

Influence of raw material properties on waste-based glass foam

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Abstract

Glass foam tablets were prepared by using mixed glass bottles (green, brown, and white), eggshell waste as a foaming agent, Na-bentonite as binder material, and red mud as an additive, respectively. The influence of the amount of red mud and fly ash on the properties (foaming temperature, mechanical strength, specimen density and porosity, thermal conductivity) of the tablets was studied. The foamed tablets contained 2.5 wt % binder material, 0.1 wt % foaming agent, 5 wt % fly ash, and red mud as additives in different ratios (0 – 40 wt %). After homogenization, the ground raw materials were pressed into tablets at 30 MPa using a hydraulic piston press. The products were heat treated at different temperatures with different heating rates. The final product's properties were measured (specimen density, thermal conductivity, and mechanical strength through falling and abrasion resistance tests). The most convenient results were shown at 5 wt % red mud in this case, the specimen density and thermal conductivity were the lowest, the values were 0.3 g/cm³ and 0.036 W/mK. If 5 wt % of fly ash was added to the mixture the density value stayed the same, the thermal conductivity increased to 0.048 W/mK, but the mechanical strength of the tablets rapidly increased.

Keywords:

recycling; glass foam; red mud; fly ash; eggshell

1. Introduction

Nowadays one of the biggest challenges of humankind is to solve sustainable development because the generated amount of waste causes environmental problems due to improper disposal and due to urbanization, the consumption of non-renewable raw materials is continuously rising (Cheng et al., 2016, Liu et al., 2019). One type of waste is glass waste which is generated in huge amounts in the packaging and construction industries (Shelby, 2017). Since it can be recycled 100%, its recycling potential is high (Sharma et al., 2021, Oss et al., 2008). As the production of new glass products is a high energy- and raw material-intensive process, recycling saves significant amounts of both (Da Costa et al., 2020, Souza et al. 2017, Song et al., 2021). In Hungary, the amount of glass waste has increased in the past few years (Szép, 2015). The problem is that there are no domestic glass factories that accept the produced amount of it and the transport cost to foreign factories are high, so they are not reused or recycled in high quantities (Seregély, 2015, Szép, 2015).

In recent years, a wide range of waste types has been used as secondary raw materials to produce environmen-

tally friendly building materials, such as glass foam which is a thermal insulation material (Scarinci et al., 2005). Glass foams are highly porous materials (Assefi et al., 2021, Cengizler et al., 2021). Due to glass foam being a lightweight, insulating material that has many positive properties, for example it is frost-resistant, chemically neutral, and non-flammable, this recycling method has many potentials.

In addition, it has relatively low transport costs and it is easy to combine with concrete (König et al., 2016, Scheffler and Paolo, 2006, Scarinci et al., 2005). It can be used on a large scale as a lightweight aggregate in concrete, as heat insulation for roofs, walls, floors, or ceilings, and as a filler for the restoration of failed slopes (Bueno et al., 2020).

It is made from glass powder and foaming agents. To produce glass foams, glass powder must be mixed with a foaming agent and must be heat treated at a high temperature in the range of 700-900°C where, due to the foaming process, gas formation occurs (König et al., 2016, Spence et al., 2016; Scarinci et al., 2005 Kálnai et al., 2007). During the heat treatment, as the furnace reaches higher temperatures, the viscosity of the glass is less than 106.6 Pa.s. where the gas formation occurs. The foaming agent particles are wrapped by the softened glass until the decomposition, or their reaction tempera-

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ture is reached, and then gases are released in the softened glass (Da Silva Fernandes et al., 2021).

The surface tension and the viscosity of the glass decrease with an increase in temperature. As a result of surface tension reduction, pore coalescence occurs, because the glass decreases the pressure over the gas bubbles and increases expansion (Da Silva Fernandes et al., 2021, Østergaard et al., 2019).

At the industrial level, most of the glass is derived from waste such as glass bottles or window glasses, but lamp glass or cathode ray tubes (CRT) are also used (Li et al., 2013, Qi et al., 2019, Federico and Chidiac, 2009, Saparuddin et al., 2020, Attila et al., 2013, Paul et al., 2018, Bueno et al., 2020).

In addition to glass, other alternative waste materials can be used in glass foam production, such as eggshells, fly ash, or red mud. Eggshells are one of the most common by-products of the food industry and are generated in huge quantities through everyday egg consumption. Its main component is calcium carbonate (94-96%), as calcite in crystalline form (Baláz, 2018). The carbonate content of eggshells makes them suitable for foaming, as they produce CO₂ gas during combustion, which causes the formation of pores (Souza et al., 2017, Fernandes et al., 2014, Ibrahim et al. 2022). Red mud is the by-product of bauxite processing plants, due to its high alkalinity it has a significant impact on the environment (Badanoiu et al., 2015).

Several studies showed (Guo et al., 2013, Xia et al., 2022, Chen et al., 2013, Pei et al., 2022, Da Silva Fernandes et al., 2021), that if red mud which is an alkaline leaching waste was added to the glass foam ceramics, a lower temperature was required during the foaming process, so in the end of the production, less energy consumption was required. Fly ash is a potential secondary raw material for several industrial uses, which is generated in coal-fired power plants in large quantities (Ambrus et al., 2019, Mucsi et al., 2019, Yalman et al., 2022). It is applicable as an additive in glass foams to increase their mechanical strength (Ambrus et al., 2019, Mucsi, 2016, Bai et al., 2014, Wang et al., 2018, Pereira et al., 2002, Zhu et al., 2016, Li et al., 2018). Abbreviations used for the sample materials in the methods and results are: ES – eggshell, FA – fly ash, RM – red mud.

The focus of the research was to create a high-added-value product with waste-based materials in an energy-efficient way, through systematic foaming experiments at different temperatures. The experiments were carried out with different red mud content to reduce the foaming temperature. In addition, 5 wt % FA was added to the tablets to increase their mechanical strength. The final products were characterized by falling and abrasion resistance tests to examine the mechanical strength, specimen density, and thermal conductivity were also tested to evaluate the heat insulation properties.

2. Materials and methods

The raw materials for the experiments were glass bottles of different colours (white, green, and brown), Na-bentonite as the binder, eggshell as the foaming agent, red mud from Ajka (Hungary), and lignite-type fly ash from Mátra (Hungary) as additives.

2.1. Raw material preparation

The glass powder was pre-crushed with a roll crusher (screen size: 1 mm), then milled for 180 minutes under dry conditions in a ball mill using stainless steel balls (Ø 20 mm) to achieve the optimal particle size of the GP (Fóris and Mucsi, 2022). The optimal particle size is < 100 µm because in other cases, the foaming process will not be sufficient (König et al., 2016, Arrigada et al., 2019, Fernandes et al., 2014, König et al., 2014, Attila et al., 2013). The abbreviation used for glass powder in the methods and results: GP – glass powder.

The foaming agent was milled for 120 minutes due to the plate-like structure of eggshells, in a ball mill under dry conditions with ceramic grinding balls (Ø 30 mm). To remove the organic content of the eggshell before milling it was heat treated in boiling water for 30 minutes.

In terms of binder and additive materials (Na-bentonite, FA, and RM), no grinding was required because their particle size was in the fine size range. In preliminary experiments, the phenomenon was observed that if the additive materials and the foaming agent were not sieved, they appeared as aggregates in the pores, which reduced their efficiency. To avoid this, sieving was required with a 32 µm mesh-size sieve under wet conditions. The raw materials were dried in a laboratory furnace at 105°C to a constant weight.

2.2. Particle size distribution

HORIBA LA-950V2 laser diffraction particle size analyzer was used to measure the particle size distribution of ground raw materials in wet conditions. Distilled water was used as dispersing media and Mie theory was applied as the evaluation method.

Table 1: Particle size of the raw materials

	GP (µm)	ES (µm)	FA (µm)	RM (µm)
X ₁₀	1.67	4.37	13.28	1.69
X ₅₀	13.63	16.38	62.50	2.39
X ₉₀	28.30	40.76	130.98	6.25

The smallest X₉₀ value was detected for GP, the materials used as additives and foaming agent had larger particle sizes. Presumably, this was the reason that if the raw materials were not sieved through, they appeared as aggregates as larger particles between the pores.

Table 2: Chemical composition of ES, GP, RM, and FA

	SiO ₂	Al ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	Fe ₂ O ₃	MnO	TiO ₂	P ₂ O ₅	S	F	LOI
Unit	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %	m/m %
ES	0.3	0.0	0.62	54.4	0.13	0.13	0.03	0.001	0.002	0.499	0.21	<0.3	45.71
GP	73.6	1.2	2.25	9.89	11.9	0.62	0.47	0.008	0.049	0.014	0.20	<0.3	-
RM	16.10	17.90	1.66	8.50	9.98	0.08	35.10	0.31	4.08	1.39	1.59	<0.3	12.8
FA	39.8	14	3.41	12.1	0.54	1.61	11.2	0.176	0.495	0.346	6.5	<0.3	4.28

2.3. Chemical composition

Chemical compositions of raw materials were measured by X-ray fluorescence (XRF) analysis. The loss of ignition (LOI) was measured at 950°C with 90 min heating time, and 60 min holding time in a static furnace. **Table 2** shows the results of the chemical composition analysis of raw materials (XRF).

Table 2 lists the chemical composition of raw materials. ES contains the highest amount of calcium oxide, which can help to produce the porous structure of the foams. Nevertheless, FA also contained a relevant amount of calcium oxide which leads us to the conclusion that it also has a role in porous structure. The RM contained the highest amount of aluminium oxide and iron oxide, and relatively low silica content which is typical of Bayer RM (Khairul et al., 2019). The XRF results indicated that FA and GP were Si-rich raw materials, this is optimal to make an effectively stable base during the foaming process. The results showed that GP is made from soda-lime glasses which are usually used in glass bottle production because they contain a high amount of calcium oxide and sodium oxide. ES has the highest value of loss of ignition because of the organic content. The LOI of RM was 12.8% and of FA was 4.28%, indicating a relatively high amount of unburned carbon content.

2.4. Systematic foaming experiments

The total mass of RM, ES, FA, Na-bentonite, and GP was 100%. The ES, the FA, Na-bentonite, and the RM were added in different ratios to the GP which are provided in **Table 3**. The rest of the mixture was GP depending on the additive materials' ratios. Homogenisation was carried out in a ceramic-lined mill for 10 minutes for each content. The tablets with 25 mm diameter were prepared with a hydraulic piston press at 30 MPa pressure. Each tablet was 10 g.

The raw tablets were heat treated in Nabertherm L (T) 3 laboratory static furnace at three different temperature ranges (700°C, 800°C and 900°C) with 120 minutes holding time (furnace heating time 90 min + 30 min holding time) using different heating rate (7.66 min, 8.77 min, 10 min). After heat treatment, their properties were investigated.

The content of ES and Na-bentonite was based on previous experiments (Fóris and Mucsi, 2021, Fóris

Table 3: Tablets' additive materials (RM and FA), foaming agent (ES), and binder material (Na-bentonite) content

ES wt %	Na-bentonite wt %	FA wt %	RM wt %
0.1	2.5	0	0
0.1	2.5	0	1
0.1	2.5	0	5
0.1	2.5	0	10
0.1	2.5	0	20
0.1	2.5	0	30
0.1	2.5	0	40
0.1	2.5	5	0
0.1	2.5	5	1
0.1	2.5	5	5
0.1	2.5	5	10
0.1	2.5	5	20
0.1	2.5	5	30
0.1	2.5	5	40

and Mucsi, 2022), as well as the FA content (Fóris et al., 2023). The RM ratio was determined by literature (Guo et al., 2013, Xia et al., 2022, Chen et al., 2013, Pei et al., 2022, Da Silva Fernandes et al., 2021).

2.5. Measurements of the properties of finished glass foam tablets

Since glass foams are lightweight products, in order to determine the specimen density values it is necessary to know pre- and after-heat treatment. The specimen density (ρ_s) was determined by their geometrical measurements, for this purpose a caliper was used, and their masses were measured with an analytical balance.

The mechanical strength and abrasion of the tablets are important, as they need to be resistant during transport. A laboratory ceramic-lined mill was used for the abrasion resistance test. 30 g tablets were tested from each RM ratio. The machine operated at 30 rpm for 10 minutes, and after removing the material, it was sieved using a 1 mm opening size sieve for the fine fraction. The degree of the abrasion was calculated using Eq. (1) from the mass of the fine fraction and the feeding material (Faitli et al., 2017).

$$\Delta m_{abr} = \frac{m_{fine}}{m_{feed}} * 100[\%] \quad (1)$$

Where:

Δm_{abr} – the amount of abrasion (%),

m_{fine} – the amount of material passed through the 1 mm sieve (g),

m_{feed} – the feeding material (30 g).

Each tablet was released in free fall from a height of 2 m onto a concrete floor repeatedly until it broke (Trinh, 2019). In each ratio, three tablets were tested.

To test the thermal insulation properties the thermal conductivity of the resulting GF tablets was measured using the Modified Transient Plane Source Method using a Therm Tci type thermal conductivity meter, which conforms to ASTM D7984 standard (Osfour et al.,

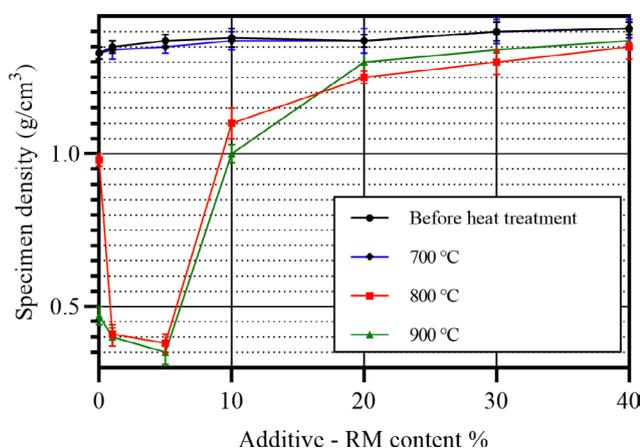


Figure 1: Specimen density values with only RM content

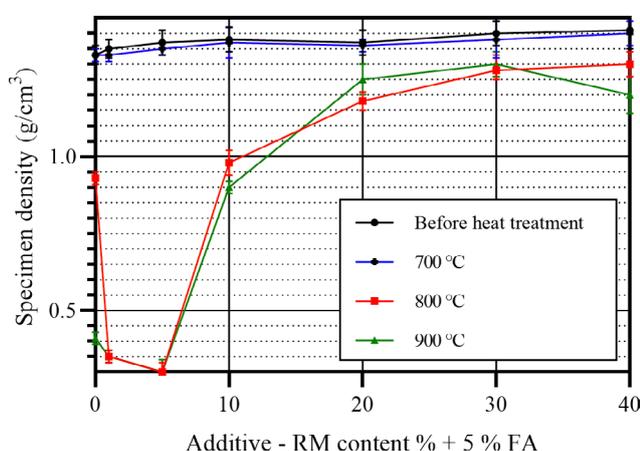


Figure 2: Specimen density values with RM content and plus 5 wt % FA

2023). For this purpose, a parallel surface was previously formed on the tablets using sandpaper.

3. Results and discussion

3.1. Specimen density before and after heat treatment

Specimen density of the tablets was measured before and after heat treatment, presented in Figures 1 and 2. The density at 700 °C was almost in the same range before heat treatment and did not change significantly after heating, this can be seen in Figure 3. However, heat treatment at 800 °C and 900 °C resulted in a significant decrease in specimen density of the tablets, especially for the 0-10 wt % RM content and 0-10 % RM + 5 wt % FA ratios.

For the tablets containing only RM, at 800 °C, the largest reduction was in the 5 wt% tablets, where the value was 0.38 g/cm³, and at 900 °C, the largest foaming was also in the 5 wt %, where the density of the tablets was 0.35 g/cm³.

In the case of tablets containing an additional 5 wt % FA, the trend was also observed that the 5 wt % RM tablets produced the largest decrease at both temperatures, with a density value of 0.30 g/cm³ in both cases. At 800 °C, no significant difference was observed between tablets containing only RM and tablets containing plus 5 wt % FA, but a difference was observed in the pore structure. The tablets containing plus 5 % FA showed less homogeneity and visibly larger pores, as shown in Figure 4.

It can be observed that above 20 wt % RM content did not change significantly compared to the pre-heat treatment condition, which leads to the conclusion that the foaming intensity decreases with the addition of RM. In the case of tablets containing only the foaming agent (0% RM), the foaming process started, and specimen density decreased to 0.98 g/cm³ even at 800 °C, but at 900 °C the value decreased to 0.47 g/cm³ by almost half compared to the previous temperature.

There is no significant difference in specimen density results between 800 °C and 900 °C treated tablets, except for 0 % RM content, suggesting that the addition of red mud can indeed reduce the foaming temperature significantly, saving energy in the process. The specimen densi-

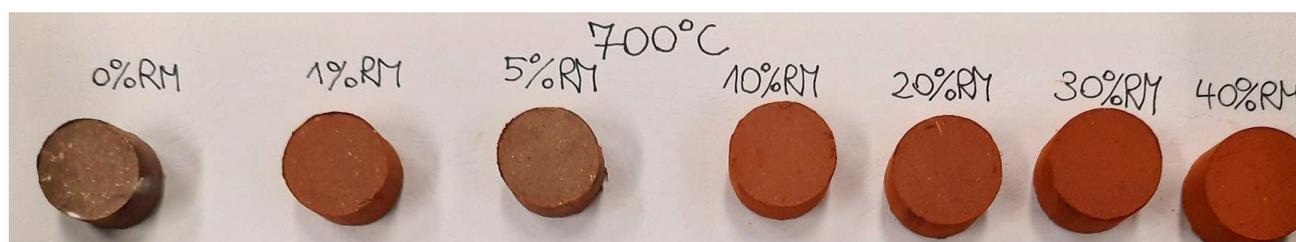


Figure 3: Tablets with different RM content after heat treatment at 700 °C



Figure 4: Tablets treated at 800°C with different RM ratios and plus 5 wt % FA

ty results at 800°C and 900°C displayed a linear trend with an increase in RM content from 10 wt % to 40 wt %.

3.2. Abrasion resistance test

Figure 5 shows the abrasion resistance values of the tablets. Overall, the abrasion rate is below 1.5 % for all tablets. It is observed that tablets with RM content above 20 wt % showed higher abrasion than tablets between 0 - 10 wt % RM. This trend was also observed for tablets containing + 5 wt % FA. Outliers are seen at 800°C for tablets containing 5 wt % RM and at 900°C also 5 wt % RM, in the range of 0-10 % RM content. The reason is the high-intensity foaming, as the specimen density results showed that this is where the maximum decrease occurred.

The lowest abrasion resistance values were 0.69 % at 700°C with 10 wt % RM plus 5 wt % FA, 0.67 % at

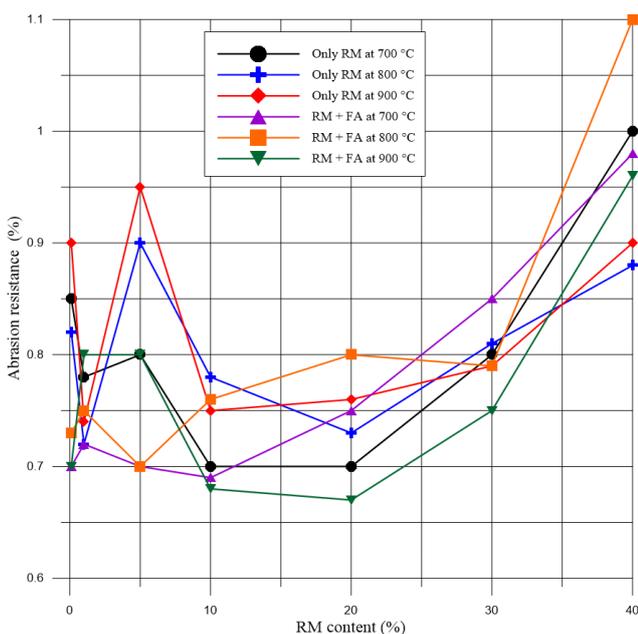


Figure 5: Abrasion resistance values

800°C with 5 wt % RM plus 5 wt % FA, and 0.67 % at 900°C with 20 wt % RM plus 5 wt % FA. Although the rate of abrasion is negligible, tablets containing an extra 5 wt % FA showed lower abrasion values than tablets containing only RM.

3.3. Falling test

Figure 6 shows the results of the falling test. The number of drops is the number of falls the sample survived undamaged. The mechanical strength of the tablets increases with the 5 wt % FA content at different temperatures, respectively. In addition, above 10 wt % RM content, the tablets broke earlier than 0 to 10 wt % RM. The more RM content the tablets had, the sooner the breakage occurred due to the impact of the falling. Furthermore, the same trend was also adequate for tablets with 5 wt % FA content at different RM contents.

If only RM was utilised to produce glass foam tablets the maximum number of unbroken drops was 20. At the same time, when 5 wt % FA was added to the mixture beside the RM content, this number increased significantly, especially for tablets containing 1-10 wt % RM. The mechanical strength showed a linear trend with the increasing RM content until 5 wt % where a significant peak occurred and after continuous decreasing was observed with the increasing RM ratio.

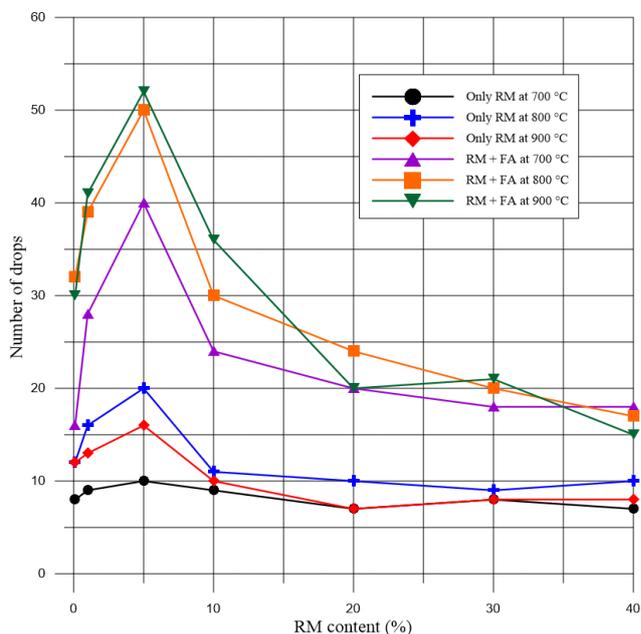


Figure 6: Results of the falling test

3.4. Thermal conductivity

Figure 7 illustrates the thermal conductivity results. Based on the literature, the average thermal conductivity of eggshell foamed tablets ranges from 0.177-0.055 W/mK (Attila et al., 2013, Cengizler et al., 2021, Chen et al., 2012, Xia et al., 2022). Overall, the results showed values below 0.08 W/mk. For this test, the tablets made

at 800°C and 900°C with 0-10 wt % RM and plus 5 wt % FA content were examined, respectively. The reason for this was that from the previous experiments, these tablets had significant foaming, so the key issue here was to see if these tablets were suitable for thermal insulation purposes. The figure clearly shows that all tablets fall within this range. Furthermore, it can be observed that the tablets containing only 5 wt % RM and 5 wt % RM plus 5 wt % FA gave the lowest value, in both temperature ranges. It can be observed that the value obtained for tablets containing 10 wt % RM is significantly higher than containing 0-5 wt % RM. A huge decrease in the values is observed for the plus 5 wt % FA content when heat treated at 800°C and for the plus 5 % FA content at 900°C, up to 5 % RM content, followed by a significant jump in the values for the 10 % RM content.

An exception to this is the case of tablets containing only RM, where a rise to 1 wt % RM is observed at both temperatures, followed by a decrease at 5 wt % RM.

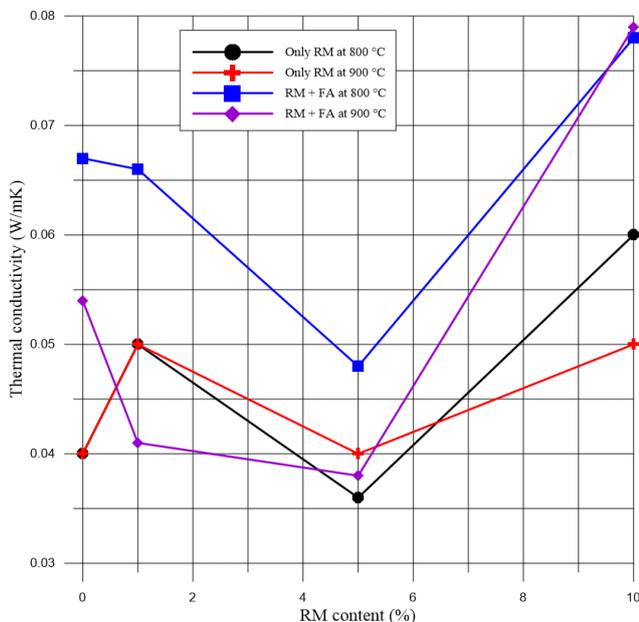


Figure 7: Thermal conductivity values for tablets containing 0- 10 wt % RM

4. Conclusions

The study highlighted the opportunity to recycle red mud and fly ash in thermal insulation by producing glass foam tablets. During the experiments systematic foaming experiments were carried out and glass foam was successfully prepared at different temperatures using ES as the foaming agent, sodium bentonite as binder material, FA and RM as additional materials. The following conclusions were observed from the measurements:

- (1) The specimen density of the tablets decreased with the addition of RM content. This trend has also occurred if 5 wt % FA is added to the mixture.
- (2) The most convenient performance was achieved when the tablets contained 5 wt % RM, treated at

800°C for 30 minutes respectively. In this case, the specimen density was 0.3 g/cm³ and the thermal conductivity value was 0.036 W/mK, which were the lowest values.

- (3) The tablets contained 5 wt % RM and 5 wt % FA, treated at the same temperature (800°C), and holding time also showed 0.3 g/cm³ specimen density value and relatively low thermal conductivity which was 0.048 W/mK, but they had much higher mechanical strength and lower abrasion resistance values than the tablets that contained only RM.
- (4) It can be concluded that using RM can reduce the foaming temperature from 900°C to 800°C in a significant way.
- (5) From the falling test and abrasion resistance test results, adding FA to the mixture significantly increased the mechanical strength of the tablets in an efficient way.
- (6) From the thermal conductivity results, the finished glass foams are suitable as thermal insulating materials, as low values were obtained, especially in the case of tablets containing 5 wt % RM.

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SAŽETAK

Utjecaj svojstava sirovina na staklenu pjenu od otpadnoga stakla

Granule staklene pjene pripremljene su korištenjem mješavine staklenih boca (zelene, smeđe i bijele boje), otpadaka ljuske jajeta kao sredstva za stvaranje pjene, Na-bentonita kao vezivnoga materijala uz dodatak crvenoga mulja. Proučavan je utjecaj količine crvenoga mulja i lebdećega pepela na svojstva tako oblikovanih granula (temperaturu pjenjenja, mehaničku čvrstoću, gustoću i poroznost uzorka, toplinsku provodljivost). Granule staklene pjene sadržavale su 2,5% masenog udjela (wt %) vezivnoga materijala, 0,1 wt % sredstva za pjenjenje, 5 wt % lebdećega pepela i crvenoga mulja kao dodataka u različitim omjerima (0 – 40 wt %). Nakon homogenizacije samljevene sirovine prešane su u granule pri 30 MPa uz pomoć hidrauličke preše. Proizvodi su toplinski obrađeni na različitim temperaturama s različitim brzinama zagrijavanja. Izmjerena su svojstva konačnoga proizvoda (gustoća uzorka, toplinska vodljivost i mehanička čvrstoća kroz ispitivanja otpornosti na udar i abraziju). Najbolji rezultati dobivani su kod 5 wt % crvenoga mulja i tu su gustoća uzorka i toplinska vodljivost bile najniže, s vrijednostima od 0,3 g/cm³ i 0,036 W/mK. Međutim, kada se mješavini dodalo 5 wt % lebdećega pepela, vrijednost gustoće ostala je ista, toplinska vodljivost povećala se na 0,048 W/mK, ali je mehanička čvrstoća granula naglo porasla.

Ključne riječi:

recikliranje, staklena pjena, crveni mulj, lebdeći pepeo, ljuske jaja

Authors' contribution

Ildikó Fóris (1) (PhD student) provided the raw material preparations, performed the laboratory work, and the presentation of the results. **Gábor Mucsi (2)** (Professor) provided the evaluation of the experimental results and the presentation of the results.