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Sulfuric acid recovery from titanium white (pigment) waste liquor using diffusion dialysis with a new series of anion exchange membranes — static runs

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Abstract

In this paper, a new series of anion exchange membranes have been produced from linear engineering plastics poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) by conducting the processes of bromination, chloromethylation and amination. By using this series of membranes, the runs of sulfuric acid recovery from titanium white waste liquor were experimentally conducted. The effects of some important factors such as ion exchange capacity, content of benzyl-halogen and the relative compositions of the liquor were experimentally investigated on the membrane dialysis process. It was found that the acid recovery rate was improved by benzyl substitution, while the selectivity improved by aryl substitution. By properly balancing them and adjusting the compositions of feed, both higher selectivity and acid recovery can be achieved. A comparison is made with a commercial diffusion dialysis membrane in China. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Ion exchange membrane; Diffusion dialysis; Titanium white waste liquor; Poly(2,6-dimethyl-1,4-phenylene oxide)

1. Introduction

Pickling of titanium fine mines with sulfuric acid is one of the key steps in manufacturing of titanium white. This pickling process is usually accomplished by precipitating of per-titanium acid and leaving large quantities of spent liquor, for example, 200–400 t of such waste liquors will be produced when 1 t of titanium white is manufactured. Generally, this kind of waste is mainly composed of hydrated TiO_2 suspensions (5–10%), free H_2SO_4 (10–20%), FeSO_4 (15–20%) and other metal ions such as vitriol, manganese, magnesium, scandium, etc. [1].

The accumulation of metal ions (especial ferrous ions) in the solution will result in a decreased efficiency of the picking agent, and thus the eventual disposal is necessitated. This disposal of spent liquor has posed a problem in the titanium white production and also the whole metal finishing industry for a very long time [2] and has prompted industries to give serious consideration to recover acid from products that are now going to wastes. If the spent liquor is directly circulated, the impurities carried therein will contaminate the pigment. Over the history of this problem, different methods of dealing with the spent pickling solutions have been applied including direct disposal and neutralization. Of course, both of them are passive and not advisable. A more practical method has been described for treatment of such solutions by concentrating the free acid, removing the ion sulfate by

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salting out, and then decomposing the sulfate for the recovery of the acid value it carries [3]. However, this method is limited by the high investment required and the cost of fuel for water evaporation, and thus is not economically feasible.

Diffusion dialysis provides an attractive acid recovery method from the view of energy saving. It relies on the great difference in the diffusivity between acids and salts and the only external power is required to circulate the solution. The separation of acid from salts is achieved by using an anion exchange membrane which has a tendency to allow the selective transport of anion across the membrane, while ideally remaining impermeable to cations other than protons because of their small volume and less positive charge. Up to now, this technology has been exploited to recover sulfuric acid and other acids from waste solutions being generated in steel, metal-refining, ion exchange resin regeneration waste and electroplating industry [4–7].

The anion exchange membrane is the linchpin for the above purpose. The membrane for diffusion dialysis use differs a lot from ordinary membrane for electrodialysis. It is demanded that it should provide high proton permeability but strong rejection of salts, relatively high water content but poor water permeability, and thus, it must retain a appropriate fixed group concentration so as to function efficiently in the diffusion dialysis recovery of acids. Nowadays, this kind of membrane is found in the market, such as Selemion DSV from Asahi Glass and Neosepta, AFN from Tokuyama Soda Co. Ltd., which are strong-base anion exchangers with polyvinyl chloride support. Recently, Stachera et al. [4] reported a dialytic membrane prepared by photoinitiated polymerization of 4-vinylpyridine with varying amounts of DVB in the pores of PP substrate. Also, there is a kind of commercial membrane S₂₀₃ which is produced from linear plastic polysulfone followed by chloromethylation and quaternary amination. However, this membrane is out of production due to environmental contamination from chloromethyl ether. To overcome the disadvantages of the original membrane and satisfy with industrial demands in China, a new series of membranes were developed in our lab recently. This new series of membranes have avoided the use of chloromethyl ether, which has been considered as a substance with serious contamination. Due to introduction of chemical cross-linking and the special choice of membrane

materials which can be both aryl and benzyl substituted, they behave extraordinarily excellent properties [8,9], especially in acid recovery. Therefore, the main purpose of this paper is to briefly introduce the nature of this new series of anion membranes and their use in the extracting sulfuric acid from titanium waste liquor.

2. Experimental descriptions

2.1. Membrane preparation

Poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) of intrinsic viscosity equal to 0.57 dl/g in chloroform at 25°C (manufactured in Sichuan special plastic plant, China) was dissolved in chloroform to form a 8% solution and this solution was subjected to the aryl bromination by some addition of bromine for 2 h under the ambient temperature [8,9]. After precipitation with methanol, the aryl-substituted polymer was obtained and benzyl-brominated in also a 8% solution of chlorobenzene for additional 2 h at boiling state. After proper addition of the necessary additives, such as inorganic salts, dimethyl formamide, etc. the produced solution was cast onto a polyvinyl chloride substrate to form a base membrane of 0.1–0.2 mm dried thickness. This base membrane was functionalized and cross-linked simultaneously by amination reaction at about $35 \pm 1^\circ\text{C}$ for at least 10 h with a properly proportional aqueous solution of trimethyl-amine, diethyl-amine, and then washed and equilibrated with 1 M NaCl solution to be transformed into chloride form.

2.2. Membrane measurements

The base membrane was conducted to ^1H NMR (unity plus 400) measurements for the precise determination of bromine contents in aryl and benzyl position, respectively. The final membrane was conducted to the conventional measurements, such as, water content (W_R), membrane area resistance, ion exchange capacity (IEC), fixed group concentration (C_R) as well as solubility in conventional solvents.

2.2.1. Water content

The water content (W_R) of the final membrane was determined after equilibrating a sample of membrane in chloride ion form with deionized water at room

temperature. The membrane samples were removed from the water and weighed immediately after blotting the free surface water. Then they were dried for over 4 h at $100 \pm 5^\circ\text{C}$. The water content was deduced from the difference in weight between the wet (W_1) and the dry (W_2) membrane in unit g $\text{H}_2\text{O}/\text{g}$ dry membrane based on the following formula:

$$W_R = \frac{W_1 - W_2}{W_2} \quad (1)$$

2.2.2. Ion exchange capacity (IEC) and fixed group concentration (C_R)

Membrane samples in the chloride form and in equilibrium with deionized water were converted to the sulfate form by leaching with a 1N Na_2SO_4 solution. The chloride ions released from the membranes were determined by a titration of 0.1 M AgNO_3 solution with K_2CrO_4 as an indicator (Mohr method), and are given as meq/g dry membrane. The fixed group concentration (C_R) was determined from the ratio of IEC to W_R and was expressed in M (mol/l).

2.2.3. Membrane area resistance (R_m)

The membrane area resistance was determined by a fast ac technique with high frequencies ($>10^4$ Hz) in a 0.1 M NaCl solution and expressed in unit $\Omega \text{ cm}^2$. The detail for this method can be found in literature [10].

2.3. Diffusion tests

Diffusion dialysis runs were carried out using a two-compartment cell of equal volume separated by the membrane with an effective membrane area of 5.07 cm^2 . Prior to the experiments, the membranes were conditioned for 1 day in the feed to be dialyzed for all runs. Initially, left chamber contained a solution of feed conforming to the conditions for each run and the other deionized water. The feed was filtrated and diluted to a composition with 0.25 M of free sulfuric acid (denoted as C_H), 0.17 M of FeSO_4 (denoted as C_{Fe}) and 0.15 M of titanium (in terms of TiO_2 , denoted as C_{Ti}), in most cases if it is not specified. The cell temperature was maintained at $25 \pm 0.5^\circ\text{C}$. Both chambers were stirred at identical rates to minimize concentration polarization effects. Diffusion was allowed to occur for a measured time and then the solutions were removed from each side of the cell and analyzed

for composition. Acid concentration was determined by titration with a standard Na_2CO_3 solution with methyl orange as an indicator. Concentration of FeSO_4 was determined by titration with a KMnO_4 solution and Ti^{4+} was analyzed with by a UV-spectrometer at 420 nm. Water transport was calculated from the mass balance of the acid.

In order to ascertain the preferential diffusion for each run, a knowledge of separation factor is essential. The separation factor (S) with respect to one species over another is given as the ratio of dialysis coefficients (U) of the two species present in the solution and can be calculated by the following formula [4,7]

$$U = \frac{M}{At\Delta C} \quad (2)$$

where M is the amount of component transported in moles, A the effective area in square meters, t the time in hours, and ΔC the average concentration between the two chambers in moles per cubic meters and defined as below [4,7]

$$\Delta C = \frac{(C_f^0 - C_f^t - C_d^t)}{\ln[(C_f^0 - C_d^t)/C_f^t]} \quad (3)$$

here C_f^0 and C_f^t are the feed concentrations at time 0 and t , respectively, and C_d^t the dialysate concentration at time (t). It should be noted that $C_f^0 - C_f^t - C_d^t \neq 0$ because of the volume changes that occur in the cell chambers caused by water transport through the membrane during the experiment [4].

3. Results and discussion

3.1. On the membrane

As mentioned above, the membranes were prepared from linear polymer following the procedures of bromination in both aryl and benzyl positions and functionalising and cross-linking with a proper mixed amine solution. Thus, the properties of the products differ significantly from those of the raw polymer. The appreciable alteration is the solubility. Due to the simultaneous introduction of the functional group and cross-linkage structure in the amination processes, the resultant membrane has extraordinarily well resistant to most conventional solvents, even at a relatively

Table 1
Properties of example membrane series developed in this paper^a

Membrane no.	ABC ^b (%)	BBC ^b (%)	IEC (meq/g dry membrane)	W_R (g H ₂ O/g dry membrane)	C_R (M)	R_m (Ω cm ²)	Burst strength (MPa)
1	0.16	0.116	0.89	0.418	2.13	4.82	
2	0.16	0.173	1.27	0.50	2.52	3.81	
3	0.16	0.285	1.94	0.593	3.26	3.72	>0.8
4	0.16	0.310	2.08	0.74	2.80	1.93	
5	0.16	0.328	2.18	0.77	2.82	1.93	
6	0.10	0.285	1.935	0.673	2.87	1.82	
7	0.28	0.285	1.952	0.478	4.08	3.81	
8	0.38	0.285	1.943	0.442	4.39	4.32	>0.8
9	0.42	0.285	1.933	0.387	4.99	4.93	
10	0.54	0.285	1.920	0.302	6.36	5.25	

^a All the measurements were conducted at temperature of 25°C.

^b ABC: aryl bromine content; BBC: benzyl bromine content. Both are assumed mono-substituted.

high temperature (50°C) compared with the raw polymer PPO and an analogous commercial membrane S₂₀₃ in China [8].

In addition to the good proof to chemical agents, the membranes prepared by the mentioned method also have good electrical, mechanical and diffusion properties as listed in Table 1. This table contains two membrane series: one is for benzyl substitution and another is for aryl substitution. Obviously, for the first membrane series, IEC are enhanced by the benzyl substitution. Nevertheless, fixed group concentration, which are expressed as the ratio of IEC to water content, decreased at first and remain approximately unchanged due to the increase in water content. While for the second membrane series, IEC remain approximately unchanged by the aryl substitution, but the fixed concentration increases with aryl substitutions due to a decrease in the water content entrapped in the membrane. Therefore, by adjusting the portions of benzyl and aryl substitutions, a desire membrane with both IEC and water content can be reasonably acquired for acid and salt separation as described in the following sections.

3.2. Effect of ion exchange capacity or benzyl substitution on the dialysis results

As shown in Table 1, IEC is directly related and approximately proportional to benzyl bromine content. First membrane series were, thus used in the dialysis tests to investigate this effect. It can be seen in Fig. 1, the dialysis coefficients of both the salts and

protons increase with IEC and then decrease until an approximate constant is attained. Moreover, the speed for proton transfer across the membrane is much fast than that of salts. Theoretical interpretation of this brings our eyes into the observations of the relationship between IEC and fixed ion group concentration as depicted in Fig. 2. Obviously, fixed group concentration increases with IEC at low BBC values due to the much low water content. At high benzyl substitutions, it reaches limiting value because both the membrane IEC and water content increase considerably at this case (cf. Table 1) when aryl substitutions keep unchanged. From academic view, permeability of ion species does not directly connect with IEC but with fixed concentration and water content. For cationic species through anion membranes, the permeability

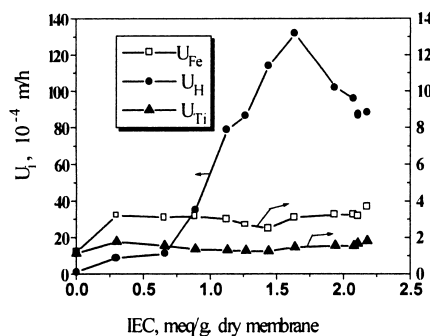


Fig. 1. Acid and salts dialysis coefficients vs. IEC or benzyl substitution with ABC = 0.16; C_H = 0.25 M; C_{Ti} = 0.15 M; C_{Fe} = 0.17 M.

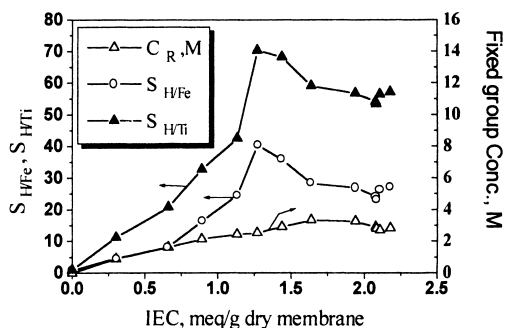


Fig. 2. Membrane IEC vs. fixed group concentration and selectivity of acid over iron and titanium with ABC = 0.16; $C_H = 0.25$ M; $C_{Ti} = 0.15$ M; $C_{Fe} = 0.17$ M.

decreases with an increase in fixed group concentration due to the electrostatic repulsion, so permeability of both iron and titanium ions are decreased with fixed group concentration. An exception to proton is that it has a strong tendency to pass the membrane to comply with the electro-neutrality because of its less positive charge and smaller volume compared with other cations (Fe^{2+} and Ti^{4+}) as well as the enhanced permeability of anions such as sulfate ions in the feed. Of course, water content itself has also significant effects on the transport of the species and so, at low IEC, both the acid and salts behave poorly permeable due to the very low water content in the membrane (see next section for further discussion). The net result for above process is that the selectivity of acid over salt ions increases with increasing fixed group concentration as the permeability of the two metal ions is markedly reduced. This is attested by the approximately same tendency for selectivity of acid over salts with IEC as that for fixed group concentration (cf. Fig. 2). It should be pointed out that some discrepancy is observed between acid dialysis coefficient and the selectivity over salts at the maximum point as shown in Figs. 1 and 2. Obviously, the maximum dialysis coefficient for acid occurs at IEC about 1.6 with a approximate value of 130 m/h, while the maximum selectivity occurs at IEC about 1.25 and with the approximate value 40 of acid over iron and 70 over titanium, respectively. This inconsistency is probably due to the effects of both water content and fixed group concentration. Therefore, for practical separations, one must make a balance between the acid dialysis rate and selectivity for the optimum.

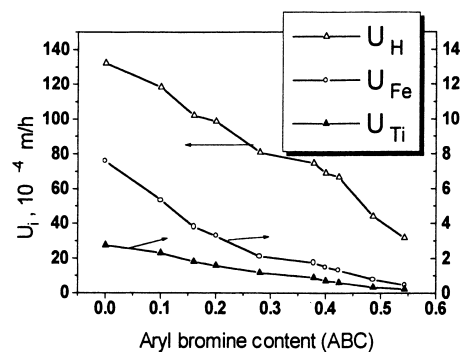


Fig. 3. Acid dialysis coefficient vs. aryl bromine content with BBC = 0.285; $C_H = 0.25$ M; $C_{Ti} = 0.15$ M; $C_{Fe} = 0.17$ M.

3.3. Effect of aryl bromine content on the dialysis selectivity

Second membrane series were used here to investigate the effects of aryl substitutions. As illustrated in Fig. 3, the dialysis coefficients of all the species decrease with increasing aryl bromine content (ABC) though the fixed group concentration is increased due to the reduction of water content in the membrane. The selectivities of acid over iron and titanium were shown in Fig. 4. A pronounced increase in selectivity was observed with increasing ABC. These results can be understood in terms of both the varying water uptake and fixed group concentration of these membranes. Following the gists in the literatures [4,11,12], the co-ions leakage through anion exchange membranes has been considered from the viewpoint of a three-phase membrane model where the membrane

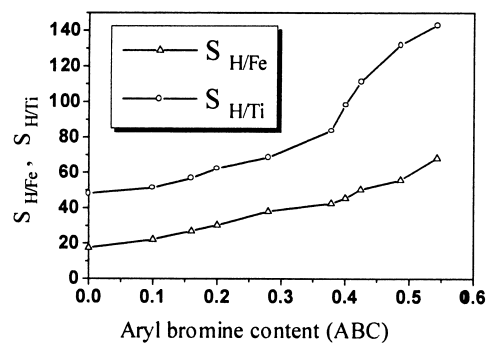


Fig. 4. Selectivity vs. Aryl bromine content with BBC = 0.285; $C_H = 0.25$ M; $C_{Ti} = 0.15$ M; $C_{Fe} = 0.17$ M.

consists of hydrophobic polymer, an active exchange zone and an interstitial zone. A counter ion transport occurs mainly through the active region where it moves by a hopping mechanism and has a greater mobility than that in the interstitial zone due to the “dragging” mechanism (restricted diffusion) [12]. The portion of water in the membrane that is found in the solvation shells of the fixed ions and their counter-ions is not free to move within the membrane and will exert drag on ions moving through the membrane. Therefore, hydrated ions will require a certain volume of water within the membrane to permit migration through the membrane. Then it is easily understood, that both fixed ion concentration and water content contained in the membrane will enhance the permeability of counter-ions, sulfate ion in our case.

Due to the minimal repulsive force caused by ion exchange sites in the interstitial zone, a co-ion transport occurs mainly through this region. And thus, the volume of the water content in the membrane should play a decisive role in co-ions permeability. In our case, permeability of iron and titanium ions decreases with water content to more greater extent compared that of proton whose diffusion is also restricted by water content but at the same time will be enhanced by transport of sulfated ions due to increase in fixed group concentration. So the net result is an increase in selectivity with increasing ABC.

3.4. Effects of acid concentration in feed solutions

Fig. 5 shows the effects of varying acid concentrations on dialysis coefficients. As can be seen that an

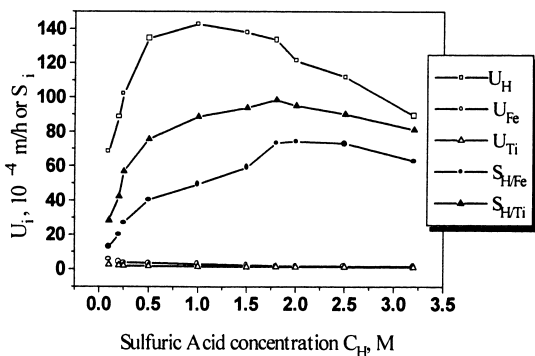


Fig. 5. Dialysis coefficient and selectivity vs. acid concentration ABC = 0.16; BBC = 0.285; $C_{Fe} = 0.17$ M; $C_{Ti} = 0.15$ M.

increase in acid concentration results in a decrease in the U_{Fe} and U_{Ti} , but for U_H , the situation is slightly different, it will increase at low acid concentration but decrease at a relatively high concentration. The main effect caused by the increase in acid concentration is an increase in ionic strength, and thus the mobilities of the transported ions (both cationic and anionic) in the membrane are decreased. However, an exception to proton is that its transport will be also enhanced by its concentration increase at the same time. So it behaves differently as other two co-ions. At low acid concentration, the latter effect is dominant and an increase in permeability is observed; while at high concentration, the former (ionic strength) effect is dominant and the permeability is decreased. It is shown that at an acid concentration of about 1.8 M, one can obtain both the desired selectivity about 73.3 for acid over iron and 98.4 for acid over titanium and relatively high permeability for acid about 133.4.

3.5. Effects of Salts ions concentration in feed solutions

Effect of $FeSO_4$ on the dialysis permeability was shown in Fig. 6. Obviously, U_{Ti} is decreased due to the increase of ionic strength in solution as described above. U_{Fe} keeps approximately unchanged because it is influenced by two effects: one is that the increase on ionic strength brings about its decrease in mobility in membrane and the other is that the increase in concentration gradient enhance its permeability in the membrane. Two factors are interacted and give the net nearly unchanged dialysis effect for ferrous ions.

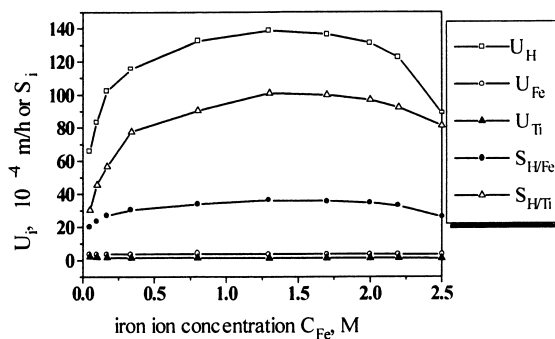


Fig. 6. Dialysis coefficient and selectivity vs. $FeSO_4$ concentration ABC = 0.16; BBC = 0.285; $C_H = 0.25$ M; $C_{Ti} = 0.15$ M.

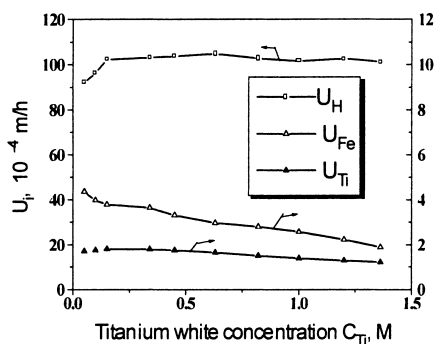


Fig. 7. Dialysis coefficients vs. titanium concentration ABC = 0.16; BBC = 0.285; $C_H = 0.25$ M; $C_{Fe} = 0.17$ M.

Compared the salt ions, permeability of protons is enhanced by the increase in concentration gradient at low $FeSO_4$ concentrations but constrained by the increase in ionic strength at high $FeSO_4$ concentrations. At the concentration of $FeSO_4$ about 1.3 M, U_H attains its maximum value about 138.7. It is interesting to find that this value is a little greater than the about maximum value, implying that some addition of salt ions with the same anion as that of the acid can promote the diffusion of protons and will be preferred in some practical processes. However, the gain in acid permeability is a sacrifice of selectivity of acid over iron, which has a value about 36.2 at this point. As the permeability of titanium is restrained by the increasing ferrous ion concentration, the selectivity of acid over titanium also has a relatively larger value about 101 than the above.

The effect of titanium ions (based on TiO_2) on the dialysis follows nearly the same trends as those of $FeSO_4$ as depicted in Fig. 7. Nevertheless, titanium ion has a larger effect on ionic strength in the solution than ferrous ion because of its high positive valence. In addition, a titanium salt has small intendance to be dissociated to increase the anionic concentration, so that the curve for U_H is more flat compared with the corresponding curve in Fig. 6. It is observed that maximum value about 104.8 occurring at $C_{Ti} = 0.63$ for acid permeability is smaller than the above values due to weak ability to be dissociated and high valence of titanium.

3.6. Comparison with the commercial membranes

Comparison was made with a commercial membrane for acid recovery in China, S_{203} , a chloromethyl-

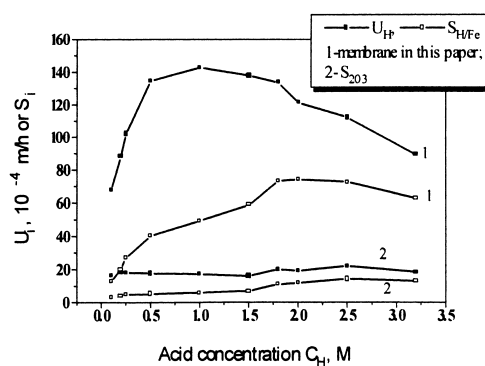


Fig. 8. Comparison of dialysis coefficient and selectivity of acid by using S_{203} and membrane in this paper with ABC = 0.16; BBC = 0.285; $C_{Fe} = 0.17$ M; $C_{Ti} = 0.15$ M.

ized polysulfone membrane followed by quaternary amination as described earlier. When the tests were conducted under the same conditions and compared with a membrane with 0.285 BBC and 0.16 ABC presented in this work, our membrane has both relatively larger acid permeability and acid-salt selectivity than S_{203} membrane in the whole sulfuric acid concentration range as shown in Fig. 8. A comparison with foreign membrane with the same conditions has not been done due to shortage of the membrane. However, from the view point of acid recovery, Stachera et al. reported that their membrane, which was prepared by photoinitiated polymerization of 4-vinylpyridine with varying amounts of DVB in the pores of PP substrate, has a dialysis coefficient about 55 (5.5 in their unit) for HCl [4]. The same tests conducted by them showed that Selemon DSV manufactured by Asahi Glass had a nearly equal acid diffusivity about 56 [4]. Of course, our membrane can easily attain this result.

4. Conclusions

This work reveals that PPO can be experiencing both aryl and benzyl substitution, producing a new series anion membranes for diffusional dialysis. Both the IEC and water content of the membrane are enhanced by benzyl substitution; while the water content of membranes decreased with aryl substitution and IEC remains approximately unchanged by the aryl substitution. Therefore, by adjusting the portions

of benzyl and aryl substitutions, one can reasonably obtain a desirable membrane with both IEC and water content for acid recovery from its salt solution.

By using these membranes, the series experimental runs for sulfuric acid — recovery from titanium white waste liquor were conducted. The effects of some important factors such as ion exchange capacity, content of benzyl-halogen and the relative compositions of the liquor were experimentally investigated on the membrane dialysis process. The acid recovery rate will be improved by benzyl substitution, while the selectivity by aryl substitution. By properly balancing them and adjusting the compositions of feed, both higher selectivity and higher acid recovery can be achieved.

Comparison with a commercial diffusion dialysis membrane in China, S₂₀₃ indicates that the membrane presented in this work perform well for acid recovery by diffusion dialysis in both acid dialysis rate and acid selectivity over its co-existing salts.

Acknowledgements

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