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Investigation of flux influence on structure of foamed slag glass with a high content of slag waste.

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ABSTRACT

General information on the chemical and phase composition of wastes from coal combustion in thermal power plants (ash and slag) is represented. Principal ways of utilizing these wastes in the construction industry are listed. A new way to utilize ash and slag – synthesis of thermal insulating materials using foam glass technology (foamed slag glass) – is represented. It was found that a slag is more suitable for foamed slag glass production than ash and was proved that the additional introduction of materials reducing the melting point of the batch (fluxes) is required when the slag content is higher than 20%. Compositions based on various kinds of fluxes (boron compounds, carbonates, fluorides) are developed. Two optimal fluxes – borax ($Na_2B_4O_7$) and sodium fluoride (NaF) – are revealed. The compositions based on mixture of these fluxes are developed, their optimal ratio is defined. The mechanism of the influence of fluxing agents' mixture on the batch melting is analyzed, the role of each component of the mixture is discovered.

Keywords: coal combustion products, slag, foam glass, flux

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INTRODUCTION

Coal combustion products – general information

Ash and slag produced when burning of solid fuels at thermal power plants (TPP) are the sizable parts of the industrial wastes. An average annual ash and slag output of one TPP exceeds 1 million tons, and it could reach 5 million tons depending on type of used fuel.

The chemical composition of TPP ash and slag, their structure and properties depend on the composition of the mineral fuel, its combustion mode and calorific value, method of ash and slag collection and removal (Ramme & Tharaniyil, 2013). The TPP waste is frequently considered as a mix of ash and slag. The conventional boundary between ash and slag is a fraction of 0.25 mm: the smaller parts of waste are referred to as the ash, the larger ones as the slag (Ash Development Association of Australia, 2013).

The modernization of TPP waste removal technology leads to the use of the separate waste removal to produce clean ash and slag (Putilov & Putilova, 2007). These products, in comparison to the previously obtained ash-slag mixture, differ in composition constancy, the main features of which are given below.

Ash is formed by small and light fractions that are carried away from the furnace by hot gases and are trapped in the TPP filters. The ash from the coal combustion is represented mainly by SiO_2 and Al_2O_3. The CaO part is typically less than 5%. The chemical composition of TPP ash is represented in Table 1. Additionally, ash composition includes SO_3, MgO, TiO_2, etc. It may also contain 0.5-20.0% of unburned organic fuel particles depending on coal type and combustion conditions.

Table 1. TPP ash chemical composition

Content of oxides, wt%					
SiO_2	*Al_2O_3*	*CaO*	*Fe_2O_3*	*Na_2O*	*K_2O*
40.0-58.0	21.0-27.0	4.0-6.0	4.0-17.0	0.4-1.4	0.4-4.7

Ash contains typically considerable amount of particles with fine closed pores. The pores are the result of molten mineral mass expansion caused by the gases produced during dehydration of the clay minerals and dissociation of limestone, gypsum and organic substances. The total pore volume may reach 60 % of the ash particles.

Slag is formed by sintering individual particles on the grate at a temperature above 1000 °C or during cooling the molten mineral portion of the fuel at a temperature over 1300 °C. The use of furnaces with liquid slag removal becomes promising due to the intensification of the solid fuel combustion. The product of liquid slag removal is granulated slag, formed because of rapid cooling of the molten incombustible fuel part by water. This slag is usually represented by dark dense granules of black or brown colors with sizes from 0.15 to 15-20 mm; sometimes it can take a form of plates and threads of light and dark-green color with maximum dimensions up to 30-35 mm. The chemical and phase composition of the slag depends on the mineral part of solid fuels, and can vary widely. Slag composition determines the activity of slag and can be described by the basic module Mo (1):

$$Mo=(CaO + MgO) / (SiO_2 + Al_2O_3) \quad (1)$$

In accordance with the above formula, fuel slag can be grouped into three categories: ultra-acidic Mo <0.1; acidic with Mo = 0.6 - 0.9; basic with Mo = 1.0 - 1.1. It should be noted that the most common type of solid fuel is black coal (anthracite), which form ultra-acidic slag when burning. So, ultra-acidic slag comprises more than 90% of the fuel slag total volume (Punshon *et al.*, 2003).

The chemical composition of slag slightly differs from the composition of the mineral part of the corresponding solid fuel. This is due to the redistribution of some mineral components of the fuel between slag and ash. Industrial slag unlike ash with similar chemical characteristics does not contain unburned fuel particles. In addition, when the mineral part of solid fuel is melting in furnaces with liquid slag removal, the major amount of alkali and SO_3 is volatilized, which provides usually very low content of these components in slag (Sajwan et al., 2006; Ash Development Association of Australia, 2014). Limits of change in the chemical and phase composition of the ultra-acidic slag are given in Table 2.

Table 2. Chemical and phase composition of ultra-acidic TPP slag

Oxide content, wt%					Phase content, wt%	
SiO_2	*Al_2O_3*	*CaO*	*Fe_2O_3*	*MgO*	Glass	Crystalline
47.4-60.4	21.9-23.9	1.9-4.4	5.7-21.6	0.8-2.8	95.0-98.0	1.0-4.0

Thus, TPP ash and slag waste (ASW) are similar in composition to the raw materials used in the manufacture of building materials. It indicates the prospects of their use as a replacement of natural raw materials for different sectors of the construction, the main of which are listed below (Dvorkin et al., 2016).

Possible applications of ash and slag waste

The possibility of using TPP wastes in building material production is primarily determined by their structure and properties. The most researched area of ash and slag applicability, where they are used widely at present, is the *cement industry* (Jang *et al.*, 2015; Rafieizonooz *et al.*, 2016). Wastes here are used mainly as an active hydraulic additive in combination with other binders, or as an additive replacing part of the fillers in concrete production. Joint milling of ash and slag with the activating additives of binders improves concrete activity and increases product durability.

TPP ash and slag are effective raw materials for the production of *wall materials*: ceramic bricks, sand-lime bricks, ash ceramic, slag blocks. The use of ash and slag in the production is enabled due to their properties: chemical reaction with lime, dispersion, sintering behavior, calorific value.

ASW can be used for the manufacture of bricks and clay tiles. Primarily, it is recommended to use a low melting ash with softening temperature up to 1200 °C. Waste containing up to 10 % of the fuel is used as replacement of sand, and waste with 10 % or more – as the fuel-containing materials. Effectiveness of ash additives depends on their particle size and grain composition. The introduction of ash and slag reduces fuel consumption by 20-70%, and raw brick drying cycle is reduced by more than 20% (Sokolar & Vodova, 2011).
By mixing slag with binder, it is possible to produce a light and durable material – slag concrete – that is used in manufacturing of slag blocks (building blocks made of concrete mixes, where slag is used as filler). According to its heat-protective qualities, a slag block is 1.5 times more efficient than a brick and is cheaper in the same proportion.

In addition to the traditional areas of ASW usage in cement and ceramic industries, utilizing of ash and slag mixtures for the production of materials in glass technology is significant and promising. It is very important that the chemical and mineralogical composition of TPP slag is close to the composition of silicate materials (glass, glass coatings, glass ceramics, and others).

Melted materials are produced from molten ASW. It is most advisable to use slag of liquid removal. Vitreous slag pumice can be produced with fine pore structure and bulk density of 600-800 kg/m^3, dense castings with compressive strength up to 400 MPa, having improved resistance to corrosive environments at elevated temperatures. A number of technological methods for producing ash ceramics is developed, where TPP waste is no longer additional, but the main raw material (60-80 % fly ash, 10-20 % clay and other additives).

Ash ceramics can serve not only as wall material having stable strength and high resistance to frost. It is characterized by high acid resistance and low friction, which enables producing paving and road slabs, and other products having high chemical and thermal stability. In the production of ash, ceramic fuel consumption can be reduced by 1.5-4 times depending on the carbon content in ash, ash content in the batch and the heating conditions.

Technology for manufacturing of mineral melts for the production of mineral fibers, in particular, high temperature resistant mineral wool, was also developed on the basis of ash and slag waste. Such materials can be used in the production of sound- or heat-insulating and fire protection materials, substances for crop production, reinforcing fibers and fibers for filtration purposes (Wang, 2016).

One of the major consumers of ASW is *road construction*, where they are used for the arrangement of underlying and lower layers of the grounds, for the partial replacement of binders for soil stabilization with cement and lime, as the mineral powder in asphalt concrete and mortar, as an additive in road cement concrete.

A relatively new and promising direction of using ash and slag waste is the development of the technology of cellular insulating glass material – foamed slag glass. This material is a glass, the distinguishing feature of which is significant amount of artificial pores in the form of cells filled with gases. Cells with predominantly spherical shape and the diameter of 0.5-3 mm are uniformly distributed in body of materials providing its low density and thermal conductivity. Foamed slag glass can be produced by foaming initial glass batch with gases released as a result of foaming agent heat treatment. Using this material, in addition to solving the problems of resource and energy saving, as well as environmental issues, also helps to improve the energy efficiency of buildings, because of reduced heat loss through the building envelope (Fernandes *et al*., 2009; Bai *et al.*, 2014; Leroy *et al.*, 2001).

Thus, the use of ash and slag in the production of new building materials is an important aspect of resource saving because it is possible to produce a wide assortment of building materials, products and structures needed in the construction of residential and industrial buildings, agricultural, road and hydraulic engineering constructions, etc.

Peculiarities of the use of ash and slag in the production of foamed slag glass

Research of ash and slag applicability in foamed slag glass production has been conducted. It was found that the slag is more promising material for synthesizing glass materials than ash due to the following features:

- non-crystalline amorphous structure (Figure 1), which promotes greater reaction activity caused by the slag production process: when coal burns in the furnace, its incombustible mineral part in the form of a melt falls under the combustion chamber where it is quenched with water to form a vitreous material;
- angular form of slag particles (Figure 2) also increases the slag reaction activity in comparison with spherical ash particles (Figure 3);
- ash now is a popular raw material used in cement production, the different types of bricks etc. On the contrary, the slag is used in small quantities, mainly as filler in road construction, in spite of the advantages mentioned above.

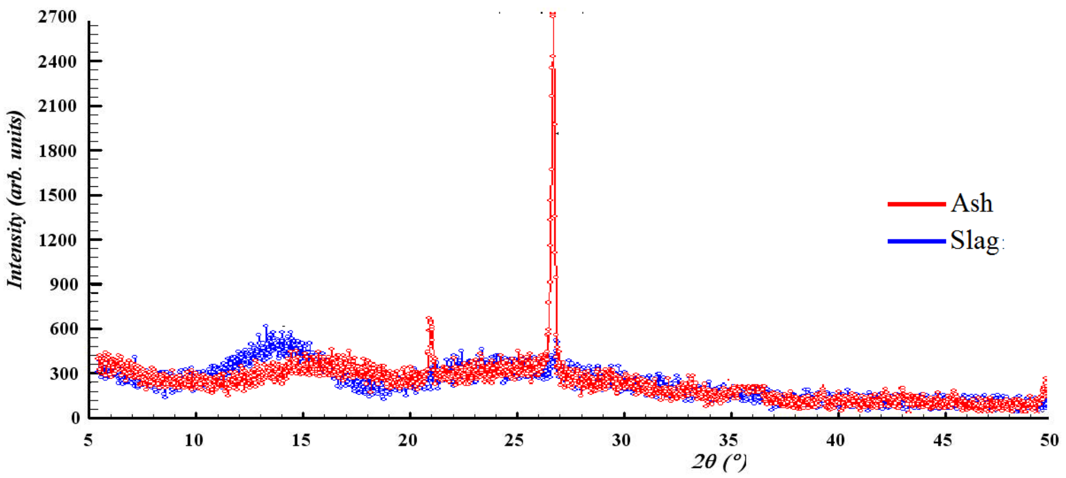

Figure 1. The XRD patterns of ash and slag

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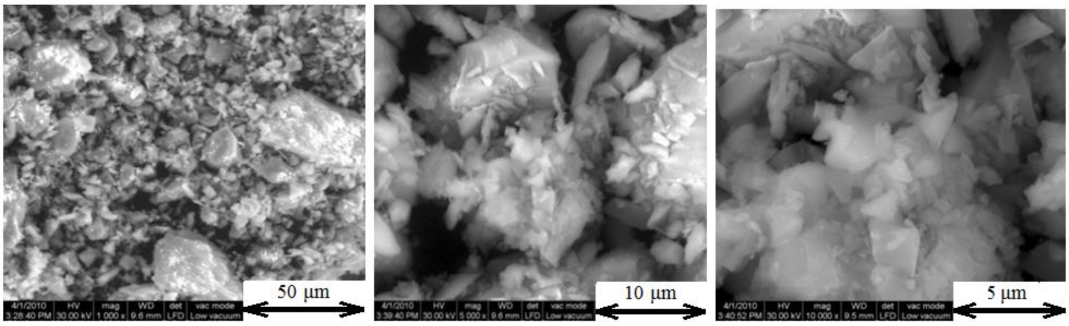

Figure 2. Slag microstructure

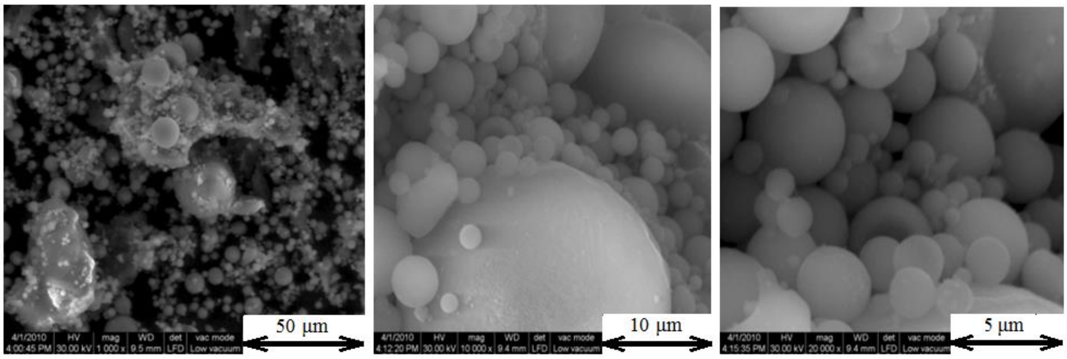

Figure 3. Ash microstructure

It was found that there is significant deterioration of porous structure formation when slag content in the batch is more than 20% due to increased content of refractory aluminum oxide. It was discovered that flux materials could be introduced in the batch to solve this problem (Yatsenko *et al.*, 2014; Yatsenko *et al.*, 2015). Fluxes in silicate industry are used for the intensification of the processes occurring during thermal processing of materials and therefore for lowering the processing temperature. Fluxes can be divided into two types. The first type fluxes are materials with lower melting point. The second type fluxes are materials with a high melting point, but they form low-melting compounds with initial raw mixture components (Sokolář & Šveda, 2016; Salem & Aghahosseini, 2012; Schoon *et al.*, 2013). In our first study borax ($Na_2B_4O_7$) was used as the flux, and its introduction allowed increasing the volume of input slag to 50%. However, studies of the influence of the flux type on the structure and properties of the resulting materials was not been carried out.

METHODS

Production of foamed slag glass (FSG) samples was conducted by the standard powder method. Raw materials (glass cullet, TPP slag waste, fluxes) were pre-dried at 120 °C. The resulting dry powders were milled to 420 microns fraction (mesh No. 40) (Aldrich Chemicals - Technical Library, *n.d.*). Chemical composition of the glass powder and slag waste was determined using energy dispersive X-ray fluorescence spectrometer ARLQUANT'X and is presented in Table 3.

Table 3. Chemical composition of raw materials

Material	Chemical composition*, wt%						
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O
Slag	57.5	23.0	10.8	1.9	1.2	3.6	0.9
Glass	71.2	2.7	0.8	3.4	7.6	0.8	13.2

* Oxides, content of which is less than 0.2%, are not shown.

Foaming mixture was prepared in a separate vessel by mixing the components in the following ratio, wt%: waterglass – 4, glycerol – 4, water – 2. Afterwards, the prepared raw materials were composed and mixed according to the following batch composition, wt%: glass – 40, slag – 50, glycerol mixture – 10, flux – 10 over 100. Then the samples from composed batches were molded into cubes with edge length of 20 mm and mass of 10 g (volume $8 \cdot 10^{-6}$ m^3, density 1250 kg/m^3). Then then samples were loaded into a furnace for heat treatment according to Figure 4.

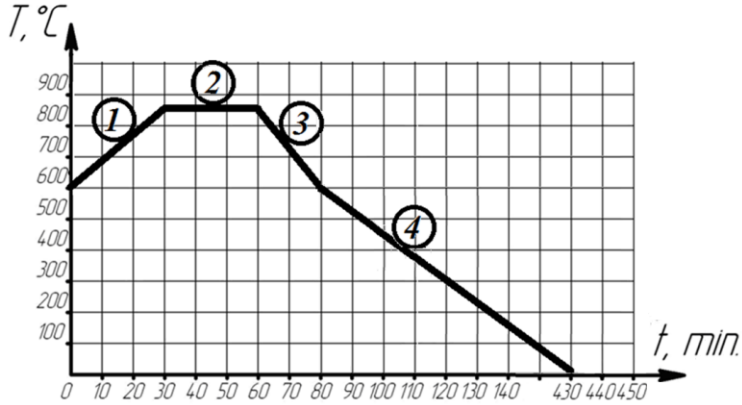

Figure 4. FSG synthesis mode:

1 - heating, 2 – foaming, 3 – rapid cooling with structure stabilization (quenching), 4 - slow cooling (annealing).

When the air inside the furnace cooled to room temperature, the samples were removed from the furnace and subjected to mechanical processing (filing) to give them the regular shape. Further, the weight of the samples of given shape was measured. Then calculations of the volume, density and foaming coefficient were performed based on the obtained data according to (2)-(4), respectively.

Volume $V = a \cdot b \cdot c$ (2)

Density $D = m / V$ (3)

Foaming Coefficient $FC_T = V_R^T / V_I$ (4)

where a – sample length, cm; b – sample width , cm; c – sample height , cm; V – sample volume, cm^3; m – sample mass, g; V_R^T –resulting sample volume after heat treatment at foaming temperature T, cm^3; V_I – initial sample volume before heat treatment, 8 cm^3.

Each recorded testing value was the mean of the results from five samples.

RESULTS

Table 4. The influence of the flux type on the foamed slag glass properties

Flux (Composition #)	Volume, cm^3			Foaming Coefficient			Density, kg/m^3		
	V_R^{850}	V_R^{875}	V_R^{900}	FC_{850}	FC_{875}	FC_{900}	D_{850}	D_{875}	D_{900}
Li_2CO_3 (F1)	10.05	10.37	10.76	1.26	1.30	1.35	995	964	929
Na_2CO_3 (F2)	8.91	9.22	9.50	1.11	1.15	1.19	1122	1085	1053
K_2CO_3 (F3)	9.14	9.61	10.11	1.14	1.20	1.26	1094	1041	989
$CaCO_3$ (F4)	8.83	9.38	10.26	1.10	1.17	1.28	1132	1066	975
$Na_2B_4O_7$ (F5)	16.21	18.83	21.74	2.03	2.35	2.72	617	531	460
BN (F6)	7.82	8.60	9.11	0.98	1.07	1.14	1278	1163	1098
H_3BO_3 (F7)	15.48	16.53	20.12	1.93	2.07	2.52	646	605	497
NaF (F8)	18.15	19.84	-	2.27	2.48	-	551	504	-
CaF_2 (F9)	7.37	7.56	7.76	0.92	0.95	0.97	1357	1322	1288
PbF_2 (F10)	6.63	6.63	6.64	0.83	0.83	0.83	1509	1509	1507

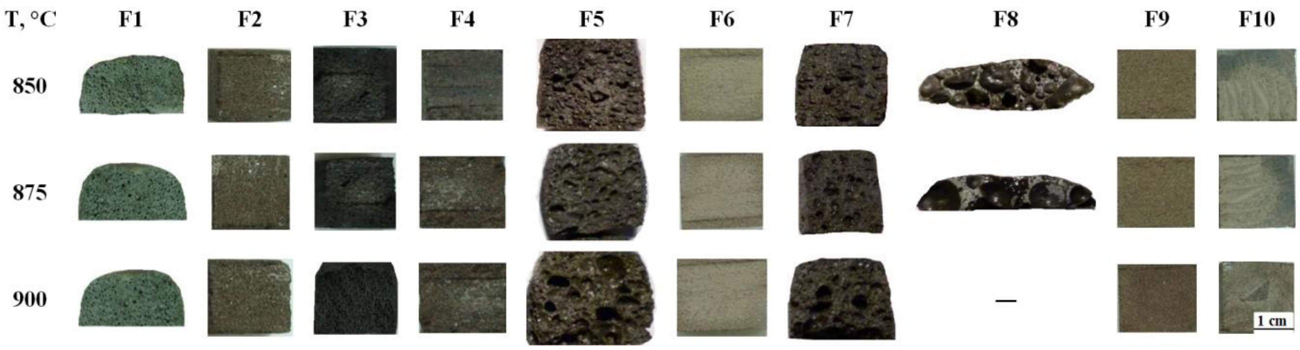

Figure 5. The influence of the flux type on the foamed slag glass structure

The materials, which play the role of flux in the silicate industry, were selected. These materials were divided into three groups: carbonates; fluorides; boron compounds (Eitel, 2012; Sharma, 1997; Singer & Singer, 1963; Bray, 2001; Philips, 2012). Thus, a number of compositions based on above composition with different fluxes was developed for the qualitative analysis of the selected fluxes influence. The compositions were subjected to heat treatment according to developed mode (Figure 4) at foaming (Stage 2) temperatures 850, 875, 900 °C. Results are represented in Table 4 and Figure 5.

Two most promising fluxes were identified on the basis of the table: composition F5, flux - borax $Na_2B_4O_7$ (large number of evenly distributed pores of different size, density less than 600 kg/m^3) and composition F8, flux - sodium fluoride NaF (uniform structure, excessive deformation of the sample under the influence of temperature up to the complete melting at 900 °C, density less than 600 kg/m^3). The composition F7 with boric acid as a flux had a relatively low density (650-500 kg/m^3), but the porous structure was very uneven. In the composition F1 with lithium carbonate as a flux small pores were observed, so the density of the composition did not fall below 900 kg/m^3. Samples of other compositions showed no tendency to form a porous structure with an appropriate density above 1100 kg/m^3.

The most active flux material was composition F8 flux – sodium fluoride NaF. The disadvantage of this composition is an excessive deformation of the sample during heat treatment. Therefore, the possibility to replace part of NaF with the other flux, which showed similar results in density (borax $Na_2B_4O_7$) was investigated. Results are presented in Table 5 and Figure 6. The density change dependence on the ratio of fluxes is presented in Figure 7.

Table 5. The influence of the fluxes ratio on the foamed slag glass properties

	Composition #				
	M1	M2	M3	M4	M5
Amount of NaF, wt%	1	2	3	4	5
Amount of $Na_2B_4O_7$, wt%	9	8	7	6	5
Synthesis temperature 850 °C					
Volume, cm^3	16.47	16.72	17.06	17.64	18.08
Foaming Coefficient	2.06	2.09	2.13	2.20	2.26
Density, kg/m^3	607	598	586	567	553
Synthesis temperature 875 °C					
Volume, cm^3	19.65	20.70	20.79	22.42	21.83
Foaming Coefficient	2.46	2.59	2.60	2.80	2.73
Density, kg/m^3	509	483	481	446	458
Synthesis temperature 900 °C					
Volume, cm^3	22.68	23.47	23.92	2.31	23.20
Foaming Coefficient	2.83	2.93	2.99	2.91	2.90
Density, kg/m^3	441	426	418	429	431

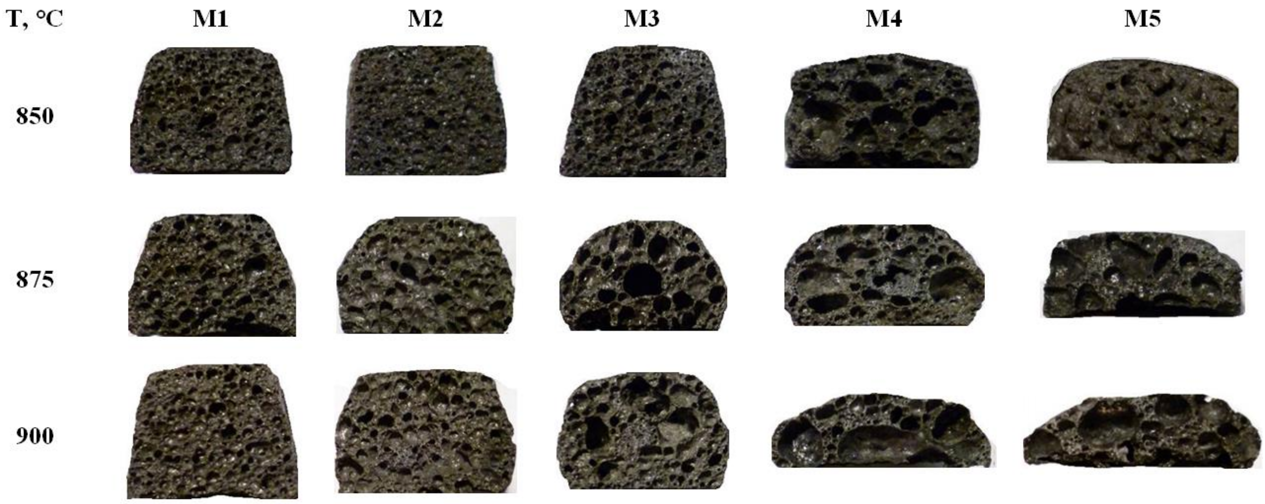

Figure 6. The influence of the fluxes ratio on the foamed slag glass structure

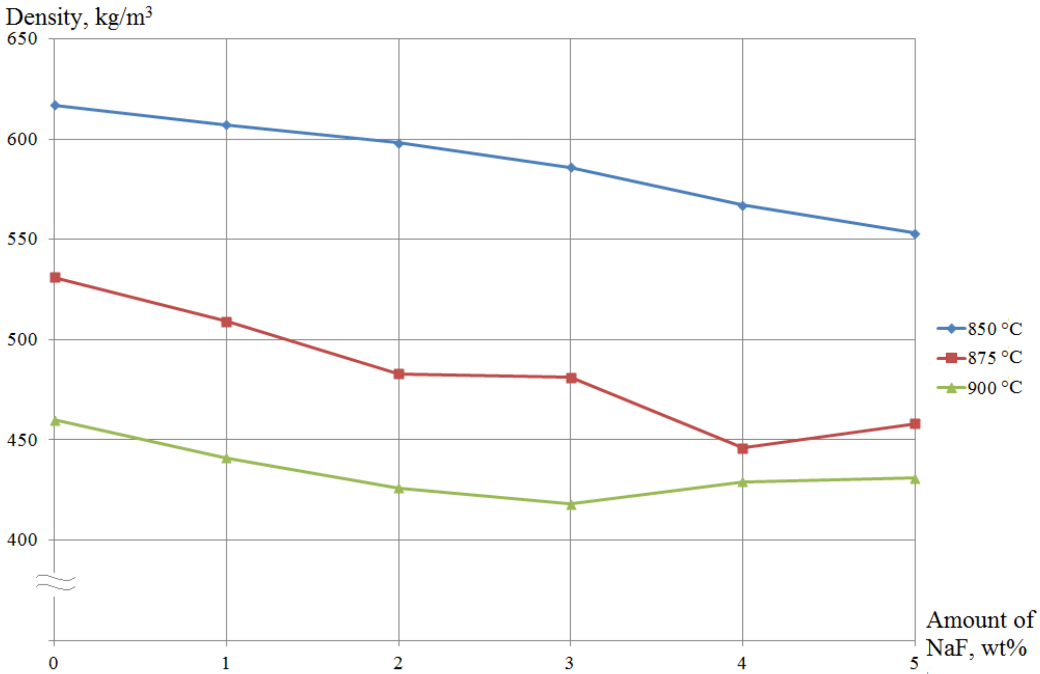

Figure 7. The density change dependence on the ratio of fluxes

The table and the figure shows that the optimum flux ratio is «$Na_2B_4O_7$: NaF = 8 : 2», which promotes the formation of an uniform porous structure with the density of about 430 kg/m^3.

DISCUSSION

The influence of the flux type and content on the structure and properties of foamed slag glass was considered. Optimal compositions F5 (borax $Na_2B_4O_7$) and F8 (sodium fluoride NaF) were selected as the optimal fluxes. Composition F7 with boric acid H_3BO_3 as a flux had a relatively low density (650-500 kg/m^3), but the porous structure was very uneven. Only small pores were observed in composition F1 with lithium carbonate Li_2CO_3 as a flux, so the density of the composition did not fall below 900 kg/m^3. Samples of other compositions showed no tendency to form a porous structure with an appropriate density above 1100 kg/m^3. Thus, two fluxes that demonstrated the highest activity (borax and sodium fluoride) were selected to develop a fluxing mixture. Study on the joint introduction of these fluxes allowed to identify their optimal ratio «$Na_2B_4O_7$: NaF = 8 : 2». The main component of the fluxing mixture is borax $Na_2B_4O_7$ (8 %). Boron compounds greatly reduce the melting point of silicate systems (Toropov, 1972; Toropov, 1974).

According to the discovered regularities, 10 % of borax is sufficient for forming a porous structure with a density of about 500 kg/m^3. However, studies have found that the most active flux is sodium fluoride NaF. Its use at a temperature of 850 °C leads to the significant deformation of the sample, and at temperatures above 875 °C – to complete melting of the sample. This high activity points to the advisability of its use as a supplement to the borax flux.

High activity of sodium fluoride is explained primarily by the fact that NaF consists of an alkali metal cation and fluorine anion, which can destroy the silicon-oxygen skeleton. The process of NaF interaction with the batch occurs likely according to the reaction (5) (Ahmed, 2005). Then the silicon tetrafluoride goes into gas phase and it interacts with the water vapor released in the process of heating foaming mixture according to the reaction (6):

$$4NaF + 3SiO_2 \rightarrow 2Na_2SiO_3 + SiF_4\uparrow \quad (5)$$

$$SiF_4 + 3H_2O \rightarrow SiO_2 + 4HF \quad (6)$$

Thus, the sodium fluoride in the fluxing mixture acts as the active component, leading to discontinuity of silicon-oxygen skeleton of the foamed slag glass, accelerating batch melting and simplifying the porous structure formation process. It explains the small number of NaF (2 %) required for batch melting.

Finally, the introduction with fluxing mixture of additional Na_2O in «Na_2O-Al_2O_3-SiO_2» system shifts equilibrium closer to low-melting compounds formation, as indicated by the arrow in Figure 8 (Toropov, 1972).

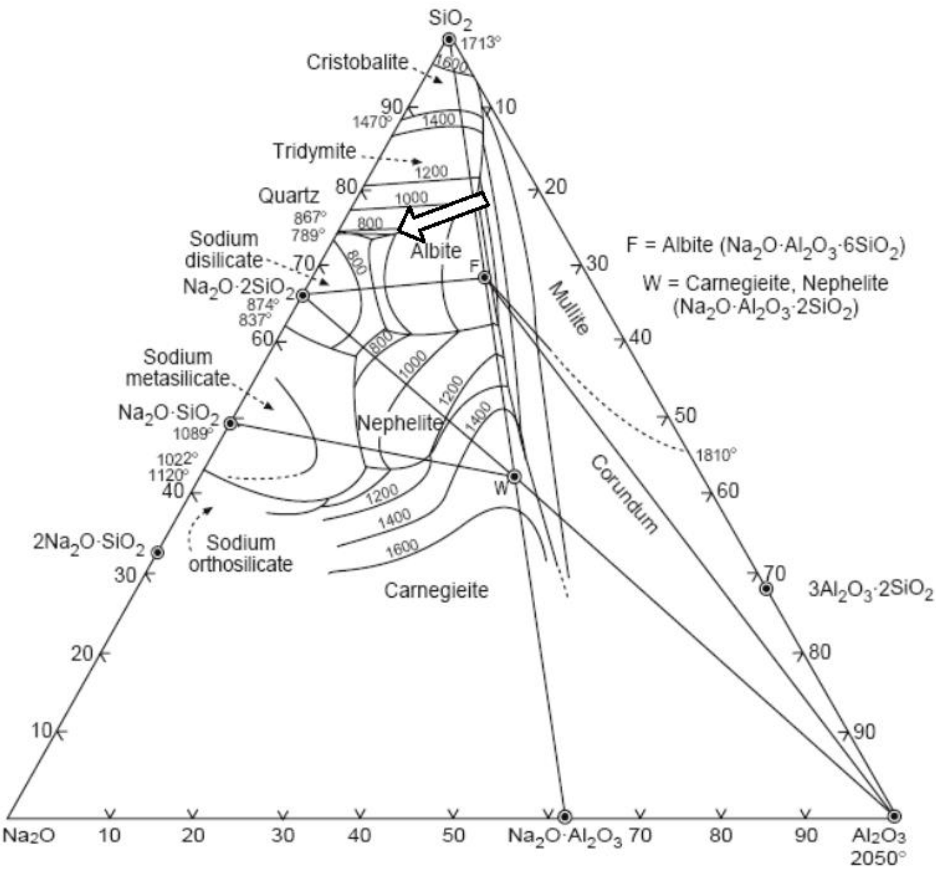

Figure 8. Phase diagram of «Na_2O-Al_2O_3-SiO_2» system (according to Osborne and Muang)

CONCLUSION

Coal combustion in thermal power plants produces large amounts of two types of wastes - ash and slag. The chemical composition of these wastes is represented by aluminosilicates and so they are suitable for use in the building industry. The great efficiency of ash and slag waste use in the production of building materials is proved by numerous scientific studies and practical experience. There are following advantages of using ASW instead of natural materials:

- Low cost of the material (it is actually determined by the cost of transportation to the construction site for slag accumulated in the dumps);
- The release of areas from under the dumps;
- The ability to save natural resources and reduce the burden on the environment.

Therefore, the ash and slag waste is quite valuable, promising and relatively cheap raw material for building materials industry. The preparation of silicate materials using slag waste is one of the most promising and cost-effective waste utilization areas.

Applications of TPP waste in the building industry are listed. The main areas of use are the production of binders, piece products manufacture, and road construction. In addition, a new trend of using ash and slag is revealed – the production of thermal insulation materials using foam glass technology – foamed slag glass. Further studies showed that the use of pure slag in this case is optimal. It was found that producing foamed slag glass compositions with high slag requires introduction of fluxes – materials reducing the melting point of the batch.

The peculiarities of flux influence on the structure of foamed slag glass was considered. It was found that the best fluxes among the tested materials were borax and sodium fluoride. Introduction of these fluxes in the ratio «$Na_2B_4O_7$: NaF = 8 : 2» leads to the formation of the uniform porous structure with a density of 430 kg/m^3. Introduction of sodium fluoride is the singularity of this composition, because NaF plays the role of an active component that interacts directly with a silicon-oxygen skeleton, accelerates the batch melting process and simplifies the porous structure formation. Additionally, it was proved that introduction with the fluxing mixture of additional Na_2O in «Na_2O-Al_2O_3-SiO_2» system shifts equilibrium closer to low-melting compounds formation.

This research should be continued for further research of physical and chemical processes of interaction between the batch components and fluxes, as well as clarification of the porous structure formation mechanisms in the process of batch heating.

The developed compositions can be used in the construction for the energy efficient buildings, with both bearing and insulating properties.

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