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SPECIAL ISSUE

# EXPERIMENTAL STUDY OF THE MECHANICAL PROPERTIES OF COMPOSITE MATERIALS FROM RUBBER GRINDS FROM END-OF-LIFE TIRES

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ABSTRACT: This paper presents the mechanical experimental measurements conducted on five different tiles made from composite materials containing rubber particles sourced from end-of-life tires (EoLT). Each specimen features varying quantities of rubber particles, quartz sand, and resin. The study focuses on collecting and organizing experimental data, which are then compiled into tables for comparative analysis. The summarized results of these measurements are also presented graphically and thoroughly discussed. The mechanical characteristics of these composite materials, including hardness, strength, and other mechanical attributes, are comprehensively analyzed to assess their viability for various applications. The findings provide valuable insights into the potential of these composites in addressing EoLT waste management challenges.

KEY WORDS: composite materials, rubber particles from end-of-life car tires, mechanical characterization of composite tiles.

## 1 INTRODUCTION

According to the Bulgarian National Waste Management Plan (NWMP-2021-2028) [1], developed by the Ministry of Environment and Water, one of the strategic objectives is focused on waste prevention. The plan aims to transition waste management into sustainable material management, prioritizing the protection of human health and the environment. It advocates for the rational and effective utilization of natural resources, promotes principles of the circular economy, and aims to reduce reliance on resource imports among EU member states. To address this environmental priority, Directive (EU) 2018/851 [2] on waste underscores the importance of waste prevention policies, highlighting several key aspects:

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- Waste prevention is essential for conserving resources and reducing harmful environmental impacts. Promoting innovative production, trade, and consumption models that prevent waste generation is crucial.
- Sustainability in production and consumption is emphasized, with a focus on resource efficiency to consider waste as a resource, facilitating the transition to sustainable resource management and a circular economy model.
- Encouraging the reuse of products containing valuable raw materials helps prevent these materials from becoming waste.

The strategic objective of the current National Waste Prevention Program (NWPP 2021-2028) [3] is to decouple economic growth and prosperity from an increase in waste quantities and their harmful impact on the environment and people. The aim is to reduce waste quantities and hazardous substances contained within them, with main measures outlined in the Action Plan for the program [4].

Additionally, addressing the challenges related to the storage and recycling of end-of-life tires (EoLT) is highly complex, labor-intensive, and expensive, not only in Bulgaria but also globally. The processing of EoLT and the extraction of valuable components or energy from them pose serious environmental risks. Direct burning of EoLT releases carcinogenic substances such as biphenyl, anthracene, and pyrene. Consequently, Directive 1999/31 [5], adopted by the European Council on April 26, 1999, introduced a ban on burning EoLT altogether from 2003. When decomposing in a natural environment or buried in the soil, EoLT release toxic substances like diphenylamine and phenanthrene, polluting soil and groundwater when washed away by rainwater. Therefore, both incineration and landfilling of EoLT are considered unacceptable methods.

The European Commission has set strict requirements regarding the recycling of EoLT. There is a requirement that recycled quantities should be no less than 50% annually from the quantity used in Bulgaria (Directive (EU) 2018/851, 2018 [1]).

However, there are certain obstacles to achieving these goals. In Bulgaria and the EU, criteria for the end of waste applicable to residues (waste) from the recovery (recycling) of non-hazardous waste, except for metal waste, have not yet been established. These problems create difficulties in realizing the obtained residues from recycling to the market and using them as raw materials in various productions. This highlights the relevance of the proposed topic and underscores the necessity for the development of new methods for treating EoLT and their ultimate utilization. Of particular interest is the possibility of producing quality composites incorporating rubber crumbs or powders from EoLT, researching their properties, and assessing how they can be used in the production of flooring, either as a primary or supplementary component together with other wastes, PVC, textiles, etc

## 2 EXPOSITION

As it was previously mentioned, one potential strategy to meet the stringent requirements of end-of-life tire (EoLT) waste management involves leveraging the beneficial properties of rubber particles to manufacture composites suitable, for instance, for floor and insulation coverings, specifically tiles.

There are some articles dealing with composite materials with rubber particles from EoLT [6–11]. Most of them present studies of composites with rubber powders from EoLT. For example authors P. Badarayan et al. [8] worked on obtaining mixtures containing rubber and sand for lining drainage systems. Other authors [9–11] are working on the development of composite materials with powdered rubber particles, using them to mix with cement, soil and asphalt for the purposes of supporting structures in road construction. This work, building on our previous research [6, 7], is dedicated to the mechanical characterization of composite materials incorporating rubber particles from end-of-life tires (EoLT) (sized 0.5 mm – 2 mm) and sand. These composites are intended for use in flooring coverings and insulation applications.

To explore more practical solutions and produce an adequate quantity of tiles from composites containing rubber particles for our experimental purposes, it was crucial for us to utilize a silicone matrix. This choice arises from the limitations encountered when employing, for example, a polyester matrix, which fails to produce components with accurate geometry, uniformity, well-defined edges, and smooth surfaces. Also for the producing composite tiles a special tooling has been developed for the injection of the composite laterally through one of the edges of the silicon mold. The resulting tiles have to be with precise dimensions corresponding to the measuring standards and yield results that are both accurate and reproducible [6, 7].

The methods employed in the present paper to investigate the mechanical properties of composite materials containing crumb rubber from end-of-live car tires are according to accepted standards and enable the obtained results to be compared with other studies of composite materials [12–14].

## 3 MATERIALS AND METHODS

This study presents composite materials with rubber particles from EoLT sized 0.5 mm – 2 mm and sand (quartz particles with a size of 0.1 mm). Fig. 1 shows these two components of the composite materials which will be here mechanically characterized.

For our mechanical experimental measurements, we produce test composite specimens (tiles) with a consistent resin content of 40% and varying proportions of rubber crumbs and quartz sand. The dimensions of the test specimens are 71 mm × 71 mm with a thickness of 4 mm, and they are fabricated using a silicon mold and a injection

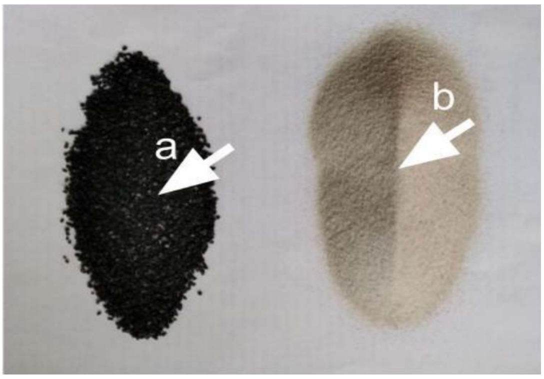

Fig. 1. Components of composite materials: (a) Rubber particles from EoLT sized 0.5 mm – 2 mm; (b) Quartz particles sized 0.1 mm.

molding equipment. The mold undergoes compaction under pressure, reaching up to 2 bar, ensuring proper formation of the tiles.

Homogenization of the composite components is conducted while maintaining the weight percentage of rubber crumbs, quartz particles, and binding agent (resin). The ratio between the components of the composite for the different test specimens is outlined in Table 1 and these 5 type specimens (tiles) can be seen in Fig. 2.

Table 1. Components of the composite material of the 5 different type specimens

| Specimen No | Resin, (%) | Quartz sand, (%) | Rubber particles, (%) |
|---|---|---|---|
| 1 | 40 | 50 | 10 |
| 2 | 40 | 40 | 20 |
| 3 | 40 | 30 | 30 |
| 4 | 40 | 20 | 40 |
| 5 | 40 | 10 | 50 |

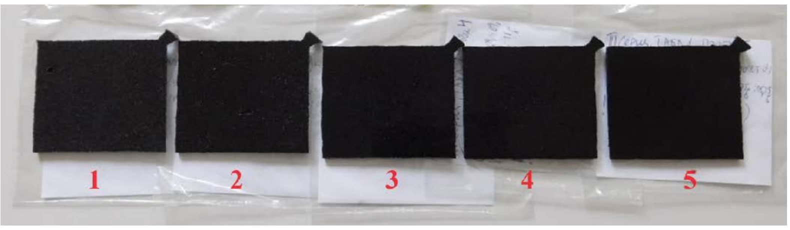

Fig. 2. Specimens with quantitative composition according to Table 1.

For mechanical characterization of these 5 type composite materials we have produced enough quantity of tiles of each type (See Table 1 and Fig. 2) according to the standard requirments [12–16]. We have made experimental measurements to characterize composite materials of each type according to Table 1 and Fig. 2.

We use the following measurement equipments (Figs. 3–7)

Fig. 3. Universal testing system INSTRON 3384.

Fig. 4. CSM REVETEST Scratch Macrotester with diamond indenter Rockwell C with radius 200 $\mu$m.

Fig. 5. Vickers hardness tester 432 SVD by Wilson-Wilpert.

Fig. 6. Mechanical Shore hardness meter (durometer) – D.

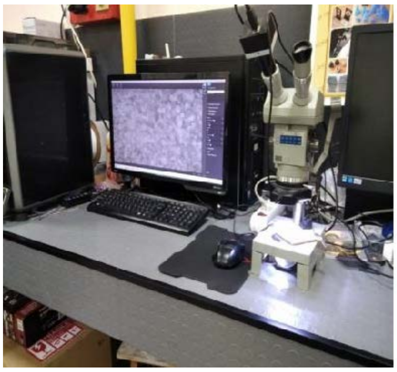

Fig. 7. Carl Zeiss Iena microscope.

- Universal testing system INSTRON 3384 for mechanical strength test in three-point bending (Fig. 3).
- CSM REVETEST Scratch Macrotester, with diamond indenter Rockwell C with radius 200 $\mu$m for scratch tests performed on the top surface of the sample. This machine also records the change in friction coefficient ($\mu$), friction force ($F_t$) and acoustic emission ($AE$) (Fig. 4).
- Vickers hardness tester 432 SVD by Wilson-Wilpert for measurements of the hardness of test specimens (Fig. 5).
- Mechanical Shore hardness meter (durometer) – D for measurements of hardness of test bodies by the Shore method (Fig. 6).
- Carl Zeiss Iena microscope with ×40 optical magnification for light optical microscopy on the top surface of the samples (Fig. 7).

Each test for a given composite type is performed for several test bodies and average results are presented and commented in the next section.

Furthermore, it is well known that statistical methods, such as regression analysis, are commonly employed for analyzing experimental data. Regression analysis usually aims to utilize statistical techniques to estimate relationships between a dependent variable and one or more independent variables. This allows for a deeper understanding of the underlying relationships and factors influencing the observed outcomes. We also will use regression analysis for some of our experimental data.

## 4 RESULTS AND DISCUSSION

All specimens are tested when the resulting composite is fully cured.

To assess the strength of the composite tiles, we utilize a universal testing machine, specifically the INSTRON 3384 (Fig. 3). Employing the Bluehill version 3.15 software, we develop a three-point bending methodology with a bending length of 71 mm and a thickness $h$ of 4 mm (Fig. 8).

The tile has dimensions of 71 mm/71 mm and thickness $h$ is 4 mm. The lower supports (rollers) are at a distance of 50 mm from each other, and the loading upper roller is located centrally between the two (support) rollers (Fig. 3, Fig. 8). The tile is mounted symmetrically to the load roller. It is located on support rollers (1b), which have a diameter of $\O = 10$ mm. The distance between the support rollers is 50 mm. The working roller (1a) is symmetrically located on the support rollers and also has a diameter of $\O = 10$ mm.

The maximum displacement (bending) is set at 20mm, with a loading speed of 10mm/s. Test data is recorded with a step of 0.5N. Testing continues until either the

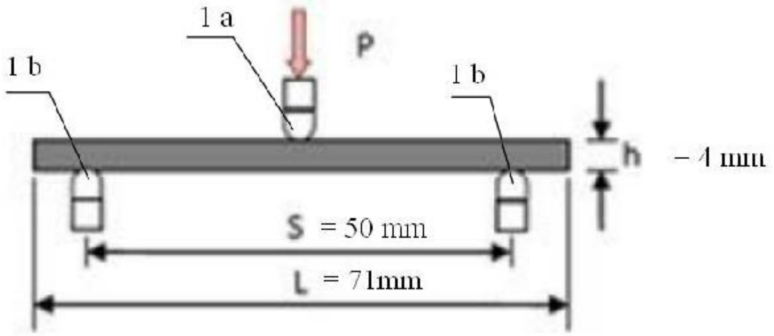

Fig. 8. Position of rollers in three-point bending and tile positioning in universal testing system INSTRON 3384. 1a — working roller; 1b — support roller.

force sharply drops (indicating tile destruction) or when the force decreases below 25%. To ensure accurate calculation of mechanical strength, the real thickness of each sample is considered, facilitating subsequent analysis.

Additionally, scratch tests are conducted on the top surface of the sample using a CSM REVETEST Scratch Macrotester equipped with a Rockwell C diamond indenter of 200 $\mu$m radius (Fig. 4). The scratch test employs a macroscratch technique with a progressive load ranging from 0 to 30 N at a speed of 1 mm/s, recording data on the change in coefficient of friction ($\mu$), friction force ($F_t$), and acoustic emission ($AE$).

A Vickers 432 SVD by Wilson-Wilpert hardness measuring device is used to measure the hardness of the test bodies according to the EN ISO 6507-1:2006 standard [6]. For the hardness measurement, the experiment is carried out with a load of 5 N for a time of 10 s (Fig. 5). The impression obtained is on the top surface. For each of the test bodies, 5 to 10 consecutive measurements are made at steps of at least 100 $\mu$m between the impressions.

Mechanical Shore hardness meter (durometer) – D (Fig. 6) is used to determine specimen hardness at loading 10 N and a hold 10 s.

Data from these experiments is tabulated (Table 2) for subsequent analysis, and comparison charts between different composite tiles are presented in Figs. 9 and 10.

A comparative analysis is conducted for the coefficient of friction ($\mu$) across the five experiments using test bodies from the series with varying percentages of rubber particles and quartz sand, while maintaining a constant percentage of the binder in the composition (RESIN - 40%) (Fig. 9, Table 2).

We use linear regression to determine relationship between $F_t$ – frictional (tangential) force and the load $F_n$ in our experimental measurements for the five composite

Table 2. Data for composition of test specimens, Vickers hardness test, Shore hardness test, flexural strength, friction coefficient and friction force (scale D standard: DIN53505; ISO R868) [7] at $T = 20.4°\text{C}$ and humidity 64.9 ‰

| Specimen | Composition resin : quartz sand : rubber particles from EoLT % | Vickers hardness at loading 5 N | Shore hardness at loading 10 N and a hold 10 s | Material strength max (MPa) | Max load force (on bending) (N) | Coefficient of friction at max (30 N) | Max friction force $F_t$ (N) |
|---|---|---|---|---|---|---|---|
| 1 | 40 : 50 : 10 | 20.07 | 23.07 | 1.86 | 527.05 | 0.42 | 13.08 |
| 2 | 40 : 40 : 20 | 17.97 | 22.93 | 1.44 | 408.93 | 0.38 | 11.72 |
| 3 | 40 : 30 : 30 | 5.33 | 23.00 | 1.15 | 327.06 | 0.44 | 13.6 |
| 4 | 40 : 20 : 40 | 4.77 | 22.03 | 0.74 | 210.93 | 0.51 | 15.56 |
| 5 | 40 : 10 : 50 | 4.77 | 21.30 | 0.74 | 210.68 | 0.48 | 15.04 |

specimens with composition of resin, quartz sand and rubber particles from EoLT according to Table 1. The regression equation represents the relationship between the tangential force ($Y$) and the independent variable load ($X$). R-squared value indicates the goodness of fit of the regression model, with higher values indicating a better fit.

Let we denote $Y = F_t$ and $X = F_n$. We use linear regression for dependent variable $Y$ and independent variable load $X$. The results could be seen in Table 3.

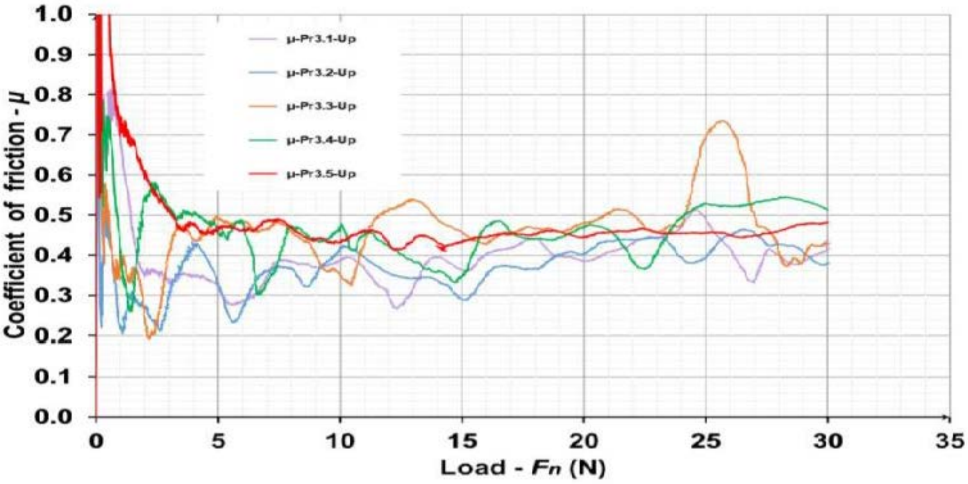

Fig. 9. Friction coefficients for test bodies 1–5 (Table 2).

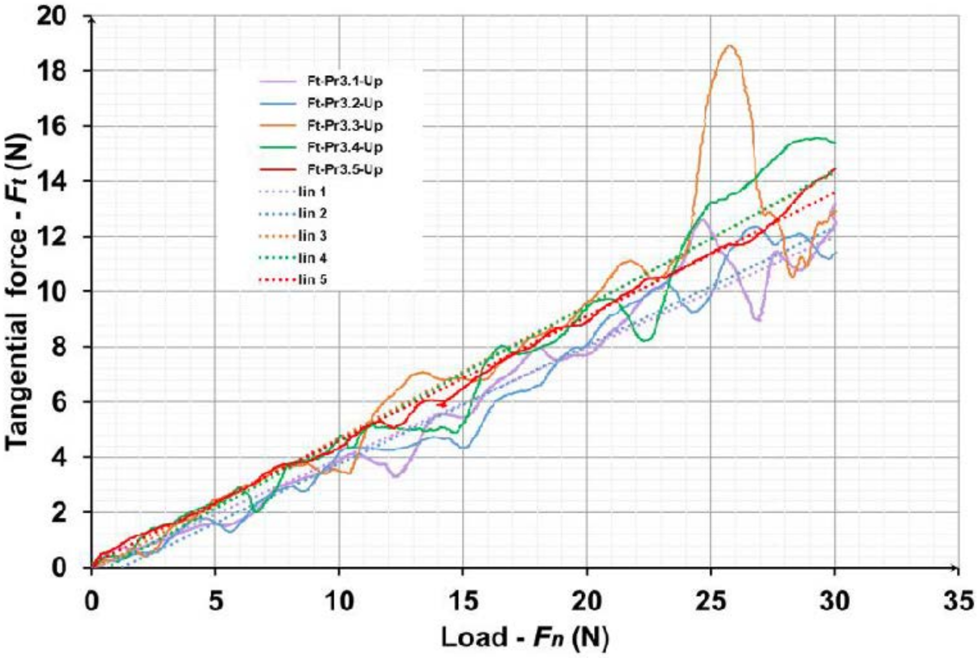

Fig. 10. $F_t$ frictional force (tangential force) on specimens from 1 to 5 at different loads $F_n$ and linear regression graphs according to Table 3, $Y = F_t$ and $X = F_n$.

The coefficient of determination ($R^2$ or r-squared) is a statistical measure in a regression model that determines the proportion of variance in the dependent variable that can be explained by the independent variable. All specimens have r-squared bigger than 0.9, which means that their tangential force is strongly dependent on the load. Especially good is the result for the 5th specimen $R^2 = 0.9961$. This good value is due to the small deviations in the friction force $F_t$, due to the high content of

Table 3. Results from the linear regression analysis of the tangential force for composite materials with composition according to Table (see also Fig. 10)

| Specimen No | Linear regression models | Coefficients of determination |
|---|---|---|
| 1 | $Y = 0.4031 \times X - 0.826$ | $R^2 = 0.9665$ |
| 2 | $Y = 0.4287 \times X - 0.5199$ | $R^2 = 0.9760$ |
| 3 | $Y = 0.4801 \times X - 0.945$ | $R^2 = 0.9119$ |
| 4 | $Y = 0.4902 \times X - 0.3102$ | $R^2 = 0.9601$ |
| 5 | $Y = 0.4495 \times X - 0.12816$ | $R^2 = 0.9961$ |

rubber, which robs the shocks compared to the small amount of the quartz particles.

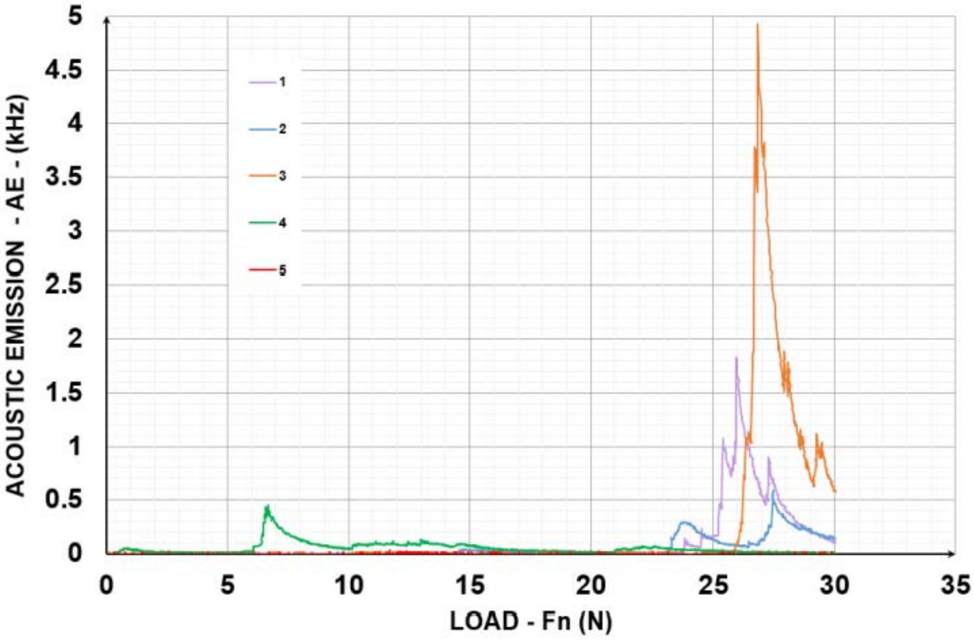

Fig. 11. Acoustic emission ($AE$) for test bodies from 1 to 5.

Further, we provide an acoustic analysis of the specimens using equipment shown in Fig. 4. Figure 11 presents acoustic emission ($AE$) for the 5 type specimens at different loads. From the analysis, the following conclusions can be drawn:

1. As the percentage content of quartz particles increases, $AE$ also increases, reaching the highest values in the third sample, where the contents of quartz and rubber particles are equal.

2. In the third test body, upon reaching a load of $F_n = 26.82$ N, $AE = 4.93$ kHz. This indicates that for composites with this ratio of components, additional noises will be created upon reaching such loads, falling within the audible range (from 100 Hz to 16 000 Hz).

3. In the last test body (Sample 5), where the composition of the composite is as follows: RESIN(%):QUARTZ SAND(%):RUBBER PARTICLES(%) $=$ 40:10:50 (Table 1), it is observed that the acoustic emission is almost absent. This means that this composite may be characterized with the best sound insulation properties.

Table 4 and Figure 12 present the results of Shore hardness measurements on the untreated upper and lower surfaces, as well as on the upper surface after treatment with 10% HCl and 10% NaOH solutions for 36 hours (scale D), following standard procedures (DIN53505; ISO R868) at a temperature of 20.4°C and humidity of 64.9 ‰ [7]. These measurements were conducted using a Mechanical Shore hardness meter (durometer) – D (Fig. 6). After treatment with chemical agents, hardness measurements for the specimens are conducted using the Shore method and the equipment shown in Fig. 6. These measurements are performed on the upper surface of the test body with a load of 10 N and a hold time of 10 seconds. All values obtained from the Shore method measurements are presented in Table 4.

Notably, after treatment, no external visual changes were observed, and there were no signs indicating chemical interaction between the components of the composite and the reagents. The solutions in the petri dishes remained clear, with only dust particles observed falling into the solution from the line of destruction (breaking of the tile). This dusting is attributed to the strength test conducted. During the treatment of the test bodies (Samples 1–5) with 10% solutions of NaOH and HCl for a period of 36 hours, the graph indicates that the deviations in Shore hardness range is from 0% to 5%.

For the first sample body (Sample 1) of the series, with the composite composition: RESIN (%): QUARTZ FLOUR (%): RUBBER PARTICLES (%) = 40:50:10 (Table 1), the average hardness values after treatment with 10% NaOH solution were: untreated : treated = 23.07 : 22.87, representing a change of 0.87% (Table 4, Fig. 12). After treatment with 10% HCl solutions, the ratio of the values obtained was: un-

Table 4. Shore hardness values of untreated upper and lower surfaces and of the upper surface after treatment separately with 10% solutions of HCl and NaOH for 36 hours (the pressure is applied at 10 N for 10 s; scale D standard: DIN53505; ISO R868) at $T = 0.4°$ and humidity 64.9 ‰

| Specimen No | Upper surface untreated | Upper surface treated with NaOH | Upper surface treated wih HCl | Untreated lower surface | Decrease in hardness of upper surface after treatment with NaOH, % | Decrease in hardness of upper surface after treatment with HCl, % |
|---|---|---|---|---|---|---|
| 1 | 23.07 | 22.87 | 22.97 | 22.47 | 0.87 | 0.44 |
| 2 | 22.93 | 22.5 | 22.27 | 22.53 | 1.91 | 2.96 |
| 3 | 23 | 21.9 | 22.03 | 22.13 | 5.02 | 4.40 |
| 4 | 22.03 | 21.37 | 21.37 | 21.07 | 3.09 | 3.09 |
| 5 | 21.3 | 21.3 | 21.23 | 21.17 | 0.00 | 0.33 |

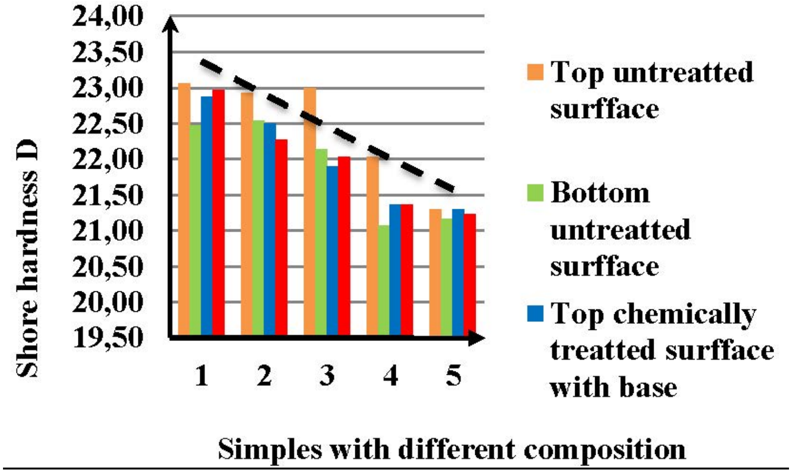

Fig. 12. Measurement of Shore hardness on upper and lower surfaces, as well as on the upper surface after treatment with 10% solutions of HCl and NaOH for 36 hours.

treated : treated = 23.07 : 22.97, indicating a change of 0.44%. The results obtained for this sample demonstrate changes within less than 1%.

For the second sample (Sample 2) from the series, with the composite composition: RESIN (%): QUARTZ FLOUR (%): RUBBER PARTICLES (%) = 40:40:20 (Table 1), the average hardness values after treatment with 10% NaOH solution were: untreated : treated = 22.93 : 22.50, showing a change of 1.91%. After treatment with 10% HCl solutions, the ratio of the values obtained was: untreated : treated = 22.93 : 22.27, representing a change of 2.96% (Table 4, Fig. 12). Deviations observed in this test body are within 3%.

For the third test body (Sample 3) in the series, with the composite composition: RESIN (%): QUARTZ FLOUR (%) : RUBBER PARTICLES (%) = 40:30:30 (Table 1), the average hardness values after treatment with 10% NaOH solution were: untreated : treated = 22.03 : 21.37, representing a change of 3.09%. (Table 1), the average hardness values after treatment with 10%

NaOH solution were: untreated : treated = 23.00 : 21.90, showing a change of 5.02%. After treatment with 10% HCl solutions, the ratio of the values obtained was: untreated : treated = 22.03 : 21.37, showing a change of 3.09% (Table 4, Fig. 12). In this case, after treatment with the working solutions, the same values were obtained in the hardness deviation, with changes up to 3.09%.

For test body (Sample 4) with the composite composition: RESIN (%): QUARTZ FLOUR (%): RUBBER PARTICLES (%) = 40:20:40 (Table 1), the average hardness values after treatment with 10% NaOH solution were: untreated : treated = 22.03 :

21.37, representing a change of 3.09%. After treatment with 10% HCl solutions, the ratio of the values obtained was: untreated : treated = 22.03 : 21.37, showing a change of 3.09% (Table 3, Fig. 9). In this case, after treatment with the working solutions, the same values were obtained in the hardness deviation, with changes up to 3.09%.

Finally, for the last test body (Sample 5), with the composite composition: RESIN (%): QUARTZ FLOUR (%): RUBBER PARTICLES (%) = 40:10:50 (Table 1), the average hardness values after treatment with 10% NaOH solution were: untreated : treated = 21.30 : 21.30, indicating no change. After treatment with 10% HCl solutions, the ratio of the obtained values was: untreated : treated = 21.30 : 21.23, showing a change of 0.33% (Table 4, Fig. 12). Despite the initially lower Shore hardness values due to the high content of rubber particles, the deviations in the studied hardness after treatment were the smallest for this sample, approaching the initial, pre-treatment, measured values.

Overall, the percentage change does not exceed 5%, indicating that the test bodies with these composite compositions are relatively resistant to the action of 10% solutions of HCl and NaOH for 36 hours. Moreover, the two separately applied solutions do not interact with the rubber particles.

Additionally, light optical microscopy is conducted on the top surface of the samples using a Carl Zeiss Iena microscope with an optical magnification of ×40 (Fig. 7), without etching the samples. To ensure comparability of the survey results for all

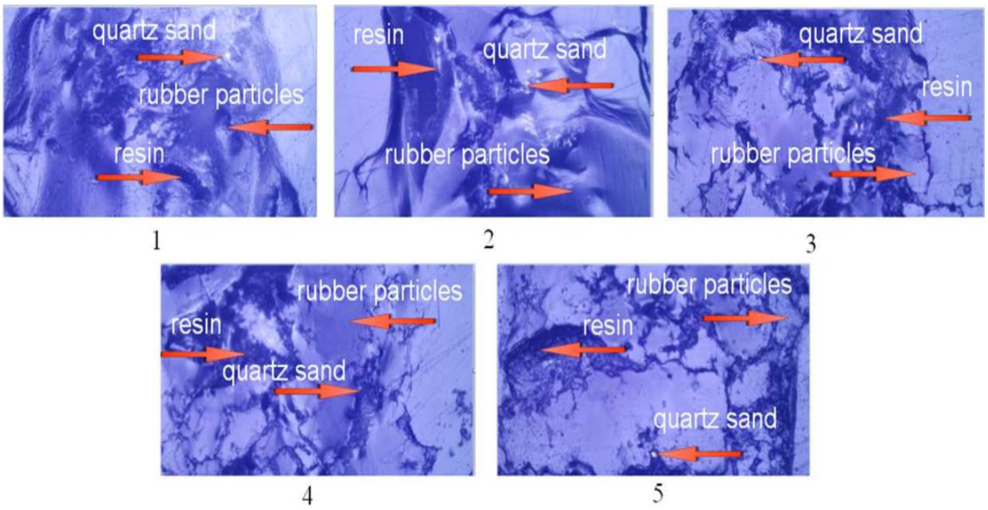

Fig. 13. Photographs of microstructural analysis in the failure zone for composite specimens 1–5 with composition according to Table 1.

samples, the same magnification is utilized. A 5-megapixel digital camera is adapted to the microscope for image acquisition. The microstructure is photographed with this digital camera to determine the structure of the composite (Fig. 13).

Microstructural analysis shows that the components of the composite are firmly connected to each other. The particles are evenly distributed in the volume of the tiles of all 5 types.

Figure 14 presents diagram for changing the mechanical properties of the composite (test bodies 1–5), depending on the content of quartz and rubber particles, where $\sigma$ is the mechanical strength (MPa) and $\varepsilon$ is the relative deformation (%). It is clear that changing the percentage ratios of the components of of the composite has a significant impact on their mechanical strength and their plasticity. An increase in the amount of quartz particles correlates with an increase in mechanical strength and a decrease in plasticity. Increasing the amount of rubber particles leads to an increase in the plasticity of the composite and a decrease in mechanical strength.

With the same ratio of both components, a compromise solution appears for plasticity and mechanical strength of the test body 3.

The higher percentage amounts of quartz particles cause an increase in mechanical strength, but the worse sound insulation ($AE$ – acoustic emission increases). As the amount of rubber particles increases and the percentage of quartz particles decreases, the strength (strength qualities) of the tiles decreases. However, the sound insulation properties of the composite are improved. The medium becomes more ho-

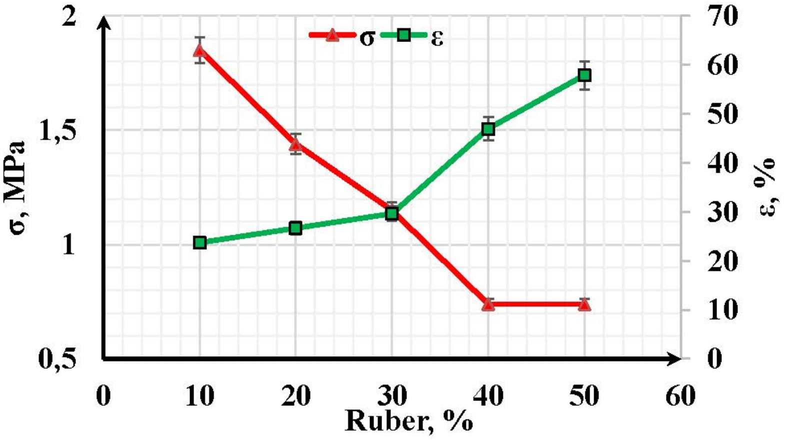

Fig. 14. Diagram for changing the mechanical properties of the composite, depending on the content of quartz and rubber particles, $\sigma$ (mechanical strength), MPa and $\varepsilon$ % – (relative deformation %) for test bodies 1–5.

mogeneous and is characterized by constant qualities throughout the volume. The characteristics change smoothly as the load increases. This is due to the rubber particles.

## 5 CONCLUSIONS

This paper centers on composite materials comprising 40% resin alongside varying compositions of quartz sand and rubber particles sourced from EoLT. Five types of experimental tiles were fabricated, and comprehensive mechanical measurements were conducted to assess their properties.

Overall, the results of all experimental measurements on these five types of composite tiles demonstrate promising characteristics, indicating their potential for both reducing EoLT waste and successful implementation, particularly in industrial or construction applications.

Especially good these composite materials could be – for floor tiles (sample 3) and for acoustic isolations (samples 4 and 5).

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