## A Study of Some Mechanical and Chemical Properties of Composite Materials Made Up of UPR and ISO/NPG Granules of the Type of Buzi/Perdido Cromate

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In this work we have measured the mechanical properties for unsaturated polyester resin (UPR) and unsaturated polyester resin mixed with isophthalic resin granules NPG (Neopentyl Glycol) of the type of Buzi/Perdido Chromate. All sets of samples underwent compression testing. We obtained the characteristic curve, the admissible resistance to compression, and the modulus of elasticity for each sample. Moreover, on the basis of FTIR analysis we measured the type of bonds (C-O, C=O, C-C and various bonds in the functional groups) in each polymer.

# Keywords: composite materials, admissible resistance to compression, modulus of elasticity, chemical bonds

Making composite materials with chemical or mechanical properties that may recommend their use in various fields of activity has always been a major challenge for researchers.

In time, obtaining primary synthetic resins (of epoxy, polymer and other types) has engendered a competition among experts in chemistry, who have sought to adapt or associate them with other materials in order to make them resistant to high temperatures, humidity, stretch, compression, and so on.

A detailed presentation of the chemical composition and some special applications of the unsaturated polyester resin can be found in Chapter 7 of the book [4]. Also in this respect, an elaborate study on the reinforcement of unsaturated polyester resin and the use of that resin as a matrix in different composite materials, employed by car, naval or construction industries has been conducted in the work [13].

Particularly, certain mechanical properties of unsaturated polyester resin have been analysed in the works [3, 8].

Given that unsaturated polyester resins are fragile and have weak resistance to fissure propagation, many studies on the chemical modification of resins have been conducted and thus their tensile strength, impact resistance, and elongation at break have been increased. This increase in resistance has been achieved through a reactive blending with polyurethane prepolymers having terminal isocyanate groups. Such a study was conducted in the work [2].

The work [7] presents different ways of using unsaturated polyester resins (which contain styrene and long-chain fatty acids as reticulation agents) in order to obtain synthetic marble and several ornaments, as well as to prepare lacquers.

Styrene is employed as a comonomer for unsaturated polyester resins. The thermic stability and mechanical properties of the resins are governed by the phase behaviour of the mixture and therefore, they can be controlled by the styrene content. All these properties are studied in the work [11].

The development of the global mechanical properties of a mixture of unsaturated polyester resin and nanodiamond powder have been analysed in the work [6].

An unsaturated polyester resin and linseed oil (in a proportion of 25% of the resin amount) have been associated in the work [10]. This combination resulted in a new polymer with a relatively high modulus of elasticity and a high glass transition temperature.

The work [14] researches the chemical recycling of unsaturated polyester resins and the composites made with their help, using selective cleavage of the ester bond.

The technology and the process of forming isophthalic resingranules NPG has been the object of several invention patents, each of them addressing a certain segment of practical applications. For example, [5, 12] present an improved procedure of forming polyester resin granules in the shape of vesicles. The technique involved in preparing porous objects of carboxylated, unsaturated polyester resin relies on dispersing a liquid medium (water, for instance) into the polyester resin in the presence of a base and then, the polymerisation of the resin. Thus, the phase of immersion in an inflating medium allows for an increase in the kinetics of the post-condension phase of the solid state.

Both the isophthalic resin granules NPG and different types of unsaturated polyester resins have been researched separately or in association with other components in many scientific works.

Thus, in the patent [5], the isophthalic resin granules NPG are used in association with polyester resins or reticulated acrylic resins (together with a hardening accelerator) as a granite-shaped cover composition, which can be atomized, useful for making simulated stone surfaces, such as polished granite. Those surfaces have a high resistance to impact, superior hardness and a pleasant appearance from an aesthetic point of view. The composition is characterized by forming covers with

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uniform thickness and quick hardening which, even with a reduced thickness, ensure a uniformly granite appearance.

The work [9] investigates the resistance to thermal shocks, some elastic properties and the water absorption of the isophthalic resin granules NPG. Their styrene emissions and some of their mechanical properties have been researched in [1].

#### **Experimental part**

We cast three sets of parallelepiped-shaped samples of the following components:

- unsaturated (orthophthalic) polyester resin - NORSODYNE S 20202 A, the samples being labelled R1-7; unsaturated polyester resin and Buzi Chromate

(M5060), the samples being labeled RC1-7; unsaturated polyester resin and Perdido Chromate (M8137), the samples being labeled RP1-6.

Buzi Chromate and Perdido Chromate granules are very stable and resistant products obtained from a mixture of isophthalic resin NPG with aluminum trihydrate and inorganic pigments (without heavy metals), hardened in a high temperature catalyzer and in a system of ovens at temperatures exceeding 160 ° C.

The size and weight of each sample, as well as the water absorption after 24 h are given in table 1.

The characteristics of the three types of samples

All three sets of samples were submitted to a compression trial. We measured the following mechanical characteristics:

the admissible resistance to compression was calculated with the formula:

$$\sigma_{\alpha c} = \frac{F_{\max}}{A_0},$$
 (1)

where  $F_{\text{max}}$  is the maximum force of compression tolerated by the sample before breaking and  $A_0$  is the area of the initial section of the sample;

-the modulus of elasticity E was obtained by Hook's Law

$$\varepsilon = \sigma_{ac} \cdot E$$
  
where  $\sigma_{ac}$  is given by (1) and

$$\varepsilon = \frac{\Delta H}{H} = \frac{H - H_c}{H} \tag{2}$$

is the specific contraction, with H the initial height of the sample and H the height after compression.

Thus we obtained that

$$E = \frac{F_{\text{max}} \cdot H}{A_0 \cdot \Delta H}.$$
(3)

Additionally, based on the data in table 1, we also calculated the humidity absorption coefficient after 24 hours using the formula:

$$MC(\%) = \frac{W_{water} - W}{W} \times 100$$

where  $W_{water}$  and W are given in table 1. Henceforth, *representative sample of a set* will mean the sample which records average values of the mechanical properties under study.

Figures 1-2 show a representative sample of each of the three sets we cast, before and after compression.

We submitted two representative samples of the composite materials we made to a FTIR analysis - Fourier-Transform Infrared Spectroscopy. This analysis is a

Sample	L <sub>1</sub> [mm]	L <sub>2</sub> [mm]	H [mm]	W [g]	W <sub>water</sub> [24h]
R1	18.45	19.08	20.79	8.3	8.34
					0.54
R2	18.27	19.37	19.16	8.0	
R3	18.50	19.07	19.85	8.2	
R4	18.45	19.94	19.53	8.5	
R5	18.34	19.05	19.44	8.0	
R6	18.31	19.10	19.62	8.0	
<b>R</b> 7	18.89	19.10	19.15	8.4	
RC1	19.05	20.23	21.28	11.3	11.39
RC2	18.47	19.74	21.20	11.2	
RC3	19.23	20.20	21.48	12.1	
RC4	18.72	20.53	21.50	12.2	
RC5	18.65	19.52	21.47	11.4	
RC6	18.44	19.50	21.30	11.4	
RC7	18.61	18.92	21.36	10.9	
RP1	18.79	20.12	21.32	11.5	11.61
RP2	19.09	20.13	21.20	11.0	
RP3	18.92	20.40	21.41	11.4	
RP4	18.75	20.15	21.34	11.2	
RP5	18.78	19.81	21.33	10.9	
RP6	18.74	20.05	21.32	11.0	

Table 1 THE CHARACTERISTICS OF THE THREE TYPES OF SAMPLES





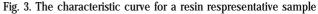


Fig. 1. The samples before compression a. resin; b. resin with buzi chromate; c. perdido chromate

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Fig.2. The samples after compression of resin with cu buzi chromate. a. resin; b. resin with buzi chromate; c. perdido chromate



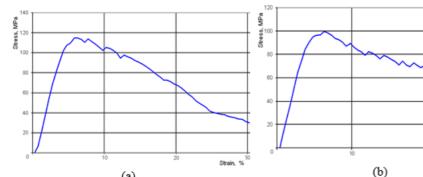


Fig. 4. The characteristic curve for a representative sample of resin and buzi chromate (a), perdido (b)

(a) **Table 2** THE HEIGHTS OF THE REPRESENTATIVE SAMPLES AFTER THE COMPRESSION TEST.

Sample	Hc
R1	18.09
RC1	17.31
RP1	17.08

 Table 3

 THE MAIN MECHANICAL CHARACTERISTICS OF THE SAMPLES SUBMITTED

 TO THE COMPRESION TEST

Strain. %

Material	Area of the	Results - compression test			
sample	section [mm <sup>2</sup> ]	F <sub>max</sub> [kN]	$\sigma_{ac}$ [Mpa]	<u>Е</u> [Мра]	Е [%]
R1	352.03	21.986	62.46	480.902	12.98
RC1	385.38	44.256	114.84	615.551	18.65
RP1	378.05	37.696	99.71	501.380	19.88

technique used for the identification and quantification of organic substances. More precisely, it determines the bond types in a polymer (C-O, C=O, C-C and different bonds in functional groups).

The infrared spectrum of the composite materials was obtained with the help of a JASCO FT/IR - 6000 series spectrometer with a measurement range of 4000 - 400 cm<sup>-1</sup>. The samples were tested as such, without grinding.

### **Results and discussions**

Figures 3 and 4 show the characteristic curves (the stress - strain diagrams) for each representative sample of the three cast sets.

The heights of the representative samples obtained after the compression test are shown in table 2.

Table 3 shows the main mechanical characteristics of the representative samples submitted to the compression test.

According to the data shown in table 1, we obtained that MC is 0.48 % for R1, 0.79 % for RC1 and 0.95 % for RP1.

The results of the FTIR analysis are shown in table 4.

A molecule spectrum may have hundreds of absorption bands. Most of them are in the region of the average infrared spectrum (4000 -400 cm<sup>-1</sup>). This spectrum can be divided into:

- 4000- 2500 cm<sup>-1</sup>, stretch region of the bond X-H; O-H produces a broad band, 3700-3600 cm<sup>-1</sup>, N-H appears in 3400-3300 cm<sup>-1</sup> with a narrower band, C-H appears between 3000-2850 cm<sup>-1</sup> in aliphatic compounds;

- 2500-2000 cm<sup>-1</sup>, the region of the triple bond C=C;

- C=C between 2300-2050 cm<sup>-1</sup>, low intensity, C=N between 2300-2200 cm<sup>-1</sup>, average intensity;

- 2000-1500 cm<sup>-1</sup>, the region of the double bond C=C; - the bond C=O appears between 1830-1650 cm<sup>-1</sup>, the most intense band, C=C much less intense, appears at 1650 cm<sup>-1</sup>, absent frequency;

- 1500-600 cm<sup>-1</sup>, the fingerprint region.

Figure 5 shows the FTIR diagram for each representative sample of the two sets of composite materials we made.

The presence of some maximums to the left of 3000  $cm^{-1}$  indicates the presence of a C=C bond or of an aromatic group in the molecule.

The presence of some maximums to the right of 3000 cm<sup>-1</sup> indicates alkyl groups present in the majority of organic molecules.

A maximum present in the region of 1760-1790 cm<sup>-1</sup> indicates the presence of a carbonyl group. If there is such a band, the additional presence of a O-H group suggests the existence of a carboxylic acid group, the presence of a maximum for the C-O group indicates an ester, for C-H an aldehyde, and for N-H an amide. The lack of additional maximums suggests a ketone.

A broad band for O-H in the region of 3500-3200 cm<sup>-1</sup> indicates the presence of an alcohol or phenol and a single band or two bands in the region of 3400-3250 cm<sup>-1</sup> indicate a secondary, or a primary amine.

If stretches of the C-O bond occur, there may be ether, or an ester if there is also a carbonyl band.

In esthers, the stretch of the C=O bond appears in the 1740-1715 cm<sup>-1</sup> range if the carbonyl carbon is conjugated with an aromatic ring, such as methyl benzoate at 1724 cm<sup>-1</sup>. The stretch of the C-O bond appears as two bands, one of them broader and more intense than the other, between 1300-1000 cm<sup>-1</sup>.

 Table 4

 CHARACTERISTICS OBTAINED FROM THE FTIR ANALYSIS

Wave no. [cm <sup>-1</sup> ]	Intensity	Width	Functional group	Range	Observations
3618	1ow	narrow	<u> </u>		Overtone C=O 1722 (n=2)
3523	1ow	narrow			
3455	average	average	C=0	3450	C=O stretch
3371	low	narrow			stretch OH
2922	average	narrow			stretch C-H vinyl or aromatic
2852	average	narrow			C-H symmetric stretch
2288	1ow	broad			
2163	low	narrow			
1979	1ow	narrow			
1723	high	narrow	C=0	1750-1740	stretch C=O, 20 cm <sup>-1</sup> if unsaturated
1599	1ow	narrow			
1590	low	narrow			
1452	low	narrow	O-CH3	1460-1430	esters, ethers, CH2/CH3 bending vibrations, CH2 around 1465
1250	average	narrow			stretch C-O+C-C+C=O
1120	average	narrow			stretch C=O asymmetric
1062	average	narrow			C-C, C-OH, C-H ring and group vibrations
1019	average	narrow			C-C, C-OH, C-H ring and group vibrations
740	average-high	narrow	-(CH2)n-	740-720	4 or more groups CH2 present band at 720
698	high	narrow	0-C=0	700-590	carboxylic acids, bending O-C=O

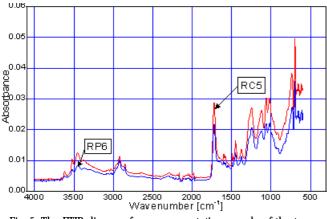


Fig. 5. The FTIR diagram for a representative sample of the two sets of composite materials

#### Conclusions

The analysis of the characteristic curves shows that the admissible resistance to compression of the composite materials obtained increases by a percentage between 50 - 85 % compared to that of the resin used as matrix. This makes these materials recommendable for use in the areas where relatively high pressure is applied to them.

Moreover, we notice that the behaviour between tension and deformation has been almost linear until the phenomenon of sample *breakage* takes place.

The maximums of reduced intensity in the region of  $3100-3000 \text{ cm}^{-1}$  indicate the presence of hydrogen bonded to hybridised carbon sp<sup>2</sup>; the compound will have at least one C=C bond, whereas the absorptions at 3000-2850 cm<sup>-1</sup> indicate that there is hydrogen bonded to hybridised carbon sp<sup>3</sup>.

The intense absorption at 1250 cm<sup>-1</sup>, together with those at 1599, 1452 cm<sup>-1</sup> and those at 3100-3000 cm<sup>-1</sup> and 746 cm<sup>-1</sup> indicate the presence of an aromatic ring, whereas the migration of the carbonyl group at smaller wave numbers is an indication that it is conjugated. All this information leads to the conclusion that the substance in question is an ester.

The esters show maximums specific to bonds in the region of 3000-2850 cm<sup>-1</sup>, produced by the hybridised C-H bond sp<sup>3</sup>, and a high intensity band, at 1723 cm<sup>-1</sup>, due to the carbonyl group of the ester, that being slightly displaced outside the 1750-1740 cm<sup>-1</sup> range because it involves an unsaturated ester and an absorption of the simple bond C-O in the ester at 1250 cm<sup>-1</sup>, which is comparable in terms of width and intensity to that of the carbonyl group.

Given the low cost, the mechanical characteristics achieved after the compression test and based on the design of these composite materials, we may claim that they can successfully be used in:

- the construction materials industry, in order to plate outside foundations, to cast industrial or home floorings, subject to high traffic (decorative floorings), to make sills and ledges, stairs, street pavements;

- the industry of sanitary wares, in order to make wash basins, bath tubs;

- the furniture industry, to make kitchen or bathroom countertops, etc.

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