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SHS membranes based on materials of mica-like structure

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Abstract. The possibility of synthesizing a functional porous material of a mica-like structure to manufacture filter membranes for purification of liquids and gases, in particular tap water, was investigated. Mixtures of available powders based on mineral raw materials — quartz sand (SiO_2) and Karelian schungite with reducing agents and secondary cryolite, a product of processing of fluorine-containing wastes of aluminum production, were used for the synthesis. The synthesis was carried out in the furnace at $T = 930°C$ in the atmosphere of air at $P = 1$ atm. A ceramic filter membrane 40 mm in diameter and ~ 5 mm in height was synthesized. The size of the open pores in the synthesized sample, which is measured according to GOST 26849-86, is 1–3 µm. The bending strength is 5.5–6 MPa. The product has a porosity of ~ 60% and an open porosity of up to 98%. The synthesized sample efficiently purifies the tap water from the high content of heavy metal ions and reduces the hardness of the water by 30%.

1. Introduction

Man-made burden on the environment can lead to irreversible changes in the composition of natural water. In particular, surface and underground water sources are contaminated by substances of anthropogenic origin - heavy metal ions, nitrates, volatile organochlorine compounds, herbicides, pesticides, radionuclides, etc. [1, 2]. Therefore the research aimed at purification of drinking water, in particular development of filter technologies using various materials, is relevant.

A comparative analysis of characteristics of filters manufactured in different countries [3] shows the expected advantages of SHS filters, especially multiple application after regeneration. It is impossible for the filters produced by traditional methods.

Fluoroflogopite is known to have a high ion-exchange capacity and can be used for heterovalent replacement of Na+ ions with metal ions that determine the water hardness (Ca2+, Mg2+, Sr2+, Ba2+) [4–7]. The authors show the possibility of SHS of a porous material based on fluoroplogopite [8, 9].

Introduction of up to 20% of CuO and Cr_2O_3 into the porous sample with the formation of spinels based on them will allow us to obtain catalytically active membranes for dehydrogenation of hydrocarbons [10] and catalytic oxidation of carbon monoxide [11].

The aim of this study is to create highly efficient, corrosion-resistant SHS membranes for purification of liquids and gases in both industrial enterprises and small plants. They allow adjusting the salt composition of water and ensuring the active human activity at its consumption.

2. Experimental methods

Quartz sand (SiO_2), Karelian schungite (Schung, table 1), aluminum PAD-1 (GOST 6058-73), and magnesium MPF-3 (GOST 6001-79) were used as initial elements in the experiments. Recycled cryolite Na_3AlF_6 was used as a source of fluorine, potassium perchlorate $KClO_4$ (TU 6-09-3801-76) – as an oxygen source. Schungite is used due to the high content of silicon oxide, as the main reagent, and carbon, which forms an oxide loosening the product. Some amounts (up to 5%) of iron and chromium oxides were introduced into the mixture as catalytically active additives. The quartz sand was crushed into powder of less than 150 and 50 μm in ball drums. The particle size was measured using a MicroSizer 201 analyzer.

Table 1. Schungite chemical composition.

1	Silicon (Si, weight %)	25
2	Oxygen (O, weight %)	30
3	Carbon (C, weight %)	32

The mixtures which can react in the SHS mode were used. The initial mixture composition was estimated by the following chemical scheme:

$$SiO_2 + Shung + Al + Mg + Na_3AlF_6 + KClO_4 \rightarrow NaMg_3Si_3AlO_{10}F_2 + KCl\uparrow + CO\uparrow \quad (1)$$

The initial billets 40 mm in diameter and ~5 mm in height were sintered in the furnace at T = 930°C in the air of atmospheric pressure. The temperature was being raised during 1 h. The exposure time at 930°C was 10 min. The phase composition of the product was determined using a DRON-3M diffractometer with Cu K_α radiation. The quantitative elemental composition of phases was carried out using a Zeiss Ultra Plus high-resolution SEM with INCA 350 Oxford Instruments X-ray microanalysis system.

The porosity of the synthesized material was determined in accordance with GOST 2409-80. The size of the open pores of the synthesized material was determined according to GOST 26849-86. The method consists in determining the minimum gas pressure required for forcing a gas bubble through the pores of a flat hydrophilic membrane impregnated with water or alcohol. The bending strength was measured on the installation Instron. The concentration of chemical elements in tap water before and after filtration was determined by the atomic absorption method. In order to reuse the synthesized membranes, they were regenerated by a reverse pulsed flow of the purified water.

3. Results

The overall view of the synthesized sample is shown in figure 1. The main phases of the synthesized material are those of spinel and fluorophlogopite in approximately equal mass fractions (figure 2). At the same time, the presence of unidentified lines indicates a more complex composition of the material.

Figure 1. Overall view of the synthesized sample.

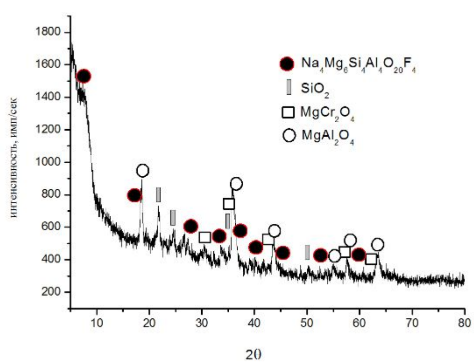

Figure 2. Phase composition of the synthesized material.

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Figure 3 demonstrates the porous structure of the synthesized material. According to the analysis, the material porosity is 60%; the open porosity is 98%. The measured value of open pores (according to GOST 26849-86) in the synthesized material is 1–3 μm. The bending strength of the sample is 5.5–6 MPa.

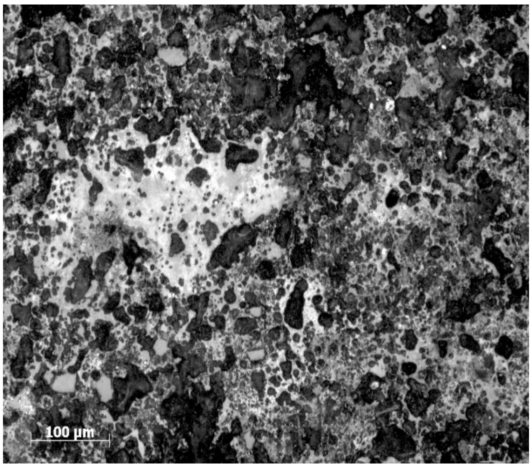
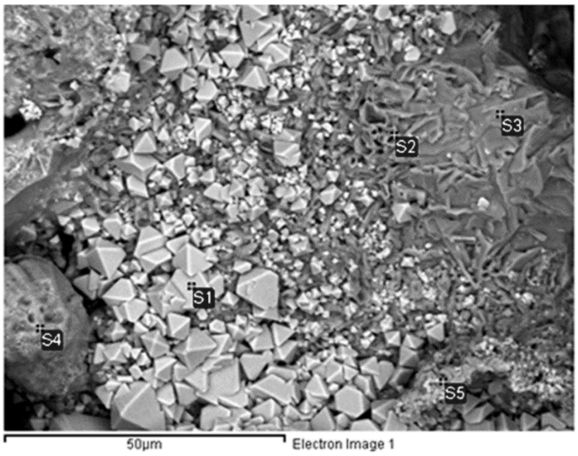


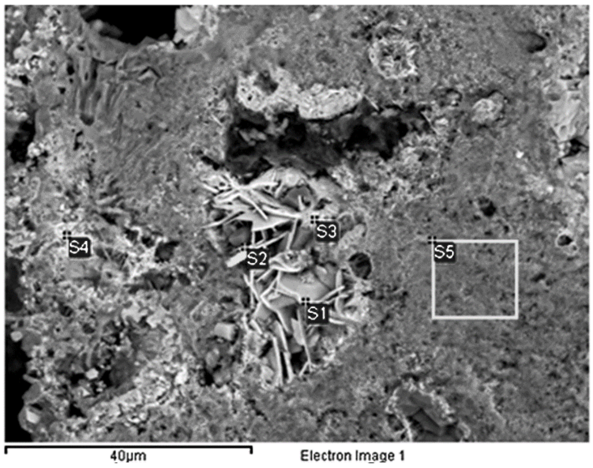
Figure 3. Synthesized material structure.

The elemental composition of the material is shown in figures 4 and 5. The analysis confirms the formation of spinel based on Cr, Mg and Al (figure 4) and the presence of fluorophlogopite (figure 5) in the material composition.



Spectrum	O	Na	Mg	Al	Si	K	Ca	Cr
S1	32.40		6.67	0.71				60.22
S2	39.09	0.74	3.03	3.61	15.88	1.36	0.72	35.56
S3	45.08	1.13	3.48	12.68	16.69	1.75		19.20
S4	49.47		3.93		4.60		25.85	16.15
S5	37.38	0.51	4.64	4.79	12.40	2.04	0.62	37.63

Figure 4. Spinel elemental composition.



Spectrum	O	F	Na	Mg	Al	Si	Cl	K	Cr
S1	37.83			0.72	0.59	1.46			59.40
S2	35.85	18.78	1.00	4.86	1.31	6.51			31.69
S3	39.54			4.75	3.71	1.01	0.37		50.62
S4	41.75		2.07	1.21	5.47	1.82		0.40	47.27
S5	39.14	18.95	0.47	10.78	4.82	15.16	0.51		10.17

Figure 5. Elemental composition of mica-like matrix.

As a result of the work, laboratory devices were developed and experiments in water purification were carried out. Table 2 demonstrates the content of metal ions in the water before and after its filtration through the synthesized membrane. The results prove that the obtained sample of the filter purifies the tap water rather efficiently from the high content of metal ions and reduces the water hardness by 30%.

Table 2. Content of Mg and Ca ions in the water before and after filtration.

Chemical element	Concentration of chemical elements in the tap water, mg/l	
	Tap water before filtration	Tap water after filtration through the synthesized filter
Mg	23.706	16.856 16.343 15.963
Ca	81.350	63.276 62.735 63.186

4. Conclusions

Membranes with mica-like structure were obtained by the direct synthesis. Their pore size was 1–3.5 µm, porosity was ~ 60%, open porosity was up to 98%. It was established that the bending strength of the obtained samples was 5.5–6 MPa, the specific surface area was 1.203 m^2/g.

Multiple regeneration of filters can be carried out by a reverse pulsed flow of the purified water, washing with solvent or high-temperature annealing.

The investigation results can be used for the development of production technologies of porous mica-crystalline filters for fine purification of water and technological gases from dispersed micro-impurities.

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