Possibilities of aerated concrete recyclate and fluid fly ash usage in autoclaved composite production

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Abstract: Waste aerated concrete grit is produced either directly in the aerated concrete plant or subsequently on the building site. The options for handling aerated concrete grit are two. Either it can be used as a construction material, e.g. in the form of light aggregate or as a raw material for the production of aerated concrete.

The subject of this article is the verification of aerated concrete grit as a raw material suitable as an additive in the technology for the production of aerated concrete. The actual solution consisted of the creation of laboratory-autoclaved composites without a porous structure to eliminate the accompanying effects of the technology. The grit was tested in different silica sand substitution ratios (10%, 20%, 30%, 40%) in the reference mixture, based on real production technology. On the basis of physico-mechanical properties and microstructural analysis (XRD, SEM), porous concrete was found to be suitable as a raw material even with 40% sand substitution, where the properties of reference aerated concrete were retained. At doses of 10 and 20%, even the performance benefits of the composite have increased.

Key-Words: Aerated concrete, fluid fly ash, lime, silica sand, autoclaved aerated concrete, autoclaving, X-ray diffraction analysis, tobermorite, recycling.

1 Introduction

Aeratedconcrete is a directly lightened inorganic composite material. Macropores are sealed and can form up to 80% of the total volume. Aerated concrete belongs to a group of directly lightened concretes, it is a special type of concrete whose bulk density is less than 2000 kg·m^{-3}. Specifically, the bulk density of autoclaved aerated concrete is in the range of 150-700 kg.m^{-3}.

The raw material composition for the production of aerated concrete has four basic types of raw materials to which water is added. They are binders, siliceous substances (fillers), gaseous substances and auxiliary raw materials. [1, 2]

The main carrier of strength in aerated concrete is tobermorite. It consists predominantly of platelets (lobular or lace) crystals of approximately 1 μm in size and with a CaO/SiO_2 (C/S) ratio of 0.8-1.0. The chemical formula of tobermorite is $C_5S_6H_5$. The hydrothermal reaction initially forms CSH II and a certain amount of CaO. These are further transformed with the remaining unreacted siliceous feed to CSH I, which has a lower CaO content. Another autoclaving produces tobermorite. [3, 4]

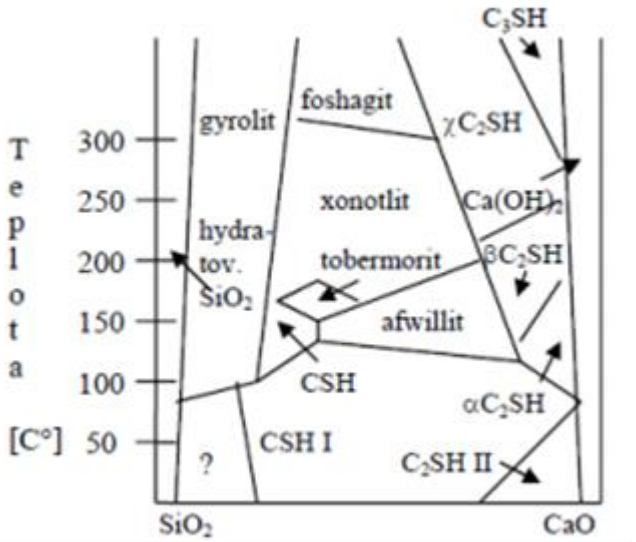
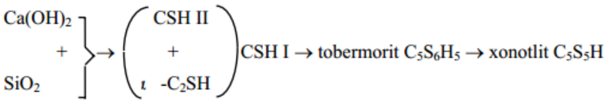


Fig. 1 $CaO – SiO_2 – H_2O$ phase diagram by Bessey

The basic condition for the formation of tobermorite and all CSH phases is the certain molar ratio of CaO/SiO_2 and the temperature of hydrothermal processes.



$$\left.\begin{matrix}Ca(OH)_2\\ +\\ SiO_2\end{matrix}\right\} \rightarrow \left(\begin{matrix}CSH\ II\\ +\\ t\ \text{-}C_2SH\end{matrix}\right) CSH\ I \rightarrow \text{tobermorit } C_5S_6H_5 \rightarrow \text{xonotlit } C_5S_5H$$

Fig. 2 equation of tobermorite formation

Some initial phases, such as the formation of CSH I (when hydrating cement), can occur under normal atmospheric conditions. They are phase-with similar structure as tobermorite but their crystalline grid is not so far developed. Under certain autoclaving temperature and molar ratio a tobermorite is produced which is only a transition phase, because it subsequently crystallizes into xonotlite which is not required due to lower strengths. Therefore, it is important to set the autoclaving cycle to produce the maximum amount of tobermorite and xonotlite not to occur.[5, 6]

The subject of this article is the assessment of the possibility of using porous concrete recyclate as a filler of the autoclaved composite. Subsequently, this filler could be used directly in the production of new aerated concrete, but for the time being, to avoid any undesirable effects, materials without a porous structure were tested. [7, 8]

2 Autoclaved composite analysis

Recycling AAC in concrete or unbound applications may cause problems because of high amounts of leachable sulfate. High pH conditions are necessary to avoid excessive sulfate leaching. Pollution of AAC waste with gypsum impurities is detrimental to sulfate immobilisation.[9, 10]

Heat power plants based on coal combustion produce fluidized bed combustion ash as a by-product of desulfurization by limestone in bed. Fluid ash is not only a by-product. It is due to its pozzolanic activity very useable as a fine additive to concrete in the building industry. Its use has not only environmental aspect, but also an economic one. Therefore, the possibility of adding fluid fly ash to the mixture has also been tested. [8, 11]

2.1 Laboratory methods

Grit originated directly from production, from scraps. Aerated concrete pieces were crushed in two phases. In the first phase by a two-roll crusher. At the second phase by hammer crusher to produce fraction 0-2 mm.

The aerated concrete grit was dried in the drying chamber to the constant weight. The dried grit was then screened to determine the grain size. Subsequently, water absorption, bulk density and crushing resistance tests were performed. The XRD diffraction analysis was used to determine the mineralogical composition of the grit.

Fig. 3 XRD device - PANalatical Empyrean

2.2 Aerated concrete grit

For research, aerated concrete grit was used directly from the crusher of the sand aerated concrete production plant.

The humidity test showed that the porous concrete contained 27.35% water. This is due to the fineness of the grit. Its porous surface results in a large surface area that binds well to water.

The results of grain size determination of porous concrete fraction 0-2 mm are shown in Fig. 4.

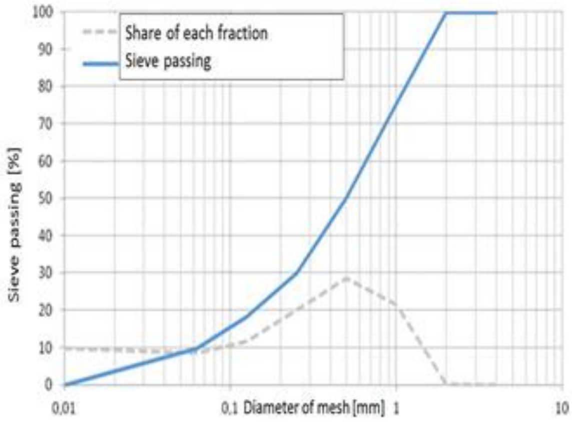


Fig. 4 grain curve of aerated concrete grit fraction 0-2 mm

Results of water absorption, bulk density and crushing resistance determination are shown in table 1.

Table 1 physico-mechanical properties of aerated concrete grit

fraction	Water absorption [% wt.]	Bulk density [kg·m^{-3}]	Crushing resistance [MPa]
0-2 mm	117	620	13

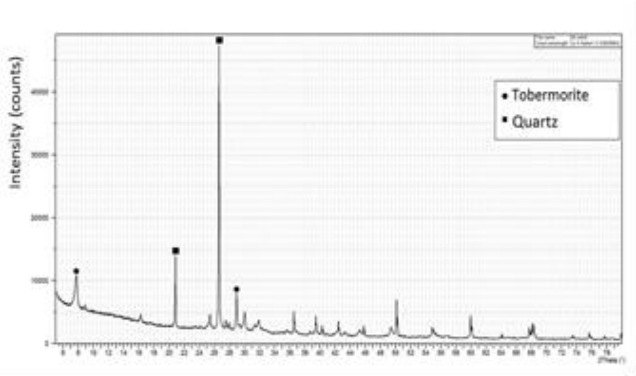


Fig. 5 results of aerated concrete grit XRD analysis

2.3 Analysis of input raw materials

Table 2 shows the percentage of the most important oxides, for aerated concrete grit in shares of dry matter. It is clear from the table that the aerated concrete grit brings both a large amount of SiO2 and a part of CaO into mixture.

Table 2 chemical composition of input raw materials

[%]	Quicklime	Cement	Silica sand	Fluid fly ash	Aerated concrete grit
Free CaO	92	-	-	-	-
Overall CaO	95.62	64.25	0.23	17.67	20.6
SiO_2	-	19.97	92.91	38.05	56.4
Al_2O_3	-	5.4	2.53	24.67	5
Fe_2O_3	-	3.06	0.84	5.82	1.28
SO_3	0.07	2.66	0.02	5.44	2.57 *
Lost on ignition	3	0.89	0.56	3.18	-

As a result of silica sand substitution by aerated concrete grit the C/S ratio is increased, which can have a considerable positive effect on the mechanical and physical properties of the resulting aerated concrete. For a more accurate evaluation, however, it would be necessary to determine whether the SiO_2 contained in the aerated concrete is amorphous or crystalline.

2.4 Mixture design

The basic ratio of each raw material was adapted to the actual raw material composition of aerated concrete in the production plant. Since the aim of the tests was to provide maximum evidence of the influence of feedstock and synthesis of tobermorite, no aluminium powder was used. The proprietary porous structure would significantly influence the results of the experiments. The composition of the reference mixture is shown in table 3.

Tab. 3 composition of reference mixture

	Lime [%]	Cement [%]	Fluid fly ash [%]	Silica sand [%]	Sulfate [%]
Ref.	11.05	16.58	6.91	62.17	3.29

Mixtures with aerated concrete grit fraction 0-2 mm (F) and fraction 0-0.2 mm (MF) were tested.

Aerated concrete grit was tested in both cases as 10% (F10 and MF10), 20% (F20 and MF20), 30% (F30 and MF30) and 40% (F40 and MF40) silica sand substitutions.

2.5 Preparation of the samples

Steel triforms were used to prepare 20x20x100 mm samples. The mixture was filled into half of the steel moulds. The compaction was followed by 2 × 10 impacts (from a height of 5 cm). The molds were fully filled and compacted in the same way.

After 24 hours, the samples were from the moulds and placed into autoclaving capsules.

Fig. 6 autoclaving capsule

Then they were autoclaved for 12 hours at 190°C. Tempering of the chamber to 190°C was carried out for about 20 minutes. The cooling was gradual and lasted 8 hours. In the same way, the autoclaving of the aerated concrete grit itself was carried out in order to verify whether phase changes occur mainly in the tobermorite.

2.6 Analysis of the samples

The bulk density and compressive strength were determined on the samples.

Fig. 7 device for determination of compressive and tensile strength

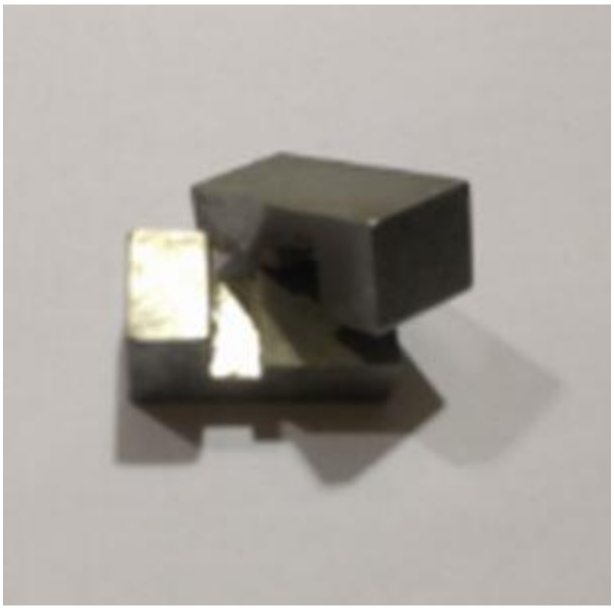


Fig. 8 application parts for determination of compressive strength

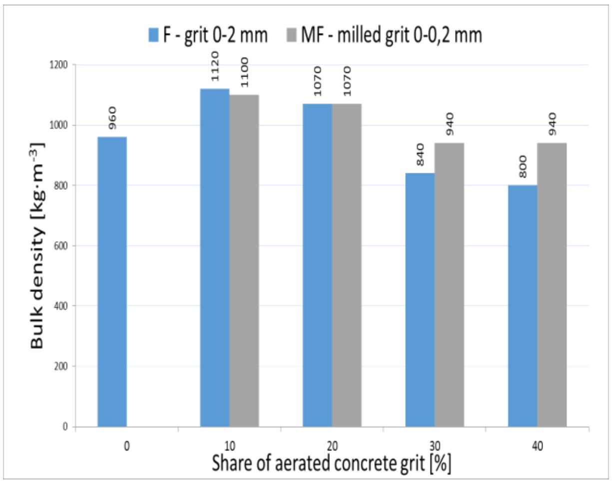


Fig. 9 Bulk density of the tested mixtures

From the results it can be seen that the coarse and milled aerated concrete grit mass in the amount of 10 and 20% increases the bulk density. In the case of 30% and 40% substitution, the bulk density remains practically unchanged for milled grit samples. For coarse grit samples bulk density is reduced even compared to the reference values.

When testing the compressive strength, a significant up to double increase in the values over the reference bodies was observed in the samples with 10% and 20% share of the aerated concrete grit (slightly higher for the coarse-grained bodies).

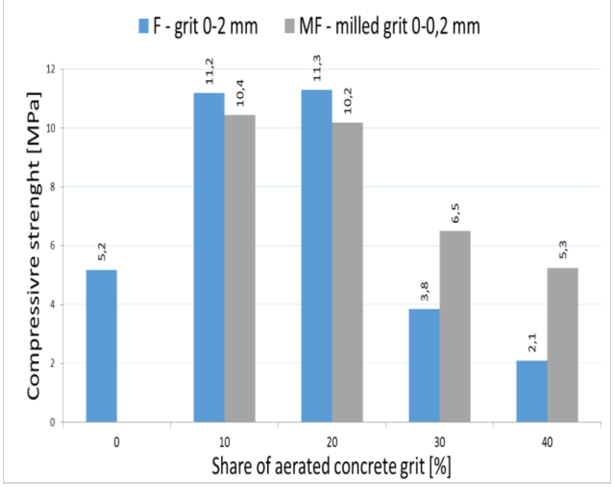


Fig. 10 Compressive strength of tested mixtures

With content 30 and 40% of milled grit, the compressive strength did not change much compared to the reference samples. Higher coarse fractions noticeably reduced compressive strengths.

Subsequently, a composite and autoclaved grit structure analysis was performed by XRD analysis (Figure 11,12 and 13) and SEM microscopy (Figures 14 and 15). Fig. 11 shows the XRD diagrams of each test sample. The reference sample diagram is placed in the correct position. The diagrams of the other samples are gradually shifted by 1 degree to the right in order to show a better change in the peak intensity of the individual minerals.

Fig. 11 SEM Tescan MIRA3 XMU

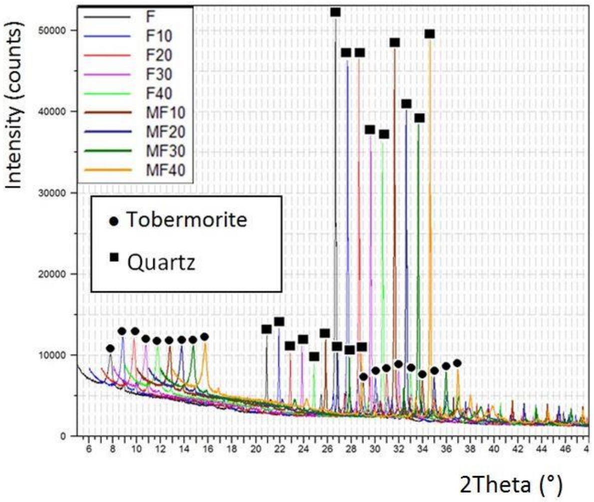


Fig. 12 Combination of roentgenograms of all mixtures

From the combination of roentgenograms of all tested samples an increase of tobermorite content is apparent with share of aerated concrete grit, either coarse or milled. On the other hand, there is a noticeable decrease in the content of quartz due to the replacement of silica sand with aerated concrete grit.

The smallest peak has a reference sample (F). This implies that the admixture of aerated concrete has always had a positive effect on the share of tobermorite. This is due both to the direct admixture of tobermorite-containing material and to the further growth of the crystals of the tobermorite in the grit. In Fig. 13 there are roentgenograms of non-autoclaved and autoclaved grit.

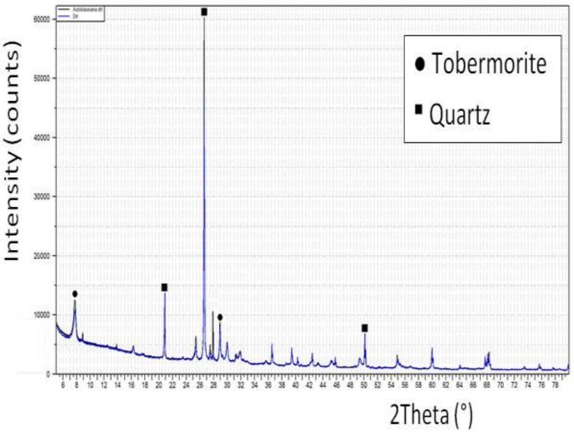


Fig. 13 roentgenograms of both autoclaved and non-autoclaved grit

On Fig. 14 below, for the sake of clarity, the areas of tobermorite in autoclaved and non-autoclaved grit are further approximated.

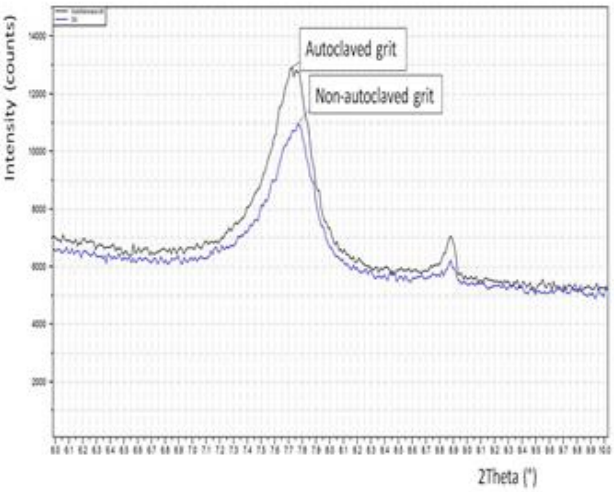


Fig. 14 approximation of the area of tobermorite content in autoclaved and non-autoclaved aerated concrete grit

As it can be seen on the diagram, tobermorite further crystallized through autoclaving and increased its peak.

The majority of the mineralogical composition of aerated concrete grit generally consists of quartz and tobermorite peaks. Xonotlite was not identified. Its occurrence can be expected after 20 hours of hydrothermal reaction. However, even after longer autoclaving, the tobermorite content remains lower than that of quartz. This could result either from a recrystallization or the fact that its solubility was too low for further CSH formation and crystallisation of tobermorite.

For SEM microscopy both aerated concrete grit samples were selected before and after autoclaving (Figure 15), same as samples with the lowest and highest content of the recyclate and reference samples. (Figure 16). In the figures, individual samples are magnified on the left side 1000 times on the right 5000 times.

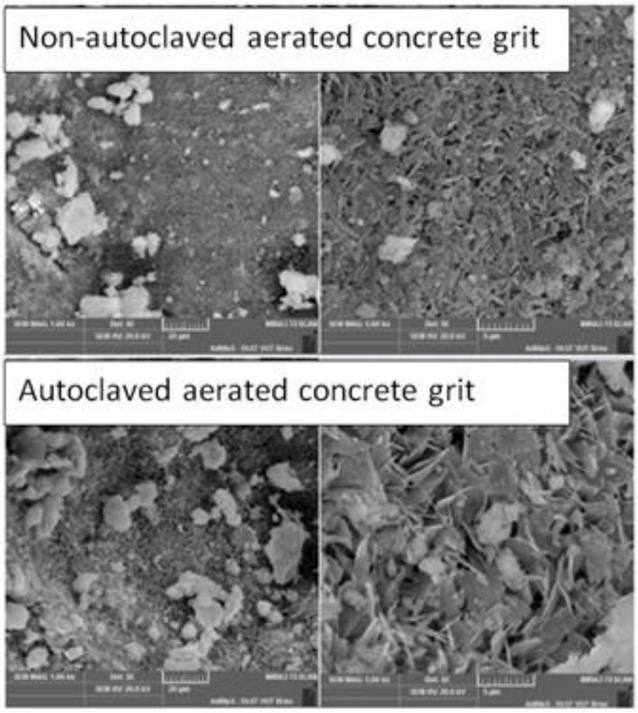


Fig. 15 SEM images of aerated concrete grit fraction 0-2 mm before autoclaving and after autoclaving for 12 hours at 190°C

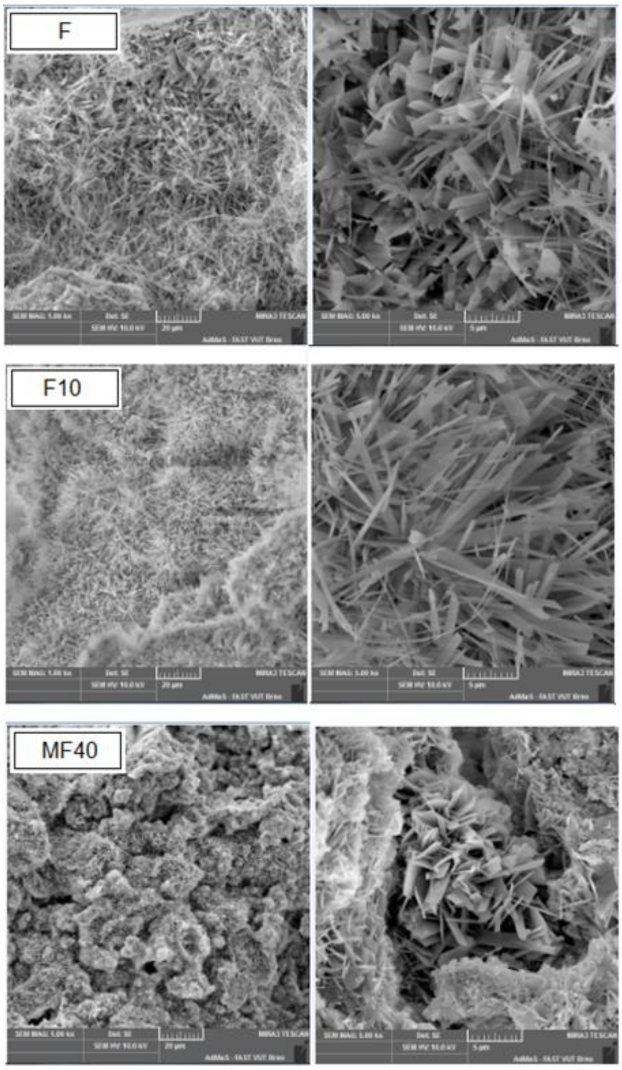


Fig. 16 SEM images of different mixtures

Due to the crushing of the aerated concrete grit, the crystals are damaged. However, it is evident that the crystal appearance has improved after repeated autoclaving of the grit. The crystals look longer and wider than the non-autoclaved grit.

In the case of the test samples F and F10, the whole structure of the tobermoritic phases can be observed. In the case of the MF40 mixture, clumps are already visible around the crystals of the aerated concrete grit.

For better imagination lower are pictured results of microstructure, where there was no fluid fly ash present.

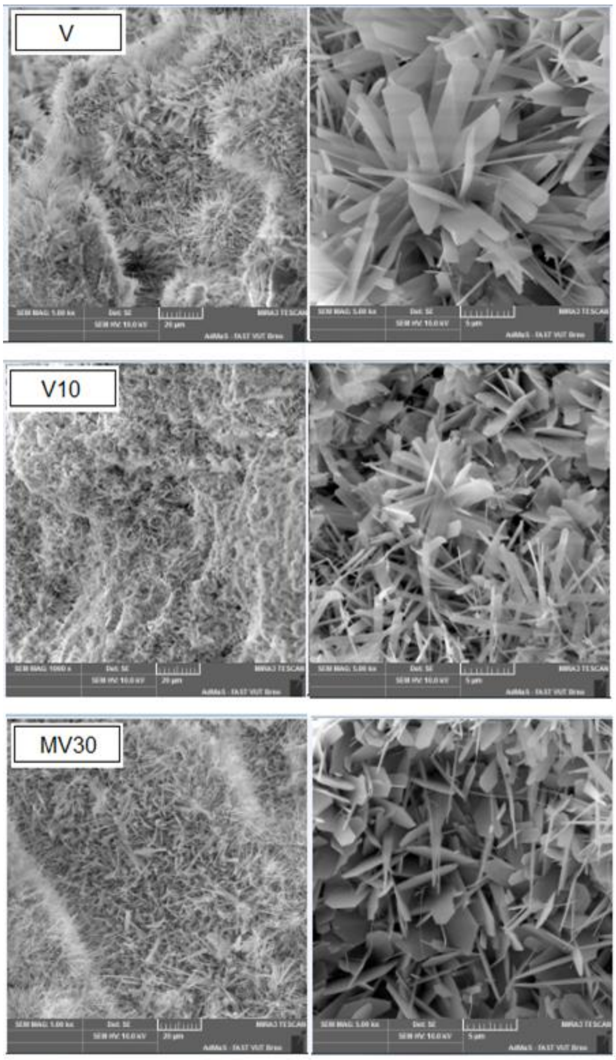


Fig. 17 SEM images of different mixtures without fluid fly ash

3 Conclusion

The results of the research show that replacement of silica sand with aerated concrete grit is possible up to 40%. The physico-mechanical properties at the 30% and 40% replacement by the grit are comparable to the reference mixture. XRD analysis results show that the share of tobermorite in the sample is always higher when the silica sand is replaced by aerated concrete grit. This is confirmed by images from a scanning electron microscope also.

From an economic and ecological point of view, replacing silica sand by aerated concrete grit is very advantageous. In practice, not only that the cost of aerated concrete production will be decreased, but the production process will also become waste-free.

For further research in this area it is possible to recommend verification of various C/S molar ratios, other temperatures of the hydrothermal reaction, verifying the possibility of shortening or extension of isothermal durability and, above all, verification of a selected set of mixtures with a porous sample structure. In the end, it will be necessary to verify the resulting variants in the real technology of the production plant.

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References:

[1] DROCHYTKA, R. *Pórobeton*. vyd. 1. Brno: VUTIUM, 1999, 157 s. ISBN 80- 214-1476-6.
[2] VÝBORNÝ, J. Nauka o materiálech 20 (21): pórobeton. Vyd. 1. Praha: Vydavatelství ČVUT, 1999. ISBN 8001020630.
[3] MATOUŠEK, M. Lehké stavební látky II. 3. opr. vyd. Praha: SNTL, 1985, 130 s.
[4] BAO, T., CHEN, T., WILLE, M-L., CHEN, D., BIAN, J., QING, CH., WU, W., FROST, R. L. Advanced wastewater treatment with autoclaved aerated concrete particles in biological aerated filters. Journal of Water Process Engineering. Volume 9, February 2016, Pages 188–194
[5] LACH, V., DAŇKOVÁ, M.: Mikrostruktura stavebních látek, Nakladatelství VUT Brno, 1991.
[6] BERGMANS, J., NIELSEN, P., SNELLINGS, R., BROOS, K. Recycling of autoclaved aerated concrete in floor screeds: Sulfate leaching reduction by ettringite formation. Construction and Building Materials. Volume 111, 15 May 2016, Pages 9–14
[7] ARAYAPRANEE, W., REMPEL, G. L. Autoclaved Aerated Concrete Waste (AACW): An Alternative Filler Material for the Natural Rubber Industry. Polymer composites. Volume 36, Issue 11 November 2015 Pages 2030–2041
[8] CERNY, V., KOCIANOVA, M., DROCHYTKA, R. Possibilities of Lightweight High Strength Concrete Production from Sintered Fly Ash Aggregate, Procedia Engineering, Volume 195, 2017, Pages 9-16, ISSN 1877-7058,
[9] HLAVÁČ, J. *Základy technologie silikátů: celostátní vysokoškolská příručka pro studenty oboru 27-06-8 Technologie silikátů*. 2. upr. vyd. Praha: Státní nakladatelství technické literatury, 1988, 517 s.
[10] FLEISCHHACKER, J. Vliv technologie výroby popílkového pórobetonu na vznik tobermoritických fází. Brno, 2015/2016. Diplomová práce. Vysoké učení technické v Brně. Fakulta stavební. Vedoucí práce prof. Ing. Rostislav DROCHYTKA, CSc., MBA
[1] POSI, P., TEERACHANWIT, CH., TANUTONG, CH., LIMKAMOLTIP, S., LERTNIMOOLCHAI, S., SATA, V., CHINDAPRASIRT, P. Lightweight geopolymer concrete containing aggregate from recycle lightweight block. Materials & Design. Volume 52, December 2013, Pages 580–586