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MANUFACTURING PROCESS OPTIMIZATION OF RESILIENT MATERIALS MADE FROM RECYCLED TYRE GRANULES

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The disposal of used tyres is a significant environmental problem in developed countries. The production of acoustic materials from rubber crumbs can therefore represent a valid alternative to incineration or to the disposal of used tyres into landfill.
The paper presents the first results of the collaboration between the Department of Industrial Engineering of the University of Perugia and the European Center of Nanostructured Polymers. The goal of this multidisciplinary partnership is to analyze and optimize the manufacturing process of impact sound insulating materials made of recycled tyre granules mixed with binders by studying the different stages of production.
Several prototypes were manufactured by means of the developed process and tested in order to evaluate their dynamic stiffness in compliance with the European standard EN 29052-1:1993. The indexes of impact sound reduction ΔL_w were also estimated.
Finally, the prototypes showing the best properties were produced in bigger size and tested in two overlapping reverberating rooms according to the standard ISO 140-8. Results confirm that the materials manufactured through the developed process have satisfactory acoustic properties, comparable to the ones of commercially available materials.

1. Introduction

Recycled tyre granules have been recently proposed for manufacturing acoustic insulating and absorbing materials, to be used for noise control in buildings and road barriers.

Materials made of rubber crumbs usually have high porosity and consequently show good sound absorbing properties over a wide frequency range. Furthermore, the use of these materials in insulating underlays shows satisfactory performance for acoustic impact insulation.

The development of novel acoustic materials made from end of life tyres can possibly be a solution for the disposal of these materials. Used tyres represent in fact a huge amount of material to be disposed. Around 2.500.000 tons of used tyres per year have been produced in the last years in the European Union; this number grows to 3.250.000 tons in the United States. The largest reuse market is tyre-derived fuel, followed by civil engineering applications such as highway embankments; the production of rubber crumbs is however growing and the subsequent applications are gaining importance [1].

Within this context, the goal of the paper is to develop an innovative process using recycled tyre granules to produce materials for acoustic applications, in particular resilient insulating layers. Since the grains themselves show no mechanical strength, it is necessary to mix them with an adequate binder and to consolidate the compound in order to create a solid structure. The parameters that mainly influence the properties of this kind of materials are grain size, binder type and concentration, compaction ratio, final thickness.

In order to develop an optimized manufacturing process, a collaboration between the Department of Industrial Engineering and the European Center of Nanostructured Polymers of the University of Perugia was started.

After a brief Literature review, the paper presents the chemical characterization of the materials, the description of the manufacturing process and the results of the experimental campaign carried out at the Acoustic Laboratory of the University of Perugia, focused on damping properties of various sample materials made from tyre granules.

2. Literature review

Several Authors studied the acoustic properties of materials made from rubber crumbs. Most of the researches found in Literature mainly focus on the acoustic absorption properties of these materials: Pfretzschener and Rodriguez [2] demonstrated among the firsts that recycled rubber products show excellent performance of broadband acoustic absorption; Horoshenkov and Swift [3] provided a comprehensive description of the sound absorption properties of both loose and consolidated rubber crumbs. Sobral et Al. [4] correlated the sound absorption properties of bound rubber crumbs to the mechanical ones. More recently, Hong et Al. [5] studied improved acoustic performance by combining double-layer structures of rubber particles with porous materials and perforated panels, obtaining satisfactory values of the absorption coefficient.

Although many studies found in Literature deal with the prediction and measurement of the damping properties of rubber-based materials [6], [7], the ones concerning rubber crumbs from recycled tyres are much more limited. Lapcik et Al. measured the dynamic stiffness of recycled rubber based mats to be used for railway track applications [8]. Previous works by the Authors [9], [10] evaluated the dynamic stiffness of rubber crumbs underlays to be used in buildings for impact sound reduction, obtaining encouraging results.

3. Materials characterization

3.1 Rubber crumbs characterization

Thanks to the instrumentation available at the European Center of Nanostructured Polymers of the University of Perugia, several analysis were first of all performed to investigate the main physical and chemical characteristics of the rubber grains derived from scrap tyres and in particular: grain size analysis, thermo-mechanical and thermo-gravimetric characterization and differential scanning calorimetry.

The composition of the rubber was studied by means of a SEM-FEG electronic microscope equipped with a EDX analysis system. The results in terms of percentage in weight of the chemical elements are: 92,0 % of Carbon, 4,9% of Oxygen, 1,6 % of Sulphur and the 1,5% of Zinc. The content of Hydrogen cannot be measured with this technique. Two images of the rubber taken by the SEM are shown in Fig.1.

Different grain sizes were available (from a thin powder to bigger pieces), but only two of them were considered suitable for the required applications: a “small” size (average diameter of the granules = 1-2 mm) and a “medium” size (3-5 mm). Larger sized grains (6-10 mm) were not used in order to achieve a good homogeneity and thickness (see also section 5).

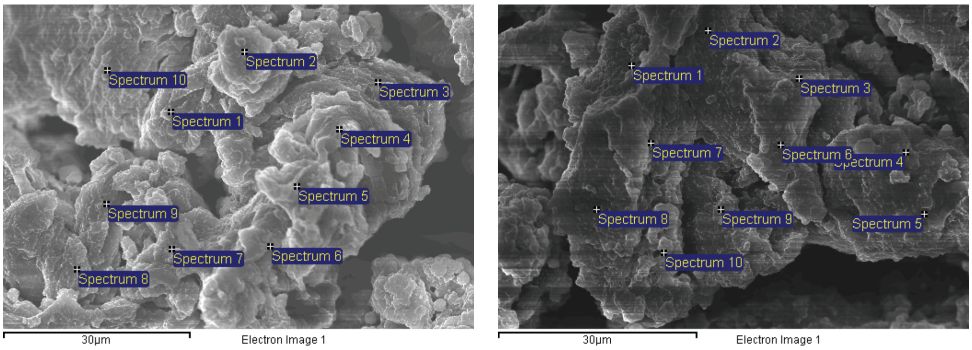


Figure 1. Rubber crumbs (images taken by SEM electronic microscope).

3.2 Binder selection and characterization

As far as the selection of the binder, previous studies by the Authors showed that the most suitable binder for this application is a pre-polymerized polyurethane [9], [10]. A polyurethane binder suggested by a partner chemical company was used for the present activity because of its particular attitude to tie granular materials, in particular rubber crumbs. The technical characteristics of the binder given by the producer are reported in Table 1.

An analysis with a rheometer was performed in order to identify the optimal condition for mixing the rubber crumbs and the binder: the lowest value of viscosity of the binder, i.e. the highest fluidity, is reached when the temperature is 55 °C.

Table 1. Technical characteristic of the binder.

Density, g/cm^3 at 25 °C	1.13
Viscosity, mPa s at 25°C	1700
Isocyanate (NCO) value, % corrected for hydrolisable chlorine	10,20
Flash Point, °C Cleveland Cup, ASTM method D92	227
Fire Point, °C Cleveland Cup, ASTM method D92	293

4. Manufacturing process

All prototypes were manufactured at the European Center of Nanostructured Polymers of the University of Perugia. The manufacturing process can be summarized as follows.

The first steps are weighing the rubber crumbs by means of a precision balance and heating the binder to 55°C, condition of lowest viscosity. Subsequently the binder is weighted and added to the rubber by means of a planetary mixer.

When the rubber crumbs are uniformly covered by the binder the mixture is put in a 26 x 26 cm square die made of steel. The die is then placed in a hot press for 15 minutes at 130°C and 10 bar. Finally, the sample is removed from the die and can be tested after at least 48 hours.

Figure 2 reports some pictures taken during different stages of the manufacturing process.

Figure 2. Different stages of the manufacturing process.

5. Preliminary results

In a previous experimental campaign, six samples were prepared and tested at the Acoustics Laboratory of the University of Perugia, in order to evaluate the influence of some characteristics of the materials – such as grain size, consolidation ratio and binder concentration - on the elastic and acoustic properties in terms of dynamic stiffness [9]. The samples were manufactured by means of a simple cold process using the tools available at the Acoustics Lab [9], [10]. Table 2 reports the main characteristics and the measured values of the dynamic stiffness of the samples.

Table 2. Main characteristics of the tested samples and measured dynamic stiffness.

Name	Grain size	Consolidation ratio (%)	Binder concentration (%)	Thickness (mm)	Dynamic Stiffness (MN/m^3)
M1	Medium	27	10	8	77,2
M2	Medium	22	10	7	71,8
G1	Large	≈0	10	9	41,0
G2	Large	≈0	7,5	12	33,7
G3	Large	≈0	5	13	28,2
GM	Large 80% Medium 20%	≈0	10	14	43,8

The samples made with larger grains show the best acoustic properties because of the inner elasticity of the rubber (in Table 2 see G1 vs. GM); moreover, they do not need to be consolidated. The main problems of the materials made only with large grain size derive from their highly non-homogeneous structure; furthermore, the thickness of the layer depends on the size of the grains.

The comparison of the results obtained for the samples M1 and M2 shows that more consolidated samples have higher dynamic stiffness. However, a certain consolidation is needed to guarantee satisfactory mechanical properties.

The results of the samples G1, G2 and G3 seems to suggest that an increase of the binder concentration causes an increase of the dynamic stiffness of the sample, but the characteristics and the type of the binder greatly influence this effect.

6. Experimental campaign

Two stages of experimental tests were executed. The first session aimed at identifying the optimal characteristics of the materials in terms of grains size and mix, density and binder concentration. In this stage dynamic stiffness measurements were carried out according to EN 29052-1 standard on several small size prototypes (20 x 20 cm.). After choosing the best performing sample, a second session of measurements was performed according to ISO 140-8 standard on a larger size sample (100 x 100 cm.), by means of two overlapping reverberation rooms in order to evaluate the impact sound reduction ΔL.

6.1 First stage: dynamic stiffness measurements

The production of the prototypes was designed taking into account the preliminary results and the evidences coming from other studies in order to optimize the values of grains mix, density and binder concentration for applications as impact insulators.

To this end, 17 typologies of prototypes were manufactured; three samples of each kind were produced (for a total of 51 samples) and the average results are reported in the following sections. The thickness of the samples was 8 mm, a common value for commercial acoustic underlays.

Because of the structure of the material, the lateral flow resistivity is always larger than 100 $kPa \cdot s/m^2$, therefore, the apparent dynamic stiffness s'_t coincides with the dynamic stiffness s'. Moreover, the index of impact sound reduction ΔL_W was estimated from the values of dynamic stiffness according to EN 12354-2 standard, in order to have a simple and immediate term of comparison.

6.1.1 Optimization of the grains' mix

As previously said (see section 3.1), two grains sizes were considered suitable for the required applications: a "small" size (average diameter of the granules = 1-2 mm, in the following indicated with the initial S) and a "medium" size (average diameter of the granules = 3-5 mm, indicated with the initial M).

Five grains'mixes were selected to be tested, keeping constant the binder concentration (13 %): 100 % M, 80 % M + 20 % S, 50 % M + 50 % S, 20 % M + 20 % S, 100 % S. The results in terms of resonance frequency f_r, dynamic stiffness s' and estimated index of impact sound reduction ΔL_W are reported in Fig.3. The best performance was achieved by the sample made of 80 % medium and 20 % small grains: consequently, this mix was considered optimal and chosen for the continuation of the experimentation.

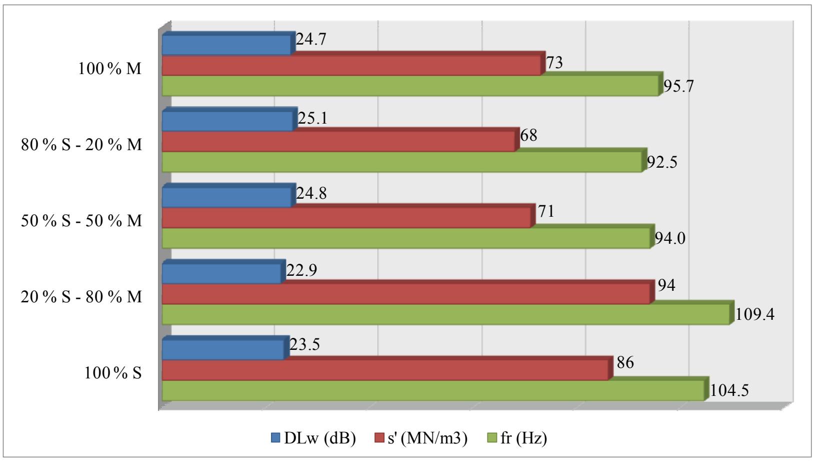


Figure 3. Effect of the grains mix on the elastic and acoustic characteristics of the material.

6.1.2 Optimization of the density

In this stage six prototypes were realized with the same binder concentration (13 %) and mix of the grain sizes (80 % M + 20 % S), changing the quantity of the compound rubber-binder put in the mould (from 292 g to 405 g), keeping constant the size and final thickness of the layer. Higher/lower density were discarded as the corresponding samples were too hard/breakable. The results are reported in Fig.4.

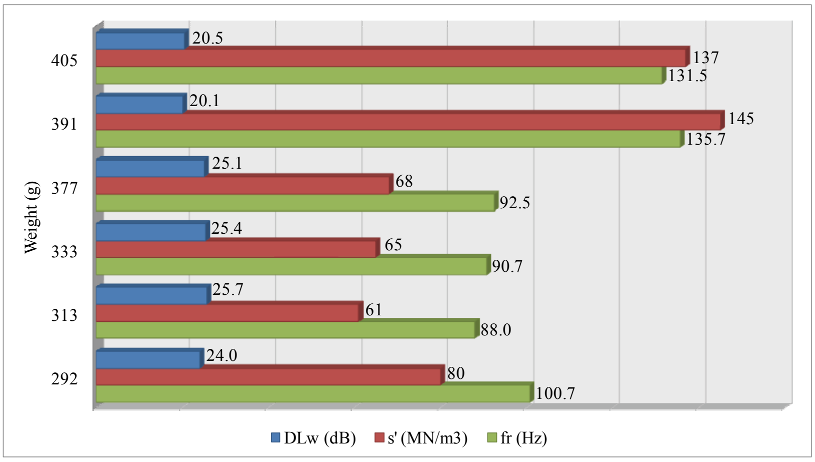


Figure 4. Effect of the density on the elastic and acoustic characteristics of the material.

As expected, denser, i.e. more consolidated, materials showed the worst performance because of the excessive hardening of the solid matrix, while the samples with intermediate density (weight between 313 and 377 g) presented the best elastic characteristics (dynamic stiffness between 61 and 68 MN/m^3).

6.1.3 Optimization of the binder concentration

The binder concentration is defined as the ratio between the mass of the binder and the total mass of the compound rubber-binder multiplied by 100 and expressed as a percentage.

For the samples under test it was observed that the optimal weight was between 313 and 317 grams. For this reason, two sessions of measurements were performed, with two series of samples weighing respectively 313 and 377 grams. The mix of the grain sizes is always 80 % M + 20 % S.

For the first session (weight = 377 g) 4 prototypes were produced with the following binder concentrations: 4,8 %, 7,0 %, 10,3 % and 13,0 %. The results are reported in Fig.5.

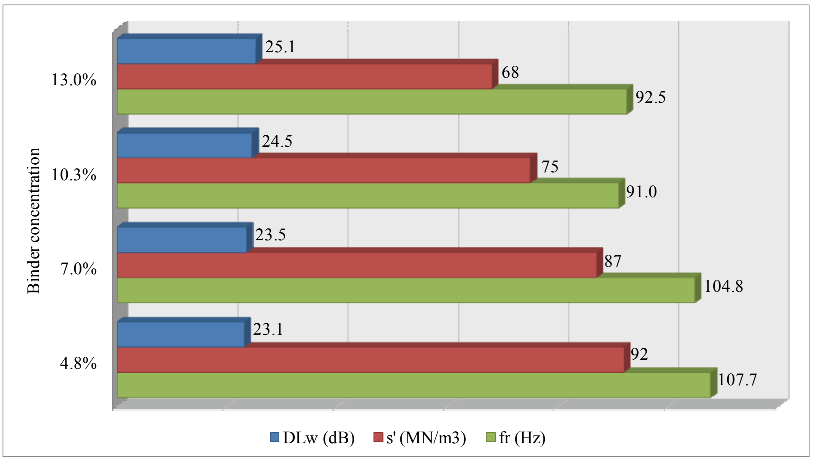


Figure 5. Effect of the binder concentration on the elastic and acoustic characteristics of the material; weight of the samples = 377 g.

The dynamic stiffness decreases almost linearly with increasing values of the binder concentrations, confirming the good elastic properties of the chosen polyurethane binder.

Five prototypes were tested in the second session. The weight of the samples was 313 g while the binder concentration was: 9,1 %, 11,1 %, 13,0 %, 14,9 %, 16,7 %. The results are reported in Fig.6. In this case, the best performance is achieved by samples with binder concentrations equal to 13,0 % and 16,7 %. The price and the environmental impact of this kind of material is principally due to the binder, thus, a binder concentration equal to 13,0 % was chosen to be the optimal one.

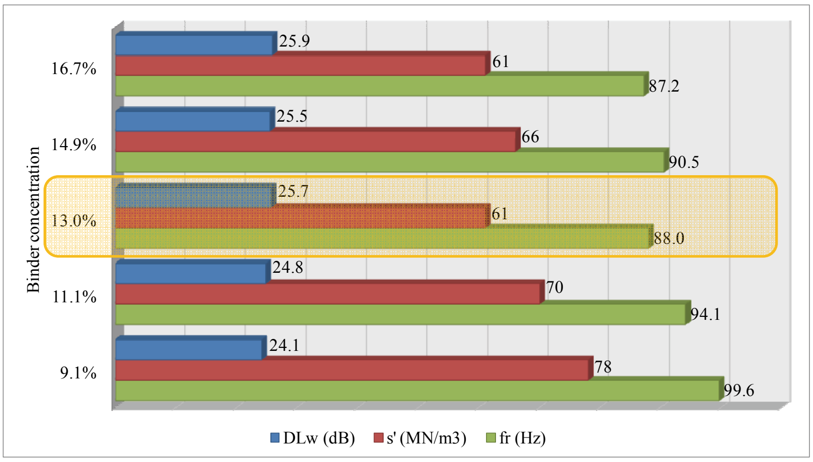


Figure 6. Effect of the binder concentration on the elastic and acoustic characteristics of the material; weight of the samples = 313 g.

6.2 Second stage: impact sound reduction measurements

The first experimental stage allowed to identify the optimal characteristics for the manufacturing of the impact insulator (highlighted in Fig. 6), i.e.:

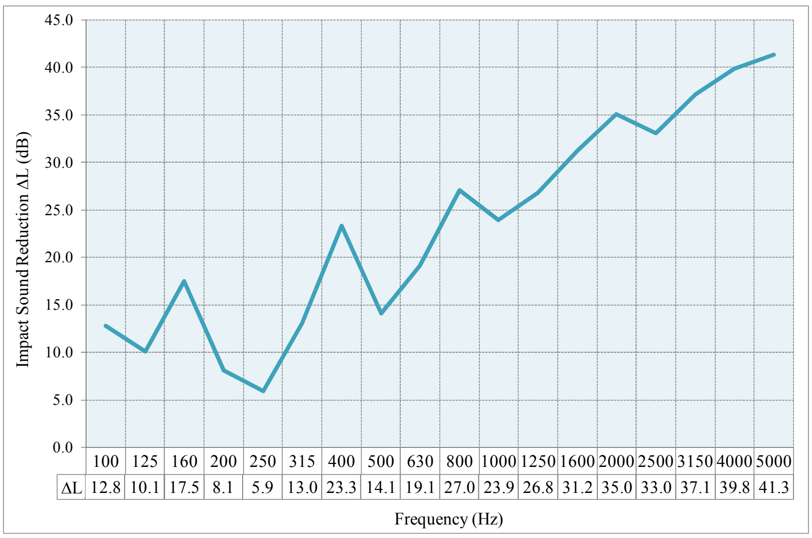
- mix of the grains size: 80 % medium size and 20 % small size;
- binder concentration: 13,0 %;
- weight: 313 g.

The second stage aimed at evaluating the acoustic performance of the optimized underlay by measuring the impact sound reduction ΔL in two overlapping reverberation rooms according to ISO 140-8. It was impossible to produce a larger sample (100 x 100 cm vs. 20 x 20 cm needed for dynamic stiffness measurements) with the manufacturing process described in Section 4: for this reason 16 square 25 cm layers were laid on the floor of the upper reverberation room and joined with tape. A 6 cm thick homogeneous concrete slab was put on the sample (see Fig.7).

The tapping machine was placed in three positions on the sample and three positions on the bare floor around the sample; the measurement was then repeated moving the sample in a different position of the floor of the upper reverberation room. Six microphone positions were used in the receiving room for a total of 72 acquisitions. Also background noise was measured but no correction was needed.

Fig. 8 reports the third-octave bands trend of the impact sound reduction ΔL. The index of impact sound reduction ΔL_W calculated according to ISO 717-2 is 26 dB, in perfect agreement with the value estimated from the dynamic stiffness (25,7 dB).

Figure 7. From left to right: picture of the tested acoustic underlay; picture of the concrete slab on the sample; picture of the tapping machine.



	100	125	160	200	250	315	400	500	630	800	1000	1250	1600	2000	2500	3150	4000	5000
ΔL	12.8	10.1	17.5	8.1	5.9	13.0	23.3	14.1	19.1	27.0	23.9	26.8	31.2	35.0	33.0	37.1	39.8	41.3

Figure 8. Impact sound reduction of the sample vs. frequency.

7. Conclusions

Used tyres represent a serious environmental problem since it is no longer possible in the EU to dispose them into landfills; also the other traditional use of end of life tyres - their incineration in concrete production plants – causes a high environmental impact.

In recent years, many research laboratories have studied, both from a theoretical and an experimental point of view, new materials made of tyre granules of various dimensions, bounded with appropriate binders. The paper presents the results of an experimental campaign carried out thanks to a collaboration between the Department of Industrial Engineering of the University of Perugia and the European Center of Nanostructured Polymersat the Acoustics Lab of the University of Perugia, and focuses on impact sound insulation properties of various samples made of tyre granules.

A wide campaign of dynamic stiffness measurements was carried out; the influence on the impact sound insulation properties of granules size, binder concentration, thickness and compaction ratio of the samples was investigated and an optimization process was carried out. The results on 8 mm thick samples made of medium and large size tyre granules with a polyurethane binder show good performance (s'_t = 61 MN/m^3, with a measured ΔL_W = 26 dB), similar to the one of commercially available rubber made materials of the same thickness [11].

The research will therefore continue with the production and testing of larger samples, together with the evaluation of further important non-acoustical parameters such as inflammability and durability.

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