Usability of waste perlite in the technology of production of autoclaved aerated concrete

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Abstract. The building industry constantly seeks ecological solutions and accents utilization of industrial wastes. For this reason, even less examined alternatives are considered. This is true also for manufacture of autoclaved aerated concrete. This paper addresses usability of waste perlite in the technology of production of autoclaved aerated concrete. Waste perlite has been examined to great extent in particular in the area of cement based composite materials, where its positive influence is known. This leads to the idea of finding out if waste perlite could contribute positively to the technology of autoclaved aerated concrete. In this basic research, four replacement amounts of quartz sand with waste perlite were tested: 10\%, 30\%, 50\% and 100\%. Replacement of 30\% appears to be most promising. At this amount, the compressive strength increased and at the same time, volume weight was reduced by 40\%.

1. Introduction

Autoclaved aerated concrete is ranked among calcium-silicate composites, or so-called aerated silicates. Porous structure gives autoclaved aerated concrete its characteristic properties like low volume weight and low thermal conductivity. This material is also inflammable; therefore, it has a wide range of application as a fireproof layer. Basic raw materials for manufacture of autoclaved aerated concrete are quartz sand, lime, cement, gypsum and aluminium powder \cite{1}\cite{2}. Quartz sand, which has highest proportion of volume of all input materials, is gained from natural resources. However, these resources become gradually exhausted, quality of sand decreases and its price rises. Not only because of these reasons but also to contribute to our environment, there are efforts of replacing primary raw materials at least partially. Ash materials have been commonly used for the technology of manufacture of autoclaved aerated concrete \cite{3}\cite{4}. Ash materials are a source of amorphous silicic oxide, which is more reactive than its natural crystalline form. Secondary raw materials usually contain amorphous silicic oxide, which is advantageous for technology of manufacture of autoclaved aerated concrete. Various materials have been examined in the efforts of finding alternative replacement of silica sand, for example ground packing glass or slag from waste incineration \cite{5}. These researches are only starting, but they are very promising. These alternative raw materials include wastes from production of expanded perlite (WEP).

Perlite is defined as a volcanic glass and therefore it can be found in amorphous form. Its microstructure binds approx. 5\% of water. This water leaves in the form of steam during rapid heating at about 1000 °C. During this relative swift reaction, highly porous structure of perlite is formed (expanded perlite). Thus, the volume of perlite is about 20 times multiplied and its specific surface...
increases, too. Perlite contains mainly SiO₂ and Al₂O₃, further oxides are detected in lower amounts: Na₂O, K₂O, MgO, Fe₂O₃ and CaO. [6][7][8][9][10]. Because of amorphous form of SiO₂ and Al₂O₃, perlite is considered to be pozzolanic material and it seems to be a promising secondary raw material [10][11]. WEP is used in the area of cement based composites and thermo-insulating mortars or various grouting materials. Researchers Pachta et al. [8] examined influence of addition of waste perlite in grouting mixes based on lime binder. It was found that addition of waste perlite considerably increased compressive strength of hydraulic grouting mixes with no negative impact on rheology. Waste perlite was also examining in production of alkali activated slag composites (AACSs). However, WEP was in this case treated by grinding unlike Patcha et al., where WEP was used without any treatment. When used as addition for AACSs, it was found, that increasing amount of addition causes reduction of compressive strength even though no significant changes were observed in the microstructure. Kotwica et al. [10] examined the influence of ground waste perlite as pozzolanic material that could be used as a partial replacement of cement binder. It was found that ground WEP in the amount of 35% of cement binder increased overall strength of cement by 50%. In the past years it was proved that addition of waste perlite in cement composites has positive effect on microstructure and compressive strength but mainly on reduction of hydration heat [7].

As regards use of waste perlite in the technology of autoclaved aerated concrete, it is almost unexplored area. Różycka and coll. [9] researched influence of WEP on properties of AAC, in particular its thermal conductivity. They state that as the amount of replacement with WEP in AAC increases, volume weight is reduced as well as compressive strength. On the other hand, the addition of WEP had a positive impact on the formation of microstructure or development of 1.1 nm tobermorite. They state that this could be caused by morphology of SiO₂ or by high fineness of WEP. It is known that when the specific surface of siliceous component is higher, the reactions are faster, hence tobermorite 1.1nm is formed easily [9]. However, this is not always true. Hartmann [11] defines in his paper the ideal limit of grain size between 0.08 mm and 0.03 mm. In case the grains are smaller than 0.03 mm, no crystallization of 1.1nm tobermorite occurs.

Töbermorite is a calcium hydro-silicate mineral (CSH), which is formed in microstructure of calcium silicate composites during hydrothermal treatment, which is between 150 °C and 220 °C and pressure of vapor 0.8 – 1.6 MPa and length of hardening 3 – 10 hours. These parameters vary based on the type of product. During this hydrothermal treatment, crystalline or amorphous SiO₂ and calcic components are dissolved in water. Thus, CSH phase is formed which subsequently becomes the binding phase and binds individual grains of non-reacted silica, which is usually in excessive amount so that some part of it can serve as filler. Thus, mechanical strength of the product is achieved. [2][3][11][12][13].

2. Raw materials and methods

Basic raw material for manufacture of autoclaved aerated concrete is quartz sand, lime, cement, gypsum and aluminum powder. Basic requirement for unslaked lime is high purity and gradual increase of temperature when hydrating. Reactivity of lime for autoclaved aerated concrete is expressed in time, when the lime paste reaches the temperature of 60 °C. Lime used in the research reached the temperature of 60 °C in 6 minutes and contains only 5% of impurities, hence it fulfills the requirement of high purity and gradual reactivity. Used cement CEM I 52.5 N assures sufficient manipulation strength before autoclaving. Energetic gypsum contains over 90% of dihydrate of calcic sulphate, which supports formation of thixotropic structure of fresh mix and supports formation of new crystalline calcium hydro-silicates (CSH). Aluminum powder is the main agent for formation of the macrostructure. It reacts with calcium hydroxide and gaseous hydrogen is released, forming porous structure of autoclaved aerated concrete. Aluminum powder is selected with respect to the standard CSN 42 0825 Aluminum powders and pastes for autoclaved aerated concrete. Silica sand contains over 92% of crystalline silica oxide and low amount of other oxides.
2.1. Waste perlite as filler
Because of high content of amorphous silica oxide, waste perlite was tested in the research as a replacement of silica sand. Volume weight of WEP was determined to be 300 kg m\(^{-3}\). This low value is considered advantageous and overall volume weight of resulting sample is expected. Determined water absorbing capacity of WEP was 260%. Chemical composition of waste perlite is in Table 1.

| Oxide | CaO | MgO | K\(_2\)O | Al\(_2\)O\(_3\) | P\(_2\)O | Fe\(_2\)O\(_3\) | MnO | Na\(_2\)O | SiO\(_2\) | TiO\(_2\) |
|-------|-----|-----|---------|---------------|--------|--------------|-----|---------|----------|---------|
| [%]   | 1.26| 0.228| 4.56   | 13.1          | <0.023 | 2.02         | 0.057| 2.14    | 74.0     | 0.157   |

2.2. Mix design and preparation
The mix-design corresponds with the mix-design used in real technology of production of autoclaved aerated concrete. To achieve high volume weight, higher amount of aluminum powder is used. WEP was used as a substitution of silica sand by 10% 30% 50% and 100%. Dosing of WEP was by volume. Water-cement ratio was constant for all mix-designs. Reference mix-design is stated in Table 2.

| Raw materials     | REF [%] |
|-------------------|---------|
| Lime              | 9       |
| Cement            | 14      |
| Gypsum            | 3       |
| Aluminum powder   | 0.137   |
| Quartz            | 75      |

Preparation consists of several stages. First, fresh mix is prepared. Lime and cement are thoroughly mixed in homogenizer. Aluminum powder is mixed with detergent and water. Sand, energo-gypsum and waste perlite are mixed in a mixer and together with water. Then, lime is added in the mixer and after mixing, suspension of aluminum powder. Time of mixing is 2 minutes from adding reactive components in water. Prepared fresh mix was poured into warmed up forms with dimension 100x100x100 mm and then the forms were placed in a dryer at 40 °C so that aeration could take place. At this stage, it is important to ensure conditions remain unchanged. After aeration and setting of the fresh mix, forms are removed, and samples placed in the autoclave, where final hardening of the mix takes place. Development of temperature is graphically presented in Figure 1.
2.3. Physical-mechanical properties
Compressive strength was determined on dried samples at (105±5) °C. The test was carried out in accordance with the standard CSN EN 679 Determination of strength of compressive strength of autoclaved concrete. Test specimen is placed in the press in the direction perpendicular to the growth of the material and is loaded at constant rate (0.1±0.05) MPa/s. Size of tested specimens was 100×100×100 mm. Volume weight was determined in accordance with the standard CSN EN 678 Determination of volume weight of dry autoclaved aerated concrete. Macrostructure was determined with digital optical microscope Keyence VHX-950F.

2.4. Preparation of samples for examination of microstructure
2.4.1. X-ray diffraction
Out of all samples, cores were prepared, which were ground to max. 0.1 mm by vibration grinder for the study of microstructure during the first stage of preparation of samples. Ground samples were then ground in isopropanol suspension with addition of comparing standard in the MC Microne grinder to the size <0.02 mm. The suspension was then dried and the powder was applied on a carrier of the Empyrean Panalitical apparatus to carry out quantitative XRD analysis.

2.4.2. Scanning electron microscope
To study microstructure with scanning electron microscope, representative fragments with size 5x5x5 mm were selected. The examination was done with microscope TESCAN MIRA 3 XMU.

3. Results
3.1. Compressive strength, volume weight and slump
Compression strength, bulk density and flow test of samples with WEP replacement are displayed in Figure 2. Based on determination of compressive strength of samples, dependence of compressive strength on the amount of WEP substituting silica sand was noted. The dependence is not linear. Compressive strength decreases more from 30% replacement with WEP. On the other side, samples with 10% and 30% of replaced amount showed compressive strength about 18% higher than reference samples. Probably, formation of CSH or 1.1 nm tobermorite was supported because of high specific surface of WEP and content of amorphous SiO₂.
Figure 2. The charts of compression strength, bulk density and flow test of samples with WEP replacement.

When the substitution with waste perlite is used, the volume weight compared to the reference sample significantly drops. As assumed, the lowest determined volume weight was that of 100% replacement of silica sand with WEP – the value decreased by more than 50%. Volume weight of samples with 10% and 30% replacement decreased by nearly 40%. Volume weight of sample with 50% replacement increased because of collapse of the porous structure during aeration. Reduction of volume weight is caused by very low volume weight of WEP.

3.2. Porous structure

Porous structure of samples is clearly visible in the Figure 3. The first image shows porous structure of reference sample and detailed view of a selected pore. The diameter of the pores is approximately 2 mm. After 10% replacement, slight increase of pore size and strengthening of inter-pore walls compared to reference sample, can be observed. This aspect causes increase of compressive strength compared to the reference sample. This trend continues in the sample with 30% replacement. Increased pore size and strengthening of inter-pore walls compared to reference and 10% replacement sample can be observed. During aeration of fresh mix of sample with 50% replacement, the structure collapsed. For this reason, the sample shows irregular porous structure with prevailing non-aerated matrix. Sample with 100% replacement shows extremely large irregular pores, which were created probably because of high slump value. The mix was not sufficiently viscous and individual pores agglomerated.
3.3. Microstructure

According to X-ray diffraction analysis, it was found that samples with 10%, 30%, 50% substitution and reference sample contained the same basic minerals, quartz, portlandite and tobermorite. Crystalline SiO$_2$ entered the sample with quartz sand, and therefore its intensity and amount decreased with increase of sand replacement with WEP. As for tobermorite, the dependence of intensity and quantity on substitution has not been confirmed here. The intensity of the tobermorite diffraction line is almost unchanged at 10% and 30% replacement of the silica sand with WEP. The same was seen in the SEM images, where there was no difference between 10% and 30% substitution and therefore no change in the morphology of tobermorite. Conversely, with 50% replacement, the most tobermorite was detected.

According to SEM images (Figure 4), it is evident that the morphology of tobermorite was different with this replacement. In the reference sample, tobermorite was found in the form of long slats, in the sample with 10% and 30% substitution, tobermorite was found in the form of thinner and shorter slats, and in the sample with 50% substitution, tobermorite was detected in the morphology of the low leafs. This is probably due to the higher proportion of amorphous SiO$_2$ from WEP. It has been suggested that with 100% substitution of silica sand with WEP, 1.1 nm tobermorite should be detected in the highest amount with respect to the reactive form of SiO$_2$ in WEP. However, the results showed that 1.1 nm tobermorite is not present. This is also likely to cause very low compressive strength of the samples.

Figure 3. The structure of samples with WEP replacement.
4. Conclusion
The research focused on possibilities of using WEP in the technology of autoclaved aerated concrete. The aim was achieving lower volume weight of autoclaved aerated concrete by using WEP. Volume weight was considerably reduced as early as with 10% replacement of sand with waste perlite. In spite of reduced volume weight, compressive strength of samples was higher up to the amount of 30% replacement. This trend can be explained by increase of thickness of walls between pores. At 50% replacement amount, thixotropic structure was not formed and after aeration, the porous structure collapsed.

Sample with 100% replacement showed very irregular porous structure with extremely large pores. However, the volume weight of the sample was lower by 50% in spite of unsuitable structure. For this reason, it is not always necessary to create porous structure and hence using expansion to decrease the volume weight. For further research of 100% replacement of silica sand with waste perlite it is necessary to adjust water cement ratio and amount of aluminum powder.

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