

# Effect of Temperature of Exposure on Properties of Cement Mortar with MSWI Bottom Ash

Z. Pavlík, M. Keppert, J. Žumár, M. Pavlíková, A. Trník, and R. Černý

**Abstract**—Effect of high temperature exposure on properties of cement mortar containing municipal solid waste incineration (MSWI) bottom ash as partial natural aggregate replacement is analyzed in the paper. The measurements of mechanical properties, bulk density, matrix density, total open porosity, sorption and desorption isotherms are done on samples exposed to the temperatures of 20°C to 1000°C. TGA analysis is performed as well. Finally, the studied samples are analyzed by IR spectroscopy in order to evaluate TGA data.

**Keywords**—Cement mortar, high temperature exposure, MSWI bottom ash, natural aggregate replacement, mechanical properties.

## I. INTRODUCTION

Municipal solid waste (MSW) needs to be set under environmentally and scientifically integrated management. The integrated management of municipal solid waste includes many aspects. In this paper, we will deal with a special treatment technology – incineration [1]. Incineration effectively reduces the volume of the waste and the volume reduction rate can reach even more than 90%. It also occupies much less area than landfill. Indispensable is also heat production during incineration that can generate electricity or hot water. Incineration is a waste treatment technology that involves the combustion of both organic and inorganic materials. It is the conversion of solid wastes into gaseous, liquid, and solid conversion products, with the concurrent or subsequent release of heat energy [2].

Since solid residuals coming from incineration of MSW possess a chemical composition that is not dissimilar to that of fly ashes from coal combustion, they represent interesting alternative materials for building industry.

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Recently, several studies on application of MSWI materials in cement industry were published and it is generally considered that these waste products can be, if meeting specific conditions, used as active as well as non-active mineral admixture [3], [4]. MSWI ashes can be beneficially used as partial Portland cement replacement pozzolana materials. On the other hand, bottom ash (BA), can be applied as fine aggregate or micro-filler [5], [6].

As one of the traditional technology, incineration is widely adopted in European countries and Japan to treat municipal MSW. However, not only in developed countries, but also in some big cities of several developing countries such as China, incinerators are also gradually being built. Hence, a great advantage in the sustainability of the concrete industry would be achieved if MSWI materials, which are available in great quantities throughout the world, could be used to produce quality concretes [7].

In this paper we refer about high temperature testing of cement mortar containing BA generated by MSWI as partial replacement of natural aggregate.

## II. EXPERIMENTAL

### A. Studied Materials and Samples Preparation

Ordinary Portland cement (CEMI 42.5 R) was used in the experimental study. MSWI BA was applied at 40% replacement level by mass of natural aggregate, while cement and water quantities were kept constant. For the sake of comparison, a reference mixture labeled MR without BA incorporation was studied as well.

Basic chemical composition of MSWI BA measured by XRF in mass % is as follows: SiO<sub>2</sub>– 33.5; Al<sub>2</sub>O<sub>3</sub>– 15.8; Fe<sub>2</sub>O<sub>3</sub>– 8.4; CaO– 19.4; MgO– 2.0; SO<sub>3</sub>– 9.3; Na<sub>2</sub>O – 3.6; ZnO– 0.8; K<sub>2</sub>O – 1.9; Cl– 1.1; TiO<sub>2</sub>– 1.5. We can see that the amount of chlorides is very low, what is beneficial from the point of view of possible reinforcement corrosion in case of application of studied materials in construction concrete. For the BA, there were measured basic material characteristics as powder density, matrix density, pH of leachate, solubility, and pozzolanic activity. The basic properties of BA are summarized in Table I. BA was firstly sieved, whereas only fraction 0-4 mm was used in this work in state “as received”. The composition of mortar mixtures is given in Table II.

TABLE I  
BASIC CHARACTERISTICS OF BA

Powder density (kg/m <sup>3</sup> )	Matrix density (kg/m <sup>3</sup> )	pH of leachate	Pozzolanic activity (%)
1 180	2 630	9.3	yes

TABLE II  
COMPOSITION OF STUDIED MATERIALS

Material		MR	MBA
CEMI 42.5 R	g	500	500
BA	g	-	600
Natural aggregate 0/4 mm	g	1500	900
Water	ml	260	260

The particular samples for material properties measurement were exposed to one of the following temperatures: 100, 200, 400, 500, 600, 800, 1000°C, whereas the time of exposure was 4 hours. Fig. 1 shows the electrical furnace that was used for sample tempering.

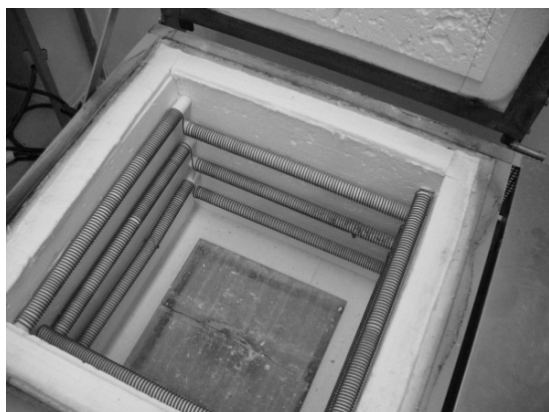


Fig. 1 View of electrical furnace

#### B. Determination of Basic Material Properties

For the analyzed mortars, measurements of bulk density, matrix density, and total open porosity were performed. The experiments were done on 5 cubic samples of side 50mm for each temperature of exposure. The relative expanded uncertainty of applied testing method was 5% and was mainly due to material in homogeneity. Bulk density was determined from the measurement of sample sizes (using digital length meter) and its dry mass. The matrix density was accessed by helium pycnometry using apparatus Pycnomatic ATC (Thermo Scientific). The accuracy of the gas volume measurement using this device is  $\pm 0.01\%$  from the measured value, whereas the accuracy of used analytical balances is  $\pm 0.0001\text{g}$ . On the basis of bulk density and matrix density measurements, the total open porosity was calculated [8], [9].

#### C. Mechanical Properties

The bending strength of mortars was determined using the procedure described in Czech standard ČSN EN 12390-5 [9] using hydraulic testing device. For the measurement, prismatic samples having dimensions of 160/40/40mm were used. The compressive strength was measured according to the standard ČSN EN 12390-3 [10] on the portions of prisms broken in bending test; the loading area was 40 x 40mm.

#### D. Measurement of Sorption and Desorption Isotherms

The dynamic vapor sorption device DVS-Advantage (Surface Measurement Systems Ltd.) was used for the measurement of adsorption and desorption isotherms of

mortars studied in this paper. The instrument measures the uptake and loss of vapor gravimetrically, using highly precise balances having resolution of  $0.1\mu\text{g}$ . The partial vapor pressure around the sample is generated by mixing the saturated and dry carrier gas streams using electronic mass flow controllers [11]. The humidity range of the instrument is 0 – 98% with accuracy  $\pm 0.5\%$  at temperatures 5 – 60°C.

Before the measurements, the particular samples of studied material were dried at first, and maintained in desiccator during cooling. Then, the sample was put into the climatic chamber of the DVS-Advantage instrument and hung on the automatic balances in the special steel tube (see Fig. 2).



Fig. 2 Placing of the sample into the DVS device

The experiment was performed at 25°C. The sample was exposed to the following partial water vapor pressure profile: 0; 20; 40; 60; 80, and 98% relative humidity. During the experiment, the DVS-Advantage instrument was running in dm/dt mode (mass variation over time variation) to decide when equilibrium was reached. A fixed dm/dt value of 0.0000% per min was selected for all relative humidity segments. This criterion permits the DVS software to automatically determine when equilibrium has been reached and complete a relative humidity step. When the rate of change of mass fell below the threshold over a determined period of time, the relative humidity set point proceeded to the next programmed level [11].

#### E. TGA Analysis

The thermogravimetric investigations were done using apparatus Labsysevo (Setaram) that allows simultaneous TGA-DTA and TGA-DSC measurements. The operating temperature range of this device is 25 – 1600°C. In our experiments, the studied samples were firstly ground using ball mill. Fig. 3 shows typical particle size distribution of powdered samples.

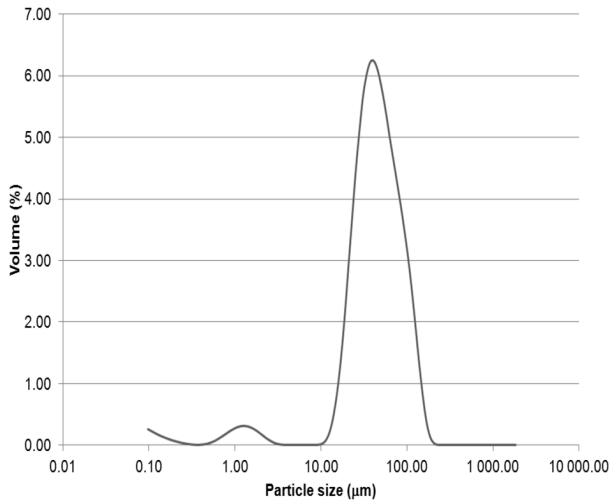


Fig. 3 Typical particle size distribution of powder sample

Within the measurement, the powdered samples were linearly heated with rate of 5°C/min up to 1000°C. The samples were measured at argon protective atmosphere. Here, TGA as well as DTG data obtained from derivation of TGA curve will be presented.

#### F. FTIR (Fourier Transform Infrared) Spectroscopy

For explanation of TGA and DTG data, the FTIR spectroscopy was used. We applied Nicolet 6700 FTIR Spectrometer (Thermo Scientific). Its spectral range varies between 7800 – 350cm<sup>-1</sup> with spectral resolution 0.4cm<sup>-1</sup>. This equipment contains pyroelectric detector HP DLaTGS, KBr beamsplitter. The mirrors are golden diamond wrought and the light source is high-intensity source EverGloTM [12], [13].

### III. RESULTS AND DISCUSSION

Results of matrix density and bulk density measurement in dependence on temperature of exposure are presented in Fig. 4. One can observe increase of matrix density for both studied materials with temperature what can be assigned to the solid matrix shrinkage that is in relation to the decrease of bulk density. This effect also leads to the high increase in porosity, especially after high temperature exposure, due to the bigger distance of solid phase compounds and possible cracks formation (see Fig. 5).

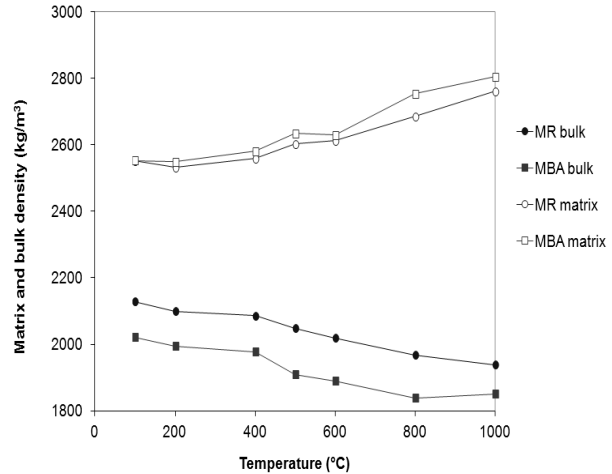


Fig. 4 Matrix and bulk density of mortars after temperature exposure

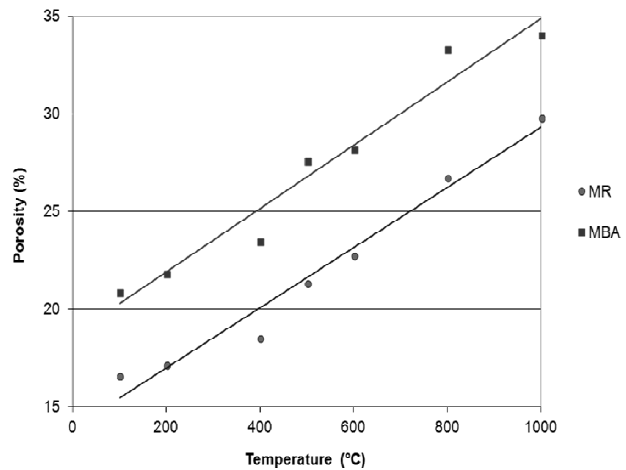


Fig. 5 Open porosity of mortars after temperature exposure

From the quantitative point of view, the reference mortar MR exhibited lower porosity, compared to mortar with BA. This resulted also in the lower compressive strength of BA modified mortar. Mechanical properties are summarized in Figs. 6 and 7. Obviously, for the temperatures higher than 200°C both compressive and bending strength of both materials decreased. The reference mortar MR had about 15% higher compressive strength than MBA up to about 600°C which corresponded with its lower porosity but then the situation was reversed; the compressive strength of BAM was then 3-5MPa higher.

The bending strength of MR was higher than BAM up to 300°C only, then its values were ≈ 0.5MPa higher for BAM. The relatively good high temperature stability of MBA despite its higher porosity can be explained by the better resistance of BA aggregates to high temperatures as compared to silica.

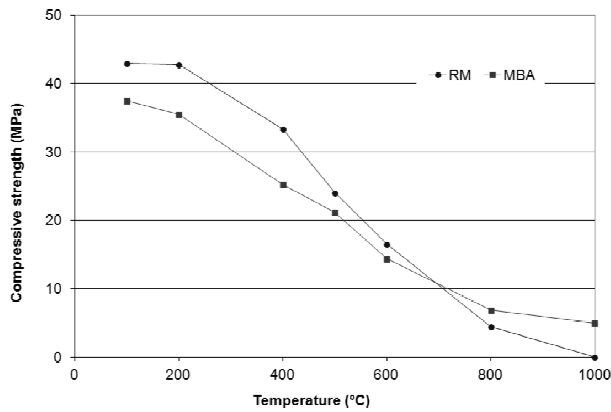


Fig. 6 Compressive strength of mortars in dependence on temperature of exposure

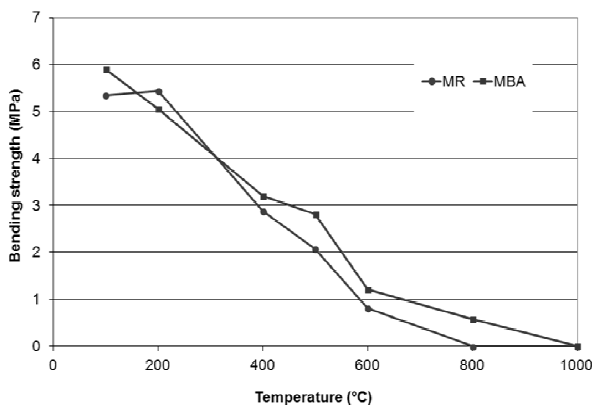


Fig. 7 Bending strength of mortars in dependence on temperature of exposure

Sorption and desorption isotherms measured by dynamic vapor sorption device in the whole relative humidity range are shown in Fig. 8. Here, only the data measured for mortars exposed to 100°C and 800°C are presented. It is quite evident that the samples exposure to 800°C led to a significant decrease of water vapor storage in both studied materials. This finding clearly supports fact that the surface properties of inner porous space influence the van der Waals forces between substrate (inner material surface) and water vapor molecules. Shrinkage of solid matrix due to the high temperature exposure decreased the total storage capacity of studied materials as well as hysteresis of moisture sorption. Although the total open porosity of both materials increased with temperature, the changes of surface properties are without question the reason of measured low moisture storage capacity. Similar results were observed also for other temperatures we investigated.

In Table III, the effect of hysteresis between sorption and desorption cycles and its differences after temperature exposure to 100°C and 800°C is summarized. One can see a decrease in moisture hysteresis after high temperature exposure that is typically two times higher for mortar with BA.

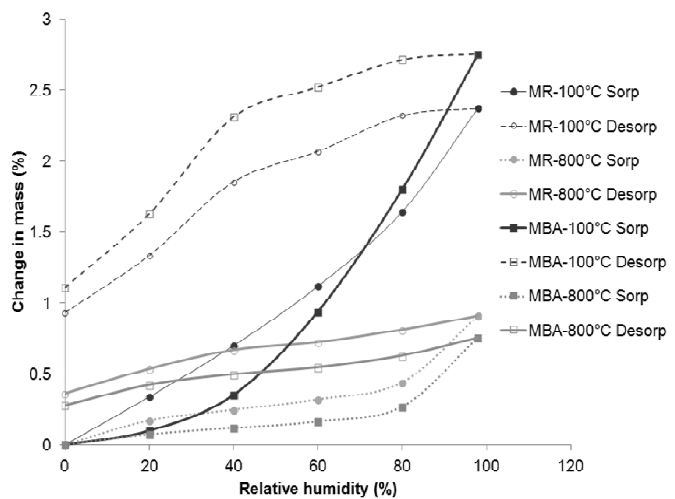


Fig. 8 Sorption and desorption isotherms of mortars in dependence on temperature of exposure

TABLE III  
HYSTERESIS IN WATER VAPOR STORAGE

	Relative humidity (%)	Hysteresis 100°C (mass %)	Hysteresis 800°C (mass %)	Hysteresis Difference (mass %)
MR	20	0.996	0.3637	0.6323
	40	1.155	0.4219	0.7331
	60	0.952	0.4037	0.5483
	80	0.678	0.3715	0.3065
MBA	20	1.529	0.3498	1.1792
	40	1.959	0.3798	1.5792
	60	1.580	0.3825	1.1975
	80	0.913	0.3602	0.5528

TGA and DTG data are presented in Figs. 9 and 10. Here, also the data for BA are given. In the DTG curves, we can distinguish several temperature ranges that are clearly defined by the edges of the characteristic peaks of DTG curve. The first characteristic peak is approximately at 105°C and the mass loss is remarkable up to 200°C. At temperatures 30 – 105°C, the evaporable water and a part of the bound water escape. It is generally considered that the removal of evaporable water is finished at 120°C. Temperature range 110 – 200°C is characteristic by decomposition of gypsum, decomposition of ettringite and loss of water from part of calcium-aluminate hydrates. At these temperatures, mortar MR exhibited lower mass loss, probably due to the higher content of sulfates in BA. Another significant peak on DTG curve is approximately at 450°C, and is due to the decomposition of portlandite. Here, mass loss of MBA was markedly lower compared to reference mortar MR. This finding indicates the lower volume of portlandite in the mortar containing MSWI BA. At temperature 500 – 700°C dissociate unstable calcium carbonates as vaterite. Temperature interval 700 – 900°C is characteristic by decomposition of calcium carbonate [14].

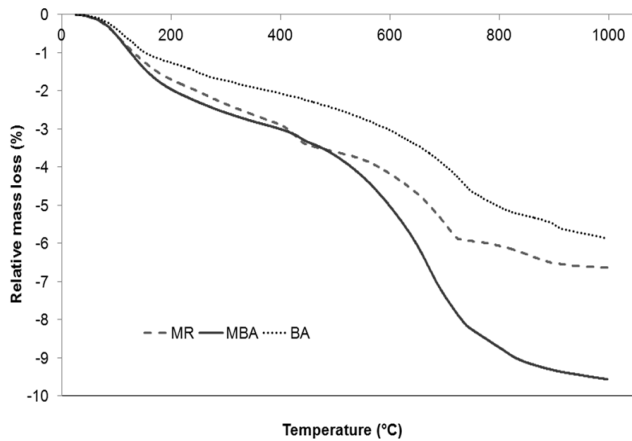


Fig. 9 TGA curves measured within linear heating of studied materials

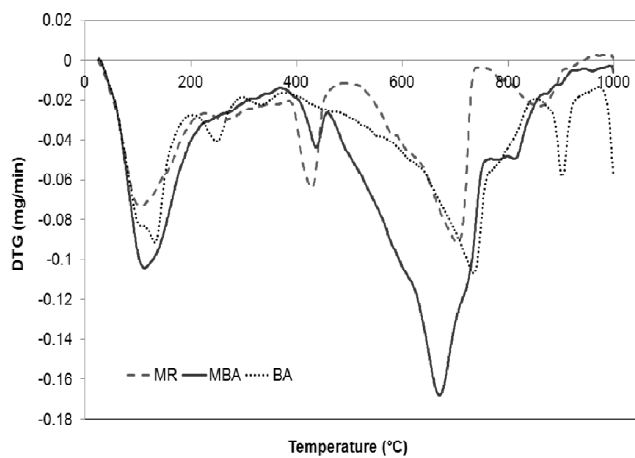


Fig. 10 DTG curves measured within linear heating of studied materials

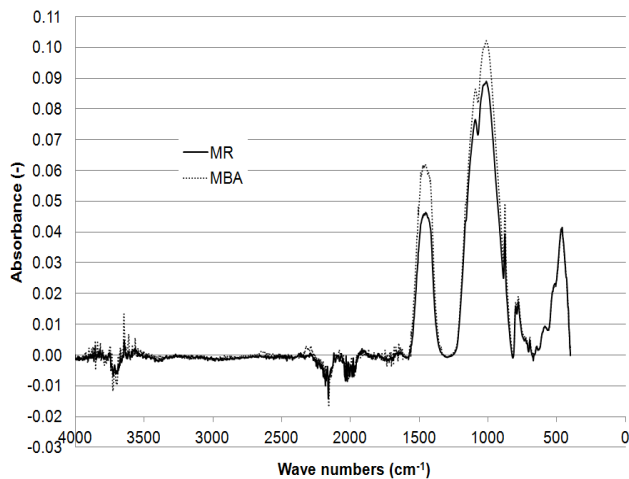


Fig. 11 Results of FTIR spectroscopy

Results of FTIR spectroscopy measured for samples without tempering are shown in Fig. 11. These results clearly

support explanation of DTG data. The peak at  $3644\text{cm}^{-1}$  corresponds to portlandite, since narrow band at approximately  $3635\text{cm}^{-1}$  is typical for O–H stretching vibrations. Peaks at  $1452$ ;  $875$ , and  $694\text{cm}^{-1}$  represent  $\text{CO}_3^{2-}$  in different forms of calcite as stated above. Peak at  $1090\text{cm}^{-1}$  corresponds to  $\text{SO}_4^{2-}$  sulfate bond and at  $966\text{cm}^{-1}$  to Si–O deformation vibration in C–S–H gel.

#### IV. CONCLUSIONS

In this paper, experimental analysis of the effect of temperature exposure on the material parameters of two types of cement based mortars was performed. Reference cement mortar MR as well as MBA exhibited high dependence of studied parameters on temperature of exposure. The significant changes of materials properties were observed for both studied materials from temperature of  $200^\circ\text{C}$  due to the evaporation of bound water, what causes decomposition of gypsum, decomposition of ettringite and loss of water from part of calcium-aluminate hydrates. The performed TGA and FTIR spectroscopy analysis provided an explanation of the reactions leading to material decomposition at high temperatures.

At higher temperatures, the changes of inner structure and chemism of hydration products led to the sharp decrease of mechanical resistivity of tested cement based mortars. Due to the high temperature of exposure, there systematically decreased water vapor storage capability, for MBA two times more as compared to reference mortar.

From the point of view of the partial natural aggregate replacement by bottom ash, it was found that application of BA led to a higher porosity of the produced mortar but did not affect negatively its behavior in high temperature range. Moreover, the usage of BA partially improved compressive and bending strength at temperature of exposure higher than  $700^\circ\text{C}$ .

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