Utilization of Meat and Bone Meal Bottom Ash in Ceramics

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During utilization of animal waste meat and bone meal (MBM) is received, realization and use of which has been stopped due to risk for the transmission of the bovine spongiform encephalopathy infection. The MBM must be safely stored or treated. Most often meat and bone meal undergoes thermal treatment. During combustion large quantities of residues (ashes) are received, the recycled use of which has been given a lot of attention lately. In this work it was investigated the impact of the additive of the bottom ash (BA) formed during combustion of the MBM on the properties of forming mass and ceramic body of hydromica clay, and also it was evaluated a possibility to use the MBM BA in manufacturing of building ceramics. After replacing the sand in porous ceramics by this additive the plasticity of the forming mass, drying and firing shrinkage as well as density of ceramic body changed insignificantly whereas the compressive strength increased by $8\% - 22\%$. So the MBM BA can be utilized in production of porous ceramics.

Keywords: porous building ceramics, MBM BA, sand, clay body.

1. INTRODUCTION

There is approximately 60 thousand tons of animal waste produced in Lithuania annually. Due to the hazard of bovine spongiform encephalopathy infection, commonly known as a mad cow disease, this waste is utilized strictly observing the European Parliament and Council Regulations [2]. In order for the spongiform encephalopathy pathogen protein species called "prions" to loose their activity, the mentioned animal waste by-products have to be continually heated for not less than 20 minutes in saturated vapor environment maintaining 3 bar absolute pressure and 133 °C average layer temperature [1, 2].]. In these conditions the so-called Meat and Bone Meal (MBM), a low risk product is received [1–6]. Due to transmission of the mentioned infection in Europe the use of the MBM for feed production has been forbidden, this is why realization and use of the meat and bone meal have been stopped [2, 6]. Practically purposeful realization and use of the meat and bone meal have been abandoned, so it has to be safely stored or processed [7–9]. For storage of the MBM premises are necessary, this is why most often meat and bone meal is thermally treated in special furnaces alone [7–12], together with other fuel [11–14] or industrial raw materials [1–3] during burning and pyrolysis [7, 9, 12]. Since the MBM is flammable and rather calorific fuel (approximately 17000 kJ/kg) [1, 3, 7, 8, 14], during burning at higher than 850 °C [2] energy is regenerated and the prions are destroyed and disrupted (like all other organic substances decompose into CO_2, H_2O, etc.) [15–17].

It has been established that after burning of 1 ton of the MBM, 100 kg – 310 kg of bottom ash (MBM BA) are received [1, 3, 9]. The research results have shown that according to the European classification of landfill waste the industrial MBM BA can be considered as non-hazardous substances [18, 19].

One of strategic objectives of waste treatment is reduction of the quantity of waste taken to rubbish dump, this is why presently a lot of attention is given to recycled use of waste. Furthermore, manufacturing technologies are being improved in order to make waste non-hazardous already during a technological process. Suitably treated MBM BA, as a raw material with prevailing calcium phosphate in its mineral composition [4, 6, 20, 21], can be used as a fertilizer or an additive in production of phosphoric acid [1], for chemical stabilization of heavy metals present in contaminated water, soil and industrial waste [21–22], also for immobilizing toxic metals [23, 24], in production of lime cement mixtures and concrete replacing sand [1, 25].

Different types of waste are utilized in ceramics. Fly ash, which is a solid waste produced from thermal power station, significantly reduces clay plasticity and increases the burning temperature of ceramics by 50 °C – 100 °C [26]. The waste formed during galvanization process increases strength of ceramic products, and toxic elements present in this waste are well-immobilized in a ceramic body [27]. For manufacture of building ceramics it is recommended to use the ashes formed during carbon gasification and by burning of orimulsion [28, 29]. Carbon ash additive (up to 20 %) at 900 °C in clays acts as a flux (quantity of K_2O increases by 4 %), it stimulates occurrence of melt this is why hydromicas start decomposed at a lower temperature. The bloatability of the clay may be increased by employing organic additives such as heavy fuel oil, fine turf, rubber waste and others [30].

Building ceramics are often produced from moulding mixtures, the composition of which besides clay also includes sand, grog and sawdust [31]. The amount and granulometric composition of these non-plastic substances define the quality of ceramic products [29]. The research results have shown that the physical properties of MBM BA are similar to those of fine sand [3], so this study has investigated a possibility to replace sand in clay forming mass with the meat and bone meal bottom ash. The objective of the work is to utilize the bottom ash which remains after the combustion of MBM in ceramics, i. e. to investigate the influence of the BA additive on properties of forming mass and the ceramic body from hydromicous

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Table 1. Chemical composition of used materials, in wt. %

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MnO	MgO	Na_2O	K_2O	P_2O_5	N_2	C	SO_3	LOI
Clay	47.69	0.76	13.35	6.19	9.05	0.06	3.59	0.96	3.32	–	–			11.9
MBM BA	1.34	<0.01	0.33	0.22	43.26	<0.03	1.24	1.59	0.72	35.2	0.44	0.38	1.02	4.98

clay and to evaluate the possibility of use of the MBM BA in manufacture of building ceramics.

2. MATERIALS AND METHODOLOGY

The clay of the Girininkai (Lithuania) pit was dried at $105\,°C - 110\,°C$, and then grained by a dismembrator into grains smaller than 1 mm. The chemical composition of the clay is presented in Table 1.

MBM BA is industrial waste resulting from meat and bone meal incineration. The bottom ash was used of natural fineness (specific surface area (S_a) – 83 m^2/kg and bulk density –1159 kg/m^3) and ground in a porcelain ball mill ($S_a = 250$ m^2/kg and bulk density – 1096 kg/m^3). The chemical composition and particle size distribution of meat bone meal bottom ash are presented in Tables 1, 2 and 3.

The sand of natural moisture and fineness from the Zatyščiai (Lithuania) pit was used of. The bulk density of the sand is 1365 kg/m^3. The particle size distribution is presented in Table 3.

The sawdust is a waste-product of freshly felled timber. It is obtained from wood-processing companies, and used of natural moisture. The maximum particle size is of 3 mm.

Grog is crushed waste of fired ceramic body. The maximum particle size is of 3 mm.

While preparing the samples dry powder of clay and additives were mixed with water for preparation of a plastic moulding mixture with humidity of $20\,\% - 24\,\%$. Cubes of the size (40×40×40) mm and slabs of the size (60×30×10) mm were formed from these mixtures, dried at $105\,°C - 110\,°C$ and fired in laboratory furnace, at a temperature bias no larger than ±3 °C. The temperature was increased up to 120 °C at 200 °C/h rate, afterwards, at 500 °C/h rate – up to the desirable temperature. The samples were fired at 1000; 1025; 1050; 1075; 1100 °C for 1 hour.

Table 2. Trace elements in MBM BA, mg/kg

Cu	Zn	Cr	Cd	Ni	Pb	Se
42	85	13	0.21	7.7	1.46	1.02

Chemical composition of raw materials was determined by atomic absorption spectrophotometer using absorption and emission methods.

The X-ray powder diffraction (XRD) data were collected with DRON-6 powder X-ray diffractometer with Bragg-Brentano geometry using Ni-filtered CuK_α radiation and graphite monochromator, operating with voltage 30 kV and emission current of 20 mA. The step-scan covered the angular range $5° - 70°$ (2Θ) in steps of $2\Theta = 0.02°$.

Simultaneous thermal analysis (STA: differential scanning calorimetry – DSC and thermogravimetry – TG) was carried out on a Netzsch instrument STA 409 PC Luxx with ceramic sample handlers and crucibles of Pt-Rh.

Heating rate of 15 °C/min, the temperature ranged from 30 °C up to 900 °C under the ambient atmosphere.

Optical microscopic analysis was performed by optical microscope "Olympus cx 31". Scanning electron microscopy (SEM) (FEI Quanta 200 FEG) was performed using an accelerating voltage of 15 kV and a working distance of 10 mm for SEM observation.

The granulometric composition of the raw materials, plasticity and moisture of clay forming mass, drying and firing shrinkage of the samples, water absorption and compressive strength of the clay body were determined according to specifications TS 5970087-06-94, TS 5970087-07-94 and standard LST EN 771-1: 2003.

3. RESULTS AND DISCUSSIONS

Chemical composition of the Girininkai clay used for research (Table 1) is similar to composition of most Lithuanian clays. The clay of Girininkai pit is hydromicous, illite (d – 1.300, 0.450, 0.333, 0.256, 0.199, 0.181 nm) and muscovite (d – 1.100, 0.449, 0.336, 0.256 nm) minerals prevail in it, there is also kaolinite (d – 0.710, 0.441, 0.356 nm) (Fig. 1, curve 1). There is a big amount of SiO_2, in the shape of quartz sand (d – 0.425; 0.334; 0.246, 0.182 nm) as well. Kalcite (d – 0.303, 0.228, 0.209, 0.191 nm), dolomite (d – 0.288, 0.219, 0.178 nm) and feldspar (d – 0.354, 0.348, 0.319 nm) are found among non-plastic admixtures (Fig. 1, curve 1).

According to the amount of particles smaller than 0.001 mm (48.18 %) the Girininkai clay is attributed to the group of dispersive clay. Such clay is characterized by good formation properties. According to the number of plasticity ($P = 14.82$) it is attributed to limited plasticity clays. Considering the fact that the amount of carbonate insertions bigger than 0.5 mm does not exceed 0.2 %, it
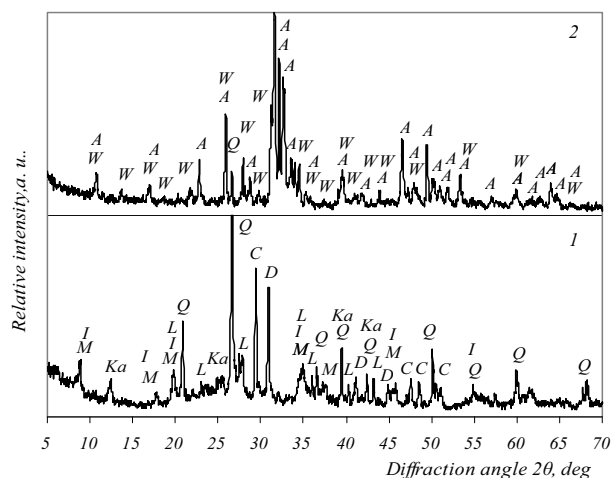

Fig. 1. X-ray difraction paterns of Girininkai clay (1) and MBM BA (2). Indexes: Q – quartz, H – hematite, M – muscovite, I – illite, C – calcite, D – dolomite, Ka – kaolinite, L – feldspar, A – hydroxyapatite, W – whitlockite

Table 3. Particle size distribution of MBM BA

Materials	Sieve number											
	3	2	1.6	1.0	0.8	0.5	0.4	0.315	0.25	0.1	0.063	>0.063
	Residue on sieve,%											
BA	2.78	2.48	7.19	11.76	5.36	15.43	4.94	9.06	6.27	26.53	6.04	2.15
Sand	–	–	–	0.1	0.15	1.76	2.36	11.38	15.83	48.87	18.83	0.1
Milled BA	–	–	–	0,3	4.84	11.94	5.09	9.23	7.55	51.69	6.53	2.82

can be stated that carbonates are finely dispersed, evenly distributed throughout the clay mass. Sintering is similar to the sintering of all calcareous clays. Water absorption of the ceramic body burned at 1025 °C is 12.73 %, and at 1075 °C – 6.71 %. With further increase of temperature water absorption rapidly decreases and at 1100 °C it is only 0.33 %. Thus, the clay sintering interval is quite narrow.

MBM BA resembles deep gray sand in colour. According to the data of optical microscopic and SEM analyses the MBM BA is composed of white, brown and black color porous grains (Fig. 2, a) of irregular shape (Fig. 2, b).


a   b
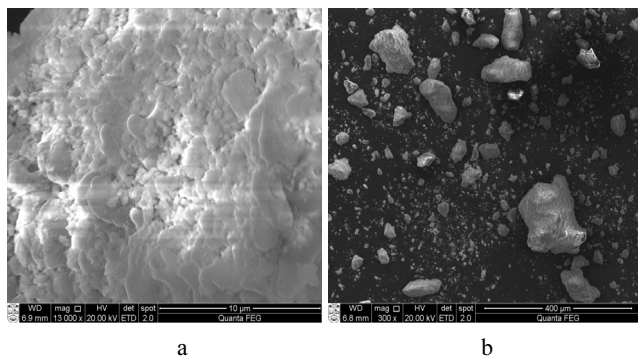
Fig. 2. Scaning electron microscope images of MBM BA particles

It has been identified that ash as well as sand predominantly contain particles of 0.1 mm, however the ash contain almost 2 times less of this fraction (Table 3). Ash contain more particles of a coarser fraction (larger than 1.6 mm), including almost 3 % of those bigger than 3 mm. In order to unify granulometric compositions of sand and ash, the latter have been ground. After grinding, the analysis of the ash granulometric composition was done and it was established that 51.59 % of the grist remained on 0.1 mm sieve, which means that although the quantity of some other fractions differs (Table 3), quantity of dominating fraction (0.1 mm) particles of ground ash and sand is very similar. Therefore it can be accepted that granulometric composition of the ground BA particles is similar to that of natural sand, which is used in preparation of molding mass for ceramics.

According to the data of chemical analysis MBM BA has high calcium and phosphorus contents (Table 1). They amount to 45.3 % of the total ash quantity. It is natural since the main non-organic component of the meat and bone meal is calcium phosphate [1, 10]. Traces of Cu, Zn, Cr, Cd, Ni, Pb, Se are found in MBM BA (Table 2). The quantity of these elements (150.4 mg/kg) compared to existing in the ashes of other burned solid municipal waste (>46000 mg/kg) [32] is very low. Most abundant element found is zinc – 85 mg/kg.

The data of X-ray diffraction analysis confirm that MBM BA predominantly contains calcium phosphates – whitlockite $Ca_3(PO)_2$ (d – 0.321, 0.288, 0.260 nm) and calcium deficient hydroxyapatite $Ca_9HPO_4(PO_4)_5OH$ (d – 0.281, 0.278, 0.272 nm) (Fig. 1, curve 2). These compounds render color to ash granules. Hydroxyapatite colours the granules in white, whereas whitlockite colours them in black. Brown colour is the result of combination of the both minerals [1].

MBM BA thermal analysis data reflect changes of hydroxyapatite during burning, in which a part of PO_4^{3-} or/and OH^- is substituted for carbonate CO_3^{2-} ions [33, 34]. DSC curve shows 3 endothermic peaks (Fig. 3, curve 1). Endothermic peak at 70 °C could be assigned to desorption of adsorbed water. Second endothermic behavior in temperature range of 671 °C – 712 °C (maximum 683 °C) may represent the decarboxylation of hydroxyapatite and the last one at 800 °C – 900 °C involves the removing of biggest part of water present in interlayers of hydroapatite crystalline lattice and decomposition of hydroapatite to β $Ca_3(PO_4)_2$ and hydroxylapatite [33, 35]. While heating up to 660 °C the mass loss is 3.8 %, whereas the mass loss in the temperature range of 660 °C – 900 °C is 2.4 % (Fig. 3, curve 2).
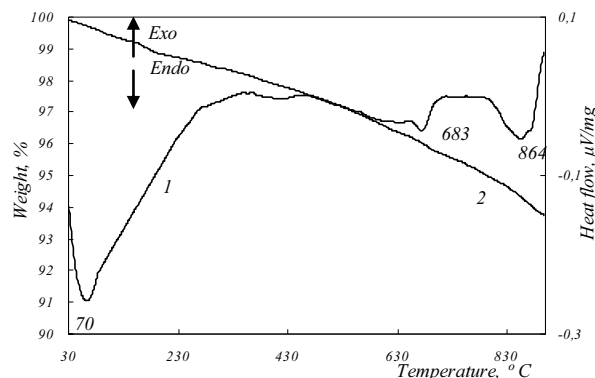
**Fig. 3.** DSC (1) and TGA (2) curves of the MBM BA

In order to establish the dependence of the MBM BA additive on properties of the Girininkai clay 3, 5, 7, 10 and 20 percent of the additive was added to the clay. Already 3 % of the ash additive reduced the plasticity of the clay mass by more than 1 percentage point, whereas 7 % and 20 % of the additive reduced it correspondingly by 3.2 and 3.8 percentage points (Fig. 4). This proves that in order to achieve an optimal consistence of clay-ash mixture it is necessary to use less water. However, with increase of the ash additive amount in the clay mass, its moisture reduces insignificantly: from 22.93 % to 21.40 % (Fig. 4). It can be explained by a porous structure of the ash particles and

their greater water adsorption [1, 3]. Thus, MBM BA just like sand is a non-plastic substance for the clay forming mass, therefore samples during drying shrink less.
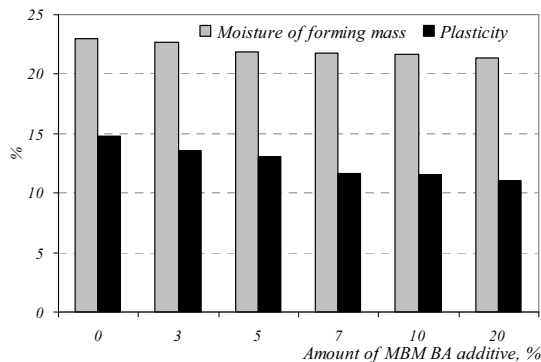

Fig. 4. Dependence of the moisture and plasticity of clay forming mass on the amount of MBM BA additive

It has been established that the MBM BA additive has an impact on burning shrinkage of the Girininkai clay ceramic body, and therefore, on sintering as well. The larger is the amount of ash in the Girininkai clay forming mass, the less is the burning shrinkage of the samples (Fig. 5).
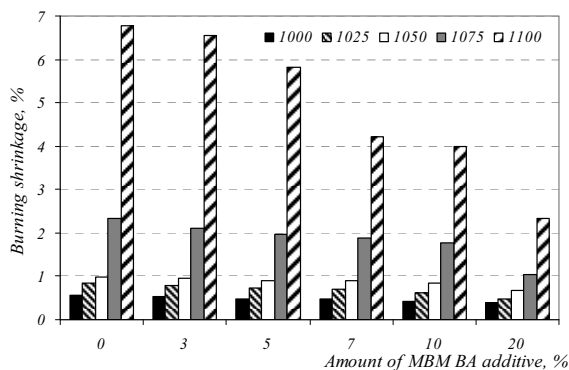

Fig. 5. Dependence of the burning shrinkage of clay body on the amount of MBM BA additive

Water absorption of the ceramic body also depends on the amount of the MBM BA additive and burning temperature. While increasing the amount of the additive in the forming mass from 0 % to 10 %, water absorption of ceramic bodies burned at 1000 °C and 1025 °C is similar – around 13 % (Fig. 6). At 1050 °C the difference of water absorption values between Girininkai clay (without the additives) ceramic body and the ceramic body with the ash additive starts to increase. This difference gets signifi-cantly high when burning the samples at 1075 °C: water absorption of the ceramic body with 3 % – 7 % of ash additive increases from 6.7 % to 9.8 %, whereas with 10 % of additive it increases up to 11 %. Thus, a ceramic body without additives at 1050 °C and 1075 °C, as well as at 1100 °C temperature sinter much better compared to one with the ash additives. At earlier mentioned temperatures the influence of a larger amount of the ash additive on the ceramic body sintering is especially evident. Water absorption of the ceramic body with 20 % of ash additive, depending on a sample burning temperature, varies from 15.06 % to 9.20 % (Fig. 6).
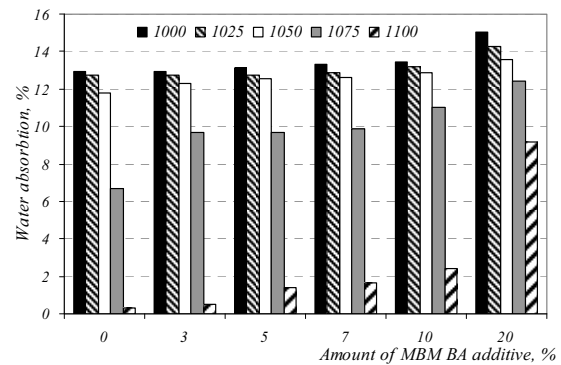

Fig. 6. Dependence of the water absorption of clay body on the amount of MBM BA additive

During X-ray diffraction analysis it has been established that in the Girininkai clay ceramic body burned at 1025 °C diopside $CaMgSi_2O_6$ (d – 0.300, 0.253, 0.252 nm) and anorthite $CaAl_2Si_2O_8$ (d – 0.320, 0.319, 0.318 nm) are formed (Fig. 7, curve 1). Traces of peaks typical of hydroxylapatite $Ca_5(PO_4)_3(OH)$ (d – 0.281, 0.277, 0.272 nm) appear in X-ray pattern of the ceramic bodies with 3 % and 7 % of ash additive burned at the same temperature. On the other hand, intensity of peaks typical of anorthite and diopside in the X-ray pattern of these bodies remain the same as in the clay ceramic body pattern (Fig. 7, curves 1 – 3). In the diffraction pattern of the ceramic body with 10 % of ash additive peaks typical of whitlockite appear next to hydroxylapatite and the intensity of anorthite peaks diminishes (Fig. 7, curve 4). This tendency is expressed even stronger in the diffraction pattern of the ceramic body with 20 % of ash additive (Fig. 7, curve 5). With increase of the additive content, the clay component part in the forming mass decreases and
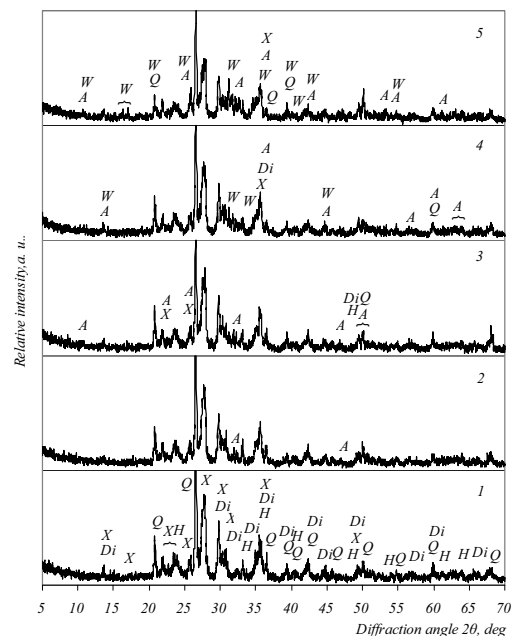
Fig. 7. X-ray difraction paterns of Girininkai clay body (1) and clay body with MBM BA additive fired at 1025 °C for 1 h. Amount of additive: 2 – 3 %, 3 – 7 %, 4 – 10 %, 5 – 20 %. Indexes: Q – quartz, H – hematite, X – anorthite, Di – diopside, A – hydroxyapatite, W – whitlockite

relatively lower amount of anorthite forms in the ceramic body during burning. While burning the samples with ash, after CO^{2-} breaks away, the formed $Ca_5(PO_4)_3(OH)$ and $Ca_3(PO_4)_2$ (already present in ash and formed during decomposition of $Ca_9HPO_4(PO_4)_5OH$) do not take part in the process of formation of a ceramic body (in liquid-phase reactions with the ceramic mass), therefore the structure of the ceramic body with 10 % and especially 20 % of ash additive is heterogeneous, more porous (Fig. 8), this is why it adsorbs more water.
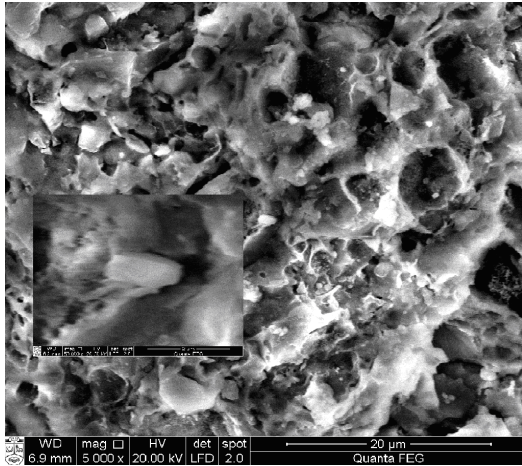
Fig. 8. Scaning electron microscope image of clay body with 7 % MBM BA additive fired at 1025 °C for 1 h

Thus, ground MBM BA and sand are non-plastic additives of clay mass with similar granuliometric composition. According to the data of technical regulations of building ceramics manufacturers grog, sawdust and, depending on clay plasticity, 3 % – 8 % of sand is added to the ceramic block moulding mixture. It has been established by previous researches that similar amount (3 % – 7 %) of ash in clay forming mass has almost no influence on ceramic body sintering at 1000 °C – 1025 °C. This is why it has been tried to identify whether it is possible to replace sand in the ceramic block (porous products) moulding mixture with MBM bottom ash. To that purpose samples of the following mixtures have been formed:
  I mixture: 83 % clay, 6 % grog, 8 % sawdust, 3 % sand;
  II mixture: 83 % clay, 6 % grog, 8 % sawdust, 3 % ash;
  III mixture: 79 % clay, 6 % grog, 8 % sawdust, 7 % sand;
  IV mixture: 79 % clay, 6 % grog, 8 % sawdust, 7 % ash.

It has been established that moisture as well as drying shrinkage of moulding mixtures I and II are very similar (Table 4). Due to high water absorption of the MBM BA moisture of formation mixture IV is higher than that of mixture II and, of course, of mixture III, since sand does not adsorb water [33].

While burning at 1000 °C, 1025 °C and 1050 °C the samples with the ash additive shrink slightly more (Table 4). However at 1075 °C and 1100 °C burning shrinkage of the samples with sand is higher. Values of water absorption of these samples start differing significantly from the values of the samples with the ash additive, after burning them already at 1050 °C temperature (Table 4). At 1050 °C, 1075 °C and 1100 °C quartz already starts to melt in the liquid phase [36, 37]. With the appearance of melt, porosity and, thus water absorption of the body decrease.

In Lithuanian ceramics manufacturing companies the highest burning temperature of building ceramic products is 1000 °C – 1025 °C. Considering ceramic body sintering at these temperatures, it can be stated that it is better to manufacture products of the moulding mixture containing 3 % of ash. On the other hand 7 % of ash also can be added to plastic clays. Water absorption value of these products varies around 22 %.

Additives of sand and ash reduce density as well as the compressive strength of the samples burned at 1000 °C – 1050 °C: the larger is the amount of these additives, the lower are the values of the mentioned parameters (Table 5). With increase of the burning temperature, the density of ceramic body increase and the ceramic body becomes stronger. Comparing compressive strength results of the samples with sand and ash, it was established that the samples with ash additive are stronger, although density of the ceramic bodies is very similar. The ceramic body with 3 % of ash burned at 1000 °C is stronger by 10.59 %, when the one burned at 1025 °C and 1050 °C is stronger by even more than 18 % (Table 5). Whereas the ceramic body with 7 % of ash additive is stronger respectively by 15.97 % and 7.66 % (at 1025 °C and 1050 °C) than the body with the same content of sand. The diffraction peaks characteristic for quartz are clearly seen in X-ray pattern of ceramic bodies of the mixtures I and III burned at 1025 °C (Fig. 9, curves 1 and 3). It means that at this burning temperature quartz still weakly participates in formation of new compounds or melt [38], and a larger amount of quartz in the sample reduces its mechanical strength [39 – 42]. Thermodynamically stable compound hydroapatite $Ca_5(PO_4)_3(OH)$ [43] renders to the ceramic body with the ash additive hardness and compressive strength [44]. While burning all mixtures mullite mineral is formed (Fig. 9, curves 1 – 4), however the peaks typical of this mineral in X-ray pattern of ceramic bodies with the ash additive are a bit more intense.

Table 4. Physical properties of samples from different forming mixtures

Mixture	Moisture, %	Drying shrinkage, %	Burning shrinkage, %					Water absorption, %				
			Firing temperature, °C									
			1000	1025	1050	1075	1100	1000	1025	1050	1075	1100
I	21.28	4.39	0.49	0.68	0.97	1.71	4.44	21.23	20.13	18.66	17.14	13.37
II	21.99	4.33	0.79	0.86	1.18	1.23	3.82	21.74	20.61	20.15	19.75	14.59
III	20.77	4.66	0.47	0.68	0.83	2.17	4.74	22.08	21.82	20.48	18.73	13.18
IV	23.21	4.63	0.63	0.79	0.94	1.10	3.56	22.29	21.94	21.90	21.00	15.34

Table 5. The influence of the firing temperature on the density and compressive strength of a ceramic body

Forming mixture	Density, kg/m³			Compressive strength, MPa		
	Firing temperature, °C					
	1000	1025	1050	1000	1025	1050
Clay	1681	1704	1759	47.25	50.33	58.45
I	1428	1435	1448	22.96	23.91	27.56
II	1439	1447	1472	25.68	30.75	33.75
III	1388	1393	1417	17.47	19.31	23.99
IV	1390	1410	1424	17.80	22.98	25.98

A higher compressive strength of the ceramic bodies can be explained by $Ca_5(PO_4)_3(OH)$ and a larger quantity of mullite in ceramic bodies with the ash additive [45, 46].
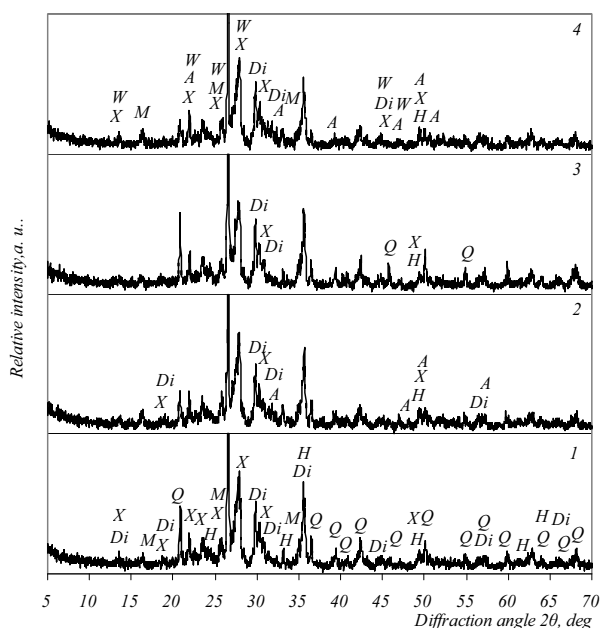
Fig. 9. X-ray difraction paterns of clay body from different forming mixtures fired at 1025 °C for 1 h. Forming mixtures: 1 – I , 2 – II, 3 – III, 4 – IV. Indexes: Q – quartz, H – hematite, X – anorthite, Di – diopside, A – hydroxyapatite, W – whitlockite

According to the data obtained from the X-ray diffraction analysis, phosphates present in the MBM BA composition at 1025 °C do not take part in the formation of new compounds with clay minerals (Figs. 7 and 9). With increase of the additive amount in the clay mass or the moulding mixture, besides minerals typical of a clay ceramic body such as, anorthite, diopside, hematite or mullite, peaks typical of only calcium phosphates appear and then become more intense. Therefore, summarizing the obtained research results, it may be stated that the MBM BA in the ceramic samples act as an inert additive. It can be used in manufacturing of ceramic products, because the moulding mass plasticity, drying and burning shrinkage, density and strength of such samples are similar to those of the samples with an analogous amount of sand.

4. CONCLUSIONS

1. MBM BA is a clay non-plastic additive: 3 % of ash additive reduce clay mass plasticity by more that 1 percentage point, whereas 7 % and 20 % of additive reduce it correspondingly by 3.2 and 3.8 percentage points.

2. With increase of the ash additive amount in the clay mass, drying and burning shrinkage of the samples decreases, water absorption of the samples increases. Sintering of the clay ceramic body and the body with 3 % and 7 % of MBM BA additive at 1000 °C and 1025 °C is similar.

3. MBM BA could be used as sand replacement in molding mixture of porous building ceramics since forming mass plasticity, drying and burning shrinkage as well as density of the ceramic body varies insignificantly, whereas compressive strength depending on amount of the additive and burning temperature increases by 8 % – 22 %.

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