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Use of sludge membrane filters as an alternative method for processing wastewater treatment

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ABSTRACT

This research highlighted the ability of sludge membrane filters (SMF) as an alternative technique for processing wastewater treatment. The sludge ash based membranes were prepared by blending cellulose acetate (CA) with sewage sludge ash (SSA) from wastewater by dry–wet phase inversion method in various proportions of CA/SSA (100/0, 90/10, 80/20 and 50/50 wt%). Characterization of prepared membranes was performed such as water content, X-ray diffraction analysis and mechanical strength. Also, the possibility of solute rejection of nickel ions from wastewater using SMF was evaluated. Effects of organic additives concentration such as polyethylene glycol (PEG 600) in the casting solution were studied in range of 0–10 wt%. Results reported that the best performance of CA/SSA blend membranes was at 50/50 wt% Furthermore, investigation of SMF in reducing the water turbidity, COD, BOD, TSS and total dissolved solids of effluent wastewater from Abu Rawash plant was depicted.

Keywords: Sludge membrane filters; Wastewater treatment; Cellulose acetate/sewage sludge ash; Phase inversion method; Characterization

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1. Introduction

The production of sewage sludge from wastewater treatment plants in Egypt was estimated to be 2 million tons dry solids in 2015 [1]. This may cause pollution if it is not well managed by incineration, landfilling, road surfacing or used as fertilizer [2,3]. Waste reuse will minimize the environmental problems associated with their build-up and reduce the use of noble starting materials [4].

One of the most common applications for the membrane separation process for water treatment is to remove, concentrate or separate various components with different sizes or dimensions, such as particles, colloids, bacteria, viruses, proteins, humic matters, organic compounds, soluble salts, heavy metal ions and detergents [5,6]. Among these components, especially, heavy metal ions such as Hg^{2+}, Cd^{2+} and Ni^{2+} are highly toxic to human bodies even at very low concentrations (ppm), therefore their removal is more crucial [7,8]. Some of the conventional membrane separation processes such as microfiltration and ultrafiltration (UF) cannot remove them because of the relatively larger pore sizes of the membrane (>50 nm). In contrast, nanofiltration and reverse osmosis processes are generally not economical because of the high operating pressures (5–80 bar) and low permeate fluxes [9]. It is always desirable to be able to remove heavy metal ions with membranes of larger pores in order to obtain high permeate fluxes and low energy consumption [10,11].

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To achieve high removal efficiency and high selectivity, this may be accomplished by using microporous adsorptive membranes that separate the desired or undesired substances from solutions through affinity adsorption, rather than size exclusion, sorption–diffusion or ion exchange principles. When the feed is made to pass through the membrane thickness, the desired components to be removed or separated will interact with the functional groups on the external and internal surfaces while the liquid or other components that have low affinity for the membranes will pass through the membrane freely, and most of the commercial polymeric membranes are prepared by the phase inversion method [12].

Phase inversion technique is a common technique and a well-known method in producing asymmetric membrane [13]. The main important component in membrane preparation via phase inversion process is the polymeric materials, which determine the characteristics and properties of the produced membranes. Some of these polymeric materials such as cellulosics (e.g., cellulose acetate (CA) and cellulose nitrate), polyacrylonitrile and related block-copolymers, polysulfone/polyethersulfone/sulfonated polysulfone/sulfonated poly(ether ether sulfone), polyvinylidene fluoride, polyamide/polyetheramide, aliphatic polyamides, polyether ketone and sulfonated poly(ether ether ketone) are used as a backbone of UF membrane [14].

The selection of polymer material as a polymer backbone to prepare an adsorptive membrane via phase inversion process is very crucial due to (1) membrane surface, (2) cost in production, (3) thermal and chemical stability, (4) fouling and (5) mechanical strength. Generally, these properties are fundamentally related to chemical and physical nature of polymers.

Shankar et al. [15] investigated the use of CA which was blended with sulfonated poly(ether ether ketone) in various compositions and subjected to different UFs. Sivakumar et al. [16] prepared CA/polyurethane membranes then studied the effect of the changed additive on blend membranes. While modified CA-based membranes were prepared in the absence and also in presence of polymeric additives such as polyvinylpyrrolidone and polyethylene glycol (PEG 600) in various composition [17].

CA has been the most widely studied polymer for adsorptive membrane, the reactivity of cellulose comes from the hydroxyl groups (–OH) on the polymer backbones [18]. However, the hydroxyl group does not cause the direct binding capability to heavy metal ions or proteins but it needs further derivation with other more reactive functional groups such as $–NH_2$, oxides, $–SO_3H$ and carbonaceous materials.

Many researchers had detected more reactive polymer to be blended with SSA to produce membrane with high porosity, high carbons content material such as activated carbon and an extended particulate surface area of the SSA [19].

The objectives of the present work are to prepare adsorptive membranes or sludge membrane filter (SMF) by blending the SSA from wastewater treatment plants with CA in different proportions and evaluate their effect in Ni ions removal from synthetic wastewater as well as actual sanitary wastewater obtained from Abu Rawash plant.

2. Materials and methods

2.1. Materials

Materials used in SMF preparation are CA purchased from Sigma-Aldrich Chemie GmbH (Germany), sewage sludge waste (SSW) sample used in this study was collected from El-Berka municipal wastewater treatment plant (Qism El-Salam, Cairo Governorate). Acetic acid (glacial, 99%–100%), poly(ethylene glycol 600) was procured from Merck (I) Ltd., and used as a non-solvent additive, Annular grade N,N-dimethylformamide (DMF) and sodium lauryl sulfate (SLS) were obtained from Sigma-Aldrich Chemicals Co.

2.2. Methods

2.2.1. Preparation of SSA

The SSW sample was dewatered to sludge cake and burned in a modular incinerator at 600°C for 2 h to provide incineration ash and then ground. The ground SSA was screened by 75 μm sieve [20].

2.2.2. Membrane preparation

The blend solutions based on CA and SSA (total polymer concentration = 20 wt%) were prepared by dissolving CA/SSA with different proportions of 100/0, 90/10, 80/20 and 50/50 wt% in the presence and also in absence of additive PEG 600 since five different concentrations were tested in range of 0–10 wt% and dissolved in DMF (80 wt%) under constant mechanical stirring at 400 rpm for 1 h at 25 ± 2°C. The homogeneous solution that was obtained was allowed to stand at room temperature (25°C) for 20 min in an airtight condition to get rid of air bubbles [21]. The preparation of sludge filter membranes is illustrated in Fig. 1.

Prior to casting, a 2 L gelation bath, consisting of 2.5% (v/v) DMF solvent (to reduce the rate of liquid–liquid demixing and macrovoids and also to increase the hardness) and 0.2 wt% surfactant, SLS (to reduce surface tension at the polymer–non-solvent interface) in distilled water (non-solvent), was prepared and kept at 20 ± 1°C.

After 1–2 h of gelation, the membranes were removed from the gelation bath and washed thoroughly with distilled water to remove all DMF and surfactant from the membranes. The membrane sheets were, subsequently, stored in distilled water, containing 0.1% formalin solution to prevent microbial growth [22].

2.2.3. Ultrafiltration process using SMF

The UF experiments using SMF were carried out in a batch mode, dead end cell as shown in Fig. 2. This cell was connected to a compressor with a pressure control valve and gauge through a feed reservoir. The experiments were performed at Chemistry lab for water and wastewater, Housing and Building Research Centre.

2.2.4. Characterization of SMF

2.2.4.1. X-ray diffraction analysis Blending of CA/SSA was tested using XRD analyzer, after placing the samples in

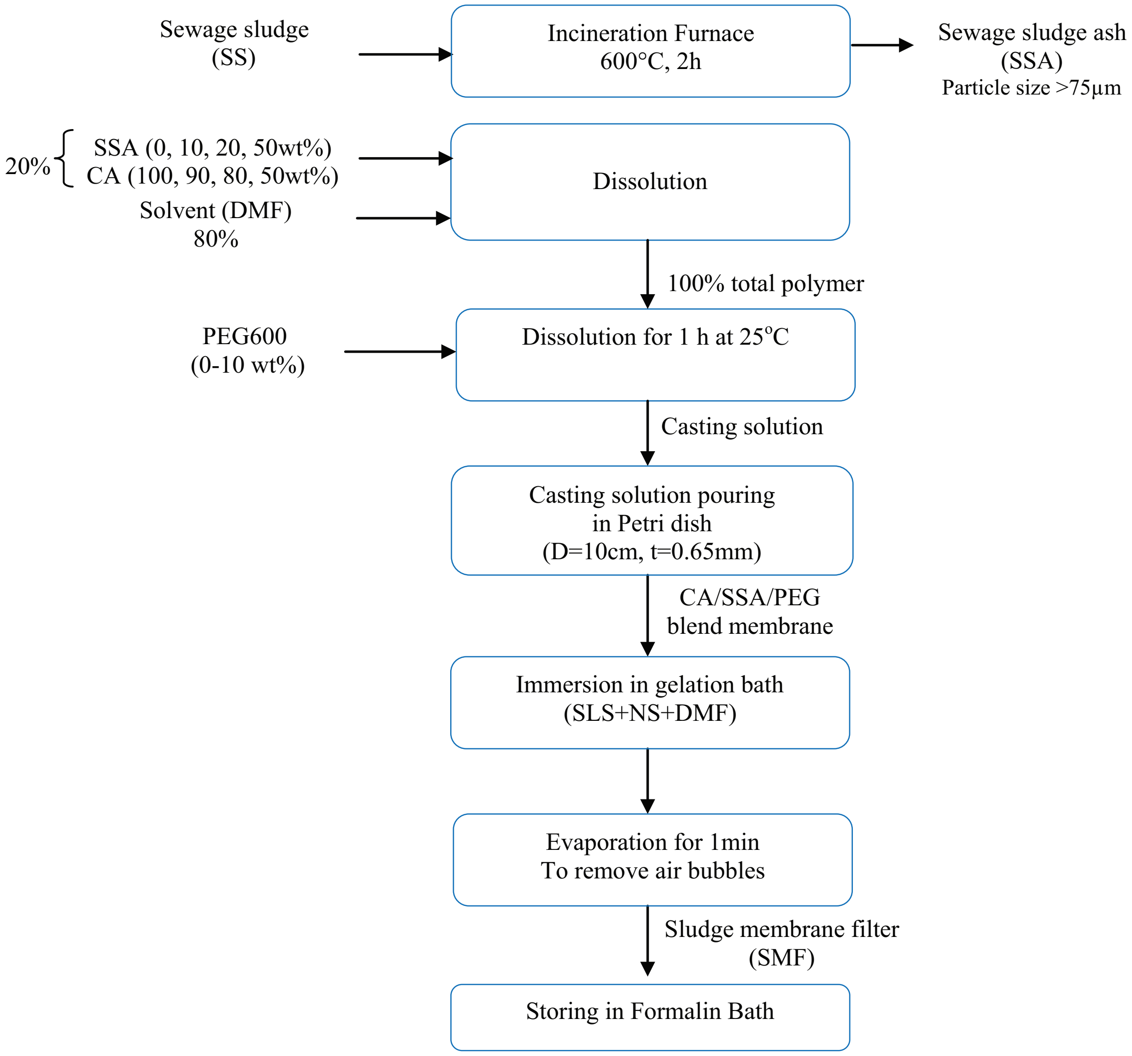

Fig. 1. Preparation of sludge membrane filter (SMF).

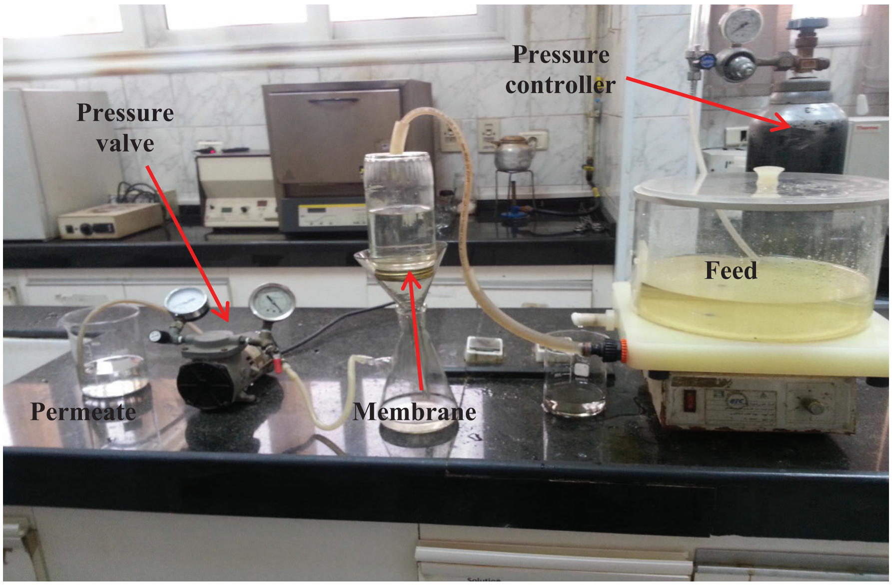

Fig. 2. Ultrafiltration process setup.

stainless steel sample holder, the XRD patterns were recorded at the radiation wavelength (Cu Kα = 1.5418 Å).The X-ray current and voltage values were 40 kV and 40 mA, respectively. The diffraction angle (2θ) ranged from 5° to 50° at a step size of 0.0167° [23]. The XRD analysis was carried out at XRD lab for materials institute.

2.2.4.2. Water uptake The water uptake (WU) of membrane samples was estimated by removing the membranes from water and weighing immediately after blotting the free surface water, and drying in oven for at least 8 h at 110°C. The percentage of WU of the prepared membranes was calculated using Eq. (1) [10].

$$\mathrm{WU} = \frac{W_w - W_d}{W_w} \times 100 \tag{1}$$

where WU is the water uptake of prepared SMF, %; W_w is the wet weight of prepared SMF, g; W_d is the dry weight of prepared SMF, g.

2.2.4.3. Mechanical strength measurement The tensile stress, elongation ratio and young's modulus values at break for the blending membranes CA/SSA/PEG are tested by Shimadzu machine, model AG-X, 100 kN, Japan [24]. Young's modulus of the polymeric films was measured using a Shimadzu autograph in air at room temperature. The correlation of elongation calculation is shown in Eq. (2) and the correlation between stress (σ in MPa) and elongation (ε in %) was determined by Eq. (3) [25]:

$$\sigma = E \times \varepsilon \tag{2}$$

where E is Young's modulus, MPa and

$$\varepsilon = \frac{Lo - L}{Lo} \times 100 \tag{3}$$

where Lo is the original length, mm; L is the length after elongation, mm.

2.2.5. Membranes testing for pollutants removal

The prepared SMF was tested for removal of nickel ions from synthetic wastewater and reduction of chemical oxygen demand (COD), biological oxygen demand after 5 days (BOD_5), total suspended solids (TSS) and total dissolved solids (TDS) from Abu Rawash wastewater treatment plant effluent.

2.2.5.1. Preparation of industrial wastewater According to Standard method for water and wastewater reported in ASTM [26], a nickel solution has been prepared and used on bench-scale experiments. The simulated stock of nickel ions (1,000 mg/L) was prepared by dissolving 4.47 g of an annular grade of respective salt in 1 L of distilled water. The salt used is nickel sulfate, $NiSO_4.6H_2O$. The stock solution was further diluted with distilled water to desired concentration of 25, 20, 10 and 5 mg/L.

2.2.5.2. Sanitary wastewater Wastewater samples were collected from effluent Abu Rawash wastewater treatment plant in Giza, Egypt. Physicochemical analysis of wastewater sample is illustrated in Table 1.

The synthetic wastewater was filled with nickel ions in the system. This was introduced to the experimental setup in Fig. 2. Filtration permeate solution was collected in a 250 mL beaker and the concentration of nickel ions was estimated using atomic absorption spectrometer (Model ICE 3000 Series – Thermo Scientific (Giza, Egypt), with air acetylene flame at wavelength of 231.6 nm). The percentage of metal ion rejection was calculated according to Eq. (4):

$$\%\mathrm{NR} = \left(1 - \frac{C_P}{C_F}\right) \times 100 \tag{4}$$

where %NR is the percentage of nickel ions rejection; C_p is the concentration of permeate; C_f is the concentration of feed.

3. Results and discussion

Adsorptive membranes prepared from CA blended with SSA in different proportions (total polymer concentrations 100 wt%) are shown in Fig. 3.

Table 1
Chemical analysis of Abu Rawash WWTP effluent before and after treatment with CA/SSA membrane

Parameter	Effluent before treated	Effluent after treated	% Removal
pH	6.9	7.0	–
DO (mg/L)	1.8	2.3	–
Temperature (°C)	28.5	25	–
Turbidity (NTU)	32.0	10.5	67
Color	Gray	Colorless	–
COD (mg/L)	167	20	88
BOD_5 (mg/L)	96	11	88.5
Ammonia (mg/L)	13.42	7.2	46
Nitrate (mg/L)	0.69	0.35	49
TS (mg/L)	606	422	30.3
TDS (mg/L)	492	319	35
TSS (mg/L)	113	12	89

Note: WWTP: wastewater treatment plant; DO: dissolved oxygen; and TS: total solids.

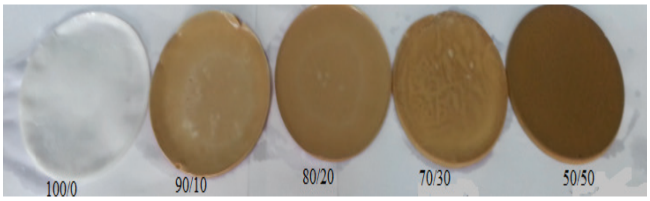

Fig. 3. CA\SSA blend membranes at various wt%.

3.1. XRD analysis of prepared SMF

Results of XRD analysis are shown in Fig. 4. It may be observed that cellulose acetate membrane (CAM) is represented by the weak intensity peaks which confirms its amorphous nature related to permeate flux (Fig. 4(a)), while SSA indicates major mineral phases in the specimens such as calcite ($CaCO_3$), anhydrite ($CaSO_4$), cristobalite (SiO_2) and quartz percentage (SiO_2) (Fig. 4(b)). Blending membrane CA/SSA at 50/50 wt% was proven the crystallinity of the prepared polymer blends (Fig. 4(c)), since it had three peaks at two diffraction angle of 2θ = 13.06°, 20.61° and 33.12° that crystallinity of membrane will improve the removal percentage of nickel ions from wastewater. The crystallinity of the prepared membranes using other proportions of CA/SSA 90/10, 80/20 and 70/30 wt% is expected to be lower than that reported for 50/50 proportion as the SSA% increased the membrane properties enhanced [25].

3.2. Water uptake of prepared SMF

The influence of SSA content and PEG 600 concentration in the casting solution of CA/SSA at 100/0, 90/10, 80/20 and 50/50 wt% in solvent DMF (80 wt%) on WU of prepared membrane (SMF) is presented in Fig. 5. It reported that the water content of SMF increased with SSA content and PEG 600 concentration increase in the studied range (0–10 wt%). This may be due to the fact that gelation was produced leading to formation of pores acting as demine of water molecules and since PEG 600 acts as hydrophilic source for attracting water molecules inside the membrane blends. This property will increase the service life of the used SMF and therefore decreasing the operating cost of wastewater treatment process.

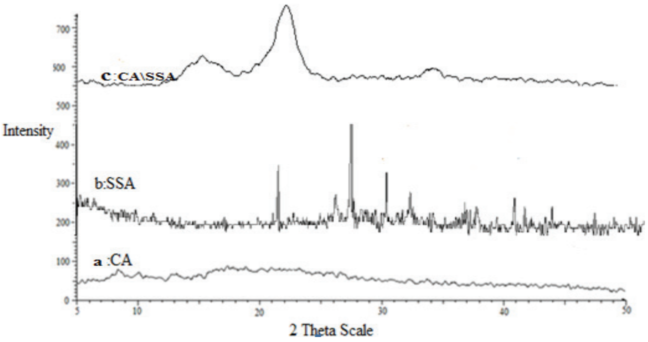

Fig. 4. XRD of CA, SSA and CA/SSA at 50/50 wt%.

3.3. Mechanical strength of prepared SMF

The mechanical strength of the prepared membranes was proven by measuring the tensile stress. The tensile stress values of blend membranes from CA/SSA at 90/10, 80/20 and 50/50 wt% are 38.4, 51.8 and 72.13 N/mm^2, respectively. It was reported that the mechanical strength increased when SSA ratio increased, hence presence of SSA in filter membrane improved its mechanical strength rather than pure CA at 100/0 which had a tensile stress of 32.5 N/mm^2.

3.4. Effect of PEG 600 on nickel rejection from wastewater

Synthetic wastewater was prepared with initial nickel concentration of 25 mg/L. The influence of PEG 600 in casting solution of CA/SSA at 100/0, 90/10, 80/20 and 50/50 wt% and DMF solvent on nickel concentration reduction is represented in Fig. 6. Rejection of the nickel ions follows a similar pattern for increasing concentrations of PEG in the membrane casting solution. However, there is a slight change in the extent of rejection. In general, the PEG added membranes provide higher rejection for nickel ions. For the CA/SSA membrane of 100/0 wt%, the rejection of Ni (II) is 6%–28% for PEG 600 (0–10 wt%), while the Ni rejection increased from 30% at CA/SSA of 100/0 wt% to 79% at CA/SSA of 50/50 wt% for increasing PEG 600. This is because of the increased porosity while adding PEG which has high hydrophilic nature. This indicated that CA/SSA blend membrane is comparatively superior in performance to a pure CAM. Overall, it was found that the membrane with the composition of 50% CA and 50% SSA and with the increase of PEG 600 loading, the repulsive force between polymer segments along with leachability of PEG is enhanced and this favors the formation of macrovoids due to increase of number of large size pores [27]. Moreover, investigation of SMF of 50/50 CA/SSA in nickel rejection from wastewater produced permeate of desirable quantities when compared with similar membranes of other weight fractions under identical conditions.

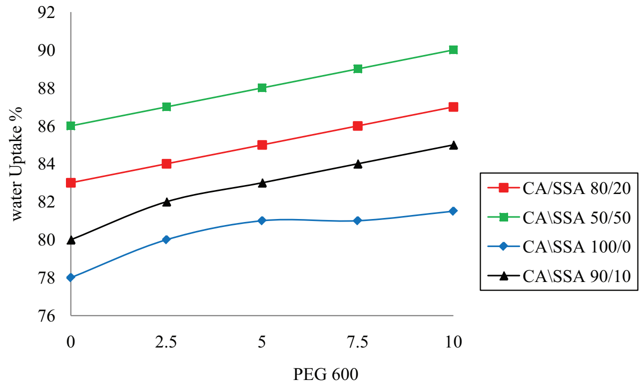

Fig. 5. Effect of PEG 600 on water content using CA/SSA blend membranes.

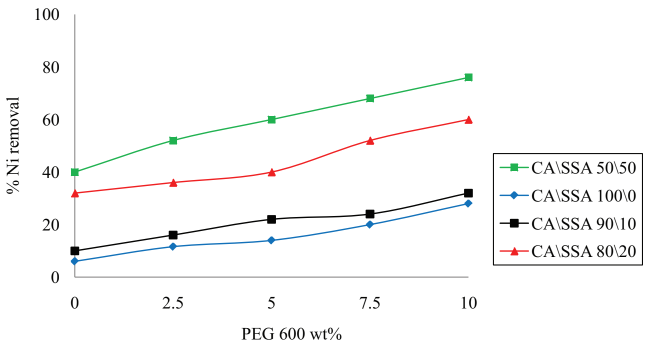

Fig. 6. Effect of PEG 600 wt% on nickel removal with initial concentration of 25 mg/L using CA/SSA blend membrane.

3.5. Effect of initial Ni concentration on the %Ni ions removal using prepared SMF

The effect of initial Ni^{2+} concentration on the removal percentage was studied in range of 5–20 mg/L using CA/SSA membrane of 50/50 wt% at fixed additive concentration of 10% PEG 600 as shown in Fig. 7. It reported that at lower initial Ni^{2+} concentration of 5 mg/L, the removal efficiency is maintained at high level (90%) and then decreased to 70% as the initial Ni^{2+} loading is further increased to 20 mg/L. This is due to the load increase on SMF. It may be observed that CA/SSA blend membrane was able to remove the nickel ions from synthetic wastewater due to the inhomogeneity arising as a result of the higher SSA content which creates voids in the produced membranes. However, the removal efficiency is acceptable if the initial Ni^{2+} concentration does not exceed 5 ppm. Similar results were reported for CA/sulfonated polyetherimide blend membranes by Nagendran et al. [28].

3.6. Effect of hydraulic retention time of the blend membrane CA/SSA on Ni^{2+} removal

The effect of the hydraulic retention time of nickel ion solution (25 mg Ni^{2+} ions/L) on the blend membrane CA/SSA of 50/50 wt% in the presence of PEG 600 (10 wt%) has been studied in range of 5–25 min in order to determine the optimum time at which the removal efficiency of nickel ions is maximized. The results indicated that the Ni^{2+} removal percentage increases by increasing the retention time of wastewater on SMF surface as shown in Fig. 8. The removal efficiency was recorded as 74%, 78%, 80%, 85% and 83% for 5, 10, 15, 20 and 25 min residence time on the blend membrane CA/SSA, respectively. The maximum efficiency was reported as 83% at 20 min since there is no real difference in removal efficiencies between 85% and 83% and this slight difference could be related to the uncertainty of the measurements, rather than changes of the rate of adsorption. Probably the system reached a condition of dynamic equilibrium at 20 min. This mechanism may include solute transfer to membrane which causes diffusion through the pores to the internal adsorption sites. In the initial stages of adsorption process of nickel ions, the concentration gradient between the film and the available pore sites is large and hence the rate of adsorption is increased. The rate of adsorption decreases in the later stages of the adsorption which is probably due to the slow pore diffusion of the solute ion into the bulk of the membrane.

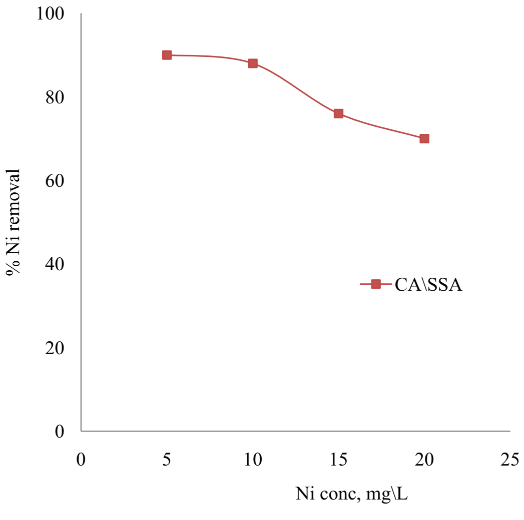

Fig. 7. Effect of nickel initial concentration on its ions removal from wastewater using CA/SSA membrane of 50/50 wt% at 10% PEG 600.

3.7. Performance evaluation of prepared SMF on Abu Rawash wastewater treatment plant effluent

The effluents of the Abu Rawash WWTP were passed through CA/SSA blend membranes in the system depicted in Fig. 2. The membrane developed with 50/50 wt% CA/SSA at 10 wt% PEG showed overall better performance compared with the other membranes. Particularly, analysis of wastewater from Abu Rawash treatment plant before and after membrane pass through UF cell is illustrated in Table 1. The results reported that the CA/SSA prepared membrane of 50/50 wt% acted effectively as a perfect filter in wastewater treatment as the TDS was reduced from 492 to 319 ppm, ammonia concentration is reduced from 13.42 to 7.2 ppm, COD reduced from 167 to 20 ppm, in addition to the removal of colloidal particles along with color. Therefore, investigation of filter membrane made from SSA and blended with CA is an active tool in pollutant removal from wastewater effluents as shown in Fig. 9.

4. Conclusions

From this research, the following points are concluded:

- The water content of prepared SMF was increased from 78% to 86% as SSA concentration increased from 0 to 50 wt%.

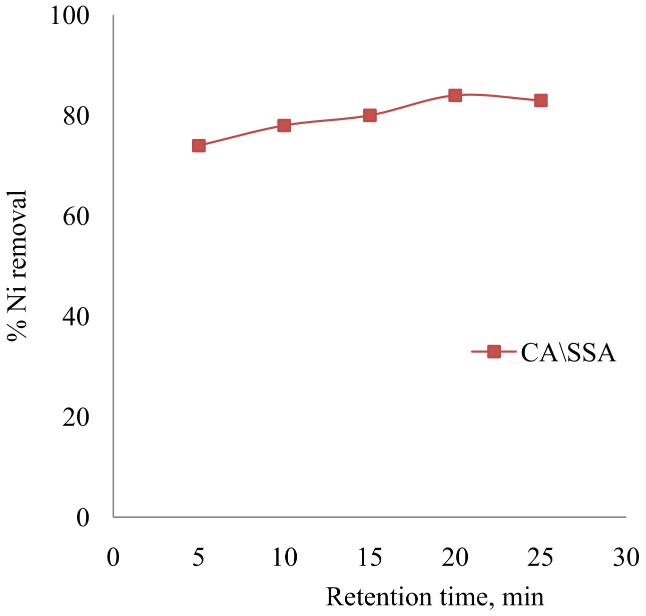

Fig. 8. Effect of hydraulic retention time on removal percentage of Ni^{2+} ions with initial concentration of 25 mg/L.

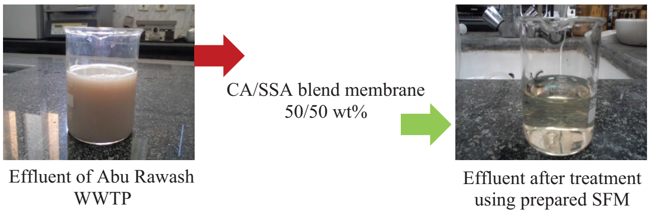

Fig. 9. Wastewater from Abu Rawash plant before and after treatment using CA/SSA blend membrane of 50/50 wt%.

- Increase of PEG 600 additive from 0 to 10 wt% will increase the pore size of the prepared membrane and hence its porosity.
- Increase of SSA from 10 to 50 wt% improved the mechanical strength of blending membranes rather than using pure CA.
- The rejection percentage of Ni^{2+} ions from industrial wastewater was recorded as a maximum (90%) at lower initial Ni^{2+} concentration of 5 mg/L.
- Applying of prepared SMF in treatment of Abu Rawash WWTP effluent proved its ability in pollutants removal as the removal percentage of TSS, COD and BOD_5 are recorded as 88%, 88.5% and 89%, respectively using CA/SSA blend membrane of 50/50 wt%.
- This research proved manufacture of an effective adsorptive membranes with high efficiency, good selectivity, low energy requirement and large permeate flux.

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