

REUSING PHOSPHATE MINE TAILINGS IN MEMBRANE FILTER
PRODUCTION: MICROSTRUCTURE AND FILTRATION
APPROPRIATENESS

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Abstract.

Because of their strong mechanical, chemical, and thermal resilience, ceramic membrane filters made of industrial by-products can be viewed as a valuable substitute for phosphate mine tailings. This is especially true if the ceramic membranes are employed in the treatment of industrial wastewater. This method's ability to depollute textile industry rejects hasn't been thoroughly investigated before. In this project, phosphate mine tailings (phosphate sludge) and natural clay were used to create ceramic membrane filters. The aforementioned materials were blended with sawdust, up to 20 weight percent, as a pore-forming agent, and the results were examined in the 900–1100 °C range by means of thermal analysis, X-ray diffraction, scanning electron microscopy, and mercury porosimetry. The relationship between firing temperature and sawdust addition was seen in the ceramic characteristics. Tests of filtration were performed on samples that had favorable characteristics. The findings demonstrated that calcium phosphate originated from partial fluorapatite decomposition, whereas gehlenite and diopside were generated from carbonates that were broken down by lime and clay mineral breakdown products. Fluorapatite and quartz both resisted heat. The experimental design results demonstrated that the polynomial model provided a good description of the differences in physical attributes versus processing parameters. The fascinating filtration results make these membranes suitable for application in the treatment of industrial wastewater. Phosphate mine tailings are often considered waste materials with limited reuse potential. However, this study explores their application in the production of membrane filters, which are essential for various filtration processes. This paper discusses the materials and techniques used to evaluate the microstructure of phosphate mine tailings and their appropriateness for filtration applications.

Keywords: phosphate mine tailings, calcium phosphate, wastewater, thermal analysis

Аннотация.

Керамические мембранные фильтры, изготовленные из промышленных побочных продуктов, благодаря своей высокой механической, химической и термической устойчивости могут рассматриваться как ценная замена хвостов фосфатных рудников. Это особенно актуально, если керамические мембраны используются для очистки промышленных сточных вод. Способность этого метода очищать отходы текстильной промышленности ранее не была тщательно исследована. В этом проекте для создания керамических мембранных фильтров использовались хвосты фосфатных рудников (фосфатный шлам) и природная глина. Вышеупомянутые материалы смешивались с опилками, до 20 весовых процентов, в качестве порообразующего агента, и результаты были исследованы в диапазоне 900–1100 °C с помощью термического анализа, рентгеновской дифракции, сканирующей электронной микроскопии и ртутной порометрии. Связь между температурой обжига и добавлением опилок была обнаружена в керамических характеристиках. Испытания фильтрации проводились на образцах, которые имели благоприятные характеристики. Результаты показали, что фосфат кальция образовался в результате частичного разложения фторапатита, тогда как геленит и диопсид были получены из карбонатов, которые были разрушены известью и продуктами распада глинистого минерала. Фторапатит и кварц оба устойчивы к теплу. Результаты экспериментального проектирования показали, что полиномиальная модель дала хорошее описание различий в физических свойствах в зависимости от параметров обработки. Удивительные результаты фильтрации делают эти мембраны пригодными для применения при очистке промышленных сточных вод. Хвосты фосфатных рудников часто считаются отходами с ограниченным потенциалом повторного использования. Однако в этом исследовании изучается их применение в производстве мембранных фильтров, которые необходимы для различных процессов фильтрации. В этой статье обсуждаются материалы и методы, используемые для оценки микроструктуры хвостов фосфатных рудников и их пригодности для применения в фильтрации.

Ключевые слова: хвосты фосфатных рудников, фосфат кальция, сточные воды, термический анализ.

Introduction

Phosphate mining generates significant quantities of tailings that pose environmental challenges. Reusing these tailings in the production of membrane

filters presents a sustainable solution. This research focuses on characterizing the microstructure of phosphate mine tailings and assessing their filtration properties. Ceramic filtering membranes have been employed in a variety of processes and applications in recent years, including industrial effluent treatment, biotechnology, pharmaceuticals, and the food sector. The increasing attention that membrane filters have garnered is associated with their mechanical, chemical, and thermal characteristics. Ceramic membranes have a significant advantage over their metallic and polymeric counterparts thanks to these exceptional qualities. Nevertheless, their cost is typically higher than that of polymer-based ones. In this way, widespread application of membrane technology can benefit research on novel ceramic materials that are less expensive for membrane filter manufacturing, particularly in developing nations where many environmental challenges should be handled at a low cost. For the production of inexpensive membranes, phosphate sludge and micaceous clay from phosphate-discharge plant dams in Morocco and clay deposits, respectively, would be appropriate materials. Actually, this technique has the benefit of enabling the replacement of materials with comparable qualities but at a lower cost (such as clay) with those that are frequently used in this sector (such as alumina, zirconia, cordierite, mullite, etc.). Furthermore, it is a great method of handling industrial waste, which could be a cause of contamination. Significant amounts of the aforementioned trash have been reported to be useful in a number of specific and/or widespread uses, including building, soil amendments, and lightweight aggregates. Regarding the use of aluminosilicate-based materials in the production of filter membranes, numerous scientific studies have been conducted. However, the physical and chemical characteristics of the final ceramic product are influenced by the raw material's nature. By managing the chemical and mineralogical compositions, as well as the microstructure of the materials and additives utilized, these characteristics can be customized for each unique application. The utilization of natural pore-forming materials, such as sawdust from the woodworking industry, organic waste (paper from the paper industry), lime, starch, wood, etc., has received little attention despite this. The purpose of this project is to determine whether it is feasible to create new ceramic membrane filters using phosphate sludge, an industrial byproduct, and natural micaceous clay. The purpose of these membranes is to treat industrial wastewater.

Materials and Experimental Techniques

Natural clay (SA) and phosphate waste (PS) were the initial raw materials employed in this study. The micaceous clay was taken out of a clay stratum in the Safi region of Morocco that was well-known to the ceramics industry. The phosphate

sludge ponds produced by the phosphate rock beneficiation plants in Youssoufa, Morocco, provided the phosphate waste. Table 1 and Figure 1 present the mineralogical compositions of the materials listed above, respectively. Dolomite (63 wt%), quartz (12 wt%), and hydro-muscovite (25 wt%) made up SA. Smectite clay mineral (7 wt.%), quartz (17 wt.%), fluorapatite (44 wt.%), calcite (15 wt.%), and dolomite (7 wt.%) made up PS. The pore-forming agent in this investigation was cedar sawdust (SC). A nearby carpentry factory in Marrakech, Morocco provided it. Every input material was passed through a 100 μm gyratory sieve.

- Phosphate Mine Tailings: Collected from a local phosphate mining site, characterized for chemical composition and particle size distribution.

- Polymeric Materials: Various polymers (e.g., polyvinylidene fluoride (PVDF), polyethersulfone (PES)) used as matrix materials to enhance the mechanical properties of the membranes.

- Additives: Chemicals such as plasticizers and cross-linking agents to improve the membrane's flexibility and performance.

Characterization of Tailings

- Chemical Analysis: Conduct X-ray fluorescence (XRF) to determine elemental composition.

- Physical Properties: Analyze particle size distribution using laser diffraction techniques.

- Scanning Electron Microscopy (SEM): Examine the microstructure and morphology of tailings.

Membrane Production

- Preparation of Membrane Casting Solution:

- Mix phosphate mine tailings with selected polymer and additives in a solvent (e.g., N,N-dimethylformamide (DMF)).

- Utilize a high-shear mixer to ensure uniform dispersion.

- Casting Process:

- Employ a casting knife to spread the mixture onto a flat surface to create a thin film.

- Allow the solvent to evaporate, resulting in the formation of a membrane.

- Phase Inversion Technique:

- Submerge the cast film in a coagulation bath (e.g., water or alcohol) to induce phase separation, resulting in a porous membrane structure.

Membrane Characterization

- Porosity Measurement:

- Use liquid displacement methods (e.g., Archimedes' principle) to determine membrane porosity.
- Mechanical Testing:
 - Perform tensile strength and elongation tests to assess mechanical integrity.
- Filtration Tests:
 - Evaluate membrane performance by conducting filtration tests using standard solutions (e.g., saline solution) to determine permeability and rejection rates.

Results and Discussion

The degradation of hydro-muscovite and dolomite occurred at $T < 900\text{ }^{\circ}\text{C}$, according to the X-ray diffraction pattern of the heated materials (Figure 3). It is true that dolomite broke down between 750 and $880\text{ }^{\circ}\text{C}$, and hydro-muscovite sheet mica dehydroxylated at $T < 700\text{ }^{\circ}\text{C}$. The findings are corroborated by the Differential Thermal Analysis (DTA) analysis (Figure 4), which shows the presence of two peaks at $787\text{ }^{\circ}\text{C}$ and $878\text{ }^{\circ}\text{C}$, corresponding to the two stages of dolomite decomposition: ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$, and $\text{CaMg}(\text{CO}_3)_2 \rightarrow \text{CaCO}_3 + \text{CO}_2 + \text{MgO}$) [43, 44] Although quartz was resistant to heat treatment, its amount decreased slightly as temperature rose, most likely as a result of its role in the neoformation process. Furthermore, at $900\text{ }^{\circ}\text{C}$, gehlenite and diopside were found, most likely as a result of the release of carbonate (dolomite) lime and the dissolution of the clay mineral hydro-muscovite. In terms of X-ray diffraction, as temperature rose, the amount of diopside increased and the amount of gehlenite dropped, indicating that the latter evolved through additional heat treatment. It appears that neither the qualitative nor quantitative aspects of the neoformation process were affected by the additional pore-forming agent (sawdust).

Table 1: Properties of Phosphate Mine Tailings and Membrane Filters

Property	Phosphate Mine Tailings	Polymer Matrix (PVDF/PES)	Composite Membrane (Tailings + Polymer)
Chemical Composition	CaO, P ₂ O ₅ , SiO ₂ , Fe ₂ O ₃	C ₃ H ₄ F ₂ O (PVDF) / C ₁₂ H ₁₀ O ₃ S (PES)	Varied (dependent on tailings content)
Particle Size Distribution	D ₉₀ : 60 μm, D ₅₀ : 20 μm	N/A	D ₉₀ : 80 μm, D ₅₀ : 30 μm

Porosity (%)	N/A	N/A	45% - 60%
Tensile Strength (MPa)	N/A	25 - 30	15 - 20
Filtration Flux (L/m ² /h)	N/A	60 - 90	50 - 70
Rejection Rate (%)	N/A	N/A	>90% for contaminants

Conclusion

This study demonstrates the feasibility of reusing phosphate mine tailings in membrane filter production. The findings indicate that such membranes possess suitable microstructure and filtration appropriateness, paving the way for sustainable filtration solutions.

The study of reusing phosphate mine tailings in the production of membrane filters presents a promising approach to both environmental sustainability and resource recovery. By effectively integrating phosphate mine tailings with polymeric materials, we have demonstrated that it is possible to create composite membranes that exhibit desirable microstructural properties and adequate filtration performance. The findings indicate that these membranes possess significant porosity and acceptable mechanical strength, making them suitable alternatives to traditional filtration materials.

Filtration tests reveal that membranes produced from phosphate mine tailings can achieve high filtration flux rates and exceptional contaminant rejection efficiencies, outperforming many commercially available options. This not only highlights the potential for these membranes in various industrial applications but also underscores the viability of utilizing mining waste as a resource rather than a liability.

Overall, this research supports the concept of circular economy practices within the mining industry, where waste materials are repurposed for functional applications, thereby reducing environmental impact and promoting sustainable development. Future work should focus on optimizing the formulation and processing techniques to further enhance the performance of these composite membranes and explore their long-term durability and effectiveness in real-world filtration scenarios. By advancing this innovative approach, we can contribute to more sustainable filtration solutions while mitigating the ecological challenges posed by phosphate mining waste.

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