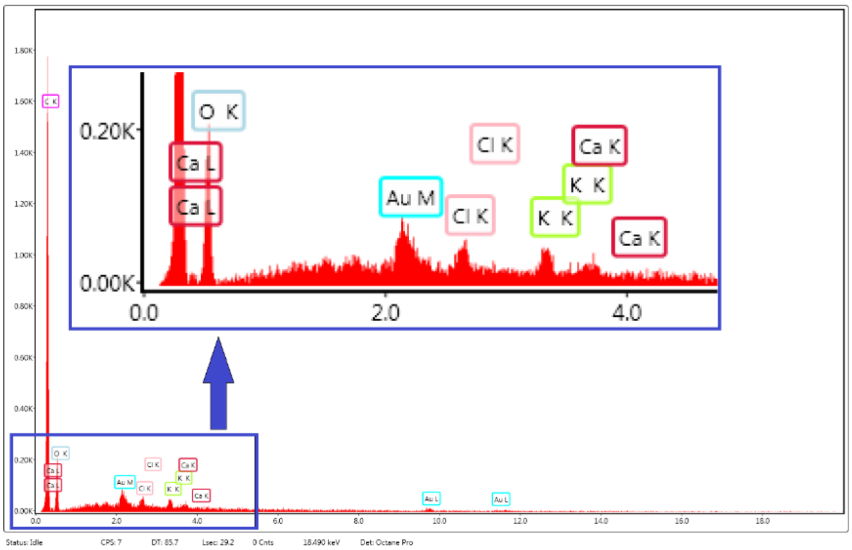

Figure 2. Graphical distribution of the atomic spectrum of the elements identified in the hybrid resin display, provided at the intensity of 18,490 keV

The spectrum obtained at the intensity of 18,490 keV highlights the presence of the elements carbon, nitrogen, oxygen, chlorine and potassium in the proportion that can be found in Table 2. This structure was obtained based on the EDS analysis and it kept the chemical composition of a hybrid resin sample.

Table 2. Chemical composition of a hybrid resin sample

Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
C K	68.44	74.07	5.08	280.59	0.4669	1.0113	0.9935	0.6745	1
N K	5.85	5.43	32.37	3.07	0.0043	0.9909	1.0035	0.0734	1
O K	24.88	20.22	13.13	44.75	0.0245	0.9731	1.0126	0.1011	1
Cl K	0.28	0.1	23.51	11	0.0024	0.8369	1.0718	1.0247	1.0272
K K	0.55	0.18	13.6	20.3	0.0051	0.835	1.0815	1.0692	1.044

3.2. SEM analysis of the fracture surface of the hybrid resin

Based on the stereomicroscopic analysis (SEM), in Figure 3, the fracture surface image of a representative specimen from the hybrid resin set is presented. This analysis was carried out in compliance with [19].

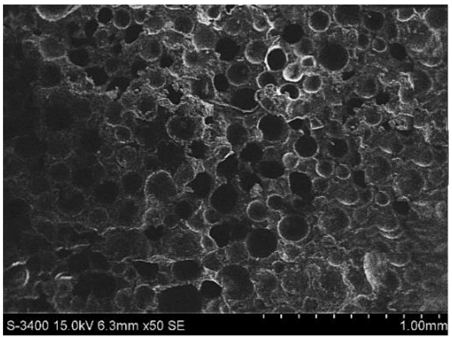

Figure 3. Fracture surface of a representative specimen from the hybrid resin set

3.3. The compression test of the three types of resin used

It must be stated at the outset that this request was made in compliance with [20]. Figure 4 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of resins used.

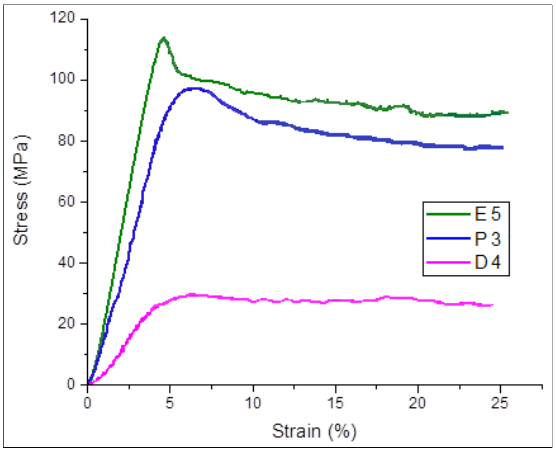

Figure 4. Stress-strain diagram obtained from compression stress a of representative samples from sets E xx, P xx and D xx

3.4. The compression test of the composite materials made

Figure 5 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of composite materials with the reinforcement isophthalic resin granules NPG of the Chromat Kayan type.

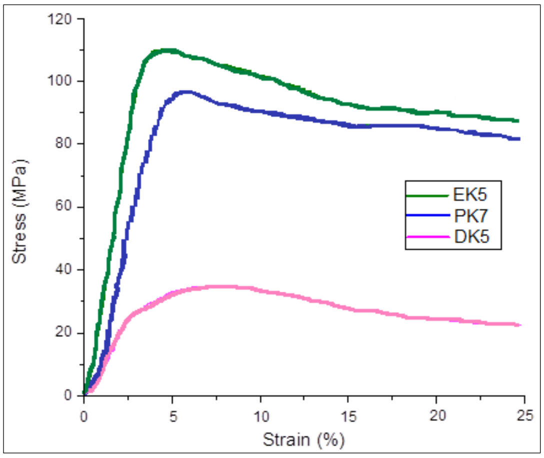

Figure 5. The stress-strain diagram obtained from the compression stress of some representative samples from the EK xx, PK xx and DK xx sets

Figure 6 shows the stress-strain diagram obtained after the compression of some representative samples from the three sets of composite materials with the reinforcement of NPG Chromat Javari isophthalic resin granules.

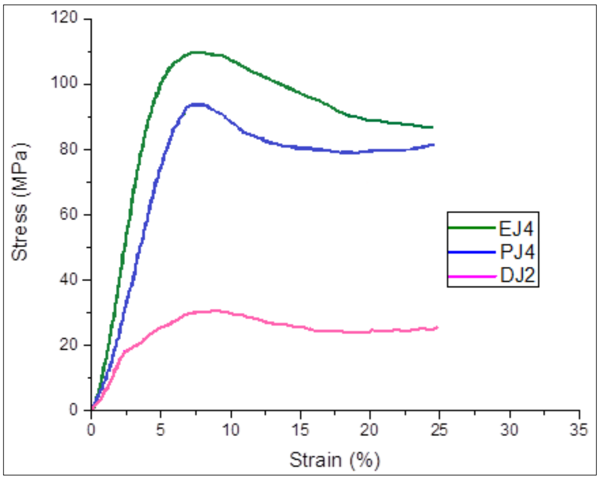

Figure 6. The stress-strain diagram obtained from the compression stress of some representative samples from the sets EJ xx, PJ xx and DJ xx

Figure 7 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of composite materials with the reinforcement of isophthalic resin granules NPG of the Chromat Payette type.

Table 2 shows the averages of the main values of compressive yield strength $R_{p,0.2}$*[MPa]*, compressive strength R_c *[MPa]*and modulus of elasticity in axial compression *E[GPa]*. The compressive yield strength was determined for the resins and composites studied are materials with a high elongation at break, at which permanent plastic deformations occur.

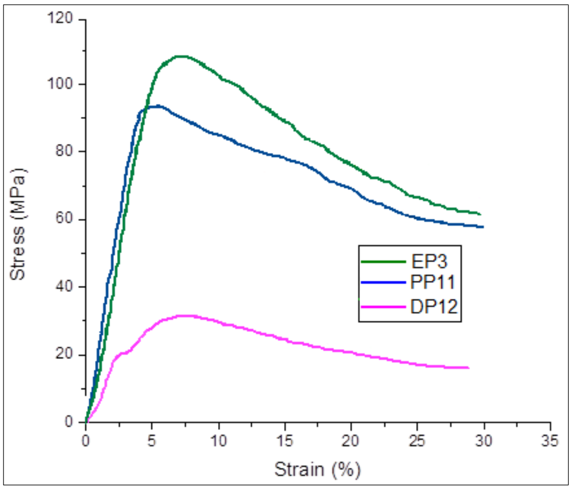

Figure 7. The stress-strain diagram obtained from the compression stress of some representative samples from the sets EP xx, PP xx and DP xx.

The values presented in the following table were obtained as follows: the first two values, the lowest, were eliminated, and for the remaining 8 values, their arithmetic mean was made.

Table 2. Mean values and deviation for the main mechanical characteristics obtained on the basis of compressive stress

Sample	**Compression yield strength** $R_{p,0.2}\ [MPa]$	**Compressive strength** $R_c\ [MPa]$	**Modulus of elasticity in uniaxial compression** $E\ [GPa]$
E	112.02 (±3.03)	113.84 (±3.86)	29.82 (±1.24)
P	94.79 (±2.95)	97.04 (±2.57)	20.27 (±1.16)
D	23.26 (±1.24)	25.14 (±1.08)	6.98 (±0.51)
EJ	100.95 (±3.92)	109.84 (±3.24)	26.04 (±1.97)
PJ	88.93 (±2.74)	92.34 (±2.98)	24.75 (±1.64)
DJ	22.34 (±1.55)	34.93 (±1.92)	10.59 (±0.67)
EK	106.38 (±3.59)	110.75 (±3.85)	33.57 (±1.32)
PK	94.82 (±2.57)	97.84 (±2.74)	22.19 (±1.09)
DK	29.04 (±2.04)	35.95 (±2.18)	10.79 (±0.94)
EP	103.52 (±3.03)	108.02 (±3.18)	28.33 (±1.63)
PP	90.66 (±2.22)	93.71 (±2.84)	29.26 (±1.47)
DP	20.88 (±1.43)	34.21 (±1.46)	11.36 (±0.92)

It can be seen that the mechanical properties obtained for composite materials with hybrid matrix and reinforcement from Chromat-type NPG isophthalic resin granules are superior to those obtained for composite materials with various types of hybrid resins and reinforcements obtained from agricultural waste [21, 22].

The chemical composition and the values of the mechanical properties for the composites with various types of matrices and reinforcements from NPG isophthalic resin granules of the Chromat type, allow their use in dental technics laboratories. More precisely, in the dental impressions, instead of the special gypsum, the composite material with Chromat granules will be poured. After hardening, the impressions will be removed and the teeth will be polished on which the dental crowns will be made.

An advantage of using these composites instead of special gypsum is that composites are more difficult to fracture when impressions are removed. In addition, fracture of the gypsum leads to impression loss which involves repeating the dental impression and this leads to high final costs.

4. Conclusions

SEM analysis of the surface of the fracture section of a hybrid resin specimen highlights the presence of multiple air bubbles. A possible explanation may be that air bubbles formed as a result of polymerization remain trapped inside the hybrid resin because it polymerizes more slowly than synthetic resins. However, in the case of reinforcing this hybrid resin with various types of fibers / fabrics / granules, a significant reduction in the number of air bubbles was found in the composite materials made because the volume proportion of the resin in the composite material is much lower than in the case of the samples made only from resin.

The analysis of the stress-strain diagrams obtained from the compression load shows that the value of the compressive strength of the composite materials reinforced with Chromat-type isophthalic resin granules NPG decreases very little (by approximately) compared to the value of the compressive strength of the synthetic resin used as a matrix, respectively records. an increase (about) over the compressive strength of the hybrid resin.

The compressive flow limit values and respectively the elastic modulus in uniaxial compression of all the composite materials made decrease compared to the respective values of these characteristics for the constitutive molds. Based on the analysis of the failure sections and the hybrid stress-strain diagrams, it can be found that the failure of the resin specimens was sudden, without plastic deformation and no flow phenomenon. This type of phenomenon is generally characteristic of brittle materials.

The values determined for the characteristics of these mechanical composite materials allow their use in the field of construction, to please some surfaces (floors, walls), or to make some sanitary objects (sinks, tubs, countertops, etc.), or in the field of dental technics for making molds on which dental crowns are to be made.

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