

염색폐수의 처리를 위한 세라믹 분리막에 대한 고찰

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A Review on Ceramic Based Membranes for Textile Wastewater Treatment

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요 약: 다양한 산업 중에서 섬유 산업은 섬유 염색을 위해 가장 많은 양의 물을 사용하는데, 이는 여러 종류의 염료를 포함한 폐수의 방대한 배출로 이어진다. 염료의 제거를 위한 방법에는 오존 처리, 흡착 등의 다양한 처리 방법이 존재한다. 하지만 이러한 처리 방법은 폐수 재사용의 문제로 인해 처리 가격이 상승하기 때문에 성공적이지 못하다. 이에 대한 대안으로 막분리 공정이 폐수의 염료 처리를 위한 가장 적절한 기술로 보고되고 있다. 이때 사용되는 분리막은 고분자 분리막과 세라믹 분리막으로 나눌 수 있다. 세라믹 분리막의 장점에는 세척의 용이함, 긴 수명, 내열성, 내화학성, 그리고 기계적 안정성이 있다. 세라믹 분리막은 다양한 원료로 만들 수 있으며, 점토, 제올라이트, 플라이 애쉬와 같은 천연 재료는 저렴하고 구하기 용이하다. 본 리뷰에서 폐수처리는 크게 한외여과(ultrafiltration), 정밀여과(microfiltration), 그리고 나노여과(nanofiltration) 세 가지 공정으로 나누어져 있다.

Abstract: Among various industries, the textile industry uses the largest amount of water for coloring textiles which leads to a large amount of wastewater containing various kinds of dye. There are various methods for the removal of dye such as flocculation, ozone treatment, adsorption, etc. But these processes are not much successful due to the issue of recycling which enhances the cost. Alternatively, the membrane separation process for the treatment of dye in wastewater is already documented as the best available technique. Polymeric membrane and ceramic membrane are two separate groups of separation membranes. Advantages of ceramic membranes include the ease of cleaning, long lifetime, good chemical and thermal resistance, and mechanical stability. Ceramic membranes can be prepared from various sources and natural materials like clay, zeolite, and fly ash are very cheap and easily available. In this review separation of wastewater is classified into mainly three groups: ultrafiltration (UF), microfiltration (MF), and nanofiltration (NF) process.

Keywords: nanofiltration, ultrafiltration, microfiltration, ceramic membrane, textile wastewater

1. Introduction

Textile wastewater is characterized by its elevated variability and complexity, as a result of various reactive agents and dyes being used in high concentrations during dyeing and finishing processes. Along with this, the textile industry consumes approximately

0.2~0.5 m³ of freshwater per kg of fabric, making it one of the most polluting industrial sectors. To remediate this issue, various textile wastewater treatment methods such as physiochemical and biological treatments with activated sludge are implemented. However, conventional wastewater treatment methods do not allow the reuse of water in the textile production process.

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Therefore, membrane technologies have been proposed as an alternative to allow for wastewater reuse. Specifically, ceramic membranes have been gaining attention due to their enhanced mechanical, chemical, and thermal resistance compared to polymer membranes. Ceramic membranes are commonly fabricated with α -alumina (Al_2O_3) and other oxides (TiO_2 , ZrO_2 , SiO_2) [1-5].

However, ceramic membranes require a high fabrication cost due to material cost and high sintering temperature. Novel low-cost ceramic membranes are prepared with materials such as geomaterials and halloysite nanotubes (HNTs). Natural magnesite has been successfully used to fabricate low-cost ceramic micro-filtration (MF) membranes with lower sintering temperatures. Dopamine modified HNTs/Graphene Oxide (GO) composite membranes also showed promising performance in dye removal, recyclability, and antifouling[6-9].

The performance of ceramic UF membranes is significantly influenced by pore size, transmembrane pressure (TMP), and feed concentration. A larger pore size leads to a decrease in flux in two stages, a rapid initial decrease due to membrane-foulant interactions and a following steady decrease due to foulant-foulant interactions. An increase in TMP and feed concentration resulted in decreasing permeation rates and flux decline. These parameters are investigated in various studies to enhance the performance of ceramic membranes[10-13]. This review is classified into three sections as presented in Fig. 1.

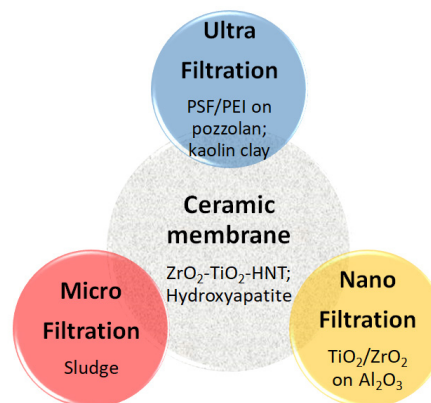


Fig. 1. Schematic of classification of the review.

2. Ceramic Membrane

Ceramic membranes are preferred over polymeric membranes due to their higher thermal and chemical stability. They can be fabricated in different forms, such as flat or tubular, and from cheap and naturally abundant minerals like clay. Generally, these membranes are prepared by heating to 500°C and kept for 2 h. Sintering is done at a higher temperature from 900 to 1200°C to investigate the properties of the membrane. The easy method of fabrication and application under harsh conditions for wastewater treatment make these kinds of membranes very popular. Halloysite nanoclay (HNT) doped ultrafiltration (UF) and tight-ultrafiltration ceramic membranes were fabricated using a sequential layer deposition method in 350°C calcination temperature and examined for mem-

Table 1. Summary of the Membrane Performance

Ceramic membrane	Water flux ($\text{L}/\text{m}^2 \cdot \text{h}$)	Contaminants	References
Halloysite nanoclay	11.2-16.2	Bovine serum albumin (BSA)	[14]
Hydroxyapatite-based	88	Cu, Fe, Zn, Cr, Cd	[15]
Tunisian natural kaolin clay	19 (raw wastewater) 30 (pretreated wastewater)	Textile dye	[18]
Drinking water treatment sludge (DWTS)	up to 100 (textile dye) up to 120(baking powder)	Textile dye, baking powder	[23]
Tight ultrafiltration (t-UF) ceramic membranes (TiO ₂ /ZrO ₂ skin layer on porous Al ₂ O ₃ support)	21.8	Reactive Brilliant Blue KN-R (RB KNR), Reactive Black 5 (RB5), and Reactive Red H-E7B (RR HE7B), Mixed Salts (NaCl/Na ₂ SO ₄)	[24]

brane performance and properties, including real wastewater treatment tests [14]. 0.45 μm commercial ceramic membranes were coated with 4 layers of HNT to create UF membranes and 0.2 μm ceramic membranes were coated with 6 layers of HNT to create tight UF membranes. Pure water flux was examined after each coating process on the membrane. Flux dramatically decreased until 3 coatings, after which flux decreased slowly. Bovine serum albumin (BSA) removal efficiency was up to 32% for UF membranes and almost 100% for tight UF membranes. The tight UF membranes were tested with 3 different wastewater samples (printing washing baths' hot discharge, mixed hot wastewater sampling points, and disperse printing washing baths). Significant chemical oxygen demand (COD) and total organic carbon (TOC) removal efficiencies were recorded, at 5-55%, 3-43% respectively. The difference in COD removal efficiency may be due to the difference in the cause of impurities and the form of COD (particulate matter or dissolved COD). The color removal efficiency was also noteworthy, at more than 40% for all samples. The flux values for the tests were recorded at 3 - 4.5 LMH, which increased when the water temperature increased to around 72°C, to around 10 LMH. However, the removal efficiencies for COD and color generally remained the same, at 40.6% and 46.5% respectively, despite the increase in flux. This is due to the temperature affecting the viscosity of the effluent while not having an effect on the active surface of the membrane. The results indicate the possible application of HNT doped membranes in the treatment of hot textile wastewater.

Hydroxyapatite-based bio-ceramic hollow fiber membranes (h-bio-CFHM) were fabricated using combined phase inversion and sintering techniques [15]. The hydroxyapatite (HAp) was prepared from waste cow bones using a calcination process. An increase in HAp content (40 wt% - 60 wt%) resulted in an increase in mechanical strength (38.9 MPa - 55.7 MPa). The increase in mechanical strength can be explained by the increase in stability of the morphology of the finger-like structures as HAp content rose. As sintering

temperature in membranes prepared with 60 wt% HAp increased from 900 to 1200°C, pore densification occurred, causing porosity to decrease from 51.3% to 35.1% while a further increase to 1300°C resulted in pinhole pores formings which led to a slight increase to 37.2%. Mechanical strength was measured at 53.2 MPa at a sintering temperature of 900°C, which then increased up to 202.5 MPa at a sintering temperature of 1200°C, finally dropping to 98.3 MPa at 1300°C due to the existence of pinole pores. COD, color, turbidity, and color removal was most efficient for membranes fabricated at 1200°C sintering temperature with 80.1, 99.9, 99.4, and 30.1% respectively. All membranes sintered from 1000 to 1200°C showed 100% removal efficiency for Cu, Fe, Zn, Cr, and Cd, due to the adsorption mechanism in the membrane which caused positively charged ions to have a high affinity towards the strong negative charge of the membranes.

The interaction mechanism between gravity-driven ceramic membrane (GDCM) system and small organic matter (SOM) was investigated using Extended Derjaguin-Landau-Verwey-Overbeek (XDLVO) theory [16]. The process was operated with 300 kDa ceramic membranes at 10, 20, 30, 60, 90, and 110 cm. The three model foulants, lysozyme (LYS), bovine serum albumin (BSA), and humic acid (HA) had significantly smaller molecular weights than the molecular weight cut off of the membranes. The LYS feed solution had a positively charged zeta potential, while the BSA and HA feed solutions had a negatively charged zeta potential. The ceramic membranes used (ceramic membrane, Fe_2O_3 deposited ceramic membrane, MnO_2 deposited ceramic membrane) showed electro negative growth trends. The decrease in operation height from 110 cm to 10 cm led to a significant flux decline from 0.53 to 0.32 $\text{L m}^{-2} \text{h}^{-1}$ (LYS filtration), from 0.68 to 0.43 $\text{L m}^{-2} \text{h}^{-1}$ (BSA filtration), and from 0.56 to 0.28 $\text{L m}^{-2} \text{h}^{-1}$ (HA filtration). A shorter operation height resulted in higher membrane fouling values. Retention rate was also affected by operation height, especially negatively charged foulants. The retention rate for negatively charged foulants significantly increased from

22% and 9% for BSA and HA at operation height of 110 cm to 48% and 33% for BSA and HA filtration at operation height of 10 cm. Positively charged foulants were slightly affected, as LYS retention rate grew from 8% to 14%. In short operating distances, Van der Waals (VdW) forces and acid-base (AB) forces were the dominant interaction force in short-distance range, while electrostatic (EL) forces were dominant in an intermediate-distance range, and permeation drag (PD) forces were dominant in a long-range distance.

2.1. Ultrafiltration

Polysulfone (PSf) and polyetherimide (PEI) composite ultrafiltration (UF) membranes were fabricated on flat pozzolan support using spin/spray-coating method and studied for its application in dye solution decoloration[17]. An increase in PEI content from 5 to 20 wt% led to growth in polymer particles in the membrane from 9.3 μm to 25.3 μm , and the surface became smoother. The smoother surface of the membrane could help reduce fouling during filtration. Water permeability dramatically decreased from 64.0 to 24.2 $\text{L/h m}^2 \text{ bar}$ following the increase in PEI content from 5 to 20 wt%. Aqueous dye solutions containing acid orange 74 (AO74) and methyl orange (MO) were used to evaluate the selectivity of the membrane. As PEI content increased, AO74 and MO rejection increases from 89.2% to 95.8% and from 72.3% to 75.0% respectively. At 3 bar pressure, the highest flux value of 122 and 125 L/h m^2 could be achieved.

An ultrafiltration (UF) membrane was fabricated using Tunisian natural kaolin clay and studied for its application in raw and pretreated textile effluent treatment[18]. At 55.25%, silica had the highest component ratio of the kaolin clay powder, followed by 24.17% of alumina, as well as trace amounts of other oxides. As sintering temperature of the membrane increased from 950 to 1000°C, the surface became more homogenous while the membrane reached the glassy phase at 1050°C. Tensile strength increased accordingly from 4.84 MPa to 13 MPa as sintering temperatures increased from 950 to 1000°C. Chemical resistance was al-

so highest in membranes sintered at 1000°C, making it the optimal membrane to use in treating both raw and biologically pretreated wastewater. The pretreatment of the wastewater improved membrane separation performance, as evidenced by the quasi-stabilized permeate flux of 19 $\text{L h}^{-1} \text{ m}^{-2}$ for raw wastewater and 30 $\text{L h}^{-1} \text{ m}^{-2}$ for pretreated wastewater at 3 bar. The membrane showed high COD, color, conductivity, and turbidity rejection rate for both raw (80%, 98%, 36.48%, and 99%) and pretreated effluent (93%, 100%, 50%, 100%). In both raw and pretreated effluent, reversible fouling was found to be the dominant fouling mechanism.

Ceramic membrane ultrafiltration was studied for its applications in textile wastewater for washing processes[19]. Four different MWCO membrane sizes (300 kDa, 50 kDa, 15 kDa, and 3 kDa) were evaluated for optimal membrane cut-offs on three different wastewater types were (disperse printing wash baths mix, mixed hot wastewater, and printing wash baths hot discharges mix). Smaller MWCO membranes showed higher removal efficiency for COD, TOC, and color, with the 3 kDa membrane showing the highest efficiency in all three wastewater types. For 3 kDa permeate, COD removal was 25%, 66%, and 85%, TOC removal was 16%, 61%, and 84%, and color removal was 99%, 90%, and 84% in disperse printing washing baths mix, mixed hot wastewater, and printing washing baths hot discharges mix respectively. Membrane flux rapidly declined at 50 kDa for disperse printing washing bath mix, mixed hot wastewater, and at 50 and 300 kDa for printing washing baths hot discharges mix, leading to the conclusion that most contaminants were 50 kDa to 300 kDa in size for all wastewater types. The larger contaminant size resulted in physically removable cake fouling (Rc) being the main fouling mechanism for all membranes. When 3 kDa membranes were used, 255 m^3 , 885 m^3 , and 2550 m^3 of hot wastewater could be removed for disperse printing washing baths mix, printing washing baths hot discharges mix, and mixed hot wastewater point respectively.

A two-stage treatment process for textile effluent treatment using ceramic membrane-based ultrafiltration (UF) process and management of vegetable waste derived biochar for dye removal was proposed[20]. Textile effluents with various COD content and dye were put through a UF process to remove COD then an adsorption unit to remove dye. 4 textile effluents with varying dye and COD content, TE₁, TE₂, TE₃, and TE₄ were collected. The UF process removed around 50% of COD, 40% of color, and > 70% of TSS and turbidity. As transmembrane pressure (P) increased from 1 to 4 bar, the permeate flux increased, though not in a linear trend. The pH of the dye solution did not significantly affect the adsorption behavior of dyes. Within the first hour, dye removal was around 80-95%, and around 99.9% removal was recorded after 3 h. The combined system showed > 99% dye removal, 70-80% COD removal, and complete removal of turbidity, making the treated effluent reusable in dye processing. For efficient TDS removal, a reverse osmosis process could be used. The biochar showed effective dye adsorption, due to the high surface area and porosity. The composted biochar showed low toxicity and high nutritional value for plants. The combined system was estimated to cost 0.02 USD/L, making it a viable cost-effective option.

Ceramic ultrafiltration (UF) membranes were studied for their application in the treatment of textile mercerization wastewater. Three different MWCO membrane sizes, 500, 2, and 1 kDa were used[21]. All three membranes show a decline in the normalized flux (J_p/J_{p0}) after 30 minutes of ultrafiltration at a 3 m s^{-1} cross-flow velocity (CFV) and 20°C . A drastic decline in flux was found in the 500 kDa membrane, while small changes were found in 1 and 2 kDa membranes. As CFVs intensified, both 500 kDa and 1kDa membranes showed a decrease in fouling. As temperature increased from 20°C to 50°C , the fouling process occurred faster for both membranes due to the decrease in viscosity according to temperature increase. In addition, 500 kDa membranes showed significant chemically irreversible fouling buildup, with only 74-78%

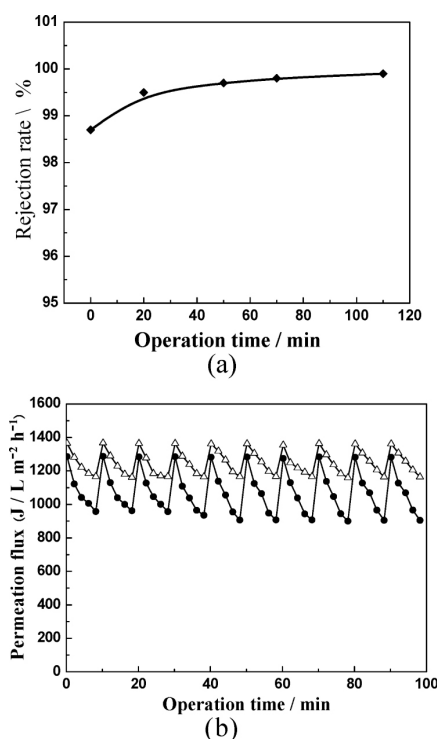


Fig. 2. (a) Change of rejection rate during ceramic microfiltration with time-on-stream. (b) Change of transmembrane permeation flux with only backflushing (●) and those with backflushing and ultrasonic irradiation (△). (Working conditions: $T = 308.0 \text{ K}$, TiO_2 concentration = 1.0 g/L , $\text{pH} = 7.0$, nominal ultrasonic power = 100.0 W , $P = 0.2 \text{ MPa}$.) (Reproduced with permission from Cui *et al.*, 23, Copyright 2011, American Chemical Society).

deionized water (DW) normalized flux of new membrane regained after four cycles. According to these characterizations, 1 kDa membranes showed the most optimal results with TSS, turbidity, and color removal of 91.9%, 98.15%, and 98.05% respectively.

2.2. Microfiltration

A novel process of incorporating sonophotocatalysis and ceramic membrane microfiltration was proposed for the degradation of methyl orange (MO)[22]. Fig. 2 represents the membrane performance.

For the degradation rate of MO, sonophotocatalysis had a higher rate than photocatalysis and sonolysis, but it was less than the sum of the rate of sonolysis and photocatalysis. pH value and TiO_2 dosage affected the effect of sonolysis, photocatalysis, and sonophotocatalysis.

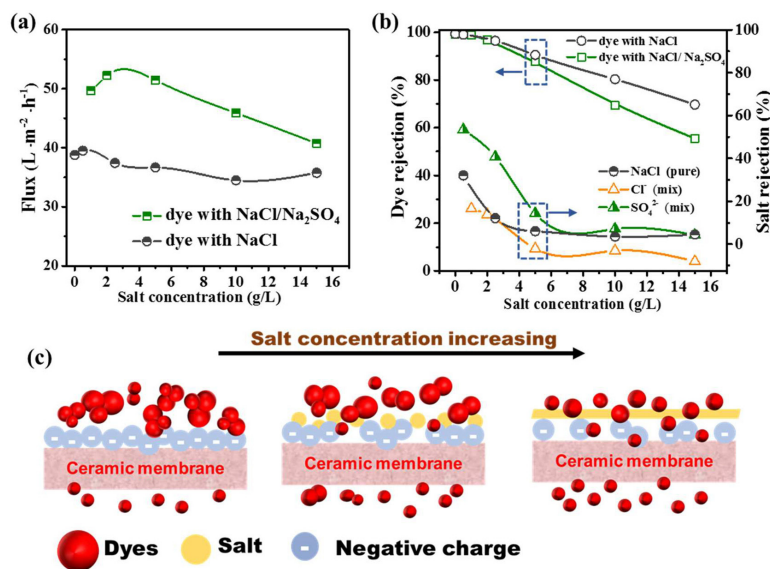


Fig. 3. Salt concentration effect on t-UF membrane separation of dye and salts: (a) permeation flux, (b) dye and salts rejections, and (c) schematic diagram of dye-salt interactions with increased salt concentration (conditions: TMP = 2 bar, CFV = 2 m/s, $C_{(\text{dye, RR HE7B})} = 0.5 \text{ g/L}$, $T = 25^\circ\text{C}$, $C_{(\text{total salts})} = 0\text{--}15 \text{ g/L}$, $\text{pH} = 6.5 \pm 0.3$) (Reproduced with permission from *Ma et al.*, 20, Copyright 2017, American Chemical Society).

A lower pH environment improved the MO degradation under photocatalysis and sonophotocatalysis but did not show much change in sonolysis. After the establishment of adsorption equilibrium, as TiO₂ dosage increased, degradation rate showed a slight decrease. Smaller TiO₂ particles were also found to be beneficial in improving the synergistic effect between sonolysis and photocatalysis. While sonication was found to potentially damage polymeric membranes, ceramic membranes did not show signs of damage due to their preparation process.

Drinking water treatment sludge (DWTS), which is alumina and silica-rich, was used to fabricate ceramic microfiltration (MF) membranes using dry pressing and sintering techniques[23]. DWTS mainly consists of Al₂O₃, which makes up 45.99% and SiO₂, which makes up 18.04%. Various sintering temperatures ranging from 950 to 1100°C were used to fabricate DWTS membranes, to examine their effects on morphology, porosity, water absorption, water permeability, and mechanical strength. DWTS membranes that were sintered at 1050°C showed optimal morphology with no cracks. As sintering temperature increased, porosity and water

absorption decreased from 64.60 to 34.15% and from 45.05 to 17.46% respectively. Water permeability improved as sintering temperature was raised, which is due to the increase in pore size. Higher mechanical strength was found with higher sintering temperatures, at 24.06 MPa for membranes sintered at 1150°C, and 11.59 MPa for membranes sintered at 950°C. Based on these characterizations, DWTS membranes sintered at 1050°C are the optimal membrane. When compared to other low-cost ceramic membranes in literature, DWTS membranes showed promising properties, with small pore size (0.92 μm) and high permeability (724.5 L/h.m².bar). In addition, in real textile wastewater treatment tests, DWTS membranes showed high COD removal (50-68%) and turbidity removal (96.5-98.6%).

2.3. Nanofiltration

Tight ultrafiltration (t-UF) ceramic membranes were fabricated and compared to polymeric nanofiltration (NF) membranes in the desalination of textile wastewater[24].

Fig. 3 represents membrane performance and schematic representation of salt dye interactions. The t-UF

membranes consists of a $\text{TiO}_2/\text{ZrO}_2$ skin layer on porous Al_2O_3 support, while the polymeric NF membrane (DK from GE) consists of polyamide. Permeability was higher for the t-UF membrane than the DK membrane for Reactive Red H-E7B (RR HE7B) dye solution, at $21.8 \text{ L m}^{-2} \text{ h}^{-2} \text{ bar}^{-1}$ and $5.3 \text{ L m}^{-2} \text{ h}^{-2} \text{ bar}^{-1}$ respectively. This is due to the difference in surface charge, where the DK membrane is more negatively charged than the t-UF membrane. Dye retention of Reactive Brilliant Blue KN-R (RB KNR), Reactive Black 5 (RB5), and RR HE7B for t-UF membranes had a competitive value of 97% compared to 99% of DK membranes. NaCl and Na_2SO_4 rejection were significantly lower for t-UF membranes than DK membranes, at 5% and 10% and 70% and 99% respectively. A significant drop in dye rejection was found with an increase in salt concentration, from 99.32% to 55.5% with NaCl/ Na_2SO_4 and from 99.32% to 69.7% with NaCl alone. The membrane and the dye have a weaker electrostatic interaction as salt concentration increases, making the dominant mechanism steric hindrance as opposed to electric exclusion. Dye charge affects salt permeation through the membrane, with negatively charged dyes being more compatible with the t-UF membrane.

A pilot-scale ceramic membrane system was used to treat caustic bath wastewater ($\text{pH} > 13$) as well as examined for reusability of the filtered wastewater[25]. The system consists of ultrafiltration (UF) and nanofiltration (NF) membranes and operates on batch and continuous modes. The system goes through three scenarios where it utilizes UF, NF, and combined UF and NF membranes. Five cycles of the system were tested. Removal efficiencies of organic carbon, chemical oxygen demand, total hardness, and color were higher in the combined UF and NF scenario compared to only UF at 67, 71, 42, and 92%, which is due to the smaller pore size of NF membranes. Sodium recovery was at least 50% for all five cycles. A combined UF and NF system was found to be the best treatment, due to its removal efficiency and sodium recovery. Cost analysis was done under the assumption of a daily caustic recovery feed volume of $1.3 \text{ m}^3/\text{day}$, filtration time of

8h, membrane area 16 m^2 , average wastewater flux of 20 LMH and recovery rate of 80%. Approximately $480 \text{ m}^3/\text{year}$ of caustic solution is recovered when ceramic membrane filtration systems were applied.

A combined hydrodynamic cavitation (HC), Fenton agent, and membrane separation system was used to treat textile wastewater[26]. Boehmite sol was prepared and coated via wet impregnation on tubular ceramic microfiltration membranes to modify its properties to nanofiltration using a layer by layer technique. Hydrodynamic cavitation (HC) performance varied with inlet pressure. Increasing inlet pressure (2.5, 5, 7, and 9 bar) resulted in increased TOC removal up to 7 bar, after which TOC removal decreased. An increase in inlet pressure results in high density vaporous cavity cloud forming, which causes a decrease in the number of OH radicals. Thus, using hydrogen peroxide (H_2O_2), which can produce auxiliary OH radicals, in combination with HC can improve contaminant removal rate. However, exceeding the optimal H_2O_2 concentration can result in the degradation of the HC system due to the scavenging of the formed OH radicals. Nanofiltration combined with HC showed 21.95% TOC removal, which was higher than HC alone, and HC, nanofiltration, and H_2O_2 addition combined showed a further increase of 36.63% TOC removal in 90 minutes. A combination of HC, nanofiltration, and Fenton method showed synergistic effects, with 58.85% TOC removal, due to the increased speed of OH radical generation and contaminant destruction. However, the treated wastewater should go through an additional reverse osmosis (RO) system for the discharge and reuse of the effluent.

3. Conclusions

Wastewater from textile industries is one of the most polluting sources which needs to be treated for the protection of our environment. It contains dye, surfactant and is usually high in chemical oxygen demand (COD) and biological oxygen demand (BOD). Heavy metal elements like cadmium, chromium and lead are present in wastewater from textile industries which is

used to prepare the color pigment. Ceramic membranes are important materials for treatment of wastewater that can be easily prepared from titanium dioxide, alumina or silica. There are very cheap sources of raw material like natural clay used for the fabrications of ceramic membranes. Thermal stability, easy processability, and long life of the membrane makes them very popular in separation membrane process. This review discusses about the preparation and wastewater treatment in three different sections: i) ultrafiltration, ii) microfiltration and iii) nanofiltration.

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