Abstract

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Degree for which submitted: **PhD** Department: **Chemical Engineering**

Title of PhD thesis: **Preparation of Low-Cost Kaolin Based Tubular Ceramic and Chabazite Zeolite-Ceramic Composite Membranes: Application in Microalgae Recovery and Wastewater Treatment**

Name of the Thesis Supervisor: **Prof. G. Pugazhenthi**

The separation and purification process is an important aspect of downstream operation as it deals with great energy consumption and operational cost in chemical and several other industries. On the other hand, the need for wastewater treatment has increased alarmingly due to urbanization and industrialization. Specifically, the harmful effects associated with discharging untreated heavy metals containing wastewater to water bodies adversely impact mankind and aquatic life. Membrane separation is regarded as a clean and simple process; in particular, the use of low-cost ceramic membranes in the membrane separation process has gained enormous popularity owing to their outstanding thermal, chemical and mechanical properties. In this work, inexpensive kaolin clay based tubular ceramic membranes were prepared with different organic additives such as guar gum (GG), carboxymethyl cellulose (CMC) and hydroxypropyl methylcellulose (HPMC). Considering the fabrication ease and properties, the membrane prepared with HPMC as a binder displayed excellent characteristics such as porosity (40%), mechanical strength (25 MPa), along with good corrosion resistance. Also, the estimated cost of an optimized membrane (Kaolin-HPMC) is found to be 253 USD/m^2 based on raw materials, energy consumption, manpower and equipment cost. The performance of the membrane was tested for recovery of microalgae from the culture broth, TiO_2 separation from aqueous suspension, and oilfield-produced water treatment. The prepared membrane displayed complete recovery (100%) of microalgae from its broth and TiO_2 nano particles from aqueous suspension. In case of oilfield-produced water treatment, the hybrid process of microfiltration followed by biological treatment efficiently reduced the COD of the treated water (COD: 11 mg/L), which was well below the discharge limits (COD: 250 mg/L) prescribed by the Central Pollution Control Board, Government of India. Furthermore, the prepared membrane was extended to use as a support to fabricate a mesoporous zeolite coated tubular ceramic composite membrane for the removal heavy metals from its aqueous solution. Different strategies were followed to fabricate the Chabazite zeolite composite membranes through the organic template-free hydrothermal synthesis method, such as coating the ceramic tubes solely on the interior surface, exterior surface and both sides. The experimental results revealed that the ceramic tube coated on both sides has the highest amount of zeolite loading, i.e., 1.82±0.18 g, along with porosity of 30.50±0.50%, water permeability of 1.74 L/m^2 h bar and average pore diameter of 36 nm. Also, the estimated cost of both sides coated membrane is found to be 868 USD/m^2. The performance of the membrane in separating heavy metal ions from their solutions was evaluated using Cd, Ni, Mg and Al salt in single, binary and tertiary mixtures. The membrane displayed the following salt rejection (R) trend: $R_{Al^{3+}}$ (99.9%) > $R_{Mg^{2+}}$ (99.7%) > $R_{Ni^{2+}}$ (99.5%) > $R_{Cd^{2+}}$ (98.5%) in a single salt system at an applied pressure of 276 kPa, feed concentration of 1000 mg/L, and pH 3.

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List of Publications

Publications in international journals:

1. **M. Purnima,** N. A. Manikandan, K. Pakshirajan, and G. Pugazhenthi, (2020). Recovery of microalgae from its broth solution using kaolin based tubular ceramic membranes prepared with different binders. ***Separation and Purification Technology***, 250, 117212.
2. **M**. **Purnima**, K. Pakshirajan, and G. Pugazhenthi, (2022). Separation of TiO_2 particles from suspension using indigenous low-cost ceramic microfiltration membrane. ***Journal of Water Process Engineering,*** 49, 103123.
3. **M. Purnima**, T. Paul, K. Pakshirajan and G. Pugazhenthi, (2022). Onshore oil field produced water treatment using a hybrid microfiltration-biological process by kaolin based ceramic membrane and oleaginous *Rhodococcus opacus*. ***Chemical Engineering Journal,*** 453, 139850.
4. P. Singh, N. A. Manikandan, **M. Purnima**, K. Pakshirajan, and G. Pugazhenthi, 2020. Recovery of lignin from water and methanol using low-cost kaolin based tubular ceramic membrane. ***Journal of Water Process Engineering***, **38**, 101615.
5. A. K. Basumatary, K. P. Goswami, **M. Purnima**, B. Deka, and G. Pugazhenthi, 2022. Fabrication and characterization of low-cost tubular ceramic membrane for microfiltration of oily wastewater. ***Journal of Water Chemistry and Technology***, 44, 175-181.
6. **M. Purnima**, K.P Goswami, M. Kumar, K. Pakshirajan and G. Pugazhenthi, Facile synthesis of organic template-free chabazite zeolite coated kaolin ceramic membrane for continuous separation of cerium from aqueous solution. ***ACS Applied Engineering Materials***, (Accepted)
7. **M. Purnima**, K.P Goswami, M. Kumar, K. Pakshirajan and G. Pugazhenthi, Low-cost and highly durable kaolin supported CHA zeolite composite membrane for efficient removal of heavy metals from aqueous solution. ***Environmental Technology & Innovation***, 30, 103102

Presentation in international/national conferences:

1. M. Purnima, N. Arul Manikandan, K. Pakshirajan and G. Pugazhenthi, Fabrication of low-cost kaolin based tubular ceramic membrane and their application for algal separation, ***International Conference on Multifunctional and Hybrid Materials for Chemical Process, Energy, Environment and Medical Applications (ICMHCEE 2019),*** 9-11 September 2019, National Institute of Technology Tiruchirappalli, India.
2. M. Purnima, N. Arul Manikandan, K. Pakshirajan and G. Pugazhenthi, Preparation and characterization of low-cost kaolin based tubular ceramic membrane for algal separation, ***Green Technologies for Sustainable Water*** **(GTSW 2019)**, 1- 5 December 2019, Rex Hotel - 141 Nguyen Hue Boulevard, District 1, Ho Chi Minh City, Vietnam.
3. M. Purnima and G. Pugazhenthi, An efficient alternative to recover titanium dioxide particles from solution using kaolin based tubular ceramic membrane, ***Emerging Trends in Separation Science and Technology (e-SESTEC-2020)***, 22-26 March 2021, Bhabha Atomic Research Centre, Mumbai 400085, India.
4. M. Purnima, K. Pakshirajan and G. Pugazhenthi, Treatment of Onshore Oil Field Produced Water using Kaolin based Tubular Ceramic Microfiltration Membrane, ***International Conference on Advances in Chemical and Environmental Engineering (ACEE-2021)***, 16-17 December 2021, National Institute of Technology Raipur, India.
5. M. Purnima and G. Pugazhenthi, Facile synthesis of OSDA-free Chabazite (CHA) zeolite crystals coated kaolin ceramic support for the lab-scale continuous recovery of cerium ions from aqueous solution, ***Second International Conference on Sustainable Technologies for Water Treatment and Desalination (STWTD – 2022)***, 28-29 January 2022, National Institute of Technology Calicut, India.
6. M. Purnima, K. P. Goswami, M. Kumar, K. Pakshirajan and G. Pugazhenthi, Development of kaolin supported CHA zeolite composite membrane for the removal of cadmium from aqueous solution, ***International Symposium on Water Sustainability & Green Technologies (WSGT 2022)***, 25-26 November 2022, Rex Hotel - 141 Nguyen Hue Boulevard, District 1, Ho Chi Minh City, Vietnam.

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