

# The Mechanical Properties of Some Polymer Composites Based on Natural Rubber

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*This paper presents the development and characterization of polymer composites based on natural rubber, crosslinked using two types of vulcanization systems, as follows: the classical method using sulfur and curing accelerators and the method using peroxide in the presence of a polyfunctional monomer - trimethylolpropanetrimethacrylate. The composites were made with 0 parts to 100 parts rubber (phr) and 40 phr filler content. The physical-mechanical characteristics of resulting composites depend on the crosslinking method and on the composition. The mechanical parameters of materials were derived by processing the transmissibility curves, derived for samples with known dimensions and density. The main vibration frequencies and the quality factor, derived in the vicinity of resonances, allow to derive the elasticity and internal damping of materials.*

**Keywords:** polymer composites, natural rubber, mechanical properties, damping, crosslinking

Elastomeric materials have a wide range of engineering applications due to their properties, including: high extensibility, high strength, high-energy absorption, and high resistance to fatigue [1]. Of these, the best known and most used are those based on natural rubber, because of its unique combination of properties. It provides high strength, outstanding fatigue resistance, high resilience, low sensitivity to strain effects in dynamic applications, in addition to good resistance to creep [2]. It is among the few elastomers that crystallize upon straining, a phenomenon that contributes to its high fatigue life and its high strength without the need for reinforcing fillers. Hence, NR compounds can exhibit a combination of low modulus, high strength and fatigue resistance, and very low damping.

Due to its elasticity and fatigue resistance properties, rubber is a fundamental material in protection against vibrations. The main properties of insulators (dampers) made of rubber are [3-5]: low dynamic compression elastic modulus, the possibility to greatly deform and the ability to take up four times higher mechanical shocks than steel springs, high damping properties, being able to dissipate about one-third of the total vibrational energy, low sound propagation rate of only 0.9% of the sound velocity of the sound in steel and 14% of the sound propagation velocity in the air, serving as a very good noise attenuator, low weight and can work assembled with steel springs, low cost, good processability etc. In comparison with metal insulators (metallic springs), natural rubber ones require no maintenance, have high energy storage capacity and non-linear load deflection characteristics, can accommodate a certain amount of misalignment and are easier to install. Although NR is highly resilient, the small amount of inherent hysteresis serves to dampen resonant vibrations. The typical engineering applications of NR include antivibration mountings, flexible couplings, bridge bearings, bearings for buildings for protection from earthquakes, dock fenders and rail pads [6].

The purpose of this article is to study the influence of the curing system and organic filler – starch, on damping,

rheological, chemical and physical-mechanical properties of mixtures based on natural rubber.

## Experimental part

### Materials

To obtain rubber mixtures the following materials were used:

- Natural rubber (NR), Crep from Sangtvon Rubber Ltd, in the form of white rubber sheets, Mooney viscosity 67.64 ML (1' + 4') 100°C, volatile matter content of 0.5%, nitrogen content 0.45%, ash content of 0.25%, impurity content of 0.026%;

- Amorphous precipitated silica BM30 Egesil -Turkey (surface area 155-195 m<sup>2</sup>/g).

- Starch - produced by Lach-Ner - soluble potato starch (water insoluble substances 0.28%; loss on drying 16.9%, easily biodegradable: BOD, -0.6 g/g- and COD - 1.2 mg/g);

- Glycerine produced by SC Chimreactiv SRL (free acidity 0.02%, density 1.26 g/cm<sup>3</sup>, purity 99.5%);

- Crosslinking agents:

- For vulcanization of mixtures with sulfur and vulcanization accelerators the following were used:

- Sulfur – yellow powder, melting point 115°C, density 1.96 g/cm<sup>3</sup>, produced by Sigma Aldrich Chemie GmbH.

- 2 mercaptobenzothiazole (MBT)-molecular weight 167.2, melting point 179°C, density 1.42 g/cm<sup>3</sup>.

- N-Cyclohexyl-2-benzothiazolesulfenamide (CBS)-molecular weight 264.4, melting point 101°C, density 1.31 g/cm<sup>3</sup>.

- Tetramethylthiuram disulfide (TMTD) - molecular weight 240.4, melting point 156°C, density 1.43 g/cm<sup>3</sup>.

- For vulcanization of mixtures with peroxide and vulcanization co-agent the following were used:

- Polyfunctional monomer trimethylol-propanetrimethacrylate (TMPT) Luvomaxx TMPT DL 75 (22% percentage of ash, pH 9.2, density 1.36g/cm<sup>3</sup>, 75.63 % active ingredient), produced by Lehmann & Voss & Co

- Perkadox 14-40B (density 160 g/cm<sup>3</sup>, 3.8% active oxygen content, 40% peroxide content, pH 7) produced by Akzo Nobel Functional Chemicals.

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-Maleic anhydride for synthesis, S6855208, produced by Merck KGaA, Germany (melting point 52°C).

-Polyethylene glycol PEG 4000 – produced by *Advance Petrochemicals Ltd.* (density 1.128 g/cm<sup>3</sup>, melting point 4-8°C).

-Other ingredients: premium quality zinc oxide (produced by SC Werco Metal SRL), stearin (produced by Stera Chemicals), promoter of adhesion between mineral filler and Luvomaxx TESPT DL50/L polymer (produced by Lehmann & Voss & Co).

### Obtaining Test Specimens

#### Plasticizing Starch

Starch is dried at 80°C for 24 h. Two parts starch are mixed with one part glycerin for 7 min until obtaining a homogeneous mixture. The required amount of plasticized starch was made for each mixture.

*Obtaining the compatibilization agent* for NR (hydrophobic) and starch (hydrophilic) - maleated natural rubber (NR-g-AM) was obtained by roll mixing NR with 5 phr (parts per 100 parts of rubber) of maleic anhydride for synthesis and 0.75 phr of Perkadox 14-40B, at a temperature of 160°C for 30 min and then used as such.

*Obtaining mixtures* - Composition of mixtures is presented in table 1.

Mixtures were made in the Brabender mixer, and vulcanization agents were added using a laboratory roll mill. Working parameters for the Brabender mixer were: rotational speed 10/115 rotations/min, temperature for 75°C up to max 110 °C.

Vulcanization agents: sulfur and vulcanization accelerators, namely peroxide and vulcanization co-agent, are embedded on the roll mill. After homogenisation of the mixture, it is taken off the roll in the form of a sheet.

*Vulcanization* in order to obtain test specimens for characterization of composites was made using the compression method in a hydraulic press. Working parameters: temperature 165°C, pressing force 300 kN, cooling time 8', at a temperature of 35°C and the required curing time is determined using the Monsanto rheometer.

### Characterization of samples

*Curing characteristics* were determined by an oscillating disk rheometer (Monsanto), at 165°C for 24 min, according to the ISO 3417/2008. Delta torque or extent of crosslinking is the maximum torque (MH) minus the minimum torque (ML). Optimum cure time ( $t_{90}$ ) is the time to reach 90 % of the delta torque above minimum.

Physical-Mechanical Characteristics Tensile strength and tearing strength tests were carried out with a Schopper

strength tester with testing speed 460 mm/min, using dumb-bell shaped specimens according to ISO 37/2012, and angular test pieces (Type II) according to EN 12771/2003, respectively. Hardness was measured by using a hardness tester according to ISO 7619-1/2011 using 6-mm thick samples. Elasticity (rebound resilience) was evaluated with a Schob test machine using 6-mm thick samples, according to ISO 4662/2009.

Determination of liquid action were performed according to ISO 1817/2015 and the action of the following liquids was tested: water and toluene. The test pieces of known weight,  $m_0$ , were immersed in various solvents in diffusion test bottles and kept at room temperature for 22 h. After immersion the samples were taken out from the solvents and the wet surfaces were quickly dried using a tissue paper and re-weighted,  $m_1$ .

To calculate the percentage change in mass  $\Delta m_{100}$  the following formula was used (1):

$$\Delta m_{100} = \frac{m_1 - m_0}{m_0} \times 100 \quad (1)$$

where:

$m_0$  is the initial mass of the test piece and

$m_1$  is the mass of the test piece after immersion.

To calculate the percentage change in volume  $\Delta V_{100}$  the following formula was used (2):

$$\Delta V_{100} = \left( \frac{m_1 - m_{i,w} + m_{s,w}}{m_0 - m_{0,w} + m_{s,w}} - 1 \right) \times 100 \quad (2)$$

where

$m_{0,w}$  is the initial mass of the test piece (plus sinker if used) in water;

$m_{1,w}$  is the mass of the test piece (plus sinker if used) after immersion in water;

$m_{s,w}$  is the mass of the sinker, if used, in water.

Report the result as the median value for the three test pieces.

*The damping evaluation method.* Disk-shaped samples were used to evaluate damping characteristics, with the following dimensions: thickness 6 mm, diameter 10 mm. The samples were placed on a vibration exciter, fixed with a double-side adhesive tape. A reference accelerometer was placed on the exciter mobile table. A second accelerometer was placed on the upper part of the sample, fixed with double-sided adhesive tape. The vibration exciter is driven through a power amplifier, by the testing signal produced on the output channel of an impedance analyzer. The SR-785 Stanford Research analyzer has one analog output channel and two input channels, with simultaneous

**Table 1**  
COMPOSITION OF NATURAL RUBBER  
MIXTURES AND PLASTICIZED  
STARCH

| Mixture symbol                         | S0C | S40C | S2x20C | S0S | S40S | S2x20S |
|--|-----|------|--------|-----|------|--------|
| Natural rubber (g)                     | 95  | 95   | 95     | 95  | 95   | 95     |
| NR-g-AM (g)                            | 5   | 5    | 5      | 5   | 5    | 5      |
| Starch plasticized with glycerin (g)   | -   | 60   | 30     | -   | 60   | 30     |
| Egesil BM30                            | -   | -    | 20     | -   | -    | 20     |
| TESPT DL50/L                           | -   | -    | 3      | -   | -    | 3      |
| Zinc oxide (g)                         | 1   | 1    | 5      | 5   | 5    | 5      |
| Stearic acid (g)                       | 0.1 | 0.1  | 0.5    | 0.5 | 0.5  | 0.5    |
| PEG 4000 (g)                           | 3   | 3    | 3      | 3   | 3    | 3      |
| Antioxidant 4010 (g)                   | 1   | 1    | 1      | 1   | 1    | 1      |
| Peroxide (g)                           | 8   | 8    | 8      | -   | -    | -      |
| TMPT (g)                               | 3   | 3    | 3      | -   | -    | -      |
| Sulfur (g)                             | -   | -    | -      | 1.5 | 1.5  | 1.5    |
| Mercaptobenzothiazole (MBT) (g)        | -   | -    | -      | 0.5 | 0.5  | 0.5    |
| Tetramethylthiuramdisulfide (TMTD) (g) | -   | -    | -      | 0.5 | 0.5  | 0.5    |
| Sulfur (g)                             | -   | -    | -      | 0.5 | 0.5  | 0.5    |

signal acquisition (fig. 1). Few testing methods are available in the vibration testing theory for measuring the transmissibility of a mechanical system, depending on the excitation signal: impulse testing, sweep sine or swept sine testing, white or pink noise testing [7-12].

The impulse testing is the fastest method but it is recommended for systems having resonance at high frequencies. In this case, preliminary tests showed low resonance frequencies, requiring for testing an excitation with an impulse not very thin in time. This excitation could be obtained with an impact hammer having a high inertial mass, producing a large displacement upon impact. The non-linear behavior, specific to composite rubber materials, is very prominent at large deformations, canceling the effectiveness of method. Also the method is not useful for systems with low damping, because there is a high risk of affecting the system [7-12].

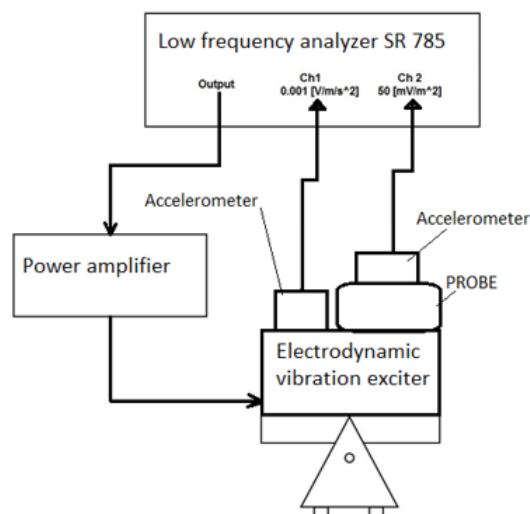


Fig. 1. The structure of experimental setup for transmissibility measurement using vibration exciter

The testing with sinusoidal signal, having a frequency modified continuously -for sweep sine testing-or in steps-for swept sine testing - in the useful range, has the disadvantage of a long testing time but very important advantages: ability to test a system with non-linear behaviour (the acquired signals are filtered with narrow band filters centered on the testing frequency, removing the unwanted harmonics at other frequencies, produced due to nonlinearities), possibility to adapt the excitation signal to the system response in real time (the tested systems having sharp resonances requires high amplitude signals at frequencies far from the resonance and low amplitude signals near the resonances).

The testing with white or pink noise is faster than sweep/ swept sine testing but more precise than impulse testing. The signal could be pre-shaped in frequency spectrum by filtering with a filter attenuating at resonance frequencies. The main disadvantage of a wide frequency excitation is still present, not recommending the method for systems with small nonlinearities.

The preliminary studies, with different types of excitation signals (for impulse testing the vibration exciter is blocked and an impact hammer with a force transducer is used), proved that the best method for this kind of materials is the swept sine testing.

The analyzer produces the excitation harmonic signals and acquires the signals from accelerometers, deriving the ratio of amplitudes (upper/lower acceleration signals) and phase difference (upper minus lower acceleration signals), for different frequencies. The resulting functions of mechanical transmissibility are graphically represented (fig. 2).

The resonance frequency depends on the Young elasticity modulus, density and damping (the damping influence is small in the resonance frequency and many times is neglected). The damping is strongly influencing the quality factor, defined as the difference between the

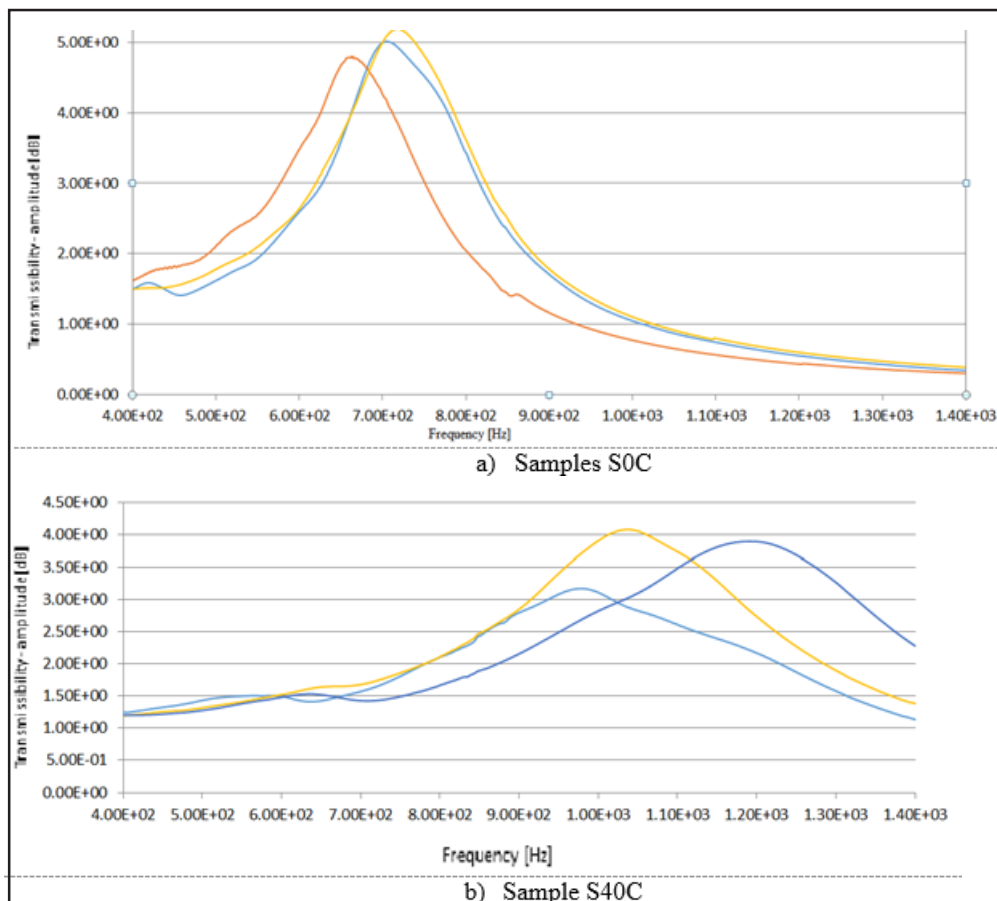


Fig. 2. The resulting functions of mechanical transmissibility for samples

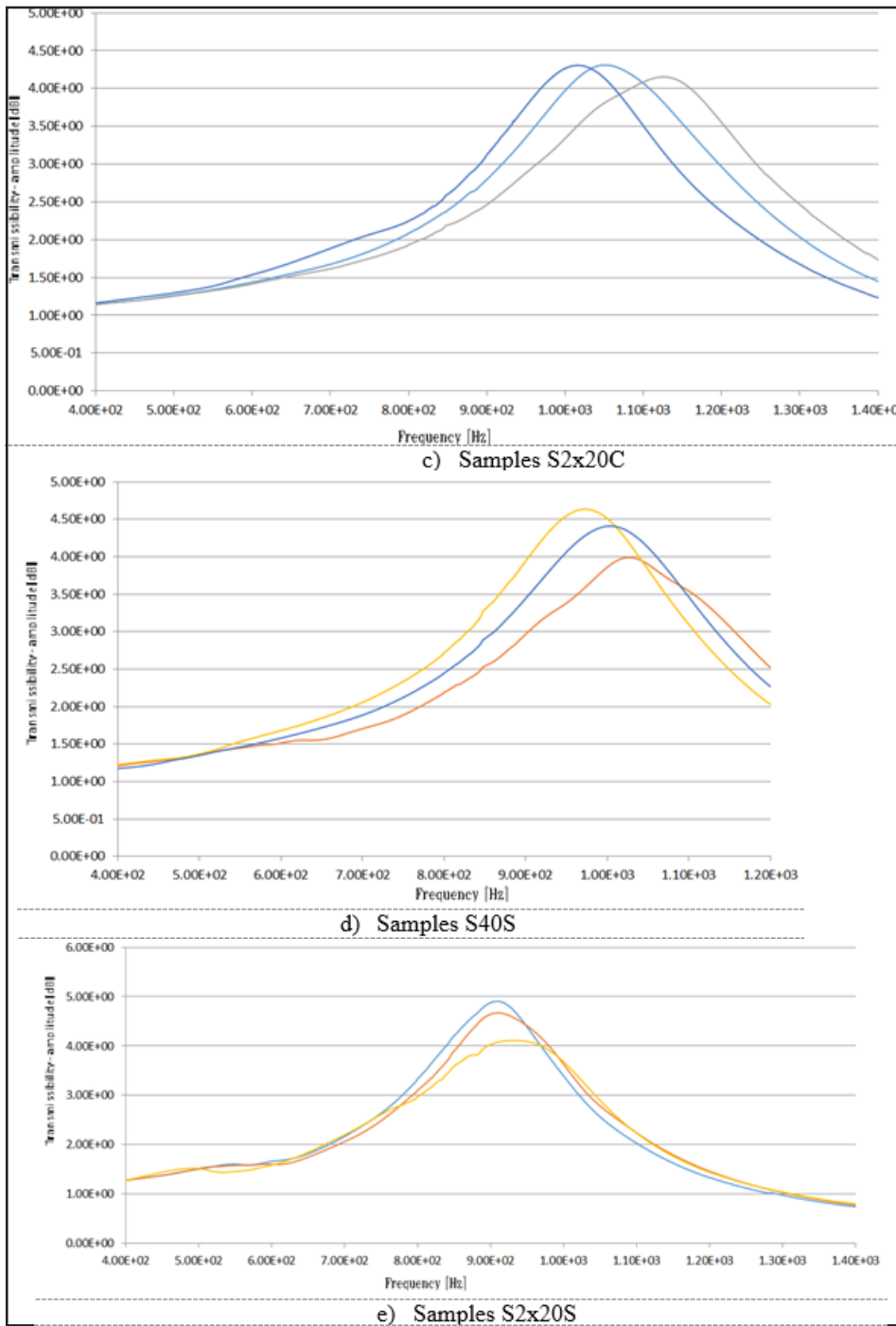


Fig. 2. Continued

frequencies with a -3dB attenuation in comparison with the maximum resonance (the half-power frequencies) and the resonance frequency.

For each type of material 3 samples were tested, the transmissibility curves are processed and the average values of damping are calculated.

The method used for deriving the damping factor assumed a Solid Type 1 (hysteretic damping) [7], which means a slow variation of Young's modulus and damping factor with frequency, being considered constant in the frequency range of interest.

$$T = \frac{\sqrt{1+\delta_E^2}}{\sqrt{(1-\Omega^2)^2 + \delta_E^2}} \quad (3)$$

where:

$\Omega = \omega / \omega_0$  - the ratio between the angular frequency  $\omega$  and the resonant frequency  $\omega_0$ ;

$\delta$  = the damping factor.

As the maximum of transmissibility occurs at  $\Omega=1$ , the damping factor could be calculated with:

$$\delta_E = \frac{1}{\sqrt{T_{max}^2 - 1}} \quad (4)$$

Other assumptions could be made (a very high hysteresis model, meaning Young's modulus is assumed to be directly proportional to the frequency and the damping factor is considered to be independent of the frequency, or a spring-damper/dashpot model) but previous studies shown small differences between methods of evaluating the damping factor using transmissibility measurements [7].

## Results and discussions

### Rheological characteristics

The analysis of data presented in table 2 shows that the minimum torque -  $M_{min}$  (a measure of rigidity of the unvulcanized material) and the maximum torque -  $M_{max}$



| Characteristics/ Mixture symbol | S0C   | S40C  | S2x20C | S0S   | S40S | S2x20S |
|---------------------------------|-------|-------|--------|-------|------|--------|
| $M_{min}$ (dNm)                 | 13.5  | 10.6  | 7.8    | 32.6  | 8.5  | 6.4    |
| $M_{max}$ (dNm)                 | 72.4  | 40.3  | 74.7   | 49    | 33.8 | 62     |
| $T_{90}$ (minutes)              | 29.57 | 25.43 | 8.6    | 18.53 | 9    | 1.36   |

**Table 2**  
RHEOLOGICAL  
CHARACTERISTICS OF  
MITURES

| Physical-mechanical properties/<br>Mixture symbol | S0C  | S40C | S2x20C | S0S  | S40S | S2x20S |
|---|------|------|--------|------|------|--------|
| Hardness, $^{0}Sh A$                              | 49   | 57   | 56     | 38   | 48   | 48     |
| Elasticity, %                                     | 58   | 32   | 40     | 56   | 30   | 36     |
| 100 % modulus, $N/mm^2$                           | 0.91 | 0.77 | 0.89   | 0.68 | 0.05 | 0.76   |
| Tensile strength, $N/mm^2$                        | 1.9  | 1.7  | 8.9    | 13.3 | 7.2  | 12.5   |
| Elongation at break, %                            | 220  | 247  | 580    | 660  | 780  | 750    |
| Residual elongation, %                            | 15   | 17   | 26     | 16   | 24   | 38     |
| Tear strength, $N/mm$                             | 8    | 12.5 | 23     | 25   | 14   | 22     |

**Table 3**  
PHYSICAL-MECHANICAL  
PROPERTIES OF  
COMPOSITES

(a measure of the rigidity of the fully vulcanized material at the curing temperature) shows lower values for samples to which plasticized starch was added because glycerin (plasticizer) reduced the rigidity of the material. Mixtures containing precipitated silica as filler yielded higher  $M_{max}$  values due to the property of active inorganic filler to reinforce rubber mixtures. The optimal curing time has lower values for filled mixtures, because the filler increased the crosslinking density of samples [13].  $M_{max}$  and  $M_{min}$  have different values in the case of crosslinking using sulfur and accelerators compared to crosslinking with peroxides because sulfur crosslinking occurs by means of an ionic mechanism, when sulfur bridges are formed between macromolecules and peroxide crosslinking occurs by means of a radical mechanism, when C-C bonds are formed between macromolecules, which are more rigid than those formed within the former crosslinking method [14-16].

#### Physical-mechanical properties

Physical-mechanical properties of resulting composites are presented in table 3.

Analyzing characteristics, it can be seen that in the case of mixtures containing starch: (a) hardness increases indicating that starch leads to reinforcement of NR, and therefore can replace active fillers such as carbon black or precipitated silica which are harmful and may cause occupational illnesses of employees in the rubber processing industry [13]; (b) elasticity decreased as a result of diminishing the amount of natural rubber in the mixtures as well as due to mixture reinforcement; (c) elongation modulus of 100%, tear and tensile strength show non-uniform variation upon increasing the starch amount; (d) elongation at break and residual elongation increase by 12-18.2%, and 13-50%, respectively.

Mixtures containing active filler such as precipitated silica in addition to plasticized starch had better values of tensile strength, elongation at break and tear strength, due to the ability of active fillers to improve physical-mechanical properties of rubber mixtures [17-19]

Comparing characteristics of mixtures crosslinked with peroxide and vulcanization co-agent with those of mixtures crosslinked with sulfur and curing accelerators, it is seen that: (1) Hardness and elasticity values are higher in mixtures crosslinked with peroxide; (2) Elasticity modulus, tensile strength, elongation at break and tear strength have better values in the case of mixtures crosslinked with sulfur and curing accelerators. These changes in the physical-mechanical characteristics are due to the types of bonds established between elastomeric chains during vulcanization. In the case of crosslinking using sulfur and curing accelerators, sulfur bridges are formed between macromolecules, there is a mobile crosslinking that shows the ability of the bond to shift under strain so that it allows good tensile and tear strengths. Instead, crosslinking with peroxides leads to the formation of C-C bonds between macromolecule chains, that are much more stable but rigid and do not allow mobility/ rotation/ shift in applying a force, which leads to lower elasticity and tensile / tear strengths compared to crosslinking with sulfur and curing accelerators [14-16].

#### Action of Liquids on composites

The results of liquid action on composites are presented in table 4. The data show that it depends on the behaviour of mixture components in immersion media; thus, natural rubber is water resistant (variation of mass and volume in water has very low values) but not resistant to sunflower seed oil (variation of mass and volume has high values); starch has a hydrophilic nature - it absorbs water and, with the increased amount of starch in the mixtures, there is

| Chemical properties /Mixture symbol          | S0C   | S40C  | S2x20S | S0S   | S40S  | S2x20S |
|--|-------|-------|--------|-------|-------|--------|
| Variation of mass in water, %                | 0.86  | 8.58  | 8.25   | 0.99  | 10.04 | 3.47   |
| Variation of volume in water, %              | 1.30  | 14.49 | 13.8   | 1.50  | 16.75 | 5.91   |
| Variation of mass in ethyl alcohol, %        | 2     | 9.55  | 6.39   | 1.82  | 10.50 | 4.7    |
| Variation of volume in ethyl alcohol, %      | 3.04  | 16.13 | 11.1   | 2.74  | 17.12 | 8.16   |
| Variation of mass in sol. 0.5% glucose, %    | 0.44  | 6.93  | 7.68   | 0.7   | 2.54  | 2.98   |
| Variation of volume in sol. 0.5% glucose, %  | 0.67  | 11.72 | 12.8   | 1.05  | 12.83 | 5.08   |
| Variation of mass in sol. 0.9% NaCl, %       | 0.48  | 6.67  | 9.04   | 0.91  | 9.17  | 3.20   |
| Variation of volume in sol. 0.9% NaCl, %     | 0.73  | 11.33 | 15.04  | 1.37  | 15.05 | 5.45   |
| Variation of mass in sunflower seed oil, %   | 11.47 | 9.17  | 12.1   | 16.67 | 15.30 | 8.64   |
| Variation of volume in sunflower seed oil, % | 17.94 | 16.07 | 21.3   | 25.04 | 25.41 | 14.67  |

**Table 4**  
VARIATION OF MASS AND  
VOLUME IN VARIOUS  
LIQUIDS

| Material           | S0C  | S40C | S2x20C | S40S | S2x20S |
|--------------------|------|------|--------|------|--------|
| Damping factor (-) | 0.20 | 0.27 | 0.24   | 0.23 | 0.22   |

**Table 5**  
THE MECHANICAL CHARACTERISTICS DERIVED  
THROUGH VIBRATION ANALYSIS

also an increase of mass and volume variation in water. The results for samples crosslinked with sulfur and curing accelerators are similar to those obtained for samples crosslinked with peroxide and vulcanization co-agent.

#### *The damping factor*

From the transmissibility curves (fig. 2) for each type of material, the average values of damping were calculated. They are presented in table 5. Results indicate that the damping factor varies both with the type of crosslinking, and with the type of filler. Higher values were obtained for mixtures crosslinked with peroxide in the presence of a curing co-agent. Filled mixtures have a better damping factor than unfilled ones. The best values were obtained for mixtures containing organic filler such as plasticized starch. These results are in accordance with the literature data on the effect of organic filler and curing on damping properties of vulcanizates [1, 20-21]

From the point of view of using these materials for damper construction, the most efficient is the material with the highest damping factor. The decision of choosing the optimum material should be correlated with the request of a high elastic modulus, if the damper also has an elastic role (if the damper is working in parallel with a spring, it is acceptable to have a low elastic modulus, if the damper acts also as a structural element, supporting the structure, it is very important to have small deformations and good capacity to sustain loads).

#### **Conclusions**

The study leads to the following observations: (1) starch has led to the reinforcement of NR and to the improvement of the damping constant, but also decreased the resistance of rubber mixtures to immersion in water or aqueous solutions, (2) mixtures cured with peroxide and curing co-agent have shown better values for hardness, elasticity and damping constant, (3) mixtures also containing active filler such as precipitated silica yielded better values of physical-mechanical properties (tensile strength, elongation at break, tear strength).

For building dampers with structural role, the S40C is optimum while for dampers working together with spring, S2x20C could be used.

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