

Performance, Characteristics And mechanical Properties Of Polyvinylidene Fluoride (Pvdf) Membrane With Addition Of Tin Dioxide (SnO₂) For Water Treatment

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Abstract:

The growth and development of the global economy makes a significant contribution resulting in increasingly uncontrolled disposal of wastewater which results in increasing environmental problems associated with the provision of clean water. Water plays a major role in most household and industrial processes. This research concerns water filtration using membrane technology with Polyvinylidene Fluoride (PVDF) polymer mixed with Tin(IV) Dioxide (SnO₂). It aims to analyze the microstructure formed through Scanning Electron Microscopy (SEM) testing and determine the mechanical properties and performance in water treatment of a mixed membrane of Polyvinylidene Fluoride (PVDF) and Tin (IV) Dioxide (SnO₂) and this research is a new method in water treatment. The research method used was initial membrane specimen formation, membrane preparation, flat sheet method, testing methods (tensile testing, Scanning Electron Microscopy (SEM) observations, and Clean Water Permeability (CWP) testing). The results of this study, namely the formation of mixed membranes showed the results which is suitable as an alternative to water filtering membranes, PVDF and SnO₂ mixed membranes were subjected to tensile testing using ASTM D638 standards, surface structures of mixed membranes using SEM showed significant differences in each concentration, and water treatment performance was carried out with Clean Water Permeability (CWP) showed an increase with increasing PVDF and SnO₂ concentration.

Keywords: Membrane, Micro Structure, Permeability, PVDF, SnO₂, Tensile Strength

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I. Introduction

Global economic growth and development make a significant contribution to human well-being, One of the influencing factors is industrialization and urbanization[1]. The development of both oil and gas and non-oil and gas industries has caused water, air and soil pollution caused by industrial waste products. Disposal of wastewater is getting out of control which results in a series of environmental problems related to the provision of clean water. Water plays a major role in most household and industrial processes[2].

Economic and industrial growth is superior, wastewater discharges from industrial and municipal sources are also growing at the same pace. The composition of wastewater produced by industry is very complex, ranging from water, pathogenic bacteria, non-pathogenic to gases such as hydrogen sulfide, carbon dioxide and methane[3]. Heavy metal ions in generated wastewater cannot be degraded microbially and therefore migrate through soil, water and air, contaminating drinking water and the food chain[4] and also can damage human health and the environment[5].

Wastewater treatment is the most important step in reducing water pollutants and improving the quality of the water environment because it offers clean water with guaranteed quantity and quality in many arid areas, coastal areas or remote locations[6]. Wastewater treatment is the result of public interest in the environmental impact caused by the discharge of untreated or partially treated wastewater and the issuance of federal and state laws on water pollution control[7].

The membrane definitively means a thin layer between two phases and functions as a selective separator. Separation of membranes works based on differences in diffusion coefficients, pressure differences, or concentration differences[8]. Membrane have been considered as a technology that can overcome global water shortages[9]. Membranes have many advantages such as low energy consumption, low use of chemicals, stable operation, easy scaling to low maintenance costs[10].

Modifications were made to the polymer membrane to increase the hydrophilicity of the membrane, such as mixing polymer with a third compound[11], chemical grafting[12], and surface modification of the

membrane[13]. *Polyvinylidene Fluoride* (PVDF) polymers are widely used in industry in various applications due to their excellent chemical stability, good thermal stability, high mechanical strength and flexibility, radiation resistance and low cost[14].

Tin (IV) Dioxide (SnO_2) is a type of metal oxide that has potential as a photocatalyst[15]. SnO_2 has been widely used in various applications because of its high chemical stability and low toxicity, SnO_2 can be used in the decomposition of compounds and toxins that are harmful to the environment in wastewater. In addition, SnO_2 has no adverse health effects and is difficult for the human body to absorb when inhaled or injected.

Membrane modification by mixing *Tin (IV) Dioxide* (SnO_2) on *Polyvinylidene Fluoride* (PVDF) is expected to improve the performance of mixed membranes, starting from the characteristics, mechanical properties to performance against water treatment. Based on the things mentioned above, this study raised the title "Pembentukan Membran *Polyvinylidene Fluoride* (PVDF) Dengan Pencampuran *Tin (IV) Dioxide* (SnO_2) : Karakteristik, Sifat Mekanis dan Kinerja Pengolahan Air".

II. Material And Methods

This research regarding the manufacture of PVDF membranes with the addition of *Tin Dioxide* (SnO_2) was carried out based on the flow chart presented in Figure 1.

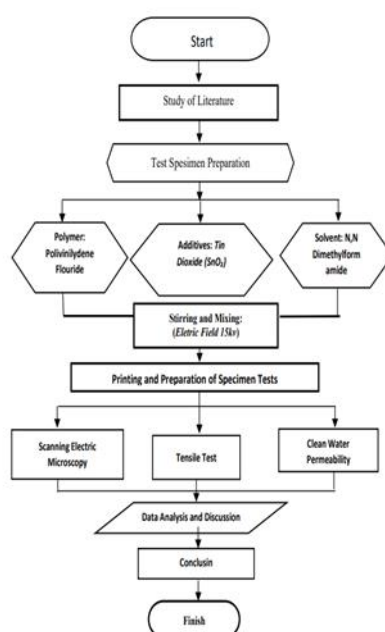


Figure 1 Flow diagram.

This study uses the preparation of membrane specimens by preparing the necessary materials and equipment. Like *Polyvinylidene Difluoride* (PVDF), *Tin Dioxide* (SnO_2) and *N,N-Dimethylformamide* (DMF) obtained from PT. Dira Sonita, Palembang. Equipment for making membranes includes: the mixing process assisted by a magnetic stirrer is carried out at the Faculty of Engineering, Sriwijaya University. Identification of natural properties assisted by *Scanning Electron Microscopy* (SEM) and identification of strength assisted by a Tensile test carried out at the Manufacturing Polytechnic, Bangka Belitung, As well as identification of permeate values assisted by *Clean Water Permeability* (CWP).

Then, carry out membrane preparation, namely making membranes on 3 specimens in the fraction ratio by weight (wt%) of the mixture dan additive substance in each sample, among others, 15wt%, 17.5wt% and 20wt%.

Table 1. Membrane Composition

Membrane	PVDF (gram)	DMF (gram)	SnO ₂ (gram)
PVDF SnO ₂ 15wt%	7.5	42.425	0.075
PVDF SnO ₂ 17.5wt%	8.75	41.1625	0.0875
PVDF SnO ₂ 20wt%	10	39.9	0.1

Tensile test:

After preparing the membrane, then printing the membrane. This study used a glass plate and duct tape as a membrane template. Membrane PVDF were prepared by phase inversion using the immersion method in a coagulation bath filled with water, the results of membrane printing with this method are in the form of flat sheets. The membrane is cut using a crankcase knife to the appropriate dimensions ASTM D638 Tipe IV (E3-95, 2016).

After the membrane was printed and cut, a test was performed on the *Polyvinylidene fluoride* membrane (PVDF). The test aims to determine the characteristics, mechanical properties, and water treatment performance of the polymer material used as a water filter membrane. In this research, tensile testing, *Scanning Electron Microscopy* (SEM) testing, and *Clean Water Permeability* (CWP). Tensile testing is a basic test used to determine the mechanical strength of a material, in this case, a membrane. The test specimens carried out in this test have the form of a flat sheet which is pulled apart, using a Tensile test tool (*ZWICK ROEL Material Testing Machine*) and with the standard of ASTM D 638. 05/2008 *Tensile Test On Plastics*.



Figure 2 ZWICK ROEL Material Testing Machine

Test Scanning Electron Microscope:

SEM observation is one of many types of testing using the electron microscope by scanning the specimen using a beam of high energy electrons on the scan in a raster pattern.

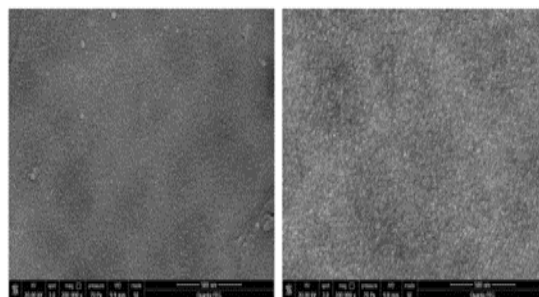


Figure 3 SEM observation results on membrane PVDF

Clean Water Permeability Test:

The *Clean Water Permeability* (CWP) test aims to determine which membranes are porous or stable over the entire surface. In this test the membrane was tested for 1 hour using a dead-end system. The dead-end system is a membrane filtering process by flowing water straight towards the membrane. In this case an angle will be formed between the direction of water flow and the membrane pores.

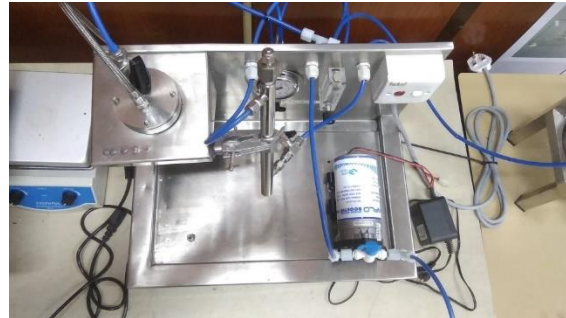


Figure 4. Clean Water Permeability

III. Result

In this researches, the mixing of *Polyvinylidene fluoride* (PVDF), *N,N-DimethylFormamide* (DMF) membrane polymer with the addition of 1% Tin Dioxide as an additive, Samples were made with 3 samples, namely PVDF & SnO₂ (15 wt%), PVDF & SnO₂ (17.5 wt%) and PVDF & SnO₂ (20 wt%). Each sample was tested for tensile strength using the *Zwick Roell Material Testing Machine* to analyze the mechanical properties of the membrane, testing the performance of water treatment with a *clean water permeability* (CWP) tool to analyze the strength of the membrane during operation, testing the surface microstructure of the membrane was analyzed using a *Scanning Electron Microscopes* (SEM).

Tensile Strength Testing:

Tensile Tests carried out on membrane PVDF with *Tin Dioxide* admixture (SnO₂) with the standard ASTM D638-04. *ZWICK ROEL Material Testing Machine* (Type BT2-FR020TH.A60) is a tensile testing tool in this test which is at the Bangka Belitung Manufacturing Polytechnic. Tensile testing on membrane PVDF and SnO₂ was carried out to analyze and determine the durability and ability of these membrane to withstand tensile loads. Tensile testing was carried out at each predetermined membrane concentration with 15wt%, 17.5wt%, and 20wt% with 5 times each test to get the best results.

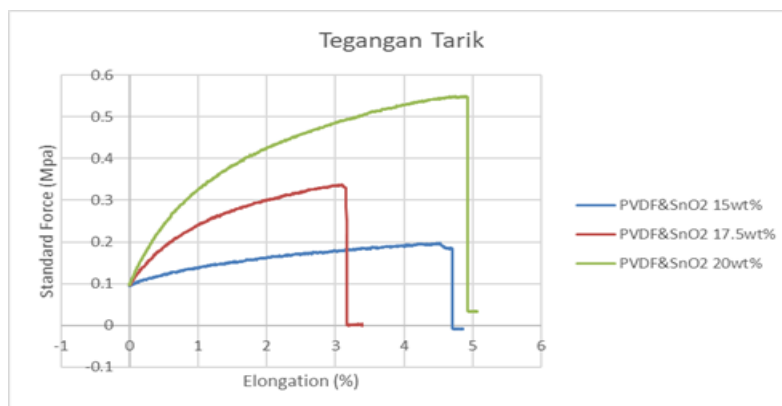


Figure 5 Graphics of the average tensile stress

Microscopic Test:

Membrane PVDF & SnO₂ with concentrations of 15%, 17.5%, and 20% using the Flat Sheet method show that as the concentration increases, the tensile strength of the membrane or the ability of the membrane to withstand loads increases. Membrane PVDF & SnO₂ with a concentration of 20% has a strength of 0.565 MPa while for a concentration of 17.5% and 15%, respectively 0.352 Mpa and 0.192 Mpa. This is directly proportional to previous research that increasing the concentration of PES and PVDF by mixing TiO₂ in the mixture for making membranes can increase the tensile strength of the membrane.

The small amount of PVDF in the printing solution causes the composition of the particles to be dispersed in the printing solution to be uneven so that during the tensile test the membrane is easily brittle and has a small tensile strength value. Membrane Morphology Mixed membrane morphology between PVDF and SnO₂ using a *Scanning Electron Microscope (SEM)*, observations of the mixed membrane structure with different weight percentages are shown in Figure 6-8.

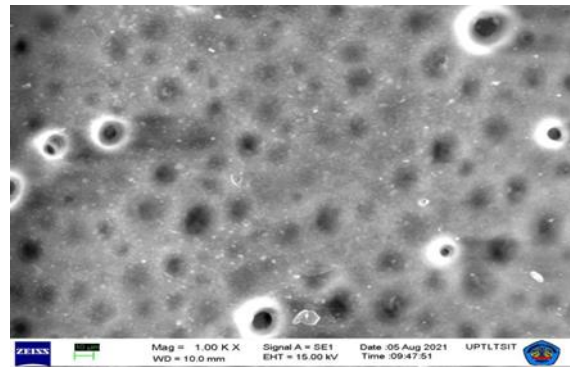


Figure 6 SEM Membrane PVDF & SnO₂ 15 wt%

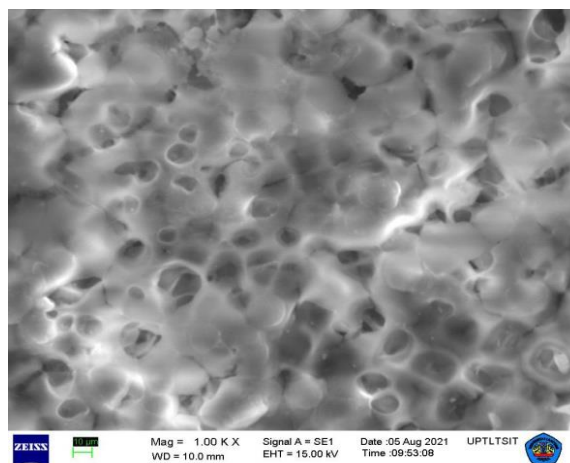


Figure 7 SEM Membrane PVDF & SnO₂ 17.5 wt%

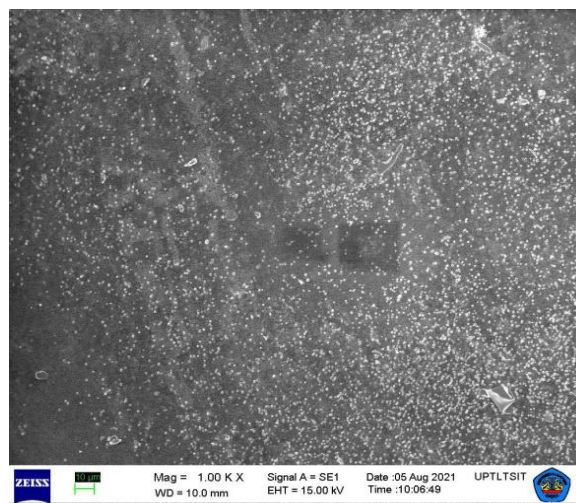


Figure 8 SEM Membrane PVDF & SnO₂ 20 wt%

The results of *Scanning Electron Microscopy (SEM)* observations with different concentrations show significant differences from the three concentrations, PVDF and SnO₂ with a concentration of 15% have large pores compared to the others and agglomerations are spread over the entire surface, whereas at a concentration of 17.5 % membrane have almost the same surface but little agglomeration occurs.

This agglomeration greatly affects the strength of the membrane in holding the load, because the agglomerates that occur become stress concentrators when water is filtered. while the pore size that is not dense has an effect on reducing the area of the membrane when the tensile test is carried out. The concentration of the PVDF membrane and SnO₂, makes the structure of the fiber braid formed denser, this makes the tendency for porosity to become smaller and denser in the membrane.

Water Treatment Performance:

The formation of mixed membrane PVDF and SnO₂ polymers with respective concentrations of 15wt%, 17.5wt% and 20wt% was carried out due to knowing and analyzing the good processing performance from previous studies. To determine the performance of water treatment from PVDF& SnO₂ membranes, *Clean Water Permeability (CWP)* was used. In this test, the membrane was tested for 1 hour, with a membrane area of 0.001809 m² using a dead-end system.

Table 2. Membrane Flux Calculation

Spesimen	Volume Permeat (L)	Luas Membran (m ²)	Waktu (h)	Fluks (L/m ² h)
PVDF & SnO ₂ 15%	0.042	0.001809	1	23,22
PVDF & SnO ₂ 17,5%	0.059	0.001809	1	32,614
PVDF & SnO ₂ 20%	0.086	0.001809	1	47,54

The increase in flux results that occur in the membrane increases significantly. The concentration of 20wt% PVDF and SnO₂ with a flux value of 47.54 Lm-2h-1 was inversely proportional to the 15wt% concentration with the lowest flux value of 23.22 Lm-2h-1. This increase in the 20wt% membrane flux shows that mixing PVDF and SnO₂ polymers succeeded in increasing the hydrophilicity of the membrane because the flux value of the membrane also increased with increasing hydrophilicity.

This hydrophilicity is one of the properties that must be possessed by a filtration membrane, because PVDF membranes are prone to fouling and low flux. This happens because the flux is the amount of water that passes through the membrane. The longer the time when water passes through the membrane, it indicates that the membrane pores are very small.

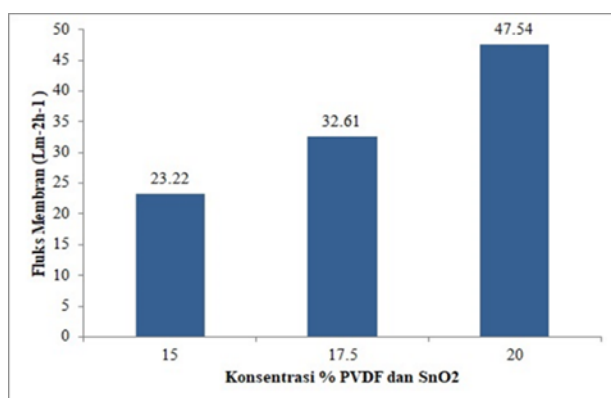


Figure 9 Graphics Flux PVDF and SnO₂

IV. Conclusion

Based on the data and research results that have been obtained on the formation of a mixed membrane of *Polyvinylidene Fluoride (PVDF)* and Tin Dioxide (SnO₂), it can be concluded as follows:

1. The formation of mixed membrane polymer Polyvinyliden Flouride (PVDF) with the addition of Tin Dioxide shows results that are as expected as an alternative to water filtration membrane.
2. The mixed PVDF and SnO₂ membranes were subjected to tensile testing using ASTM D638 standards, this test showed an increase in the average tensile stress of each concentration, namely a 20% concentration had a strength of 0.565 MPa while for a concentration of 17.5% and 15% each 0.352 MPa and 0.192 MPa.

3. The surface structure of the mixed membrane using SEM shows significant differences in each concentration, the occurrence of agglomeration on the surface of the membrane is caused by uneven mixing. The dense pore size of the membrane is directly proportional to the tensile stress that occurs in the PVDF membrane.
4. The performance of water treatment using *Clean Water Permeability (CWP)* showed an increase with increasing PVDF & SnO₂ concentration, an increase in flux at a concentration of 20wt% which was 47.54 Lm-2h-1, and for a concentration of 17.5wt% with a flux of 32.61 Lm-2h-1, whereas at a concentration of 15wt% the lowest flux value was 23.22 Lm-2h-1.

V. ACKNOWLEDGMENTS

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