THE EFFECT OF THE TYPE AND CONTENT OF CARBONATES ON THE CHARACTERISTICS OF POROSITY OF CERAMIC MEMBRANES

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The main issue considered in the work is the creation of a matrix for ceramic membranes, which would be distinguished by low cost and high porosity. In order to reduce the cost of ceramic membranes, a widespread approach was chosen, which is powerfully used by many researchers, namely the use of natural minerals - kaolin and saponite - as the main components of the matrix. Carbonates were used as pore generating agents to achieve the required porosity value. Three series of ceramic membranes (CM) differing in chemical composition were synthesized in the work: KP1, KP2, and KP3 series. The main composition of CM includes: kaolin, saponite, sodium silicate and carbonates. The effect of the type and amount of carbonates ($CaCO_3$, NH_4HCO_3, $(NH_4)_2CO_3$) as pore generating agents on the formation of the porous structure of the samples was studied. The type and content of carbonates varied in different samples from 8 wt% up to 40 wt%. The dry pressing method using pressure equivalent to 8 tons and 15 tons was used to form ceramic membranes. The main parameters by which the properties of ceramic membranes were evaluated: water absorption, total and open porosity. Standard methods were used to determine these parameters for synthesized samples. The chemical composition of the dry mixture of the ceramic membrane, for which the porosity is the maximum in the series of manufactured samples, was established. It was established that the addition of calcium carbonate components to the dry mixture increases the porosity of ceramic membranes and water absorption. The chemical composition of a ceramic membrane sample with the best indicators of total and open porosity in the KP2 series is given. The chemical and phase composition of samples of the KP2 series, as well as their structural and adsorption characteristics, were investigated by X-ray fluorescence and diffraction methods of analysis and low-temperature nitrogen adsorption-desorption. The effect of the addition of silicon carbide on the characteristics of the porosity of the samples was determined.

Keywords: *calcium carbonate, ceramic membranes, chemical composition, dry pressing method, pore generating agent, porosity*

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1. Introduction

The global problem of water pollution is becoming increasingly acute due to rapid industrialization. The need for effective purification of water of various origins, in particular in conditions of remote location of treatment facilities, requires the use of membrane technologies, such as allow creating convenient mobile membrane modular installations. Membrane methods are universal and free of many disadvantages of other water purification methods, such as oxidation, adsorption, coagulation and

flocculation (Yang, 2020; Delcolle, 2017). Membrane separation, in particular with the use of ceramic membranes, has a number of advantages: ease of operation and regeneration, absence of additional reagents, high productivity, long service life, etc. (Ha, 2015). Recently, the methods of using natural materials to create ceramic membranes are gaining increasingly popularity in order to make cheaper of CM, since the high cost of their production still remains one of the disadvantages of using CM (Kuzminchuk, 2023; Serhiienko, 2023).

One of the important properties of ceramic membranes is the porosity of the membrane matrix, which ensures its transport characteristics, and therefore its efficiency. The porosity of ceramic membranes is ensured by the addition of so-called pore generating agents. It is possible to regulate the overall porosity of the ceramic matrix by changing the type and amount of pore generating agent. For example, in work (Kaur, 2016) different amounts of calcium and sodium carbonate were added to kaolin-based ceramic matrices. The matrix composition was as follows: kaolin (55–95 wt%), Na_2CO_3 (0–40 wt%), $CaCO_3$ (0–40 wt%), boric acid (2.5 wt%), sodium metasilicate (2.5 wt%). The addition of calcium carbonate increased the porosity and chemical stability of the samples, and the addition of sodium carbonate increased the pore-density by reducing their diameter. It was concluded that the content of $CaCO_3$ is 20 wt % makes it possible to obtain a sample with a pore size of 0.5 μm, a porosity of 37 % and a bending strength of 48 MPa. This sample showed good results in the separation of oil-in-water emulsions – 98 % oil rejection at a transmembrane pressure of 103 kPa.

In (Simão, 2015), the authors studied the influence of the amount of limestone (10 wt% and 20 wt%) on the porosity and bending strength of ceramic bodies that were formed from kaolin, potassium feldspar ($KAlSi_3O_8$), albite ($NaAlSi_3O_8$), quartz and white clay in different ratios The obtained ceramic bodies showed an apparent porosity from 28 wt% to 32 wt%, flexural strength from 7 MPa to 29 MPa. A sample containing kaolin (50 wt%), limestone (20 wt%), potassium feldspar (10 wt%), albite (10 wt%) and quartz (10 wt%) showed the best microfiltration properties for removing suspended solids from liquid suspensions.

To prepare inexpensive ceramic matrices, the authors (Lorente-Ayza, 2015) used the following components: a Spanish clay mixture, chamotte taken from fired calcite and potato starch. Calcite was used as a pore generating agent in the amount of 15 wt% and 20 wt%. Studies have shown that such ceramic composites are cheap and can serve as microporous material with porosity values ranging from 28 % to 31 %.

The study (Aripin, 2022) shows the use of calcium carbonate from onyx stone as a pore generating agent in a ceramic membrane based on kaolin, zeolite, and silica xerogel composites. Four different samples were made with different calcium carbonate content (from 5 wt% to 30 wt%), the sintering temperature of the samples was 1200 °C. It was established that the volume fraction of interconnected pores depends on the content of calcium carbonate. The sample with its content of 30 wt% provides the largest volume fraction of homogeneously interconnected pores, which in turn provides the largest value of the permeate flow. The effectiveness of the separation properties of the membrane was tested in the process of

removing non-sugar impurities in coconut juice.

2. Materials and Methods

The following raw materials and reagents were used for the synthesis of samples of ceramic membranes: kaolin (kaolin from the Prosyansky deposit of primary kaolins in the Dnipro region (Ukraine)), saponite (saponite clay from the Tashkiv deposit in the Khmelnytskyi region (Ukraine)), sodium silicate (sodium siliceous (silicate), pure Na_2SO_3), calcium carbonate (calcium carbonate, pure $CaCO_3$ (Ukraine)), silicon carbide (silicon carbide black SiC 54S F22 (Ukraine)), ammonium carbonate (ammonium carbonate, pure $(NH_4)_2CO_3$ (China)), ammonium hydrogen carbonate (ammonium carbonate acidic, pure NH_4HCO_3 (Ukraine)).

2.1 Determination of porosity and water absorption

The membrane sample dried for two hours at a temperature of 105 °C was weighed (m_{dry}). The weighed sample was immersed in distilled water and vacuumed in a desiccator at a constant pressure of 1 bar for 30 min. The wet sample was weighed to four decimal places ($m_{satur.}$). Next, this sample was weighed in the state immersed in distilled water (m). The calculation of research results was carried out according to the following formulas (ISO 5017:1998, IDT):

$$W_{intake} = \frac{m_{satur.} - m_{dry}}{m_{dry}} \cdot \frac{\rho_{aqua}}{\rho_{solv.}} \cdot 100, \quad (1)$$

$$\rho_{cond.} = \frac{m_{dry}}{m_{satur.} - m} \cdot \rho_{solv.}, \quad (2)$$

$$P_{open} = \frac{m_{satur.} - m_{dry}}{m_{satur.} - m} \cdot 100, \quad (3)$$

$$P_{total} = \frac{\rho - \rho_{cond.}}{\rho}, \quad 4)$$

where W_{intake} is water absorption, %; $m_{satur.}$ − mass of the liquid-saturated sample, g; m_{dry} − dry sample mass, g; ρ_{aqua} – the density of water at a temperature of 20 °C (293K), g/cm^3; $\rho_{solv.}$ – density of the liquid-saturating the sample at ambient temperature, g/cm^3, for distilled water – a reference value; $\rho_{cond.}$ − conventional density, g/cm^3; m – the mass of the sample measured when immersed in a glass, g; P_{open} – open porosity, %; P_{total} – total porosity, %.

2.2 Dry pressing method

The method of dry pressing with subsequent sintering of the pressed samples was used to form ceramic membranes. For this, the substances included in the mixture were crushed, mixed in the appropriate proportions, the resulting powdery mixture was weighed and poured into a mold. Using a manual hydraulic press, a pressure equivalent to 8 tons or 15 tons was created for 10 min. After removing samples of round ceramic membranes from the mold, they were sintered at temperatures from 800 °C to 1100 °C to increase their strength. Heat treatment was carried out in air in a high-temperature muffle furnace at a heating rate of 1 °C/min to 95 °C with an isothermal hold for 30 min, at a rate of 3 °C/min to 350 °C and at a rate of 2 °C/min to 950 °C with the next exposure at the final temperature for 60 min. After that, the samples were cooled in air to room temperature.

2.3 X-ray fluorescence analysis

X-ray fluorescence analysis was performed using a mobile precision analyzer EXPERT 3L, INAM (Ukraine). The range of chemical elements (control range) detected by this analyzer is from magnesium (12Mg) to uranium (92U). The range of measurement of mass percentages (concentrations) of elements is from 0.005 wt% to 100 wt%.

2.4 Diffraction analysis

Diffraction analysis was performed on an X-ray diffractometer Rigaku Ultima IV (Japan) with CuKα radiation. The samples were examined in copper radiation (Cukα = 0.15418 nm, 40 kW, 30 mA). For this, the Bragg-Brentano focusing scheme was used. Research conditions: interval of the studied angles 2Θ = 5–80°, shooting step – 0.04°, exposure time at a point – 2 s; the duration of shooting one sample was 90 min. The phase composition and average crystallite size were calculated automatically using the PDXL software application based on standard maps (ICDD and PDF-2/Release 2011 RDB databases) and Scherer's formula (Lee, 2016).

2.5 The method of low-temperature adsorption-desorption of nitrogen

Nitrogen adsorption-desorption isotherms were obtained using a Quantachrome® Nova 4200e analyzer (USA). Nitrogen desorption was carried out at a temperature of 393 K. The mass of nitrogen adsorbed and desorbed by the sample under study was determined by the algorithm built into the software of the device, based on the Langmuir and Brunauer-Emmett-Teller (BET) isotherm equations.

3. Results and Discussion

3.1. The effect of the carbonate type on the porosity of ceramic membranes

To determine the chemical composition of the ceramic membrane corresponding to the largest value of total (P_{total}, %) and open (P_{open}, %) porosity, as well as the value of water absorption (W_{intake}, %) (paragraph 2.1), a series of membrane membranes was synthesized (paragraph 2.2). For this purpose, the type of pore generating agents ($CaCO_3$, NH_4HCO_3, $(NH_4)_2CO_3$), its content (15 wt%, 20 wt%, 8 wt% − 40 wt%), the pressing pressure of the mixture of components were changed (8 tons, 15 tons).

To determine the type of carbonate, the addition of which to CM samples affects the change in total and open porosity values, a series of KP1 samples was synthesized and studied. The constant basis of the mixture for this series of samples was a mixture of the following composition: kaolin (65 wt%), saponite (10 wt%), sodium silicate (10 wt%). Carbonates ($CaCO_3$, NH_4HCO_3, $(NH_4)_2CO_3$) were added to the given mixture in the amount of 15 wt%. The pressing pressure applied during the formation of the ceramic membrane was 8 tons. The composition of the variable part of the samples of the KP1 series (carbonate content) is shown in Table 1.

Total, open porosity and water absorption in samples of the KP1 series were determined. It was established that sample KP1-1, which contains 15 wt% of $CaCO_3$, has the highest total porosity, namely − 42.95 %. The open porosity characteristic of this sample is 22.89 % and is the maximum in the series of KP1 samples. The water absorption for sample KP1-1 is 11.25 %, which is also the highest water absorption values among other samples of the KP1 series.

In order to further study the change in porosity of ceramic membrane samples, it was decided to synthesize a series of KP2 samples in which the content of carbonate pore generating agents was increased to 20 wt%. It was also decided to apply different pressing pressures of dry mixtures of membranes, namely 8 tons and 15 tons. The constant basis of the mixture was a mixture of the following composition: kaolin (60 wt%), saponite (10 wt%), sodium silicate (10 wt%). The mass percentage composition of the variable part of

the starting mixtures for CM of the KP2 series (carbonate content) is shown in Table 2.

The study of the synthesized samples of the KP2 series showed an increase in the values of total and open porosity in the samples with an increase in the content of the pore generating agents in the initial CM mixtures.

Table 1. *Carbonate content in CM samples of the KP1 series (pressing pressure – 8 tons)*

Sample	wt% ($CaCO_3$)	wt% (NH_4HCO_3)	wt% ($(NH_4)_2CO_3$)
KP1-1: Kaolin (65 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	15	–	–
KP1-2: Kaolin (65 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	–	15	–
KP1-3: Kaolin (65 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	–	–	15

Table 2. *Carbonate content in CM samples of the KP2 series (pressing pressure – 8 tons and 15 tons)*

Sample	Pressing pressure, tons	wt% ($CaCO_3$)	wt% (NH_4HCO_3)	wt% ($(NH_4)_2CO_3$)
KP2-8-1: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	8	20	–	–
KP2-8-2: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	8	–	20	–
KP2-8-3: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	8	–	–	20
KP2-15-1: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	15	20	–	–
KP2-15-2: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	15	–	20	–
KP2-15-3: Kaolin (60 wt%), Saponite (10 wt%), Sodium silicate (10 wt%)	15	–	–	20

The obtained results regarding the porosity of ceramic membranes of the KP2 series with different pore formers − ammonium carbonate, ammonium bicarbonate, calcium

carbonate (Table 2), testify to the positive effect of lower pressing pressure of dry CM mixtures on the formation of greater porosity in the samples. So, KP2-8-1 sample, which contains 20 wt% of $CaCO_3$ and was pressed under a pressure of 8 tons, has the greatest value the total porosity (46.93 %). A sample of similar composition (KP2-15-1), which was pressed under a pressure of 15 tons, had a total porosity − 40.47 %. The open porosity for samples KP2-8-1 and KP2-15-1 is 20.43 % and 13.72 %, respectively, which are the largest values in the series of samples and also indicate the positive effect of reducing the pressing pressure of dry CM mixtures on the formation of higher porosity.

The study of the porosity of samples of the KP2 series also showed that when using NH_4HCO_3 as a pore generating agents, it is possible to obtain values of the total porosity in the samples at the level of only 46.53 % and 42.88 % at pressing pressures of 8 tons and 15 tons, respectively. These values are lower than the value of this parameter in sample KP2-8-1, in which $CaCO_3$ was used as a pore generating agents. The use of $(NH_4)_2CO_3$ as a pore generating agents leads to a decrease in total porosity values to 42.18% (pressing pressure – 8 tons) and to 39.45% (pressing pressure – 15 tons). This result confirms the thesis that the optimal pore former in the studied ceramic membranes is $CaCO_3$ in the amount of 20 wt%, and the pressing pressure of dry mixtures is 8 tons. It should also be noted that the value of water absorption for sample KP2-8-1 is the largest among samples of this series, namely − 10.27 %.

Thus, it can be stated that sample KP2-8-1 has the best porous characteristics, in which water absorption is 10.27%, total porosity is 46.93%, open porosity is 20.43%.

3.2. Characterization of ceramic membrane samples

In addition, the sample KP2-8-1 (sintering of the sample was carried out at a temperature of 950 °C) was examined in more detail by X-ray fluorescence and diffraction methods of analysis (paragraph 2.3, paragraph 2.4). Table 3 shows the mass content of the main elements for sample KP2-8-1 according to the results of fluorescence analysis. The main elements for sample KP2-8-1 are Ca (63.06 wt%), Si (17.97 wt%), Al (10.46 wt%), Fe (6.04 wt%) and Ti (2.14 wt%), as well as the following elements were found in minor quantities: V, Mn, Sr, Zr, Ga, Cu, Zn, Rb, Y, Nb.

Figure 1 shows an X-ray pattern of sample KP2-8-1, which was identified as a mixture of kaolinite $Al_4(OH)_8(Si_4O_{10})$ and calcium and aluminum silicates $CaAl_{l1.77}Si_{2.23}O_8$, with crystallite sizes of − 22 nm.

Samples of ceramic membranes KP2-8-2 and KP2-8-3 were also investigated by the diffractometric method (Fig. 2). Peaks characteristic of kaolin are not observed for both samples, indicating the transformation of kaolinite into amorphous metakaolin at the sintering temperature. On the other hand, the peaks for quartz SiO_2 are clearly defined for both samples (Card ICDD 01-087-2096).

In addition, samples KP2-8-1, KP2-8-2, and KP2-8-3 were investigated by the method of low-temperature nitrogen adsorption-desorption on the surface and porous structure analyzer (paragraph 2.5). Table 4 shows the structural and adsorption characteristics of these samples (specific surface area (BET) − S, m^2/g, volume of mesopores − V_{mes}, sm^3/g, diameter of pores − D_{por}, nm).

Table 3. *Chemical analysis of ceramic membrane KP2-8-1*

Element	**Ca**	**Si**	**Al**	**Fe**	**Ti**	**V**	**Cu**	**Mn**
wt%	63.06	17.97	10.46	6.04	2.14	0,11	0.02	0,07
Element	**Sr**	**Zr**	**Ga**	**Zn**	**Rb**	**Y**	**Nb**	
wt%	0.04	0.03	0.02	0.01	0.01	0,01	0.01	

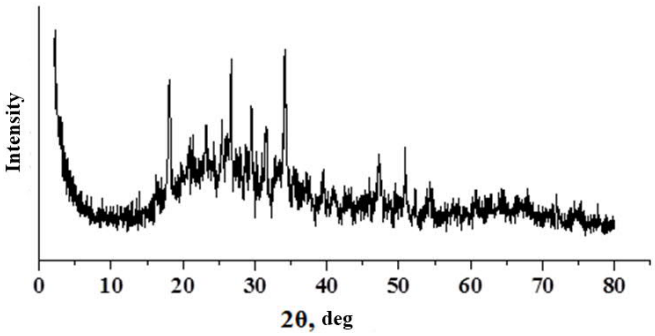

Fig. 1. *X-ray pattern of ceramic membrane KP2-8-1*

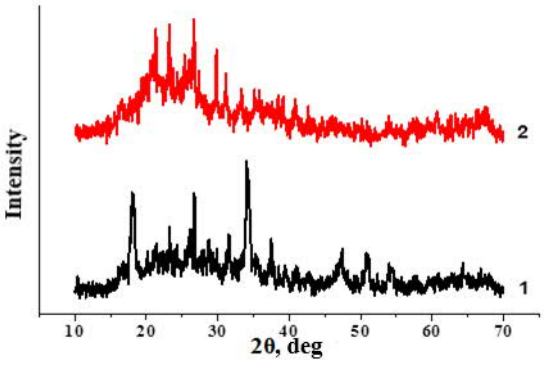

Fig. 2. *X-ray pattern of ceramic membranes of the KP2 series: 1 – KP2-8-2, 2 – KP2-8-3*

The obtained isotherms for these samples indicate that they are macroporous, and the values of the structural and adsorption characteristics of the samples do not differ significantly from each other.

Table 4. *Structural and adsorption characteristics of ceramic membrane samples of the KP2 series*

Parameters	**KP2-8-1**	**KP2-8-2**	**KP2-8-3**
S, m^2/g	4	6	5
V_{mes}, sm^3/g	0.86	1.28	1.10
D_{por}, nm	2.1	2.0	2.1

3.3. The effect of varying the carbonate content and the addition of silicon carbide on the characteristics of CM

Since the structural-adsorption analysis showed low values of the specific surface of samples KP2-8-1, KP2-8-2 and KP2-8-3, it was assumed that a further increase in the content of calcium carbonate in the samples can have a positive effect not only on the increase in porosity, but also therefore, in our opinion, to increase the specific surface of the samples. Thus, a series of KP3 samples were synthesized, in which the content of $CaCO_3$ in the composition of the initial mixtures of CM varied from 8 wt% to 40 wt%. In addition, it was decided to add 10 wt% silicon carbide to mixture, the presence of which in the structure of CM, according to the analysis of scientific literature over the last 10 years, is capable of increasing the mechanical strength of ceramic materials.

The formation of CM was carried out using a pressure of 8 tons. Table 5 shows the compositions of the starting mixtures for CM of the KR3 series.

Table 6 shows the values of water absorption and porosity of samples of CM series KP3.

According to the research results, the best porous characteristics are characteristic of sample KP3-3 (total porosity − 46.11 %

and open porosity − 27.97 %), but this sample loses its strength, as evidenced by the appearance of cracks in it, which are visible visually (Fig. 3 b).

In further studies, samples of the KP3 series, which contained more than 24 wt% $CaCO_3$ as pore generating agents, were found to be mechanically unstable and crumbled.

Table 5 *Mass-percentage composition of the starting mixture for CM samples of the KP3 series (pressing pressure – 8 tons)*

Sample	wt% (Kaolin)	wt% (Saponit)	wt% (Na_2SiO_3)	wt% (SiC)	wt% ($CaCO_3$)
KP3-1	62	10	10	10	8
KP3-2	54	10	10	10	16
KP3-3	46	10	10	10	24
KP3-4	38	10	10	10	32
KP3-5	30	10	10	10	40

Table 6. *Water absorption and porous characteristics of CM samples of the KP3 series (pressing pressure – 8 tons)*

Sample	*Water absorption,* W_{intake}, %	Open porosity, P_{open}, %	Total porosity, P_{total}, %
KP4-1	7.94	16.96	38.60
KP4-2	8.93	18.73	37.50
KP4-3	15.08	27.97	46.11
KP4-4	-	-	-
KP4-5	-	-	-

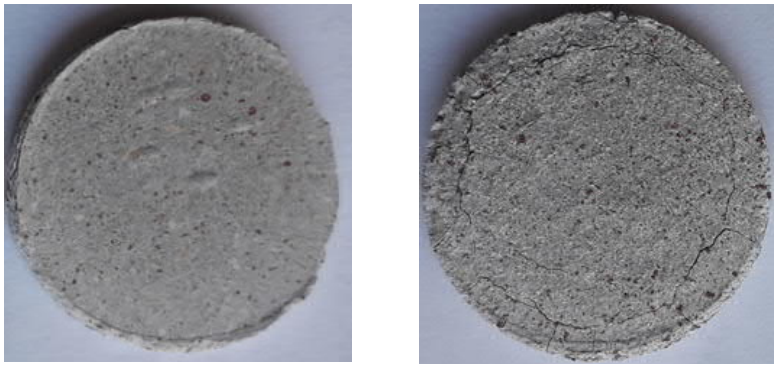

Fig. 2. *Photo of CM samples of the KP3 series*

a – sample KP3-1; b – sample KP3-3

The obtained results allow us to conclude that an increase in the content of $CaCO_3$ in the dry mixture by more than 24 wt% does not lead to an improvement in the characteristics of porosity and strength of ceramic membranes. Instead, the addition of silicon carbide in the amount of 10 wt% and increasing the content of calcium carbonate to 24 wt% allows to increase the value of open porosity to 27.97 % and the value of water absorption to 15.08 %.

4. Conclusions

In the work, the synthesis of three series of samples of ceramic membranes was carried out by dry pressing using a pressure of 8 tons and 15 tons with varying their chemical composition, namely the content of $CaCO_3$, NH_4HCO_3, $(NH_4)_2CO_3$ from 15 wt% to 40 wt%. Studies of total and open porosity, as well as water absorption of the samples showed that the best characteristics are provided by the use of calcium carbonate as pore generating agents in the amount of 20 wt%. The study of the chemical and phase composition of the samples of the KP2 series, as well as their structural and adsorption characteristics by the methods of X-ray fluorescence, diffraction analysis and low-temperature nitrogen adsorption-desorption showed that the specific surface of all samples is low (4−6 m^2/g). Addition of silicon carbide to the samples in the amount of 10 wt% and increasing the content of calcium carbonate to 24 wt% allows you to obtain a sample with best porosity and water absorption indicators, namely sample KP3-3 of the following composition: kaolin (46 wt%), saponite (10 wt%), sodium silicate (10 wt%), silicon carbide (10 wt%), calcium carbonate (24 wt%) (sintering at 950 °C, pressing pressure is 8 tons). For this sample, the water absorption value is 15.08 %, the total porosity is 46.11%, and the open porosity is 27.97 %.

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ВПЛИВ ТИПУ ТА ВМІСТУ КАРБОНАТІВ НА ХАРАКТЕРИСТИКИ ПОРИСТОСТІ КЕРАМІЧНИХ МЕМБРАН

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Основним питанням, яке розглядається в роботі, є створення матриці для керамічних мембран, яка б відрізнялася низькою вартістю та високою пористістю. З метою здешевлення керамічних мембран був обраний широко поширений підхід, який активно використовується багатьма дослідниками, а саме використання як основних компонентів матриці природних мінералів – каоліну і сапоніту. Для досягнення необхідного значення пористості використовували карбонати як пороутворювачі. У роботі синтезовано три різні за хімічним складом серії керамічних мембран (КМ): серії КР1, КР2 і КР3. До основного складу КМ входять: каолін, сапоніт, силікат натрію і карбонати. Досліджено вплив типу та кількості карбонатів ($CaCO_3$, NH_4HCO_3, $(NH_4)_2CO_3$) як пороутворювачів на формування пористої структури зразків. Тип і вміст карбонатів коливався в різних зразках від 8 % мас. до 40 % мас. Для формування керамічних мембран використовувався метод сухого пресування з використанням тиску, еквівалентного 8 т і 15 т. Основні параметри, за якими оцінювали властивості керамічних мембран: водопоглинання, загальна та відкрита пористість. Для визначення цих параметрів для синтезованих зразків використовували стандартні методики. Встановлено хімічний склад сухої суміші керамічної мембрани, для якої пористість є максимальною в серії виготовлених зразків. Встановлено, що додавання до сухої суміші компонентів кальцій карбонату підвищує пористість керамічних мембран і водопоглинання. Наведено хімічний склад зразка керамічної мембрани з найкращими показниками загальної та відкритої пористості в серії КР2. Хімічний і фазовий склад зразків серії КР2, а також їх структурно-адсорбційні характеристики досліджено рентгенофлуоресцентним, дифракційним методами аналізу та низькотемпературною адсорбцією-десорбцією азоту. Визначено вплив додавання карбіду кремнію на характеристики пористості зразків.

Ключові слова: *кальцій карбонат, керамічні мембрани, метод сухого пресування, пористість, пороутворювач, хімічний склад*