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INVESTIGATION OF THE PROCESS OF PORE FORMATION BASED MATERIALS HYDROSILICATES

Abstract: *Research porosity thermal insulation of refractory materials is the important task of power engineering, because the thermal conductivity of porous materials depends on the shape and especially location of pore.*

Analytical review of existing technologies shows that research in this area focused on the study of a process separately and generalized theories is not sufficient to clear analysis and model building process heat mass transfer of alumina porous material. Experimental and generalization of the characteristics of heat and mass transfer in porous materials that swelling is actual scientific problem.

In this paper analyzes the different composition of aluminous minerals, aluminum effect of additives on the formation of pores, as well as the influence of various impurities on the thermal conductivity of the material. The effect of temperature on the thermal conductivity of porous materials.

Keywords*: thermal conductivity, porosity, swelling, heat insulation.*

Introduction

Argil – widespread rock formation unstable composition and physical properties. Pure argil – clay without any impurities, is rock consisting of small dispersed particles of a certain chemical composition (which includes the base hydroaluminosilicates). Argil has a property to become plastic when saturated with moisture and maintain its form during drying. Argil is a silicate which includes alumina, silica, bound water, sand, lime carbonate, etc. The density of the argil is usually in the natural humidity of 25% the total of 1500÷1600 kg/m^3. From argil made such important heat insulation materials such as brick and ceramsite. The properties of these materials depend on the chemical composition of particulate matter fraction included in its composition [1].

Many experimental data indicate the presence of relationship between the porosity of the material and its thermophysical properties [2]. Influence of porosity on the thermal conductivity of the material can be considered following the example of experimental data [3]. The values of thermal conductivity of iron (58.19 W/(m·K)) and a rock formation (3.26 W/(m·K)) are different almost 18 times, but the filling of iron balls and balls a rock formation of the same porosity of 62.5% has nearly the same coefficient of thermal conductivity (0.0403 W/(m·K) and 0.0402 W/(m·K) respectively. However, the way of forming the porous structure has not yet been investigated, and accurate relationship between the porosity and the physical properties of the material not found.

In [2] analyzed the basics the formation of pores, but there is no end link of the form of the porous structure of a material with its thermophysical characteristics. It is also still not clear how necessary to evaluate the porous structure. Most authors studying porous materials are evaluated only quantitative indicator - porosity. The question of the adequacy of the criterion has not yet been raised.

The main part of research

To conduct a series of experiments on the swelling of hydrosilicates chosen argil different places of birth, with a different chemical composition. For the heat treatment used muffle furnace with thermocouples HC-0.1.

The used argil was classified visually ductility and the results are summarized in table 1. We used the following scale:

0 – dry powder,

1 – not plastic, at low load is divided into small pieces,

2 – not plastic, at low load is divided into large pieces,

3 – not plastic, destroyed only under heavy load,

4 – plastic.

TABLE 1. *The characteristics of the argil*

No. sample	Colour	The plasticity	The presence of impurities
1	yellow	3	small
2	dark grey	2	medium
3	mustard	4	small
4	mustard	3	medium
5	gray	3	large
6	gray	3	large
7	gray	3	large
8	gray	3	large
9	mustard	4	medium
10	dark grey	3	medium
11	white argil	0	not available

These samples were filled to the maximum humidity. A portion of each sample was dried and rapid indirect method (10 minutes of drying time, oven temperature 130°C) was determined by the humidity of the samples (table 2). The color of the resulting material can be seen on the progress of the main reactions. So red-brown color of the material will indicate oxidation processes, dark gray color of the recovery process.

The dried samples had the following characteristics:

- part of the sample No. 1 - color - yellow mustard, appeared pores fragile;
- part of the sample No. 2 - color - has not changed, visible small pores and cracks, more robust;
- part of the sample No. 3 - color - yellow mustard, you can see a few small pores;
- part of the sample No. 4 - color - yellow mustard, clearly visible pores and layers, brittle;
- part of the sample No. 5 - color - has not changed, visible layers;
- part of the sample No. 6 - color - has not changed, there is clearly split into two main layer;
- part of the sample No. 7 - color - has not changed, there were pores and cracks;
- part of the sample No. 8 - color - mustard-gray, appeared pores and layers;
- part of the sample No. 9 - color - yellow mustard, were pores and cracks;

- part of the sample No. 10 - color - has not changed, visible small pores and cracks;
- part of the sample No. 11 - color - has not changed, clearly visible spherical pores, not available layers and cracks, brittle.

***TABLE 2.** Determine the moisture content of the initial mixture*

No. sample	Weight before drying	Weight after drying	Weight of absorbed water	Humidity W,%
1	12.9	12	0.9	6.9767
2	17.7	16.9	0.8	4.5198
3	20.2	11.4	8.8	43.564
4	17.1	14.3	2.8	16.374
5	15	10.4	4.6	30.667
6	13.7	9.5	4.2	30.657
7	20.8	14.6	6.2	29.808
8	17.9	13.1	4.8	26.816
9	19.5	13.9	5.6	28.718
10	10.4	7.3	3.1	29.808
11	10.4	6.5	3.9	37.5

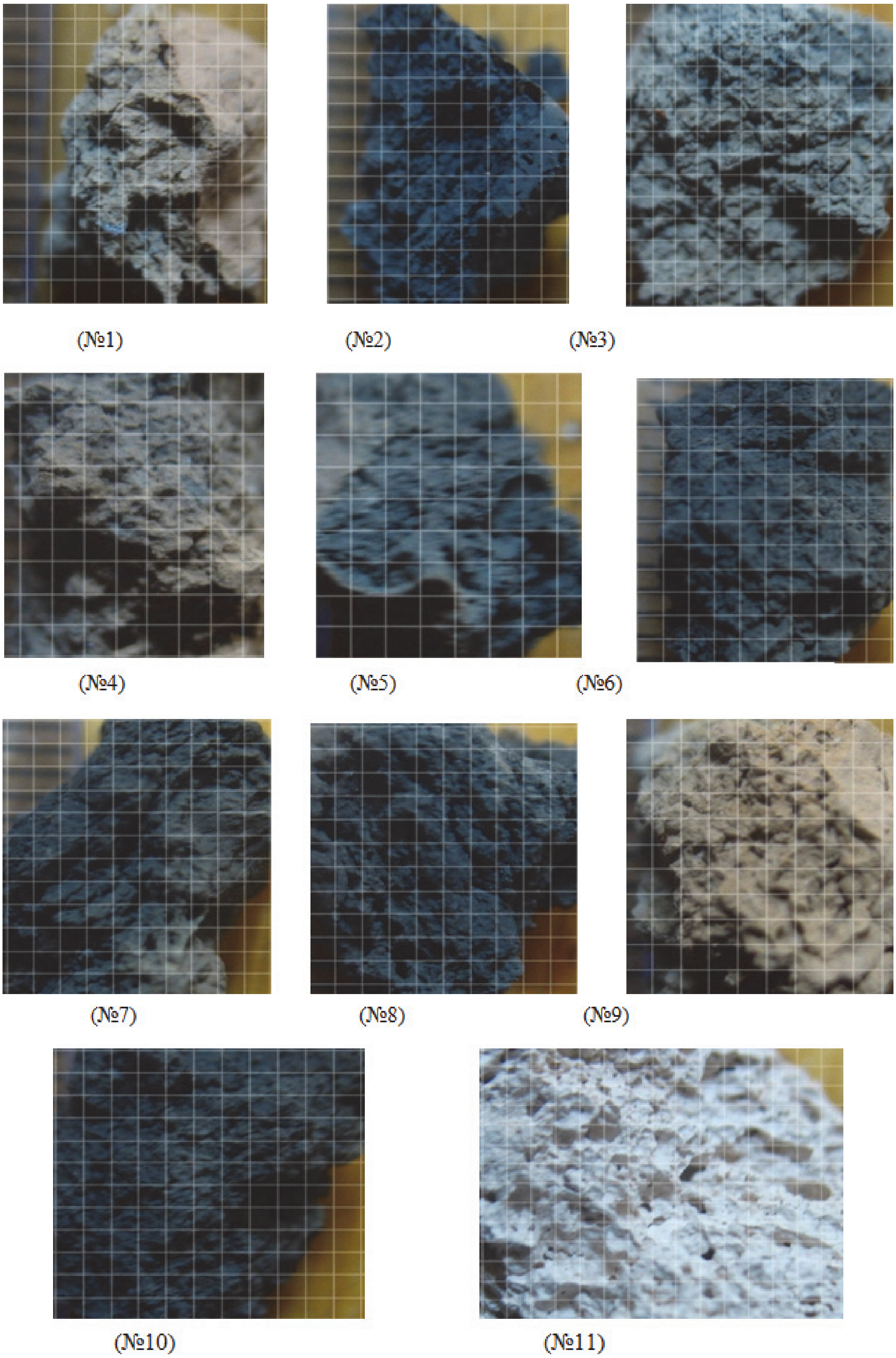
From the experiment it is seen that the evaporation of moisture causes the formation of pores inside the argil. It has highest porosity pure argil, wherein the pores are spherical in nature. The presence of impurities reduces the porosity of the material, as it increases the viscosity of argil. It should be noted that the impurities will also affect the shape of the pores. The pores are becoming stretched perpendicular to the lines of diffusion of moisture, the material is separated into individual layers. A large number of impurities leading to formation of cracks. This is due to the uneven distribution of impurities in volume, thus creating different tensions within the material in pore formation. Also impurities increase the final strength of the material.

To determine the effect of triatomic gases swelling process samples available swelling at 750°C for 8 minutes. This temperature is enough for allocation the gases, but it is lower than the melting temperature of the argil. After heat treatment, all samples were observed following changes: pore – are bigger than in the previous experiment, but is not spherical; sometimes there is a great time to within the material; the material becomes more durable, that is connected with chemical reactions; there are more distinct crack; material from pure argil is still fragile, but more irregular pores, and various size.

Investigation of the effect of additives on aluminum swelling of argil

Presumably aluminum must enter into relationship with hydroxides and water, thereby forming a new chemical compound. The increase in aluminum oxide will change the structure and physical properties of argil. The samples were saturated with moisture, and they added different amounts of aluminum. The heat treatment temperature is equal to 750°C, time 8 minutes. The experimental data are listed in the tables 3 and 4. Moisture and devolatilization gases was determined by the indirect method, accelerated.

Results of the experiment are summarized in figure 1 grid is applied to calculate the porosity. Graduation 1 mm.


(№1) (№2) (№3)

(№4) (№5) (№6)

(№7) (№8) (№9)

(№10) (№11)

Figure 1. *Expanded samples of argil with the addition of aluminum*

Expanded samples had the following characteristics:

- part of the sample No. 1 - a color in the middle of - sand, crust color - yellow with orange flavor, closer to the center there is separation of layers (elongated wedge-shaped pores), harder than without the addition of aluminum;

- part of the sample No. 2 - color - black, visible small pores and cracks, pores are quite a bit larger than the pore formation of moisture, more robust; aluminum particles are seen, which did not react with the silica;
- part of the sample No. 3 - color - sandy, more clearly visible pores, there are small cracks, well traced the inner layer with low porosity;
- part of the sample No. 4 - color - gradient from gray to yellow-orange, clearly visible pores of different sizes, more durable;
- part of the sample No. 5 - the color of the outside - orange hue color of inside - gray, fine porosity, and visible separation into layers, in some places there are large pores;
- part of the sample No. 6 - color - black, visible small pores and cracks, a strong, visible aluminum particles that do not react with the silica;
- part of the sample No. 7 - color outside - dark yellow inside color - black, clearly visible separation into layers, the surface layer of small spherical pores visible, the aluminum particles are visible which are not reacted with the silica;
- part of the sample No. 8 - color outside - gray inside color - black, clearly visible separation into layers, the surface layer of small spherical pores visible, the aluminum particles are visible which are not reacted with the silica;
- part of the sample No. 9 - color - yellow-orange with a small plot of gray inside the sample, clearly visible pores and small cracks;
- part of the sample No. 10 - color outside - gray visible small spherical pores, but the pores predominate vermiculite, aluminum particles are seen, which did not react with the silica;
- part of the sample No. 11 - appeared in some places a shade gray in color, the pores are larger, there was strongly pronounced channel porosity. Channel porosity occurred during draining different pores.

After the experiments it can be concluded that aluminum in argil with a large amount of impurities and a high content of humus practically does not react and no effect on swelling of. In the argil with a small amount of impurities – small additions of aluminum is slightly increased strength properties and slightly increases the porosity. In pure argil small additions of aluminum greatly increase pore formation. In general, all the samples of aluminum additives do not affect the shape of the pores. In samples where aluminum is not reacted, it can be seen near the germ pores aluminum particles. In pure argil all also absent vermiculite pores.

TABLE 3. *Moisture and the amount of volatile gases in the test samples*

No. sample	Humidity W, %	Weight of the pure sample before drying, g	Al weight before drying, g	Al weight after drying, g	Weight Al, g	Al, %	Vro$_2$+W, %	Vro$_2$, %
1	6.9767	6.1	6.3	4.1	0.2	3.1746	34.921	27.944
2	4.5198	3.5	3.7	2.5	0.2	5.4054	32.432	27.913
3	43.564	5.1	5.2	2.7	0.1	1.9231	48.077	4.5126
4	16.374	6.3	6.5	4.6	0.2	3.0769	29.231	12.857
5	30.667	7.3	7.4	4.7	0.1	1.3514	36.486	5.8198
6	30.657	6.8	6.9	4.5	0.1	1.4493	34.783	4.1257
7	29.808	9.8	9.9	6.6	0.1	1.0101	33.333	3.5256
8	26.816	7.6	7.7	5	0.1	1.2987	35.065	8.2493
9	28.718	5.6	5.8	3.6	0.2	3.4483	37.931	9.2131
10	29.808	6.7	6.8	4.3	0.1	1.4706	36.765	6.957
11	37.5	7	7.2	3.4	0.2	2.7778	52.778	15.278

***TABLE 4.** The porosity and frost resistance of test samples*

No. sample	The number of pores per 20 mm²						Limit the number of cycles of wetting - drying	Limit the number of cycles freeze defrosting
	vermiculite			spherical				
	<1 mm	1-3 mm	>3 mm	<1 mm	1-3 mm	>3 mm		
1	7	1	0	11	0	1	15	17
2	6	1	1	0	0	0	7	9
3	10	1	1	10	1	0	10	11
4	4	0	0	25	3	0	8	9
5	5	1	0	7	1	0	15	13
6	12	1	1	21	0	0	9	8
7	5	4	1	1	0	0	15	14
8	13	3	1	6	0	0	12	13
9	6	1	0	8	2	0	12	13
10	4	2	0	7	0	0	11	11
11	0	0	0	28	4	1	5	3

Determination of the effect of porosity on thermal conductivity

Previously described numerous analytical and empirical studies of the effect of porosity and bulk density on the thermal conductivity of the final material or layer. It was shown that all dependencies have their uses and are not suitable for general cases. Some dependencies absolutely not suitable for calculating the thermal conductivity of porous material. Therefore, experiments were conducted to determine the thermal conductivity of different porous materials. We investigated samples of expanded argil (sample number 2, 3, 4), the sample $3CaO \cdot Al_2O_3 \cdot 6H_2O$ (sample No. 5) and pressed gypsum powder (sample No. 1). Samples of expanded argil differed only by the temperature of heat treatment. The temperature of the heat treatment of the sample No. 2 was 800°C, sample No. 3 – 650°C, sample No. 4 – 750°C. Measurements of thermal conductivity were performed on IT-λ-400. This measuring instrument is designed to study the temperature dependence of the thermal conductivity of solid, machined materials in the heating mode, a monotone, which allows one to get the experiment right temperature dependence of the studied parameters and provide high performance. The theoretical basis of the method described in [4].

Working calculation formulas for the thermal resistance of the sample and its thermal conductivity are given below [5].

The thermal resistance of the sample was the following formula

$$P_0 = \frac{\nu_0 \cdot S \cdot (1+\sigma_c)}{\nu_T \cdot K_T} - P_k$$

where:

K_T – coefficient of proportionality is characterized by the effective thermal conductivity of the plate (constant value for the device is in the calibration), W/K,

P_k – thermal contact resistance (constant value for the device),

σ_c – the amendment taking into account the heat capacity of the sample, the contribution does not exceed 10%,

v_0 – temperature difference across the sample, K,

v_T – temperature difference on the plate, K.

The amendment takes into account the heat capacity of the sample was determined by the following formula [6]

$$\sigma_c = \frac{C_0}{C_0 + 2C_r}$$

where:

C_0 – the total heat capacity of the sample, $C_0 = c_0(t) \cdot m_0$;

C_r – the total heat capacity of the rod, $C_r = c_c(t) \cdot m_r$;

c_0 – the approximate value of the mass specific heat of the sample, J/(kg·K);

c_c – the value of the mass the specific heat capacity of copper, J/(kg·K);

m_0 – sample weight, kg;

m_r – weight of the rod, kg.

This formula differs from the formula proposed by [5]. Its choices are substantiated by the fact that calibration ratio is calculated before it under this formula.

The thermal conductivity of the sample is determined by the following formula

$$\lambda = \frac{h}{P_0}$$

where *h* is sample height, m.

The calculated values of the thermal conductivity of the sample were attributed to the average temperature of the sample, which is determined by the formula

$$\bar{t} = t_c + 0.5 \cdot A_t \cdot n_0$$

where:

$\bar{t}$ – the average temperature of the sample, °C;

t_c – the temperature at which the measurements, °C;

A_t – the sensitivity of the thermocouple CA, K/mV;

n_0 – temperature difference across the sample, mV.

The results of experiments and calculations were analyzed. With an increase in temperature from °C to 275°C the thermal conductivity of the porous material is increased. Moreover, the thermal conductivity pure Al_2O_3 when the temperature rises (within the specified range) should decrease [7]. This confirms the possibility of regulating thermal conductivity by increasing the content of Al_2O_3. But based on previous experiments, it can be argued that the increase in content Al_2O_3 into alumina will be justified only when a minimum of impurities.

For all of the samples characteristic that all of the temperature dependence of the thermal conductivity of most accurately describes the logarithmic dependence.

Figure 2 shows the dependence of the thermal conductivity of five samples of the temperature. From this graph it can be concluded that the behavior of the thermal conductivity of all the samples of the same, but the dependence of aerated concrete has a gentle nature, i.e. It is minimally dependent on the temperature.

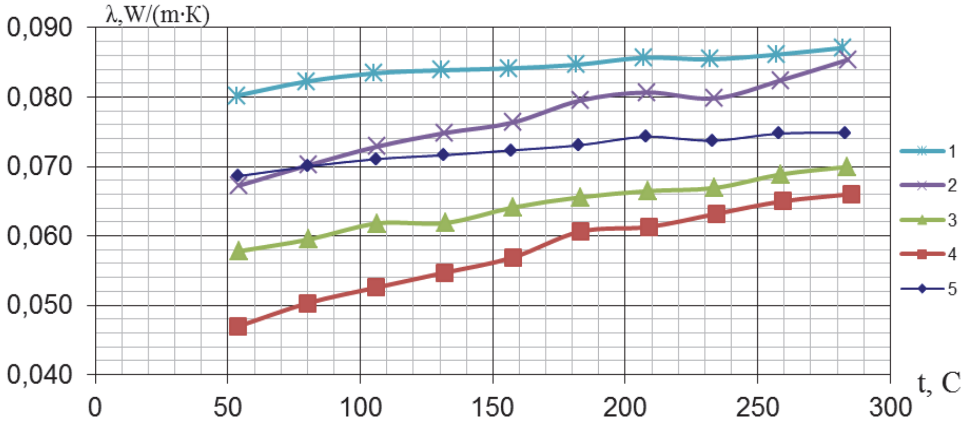


FIGURE 2. *Dependence of thermal conductivity of samples on temperature*

Dependence of the temperature swelling argil on the thermal conductivity is ambiguous.

The minimum thermal conductivity was achieved at 750°C. Moreover during the heat treatment of 650°C slightly higher than the thermal conductivity of the material, and at 800°C – substantially higher than twice. This is due to the uniformity of the pore distribution and the same size during the heat treatment of 750°C. Thus, when the heat treatment of 650°C – less uniform pores and smaller in size (sample less porous). At 800°C – a considerable increase in for some time, but disappear smaller pores and the total porosity of the material at greater than 750°C heat treatment, but the porosity is non-uniform. Also at 750°C and 800°C will be leakage of different chemical reactions that also significantly affected the results.

It can be concluded that the uniformity of the porosity has a significant effect on the thermal conductivity of porous building materials.

Conclusions

The presence of impurities in the intumescent of aluminous materials reduces the of the final porosity material. The pores are becoming stretched perpendicular to the lines of diffusion of moisture, the material is separated into individual layers. A large number of impurities leading to formation of cracks.

The amount of aluminum in the alumina has different effects on the process of swelling. The alumina with a large amount of impurities – aluminum practically does not react and do not affect the swelling. In the argil with a small amount of impurities – small additions of aluminum are slightly increased strength properties and slightly increase the porosity. In pure argil small additions of aluminum greatly increase pore formation. In general, all the samples of aluminum additives do not affect the shape of the pores.

Small content of Al_2O_3 mixture increases the duration blistering, and also increases the viscosity of the mixture. The iron content in the intumescent mixture should be kept to a minimum.

Dependence of the temperature swelling argil in the final thermal conductivity of the material is nonlinear.

The uniformity of porosity has a significant effect on the thermal conductivity of porous building materials.

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