Application of brick grind dust in systems based on Portland cement.

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Abstract. This paper deals with possibilities of application brick-grind-dust which is a secondary product from manufacturing of grinded bricks. This product is usually partially returned to brick manufacturing or buried on a land field site. The basic idea of using this material in systems based on cement comes from their mineral composition, origin and particle size. These materials are potentially pozzolan active, which is interesting for use in hydraulic binders. Also red colour of brick dust can be interesting for visual application. First physical and chemical properties of brick-grind-dust was studied, then binders based on the material and Portland cement were prepared. Mechanical properties of prepared binders were studied over time. Hydration of the selected binders was analysed with isothermal calorimetry.

1. Introduction
In recent years, the brick industry has been focusing on the production of ground bricks with precise dimension and high level of flatness. These bricks are used for rapid and accurate bricklaying to thin layer of special mortar or polyurethane foam. Secondary product of manufacturing these bricks is a fine dust with some fragments from bricks. This material is partially reused in the production of bricks, but the remaining production is landfill.

Production of brick is based on drying and burning brick clay, soils and other components in tunnel furnace about 800°C exceptionally up to 1000°C. Brick materials typically contain relatively low and varying amounts of clay minerals (i.e. kaolinite, illite, montmorillonite, chlorite and their mixed structures). The predominant fraction is composed of soil containing silica, mica and rock fragments including carbonates. There is also an iron oxide pigment [1].

Dehydroxylation of clay minerals at these temperatures made these materials potentially pozzolan active. The American Standard ASTM C 125 defines pozzolans as silicate and alumina materials which in themselves bear little or no binding properties. However, in finely ground form, in the presence of water, they react with calcium hydroxide at ambient temperature to produce compounds having binding properties. Pozzolans are chemically referred to as silicates or alumino-silicate inorganic substances, mostly without or with little lime [2].

Pozzolan activity gives information about amount of calcium hydroxide which can react with some pozzolan. Like pozzolan activity is so defined reaction velocity between these substances. Both parameters are depended on the type of pozzolan, surface area, chemical and phase composition (Amorphous content), CaO/pozzolan ratio and so with curing parameters [3].
Hydration products formed by pozzolan activity are very close to product of hydration calcium-silicate or calcium-aluminate phases which is called C-S-H gel and C-A-H gel. Pozzolan reaction can be written in most simply way like schema: puzzolan + CH + H → C-S-H*.

There is C-S-H* gel marked with the star like a secondary C-S-H gel in spite of primary C-S-H gel from hydration of calcium-silicates from Portland cement. Pozzolan reaction takes much longer than hydration of clinker minerals. It is reason for less porosity and more compact structure for pozzolan cement [4].

2. Materials and methods

Brick-grind-dust was produced by manufacturing bricks called Family in plant Hevlin (HELUZ). Mineralogical composition was analyzed by powder XRD (Empyrean, Panalytical) with Rietveld analysis. For pozzolan reaction is most important amorphous phase content, which is 28%. Others phases contents are said in Table 1.

Table 1. Phase composition of brick rind dust.

| Phase     | Amorphous | Quartz | Augite | Albite | Muscovite | Orthoclase | Akermanite | Hematite |
|-----------|-----------|--------|--------|--------|-----------|------------|------------|----------|
| (%)       | 28.4      | 30.3   | 5.6    | 17.1   | 11.3      | 2.0        | 4.3        | 0.9      |

Particle size distribution was analyzed by six sieve 1.4 mm, 0.8 mm, 0.4 mm, 0.2 mm, 0.105 mm, 0.063 mm and results are shown in Table 2. Fraction less than 0.063 mm was analyzed by laser analysis of particle size (HELOS, Sympatec). Particle size distribution of Portland cement CEM I 42.5 R plant Mokrá was analyzed to by HELOS for comparison with particle size distribution of brick grind dust. Cement has a much smaller grain size scatter, but D90 of both samples are in this very close. Modus and D50 of dust are approximately half than cement. Smaller particles in case of brick dust can apply the filler effect.

Table 2. Sieve analysis of brick-grind-dust.

| Fraction (mm) | <0.063 | 0.063–0.105 | 0.105–0.2 | 0.2–0.4 | 0.4–0.8 | 0.8–1.4 | >1.4 |
|---------------|--------|--------------|------------|----------|---------|---------|------|
| (%)           | 66.8   | 14.2         | 4.8        | 3.8      | 5.1     | 3.6     | 1.6  |

Figure 1. Particle size of brick-grind-dust.

Figure 2. Particle size of CEM I 42.5 R.
Table 3. Particle size parameters.

| Sample                | D$_{50}$ (μm) | D$_{90}$ (μm) | Modus (μm) |
|-----------------------|---------------|---------------|------------|
| Brick-grind-dust      | 4.10          | 33.47         | 11.55      |
| CEM I 42.5 R          | 10.35         | 36.56         | 20.51      |

Mechanical properties were tested on three 20 × 20 × 100 mm test specimens from each sample and time on the equipment for testing of building materials (DESTTEST, BETONSYSTEM). The average values of compressive strength are shown in the graph in the Figure 3. Composition of prepared samples is shown in Table 4. Water/cement ratio is marked like W/C and W/S is water/solid components ratio. From the samples for analyzing mechanical properties were, after 28 days, a representative of 5 analyzed to compare the phase composition by XRD.

Table 4. Composition of prepared samples.

| Sample                | A   | B   | C   | D   | E   | F   | G   | H   | I   | J   |
|-----------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Brick grind dust [g]   | 0   | 100 | 200 | 300 | 400 | 500 | 600 | 700 | 800 | 900 |
| CEM I 42.5 R [g]       | 1000| 900 | 800 | 700 | 600 | 500 | 400 | 300 | 200 | 100 |
| H$_2$O [g]             | 333 | 334 | 347 | 353 | 360 | 370 | 378 | 395 | 413 | 424 |
| W/C                   | 0.33| 0.37| 0.43| 0.50| 0.60| 0.74| 0.95| 1.32| 2.07| 4.24|
| W/S                   | 0.33| 0.33| 0.35| 0.35| 0.36| 0.37| 0.38| 0.40| 0.41| 0.42|

For the study of hydration processes using isothermal calorimetry (TAM Air, TA Instruments), 4 g samples were prepared. Samples were the same composition as samples for testing mechanical properties or XRD analysis, but amount of water was increased by 20% in all blends to improve homogenization of the system. Increasing the water factor by such an amount would affect the mechanical properties, but the change in the chemistry of hydration is not expected. Reaction should be only accelerated and it does not affect for the comparison of the samples.

Samples were mixed with water for 1 minute outside the calorimeter. Subsequently, together with the reference (distilled water), they were inserted into the calorimeter. The temperature was set at 40°C, which would be sufficient to capture all significant hydration processes within 100 hours. Since the measurement was at 40°C, the start of the recording (about 15 minutes) is subject to considerable heat flow, which is caused by the temperature of the samples from 25 to 40°C. For this reason, the first peak does not represent chemical processes, so it will not be discussed in more detail.

3. Results and discussion
The prepared samples were all able to handling after 24 hours, but samples I, J exhibited zero compressive strengths. Generally, the compressive strength (Figure 3) can be read by the trend of a gradual increase in strengths, even after 28 days. It may be a proof of the pozzolan activity of the brickwork. But after 180 days is here little decrease for all samples. Trend of increasing compressive strengths is well visible after 60 days in blends with brick ash contents above 40%. At 40% brick ash (E), the compressive strengths after 28 days were 60–70 MPa scattered, and with each 10% brick ash decreased the strength by approximately 10 MPa. So the system was probably overload with brick dust. Sample J containing 10% of cement showed a compressive strength of more than 13 MPa after 28 days, and sample I with 20% cement showed strength of 26 MPa. These values are for some applications fully sufficient and economically interesting. The problem could be unstable strength after 24 hours, which could complicate the manipulation of products. The solution could be found in technological parameters such as prolonged aging time before deformation, increased curing temperature, or the use of a suitable solidification accelerator.
The bulk weights of the prepared bodies (Figure 4) stored in the humid deposit decreased for the first 10 days, than were then approximately constant. As the brick ash content increased, the final bulk density from 1.95 g·cm$^{-3}$ to the body from pure cement to 1.43 g·cm$^{-3}$ in the 90% brick ash was reduced.

![Figure 3](image1.png)  
**Figure 3.** Graph of development compressive strength in time.

![Figure 4](image2.png)  
**Figure 4.** Graph of development bulk density in time.

![Figure 5](image3.png)  
**Figure 5.** Colour change depending on cement/brick dust ratio.

![Figure 6](image4.png)  
**Figure 6.** Comparison of diffractograms for selected samples.

The color of the samples (Figure 5) was changing with increasing brick ash content from gray to brick-orange. The color of the bodies and the variability of their strengths could be attractive for visual applications or concrete outdoor products.

In the Figure 6 are the individual diffractograms of samples A, B, C, F, I, J showed. The arrows indicate the phases of clinker minerals (C$_3$S, C$_2$S, C$_{4}$AF), Portlandite (CH) as the product of Portland cement hydration and the potential reactant for pozzolan reaction and quartz as one of the most striking phase occurring in the brick dust. Generally quartz is believed to be unreactive or very few. With a simple comparison of the diffractograms, the peak intensity of the cements and the Portlandite hydration product decrease with increasing brick content. It is observed due to the dilution of the cement. The increase in intensity in same way can be observed for the peak representing quartz. This is the content which increases as the brick ash content increases. Samples contains many phases of both...
cement and brick. This complicates their phase analysis, however, no new crystalline phases of react product have been proven. The pozzolan reaction is based on the reaction of amorphous phases of silicates or alumino-silicates with Ca(OH)$_2$ to form the C-S-H or C-S-(A)-H gel. Although the intensity of the Portlandite diffraction decreases with the brick ash content, it is rather due to dilution of the cement with the brick ash rather than its consumption for the formation of the C-S-H gel. In order to verify the pozzolan reaction and to understand the hydration of these mixtures, the following experiment, isothermal calorimetry, was proposed.

![Figure 7. Calorimetric curves of samples A, B, C, F, I, J.](image)

![Figure 8. Graph of total hydration heat of samples A, B, C, F, I, J.](image)

**Table 5.** Table of approximate times from the calorimetric curve and total hydration heat.

| Sample | End of induction period (h) | Main hydration peak (h) | Sulphate depletion peak (h) | Ettringite conversion (h) | Heat (J/g) |
|--------|-----------------------------|-------------------------|-----------------------------|--------------------------|------------|
| A      | 2.0                         | 6.5                     | 11.5                        | 35                       | 317        |
| B      | 2.1                         | 6.5                     | 10.8                        | 36                       | 307        |
| C      | 2.1                         | 6.4                     | 10.8                        | 46                       | 276        |
| F      | 2.3                         | 6.4                     | 9.4                         | -                        | 198        |
| I      | 3.0                         | -                       | 7.6                         | -                        | 123        |
| J      | 3.5                         | -                       | 7.4                         | 20                       | 84         |

In the Table 5 are the approximate times of individual hydration steps subtracted from the calorimetric curve (Figure 7) and total hydration heat listed. The induction period of the cement without brick dust lasts for one hour and increases with the addition of brick dust for up to 2.5 hours. It is a sign of prolonging workability, which is related to the slower start-up of the strengths.

The effect of brick ash addition on the main hydration position over time is not significant. The maximum heat flux is reached for samples up to 50% (inclusive) of brick ash in about 6.5 hours. For samples with brick ash content higher than 50%, the main hydration peak overlaps with the sulphate depletion peak, so it is not possible to determine exactly its position.

Brick dust does not affect the main hydration peak with only a dilution effect because the drop in heat flux is not directly proportional to the loss of cement in the samples. Due to particle size of brick ash, the effect of hydration is influenced by the "filler effect". The explanation of the effect filler is based on two mechanisms. In the first instance, a cement containing filler like inert powder facilitates hydration, since, while maintaining the W/C ratio, the inert material does not engage in hydration. It does not form
hydration products, and there is more room for hydration of the clinker minerals. The second mechanism is to introduce a new surface with an inert material on which hydration products can form. When it is an inert material of quartz, an amorphous layer may be formed on the surface by grinding, which may additionally be a pozzolan active [5].

The most significant change in the calorimetric curve due to the addition of brick ash is observed on the third hydration peak representing sulfate depletion [6]. This is also possible in the area where diffusion-controlled chemical reactions revert to the heat flux directive to positive values. This peak appears on a calorimetric curve with increasing brick ash content still earlier (11.5 hours for sample A and 7.4 hours for sample J). Gradually, it moves from the diffusion-controlled area to the C-S-H gel surface-driven region [7]. There is a rapid increase in the intensity of this peak. The intensity is strongly influenced by concentration of sulphate and aluminate ions over time. The main source of sulphate ions is gypsum, which is added to the cement. The ratio between clinker minerals and gypsum in the samples is therefore constant. It is expected that peak shifts cause alumina ions to so-called "active alumina" from brick ash and aluminate ions are introduced into the solution by the influence of the pozzolan reaction. For optimum hydration, this active alumina should be compensated for by addition of gypsum [8]. Optimal gypsum content in cement binders is such that sulphate depletion occurs several hours after the main hydration peak. If the peak is present before the main hydration peak, the strength decreases due to the formation of the defective C-S-H gel. If it is found for more than a few hours, the cement dilution effect by gypsum [9]. In this case, the condition of the optimal gypsum content would be samples A, B, C, sample F is in the transition region, and Samples I and J suffer from a considerable deficiency of gypsum.

The total heat (Figure 8) is almost constant after 90 hours at 40°C, so it is believed that hydration is controlled by diffusion and is very slow and total heat can be compared. Assuming that the brick is only non-reactive filler, the 10% decrease in brick ash should be the one-tenth of the released heat by about 31 J/g. However, the difference is only 10 J/g. Likewise, for sample F with a brick ash content of 50%, the hydration heat should be approximately 158 J/g and in fact is 198 J/g. Increasing the hydration heat is therefore a proof of the ongoing pozzolan reaction. Its thermal expression is not significantly affected by the thermal flow over time. So the reaction is going on for a long time. The benefit of brick dust for Portland cement bonding systems is probably a combination of pozzolan reaction and filler effect.

4. Conclusion

Influence of adding brick-grind-dust was showed by the growth of long-term compressive strengths, in case samples with addition of brick dust up to 40%. So decreasing of compressive strengths, in case of samples with more content of brick dust, was not directly proportional to the loss of cement, which was attributed to pozzolan activity.

X-ray diffraction analysis of the prepared samples did not show presence of a new crystalline phase of hydration products of cement and brick, which encouraged the likelihood of the formation of amorphous hydration products of this pozzolan.

The basic comparison of the total hydration heat, confirmed that a pozzolan reaction occurred because the decrease of the hydration heat is not directly proportional to the reduction of the cement content. Another mechanism influencing the positive properties of these alternative binders, resulting from the isothermal calorimetry and from the particle size of brick ash, is filler effect. This theory should be confirmed by other experiments.

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References

[1] Pytlík P 1995 Cihlářství (Brno: CERM)
[2] ASTM C125-13 2007 Standard Terminology Relating to Concrete and Concrete Aggregates
(American Society for Testing and Materials)

[3] Moropoulou A, Bakolas A and Aggelakopoulou E 2004 Evaluation of pozzolanic activity of natural and artificial pozzolans by thermal analysis Thermochim. Acta 420 pp 135–40

[4] CollepardiM 2009 Moderní beton (Praha: Ic ČKAIT)

[5] Lothenbach B, Scrivener K and Hooton R D 2011 The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes Cem. Concr. Res. 41 pp 217–29

[6] Lerch W 1946 Am. Soc. Test. Mater. 46 pp 1252–97

[7] Bullard J W, Jennings H, Livingat0n A, Jennings M, Nonat A, Sherer G, Svheweitzer J, Scrivener K and Thomas J 2011 Mechanisms of cement hydration Cem. Concr. Res. 41 pp 1208–23

[8] Novotný R, Bartoníčková E, Švec J and Mončeková M 2016 Influence of Active Alumina on the Hydration Process of Portland Cement Procedia Eng. 151 pp 80–6