Utilization of waste quarry washing sludge for the production of dry pressed ceramic bodies

Radomir SOKOLAR1 and Simona GRYGAROVA
Brno University of Technology, Faculty of Civil Engineering, Institute of Technology of Building Materials and Components, Veveri 331/95, 602 00 Brno, Czech Republic

Sludge from the washing process of natural aggregate was used as the raw material for the production of dry pressed ceramic bodies. After fast firing at temperatures of 1000–1090°C with a 10-min soaking time, bodies based on waste sludge show considerably lower porosity (water absorption; bulk density), higher bending strength and better pore size distribution with respect to frost resistance than similar compared bodies based on spray granulate used for the production of dry pressed ceramic tiles.

©2013 The Ceramic Society of Japan. All rights reserved.

Key-words : Washing sludge, Dry pressed body, Fast firing, Porosity

Natural aggregate is often washed–clay and dust grains are removed by water. A considerable quantity of waste washing sludge is produced all over the world. The growing trend of using waste for ceramic technology has given rise to many studies and specialized analyses, mostly concerning using granite sludges or dusts, marble wastes or their combination in connection with local clays or other wastes, for example fly ash. Generally all tested stone sludges or dusts in raw material mixtures decreased the porosity of fired ceramic bodies or decreased the firing temperature—they are used as fluxes with low vitrification temperature.

The note presents the sludge (“SV”) which originates during the washing process of crushed aggregate (Moravian Greywacke, fraction 0–4 mm), and is used as a raw material for the production of dry pressed ceramic bodies. The quarry produces on average 30,000 tonnes of the sludge per year. The objective of the research was to assess the properties of the green and the fired bodies produced from the sludge, depending on the firing temperature, in comparison to the parameters of ceramic bodies produced from industrially prepared spray granulate (“SG”) intended for the production of dry pressed wall ceramic tiles with water absorption about 15%.

The granulometry of both compared raw materials was analysed according to residue (in mass %) on a sieve with a mesh diameter of 63 µm-SG 3.5%, SV 2.1%. This result is acceptable for the production of dry pressed ceramic tiles without further milling of the sludge.

The chemical and mineralogical composition of both compared raw materials (SG, SV) is very similar. The only remarkable difference is in the content of dolomite, which increases the content of CaO in the spray granulate (4.27%), in comparison to the sludge (0.88%) in question. The comparable content of MgO (SG 2.84%, SV 2.06%) in both raw materials is given by the content of dolomite in the granulate and brucite Mg(OH)2 in the sludge. The higher content of Na2O in the assessed sludge (3.27%) is attributed to the presence of sodium feldspar (albite). As well as kaolinite, the sludge also contains other types of clay minerals (illite and montmorillonite), in comparison to the spray granulate which is based on kaolinite only. The markedly higher content of Fe2O3 in the sludge (5.36%) presumes creation of coloured ceramic bodies after firing, and may bring problems during fast firing of a ceramic body (bloating).

The pressing granulate SG was prepared by pressing of the moistened sludge (6%) through a sieve of 1.0 mm mesh size. The granulate was left for 48 h for homogenization in a laboratory homogenizer, to obtain a homogenous mixture. The spray granulate was used as reference material and it was produced by the relevant industrial procedure. Test samples with dimensions 100 × 50 × 10 mm3 were unaxially pressed at 20 MPa with a 30-s soaking time at maximum pressing pressure. The green bodies were fired in an electric laboratory furnace at temperatures of 1000, 1030, 1060 and 1090°C with a heating rate of 10°C/min and 10 min’ soaking time at the maximum temperature. The subsequent cooling proceeded spontaneously. Relative expansion during the firing of dry pressed green samples was measured by thermadiatometric analysis according to American Standard ASTM E 228-95. Properties of the fired bodies were tested according to the European Testing Standard EN ISO 10545 (water absorption WA, bulk density ρb, bending strength σ). The pore size distribution was defined by means of the method of high-pressure mercury porosimetry (Thermo Finnigan Pascal 140/240).

The bending strength of the dry green bodies made from the reference spray granulate is a higher (2.6 MPa) than the green body made of sludge (1.8 MPa). The technology of production in this case requires a bending strength within 1.5–2.0 MPa, depending on the size format of the tile.

In comparison to the ceramic bodies made of the reference mixture SG, the sludge-based ceramic bodies SV show lower porosity at all tested firing temperatures. The porosity was assessed according to water absorption (Fig. 1) and bulk density (Fig. 2). The higher firing temperature of ceramic bodies based on the sludge, the more pronounced the sintering of the ceramic bodies, due to a much more distinct decrease in their water absorption (Fig. 1). An optimum for the respective production of dry pressed ceramic bodies (for example wall ceramic tiles),
also from the economic point of view, seems to be a firing temperature of 1000°C, when the firing shrinkage of body SV is comparable to the reference body SG (Fig. 3), at a comparable bending strength (Fig. 2) - 19.8 MPa (SV) or 20.2 MPa (SG), respectively.

A ceramic body based on sludge (SV) has higher total firing shrinkage compared with the reference body (SG) according to the thermal dilatometric curves (Fig. 3). Within the temperature interval of about 500 to 850°C, the sludge-based ceramic body shows a higher volume increase due to decomposition of illite and mica structures in the raw material. Both compared raw materials start to shrink from about 900°C. However, for the reference samples SG, a temperature of 950°C means a decrease in shrinkage during firing due to the creation of the mineral anorthite\(^{17}\) (Fig. 4), the creation of which depends on the content of calcium oxide (CaO) in the ceramic body. The creation of anorthite is accompanied by the volume increase of the ceramic body and the firing shrinkage is eliminated in this way during the sintering process. Ceramic bodies SG have therefore only 0.9% of firing shrinkage (Fig. 3) after the firing at 1090°C without the soaking time at the maximum temperature during the thermodilatometric analysis. The firing shrinkage of the SV sample is about 2.6% (Fig. 3) because the sludge SV contains only minimum of CaO for the creation of anorthite and higher content of fluxing oxides (Fe\(_2\)O\(_3\) and Na\(_2\)O) supports the sintering process. The firing shrinkage at a temperature of about 1040°C is approximately the same for both compared bodies from tested mixtures SV and SG (Fig. 3).

Ceramic bodies with comparable water absorption (SV1000 and SG1090) have an incomparable bending strength, in favour of the dry pressed anorthitic ceramic body SG - anorthite increases the strength of the body.\(^{18}\)

At a comparable firing temperature (tested at firing temperatures 1000 and 1060°C), the ceramic body based on sludge SV has a lower total volume of pores in comparison with the ceramic body from spray granulate (SG) prepared by the industrial method (Fig. 5). With the increase of firing temperature, both types of ceramic bodies show an increase in mean pore radius.
The sludge obtained from the washing process of aggregate represents a ceramic raw material suitable for preparation of dry pressed ceramic bodies. The advantageous granulometry of the sludge does not require milling, as regards the preparation of dry pressed ceramic bodies. Owing to the very good sinterability, it is possible to fire ceramic bodies based on sludge at firing temperatures lower than the compared ceramic bodies made of spray granulate, intended for the production of dry pressed interior wall ceramic tiles.

This Research project was financed by the Czech Science Foundation, research project No. P104/10/0885 “Analysis of Stone Dusts and Sludges Influence on Ceramic Body Properties and Microstructure” and the project “SUPMAT—Promotion of further education of research workers from advanced building material centre”. Registration number CZ.1.07/2.3.00/20.0111.

References
1) A. Mostafa, *Interceram*, 57, 26–30 (2008).
2) P. Torres and H. Fernandes, *J. Eur. Ceram. Soc.*, 24, 3177–3185 (2004).
3) C. Vieira, *Mater. Sci. Eng.*, 373, 115–121 (2004).
4) M. Hojamberdiev, A. Eminov and Y. Xu, *Ceram. Int.*, 37, 871–876 (2011).
5) S. N. Monteiro, L. A. Peçanha and C. Vieira, *J. Eur. Ceram. Soc.*, 24, 2349–2356 (2004).
6) M. Raigon-Pichardo, G. Garcia-Ramos and P. Sanchez-Soto, *Resour. Conserv. Recycling*, 17, 109–124 (1996).
7) R. Sarkar and S. Das, *Tile Brick Int.*, 19, 24–27 (2003).
8) J. Garcia-Ten and A. Fernandez, *DKG*, 80, E84–E884 (2003).
9) J. Garcia-Ten and A. Fernandez, *DKG*, 80, E30–E34 (2003).
10) L. Catarino, J. Sousa, I. M. Martins, M. T. Vieira and M. M. Oliveira, *J. Mater. Process. Technol.*, 143–144, 843–845 (2003).
11) W. Acchar, F. A. Vieira and D. Hotza, *Mater. Sci. Eng.*, 27, 306–309 (2006).
12) A. M. Segadães, M. A. Carvalho and W. Acchar, *Appl. Clay Sci.*, 30, 42–52 (2005).
13) P. Torres and R. Manjate, *J. Eur. Ceram. Soc.*, 27, 4649–4655 (2007).
14) M. Hernandez-Crespo, *Ceram. Int.*, 27, 713–720 (2001).
15) M. Vieira, *J. Mater. Process. Technol.*, 92–93, 97–101 (1999).
16) G. Biffi, “Book for the production of ceramic Tiles”, Gruppo Editoriale Faenza Editrice S.p.A. Faenza (2003).
17) K. Traoré, T. S. Kabré and P. Blanchart, *Ceram. Int.*, 29, 377–383 (2003).
18) S. Kurama and E. Ozel, *Ceram. Int.*, 35, 827–830 (2009).
19) M. Sveda, *Ziegelindustrie International*, 59 (2004).