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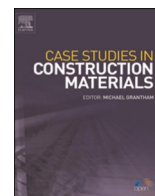
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Short communication

## Effect of rubber size and shape on Proctor elements of CBC mixtures

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### ABSTRACT

The application of recycled rubber in cement bound base courses (CBC) has recently become a subject of research. There are several advantages of the application of rubber in CBC. One is the environmental benefit considering the major problem of disposing of end-of-life tires (ELT), whose number is rapidly increasing in the modern world. On the other hand, CBC is known as the most rigid course of pavement, and is very prone to cracking. Rubber has the potential to reduce the cracking, which is another benefit of its use in those courses. This paper presents laboratory research on a reference mixture and five rubberized mixtures with different fractions of recycled rubber. The mixtures are modified with 20 % of rubber as a replacement for sand. Three fractions of granulated rubber and two fractions of rubber threads are used. The research encompasses the impact of rubber of different sizes and shapes on the Proctor elements of the CBC mixture. Furthermore, specific surface area (SSA) and SEM analysis is presented. The SSA value was found to be the most influential factor on the optimal moisture content (OMC) values, which is confirmed by SEM images. SEM analysis showed that larger particles have a flatter surface and demand higher water content. Furthermore, the rubber particle shape has a greater impact on the mixture MDD compared to the particle size, while the rubber particle size has a greater effect on the OMC compared to the rubber particle shape. Finally, the effect of the rubber particle size and shape on the OMC and MDD can be neglected and the production of cement bound aggregate incorporating waste rubber can be designed and built using reference values of the OMC and MDD.

### 1. Introduction

Nowadays, recycled rubber is increasingly considered as a building material and less as a waste. According to Directive 2008/98/EC of the European Parliament, waste is not considered as waste when a market or demand exists for it [1]. To achieve this status, usually waste material has to go through a certain processing process. End-of-life tires (ELT) are mainly subjected to mechanical processing such as grinding and shredding. Those processes can be conducted at ambient temperature, which is called ambient grinding, or at reduced temperatures, which is called cryogenic grinding. Ambient grinding can be dry or wet. Cryogenic grinding is carried out after cooling the rubber in liquid nitrogen ( $N_2$ ) until the glassy state is reached in which the rubber crumbles very easily [2, 3]. The shape and size of rubber particles depend on the production process, and in turn affect its potential use as a building material.

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Due to its elasticity, rubber differs greatly from conventional construction materials such as quality stone material, gravel or sand. For this reason, rubber is used in building construction, mainly in smaller quantities. To utilize a significant amount of waste and recycled rubber, it is necessary to use it in large construction projects. Due to the large amounts of materials used for pavement construction, roads are suitable for using recycled rubber, especially in the bearing layers. In cement bound base courses (CBC), waste rubber particles are used as a partial replacement for fine aggregate fractions, usually in 1–6 % of the total volume of the aggregate [4,5], due to their lower density compared to natural aggregate (particularly sand). The result of fine fraction replacement by waste rubber is a reduction in compressive and tensile strength [5–8], but the benefits of rubber incorporation can be found in the elevated frost resistance of the rubberized mixtures [9]. As a material that can absorb higher energy compared to natural aggregate, rubber can take internal tensile stresses resulting from shrinkage of the CBC mixture. Also, its better elastic properties can accommodate tensile stresses from cyclic loads (traffic) at the bottom of the bearing layers. The production of less stiff material by the incorporation of recycled rubber has been proven in papers [7,10], which is evident in the lower dynamic modulus of elasticity values as a measure of material stiffness.

When using nonconventional materials as a replacement for materials usually used in construction, one has to be careful. Road material characterization starts by defining its compaction characteristics, i.e., the Proctor elements: maximum dry density (MDD) and optimal moisture content (OMC), defined by standard [11]. For the influence of rubber on the MDD, there is a consensus among different researchers that the addition of rubber results in the MDD decreasing due to its low specific gravity. However, there are two main views on the influence of rubber incorporation on the OMC. Rubber is mainly used as a substitute for fine-grained material that is prone to water absorption. So, the decrease in the OMC with rubber incorporation is highlighted due to its hydrophobic nature and lower water absorption capacity. Secondly, contrary to previous theory, the increase in the OMC due to rubber's rough surface and large specific surface area is highlighted. This view is explained by a greater need for water to lubricate the entire matrix of the mixture. As stated in papers [12,13], rubber has a rough surface that entraps water molecules, while sand or similar fine-grained materials have a water absorption ability. These water molecules remain unreacted with cement. Furthermore, in rubberized CBC mixture production, only physical and mechanical interactions between the rubber and cement particles can be achieved. Chemical bonds do not appear [14]. In the literature review presented by Wen et al. [15], it was concluded that the implementation of rubber particles in the matrix does not form new hydration products.

As previously mentioned, some researchers claim that increasing the amount of rubber causes an increase in the OMC value. Nakhaei et al. [16] added recycled rubber fractions up to 10 mm to granular soil in amounts of 8 %, 10 % and 14 %. The increase in rubber content was followed by an increase in the OMC. For instance, the OMC of the mixture with 10 % of rubber was higher by about 2 % than the reference mixture OMC. Furthermore, several research works concluded an opposite trend when defining the Proctor elements. The decrease in the MDD and OMC with an increase in the rubber content in clayey soils is pointed out in [17–21]. In [17] rubber particles up to 2 mm were used and the OMC decreased by about 1 % compared to the reference mixture. Rubber sizing between 5 and 10 mm was used in [18], where an increase in rubber content of 4 % brought a decrease of the OMC by about 1 %. Larger rubber particles, rubber fibers up to 15 mm, were used in [19], where an increase of rubber content by 10 % was followed by a decrease of the OMC by about 2 %. A larger amount of rubber, up to 50 %, was used in [20], where a 50 % increase of rubber ground tire particles caused a decrease in the OMC value of about 3.5 %. Within the same research, the amount of rubber was increased in increments of 10 % and the reduction of the OMC was not significant up to a rubber proportion of 30 %. The reasons for this phenomenon are stated as the difference in water adsorption between the materials [17,19–21] and the large specific surface area (SSA) of the rubber particles [18]. However, there is research that claims that an increase in the rubber fibers content in stabilized clayey soil does not affect the OMC [22] because of rubber's hydrophobicity.

Some researchers have investigated the effect of rubber on CBC properties, but they used a constant amount of water, regardless of the rubber content of the mixture. Sun et al. [5] replaced the fine fraction of limestone with 40# rubber in three proportions, whereby they added water in the amount of 4.5 % of the aggregate mass. Farhan et al. [4] also replaced limestone by rubber particles up to 6 mm, while retaining the water amount as 4.6 % of the total mixture mass. Furthermore, Pham et al. [9] replaced sand with rubber of the same gradation while retaining the same amount of water as for the reference mixture. Loganathan and Mohammed [23] varied three rubber proportions as a replacement for river sand, but kept the water–cement ratio of 0.15. Petrella and Notarnicola [24] replaced sand with two rubber fractions in three different mass proportions while retaining the same water–cement ratio. However, a decrease of the OMC with increase of the rubber content in CBC is presented in [13], and the decrease was lower than 0.5 % for 60 % replacement of the fine aggregate fraction by granulated rubber of a similar grain size distribution. Kowalska [25] studied the compactness of four different fractions of scrap tire rubber in a standard Proctor test. The fractions were 0.1–1 mm, 0.5–2 mm, 2–5 mm and 10–40 mm, and for comparison river quartz sand was used as a reference material. The author concluded that the bulk density of all specimens increased with the increase in the water content. The MDD increased slightly as the size of the grains decreased and the maximum amount of water retained in the specimens decreased with the increase in rubber particle sizes.

From the presented literature review it is evident that a large array of different rubber shapes and fractions can be found on the market. Waste rubber threads and granules are used as a volume replacement for the fine aggregate fraction depending on the grain size distribution. Generally, rubber of all sizes and shapes in clayey soils increases the OMC values, while in granulated material researchers tend to retain the moisture value as for the reference mixture. Also, there is research that claims both an decrease and an increase in the OMC with increase of the rubber content in the mixture.

Within this research, laboratory tests are conducted comparing the Proctor elements of the reference CBC mixture with natural aggregate and five rubberized mixtures with different sizes and shapes of rubber particles. The aim of this paper is to determine the influence of the rubber particle size and shape on the Proctor elements. The novelty of the research presented here lies in the holistic approach to the influence of the waste rubber particle characteristics on the CBC mix design, since most of the previous research

presents general conclusions on their influence on the Proctor elements based on only one waste rubber fraction.

## 2. Materials and methods

The cement bound mixtures for this research were composed of three different natural river gravel fractions from Sava river (0–4 mm, 4–8 mm, 8–16 mm), sand from Drava river (0–2 mm) and rubber derived from end-of-life tires (ELT) in five different fractions. Two different types of rubber fractions were used, granulated rubber and rubber threads. The particle sizes of the granulated rubber were 0–0.5 mm, 0.5–2 mm and 2–3.5 mm, while the rubber threads particles were 0–0.8 mm and 0.8–3 mm in size (Fig. 1).

The named rubber grain sizes represent the designations of rubber used available on the local market. The defined grain size distributions of the materials used are presented in Fig. 2. The densities of the natural aggregates used (0–4 mm, 4–8 mm, 8–16 mm and sand) are 2.96, 2.63, 2.70 and 2.86 g/cm<sup>3</sup>, respectively, determined by EN 1097–6:2013 [26]. The reference mixture is composed of equal proportions of each natural aggregate fraction (25 % each). The mixture gradation is designed in such a way as to meet the upper and lower limits for hydraulically bound mixtures according to EN 14227–1 [27]. The rubberized mixtures are composed by replacing 20 % of sand volume by rubber. The rubber content is selected according to previous preliminary research results indicating that higher rubber content significantly influences the compressive strength reduction [13,28]. Volume replacement is chosen due to the low density of rubber, i.e., 1.123 g/cm<sup>3</sup>, determined by EN 1097–6:2013 [26]. As a binder, Portland cement of grade 32.5 (CEM II B/M (P-S) 32.5 R) was used, comprising 5 % of the total mass of the aggregate for all tested mixtures. According to the procedure described in standard EN 196–6 [29], the binder density is 2.92 g/cm<sup>3</sup>. The optimal binder content is determined based on previously conducted research [13]. Due to the same density of all the rubber fractions, the volume and mass proportion of the constituents in the rubberized mixtures are the same.

The grain size distributions of the sand and rubber fractions are presented in Fig. 2. From the plotted graph it can be concluded that the smallest rubber fractions (0–0.5 granulated rubber and 0–0.8 rubber thread) have a gradation similar to sand. The major similarity can be observed between sand and granulated rubber at 0–0.5 mm. Also, the 0.5–2 and 0.8–3 rubber fractions are very similar (yellow and green lines) to each other. All rubber fractions were shown to be coarser than sand particles. What can also be concluded from the plotted graph is that the market names of recycled rubber do not fully reflect the real grain size distribution of rubber packing; however, for simplification those names will be retained within the paper.

Despite major differences in the grain size distribution of the fine fractions used (Fig. 2), in the grain size distribution of the final mixtures those differences have a minor impact (Fig. 3). This is due to the small mass proportion of rubber in the rubberized mixtures. The grain size distributions of the reference and rubberized mixtures are presented in Fig. 3. Considering the replacement of the fine fractions, differences in the grain size distribution can be found up to the grain size of 4 mm. From Fig. 3, it can be concluded that, for the rubberized mixtures, the position of the grain size distribution curve was similar to the reference mixture.

For OMC and MDD determination, the Proctor test procedure was used according to EN 13286–2 [30]. The Proctor procedure considers the compaction of five test specimens with different amounts of water varied by 1 % or 2 %. Due to the mixtures compositions and following the recommendations of standard EN 13286–2 [30], the modified Proctor energy of 2.7 MJ/m<sup>3</sup> and Proctor mold B with 150 mm diameter and 120 mm height were used. This procedure is described in more detail in [13]. Furthermore, the specific surface area (SSA) of the fine solid particles, sand and rubber particles was determined in the Brunauer, Emmett and Teller (BET) machine according to the ISO 9277:2010 standard [31]. This procedure considers the determination of the amount of adsorbate or absorptive gas (in this case, liquid nitrogen), to cover the external and accessible internal pore surfaces of solid particles. More precisely, the result of this testing is the specific surface area (SSA) of the particle with all its irregularities and roughness. The test was conducted on four specimens prepared according to the ISO 8213 standard [32] for each aggregate fraction. The first step is degassing the specimen, where the specimen is cleaned of all impurities and moisture. Despite the precise quantitative evaluation of particle SSA by the BET procedure, there is still a lack of assessment of the particle surface flatness. Bearing in mind the sand replacement by rubber, an important step is to compare the surface area of sand particles with the surface area of the rubber particles, as well as to compare the surface areas of particles of different rubber fractions. This kind of analysis was conducted by Scanning Electron Microscopy (SEM) at a

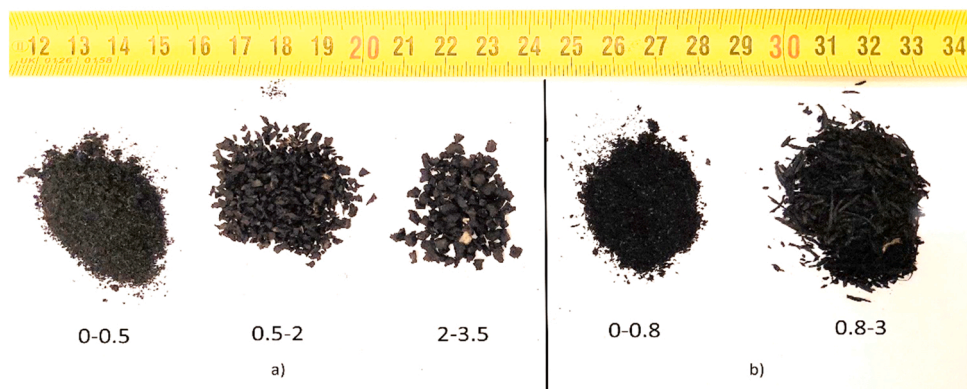


Fig. 1. Rubber fractions used in laboratory research: a) rubber granules and b) rubber threads.

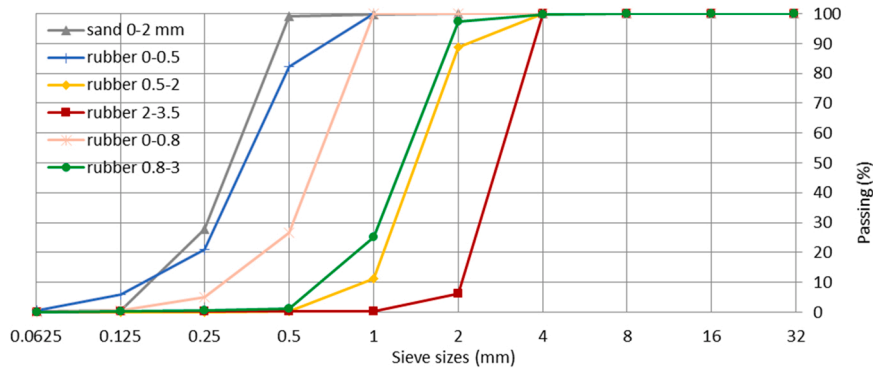


Fig. 2. Grain size distribution of sand and different rubber fractions.

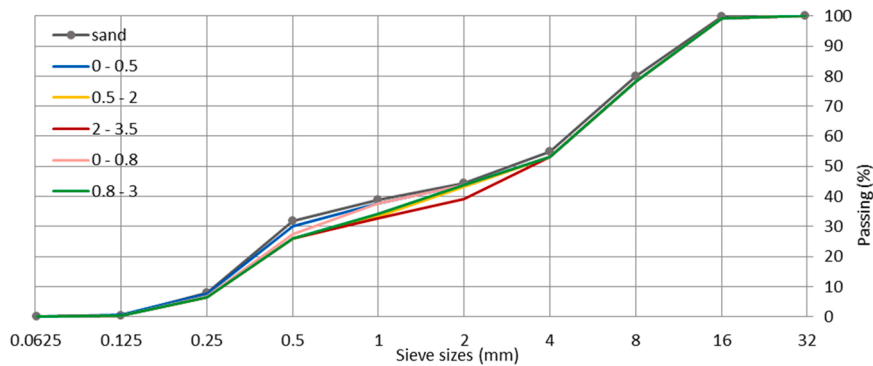


Fig. 3. Grain size distribution curves of reference and rubberized mixtures.

1000 times magnification. Preparation of specimens for SEM analysis involves drying the samples and then covering them with a conductive material (in this case, gold).

### 3. Results and discussion

The results of the Proctor compaction elements, OMC and MDD are presented in Table 1. Mixture “Sand” represent reference mixture comprised of natural aggregate only while other mixtures represent rubberized mixtures with various rubber fraction replacing 20 % of sand volume. Rubberized mixtures are named according to the rubber fraction they contain.

It is evident that the MDD values differ very little between mixtures, while greater differences are observed in the OMC values. The Proctor compaction curves for all the tested mixtures are presented in Fig. 4. According to the Proctor compaction curves, mixtures with 0.5–2 and 0.8–3 rubber granules have similarly shaped curves. The curves for these mixtures have a milder slope, representing material that is more suitable for on-site compaction, since a greater change in humidity will not significantly affect the density of the material. The grain size distributions of these fractions are also similar, as pointed out earlier in the text (Fig. 2). It is important to point out the similarity of the 0.5–2 and 0.8–3 Proctor curves and that for the sand.

Fig. 5 presents the variation in MDD and OMC depending on the rubber size and shape: Fig. 5.a. for granulated rubber and 5.b. for rubber threads. Regardless of the rubber particle shape and size, a decrease in the MDD is recorded compared to the sand mixture, which is in line with previous research presented in the Introduction. This MDD drop is from 0.94 % to 1.83 % for granulated rubber and 2.75 % for rubber threads. A higher drop in the MDD can be observed for rubber threads, but there is no general trend in MDD variation depending on the rubber particle size. A decrease in the MDD can be partially due to the lower density of the rubber used compared to sand, which resulted in a mix density reduction of 2.89 %. Namely, the calculated densities of the reference and rubberized mixtures are 2.79 g/cm<sup>3</sup> and 2.71 g/cm<sup>3</sup>, respectively. However, the MDD drop is less than 2.89 %, indicating the rubber particles’ elasticity

Table 1  
Results of OMC and MDD.

Mixture	Sand	0–0.5	0.5–2	2–3.5	0–0.8	0.8–3
OMC (%)	6.70	5.79	6.81	6.66	5.46	6.28
MDD (%)	2.18	2.14	2.14	2.16	2.12	2.12

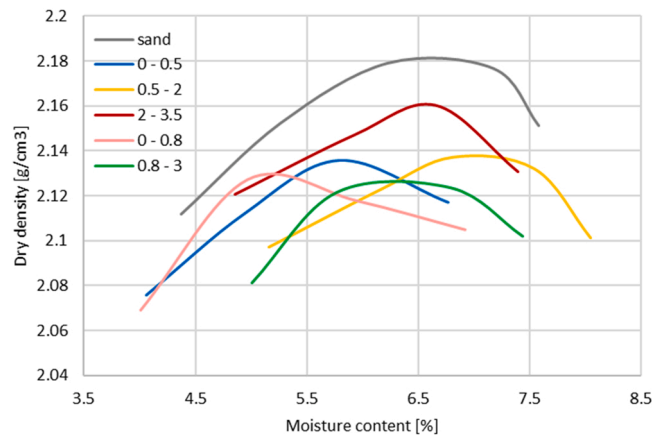


Fig. 4. Proctor compaction curves of tested CBC mixtures.

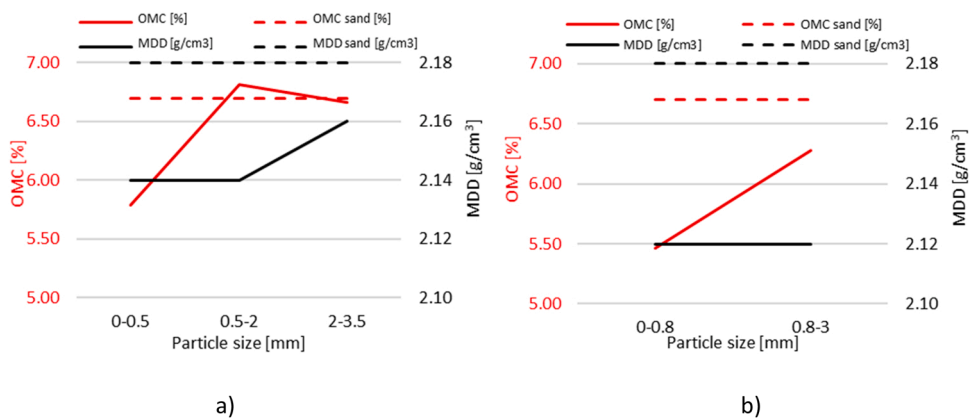


Fig. 5. OMC and MDD values for mixtures with: a) granulated rubber; b) rubber threads.

and deformability having a positive effect on compaction, since the lower rubber density was “lost”, compensated during compaction.

From the results presented in Fig. 5, a general trend of OMC decrease compared to the sand mixture can be observed. The drop in the OMC compared to the sand mixture is between 0.6 % and 13.6 % for granulated rubber and 6.3 % and 18.5 % for rubber threads. However, an increase in the OMC with increase of the rubber particle size is also recorded, regardless of the rubber particle shape. There is an indication of a higher rubber particle size effect on the OMC compared to the rubber particle shape. However, comparing the sand and rubber mixtures, the difference in the OMC is from 0.04 % to 1.24 %, which are rather negligible values.

As stated in [12,13], rubber has an irregular surface which entraps the water molecules, thus leaving a certain amount of water unreacted. Furthermore, the hydrophobicity of rubber causes a migration of hydraulic phases from the rubber, thus producing lower bonds between the rubber and other particles in the matrix [14]. Considering the different processes used in the production of granulated and rubber threads, the key to OMC development lies in the surface area of the rubber particles. The same indication occurred when analyzing the MDD, where the flat and elongated shapes of the rubber particles occupy more space within the matrix during compaction, which is evident from Fig. 6. This is emphasized following the SSA and SEM analyses. The SSA of the sand and rubber particles is presented in Table 2.

All the tested rubber particles have a higher SSA comparing to sand and there is a decrease in the SSA with increases of the rubber particle size. This indicates a large difference in the rubber particle surface shapes and appearance. Also, the SSA results are consistent with grain size distribution similarities between the 0–0.5–0–0.8 and 0.5–2–0.8–3 fraction rubber particles. The rubber is produced by grinding and shredding, which leads to an irregular surface due to the rupture of the bonds in the rubber structures. Small rubber particles, particularly the 0–0.5 and 0–0.8 fractions, act as a filler occupying smaller pores, resulting in less absorption capacity and an OMC drop compared to the sand mixture, which is also pointed out in [33].

Furthermore, the sand and all the rubber particles were observed on a Scanning Electron Microscope (SEM) magnified 1000 times. Those laboratory procedures were conducted in order to more closely analyze the influence of the rubber’s surface shape and area on the OMC values.

In Fig. 7, significant differences in the particles’ surface shapes can be observed. Sand has the flattest surface of all the observed particles. Fine rubber fractions (0–0.5 and 0–0.8) have a rougher surface, while coarse fractions (0.5–2, 2–3.5 and 0.8–3) have a

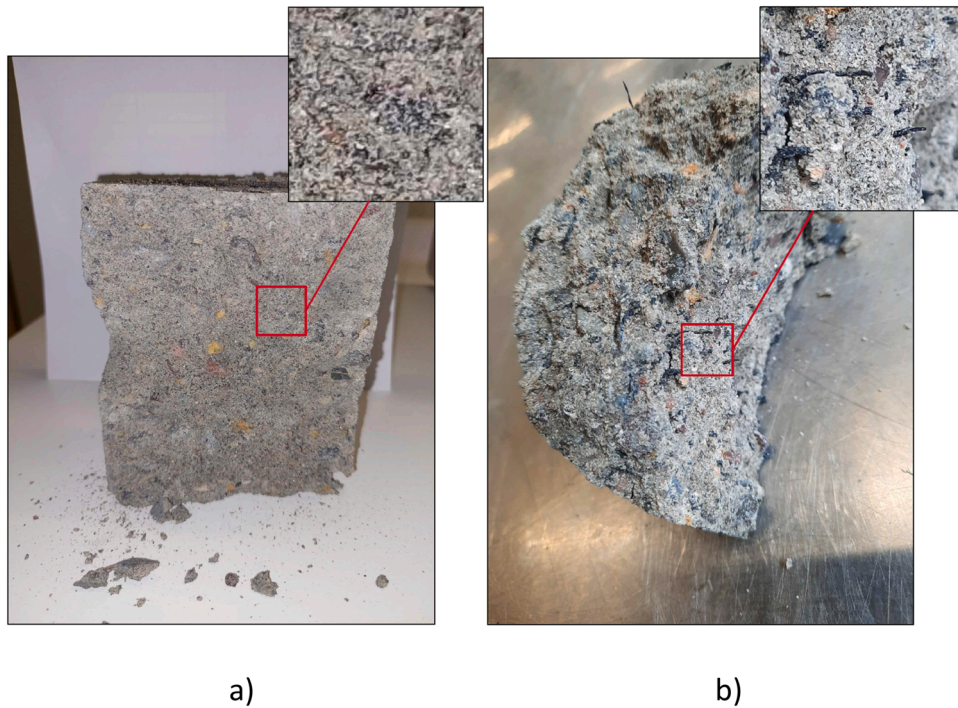


Fig. 6. Rubber granulated (a) and threads (b) particles within cement bound aggregate mixture.

**Table 2**  
SSA measurement results.

Fraction	SSA (m <sup>2</sup> /g)	SD (m <sup>2</sup> /g)	CoV ( %)
sand	3.14	0.21	6.56
0–0.5	12.79	0.46	3.61
0.5–2	7.77	0.44	5.63
2–3.5	6.43	0.58	9.03
0–0.8	10.06	0.05	0.46
0.8–3	7.88	0.70	8.87

noticeably flatter surface. The surface shapes from the SEM analysis follow the SSA results presented in Table 2. More precisely, those fractions characterized as flatter have lower values of SSA. An analogous rule applies to the fractions with rough surfaces. Compared to the OMC values, it is shown that flatter particles, both sand and rubber, require larger amounts of water. Furthermore, more irregular rubber particles obtained lower OMC values. As generally observed, rubberized mixtures need lower amounts of water than the reference mixture, which could be attributed to sand's water absorption capability, which is not the case with rubber particles.

Most interesting are the results of the 2–3.5 mm fraction. Its grain size distribution deviates the most from the reference mixture. But, the OMC and MDD values obtained are the closest to the values of the reference mixtures, among all the other tested mixtures. The particle surface can be classified as a flat one, based on Fig. 6. Furthermore, the SSA of this fraction is the lowest of all the rubber fractions and thus the closest to the sand's SSA. Based on these results, the SSA of the nonabsorptive alternative material was found to be the most influential factor on the Proctor elements of the mixture. Overall, the differences in OMC values range up to 1.5 %, which is actually a very small percentage. Considering the fact that the permitted deviation from the material's OMC during construction on the construction site is up to  $\pm 2$  % of the optimal value, it can be concluded that the shape and size of the rubber particle have no effect on the OMC and that the OMC of the natural aggregate mixture can be adopted for the rubberized mixtures. This is significant from the perspective of the potential commercialization and implementation of rubber into CBC construction, since no additional tests need to be conducted when defining the optimal mix composition for implementing waste rubber.

#### 4. Conclusions

The aim of this research is to determine the impact of the rubber's size and shape on the Proctor elements of rubberized cement bound mixtures. Besides the Proctor elements, BET and SEM analysis was conducted in order to investigate the surface of the rubber particles in more detail. The following conclusions are drawn:

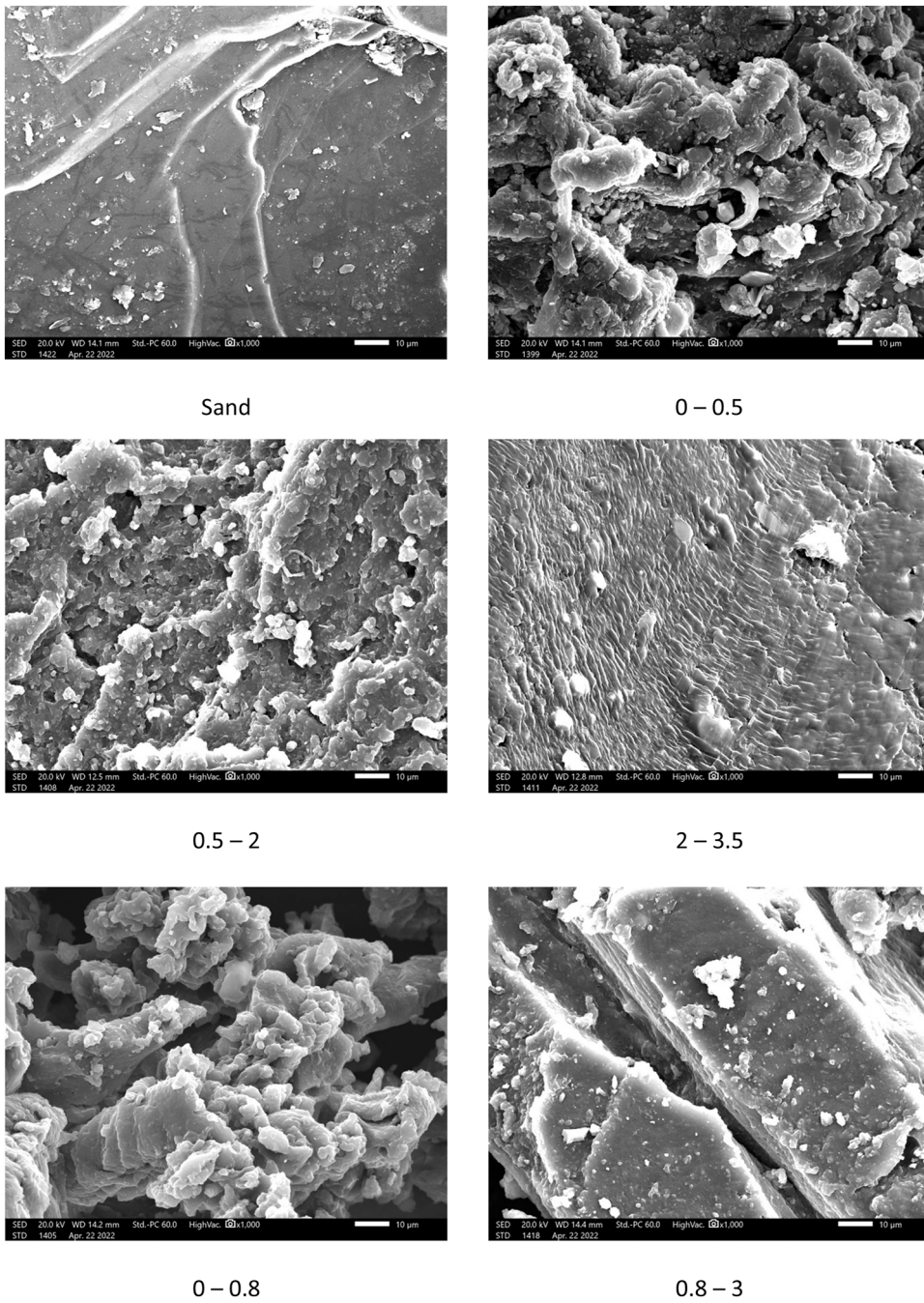


Fig. 7. Sand and rubber particle surfaces obtained by SEM (x1000).

- Rubberized mixtures obtained lower MDD values compared to the reference mixture (0.94–1.83 % for granulated rubber and 2.75 % for rubber threads). The rubber particle shape has a higher impact on the MDD compared to the particle size. Also, rubber particles' deformability has a positive effect on compaction, since the lower rubber density was "lost", compensated during the compaction process of the mix.
- In rubberized mixtures the OMC decreases compared to the sand mixture and increases with increase of the rubber particle size (between 0.6 % and 13.6 % for granulated rubber and 6.3 % and 18.5 % for rubber threads). The rubber particle size has a greater effect on the OMC compared to the rubber particle shape.
- Generally, flatter particles require higher water content, while coarser particles require lower water content.



- The effect of the rubber particle size and shape on the OMC and MDD values can be neglected (difference in the OMC is from 0.04 % to 1.24 % and in the MDD is from 0,94 % to 2,75 %) and the production of cement bound aggregate incorporating waste rubber can be designed and built using reference values of OMC and MDD for small rubber content (up to 20 %). For higher rubber content, additional research is to be conducted, but it should be noted that a higher rubber content will significantly reduce the CBC strength characteristics.

Here presented results and conclusions are drawn from the conducted research and apply to here used materials with its characteristics and geometry as defined within the previous sections. To set some general conclusions, more tests are to be done on different shape and size rubber samples as on different cement, rubber and sand ratios.

### Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Ivana Barisic reports financial support was provided by Croatian Science Foundation.

### Data Availability

No data was used for the research described in the article.

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