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ORIGINAL ARTICLE

Heavy metals and color retention by a synthesized inorganic membrane

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Abstract In this study, a new type of a double-layer ceramic membrane was used for the filtration of wastewater. The synthesized membrane consists of a macroporous substrate (with pore size of about 0.1 μm) prepared following the colloid filtration technique and a thin film functional layer (with pore size of about 10 nm) carried out according to the sol–gel preparation method.

The ceramic membranes were tested for the removal of cadmium, zinc, Methylene Blue and Malachite Green from water under a pressure of 5 bar and a treatment time of 2 h. Liquid filtration and flow tests through these membranes resulted in a rejection rate of 100% for Methylene Blue and Malachite Green. This paper also presents the ability of the tubular membrane prepared to separate heavy metals (cadmium and zinc) from their synthetic aqueous solutions. The influence of the applied pressure, feed solute concentration, feed pH on the rejection of cadmium and zinc ions was studied. Retention rates of cadmium and zinc ions of 100% were observed for an initial feed concentration of 10^{-4} mol/L.

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1. Introduction

In recent years, there has been a growing concern about environmental issues and specifically about the presence of dyes and heavy metals in water.

The treatment of industrial wastewater containing heavy metals is a major concern because of their high toxicity (Hestekin et al., 1998; Seiler et al., 1988). The separation of dyes from industrial effluents is one of the most important environmental concerns (Gonzalez-Mŭnoz et al., 2006; Vieira et al., 2001). Increased awareness on the toxicity of metals prompted the implementation of strict regulations for their disposal. Several physicochemical techniques have been used for the removal of contaminants such as cadmium and zinc including: straining, sedimentation, impaction, interception, adhesion, chemical adsorption, physical adsorption, flocculation, and biological growth (Xu et al., 1999; Zollinger, 1987; Ghezzi et al., 2008; Drew et al., 2006; Polat and Erdogan, 2007; Cioffi et al., 1996). Zinc and cadmium ions are toxic pollutants, which are released into the environment through a variety of industrial operations. They exhibit a long environmental persistence and bioaccumulation in living organisms with the potential to cause kidney and liver damage and blood (Monser and Adhoum, 2002; Van der Bruggen et al., 2001a,b; Ritchie and Bhattacharyya, 2002). Membrane filtration has received considerable attention for the treatment of inorganic effluents,

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since it is capable of removing not only suspended solid and organic compounds, but also inorganic contaminants such as heavy metals as well.

On the other hand, industries such as textile, paper, and plastic industries release colored matter to water bodies often without treatment.

Membrane technology had gained its popularity in dye and metal separation by ultrafiltration, nanofiltration and reverse osmosis (Strano et al., 2002; Song et al., 2006; Tan et al., 2001; Belouatek et al., 2008). Industrial applications of porous ceramic membranes with their various advantages, such as better thermal, chemical and mechanical resistance, controllable micro-structure and little pollution to our environment, have been attracting much attention in the scientific community recently (Belouatek, 1998; Belouatek et al., 2005).

In recent years, mineral-based porous ceramic membranes have been considered for their low costs and additional functions. Tubular ceramic membranes with good performance parameters, such as Permeate flux and filtration efficiency can be obtained by using an asymmetric configuration in which different layers are coated on a porous support that offers sufficient mechanical strength and thermal shock resistance, which is desirable for hot gas filtration application (Belouatek, 1998; Belouatek et al., 2005; Ezziane et al., 2010). Transport properties of the ceramic membranes are significantly influenced by the composition of the treated solution. Cations such as mercury (Hg^{2+}), cadmium (Cd^{2+}), zinc (Zn^{2+}) and lead (Pb^{2+}), and anions such as nitrate (NO_3^-) and chromate ($Cr_2O_7^-$) can be easily removed via facilitated transport (Elmaleh and Naceur, 1992; Mulder, 1996).

In this work, the feasibility of a new ceramic membrane synthesized for ultrafiltration application was investigated. The main objective of this study was to evaluate the ability of the prepared membrane to separate polluting substances (dyes and heavy metals) from wastewater.

2. Materials and methods

The tubular support was synthesized from high purity kaolin. Tubular supports were prepared in the laboratory by the extrusion method. A powder of oxide was mixed with kaolin powder (particle size 0.1–0.2 μm) and then with water in the proportion of 30–35% of the powder weight. The kaolin barbotine had a 65–75 centipoise final viscosity as measured by a torsion Gallenkamp viscometer.

After mixing, the obtained kaolin barbotine was put in plaster molds. The plaster was then extruded to obtain a tubular support of 4 mm internal diameter and 30 cm length. After drying, the support was sintered at 1373 K for consolidation (Belouatek et al., 2005, 2008; Belouatek, 1998; Ezziane et al., 2010).

A microfiltration layer consisting of an intermediate mixture of 50% kaolin and 50% ZrO_2 was then coated on the ceramic support before depositing the final sol–gel layer. This intermediate layer allows the membrane to be maintained without infiltration into the support and serves to bridge the gap between the macroporous support and the ultrafiltration layer. After firing the assembly at 1373 K for 2 h, a double layer support named (DLS) with a mixture layer of 0.1 μm pore diameter and a thickness of 10 μm was obtained.

To improve the membrane performance, a thin layer of alkoxide (98% tetraethylorthosilicate) was added onto the DLS support to obtain a ceramic membrane named activated double layer support (ADLS). The third layer was prepared via a dip coating technique from colloidal boehmite nanoparticle suspensions synthesized with the alkoxide prepared. Hydrolysis was followed by peptization under acidic conditions. The slip-casting procedure was followed by drying and calcination at a temperature of 753 K.

Filtration was of tangential type under an applied pressure of 5 bar (Belouatek et al., 2005, 2008; Belouatek, 1998; Ezziane et al., 2010). The feed colored solutions were prepared from Malachite Green (MG) and Methylene Blue (MB) of analytical grades. The rate of retention (R (%) $= 1 - C_P/C_o$ where C_o is the initial solution concentration and C_P the concentration in the filtrate) of the colored solution was measured by visible spectrophotometry (Optizen 120 UV). The maximum wavelength at $\lambda_{max} = 615$ nm and $\lambda_{max} = 665$ nm for MG and MB, respectively (Khattri and Singh, 2009; Mukerjee and Gosh, 1970).

The structure and morphology of the ADLS membrane were characterized by scanning electron microscopy (Philips SEM5050 microscope 3000F). The porosity of the samples was calculated by using an AutoporeII 9220 V3.01 mercury porosimeter (down to 3 nm pore diameter). The thermal stability of the membranes was determined by thermogravimetric analysis (TGA) using a (Setaram–setaram) thermogravimeter. Phases present in the mixture of 50% kaolin and 50% ZrO_2 (raw powder and heated to 1373 K) were analyzed using an X-ray diffractometer (Siemens, Germany) with CuKα1 radiation ($\lambda = 1.54056$ Å).

3. Results and discussion

3.1. Thermal analysis

Thermal analysis was used to determine the weight changes within the membrane as a function of temperature. Calcination has great influences on the structure and permselectivity of the DLS and ADLS membranes. In order to establish an efficient heat treatment procedure, the modified membrane support powders were examined by thermogravimetric analysis as shown in Fig. 1.

The measurement was carried out under air in the temperature range of 293–1273 K with a heating rate of 283 K/min. The thermogravimetric analysis curve of membrane powders showed that the total weight loss was about 2.5% and

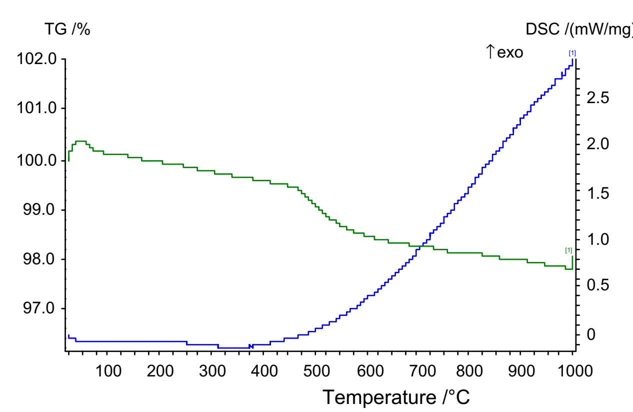

Figure 1 Thermogravimetric analysis of DLS membrane.

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