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# Effect of curing methods and scrap tyre addition on properties of mortars

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Research Paper

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## Effect of curing methods and scrap tyre addition on properties of mortars

Mechanical, thermal conductivity, and porosity properties of scrap tyre incorporated mortars are examined experimentally in this study for various curing conditions. Cement mortars were prepared by adding 0%, 1%, 5%, 10% and 18% of scrap tyre by cement weight, and the mortars were subjected to three curing procedures for 14 weeks. The lowest thermal conductivity values were obtained on samples containing 18% of scrap tyre by water curing and wetting-drying curing as 1.09 and 1.14 W/mK, respectively. The maximum compressive strength of 61.5 MPa was obtained on a pure sample cured by wetting and drying, while the flexural strength of pure and a 1% additive incorporated samples, which were also cured by wetting-drying, amounted to 9.1 and 8.2 MPa, respectively.

### Key words:

mortar, scrap tyre, curing procedure, thermal conductivity, compressive strength

Prethodno priopćenje

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## Utjecaj metoda njege i udjela otpadne gume na svojstva morta

U radu se eksperimentalno istražuju poroznost te mehanička i toplinska svojstva morta s dodatkom otpadne gume u različitim uvjetima njege. Mješavinama morta dodana je otpadna guma u udjelu od 0 %, 1 %, 5 %, 10 % i 18 % mase cementa, nakon čega su provedena tri postupka njege u razdoblju od 14 tjedana. Najniže vrijednosti toplinske provodljivosti od 1,09 tj. 1,14 W/mK dobivene su kod udjela otpadne gume od 18 % na uzorcima koji su njegovani vodom te vlaženjem i sušenjem. Najviša vrijednost tlačne čvrstoće od 61,5 MPa dobivena je na uzorku bez dodatka otpadne gume koji je njegovan vlaženjem i sušenjem, dok je čvrstoća na savijanje uzoraka bez gume i s 1 % otpadne gume iznosila 9,1 odnosno 8,2 MPa.

### Ključne riječi:

mort, otpadna guma, postupak njege, toplinska provodljivost, tlačna čvrstoća

Vorherige Mitteilung

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## Einfluss der Pflegemethode und des Gummiabfallgehalts auf die Eigenschaften von Mörtel

In der Abhandlung werden experimentell die Porosität sowie die mechanischen und thermischen Eigenschaften von Mörtel unter Zusatz von Gummiabfällen unter verschiedenen Pflegebedingungen untersucht. Den Mörtelmischungen wurden Gummiabfälle in einem Anteil von 0%, 1%, 5%, 10% und 18% Zementmasse hinzugefügt, wonach drei Pflegeverfahren in einem Zeitraum von 14 Wochen durchgeführt wurden. Die niedrigsten Wärmeleitfähigkeitswerte von 1,09, d.h. 1,14 W/mK wurden bei Gummiabfallgehalten von 18% bei Proben erreicht, die mit Wasser sowie durch Nässen und Trocknen gepflegt wurden. Den höchsten Druckfestigkeitswert von 61,5 MPa erhielt man mit der Probe ohne Zusatz von Gummiabfall, die mit Nässen und Trocknen gepflegt wurde, während die Biegefestigkeit der Proben ohne Gummi und mit 1% Gummiabfall 9,1 beziehungsweise 8,2 MPa betrug.

### Schlüsselwörter:

Mörtel, Gummiabfall, Pflegeverfahren, Wärmeleitfähigkeit, Druckfestigkeit

## 1. Introduction

Rapid population growth in developing countries has boosted the demand for transportation vehicles and, as a result, the automobile industry has been growing quite rapidly. Due to this continued growth of automobile industry, it is estimated that 1.4 billion tires are produced annually all over the world [1, 2]. Tires, the main components of which are the natural and artificial synthetics, carbon black and additional additives such as sulphur, zinc oxide, stearic acid, have very high resistance to abrasion and corrosion. Therefore, degradation of tires on their own is very difficult and unlikely [3-6].

In developed countries, a series of regulations are currently in place to provide for the recycling of tires whose lifecycle is over. The recycling process is important in terms of an efficient use of scarce energy resources, and it contributes to the reduction of environmental pollution and nature protection, as raw materials of rubber are oil-based [7]. At the present time, scrap tires could be technically employed as fuel in industries that need high-energy consumption, however, they are not attractive compared to equivalent alternatives due to economic issues [8]. Automobile tire, which are very difficult to degrade by themselves in the environment, currently present a serious threat to nature. Therefore, recycling and reutilization of the tires at the end of their lifecycles has become even more important in terms of economy and nature [9].

In concrete technology, the recycling of scrap tires has become, in recent years, a very crucial and scientifically attractive idea. In most previous studies, it was established that the compressive strengths of concrete decreases when scrap tire is added as partial replacement of aggregate and, in that respect, various analyses were made to determine the size and distribution of tire particles that would minimize the loss of strength. Concrete containing scrap tire is currently used in structural elements for which the risk of earthquake and accompanying effects does not exist [10].

The effect of scrap tire addition on the mechanical strength of mortar has been investigated experimentally in a number of studies. Marques et al. used treated and untreated tire rubber in the proportion of 12 % by volume, and they observed 40 % and 47 % reduction in compressive strength, respectively, compared to the control sample [11]. In their experimental work on high strength mortar, Angelin et al. found that mortar specimens containing 30 % of tire rubber by weight exhibited 12 times loss in compressive strength and 4 times loss in bending strength compared to control sample [12]. Oikonomou and Mavridou used up to 15 % of the worn automobile tires additive in mortar mixtures and found improvement in mortar chlorine permeability, although reduction in compressive strength proved inevitable for all tire additive contents [13]. Boukour and Benmalek attempted to reduce the compression and bending losses caused by scrap tire addition with brick waste filler, which improved durability properties of mortars including scrap tire [14].

Mass transfer between aggregate (2.45-5.20 W/mK) with a high thermal conductivity coefficient and scrap tire with a low coefficient of thermal conductivity (0.25-0.50 W/mK) reduces the thermal conductivity coefficient of mortar containing scrap tire, and the conductivity drop is related not only to the conductivity of displaced materials but also to pore structure, moisture, and degree of crystallization [15].

Despite the decrease in mechanical strength of mortar with an increase in scrap tire content, the positive effect of the scrap tire addition on the durability and thermal conductivity coefficient is remarkable. When 10 % and 30 % of scrap tire is added to mortar mixtures, the mechanical strength decreases, but the thermal conductivity values are reduced by as much as 12 % and 20 %, respectively [16]. Using 40 % crumb rubber addition in their experimental study, Fadiel et al. found a 28 % decrease in thermal conductivity coefficient compared to traditional mortar [17].

Although the scrap tyre addition is so useful in terms of significant reduction in the thermal conductivity coefficient of mortars, the simultaneous decrease in both compressive and flexural strengths limits the current use of scrap tyre additive. One of alternative solutions for compensating the decrease in mechanical strength values caused by the scrap tyre additive is the application of appropriate curing methods. Recent technological developments in cement technology have enabled introduction of a number of new curing methods in the production of structural elements. Although there are many newly-developed curing methods, the reliability, the efficiency and the effect of structural curing on the mechanical, thermal, durability and microstructure properties of structural elements are still being debated. Individual curing methods act in a variety of ways, but the main point in all curing methods is to maintain relative humidity in the calcium-silicate structure and to ensure the continuity of the hydration. Low relative humidity in the capillary structure is an obstacle to the continuity of chemical reactions due to insufficient water content. It results in ending the hydration process earlier than the desired time and the mechanical, thermal, durability and microstructural properties of structural elements are thus adversely affected [18].

Several curing methods are applied in the production of calcium-silicate based structural elements, e.g. traditional curing in water, steam curing, curing by heat, and membrane curing. Traditional water curing method is considered to be the reference curing method that can be used to demonstrate the effectiveness and efficiency of other alternative curing methods. Samples produced in the water curing process are usually kept in the mould for 1 day at the beginning of the process and the next day the samples taken from the mould are kept in a sealed tank so that they are completely submerged in water until mechanical testing. Water curing method is very important as it is one of the curing methods representing curing conditions in the field in the production of structural elements [19, 20]. In their experimental work, Nasr et al. investigated the efficiency of ambient and hydrothermal curing methods, considering

water curing conditions as a reference, for mortar samples which were produced under various curing conditions. Kong et al. investigated the effectiveness of alternative steam curing and microwave pre-curing methods, and they also used water curing as a reference [21]. Moreover, the results in our previous study demonstrated that enhanced compressive and flexural strengths were detected in mortar samples that cured under the effect of wetting – drying in comparison to conventional water curing regime [22].

In this study, it is discussed whether the curing methods, i.e. the wetting - drying curing method and the  $MgSO_4$  method, can be used as an alternative to water curing method, considering the water curing method as reference. The mechanical strength and thermal insulation properties of mortar samples are investigated in detail based on the change of pore structure and porosimetric parameters under alternative curing conditions.

## 2. Materials and methods

### 2.1. Materials

The scrap tyre used in the mortar produced for the experimental works has been obtained from Isfalt A.S., and the particle size varied from 0.18 to 0.425 mm. The particle distribution and other properties of the scrap tyre used in experimental studies are given in Figure 1 and Table 1, respectively. Other components used for mortar production are CEM I 42.5 R type Portland cement produced by Limak Cement Co. and standard CEN sand. Physical and chemical properties of CEM I 42.5 R type cement are presented in Table 2.

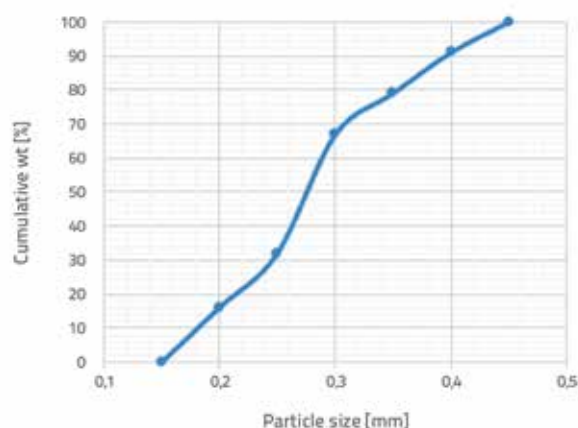


Figure 1. Particle distribution of used scrap tyre

Table 1. Physical and chemical properties of used scrap tyre

Specific gravity [ $kg/m^3$ ]	1140
Particle size [mm]	0.18 - 0.45
Total polymer content [%]	43
Carbon black [%]	38

Table 2. Chemical and physical properties of CEM I 42.5 [22]

Chemical composition [wt/ %wt]		
SiO <sub>2</sub> (solute)		18.99
Al <sub>2</sub> O <sub>3</sub>		4.77
Fe <sub>2</sub> O <sub>3</sub>		3.15
CaO		63.69
MgO		1.12
SO <sub>3</sub>		2.96
K <sub>2</sub> O		0.64
Na <sub>2</sub> O		0.17
Cl -		0.0099
Loss on ignition		3.70
Insoluble residue		0.38
Total alkali		0.59
C <sub>3</sub> A		7.32
Physical properties		
Setting time [min]	Initial	150
	Final	195
Specific gravity		3.11
Specific surface area [ $cm^2/g$ ]		3769
Volume stability [mm]		1

### 2.2. Mix proportions

In the scope of experimental studies, five different mixtures with different addition proportions were prepared in order to determine thermal and mechanical properties of scrap tyre incorporated mortars under different curing conditions. The curing of mixture samples was carried out in three different curing conditions, namely the wetting-drying,  $MgSO_4$  effect, and water-curing. Standard CEN sand conforming to TS EN 196-1 specification, and CEM I 42.5 R type Portland cement conforming to TS EN 197-1 specification were used in all mixtures. The scrap tyre was added to mortar mixtures as a partial replacement of cement in four proportions: 1.0 %, 5.0 %, 10.0 %, and 18 % by weight. Standard sand and scrap tyre to be added to mortar mixtures were stored before usage in a humid chamber where relative humidity was above 50 %. By doing this, it was ensured that sand and scrap tyre were in an appropriate saturated-surface-dry state and they were added to mixture when they were in saturated surface - dry state moisture condition. Mix proportions of scrap tyre incorporated mortars are presented in Table 3.

A dry mixing process was initially applied due to the porous structure and surface shape of the scrap tyre particles used in the experiments. In order to increase the homogeneity of the mixture, the water was added to the mixture gradually and all scrap tyre particles were completely covered with cement

Table 3. Mix proportions of scrap tyre incorporated mortars

Mix No.	Scrap tyre content [%]	Cement content [%]	Scrap tyre content [g]	Cement [g]	Water/cement	Water [ml]	Sand [g]
L0	0	100	0	450	0.50	225	1350
L1	1	99	4.5	445.5			
L2	5	95	22.5	427.5			
L3	10	90	45	405			
L4	18	82	81	369			

mortar in an attempt to increase the adhesion quality of mortars. The mixtures were set to have a constant water/binder ratio of 0.5. The total amount of cement and scrap tyre was maintained constant at 1/3 of sand by weight in the mixtures.

Once the mixing process was completed, the cement mortar was placed into standard 40 mm x 40 mm x 160 mm stainless steel moulds and an effective compacting process was carried out in order to prevent segregation of the mixture. Mixture samples were removed from the moulds after being kept in moulds for 24 hours at room temperature ( $20 \pm 3^\circ \text{C}$ ). Three samples from each of the mixtures were subjected to different curing conditions, which was the original intention of the experimental work.

### 2.3. Curing methods

In order to investigate mechanical, thermal and porosity properties of scrap tyre incorporated mortars under different curing conditions, 3 different curing methods were prepared for each mixture sample group, namely the wetting-drying method,  $\text{MgSO}_4$  method and water curing method, as recommended in the paper [22]. The water-cured sample group was kept in a lime-saturated curing tank at a temperature of  $21^\circ \text{C}$  ( $\pm 2^\circ \text{C}$ ) continuously for 14 weeks. In the case of the wetting-drying curing, one cycle involved subjecting the samples to the wetting-drying curing conditions in a lime-saturated curing tank at a temperature of  $21^\circ \text{C}$  ( $\pm 2^\circ \text{C}$ ) for 1 week, and the following week they were kept at room temperature ( $21^\circ \text{C} \pm 2^\circ \text{C}$ ). This cycle was repeated 7 times to complete the total of 14 weeks of curing. For the sample group cured under the effects of  $\text{MgSO}_4$ , a solution containing 13%  $\text{MgSO}_4$  by weight was prepared and the samples were kept in the  $\text{MgSO}_4$ -containing solution for 1 week, while the following week the same samples were dried in oven at  $105^\circ \text{C}$ . This cycle was also repeated 7 times to complete the total of 14 weeks of curing. For each curing method, 0% scrap tyre incorporated samples were considered

as reference samples for the respective curing method. In experimental studies, water of the same chemical composition was used in all curing methods.

### 2.4. Testing

After having completed the 14-week curing process for water curing, wetting-drying, and  $\text{MgSO}_4$  effects, the samples were subjected to flexural and compressive strength tests, thermal conductivity coefficient measurement, and mercury porosimetry (MIP) tests, respectively. Five different mixtures were determined in the scope of experimental studies, and nine samples were prepared for each mixture. Each mixture was divided into three different curing groups to be represented by three samples and subjected to mechanical strength tests. Unlike the mechanical strength test, the mercury porosimetry and thermal conductivity coefficient were measured on the samples with the highest compressive strengths. In the measurement of the thermal conductivity coefficient, the measurement was repeated for five different regions of the sample. Experimental test programme is in accordance with recommendation in our previous study [22] and is shown in Table 4.

## 3. Results and discussion

### 3.1. Compressive strength results

The compressive strengths of the 0%, 1%, 5%, 10%, and 18% scrap tyre incorporated mortars subjected to water curing, wetting-drying, and  $\text{MgSO}_4$  effects are shown in Figure 2. The compressive strength results are in range from 17.1 to 54 MPa with a maximum standard deviation of 0.45 MPa for the water curing group, while the compressive strengths of wetting-drying curing group and  $\text{MgSO}_4$  curing group vary in the range from 12.3 to 61.5 MPa with a maximum standard deviation of 0.96 MPa and 7.3-47.4 MPa, and with a maximum standard deviation of

Table 4. Experimental test programme

Test	No. of specimens	Dimension [mm]	Standards
Compressive strength	45	40 x 40 x 160	TS EN 196-1
Flexural strength	45	40 x 40 x 160	TS EN 196-2
Thermal conductivity	15 x 5 puta	40 x 40 x 160	ASTM C-518
Mercury intrusion porosimetry	15	40 x 40 x 160	ASTM D-7984

0.62 MPa, respectively. Considering the compressive strength, under all curing conditions, the compressive strength of mortar samples decreases with an increase in scrap tyre content.

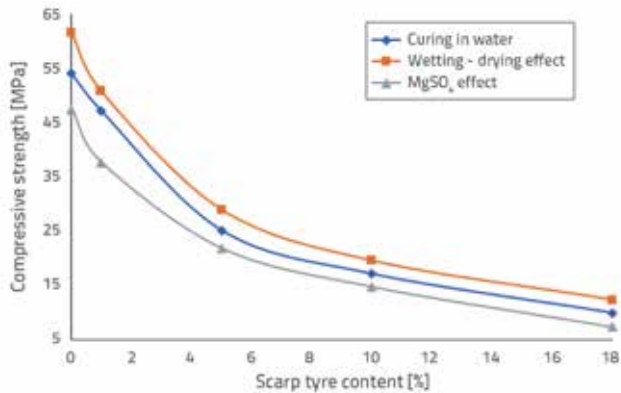


Figure 2. Relation between scrap tyre content and compressive strength test results for scrap tyre incorporated mortars

The inclusion of scrap tyre particles in mortar mixtures reduces the quality of the interfacial transition zone between scrap tyre particles and cement paste. Especially, the rough surface and nonpolar nature of the scrap tyre particles lead to a weak bond with the cement paste. The ability of rough scrap tyre surface to entrap air leads to an increase in air content with an increase in the scrap tyre content. Previous studies have indicated that the weak interface, poor adhesion, insufficient bonding, increased porosity, and low density, are the main reasons for the low strength of mortars containing scrap tyre. Because of the porous characteristics of scrap tyre – cement matrix transition zone, the compressive strength decreases with an increase in scrap tyre content under all curing conditions [16, 23-26].

For all mixture samples, the highest compressive strengths were obtained as 54, 61.5, and 47.4 MPa under water curing, wetting-drying, and MgSO<sub>4</sub> effects, respectively. For all mixture samples, the highest compressive strengths at equal scrap tyre contents were obtained from wetting-drying mortar samples, and the lowest ones were observed in samples exposed to MgSO<sub>4</sub> action. Under all curing conditions, sudden and significant losses in compressive strength were detected in samples with a 5 % content of scrap tyre (L2). In the case of the 5 % content, the compressive strength losses, which are 53.0 % at wetting-drying, 53.5 % under water curing, and 54.0 % under MgSO<sub>4</sub> effect, are remarkable when compared to control samples (L0).

### 3.2. Flexural strength results

Figure 3 presents flexural strengths for the 0 to 18 % scrap tyre incorporated mortar samples subjected to wetting-drying, MgSO<sub>4</sub>, and water curing effects. Flexural strength results are in the range of 2.7 - 7.2 MPa with a maximum standard deviation of 0.2 MPa for water curing group, while the flexural strengths of the wetting-drying curing group and the MgSO<sub>4</sub> curing group vary within the range from 3.9 to 9.1, MPa with a maximum

standard deviation of 0.3 MPa and 4.0 - 8.1 MPa, and with a maximum standard deviation of 0.43 MPa, respectively. Under all curing conditions, the flexural strength of mortar samples decreases as scrap tyre content replaced by cement increases in the mixtures (except for sample L1 exposed to MgSO<sub>4</sub>), which is similar to the compressive strength results.

For all mixture samples, the highest flexural strengths are obtained as 7.2, 9.1 and 8.7 MPa under water curing, wetting-drying, and MgSO<sub>4</sub> effects, respectively. A sudden decrease in flexural strength is observed when the scrap tyre content reaches 5 % (L2) in the wetting-drying curing method. This decrease is only 4.1 % for the 1 % content (L1) compared to the control sample (L0). However, the loss exceeds 30 % when the content is 5 %. In addition to this, water cured samples give the lowest flexural strength for all scrap tyre content intervals and for all curing conditions.

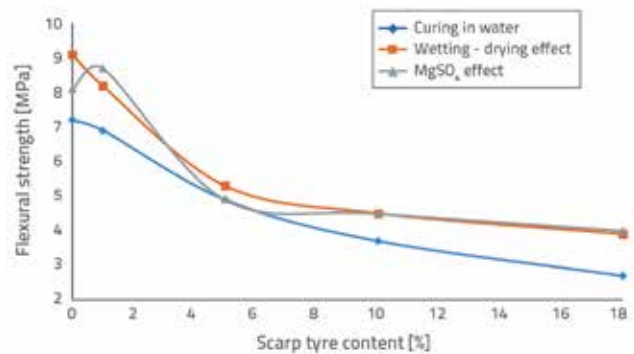


Figure 3. Relation between scrap tyre content and flexural strength test results for scrap tyre incorporated mortars

The control sample (L0) subjected to the wetting-drying effect has a higher flexural strength (9.1 MPa) than the control samples obtained for the remaining two curing conditions. When the scrap tyre ratio reaches 18 % (L4 samples), the flexural strength of water cured samples is the lowest compared to the strengths obtained for the other two curing media (3.9 MPa).

Under the wetting-drying and MgSO<sub>4</sub> curing conditions, samples (L3) and (L4) with 10 and 18 % of the scrap tyre content exhibit a nearly flexural behaviour. For 10 % scrap tyre content samples, a 44.4 % strength loss is observed in the case of flexural strength compared to the control sample without scrap content. The strength difference between the 10 % and 18 % content samples was obtained as 0.50 MPa under the MgSO<sub>4</sub> effect. This difference is 0.6 MPa under the wetting-drying curing conditions. It can be observed that, under this curing condition, the scrap tyre content in the range of 5 % is effective on the loss of flexural strength and causes sudden decreases.

### 3.3. Thermal conductivity test results

Thermal conductivity test results for scrap tyre incorporated mortars with the 0 %, 1 %, 5 %, 10 %, 18 % scrap tyre content are presented in Figure 4 for water curing, wetting-drying, and MgSO<sub>4</sub> curing conditions.

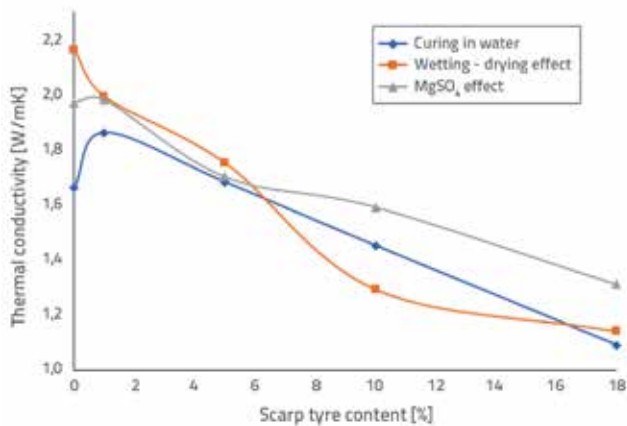


Figure 4. Relation between scrap tyre content and thermal conductivity test results of scrap tyre incorporated mortars

Under all curing conditions, the coefficients of thermal conductivity of all samples containing scrap tyre (L1, L2, L3, and L4) decreases while the content ratio increases. It has been established that the scrap tyre content should be no less than 10 % in order to achieve significant reductions in the coefficient of thermal conductivity under water curing. The coefficient of thermal conductivity which has been measured for the 5 % scrap tyre content is close to the control sample's value. In the sample with the 10 % content (L3), the coefficient of thermal conductivity decreased by 12.6 % compared to the control sample (L0). The increase of the content ratio up to 18 % (L4), brings about the decrease in the coefficient of thermal conductivity which amounts to 34.3 %.

Under the effect of MgSO<sub>4</sub>, thermal conductivity coefficient at the 1 % content is close to the value of the control sample. Significant reductions in the thermal conductivity coefficient could be achieved at a scrap tyre ratio of 5 % and above. Thermal conductivity coefficient decreases were found as 13.7 %, 19.2 % and 33.5 % for 5 %, 10 % and 18 % scrap tyre contents (L2, L3, and L4 samples), respectively.

In all samples exposed to wetting-drying (L0, L1, L2, L3, and L4), the increase in additive ratio resulted in the decrease in thermal conductivity coefficient. In the range of 0 - 5 % content ratio, the highest thermal conductivity coefficients were determined by wetting-drying effect. It allows the reduction of the thermal

conductivity coefficient even at low content rates. For samples subjected to the wetting-drying effect, the decrease values of 40.2 % and 47.2 % in thermal conductivity coefficient, which have been determined at the high waste tyre additive ratio compared to the control sample, are remarkable. The positive effect of the wetting-drying curing conditions on the decrease in the thermal conductivity coefficient in the mortars compared to the water curing and MgSO<sub>4</sub> effect is particularly prominent at the content rates of 10 % and 18 %.

### 3.4. MIP test results

Table 5 presents the results of porosimetry analysis of scrap tyre incorporated mortars exposed to curing in water, wetting-drying and MgSO<sub>4</sub> effects.

Under all curing conditions, the total porosity of mortar samples and the median pore diameter-volume increased along with an increase in the scrap tyre content. When evaluating the compressive strength-total porosity-median pore diameter relation, it was observed that high compressive strength is associated with low total porosity and low median pore diameter - volume in all curing methods.

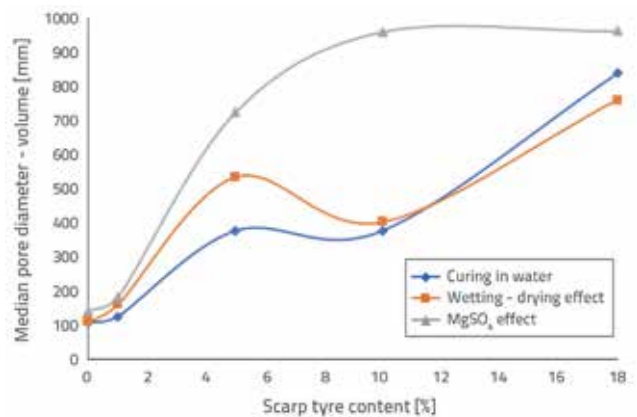
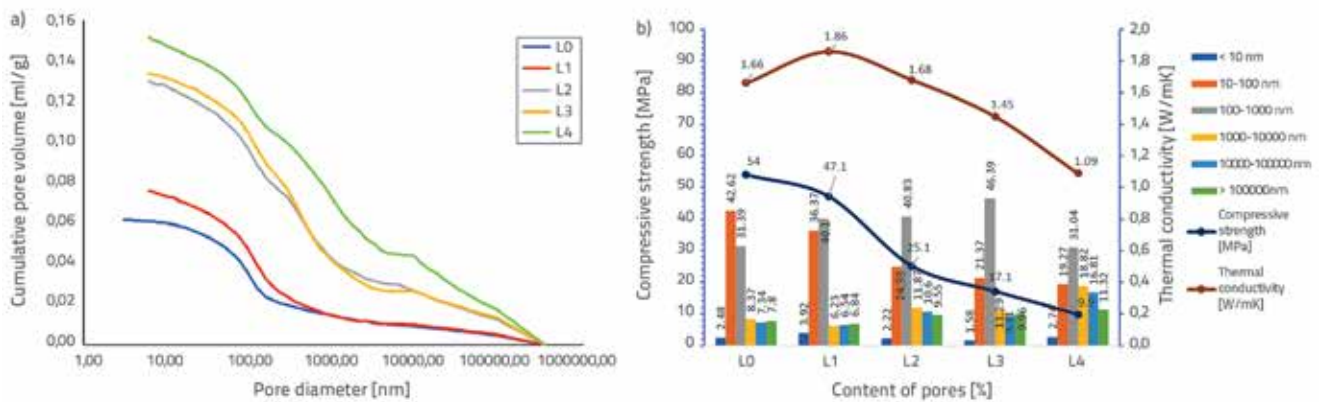


Figure 5. Relation between scrap tyre content and median pore diameter - volume of scrap tyre incorporated mortars

Figure 5 shows the median pore diameter-volume values at all content rates. Under the effect of MgSO<sub>4</sub> they are higher than

Table 5. MIP test results of scrap tyre incorporated mortars (MIP - mercury porosimetry)

Mix No.	Curing in water		Wetting - drying effect		MgSO <sub>4</sub> effect	
	Total porosity [%]	Median pore diameter - volume [nm]	Total porosity [%]	Median pore diameter - volume [nm]	Total porosity [%]	Median pore diameter - volume [nm]
L0	13.05	109.6	12.46	111.1	2.32	143.7
L1	15.57	126.5	14.05	161.8	15.89	182.5
L2	23.65	377.5	21.85	534.5	21.6	724.6
L3	23.99	377	23.24	402.5	21.01	961.1
L4	25.83	838.5	26.77	760.5	24.24	963.4



Slika 6. a) Pore size distribution for all samples cured in water obtained from MIP data; b) Relation between content of pores – thermal conductivity – compressive strength for water curing group

those in the other two media. This state, which is particularly pronounced at the scrap tyre content of 5 % and more, caused lower compressive strength at all scrap tyre content ratios from unmixed state to the maximum content proportion under  $MgSO_4$  effect, compared to compressive strength obtained from the remaining two media.

The cumulative pore volume - pore size distribution, and the relation between the content of pores – thermal conductivity - compressive strength of the samples that completed the curing process in water is shown in Figure 6a and Figure 6b, respectively. The samples exhibit a stable distribution behaviour over the entire diameter range in proportion to the total porosity and median pore diameter-volume values.

The sample L4 with the highest total porosity (25.83 %) and median pore diameter-volume (838.8 nm), and the lowest compressive strength (9.9 MPa) and flexural strength (2.7 MPa) also has the highest cumulative pore volume over the entire pore size distribution range. A persistent behaviour can be observed for the samples L0, L1, L2, and L3. Thus, it can be seen from the graph that samples L2 and L3 (377.5 nm and 377 nm), whose median pore diameter-volume values are very close to each other, exhibit characteristics that are quite similar across all diameter ranges. When assessing thermal conductivity coefficients, it can be observed that the decrease in the coefficient of thermal conductivity appears to correspond to a high cumulative pore volume throughout the graph, depending on the proportion of content in samples L1 to L4. This behaviour remains uncovered only by the L0 sample, which is a pure sample, in the 3-800 nm diameter range, and for the distributions of diameters higher than 800 nm, the curves for the L0 and L1 samples almost overlaps.

As can be seen in Figure 6.b, the pore analysis revealed that macro pore values for samples L0 (1.66 W/mK) and L1 (1.86 W/mK) amount to 15.1 % and 13.3 %, respectively. Therefore, it was determined that the thermal conductivity coefficient of L0, which has higher macro pores content, was lower than that of L1 due to the dominant effect of macro pores on the thermal conductivity coefficient, even though the L0 sample has a lower

cumulative pore volume at a diameter range of 3 to 800 nm. The macro pores formation detected in samples L2, L3, and L4 were 20.1 %, 19.3 %, and 28.1 %, and their thermal conductivity coefficients were determined as 1.68, 1.45, and 1.09 W/mK, respectively. The dominant effect of the increased macro pores formation on the thermal conductivity coefficient can be observed in these samples as well. Although a lower level of macro pores formation is observed in sample L3 compared to sample L2, the capillary pore formation detected in the 100-1000 nm range in sample L3 is at the maximum level (46.39 %). For a similar level of macro pores, a significant reduction in thermal conductivity coefficient was achieved with an increase in capillary pores formation.

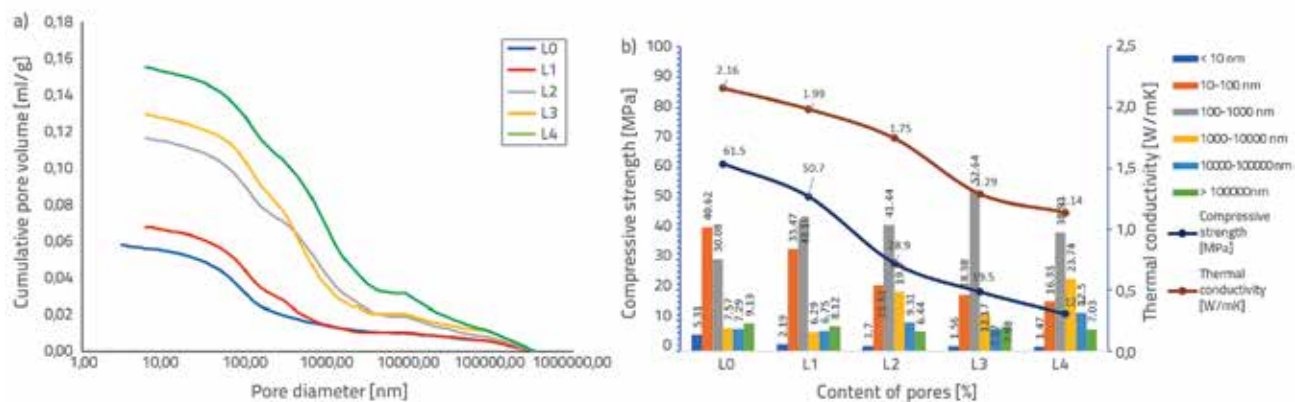
Figure 7.a shows the cumulative pore volume - pore size distribution and the relation between the content of pores – thermal conductivity - compressive strength of the samples under wetting-drying curing conditions.

As can be seen in Figure 7.a, a persistent distribution behaviour can be observed for all samples from L0 to L4, which depends on a high cumulative pore volume corresponding to a high total porosity and median pore diameter-volume, between compressive and flexural strengths, with the thermal conductivity coefficient characteristics as in water curing conditions.

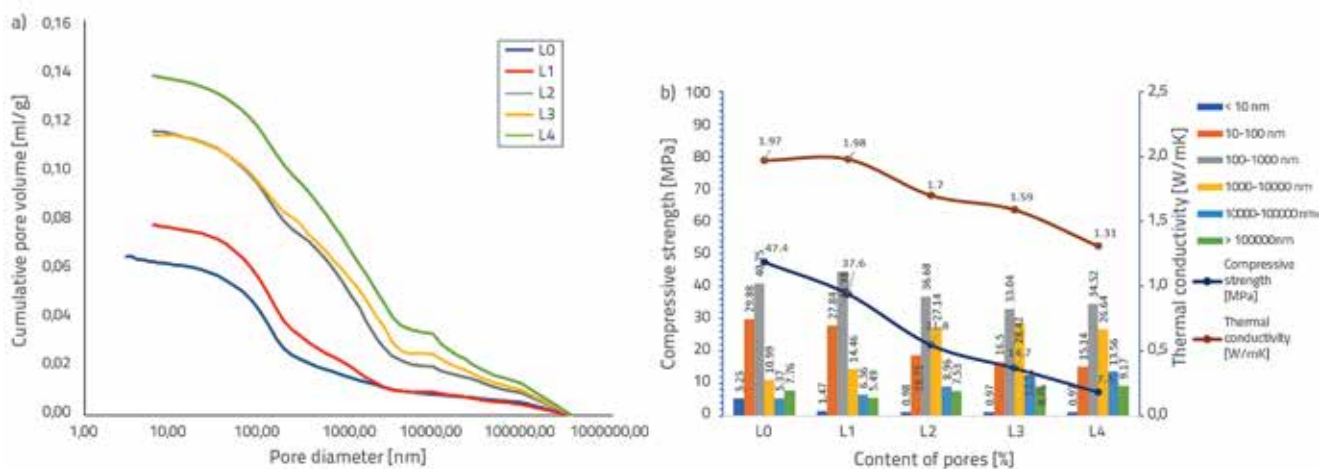
For the pore size distribution behaviour of samples L0 and L1, the curves coincide at high diameters, which is similar to the behaviour in water curing. However, the coincidence occurs at about 1545 nm and beyond, which differs from curing in the water.

Sample L1, which exhibits a higher cumulative pore volume up to the range of 3-1545 nm capillary diameters, has a lower thermal conductivity coefficient compared to sample L0. Therefore, the thermal conductivity coefficient difference was determined as 7.8 %.

Regarding the curves for samples L2 (1.75 W/m.K) and L3 (1.29 W/m.K), the latter sample had higher content ratio and lower thermal conductivity coefficient, and it exhibited a lower cumulative pore volume over an approximate diameter range of



Slika 7. a) Pore size distribution for all samples subjected to wetting – drying cycles obtained from MIP data; b) Relation between content of pores – thermal conductivity – compressive strength for wetting-drying curing group



Slika 8. a) Pore size distribution for all samples subjected to MgSO<sub>4</sub> effect obtained from MIP data; b) Relation between content of pores – thermal conductivity – compressive strength for MgSO<sub>4</sub> curing group

1000 to 9045 nm even though it was likely to exhibit a higher cumulative pore volume compared to sample L2 overall diameter ranges. However, increasing the graph along the diameter range at the macro level, the total porosity value reaches 23.24 % and a lower conductivity coefficient is observed compared to sample L2 with a total porosity of 21.85 %.

As shown in Figure 7.b, under the wetting-drying curing method the change in macro pores formation is more limited compared to the water-curing method. In spite of the limited macro pores change, the regular increase in capillary pores (30.0-52.6 %) in the range of 100 -1000 nm allowed a linear reduction of the thermal conductivity coefficient. The minimum thermal conductivity coefficient amounted to 1.14 W/mK for sample L4, at its maximum macro pores formation (19.5 %).

Figure 8.a shows the cumulative pore volume - pore size distribution and the relation between content of pores – thermal conductivity - compressive strength of samples under MgSO<sub>4</sub> effect. In the porosimetry analysis, for all samples from L0 to L4, a persistent distribution behaviour, which depends on high cumulative pore volume corresponding to high total porosity and median pore diameter-volume, exists between the compressive

and flexural strengths with the thermal conductivity coefficient characteristics as in the other two curing conditions.

The thermal conductivity values of samples L0 and L1 are very close to each other (1.97 W/mK and 1.98 W/mK). The dispersion curves coincide from approximately 2000 nm, however, sample L1 has a higher cumulative pore volume before this point. Initially, the partial cumulative pore volumetric difference between curves is not reflected in the thermal conductivity coefficient results. Therefore, macro pores diameter exerts a considerable influence on the thermal conductivity coefficient. An almost identical situation is observed for samples L2 (1.70 W/mK) and L3 (1.59 W / mK). The difference between the thermal conductivity coefficients is also related to the difference between macro pores (16.4 % and 21.0 %) as the pore diameter distribution up to 150 nm is almost the same. As shown in Figure 8.b, the change in macro pores formation (11.8-22.7 %) under the MgSO<sub>4</sub> effect controls the level of thermal conductivity coefficient.

Figure 9 shows the distribution of gel pores in the mortar pore structure, depending on the increased scrap tyre content. As a result of curing in water, the maximum gel pores (3.9 %



formation in the water-cured sample group is observed at sample L1 with 1 % scrap tyre content, which has the highest thermal conductivity coefficient (1.86 W/mK). Under the wetting-drying and  $MgSO_4$  effects, the gel pores values were 5.2 % and 5.2 %, respectively, for L0 samples, which does not include scrap tyre content. These values decrease suddenly at 1 % content, and gel pores formation continues by decreasing slightly with the increase of content rate.

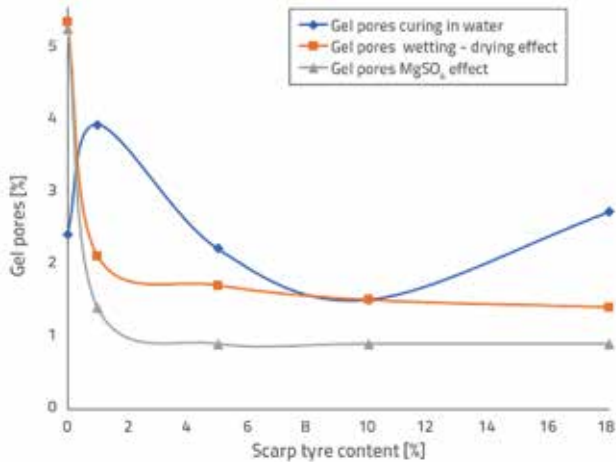


Figure 9. Relation between scrap tyre content and distribution of gel pores of scrap tyre incorporated mortars

Figure 10 shows the distribution of capillary pores in mortar pore structure, depending on the increase of scrap tyre content.

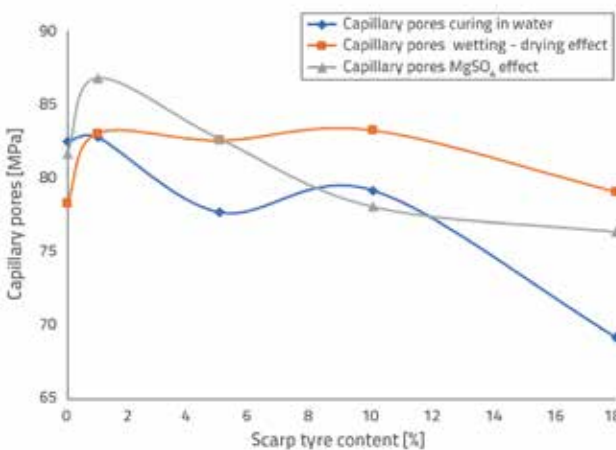


Figure 10. Relation between scrap tyre content and distribution of capillary pores of scrap tyre incorporated mortars

As a result of water curing, the maximum capillary pores (83.1 %) formation in the water-cured sample group is observed at sample L1 with 1 % scrap tyre content, which has the highest thermal conductivity coefficient (1.86 W/mK). Under  $MgSO_4$  effect, for sample L1 with 1 % scrap tyre content, in which the highest thermal conductivity coefficient (1.98 W/mK) was obtained, the percentage of capillary pores reaches its maximum value (86.8 %) in parallel with curing in water. Under the effect of

wetting-drying, the capillary pores reached the maximum value (83.1 %) in the case of 1 % content (L1).

Figure 11 shows the distribution of macro pores in mortar pore structure, depending on the increase of scrap tyre content ratio.

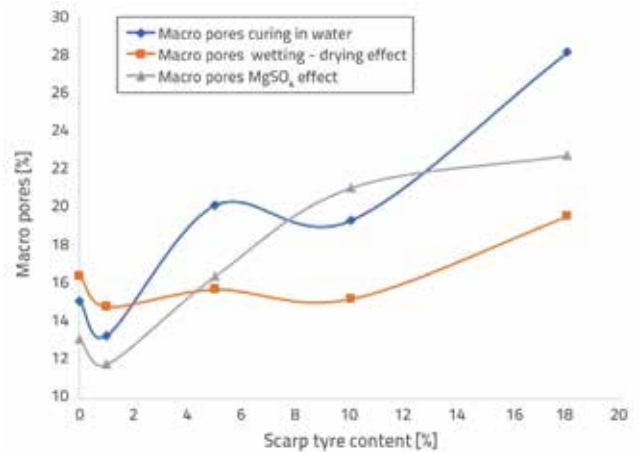


Figure 11. Relation between scrap tyre content and distribution of macro pores of scrap tyre incorporated mortars

Under all curing conditions, in the case of 1 % scrap tyre content, the percentage of macro pores in mortars reduces to the minimum value for all content cases and, accordingly, the thermal conductivity coefficients reach the maximum value.

### 3.5. Relation between test results and mechanisms behind each curing method

Figure 12 shows the standard deviation of strength results. Standard deviations were calculated for each curing method. As shown in Figure 12, standard deviations of compressive strength results are in range of 0.2-0.45 MPa for the water curing group, while the standard deviations of the wetting-drying curing group and the  $MgSO_4$  curing group vary in the range of 0.43-0.96 MPa and 0.17-0.62 MPa, respectively. Similar limited deviation results were also obtained by means of flexural strength tests. The standard deviation of flexural strength results ranges from 0.1 to 0.2 MPa for the water curing group, while the standard deviation of the wetting-drying curing group and the  $MgSO_4$  curing group varies from 0.17 to 0.26 MPa and from 0.1 to 0.43 MPa, respectively. The evaluation of standard deviations clearly shows that the results are quite consistent.

The evaluation of mechanical strength results obtained by water curing and wetting-drying curing conditions has revealed that the higher strength values can be reached by applying the wetting-drying curing method for all waste rubber content ratios. The cycles formed by the wetting-drying cure method contribute to the creation of a pore structure showing a decrease in total porosity compared to the water curing method. The decrease in total porosity allows the cavities in the mortar pore structure to be filled with calcium-silicate hydrates. With an increase in the number of C-S-H bonds, both compressive and

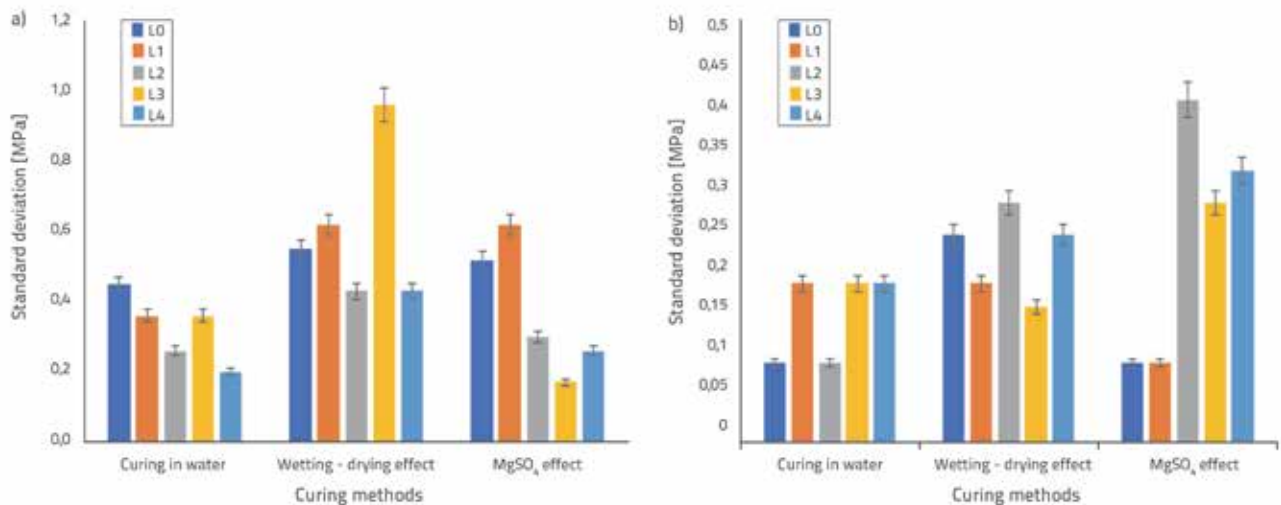


Figure 12. Standard deviations of: a) compressive strength results; b) flexural strength results

flexural strengths are higher in the case of the wetting-drying curing method compared to the reference water curing method. Compared to the water curing method, in the wetting-drying curing method - where positive and satisfactory mechanical strength results were obtained - it is very important to plan the drying cycle from the 7<sup>th</sup> day considering the relative humidity-hydration interaction for the first 7 days of hydration, and taking into consideration that 70 % of ultimate endurance of mortar samples is gained in the first 7 days.

In the MgSO<sub>4</sub> curing method, it was observed that higher median pore diameters – volume are formed despite the overall total porosity compared to the reference water curing method for all content ratios. Compared to the results of curing in water, while the decreasing porosity condition has a positive effect on flexural strengths, the increasing volumetric porous diameters cause decrease in compressive strength. In the MgSO<sub>4</sub> curing conditions, an increase in the median pore diameters –volume results in bond formation during hydration that is more brittle compared to water curing conditions. The MgSO<sub>4</sub> salt that was added to the curing water in the proportion of 13 % by weight causes the formation of magnesium-silicate hydrates during hydration. Magnesium - silicate hydrates, which are extremely brittle, reduce the amount of calcium - silicate bond formation that has a positive effect on compressive strength. Therefore, lower compressive strengths were determined for all content ratios in curing conditions under the influence of MgSO<sub>4</sub> compared to the water curing method. The positive effect of the low porosity character on the flexural strength necessitates a higher level of energy requirement for the formation of deformation in the cement matrix.

#### 4. Conclusions

The effect of curing methods and scrap tyre addition on the mechanical, thermal conductivity and pore structure properties of mortars were investigated.

Based on the results of the study, the following conclusions can be drawn under different curing methods:

##### Evaluating compressive strength results

- Considering the compressive strength results under different curing conditions, the maximum and minimum compressive strength values for equal scrap tyre content ratio were always obtained from samples under wetting-drying curing effect and MgSO<sub>4</sub> effect, respectively. Curing in water provides the results between the values of both remaining curing conditions.
- In the 0-18 % scrap tyre content interval in which experimental work was carried out, the positive effect of the wetting-drying curing condition on the compressive strength is remarkable. Since lower total porosity (0 - 5 %) at relatively lower content rates and lower median pore diameter volume at higher content ratios (10 - 18 %) were developed, the compressive strength showed higher values under the wetting-drying effect compared to the other two curing methods.
- Median pore diameter volume values reach the maximum for all additive ratios under MgSO<sub>4</sub> effect compared to the remaining curing media and, therefore, the highest losses in the compressive strengths have been observed under the MgSO<sub>4</sub> exposure.

##### Evaluating flexural strength results

- The lowest flexural strengths for all scrap tyre content ratios were determined for samples obtained from the water curing condition.
- Sudden decreases in flexural strength were found at 5 % scrap tyre content ratio for curing in wetting-drying and MgSO<sub>4</sub> effect. However, the decrease in flexural strength at 10-18 % content range under wetting-drying curing conditions and MgSO<sub>4</sub> effect is negligible.

##### Evaluating thermal conductivity – Pore structure results

- By assessing the thermal conductivity coefficient results under different curing conditions, the decrease in thermal conductivity coefficient values at all scrap tyre content ratios are observed from the point of 1 % under the wetting-drying effect. Even at 1 % content rate, a 7.8 % decrease was observed under the wetting-drying curing condition. Under  $MgSO_4$  and water curing effects, however, the decrease of 13.7 % and 12.6 % has been observed at 5 and 10 % content ratios, respectively.
- Under the wetting-drying effect, gel pores formation reduces due to the increase of content rate in mortar pore structure. For the  $MgSO_4$  curing, due to an additive amount increase in the range of 0-5 %, the gel pores formation gradually decreases and is not influenced by the increase of content.
- Under the different curing conditions, the highest capillary pores and the lowest amount of macro pores in the mortar pore structure are observed at 1 % scrap tyre content ratio. Accordingly, in the content range of 0 to 18 %, the thermal conductivity coefficient value reaches the maximum at 1 % scrap tyre content ratio under  $MgSO_4$  and water curing

conditions. In this study, using 18 % of the scrap tyre content, a remarkable effect on the thermal conductivity value was detected compared to the control sample in its curing condition. The effect was found at decrease rates of 33.5 %, 34.3 % and 47.2 % for  $MgSO_4$ , water, and wetting-drying curing conditions, respectively.

Regarding the mechanical strengths and thermal conductivity coefficient results for different mixture content ratios, scrap tyre incorporated mortar designs, which are created considering the design parameters based on the intended use and expectancy, are able to become an alternative to traditional mortars. Especially the wetting – drying curing method provides both higher compressive and flexural strengths, while the  $MgSO_4$  curing method provides higher flexural strengths for all additive ratios compared to the conventional water curing method. The strength results are promising for the production of prefabricated bending elements with alternative wetting–drying and  $MgSO_4$  curing methods.

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