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LATE-MICROWAVE IRRADIATION OF ALKALI-ACTIVATED WASTE GLASS WOOL: LINKING DEHYDRATION RATE WITH THERMOMECHANICAL BEHAVIOUR

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Building and civil engineering conventional inorganic binding materials represent a significant burden for the environment, leading to the search for more sustainable materials. One of the potential solutions is alkali-activated materials (AAMs), which can be made solely from waste materials and at lower temperatures. However, reaction rate and solidification time depend on the precursor used. To enhance the speed of solidification, the curing temperature can be increased, but from the inside out not to create the crust on the surface, which would hinder the dehydration. Therefore, three mixtures of alkali-activated glass wool, a slow-in-alkali media solidifying precursor, were irradiated with low- and high-power microwaves in the late curing stage till complete dehydration, to determine the influence of artificial volumetric solidification onto thermomechanical behaviour of the AAM. As the electromagnetic power increased, the damages in the AAM became more severe, resulting in a reduction in mechanical strength.

Keywords:
microwave irradiation,
electromagnetic
dehydration,
glass wool,
alkali-activated material,
mechanical performance

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1 Introduction

Building and civil engineering inorganic binding materials currently available on the market (e.g. cement, mortar, concrete, ceramics) represent a significant burden for the environment ('The Industry Creating a Third of the World's Waste', n.d.):

- Continuous consumption of raw materials (yearly almost 70 $m_{\%}$ of the mass of Mount Everest),
- The high energy demands of production leading to a significant carbon footprint (approximately 40% of man-caused CO_2), and
- Constant waste generation (more than 30 $m_{\%}$ of global waste).

While building materials already act as a "black hole" for various types of waste, climate change calls for an urgent transition to more sustainable alternatives, like alkali-activated materials (AAMs) (Škvára 2007; Obonyo et al. 2014). AAMs can be made solely from (local) waste materials and at lower temperatures (Pacheco-Torgal, Castro-Gomes, and Jalali 2008), which both positively affect the influence on the environment. The only requirement from the waste material is that it contains enough Si and Al in the amorphous content (Horvat and Ducman 2019), like glass wool (GW) (Horvat et al. 2018).

GW is a fibrous material that consumes at the end of its use a lot of space in landfills due to its low density (Horvat et al. 2018), therefore, using waste GW as the source of Si and Al in alkali activation for building products is an ideal solution. However, GW needs a longer time and/or higher temperature for solidification (Pavlin et al. 2021), which is a significant environmental and economic drawback.

To speed up the solidification process in AAMs based on GW without the use of conventional heat chambers and driers, surface heating can be replaced with volumetric heating. Unlike conventional surface heating, which requires hours or days, volumetric heating can finalize the AAM within minutes (Horvat et al. 2022; 2023). However, the rapid finalisation of a freshly prepared mixture of the precursor and alkali (slurry) with high-power microwaves ends as a foam and not a potential load-bearing material (Horvat et al. 2023).

Therefore, when foamed AAM is not desired, volumetric heating can be applied after the initial conventional heating (Tesovnik and Horvat 2024a; 2024b). However, low-power microwaves can lead to foamed material when the curing stage is not completely finalised (Tesovnik and Horvat 2024a), which insists that AAM can be exposed to microwaves for a period shorter than the time needed for complete dehydration.

Nonetheless, before evaluating the point to which material can be exposed to electromagnetic irradiation, it is necessary to study the exposure limit beyond which no further changes occur. Therefore, this study analysed three mixtures of alkali-activated GW irradiated with low- and high-power microwaves in the late curing stage till complete dehydration to determine the influence on the thermomechanical and structural behaviour of the AAM. The study showed that high-power microwaves lead to macro-fractures which cause a significant decrease in mechanical performance, while low-power microwaves cause less significant damage. The most important finding in this study was the existence of a critical point of microwave power, where the amount of water does not influence the time of dehydration.

2 Experimental

2.1 Materials and characterisation of the materials

The following ingredients were used in the synthesis of AAMs:

- Waste GW with organic material on its surface (loss on ignition at 550 °C is 5.7 $m_{\%}$) was used as a precursor. GW was milled in a vibrating disk mill (Labor-Scheibenschwingmühle TS.250, Siebtechnik GmbH) and sieved below 63 μm.
- Na-silicate solution (Geosil, 344/7, Woelner, 16.9 $m_{\%}$ Na_2O, 27.5 $m_{\%}$ SiO_2, 55.6 H_2O), which was used as received as the only (aqueous liquid) alkali.

In the present study, there was no theoretical limit (Horvat and Ducman 2019) on the amount of alkali added to prevent efflorescence, as GW naturally contains such a high amount of Na (Horvat et al. 2018) that efflorescence in alkali-activated GW would happen without added alkali (see Tables 1 and 2) – of course, if GW would dissolve and react in water itself. Namely, the molar ratio between amorphous elements from the first group of the periodic table and amorphous Al is higher than 1.

To evaluate the amount of excessive alkali in the mixtures (and GW), GW (dried, milled and sieved below 63 µm) was analysed by:

- X-ray fluorescence (XRF; Thermo Scientific ARL Perform'X Sequential XRF) to determine the chemical composition (elements from fluorine to americium). The main results are collected in Table 1, raw analysis is in the open repository.
- X-ray diffraction (XRD; Empyrean PANalytical X-ray Diffractometer, Cu X-Ray source; under clean room conditions in the 2θ range from 4 to 70° and step 0.0263°) to qualitatively determine the minerals. Results are shown in Figure 1. Because the determination of the most prominent peak at ~44.5 ° (and a few more on the amorphous halo) might lead to a false positive result, the mostly amorphous pattern was considered as completely amorphous.

Therefore, elements present in the amorphous content, which is calculated as the difference between XRF and XRD (Horvat and Ducman 2019), are elements measured by XRF. The results of the GW evaluation are shown in Table 1.

Table 1: Mass percentage ($m_{\%}$) of crucial elements in alkali activation.

Elements [$m_{\%}$]	Na	K	Mg	Ca	Al	Si	Content [$m_{\%}$]
XRF	13.5	0.5	5.6	8.7	6.1	62.5	100
XRD	0	0	0	0	0	0	0*
Amorphous	13.5	0.5	5.6	8.7	6.1	62.5	100

*The one prominent peak (at 2ϑ ~ 44.5 °), which indicates that crystalline material is present in GW, was not enough to determine the mineral(s) present in GW. Nonetheless, their amount is insignificant.

The response of GW and all AAM samples to thermal exposure was measured in platinum crucibles using thermogravimetric analysis (TGA; TGA Q5000IR analyser, TA Instruments, New Castel, DE, United States) in constant airflow. Masses of (milled and sieved below 63 µm) materials used for analysis ranged from 6±2 mg. Because the focus of TGA analysis was only on water and organic compound behaviour, which can be influenced by microwave irradiation, the temperature range was from 25 to 600 °C. The heating rate was 5 °C/min.

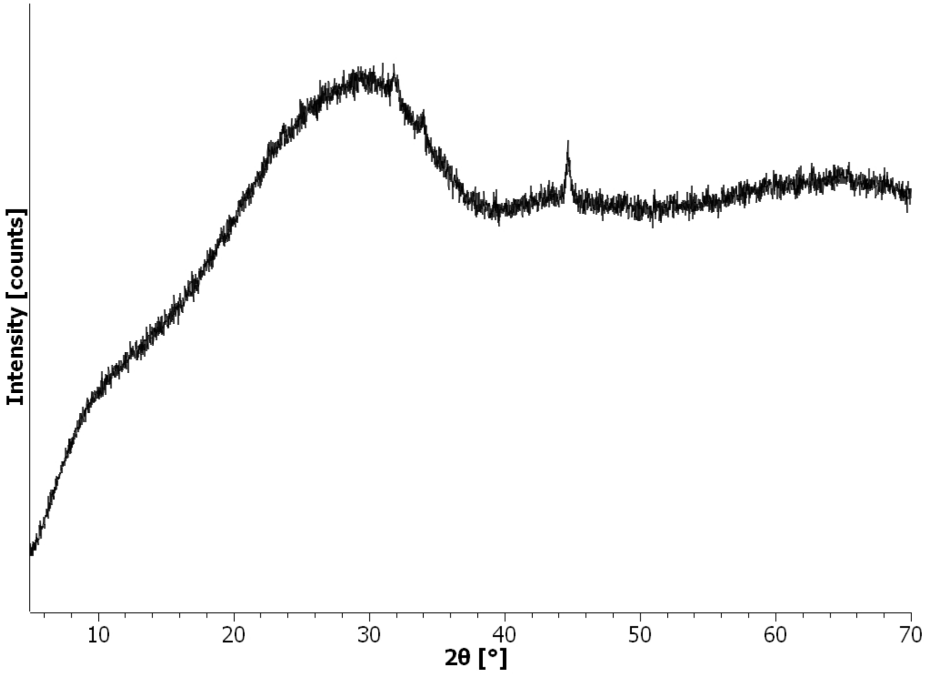

Figure 1: XRD pattern of waste GW.
Source: own.

The compressive strength, which determines if the building industry product has the potential to bear the load, was measured with the compressive and bending strength testing machine (ToniTechnik ToniNORM) on 7- and 21-day-old AAM prisms.

2.2 Synthesis of the material

AAMs were prepared in three different recipes (100-50, 100-80, and 100-100, where the numbers indicate the mass of precursor to the mass of alkali) shown in Table 2 along with their chemical compositions, which are relevant to the potential of efflorescence occurrence and compactness of AAM (Duxson et al. 2005) as follows:

- If the molar ratio between amorphous elements from the first group of the periodic table and Al (1^{st}/Al), is higher than 1, efflorescence will eventually occur.

- If the molar ratio between amorphous Si and Al (Si/Al) is 1.9, compressive strength will be the highest, if this ratio is below 1.4, the structure will be porous, if it is above 1.9, compressive strength will be lower than when the molar ratio is 1.9.

The theoretical molar ratio of GW and AAM mixtures (shown in Table 2) was calculated using software designed in MS Excel platform, developed in project no. C3330-17-529032 "Raziskovalci-2.0-ZAG-529032" (Horvat and Ducman 2019) and upgraded in the ARIS project under grant no. J2-3035.

Table 2: Chemical composition of the samples (precursor and AAMs) with mixture recipes.

Sample	1st/Al [mol/mol]	Si/Al [mol/mol]	Precursor [g]	Alkali [g]	H_2O in alkali [g]	Na in alkali [g]
GW	3.6	8.5	/	/	/	/
100-50	5.9	10.5	100	50	27.8	6.3
100-80	7.3	11.6	100	80	44.5	10.0
100-100	8.2	12.4	100	100	55.6	12.5

The prechosen masses of GW and alkali were mixed at up to 1000 rpm. Freshly mixed slurries were moulded into 2×2×8 cm^3 prisms in a rubber mould, and cured for 6 days at 40 °C in the heating-drying chamber (Memmert, Universal Oven U). On day 7, prisms were demoulded and treated further: one-third of the samples was irradiated with microwaves at 100 W, the second third at 1000 W, both until complete dehydration, and the last third was left to evolve further at room conditions as a reference.

Dehydration was performed in an inverter microwave oven (Panasonic, NN-CD575M; frequency 2.45 GHz, magnetron source; working in mode with constant presence of microwaves) following the procedure shown in Figure 2:

- A ceramic plate (cooled to room temperature) was put into the centre of the microwave chamber.
- 7-day-old bulk prisms were placed individually into the centre of the ceramic plate.
- The prism on the ceramic plate was covered by a clean and dry 1000 ml beaker (cooled to room temperature) placed upside-down, without touching

the prism (the beaker cannot withstand the temperatures created in the prism by electromagnetic irradiation – the beaker explodes if it gets into contact with the heated prism). On the left and right side of the prism, a paper towel was placed to absorb condensed water to disable its absorption by the prism.

- Set-up was irradiated at the prechosen power (100 W, the lowest possible power, and 1000 W, the highest possible power) first for 30 s, then every additional irradiation for 10 s, until there was no condensed moisture seen on the beaker.
- The beaker and plate were replaced with clean, dry and cooled to room temperature beaker and plate for every additional irradiation. The time of the prism kept at the room conditions between irradiations was as minimal as possible.

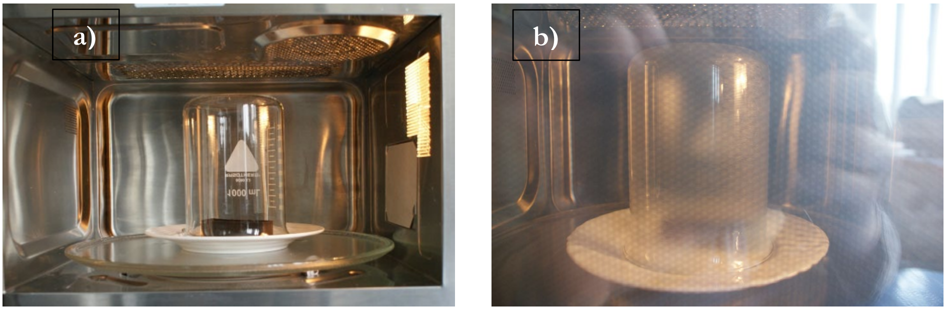

Figure 2: Dehydration set up a) before irradiation and b) during irradiation.

Surce: own.

3 Results and discussion

7-day-old AAM prisms of all three mixtures are shown in Figure 3, from which it can be concluded, that the amount of liquid defines the rheology of the slurry (fresh mixture of precursor and liquid alkali) and the moulding procedure. If there is not enough liquid, AAM can end as ellipsoidal aggregates, unless the aggregates (or wetted powder) are physically pressed in the mould (mixture 100-50) into a prism or a tablet. Therefore, for this study, the mixture 100-50 was manually pressed directly into the mould (layer by layer), while both other mixtures (100-80 and 100-100) were poured into the mould. While there is no inhomogeneity obvious in the mixture 100-

50 (the sample is overall made from small spheres of the same colour), for mixtures 100-80 and 100-100 buoyancy is expressed by the lower lighter coloured part of the prism, and the upper darker part of the prism. The latter expresses a sticky nature (when touched with fingers), while the lower part is solid and "non-wet", meaning that with alkali-reacted organic material (which is present on the surface of the GW) travels towards the top surface of the prism. Therefore, the physically optimal mixture is somewhere between 100-50 and 100-80, when buoyancy on a large scale will not occur.

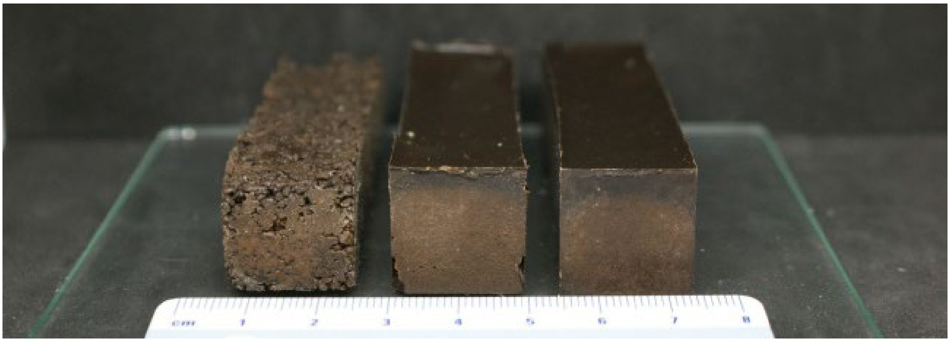

Figure 3: 7-day-old non-irradiated prisms. Mixtures from left to right: 100-50, 100-80, and 100-100.
Source: own.

From the cross-sections of the prisms, shown in Figure 4, local inhomogeneities present in samples not exposed to the microwave irradiation (cracks in the case of the mixture 100-50, and spheres in the case of the mixtures 100-80 and 100-100) are a consequence of the manual moulding. Cracks occurred because layers were not evenly pressed over the whole top-surface area, and because not all layers were pressed with the same force. Spherical bubbles, on the other hand, can occur in all AAMs that are conventionally moulded (slurry is poured into the mould) as remaining bubbles from mixing powder and liquid. While the cracks can be avoided by machine pressing, bubbles can be removed by shaking freshly moulded prisms on vibration tables for concrete.

Nonetheless, microwave irradiation induced additional spherical pores and elongated cracks. Spherical pores in irradiated samples were a consequence of 7-day-old AAMs not being completely finalised (curing at 40 °C for 6 days was not enough to completely cure the sample), which left an open window for late volumetric foaming (Tesovnik and Horvat 2024a). However, because irradiation did not stop when induced porosity would be only spherical, but was prolonged until there was any steam coming out from the prism, the pressure in the prism increased to the point that the aluminosilicate network opened to depressurise and let the steam out through artificially formed elongated cracks (Tesovnik and Horvat 2024a). The severity of the microwave-induced porosity was visually more notable for mixtures synthesised with a higher amount of liquid (mixtures 100-80 and 100-100), ending with more spherical pores compared to the mixture 100-50. The most severe damage in the aluminosilicate network was caused by higher-power microwave irradiation (1000 W) for mixtures with more liquid (100-80 and 100-100), while the mixture with the smallest amount of liquid had no clear difference between low-power (100 W) and high-power (1000 W) irradiation.

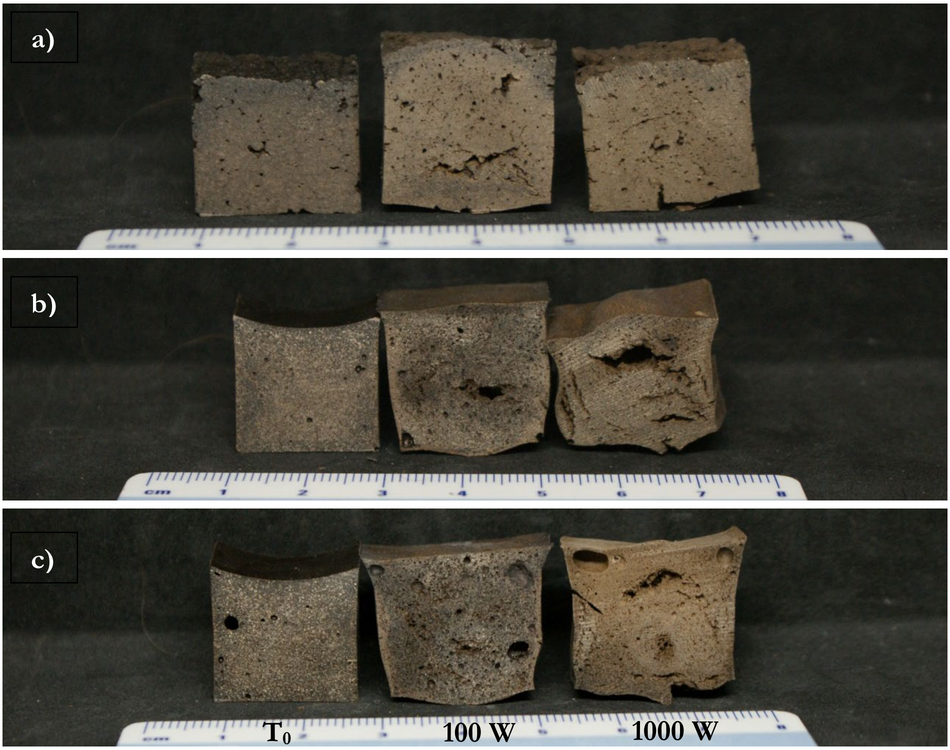

Figure 4: Cross-section of 14-day-old mixtures a) 100-50, b) 100-80, and c) 100-100, that were (from left to right) kept at room conditions, irradiated at 100 W, and at 1000 W.

Source: own.

The compressive strength, shown in Figure 5 (measurements are available in the open repository), decreased with the addition of liquid alkali, i.e. mixture 100-50 had the highest compressive strength and was the least impacted by irradiation. This phenomenon can be explained by Figure 4 where irradiated mixture 100-50 shows the highest structural integrity among the mixtures.

While the compressive strength for the non-irradiated sample slightly increased with time (Figure 5a compared to Figure 5b), for the samples irradiated with high-power microwaves the compressive strength decreased.

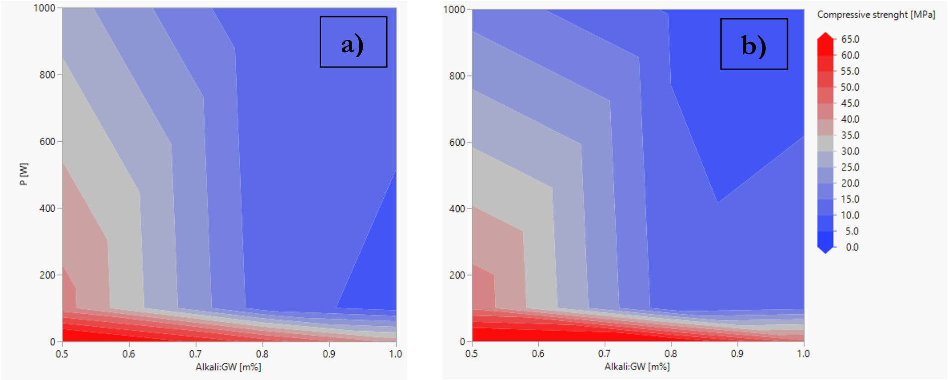

Figure 5: Delaunay triangulation of compressive strength of a) 7 and b) 21-day-old AAM prisms from GW in different mass ratios of GW and alkali (0.5, 0.8, and 1; mixtures 100-50, 100-80, 100-100, respectively).
Source: own.

The irradiation time, shown in Figure 6a, needed to reach the dehydration stage when no visible steam appears on the beaker anymore depends on: (i) the mass of water, (ii) the compactness of the irradiated material (initial cracks/voids/pores), (iii) the strength of the chemical bonds holding the material together, (iv) power of microwaves, (v) frequency of the microwaves, and (vi) the dimension of the irradiated material. While there was no option to vary the frequency of microwaves, also the dimensions of the prisms, chemical bonds and compactness of the material were assessed as comparable among the samples, and therefore a constant. The only parameters heavily influencing the outcomes of the irradiation were the mass of water and the power of microwaves.

Samples containing the same amount of water needed less time for dehydration if the power of microwaves was higher. This is the same as in conventional heat treatment, i.e. the material exposed to higher temperatures reaches higher temperatures faster. However, the dependence of the time of the irradiation till complete dehydration at a constant power is heavily dependent on the amount of water and the pre-chosen power of microwaves. In Figure 6a are presented both behaviours:

- Subcritical: at the low power of microwaves the time of irradiation increases with the amount of mass, just as in conventional heat treatment.
- Supercritical: at the high power of microwaves the time or irradiation shortens with the amount of water, which is paradoxical in the conventional heat treatment.

The reversed phenomenon at high-power microwave exposure (more water leading to a shorter time for dehydration), which cannot happen with surface heating methods, happens because more water molecules in the slurry absorb more microwaves. Water molecules can also not absorb microwaves, but if the water molecules are in the "strong part" of the electromagnetic standing wave, they try to align with the rapidly changing outer electromagnetic field, and enhance temperature by rubbing the neighbouring material. The absorbed microwaves are replaced by "new microwaves" in the microwave chamber, i.e., the inverter microwave tries to maintain the chosen output power at a constant level, meaning that there is no decrease in the source power used for the heat treatment. The more molecules that are simultaneously impacted by microwave irradiation, the faster the temperature increases inside the irradiated AAM. Water molecules that were not impacted by the microwave field are used as a heat dissipation media (cooling system), i.e., which is secondary (conventional) heating in the material. When there are more by-microwaves-impacted water molecules (acting as the inner heat sources, i.e., the secondary heating), the temperature rises faster. If there is more water, i.e., more potential heat sources, the system at high microwave power internally heats up faster.

In the presented systems (the size of the samples, compactness of the material, frequency of the microwave were constant), the critical power of microwaves (when all three mixtures need the same time for dehydration) is between 100 and 1000 W. Above the critical power the system is supercritical (1000 W: more water leads to faster dehydration), below the critical power the system is subcritical (100 W: more water leads to slower dehydration).

As shown in Figure 6a, a mixture 100-100, which contains the highest amount of water, dehydrates at 1000 W fastest among all mixtures but just slightly faster than mixture 100-80. The fastest dehydration (for both mixtures with higher amounts of alkali) damages the material the most (Figure 4b and c: the right cross-sections have the most prominent damage) and negatively influences the compressive strength (Figure 5).

The relative changes in mass (δm), volume (δV) and geometrical density (δϱ) of AAMs, shown in Figure 6b, were calculated as the difference between the values on non-irradiated samples and their thermally treated 7-day-old counterparts, normalised to the non-irradiated values. While the mass and geometrical density of all microwave-irradiated mixtures decreased, the volumes increased. Samples containing more liquid alkali (mixture 100-100) expressed the biggest changes, which are attributed to the loss of water (loss of mass) and the creation of more voids/pores/cracks by the removal of a higher amount of liquid from the system. However, the difference in mass, volume and geometrical density between low and high-power microwave treatment is comparable. Therefore, the irradiation with high-power microwaves only notably decreased the time of dehydration, while other changes were just slightly more expressed. The smaller macro-difference in prisms between low and high-power treatment cannot have caused a severe decrease in compressive strength (Figure 5). Therefore, the cause behind the destruction of the mechanical performance of AAMs treated with high-power microwaves is the larger fractures in the micro/mili-structure of the aluminosilicate network (Figure 4). Therefore, dehydration must be performed with low-power microwaves when dehydrating samples still have to express significant mechanical performance.

The thermogravimetric behaviour of GW and 7-day-old mixtures, non-irradiated and irradiated at different powers, is shown in Figure 7. GW does not contain water, therefore there is no mass loss up to 300 °C. At 300 °C GW starts expressing approximately linear mass loss in the remaining measurement area (from 300 to 600 °C; 600 °C was the measurement limit to avoid GW melting which can cause irreversible damage to used crucibles). The thermogravimetric mass loss corresponds to the measured value of gravimetric loss on ignition (~ 6 $m_{\%}$).

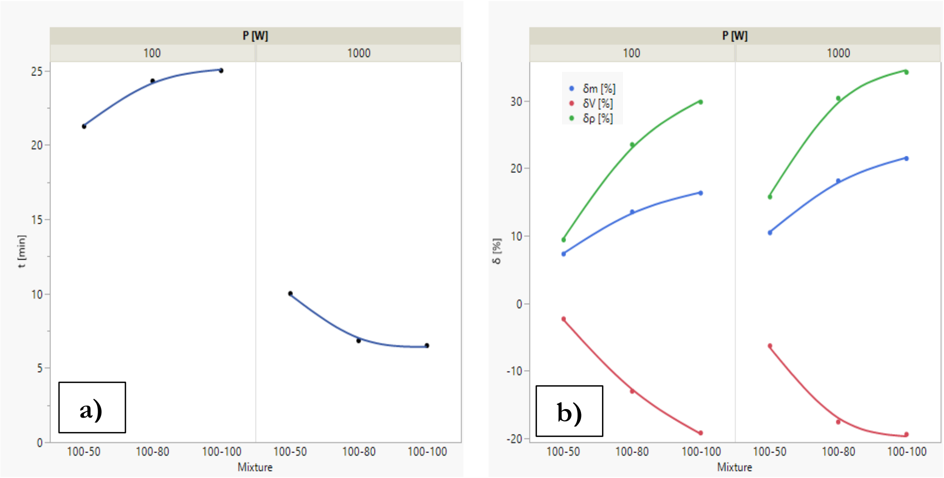

Figure 6: a) Dehydration time and b) changes in mass/volume/density (δm/δV/δϱ) of AAM prisms per irradiation power and mixture.

Source: own.

The linear mass loss is present for all alkali-activated samples in the same range as for GW. Because AAMs and GW have also a comparable mass loss rate in the higher temperature range, this loss is attributed to the thermal degradation of the remaining organic compound which did not react with alkali. Therefore, the aluminosilicate network of all AAMs can be considered thermally stable at least up to 600 °C.

The water loss is represented by the steepest slope in the lowest temperature range (from approximately 50 °C to slightly more than 100 °C). When comparing the AAMs treated in the same manner in the "water range", the AAM with the highest amount of liquid (mixture 100-100) had the highest mass loss and the AAM with the lowest amount of liquid alkali (mixture 100-50) had the lowest mass loss. However, when comparing the behaviour of AAM mixtures that were irradiated with low-power microwaves, high-power microwaves, or were not irradiated, the mass loss in the "water range" is highest for non-irradiated samples and the smallest loss is for the irradiated with high-power microwaves samples. Nonetheless, Figure 7 shows that dehydration was either incomplete or samples should be put into a waterless environment directly after irradiation due to the hygroscopic nature of AAMs made from mineral wools.

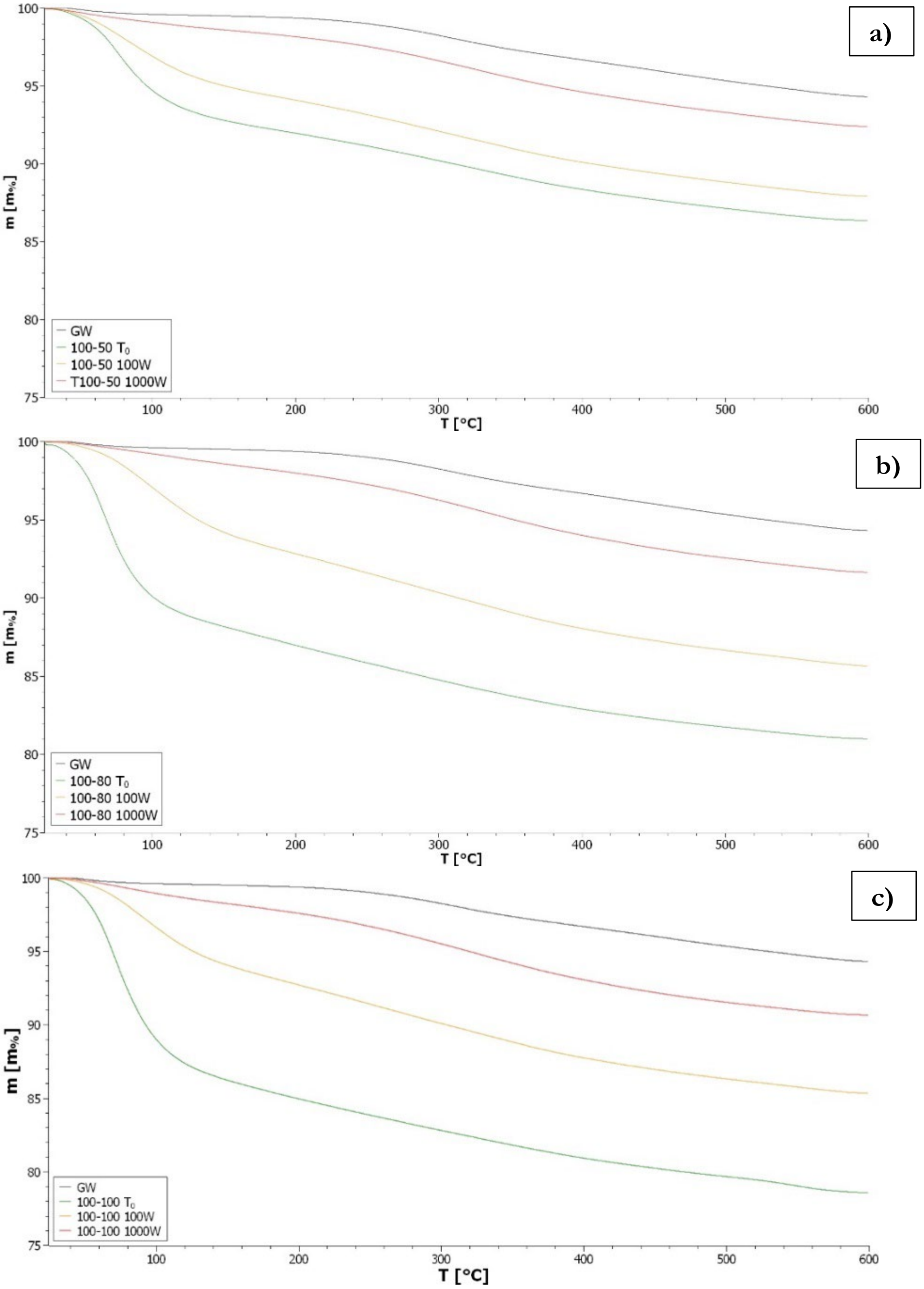

Figure 7: Thermogravimetric behaviour of GW and 7-day-old AAM (dehydrated) mixtures.

Source: own.

In the middle-temperature range (between approximately 100 °C and 300 °C), where there is no mass loss in GW, all AAMs express slightly different mass-loss rates than the rate attributed to the in-alkali-not-reacted organic compound originally present on the surface of the GW. Therefore, the mass loss in the middle range belongs to organic compounds that have at least partially reacted with alkali, which is expressed by the released scent during the reaction and release of colour when AAMs made from GW with organic material on the surface are put into water (video in the supplementary material: Tesovnik and Horvat 2024b).

4 Conclusion

Although all alkali-activated mixtures based on the waste glass wool prepared in this study will never end on the market, namely they will all eventually deteriorate because of the chemical affinity to efflorescence, they suit as an explanation of the non-trivial connection between compressive strength, microwave irradiation and time needed for dehydration of conventionally pre-cured alkali-activated mixtures. In subcritical microwave heating (lower microwave power), where a higher amount of water leads to longer dehydration time, the damage to the inner structure of the material followed by a decrease in compressive strength is smaller when compared to material irradiated in the supercritical conditions (higher microwave power), where more water leads to faster dehydration.

When the compressive strength of solidified materials should be kept as high as possible also after microwave irradiation, the irradiation should be performed in subcritical conditions. However, if the material is dehydrated solely to remove water and test chemical and mineralogical composition, supercritical conditions decrease the time for preparation: water is removed from the system faster and the compressive strength of the material becomes weaker allowing easier pulverisation.

Nonetheless, the finding that in microwave irradiation exists a critical point where the time of irradiation does not depend on the amount of water, can open a new field in research of the synthesis of materials with microwaves.

Acknowledgements

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Data availability status

The original data presented in the study are openly available in the repository Zenodo at https://zenodo.org/records/14515939 with DOI: 10.5281/zenodo.14515939.

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