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Synthesis Process of Silica–Activated Carbon Composite Ceramic Membranes for Integrated Filtration and Adsorption in Acid Mine Drainage

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ABSTRACT
Acid Mine Drainage (AMD) is a liquid waste from coal mining by-products. AMD is a highly corrosive liquid (pH 1.5 – 4.0) and contains heavy metals, which is one of the problems in coal mining. Filtration technology using ceramic membranes can offer greater corrosion resistance than other membranes. Therefore, the study aims to fabricate and characterize composite ceramic membranes made from silica extracted from rice husk ash (RHA) and modified with high-performance activated carbon (Maxsorb III) to boost heavy metal adsorption capacity. The membranes are produced through extrusion molding and stepwise sintering processes. The results were analyzed using SEM-EDS, XRF, XRD, and BET. XRF analysis showed that the silica purity increased from 88.42 to 98.62 wt%, while XRD confirmed a predominantly amorphous structure with a broad peak centered at approximately 22° (2θ). SEM-EDS revealed the formation and distribution of pores, with Si, O, and C as the main elements. Increasing the activated carbon content from 10 to 20 wt% increased the BET specific surface area from 238 to 412 m²/g and the average pore diameter from 16 to 25 nm, indicating increased pore development and surface area. These results indicate that activated carbon effectively modifies the porous structure and textural properties of the ceramic membrane.

Keywords: filtration, ceramic membrane, wastewater, heavy metal adsorption.

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Introduction

Globally, the coal mining industry continues to play a crucial role in meeting energy needs, particularly in countries that rely on coal as a primary source of electricity generation and industrial energy. In this global context, Indonesia is a significant case in point due to its extensive coal mining activities and significant contribution to its energy sector and economy. The growth of the coal mining industry in Indonesia has supported the national energy supply, industrial development, employment, and export revenues. However, this activity also produces liquid waste in the form of Acid Mine Drainage (AMD), which remains a problem in mining environmental management [1]. AMD is formed due to the oxidation of sulfide minerals, especially pyrite (FeS_2), when in contact with water and oxygen, resulting in water with a very low pH (1.5–4.0). AMD also contains dissolved heavy metals such as Fe, Mn, Cu, Zn, Al, and sulfate in high concentrations. If not adequately treated, AMD can cause water pollution, soil degradation, damage to aquatic ecosystems, and increased health risks for communities around mining areas [2]. Therefore, the development of effective, economical, and sustainable AMD processing technology is a priority in environmental engineering and mining technology.

The Indonesian government, through the Decree of the Minister of Environment Number 113 of 2003, has established wastewater quality standards for coal mining activities that must be met before the wastewater is discharged into the environment. However, the characteristics of highly acidic AMD and containing heavy metals in high concentrations make the treatment process complex [3]. Conventional technologies such as neutralization using lime, chemical precipitation, coagulation-flocculation, bioremediation, and adsorption have been widely applied on an industrial scale. However, these methods still face various limitations, including high chemical consumption, the formation of large amounts of secondary sludge, the need for relatively long processing times, and high operating and maintenance costs [4]. These conditions indicate that alternative technologies are still needed that have high efficiency and are able to reduce the environmental impact of the waste processing process itself.

In recent years, membrane technology has emerged as a promising separation method due to its ability to produce high-quality effluent, small land footprint, and easy integration with other treatment processes. However.

However, most membranes used in wastewater treatment are still polymer-based and lack chemical and thermal stability. Their performance and structural stability can be negatively affected by extreme pH and high-temperature conditions, making them susceptible to degradation, fouling, permeability changes, and reduced service life when applied to acid mine wastewater (AMD) treatment [5].

As an alternative, ceramic membranes offer significantly better mechanical, thermal, and chemical stability, enabling them to operate in highly corrosive environments. In addition to their longer lifespan, ceramic membranes can also be regenerated through chemical or thermal cleaning processes without significant structural damage [6]. Therefore, the development of ceramic membranes based on local materials is an important research direction in producing more efficient and sustainable AMD processing technology.

In line with the increasing attention to the concept of circular economy and waste valorization, the utilization of agricultural waste as raw material for advanced materials has become a rapidly growing research topic. Indonesia is one of the largest rice producers in the world, producing millions of tons of rice husks annually. Burning rice husks produces rice husk ash (RHA), which contains around 80–95% amorphous silica, depending on the combustion conditions and extraction process [7]. To date, most of the rice husk ash is still used as backfill or even disposed of as solid waste, thus not providing optimal added value. In fact, the high silica content makes RHA a potential source of raw materials for the synthesis of ceramic materials, adsorbents, catalysts, and membranes. The utilization of silica from rice husk ash not only reduces agricultural waste but also supports the development of environmentally friendly materials based on renewable resources.

Silica extracted from rice husk ash has high chemical stability in acidic environments, purity that can be improved through a purification process, and an amorphous structure that supports the formation of ceramic membranes. However, silica has a relatively limited specific surface area, so the number of active sites for heavy metal adsorption is still not optimal [8]. One approach to improving these characteristics is to combine silica with highly porous activated carbon. Maxsorb III is a commercial activated carbon with a specific surface area of approximately 3,000 m^2/g, thus potentially increasing the membrane's adsorption capacity for heavy metal ions. In addition to functioning as an adsorbent, activated carbon can also act as a pore-forming agent during the sintering process. During the combustion process, activated carbon will decompose and leave micropore cavities, thereby increasing the porosity, effective surface area, and permeability of the membrane [[9], [10]]. Controlling the pore structure is one of the main factors that determine the performance of ceramic membranes in the filtration process. In this study, Maxsorb III was selected because of its highly porous structure and exceptionally high specific surface area, which can provide additional active sites for adsorption and facilitate the development of membrane porosity. However, the low bulk density and relatively low thermal conductivity of Maxsorb III should also be considered in the design of the composite membrane. The low density may affect particle packing and interparticle contact within the silica matrix, while the low thermal conductivity can influence heat transfer during thermal treatment. Excessive incorporation of activated carbon may therefore result in increased porosity at the expense of structural integrity and mechanical strength if the pore-forming phase is not adequately controlled. Consequently, optimizing the activated carbon content is essential to achieve a balance between porosity, permeability, adsorption capacity, and mechanical stability. Controlling the pore structure is therefore one of the key factors determining the filtration performance and durability of ceramic membranes.

Although research on the utilization of rice husk ash as a source of silica and the development of ceramic membranes has been widely reported, most of the research is still focused on its application as an adsorbent, geopolymer, catalyst, or membrane for domestic water treatment and general industrial wastewater. Research that integrates silica extracted from rice husk ash with Maxsorb III activated carbon as a ceramic membrane component for Acid Mine Drainage treatment applications is still very limited [11]. In addition, the effect of variations in activated carbon content on the development of pore structure, specific surface area, and morphology of RHA silica-based membranes has not been studied systematically. This research gap indicates the need for the development of new membrane materials that not only have high resistance to acidic environments, but are also able to utilize biomass waste as a source of value-added raw materials.

Based on the description, the novelty of this research lies in the development of a silica-based composite ceramic membrane extracted from rice husk ash with the addition of Maxsorb III activated carbon, which functions as an adsorbent and pore-forming agent. This approach integrates the concept of biomass waste utilization (waste-to-value) with ceramic membrane technology to produce a material that has higher porosity, larger specific surface area, and good chemical resistance to acidic environments. Thus, this research not only offers a solution to the problem of Acid Mine Drainage treatment, but also contributes to the development of sustainable material technology that supports a circular economy and the achievement of the Sustainable Development Goals (SDGs), especially SDG 6 (Clean Water and Sanitation), SDG 9 (Industry, Innovation and Infrastructure), and SDG 12 (Responsible Consumption and Production).

This article aims to fabricate and charatarization of new type of membrane for integrated filtration and adsorption in acid mine drainage. It also aims to characterize the morphology, porosity, and specific surface area of the resulting membranes, and analyze the effect of the addition of activated carbon on the physical characteristics of the membrane as a basis for evaluating its potential for Acid Mine Drainage treatment applications.

Experimental part

Materials and tools

The main raw material used in this study was rice husk obtained from a local rice mill in Padang City, Indonesia. It was rice husk freshly collected in April, 2026. The rice husk was collected immediately after the rice milling process and was used as the starting material for subsequent washing, drying, and preparation.

The chemicals used included a 10% hydrochloric acid (HCl) solution as an acid leaching agent, Maxsorb III activated carbon as a pore-forming agent, polyvinyl alcohol (PVA) as a binder, sodium hydroxide (NaOH) for the silica extraction process, and deionized water as a solvent and washing medium.

The main equipment used includes an electric furnace for calcination and sintering processes, a ball mill for powder refinement, a 200-mesh sieve, a hydraulic extruder for tubular membrane molding, a drying oven, and an analytical balance. The characterizations were conducted using X-Ray Fluorescence (XRF) to determine chemical composition, X-Ray Diffraction (XRD) to identify crystal phases, Scanning Electron Microscopy (SEM) to observe surface morphology, and a Brunauer–Emmett–Teller (BET) Surface Area Analyzer to determine specific surface area and pore characteristics.

Silica Extraction from Rice Husk Ash

Silica is extracted from rice husk ash using a combination of acid dissolution and calcination. The extraction process includes preparing the rice husk, dissolving it with acid, calcination, and extracting the silica.

Rice Husk Preparation

The rice husks were gently rinsed with running water to remove surface dirt. The husks were then dried in an oven at 105°C for 24 hours until a constant weight was achieved. The rice husks are gently rinsed with running water to remove surface dirt and other adhering impurities. The washed husks are then dried in an oven at 105°C for 24 hours, or until a constant weight is reached. The drying temperature of 105°C was chosen to effectively remove free and physically bound moisture from the rice husks while minimizing thermal changes or decomposition of their organic constituents. Achieving a constant weight ensures that the moisture content is sufficiently stable before further processing. After drying, the samples were weighed as a reference for the next extraction process [12].

Acid Leaching

Dry rice husks were soaked in a 10% HCl solution for 24 hours at room temperature to remove metal oxides and mineral impurities. Then, they were washed with deionized water until a neutral pH was reached. Once the filtrate reached neutral pH, the solution was dried for 24 hours at 105°C.

Calcination

Rice husk ash (RHA) is obtained by calcining rice husks from an acid leaching process for 4 hours at 650°C with a heating rate of 5°C per minute. This is done to keep the silica in an amorphous phase, making it suitable as a raw material for ceramic membranes. After naturally cooling in the furnace, the rice husk ash is ground using a ball mill and then sieved through a 200-mesh screen to ensure uniform size [7].

Silica Extraction

Silica is extracted from rice husk ash using the alkali–sol–gel method [[13], [14]]. 50 g of rice husk ash is heated in a 1 M NaOH solution at 100°C for 1 hour. The solution is filtered to separate the solid residue. The filtered solution is then titrated with 1 M HCl until reaching pH 7 to form a silica gel precipitate and aged overnight. Next, it is rinsed with deionized water until free of chloride ions. The resulting silica gel is dried at 105°C for 24 hours and then calcined again for 2 hours at 600°C. This is done to obtain amorphous silica powder. The resulting amorphous silica powder is analyzed for its composition using X-Ray Fluorescence (XRF) to determine the SiO_2 content, oxide impurities, and the effectiveness of the purification process. The crystal structure was characterized using XRD, while the specific surface area and pore distribution were analyzed using a BET Surface Area Analyzer.

Composite Ceramic Membrane Fabrication

Composite ceramic membranes were fabricated using an extrusion method followed by drying and sintering [[13], [15]]. The extracted silica was mixed with Maxsorb III activated carbon at varying compositions of 10% (membrane i) and 20 wt% (membrane ii) by weight of silica. Before mixing, the activated carbon was dried at 100°C for 12 hours to remove moisture that could affect the homogeneity of the mixture.

A mixture of silica and activated carbon was added with a 5 wt% PVA solution as a binder, then mixed with deionized water to obtain a plastic mixture with a water content of around 20–25 wt%. The mixture was homogenized using a mechanical mixer at 300 rpm for 30 min to ensure uniform dispersion of the silica, activated carbon, PVA binder, and water throughout the mixture. The mixing speed and duration were kept constant for all samples to maintain consistent processing conditions and minimize variations in the homogeneity and subsequent structural properties of the ceramic membrane.

The plastic mixture was molded into a tubular membrane using a hydraulic extruder. The ceramic membrane in this study is 3 cm long with an inner diameter of 0.6 cm and an outer diameter of 0.8 cm [16]. To get a uniform membrane thickness and diameter, the pressure was kept constant during the extrusion process. The process continued with slow drying. The dry membrane then underwent sintering with a heating rate up to 1000°C at a rate of 3°C/min. The heating was held for 2 hours before allowing it to cool naturally in the furnace. This process decomposes the PVA and burns the activated carbon, creating pores in the silica matrix and acting as a pore-forming agent [[17], [18]].

Membrane Characterization

After the sintering process, the resulting membrane is characterized, including its morphological structure and elemental composition, crystal structure, surface area, and porosity.

Morphological Characterization and Elemental Composition (SEM)

The surface morphology of the membrane was analyzed using a Scanning Electron Microscope (SEM) at magnifications of 750x and 2000x with an accelerating voltage of 5–15 kV. This analysis was done to determine the membrane's microstructure in terms of shape and pore distribution of the ceramic membrane made from rice husk ash silica and activated carbon.

Crystal Structure Characterization (XRD)

The crystal structure was analyzed using X-Ray Diffraction (XRD). The wavelength of Cu-Kα radiation is 1.5406 Å with a scan speed of 2°/minute in a diffraction angle range of 2θ = 10–80°. The presence of amorphous silica was identified by the appearance of a broad halo at an angle of around 2θ = 22°, while the formation of crystalline silica, such as quartz or cristobalite, was indicated by the appearance of sharp diffraction peaks at their characteristic positions. Phase identification was carried out by matching the diffraction pattern to the International Centre for Diffraction Data (ICDD PDF-4) database [19].

Surface Area and Porosity (BET) Characterization

The membrane texture characteristics were analyzed using a Brunauer–Emmett–Teller (BET) Surface Area Analyzer through the nitrogen adsorption–desorption method at a temperature of -196°C (77 K). Before testing, the samples were degassed at 180°C for 4 hours under vacuum conditions to remove water and gas molecules adsorbed on the surface.

The specific surface area was calculated using the BET equation, while the pore size distribution and pore volume were analyzed using the Barrett–Joyner–Halenda (BJH) method. The parameters obtained included the specific surface area (m^2/g), total pore volume (cm^3/g), and average pore diameter (nm). These parameters were used to evaluate the effect of variations in activated carbon content on the development of the membrane pore structure.

Chemical Composition Characterization (XRF)

The chemical composition of rice husk ash, extracted silica, and composite ceramic membranes was analyzed using X-Ray Fluorescence (XRF) with the Fundamental Parameters (FP) method. Prior to testing, the samples were dried to constant weight, ground until homogeneous, and then pressed into pellets according to the instrument's standard preparation procedure.

XRF analysis was conducted to determine the content of major oxides and impurities, so that the effectiveness of the silica purification process could be evaluated quantitatively. The parameters analyzed included the content of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, K_2O, Na_2O, TiO_2, P_2O_5, and other minor oxides expressed in weight percent (wt%). The analysis results were used to ensure that the rice husk extraction process did indeed contain silica.

Results and Discussions

Characteristics of Silica Raw Materials

XRF results show that quartz sand contains 97.3% SiO_2, with major impurities Al_2O_3 (1.4%) and Fe_2O_3 (0.5%).

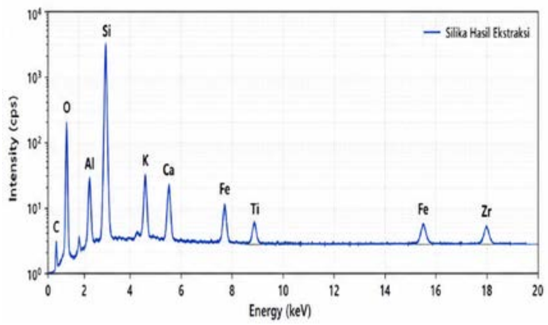

Figure 1 - XRF Spectrum of Silica Extracted from Rice Husk

The overall XRF results can be seen in Table 1.

Table 1 - Rice husk components and extraction results

Parameter	Component	
	RHA (wt%)	***Silica from extraction***
SiO_2	88.42	98.62
Al_2O_3	1.86	0.18
Fe_2O_3	0.92	0.07
CaO	1.48	0.05
MgO	0.71	0.04
K_2O	2.73	0.12
Na_2O	0.32	0.03

The XRF analysis results showed that the extraction process successfully increased the purity of silica from 88.42 wt% to 98.62 wt% (10.20 wt%). The content of the main impurities (Al_2O_3, Fe_2O_3, CaO, MgO, K_2O, Na_2O) decreased significantly (>90%). The LOI value also decreased from 3% to 0.84%, indicating a reduction in organic/volatile compounds after purification. Silica produced from rice husks is generally amorphous (not crystalline), which is very good for adsorption and membrane applications because it has high surface reactivity [[3], [20]].

Characteristics of Silica Crystal Structure from RHA Extraction

XRD results show that silica from rice husk has an amorphous structure, which is characterized by a broad diffraction peak at an angle of 2θ of around 20–30° (the main peak is around 22°) and no sharp peaks indicating a crystalline structure (such as quartz). Pretreatment with acid (HCl) can maintain the amorphous structure of silica up to a firing temperature of 750°C-800°C [21].

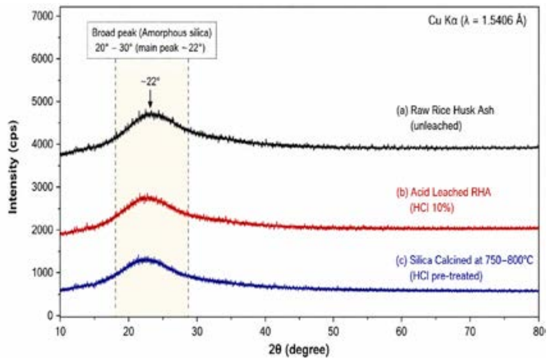

Figure 2 - XRD result of Silica from Rice Husk Ash

Characteristics of Ceramic Membranes from Rice Husk Silica

The morphology of ceramic membranes with a composition of 10% activated carbon (membrane i) and a composition of 20% (membrane ii) at various magnifications can be seen in Figure 3.

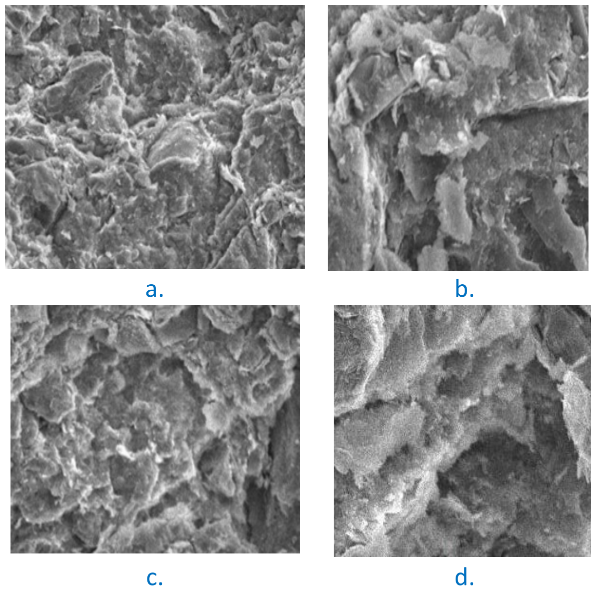

Figure 3 - Morphology of membrane i at zoom a. 750x; b. 2000x; and Morphology of membrane ii at zoom c. 750x; d. 2000x

Activated carbon clumps form interparticle bonds that overlap, leading to a visible reduction in pore size from the surface to the interior. This indicates the formation of a microporous membrane. Activated carbon forms interparticle bonds that overlap, leading to a visible reduction in pore size from the surface to the interior. The morphology of extracted silica particles is generally irregular with a porous structure [21].

SEM morphological structures of each membrane at various magnifications show that the membrane pore structure is random. From the SEM observations, the pores and uneven membrane surface texture are visible due to the influence of temperature. The pores in the membrane with the addition of activated carbon. The more activated carbon added, the more pores formed and still appear less uniform (asymmetric). It can be seen that the pores formed are already small enough to be used in the ultrafiltration process with a pore size of 0.001 – 0.1 µm. The pore surface appears to be evenly distributed on each membrane. For membranes with different concentrations, it can be seen that the pores become denser with increasing amounts of activated carbon additives, and the pores are evenly distributed on the membrane surface due to the addition of these additives [13]. This behavior can be attributed to the porous nature of activated carbon and its role as a pore-forming agent; a higher amount of activated carbon provides more sites for void formation within the silica matrix during subsequent thermal treatment, thereby resulting in a denser pore structure.

Surface Area Characterization (BET)

Analysis using N_2 physisorption at -196 °C (77 K) showed very significant differences in textural characteristics.

Table 2 - Membrane Surface Area Characteristics

Parameter	Membrane I (10%)	Membrane II (20%)
Surface area (m^2/g)	23 8 ± 12	412 ± 18
Total pore volume (cm^3/g)	0.38 ± 0.02	0.72 ± 0.04
Average pore diameter (nm)	16.20	24.80

Increasing the activated carbon content from 10% to 20% resulted in a nearly twofold increase in surface area (from 238 to 412 m^2/g). This was due not only to the remaining unburned carbon, but primarily to the micro-etching of the silica surface by the CO_2 gas released during combustion [22].

The shift in pore diameter from 16 nm to 25 nm indicates that in M-II, the pore structure shifts from mesopore dominance towards macropores, which is very beneficial for increasing permeability, but slightly reduces the adsorption capacity per unit volume [23].

Conclusion

This study has developed a potential ceramic membrane based on agricultural waste and carbon activation. The utilization of rice husk as an agricultural waste contributes significant impact on solid waste solutions and increases the added value of rice husk. Furthermore, the manufacturing process is relatively simple, using extrusion and sintering techniques, which makes this technology adoptable for small- and medium-scale industries.

The recommended further research includes: (1) modifying membranes with metal oxides like ZnO and TiO2, to be able to improve the simultaneous adsorption capacity of heavy metals and sulfate; (2) evaluating the technical and economic feasibility in pilot plant testing.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **S.R.P.Primandari:** Conceptualization, Methodology, Investigation; **Mulianti**: Data curation, Writing draft preparation; **T. Aprianti**: Supervision, Validation; **Y. Sugiarto**: Reviewing and Editing; **T. Elshaarani:** Data analysis and validation.

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Қышқыл шахта ағындыларын кешенді сүзу және адсорбциялау үшін кремний диоксиді мен белсендірілген көмірден композиттік керамикалық мембраналарды синтездеу процесі

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ТҮЙІНДЕМЕ
Қышқылды шахта ағындылары (ҚША) – бұл көмірді өңдеу нәтижесінде пайда болған сұйық қалдықтар. ҚША – жоғары коррозиялық сұйықтық (pH 1,5 – 4,0) және құрамында ауыр металдар бар, бұл көмір өнеркәсібінің мәселелерінің бірі болып табылады. Керамикалық мембраналарды пайдаланатын сүзу технологиясы басқа мембраналарға қарағанда коррозияға төзімділікті арттыра алады. Бұл зерттеудің мақсаты- күріш қабығының күлінен (RHA) алынған және ауыр металдардың адсорбциялық қабілетін арттыру үшін жоғары өнімді белсендірілген көмірмен (Maxsorb III) модификацияланған кремний диоксидінен жасалған композиттік керамикалық мембраналарды жасау және сипаттау. Мембраналар экструзиялық қалыптау және сатылы күйдіру процестері арқылы өндіріледі. Нәтижелер SEM-EDS, XRF, XRD және BET көмегімен талданды. XRF талдауы кремний диоксидінің тазалығы 88,42-ден 98,62 салмақтық%-ға дейін өскенін, ал XRD шамамен 22° (2θ) орталығында орналасқан кең шыңы бар негізінен аморфты құрылымды растағанын көрсетті. SEM-EDS әдісі негізгі элементтер ретінде Si, O және C бар кеуектердің пайда болуы мен таралуын анықтады. Белсендірілген көміртегі мөлшерін 10-нан 20 салмақтық пайызға дейін арттыру BET меншікті бетінің ауданын 238-ден 412 м²/г-ға дейін және орташа кеуек диаметрін 16-дан 25 нм-ге дейін арттырды, бұл кеуектердің өсуі мен бетік ауданының артқанын көрсетеді. Бұл нәтижелер белсендірілген көмірдің керамикалық мембрананың кеуекті құрылымы мен текстуралық қасиеттерін тиімді түрде өзгертетінін көрсетеді.

Түйін сөздер: сүзу, керамикалық мембрана, ағынды сулар, ауыр металдардың адсорбциясы.

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Процесс синтеза композитных керамических мембран из диоксида кремния и активированного угля для комплексной фильтрации и адсорбции кислых шахтных стоков

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АННОТАЦИЯ

Кислотные шахтные стоки (КШС) — это жидкие отходы, образующиеся в результате переработки угля. КШС представляют собой высококоррозионную жидкость (pH 1,5–4,0) и содержат тяжелые металлы, что является одной из проблем угольной промышленности. Технология фильтрации с использованием керамических мембран может обеспечить более высокую коррозионную стойкость, чем другие мембраны. Поэтому целью данного исследования является изготовление и характеристика композитных керамических мембран из диоксида кремния, извлеченного из золы рисовой шелухи (ЗРШ), и модифицированных высокоэффективным активированным углем (Maxsorb III) для повышения адсорбционной способности по отношению к тяжелым металлам. Мембраны изготавливаются методом экструзионного формования и ступенчатого спекания. Результаты анализировались с помощью SEM-EDS, XRF, XRD и BET. Анализ XRF показал, что чистота диоксида кремния увеличилась с 88,42 до 98,62 мас.%, в то время как XRD подтвердил преимущественно аморфную структуру с широким пиком, центрированным примерно на 22° (2θ). Метод SEM-EDS выявил образование и распределение пор с основными элементами Si, O и C. Увеличение содержания активированного угля с 10 до 20 мас.% привело к увеличению удельной поверхности по методу BET с 238 до 412 м²/г и среднего диаметра пор с 16 до 25 нм, что указывает на увеличение развития пор и площади поверхности. Эти результаты показывают, что активированный уголь эффективно модифицирует пористую структуру и текстурные свойства керамической мембраны.

Ключевые слова: фильтрация, керамическая мембрана, сточные воды, адсорбция тяжелых металлов.

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