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Performance study of hydroxyapatite cow bone based polysulfone mixed matrix membrane: Effect of hydroxyapatite morphology

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Abstract. In this work, polysulfone (PSf)/ hydroxyapatite (HAp) ultrafiltration mixed matrix membranes (MMMs) were prepared for wastewater treatment by using phase inversion method. The effect of hydroxyapatite (HAp) dispersion on the PSf/PVP membrane surface were investigated. Characterizations were done to observe the distribution of HAp on the membrane surface by using X-ray diffractometer (XRD) and scanning electron microscope (SEM). The XRD analysis showed the crystallinity of the HAp derived from cow bone. The dispersion of HAp in polymer matrix is a promising materials that can change the structure of the PSf membrane.

1. Introduction

Increasing population trends in major cities have put tremendous strain on natural water supplies, while insufficient method to provide water have resulted in severe shortages [1]. Membrane technology has been recognised as an effective separation process for water treatment due to the durability and efficiency of contaminant rejection, as well as the flexibility provided by a range of membrane materials such as ceramics, silica, alumina, titanium and polymer [2]. Due to its simplicity in operation, no addition of chemical additives (or less), cost-effective, no phase shift, high efficiency, simple scaling up and high removal power, membrane technology contributes up to 53% of the total world processes for clean water supply and is an efficient approach for water treatment [3].



Polymeric membrane is mainly used due to their greater durability, strong film forming properties, mechanical strength, high selectivity, low cost for fabrication and sufficient pore size for pressure-driven processes [4][5]. Due to its properties, such as chemical, mechanical and thermal stress resistance, and excellent film forming capabilities, polysulfone (PSf) is a widely used polymer for the preparation of membranes [6]. However, PSf has hydrophobic nature and susceptible towards fouling problems which can affect the membrane's performance such as rejection and flux permeation [7].

As a result, various methods of modification have been approached to solve the problems of the polymer membrane over the last decade. Mixed matrix membranes (MMMs) have gained significant importance in meeting new demands and requirements due to their ability to combine the characteristics of above-mentioned membrane materials, providing better approaches in term of efficiency, fouling, permeate consistency and longevity. MMMs are classified as membrane with inorganic fillers dispersed in a continuous phase (solid, liquid, or both) which is made up of polymeric materials [8]. Incorporation of inorganic additive into polymer matrix could provide the desired properties in the membrane such as high selectivity and high mechanical, chemical and thermal strength [9][10].

Hydroxyapatite, HAp ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) is a low cost material, high adsorption capacity, low water solubility and high stability at low temperature and oxidation condition. HAp has a large number of hydroxyl group (-OH) on the surface which can increase the hydrophilicity of the polymer membrane [11]. The increasing hydrophilicity of the membrane could reduce fouling effect and also increasing the permeation performance of membrane.

In this work, a new adsorptive membrane by dispersing HAp into PSf membrane was prepared via phase inversion technique. The effect of addition HAp into the structure of polysulfone membrane were investigated.

2. Materials and methods

2.1. Preparation of hydroxyapatite

Hydroxyapatite were prepared by using cow bones. The bones were crashed into smaller pieces and boiled into 100°C hot water. The bones were then dried in oven for 24 hours. Then, the bones were crushed and calcined in furnace at 800°C for 3 hours. The bones were then ground into powder using ball mill and were sieved to get fine powder with average size 20µm.

2.2. Membrane preparation

Polysulfone (PSf) mixed matrix membrane were prepared using immersion precipitation by phase inversion method. PVP were first mixed with NMP and stir with magnetic stirrer for 15 minutes. Then, HAp powder was added and continuously stirred for 6 hours to ensure uniform powder dispersion in the solution. After that, PSf was added and stirred at heating temperature 60°C until the solution fully homogeneous. The casting solution was then sonicated for 1 hour to release the bubble. Next, the solution was cast using a flat sheet membrane casting method and immersed in distilled water for coagulation bath. The flat sheet were then dried for 24 hours.

2.3. Membrane analysis

The XRD analysis was done by using XRD Bruker D8 advance with 40kV scaled copper tube as source and a graphite crystal as monochromator. The diffraction angles of 2θ used were in range between 10-60°. The membrane surface were observed using JEOL JSM-6380LA scanning electron microscope (SEM).

3. Result and Discussion

3.1. X-ray diffraction (XRD)

Figure 1 demonstrated the XRD result for HAp derived from cow bone through calcination at 800°C. The XRD pattern were observed from 2θ range of 10° to 60° at 40kV and 20mA. From the graph, it showed the crystallinity of the HAp via the sharp peaks that are present in the XRD spectrum. The graph was almost in line with the result demonstrated by [12] for natural HAp. The highest peak was obtained at the 2θ value of 31.7823° .

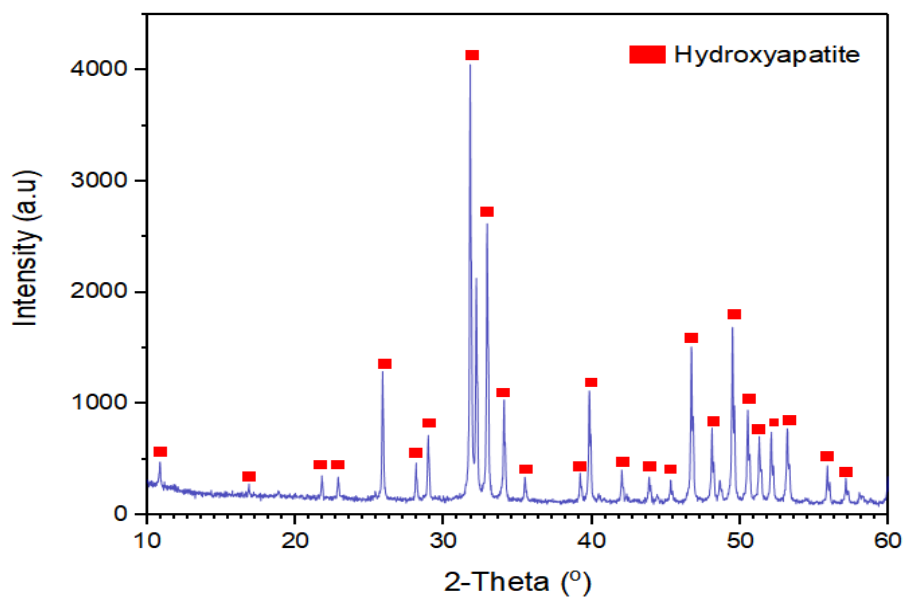


Figure 1: Diffraction patterns for hydroxyapatite derived from cow bone

3.2. Membrane Morphology

The top layer surface view of mixed matrix membrane is depicted in Figure 2 with various loading of HAp into polymer matrix. The SEM image indicated that the dispersion of HAp has successfully occur on the membrane. The formation of pore and surface porosity in the fabricated membrane is resulted from the presence of HAp as fillers [13]. The fabricated membrane have more porous surface that neat membrane as the HAp concentration increase. This situation might due to the hydrophilicity nature of the fillers. The increasing pores can lead to the increasing porosity as well as membrane performance [14].

The SEM image also demonstrated the cross section of the membrane. It can be seen the membrane having an asymmetric structure that consists a dense surface layer at the top of the membrane and a porous sub-layer. The presence of HAp in the polymer matrix of the PSf membrane resulting in the formation of finger-like porous structure [15]. The finger-like structure in fabricated membrane is more bigger than the neat membrane.

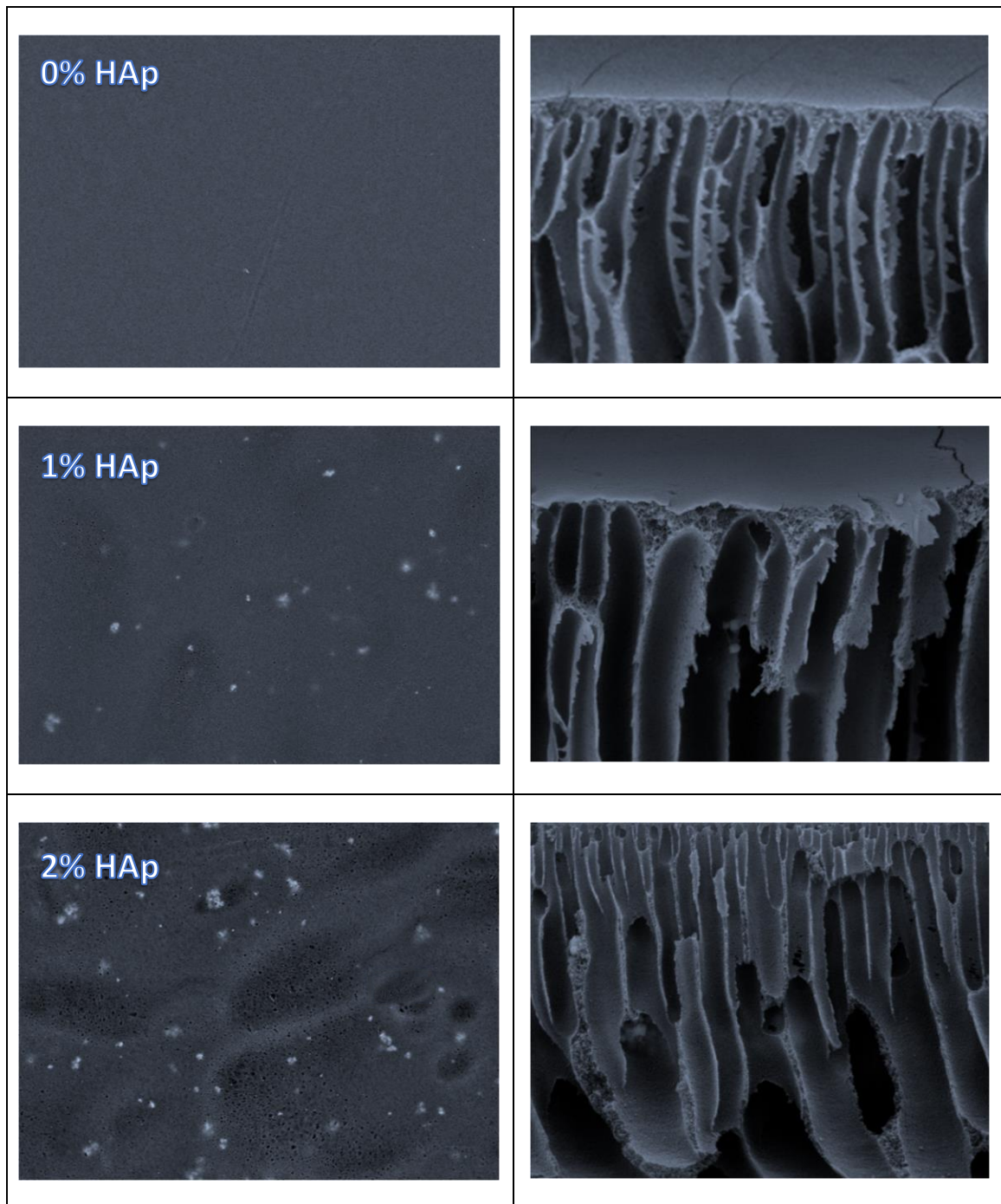


Figure 2: SEM images of top surface (left) and cross section (right) of membrane

4. Conclusion

Both hydroxyapatite and fabrication of PSf/HAp were successfully synthesized and prepared using thermal treatment and phase inversion technique. In this work, the effect on HAp on membrane morphology was investigated. The XRD result showed that the HAp was synthesized perfectly as the sharp peak exists in the XRD graph. The SEM image demonstrated that the dispersion of HAp in the polymer matrix can be seen in the membrane surface. Furthermore, the enlarged, increasing number of pores and finger-like structure of the membrane morphology indicated that the porosity of the fabricated membrane is enhanced from neat membrane. The enhanced porosity will intensify the membrane performance and pure water flux.

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