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## **SYNTHESIS OF BACTERICIDAL MICROFILTRATION CERAMIC MEMBRANES**

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### **Abstract**

*Membrane technologies have become widely used in filtration and separation processes in chemical, oil, food, pharmaceutical, medical, environmental, textile industries, etc. Recently, there is a growing demand for the use of bactericidal membranes due to their effectiveness in neutralizing microorganisms. The aim of this work is to synthesize microfiltration ceramic membranes modified with silver nanoparticles to provide them with bactericidal properties. In this*

*work, ceramic membranes are synthesized by dry pressing followed by sintering from natural raw materials of Ukrainian origin, namely: kaolin and saponite, as well as with the addition of silicon carbide, sodium silicate, and calcium carbonate. To provide ceramic membranes with bactericidal properties, they were modified with particles of silver. The synthesized ceramic membranes were characterized by X-ray diffraction and fluorescence analysis, and their bactericidal ability has been established. The ceramic filtration membranes after modification by silvers exhibited an inhibitory effect on the growth of Gram-positive (*B. subtilis.*) and Gram-negative (*Escherichia coli*) pathogens. Thus, the obtained bactericidal ceramic membranes are of great interest for their use for biofouling control.*

### **Keywords**

Bactericidal Ceramic Membranes, Microfiltration, Silver, Water Treatment

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## **1. Introduction**

Water is the basis of all organic life, without which neither human existence nor the development of mankind as a whole is possible. Along with the problem of limited water resources, the issue of their quality is no less acute. Many countries are at risk of a water crisis. It is anticipated that the need for access to water resources shall increase in the coming years (Duzdaban, 2021). Water pollution is a serious problem for many countries and needs to be resolved as soon as possible (Aini Zakaria, Wan Mansor, & Shahrin, 2018). Its solution requires the development of traditional methods and the creation of new methods and technologies. One of the most advanced methods of filtering water is its membrane purification.

Membrane technologies are a high-tech method of water purification and occupy a special place in the field of water treatment. This technology is used to filter water from mechanical and organic contaminants, bacteria and viruses, and in solution separation, water softening, wastewater treatment and production of sterile liquids. These processes find applications in chemical, oil, food, pharmaceutical, medical, environmental, textile industries, etc. Membrane filters are thin and have a fairly controlled pore size. Nowadays, the filter materials are usually polymers and ceramics.

Composite membranes have recently attracted attention as each layer can be independently modified to achieve the desired membrane characteristics (Hong Anh Ngo, & Thi Tran, 2017). In particular, ceramic membranes remain one of the most promising technologies among membrane processes due to their superior performance, hydrophilicity, mechanical robustness, and high thermal and chemical stabilities. Ceramic membranes are characterized by reliability and long

service life under routine conditions, as well as the possibility to restore the filtration properties by membrane regeneration, including backwashing. (Gitis & Rothenberg, 2016, Serhiienko, Dontsova, Yanushevskaya, Nahirniak, & Ahmad, 2020). Ceramic membranes are used for different types of filtration depending on the average pore size: ultrafiltration, microfiltration and nanofiltration. However, despite several advantages, ceramic membranes are not protected from biofouling. Even under correct operating conditions, it is inevitable that the membrane will become contaminated after prolonged operation. Membrane fouling is a serious problem in the application of a ceramic membrane, which is mainly caused by the deposition and adsorption of certain components in the filtrate at the membrane surface or in the pores of the membrane. Bacteria may also be growing in the pores of the membrane or on its surface, reducing the membrane's permeability and causing it to become contaminated (Zuo, Zhang, Ma, Lv, & Li, 2020). Therefore, the issue of the production of ceramic membranes endowed with bactericidal properties is of interest.

## **2. Literature Review**

Many works have been devoted to the study of the performance of ceramic membranes. Various kinds of inorganic membrane materials are available on the market. The properties of ceramic membranes are basically defined by their composition, the choice of pore formers and the temperature of heat treatment. According to the literature, most often ceramic membranes are made of Al, Ti, Si, and Zr oxides (Samaei, Gato-Trinidad, & Altaee, 2018). However, the production of membranes based on these materials requires high economic costs. To reduce costs, recent research has focused on the use of natural materials such as clays, natural zeolites, apatites, quartz sand, and others for the production of ceramic membranes (Abdullayev, Bekheet, Hanaor, & Gurlo, 2019). Clays are widespread around the world and require minimal processing before use in the production of ceramic membranes. Kaolin is the most common type of clay whose main mineral form consists of kaolinite. Kaolin-based ceramic membranes are characterized by mechanical stability and high porosity, which are achieved by heat treatment.

There are different methods for producing ceramic membranes from raw materials such as slip casting, pressing, extrusion, dip coating, sol-gel method, etc. The choice of any preparation method depends on the desired membrane structure and future use (Amin, Abdallah, Roushdy, & El-Sherbiny, 2016).

The traditional method of obtaining porous ceramic substrates is sintering powders with binders, which can be liquid glass, clay minerals (kaolinite, montmorillonite), aluminophosphate binder, organics and others. To increase the porosity of ceramics in some cases, burnout (sawdust, flour, starch) or gas-forming (calcite, magnesite) additives are introduced. By regulating the dispersion of powders, the amount and nature of binders, additives and the method of heat treatment of the mixture, a ceramic support with different porosity and permeability is obtained.

There are many research papers devoted to the study of membrane fouling control strategies. Membrane cleaning methods can be divided into physical methods (reversible fouling cleaning) and chemical methods (irreversible fouling cleaning). Physical methods are reagent-free methods of membrane cleaning, and include Hydraulic flushing, backwashing, backpulsing, air enhanced backflushing, use of ultrasound, use of electric field and others. Chemical cleaning of membranes is a so-called cleaning-in-place (CIP) using the following typical reagents: alkalis, oxidants/disinfectants, acids, chelating agents, surfactants and enzymes (Gruskevica & Mezule, 2021).

Ways to prevent membrane fouling include water pretreatment, timely hydraulic and chemical flushing, reagent dosing, or special membrane design. One way to prevent the biological fouling of ceramic membranes is to modify the membranes with certain metal nanoparticles, such as Ag and Cu, which are well-known for their antibacterial properties (Lu et al., 2016, Kim & Van, 2010). Previous investigations have shown the use of silver particles to reduce membrane biofouling using various methods of membrane production. Basically, there are two main ways of adding Ag nanoparticles (Ag NPs) to the membrane surface, namely, making a membrane from a mixture that contains Ag NPs in advance and modifying an existing membrane by the coating method. There is evidence in the literature that Ag NPs not only provide disinfection but also reduce contamination by reducing biofilms on the membrane surface (Bolto & Xie, 2018, Diagne et al., 2012, Le et al., 2019).

By incorporation of Ag nanoparticles (NPs) into the ceramic matrix, antibacterial properties can be added to the ceramic membranes. In (Ngoc Dung, Phan Thi, Nam, Nhan, & Quang, 2019) a highly porous ceramic filter with silver particles incorporated into its structure, obtained by the in situ chemical reduction method, was fabricated. For this purpose, the  $\text{AgNO}_3$  was first impregnated with activated carbon with a further addition to the ceramic filter production

mixture. In doing so, the silver particles are distributed evenly throughout the porous ceramic filter, allowing it to slowly release and maintain its antibacterial effectiveness for prolonged filtration.

In another research (Lv et al., 2009) to fix silver nanoparticles in porous ceramic composites using as a binder aminosilane binding agent, namely 3- aminopropyltriethoxysilane. After passing inlet water with a bacterial load of  $\sim 10^5$  colony-forming units (CFU) per milliliter through the silver-decorated porous ceramic at a flow rate of 0.01 l/min, no E. coli was detected in the output water.

In the paper (Singh, Rento, Son, Turner, & Smith, 2019), ceramic tablets were made in different ways by changing the pressure in the mold, temperature and sintering rates, sawdust composition, mixing protocols of starting materials and granulation. The prepared ceramic samples were investigated for the release of silver ions into water for periods of 8 to 24 hours.

An alternative method of adding silver to ceramic filters before sintering is compared in the literature with the traditional methods of applying silver particles after heat treatment of the product (Jackson, & Smith, 2018). The preparation of ceramic water filters by adding silver nitrate to the initial mixture of clay, sawdust and water can improve the retention of silver in the filter, support effective pathogen removal, reduce production costs, and minimize the risk of inhalation of silver nanoparticles by workers. Filters were evaluated using a series of mixing flow experiments using a non-pathogenic strain of E. coli and [ $^3\text{H}$ ]H<sub>2</sub>O.

Another major study (Mukaratirwa-Muchanyereyi et al., 2020) describes ceramic water purification filters made from clay, broken clay pots and flour, with the addition of silver nitrate in one case and silver nanoparticles in the other by immersing the formed discs in the respective solutions. The effectiveness in reducing the pH of the original water, water hardness, biological oxygen demand (BOD), chloride and nitrate concentrations after passing through the ceramic filters are compared. The paper (Kallman, Oyanedel-Craver, & Smith, 2011) mentions ceramic filters prepared by mixing clay, sawdust and water, followed by impregnation with silver nanoparticles. The filters have been successfully tested for the transfer and removal of E. coli under laboratory and field conditions.

Recently, Ag NPs on ceramic materials has attracted considerable attention due to their disinfection and reduction of biofouling for household (point-of-use) water treatment (Shepard, Lux, & Oyanedel-Craver, 2020). Ceramic filters containing sawdust and commercial clays and

similar filters with added AgNPs were evaluated for flow rate, turbidity removal, bacteria removal and silver leaching.

The aim of this work is to synthesize bactericidal microfiltration ceramic membranes. For this purpose, ceramic membranes were modified with silver nanoparticles. The effect of silver content on the bactericidal properties of the membrane is described.

### **3. Methodology**

Ceramic membrane supports were prepared by dry pressing followed by sintering. This method is suitable to produce flat round ceramic membranes for research of their properties. In general, ceramic membranes made by pressing have well-defined characteristics, such as uniform porosity and homogeneous physical properties over the entire membrane area and are widely used for fundamental research (Issaoui & Limousy, 2019). This method is based on pressing dry powder using a press. To impart strength, the formed membrane is calcined at a temperature sufficient for sintering the initial substances.

To prepare sample CM0, the feedstock was ground and mixed in the following ratio: Kaolin 60 %, Saponite 10 %, Sodium silicate 10 %, Calcium carbonate 20 %. 2 g of the mixture of powders was weighed and then poured into the 2.2 cm diameter press mould. Round ceramic membrane supports were made by hand hydraulic pressing at 8 tons of pressure. Heat treatment was performed in the air in a high-temperature muffle furnace at a heating rate of 1 °C/min to 95 °C with isothermal holding for 30 min, at a rate of 3 °C per min to 350 °C and at a rate of 2 °C per min up to 950 °C, followed by holding at this temperature for 60 minutes. Finally, the samples spontaneously cooled to room temperature.

Samples of bactericidal ceramic membranes modified with silver were prepared similarly, only at the stage of mixing the starting materials was added silver nitrate in terms of Ag in the following contents: 0.5 %, 1 %, 2 %, 4 % and 5 %.

X-ray diffraction analysis was performed using a Rigaku Ultima IV X-ray diffractometer (Japan) with CuK $\alpha$  radiation. The size of the crystallites was calculated using Scherer's formula.

The chemical composition was determined using an EXPERT 3L INAM X-ray fluorescence analyzer (Ukraine).

Test strains, such as Gram-negative bacteria *Escherichia coli* UKM B-906 and Gram-positive (*B. subtilis*) were obtained from Danylo Zabolotny Institute of Microbiology and Virology

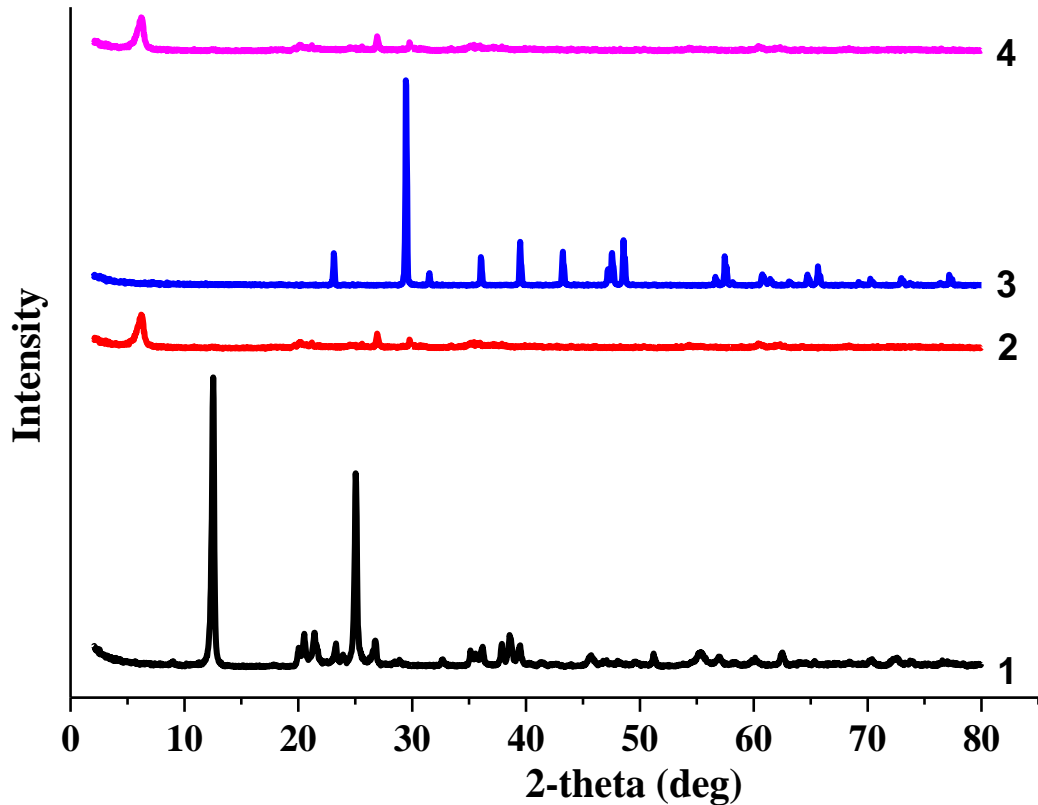
of the National Academy of Science of Ukraine. Before testing inoculums were cultivated in liquid medium meat-and-peptone broth for 6 hours at 37 °C. The inoculum suspension was diluted to an optical density of 0.5 according to the McFarland standard scale, which corresponds to  $10^8$  colony-forming units (CFU/cm<sup>3</sup>). The test was conducted by the diffusion method on a solid medium which is based on ceramic filtration membranes in nutrient agar and inhibition of bacteria growth. The inoculum was densely touched by swab on sterile agar in Petri dishes, after that the pieces of catgut threads were placed on the surface. Petri dishes were incubated at 37 °C for 24 hours. Evaluation of antibacterial activity was provided by measuring of size of the inhibition zones around the threads.

## **4. Analysis**

The ceramic membrane CM0 and its initial components, namely Kaolin, Saponite, and Calcium Carbonate, were characterized by X-ray diffraction and fluorescence analysis. The bactericidal properties of ceramic membrane samples with and without silver modification were examined using the inhibition zone test.

### **4.1. XRD**

Fig. 1 shows diffraction patterns for the components of ceramic membranes (Kaolin, Saponite, Calcium carbonate) and CM0 sample of a ceramic membrane made from them. X-ray diffraction analysis was performed using a Rigaku Ultima IV X-ray diffractometer (Japan) with CuK $\alpha$  radiation. According to standard cards (ICDD 01-078-2110 (Kaolinite), ICDD 01-087-2096 (Quartz), ICDD 01-083-0578 (Calcite), ICDD 01-073-2337 (Magnesium aluminosilicate)) the components of ceramic membranes contain phases characteristic of them (Table 1), and CM0 ceramic membrane contains Magnesium aluminosilicate (Saponite). Thus, it can be concluded that as a result of the preparation and subsequent heating of the ceramic membrane, kaolinite is converted into metakaolin, which has an amorphous structure, and therefore, the diffraction pattern of the CM0 sample exhibits peaks characteristic only of the layered structure of saponite. Calcium carbonate decomposes to Calcium oxide, which also has an amorphous structure.



**Figure 1:** XRD Patterns of Samples: 1 – Kaolin; 2 – Saponite; 3 – Calcium Carbonate; 4 – CM0  
 (Source: Self)

The presented crystallite size in Table 1 indicates the nanocrystalline state of both the initial components and ceramic membrane (matrix).

**Table 1:** Composition and Crystallite Size of Samples

Sample	Composition	Crystallite size, nm
Kaolin	Kaolinite	23
Saponite	Magnesium aluminosilicate (Saponite), Quartz	13
Calcium carbonate	Calcium carbonate	58
CM0	Magnesium aluminosilicate (Saponite)	18

(Source: Self)

#### 4.2. Chemical composition

Chemical analysis was used to determine the elemental composition of the samples. The samples were loaded in the EXPERT 3L INAM X-ray fluorescence analyzer (Ukraine) for elemental analysis. The chemical analysis is presented in Table 2. The chemical analysis of the samples revealed the main chemical elements inherent in the purposeful phases.



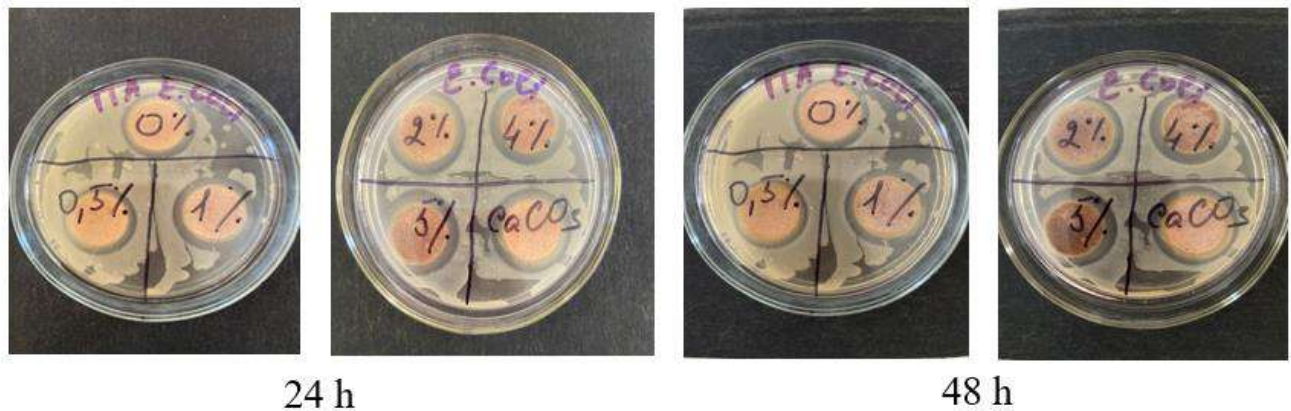
**Table 2:** Chemical Analysis of Samples

Element	Composition, %			
	Kaolin	Saponite	Calcium carbonate	CM0
Si	<b>57.75</b>	<b>36.15</b>	0.22	17.98
Al	<b>32.34</b>	-	-	-
Ti	<b>3.95</b>	-	-	-
K	<b>2.57</b>	-	-	-
Fe	<b>1.97</b>	<b>40.15</b>	0.03	6.04
Al	-	<b>7.63</b>	-	10.46
Ca	0.89	<b>6.80</b>	<b>99.28</b>	63.06
Mg	-	<b>5.19</b>	-	-
S	0.30	-	-	-
Ti	-	<b>2.85</b>	0.22	2.14
Ga	0.03	-	-	0.02
Zr	0.02	0.04	-	0.03
Mn	-	0.59	-	0.07
V	0.15	0.17	0.09	0.11
Re	0.01	-	-	-
Ni	0.01	0.09	-	-
Cu	0.01	0.13	-	0.02
Zn	-	0.10	0.01	0.01
Cr	-	0.04	-	-
Sr	-	0.03	0.13	0.04
Rb	-	0.02	0.01	0.01
Y	-	0.02	0.01	0.01

(Source: Self)

### 4.3. Bactericidal ability

The bactericidal ability of ceramic membranes was investigated according to the method described in the "Methodology" section. The size of the zone of inhibition clearly shows the efficiency of modified ceramic membranes as shown in Fig. 2 and Fig. 3.



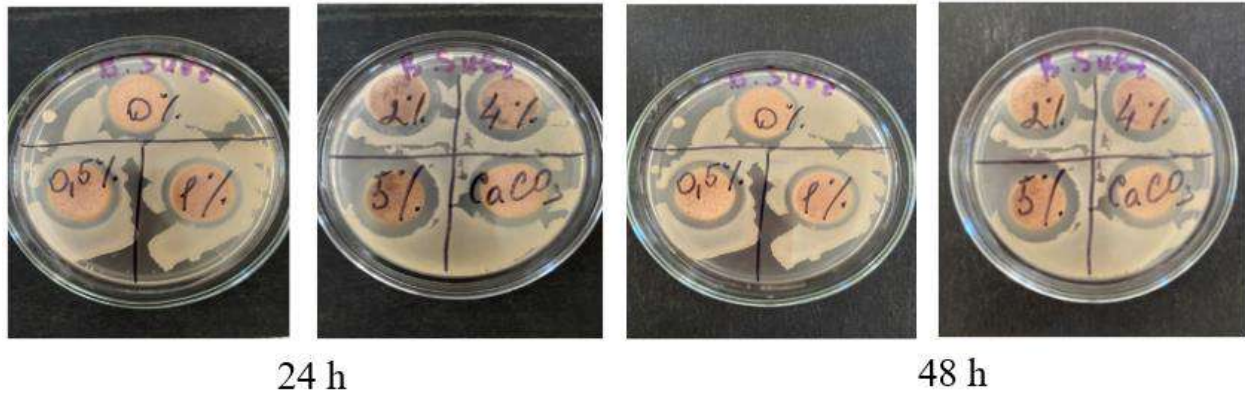
**Figure 2:** Inhibition Zone of Functionalization Ceramic Filtration Membranes against *E. coli* after Incubation for 24 And 48 H

(Source: Self)

As a general result, it was observed that the modified ceramic membranes showed higher activity against *B. subtilis* compared with *E. coli*. The inhibitory effect of ceramic membranes on the growth of bacteria were observed after incubation at 37 °C for 24 and 48 h. Further, it was noticed that uncoated membranes have a slight influence on the removal of the bacteria under study.

To study the effect of silver concentration, its content was varied from 0.5 to 5 % against both strains of bacteria. The inhibitory activity of bactericidal ceramic membranes with increased silver concentration was increased. It is noticed that Ag is evenly covered on the membrane surface. At the same time, a small amount of 0.5% showed a significant effect on the removal of *B. subtilis* and *E. coli*. The radius of inhibition is uniformly uniform. Additional control on the second day and after one week of cultivation did not demonstrate any difference.

These results showed that the antibacterial properties presented by functionalized ceramic filtration membranes can be ascribed to the presence of silver over their large surface area that ensures greater surface contact with microorganisms. Antimicrobial activity depends mainly on the release of silver ions from the surface of bactericidal ceramic membranes.



**Figure 3:** Inhibition Zone of Functionalization Ceramic Filtration Membranes against *B. Subtilis* after Incubation For 24 And 48 H

(Source: Self)

## 5. Conclusion

The raw components and obtaining ceramic membranes were characterized by X-ray methods, as a result of which the transformation of kaolin and calcium carbonate into metakaolin and calcium oxide was observed. The saponite phase does not undergo any changes during heating.

The antimicrobial properties of silver-modified ceramic membranes were investigated. The test of the zone of inhibition established an increased antimicrobial efficiency of the material, APMB, against the two bacteria (Gram-negative *Escherichia coli* and Gram-positive *Bacillus subtilis*) and the level for both bacteria showed dose-dependent antibacterial activity. The results indicate that the silver content in the amount of 0.5% exhibits high antibacterial activity against *B. subtilis* and *E. Coli*. To confirm the results obtained, more experimental studies need to be carried out. In the future, it is planned to study the mechanical and transport characteristics of obtaining ceramic membranes.

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