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Characteristic Analysis Of Polyethersulfone (PES) Based Membrants With Silver Nitrate ($AgNO_3$) Addition As Water Treatment Application

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Abstract:
Water is one of the essential elements for human life, as it plays a crucial role in various aspects of life. Therefore, the fulfillment of the need for adequate clean water is one of the basic rights of the community that must be guaranteed. This study aims to analyze the characteristics of Polyethersulfone (PES) based membrane with the addition of Silver Nitrate ($AgNO_3$) as an alternative material in water treatment applications. The modification was done through molding method with 25 kV DC electric field and $AgNO_3$ concentration variation of 1%, 1.5%, and 2%. Characterization was carried out through tensile testing (ASTM D638) Type IV: 2008 Standard Test Method for Tensile Properties of Plastics, Scanning Electron Microscope (SEM), and Clean Water Permeability (CWP). The results showed that the addition of $AgNO_3$ had an impact on mechanical strength and water permeability. Membranes with 1% $AgNO_3$ concentration showed the highest tensile strength and water flux, at 5.0009 MPa and 6.117 $L\text{-}m^{-2}\text{-}h^{-1}\text{-}bar^{-1}$, respectively. Meanwhile, an increase in $AgNO_3$ concentration caused particle agglomeration, pore irregularity, and a decrease in porosity and mechanical strength. SEM observations revealed differences in microstructure between the concentration variations, where the membrane with 2% $AgNO_3$ showed a rough texture and uneven pore distribution. These results suggest that the use of $AgNO_3$ in optimum levels can increase the effectiveness of membranes in water treatment applications.
Keywords: *Membrane, polyethersulfone (PES), Silver nitrate ($AgNO_3$), Electric Field, Clean Water Permeability (CWP), Scanning Electron Microscope (SEM).*

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I. Introduction

Water is one of the important elements for human life, because it has a crucial role in various aspects of life. Therefore, the fulfillment of the need for adequate clean water is one of the basic rights of the community that must be guaranteed [1].

With the rapid rate of population growth, the need for clean water is increasing. Urbanization, widespread industrialization, and increased human activity to meet various household needs according to the ever-changing demands of life are driving factors [2].

Faced with the importance of clean water needs, many people have begun to look for alternatives to ensure the availability of adequate clean water, here membrane technology has become one of the most talked about water treatment options around the world because of its highly selective separation media capabilities [3].

Membrane technology has advantages such as low energy use, easy manufacturing process, environmentally friendly, does not require many additional tools, is easy to operate, and produces high quality water [4].

Although membrane technology has various advantages, its application on an industrial scale still experiences obstacles, one of which is the problem of *fouling*. *Fouling* occurs due to adsorption and accumulation of pollutants on the surface and in the pores of the membrane [5].

Therefore, the addition of Silver Nitrate ($AgNO_3$) is very effective in suppressing the growth of *coliform* bacteria, which is the main cause of *biofouling* on water filtration membranes [6].

The addition of *N,N- Dimethylformamide* (DMF) is also proven to increase the mechanical strength of the resulting material [7], and to increase the effectiveness and efficiency of the membrane requires modification in terms of the membrane surface involving the *electric* field method (*Electric Field*) using a DC current of 25 kV which serves to refine the membrane surface and produce a uniform pore size [8].

To determine the characteristics of membranes made from *Polyethersulfone* (PES) with the addition of Silver Nitrate ($AgNO_3$) which is reviewed through testing mechanical properties using tensile testing, *Scanning Electron Microscope* (SEM), and water permeability using *Clean Water Permeability* (CWP) testing.

II. Material And Methods

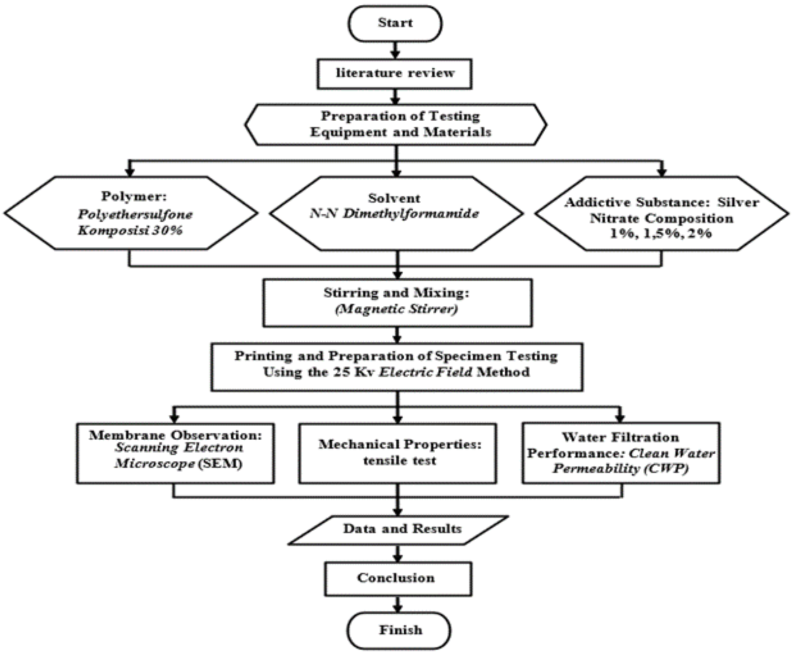

Figure 1. Research Flow Chart

Membrane Preparation

In the initial preparation that must be done is to prepare the materials and instruments needed, namely starting from *Polyethersulfone (PES)*, which is the main material of the membrane. *Polyethersulfone* (PES), *N,N-Dimethylformamide* (DMF) and Silver Nitrate ($AgNO_3$) additives. The membrane manufacturing equipment includes a magnetic stirrer and *Electric field* or also a copper mold with a 25 Kv DC electric current using a tool located at the Sriwijaya University Engineering Materials Laboratory. *Scanning Electron Microscope* (SEM) observations were made at Sriwijaya University, South Sumatra. Tensile testing and water filtration performance with the *Clean Water Permeability* (CWP) tool were carried out at the Sriwijaya University Engineering Materials Laboratory

Preparation of Mixing Process

Membranes were prepared in 3 specimens of fraction by weight (wt%) with the same polymer blend in each sample 30% PES composition. Based on research, the use of 30% PES polymer composition produces a type of membrane with good characteristics, where the use of polymers with higher concentrations will make the structure and pore bonds of the membrane stronger which has a good impact on the tensile strength value. In this study, the initial process carried out was dissolving PES and DMF, mixing the PES with DMF. $AgNO_3$ as a comparison in each sample, including 1%, 1.5% and 2% at each PES concentration. Mixing the solution (50gr) using a *magnetic stirrer*, at a normal temperature of approximately 40°C with a time of 7 - 8 hours until homogeneous, the PES membrane is poured into an airtight glass which aims to process the precipitation and review of the solution that has not been mixed [9]. The following is a description of the membrane mixing composition:

Table 1. Membrane Mixing Composition

Membrane	PES (gram)	DMF (gram)	$AgNO_3$ (gram)
PES 30wt% @$AgNO_3$ 1wt%	15	34,85	0,15
PES 30wt% @$AgNO_3$ 1,5wt%	15	34,775	0,225
PES 30wt% @$AgNO_3$ 2wt%	15	34,7	0,3

Electric Field Molding Method

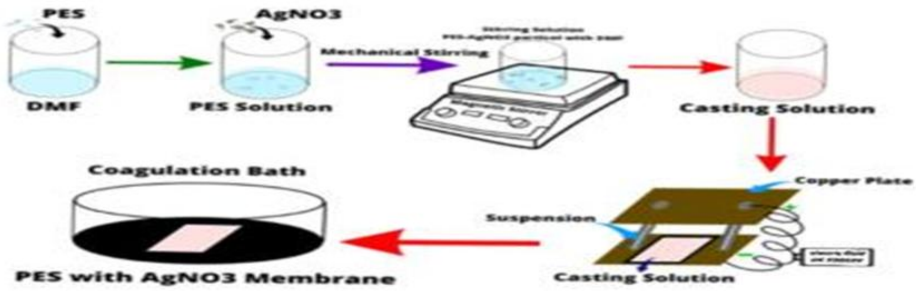

Figure 2. Membrane Preparation Process

In this study, surface modification will be carried out using the *Electric Field* method with a DC current of 25 Kv. The research was conducted using a copper plate that was electrified with a 25 Kv DC current, generated by an *Electric Field* device sourced from a 3V battery, then raised to 25 Kv using a *step-up transformer*. The process of making specimens is carried out through general procedures in membrane manufacturing, namely inversion phase printing, *electric field* current flow, and immersion in certain media. The resulting specimen is *flatsheet-shaped*. The mixture that has gone through the precipitation stage will proceed to the molding process with the phase inversion method, then flowed with a DC current of 25 Kv for 30 seconds. After that, the specimen was immersed until it was detached from the copper plate, and dried at room temperature for 24 hours.

Scanning Electron Microscopy (SEM)

Figure 3. *Scanning Electron Microscopy* (SEM)

A Scanning Electron Microscope (SEM) is a type of advanced microscope that uses electron-based technology to produce high-resolution images of sample surfaces, enabling detailed observation down to the microscopic level. SEM utilizes electron beams to reveal surface structures and characteristics with great clarity and accuracy. This technology enables researchers and practitioners to analyze material structure details, such as morphology, texture, and element distribution, which cannot be observed with conventional microscopes. With its high-resolution capability and extraordinary detail, SEM is widely used in various fields, including materials science, nanotechnology, biology, forensics, and engineering. The main advantage of SEM is its ability to magnify up to millions of times, allowing in-depth observation of surface structure, topography, and chemical composition of the sample being tested. With its high magnification and image clarity, SEM is a very useful tool in various fields of research to understand the microscopic details of a material or object in greater detail.

Tensile Testing

Figure 4. ALIYIQI AMF-20 Tensile Testing Tool

The specifications of this ALIQIYI AMF-20 tool are
•Range: 20N; 2kg; 4.5Lb; 72 Oz
•Accuracy: : ± 1 %
•Power: 2 AAA; 1.5 V battery

Tensile test is a fundamental test used to determine the mechanical strength of a material, one of which is membrane. In theory, the tensile test method is a test by pulling the specimen until it breaks and then the tensile load of the specimen will be seen to get the tensile stress. In this test, the test specimen used has a flat sheet shape which is pulled until it breaks using a tensile tester and using the ASTM D 638 Type IV standard: 2008 *Standard Test Method for Tensile Properties of Plastics.* This tensile testing method is also used as a reference for specimens to determine how much tensile strength the material has. *Tensile strength* (*tensile strength, ultimate tensile strength*) is the maximum voltage that a material can withstand when stretched or pulled, before the material breaks.

***Clean Water Permeability* (CWP)**

Figure 5. *Clean Water Permeability* (CWP)

Clean Water Permeability (CWP) or water flux is an important parameter used to measure the rate at which water particles pass through a membrane per unit of time and surface area. Water flux provides insight into the characteristics of the membrane and its performance in separation applications. Specifically, flux is defined as the amount of permeate (clean water) produced during membrane operation per unit of membrane surface area and per unit of time. Flux values not only reflect the membrane's ability to transport water but also serve as a key indicator for assessing separation efficiency. The steps for testing CWP are as follows:

1. Cut the specimen to size on the CWP tool, with d = 0.048 m.
2. Install the CWP tool and prepare water as a testing medium.
3. Place the specimen on the membrane holder in the tool.
4. Turn on the CWP device, then set the pressure used.

5. Prepare a chemical tube as a water permeate counter medium.
6. Calculate the test time until completion.
7. The test data is calculated with the formula already listed, and that is the result of the test *flux* value.

Result And Discussion

Sample	Tensile Stress (N)	Thickness (mm)	wide (mm)	Surface Area (mm^2)	Maximum voltage (N/mm^2)	Average	Standard Deviation
PES 30% $AgNO_3$ 1%	3,7	0,5	6	3	1,233	**5,0009**	2,719
	18,8	0,5		3	6,266		
	27,1	0,6		3,6	7,527		
PES 30% $AgNO_3$ 1,5%	14	0,6		3,6	3,888	**4,520**	1,932
	25,7	0,6		3,6	7,138		
	7,6	0,5		3	2,533		
PES 30% $AgNO_3$ 2%	4,4	0,3		1,8	2,444	**3,098**	0,828
	12,8	0,5		3	4,266		
	6,2	0,4		2,4	2,583		

Table 2. Tensile Testing Value Calculation Data

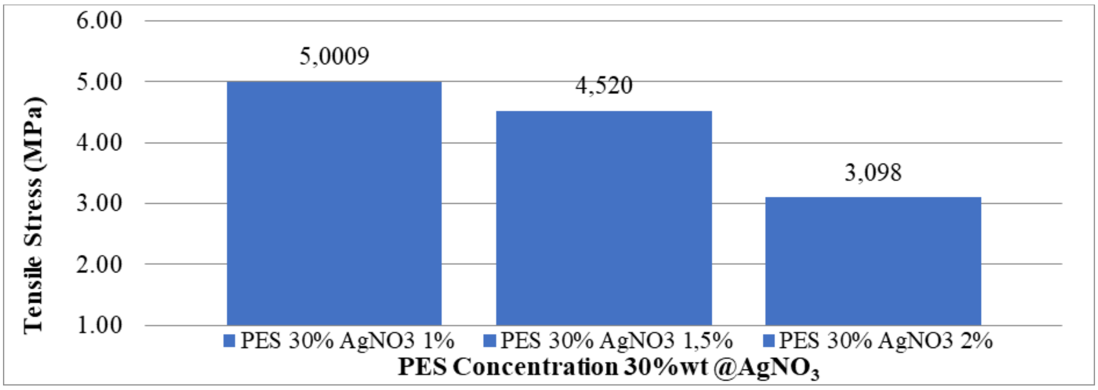

Figure 6: Graph of Average Tensile Strength of Membrane in Each Concentration

Based on Figure 6, it is known that of the three PES membrane samples modified with the addition of Silver Nitrate ($AgNO_3$) at concentration variations of 1wt%, 1.5wt%, and 2wt% experienced a decrease in tensile strength as the concentration of ($AgNO_3$) increased. Membranes with the addition of $AgNO_3$ at 1wt% concentration produced the highest average tensile strength, which was 5.0009 MPa. In contrast, the lowest value was found in the membrane with 2wt% concentration, which amounted to 3.098 MPa. Meanwhile, the membrane having a concentration of 1.5wt% showed an average tensile strength of 4.520 MPa. According to the data results shown in Table 2, it can be observed that increasing the concentration of Silver Nitrate ($AgNO_3$) in PES@$AgNO_3$ solution shows an inverse relationship to the value of tensile stress of the membrane. Membranes with 1wt% $AgNO_3$ content showed higher tensile stress compared to membranes with 1.5wt% and 2wt% concentrations. Presumably, at a concentration of 1wt%, a more stable and complex network of polymer bonds is formed, so as to strengthen the tensile resistance between polymer chains. In contrast, the addition of higher amounts of $AgNO_3$ can disrupt the membrane structure, causing the formation of inhomogeneous morphology due to uneven mixing. As a result, the pore distribution becomes non-uniform, the pore size tends to decrease, and the membrane surface formed has weak inter- particle bonds.

Sampel	Permeat Volume (L)	Membrane Area (m^2)	Time (h)	Preasure (bar)	Flux ($L.m^{-2}.h^{-1}.bar^{-1}$)	Std. Deviation
A1	0,06	0,001809	0,25	2	4,643	1,611
A2	0,0055	0,001809	0,25	2	2,211	
A3	0,0051	0,001809	0,25	2	4,201	
Average					6,117	

Table 3. Calculation of *Flux* Value of PES Membrane 30wt%@$AgNO_3$ 1wt%

Sampel	Permeat Volume (L)	Membrane Area (m^2)	Time (h)	Preasure (bar)	Flux ($L.m^{-2}.h^{-1}.bar^{-1}$)	Std. Deviation

A1	0,0052	0,001809	0,25	2	4,643	1,611
A2	0,0027	0,001809	0,25	2	2,211	
A3	0,0031	0,001809	0,25	2	4,201	
Average					6,117	

Table 4. Calculation of *Flux* Value of PES Membrane 30wt%@$AgNO_3$ 1,5wt%

Sampel	Permeat Volume (L)	Membrane Area (m^2)	Time (h)	Preasure (bar)	Flux ($L.m^{-2}.h^{-1}.bar^{-1}$)	Std. Deviation
A1	0,06	0,001809	0,25	2	4,643	1,611
A2	0,0055	0,001809	0,25	2	2,211	
A3	0,0051	0,001809	0,25	2	4,201	
Average					6,117	

Table 5. Calculation of *Flux* Value of PES Membrane 30wt%@$AgNO_3$ 2wt%

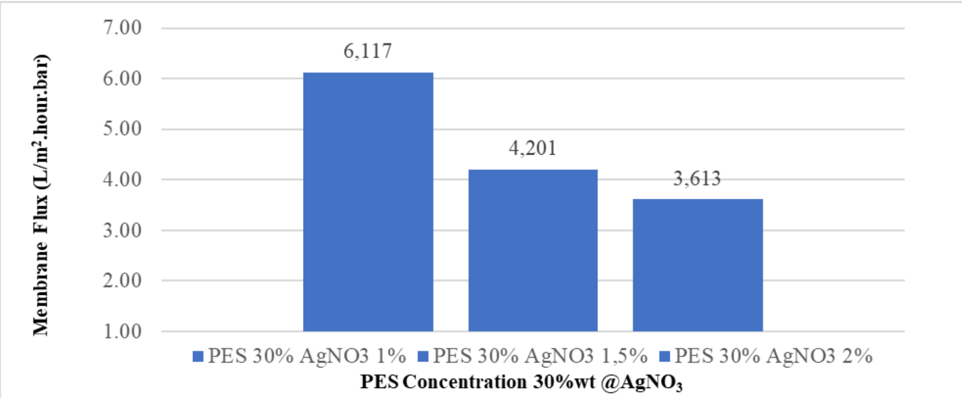

Figure 7. Graph of Average *Flux* Value of Membrane At Each Concentration

Based on Figure 7, there is a significant decrease in the membrane *flux* value as the concentration of Silver Nitrate ($AgNO_3$) in the mixture increases. The highest *flux* value was recorded at the lowest concentration, PES@$AgNO_3$1wt%, with a value of 6.117 $L\text{-}m^{-2}\text{-}h^{-1}\text{-}bar^{-1}$. At 1.5wt% concentration, the *flux* value decreased to 4.201 $L\text{-}m^{-2}\text{-}h^{-1}\text{-}bar^{-1}$, while 2wt% concentration showed the lowest *flux* value of 3.613 $L\text{-}m^{-2}\text{-}h^{-1}\text{-}bar^{-1}$. This decrease in *flux* can be attributed to the increasing concentration of $AgNO_3$, there is an increase in the number and size of silver particles formed on the membrane surface. The accumulation of these particles leads to an increase in the density of the mixing solution. Not This homogeneity eventually triggers the formation of particle agglomeration on the membrane surface, so decrease permeability and efficiency of water flow through the membrane.

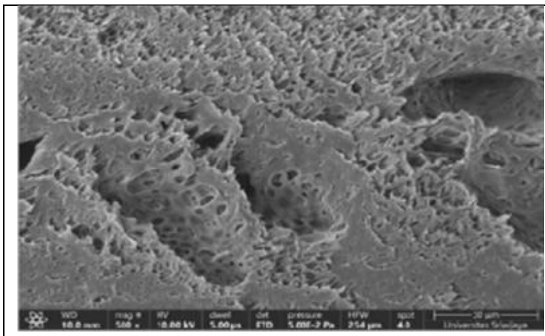

Figure 8. SEM Observation Result of *Cross- Section* of PES Membrane 30wt%@$AgNO_3$ 1wt%

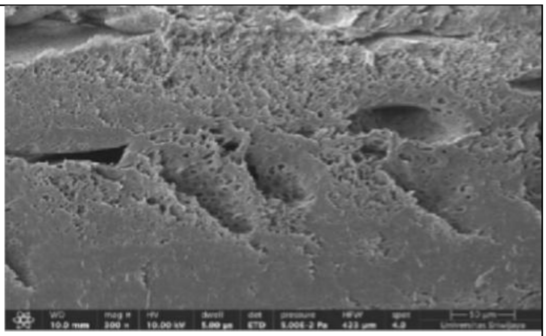

Figure 9. SEM Observation Result of Structure section of PES Membrane 30wt%@$AgNO_3$ 1wt%

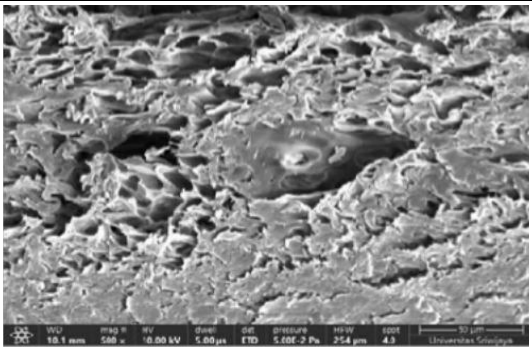
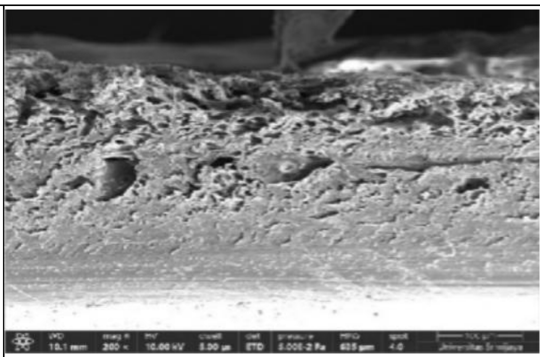

Figure 10. SEM Observation Result of *Cross-Section* of PES Membrane 30wt%@$AgNO_3$ 2wt%	**Figure 10.** SEM Observation Result of Structure section of PES Membrane 30wt%@$AgNO_3$ 2wt%

The results of surface morphology characterization using a Scanning Electron Microscope (SEM) showed significant morphological differences between the two membrane compositions tested, indicating that variations in composition have a significant effect on the microstructure of the membranes formed. The membrane structure exhibited a sponge-like, porous shape and showed differences in the agglomeration patterns of particles on its surface. In Figures 8 and 9, which show the Polyethersulfone (PES) membrane with a composition of 30wt%@$AgNO_3$ 1wt%, the surface has a smooth texture with a fairly uniform distribution of small pores, although the agglomeration is not entirely uniform. Conversely, in Figures 10 and 11 for the PES membrane with a composition of 30 wt% $AgNO_3$ and 2 wt% $AgNO_3$, the surface appears rougher. However, the pores formed appear to be unevenly distributed and tend to be larger in size. This condition is thought to be due to increased agglomeration of silver nitrate particles, which causes resistance in pore formation and a decrease in porosity in the membrane. Therefore, the application of an electric field treatment with a direct current (DC) of 25 kV for 15 seconds during the membrane printing stage plays a role in helping to stretch the solution, thereby facilitating pore formation in the membrane structure.

III. Conclusion

From the results of observations using a Scanning Electron Microscope (SEM), the PES membrane with a composition of 30wt%@$AgNO_3$ 1wt% was found to have a smooth surface with a fairly uniform distribution of small pores, although there was some agglomeration that was not very uniform. Meanwhile, the surface of the PES membrane with a composition of 30wt%@$AgNO_3$ 2wt% appears rougher. This condition is suspected to be caused by increased agglomeration of silver nitrate particles, which hinders pore formation and reduces porosity. A significant decrease in membrane flux values was observed with increasing $AgNO_3$ concentration in the membrane mixture. The highest flux value was recorded for the PES@$AgNO_3$ 1wt% membrane at 6.117 $L\cdot m^{-2}\cdot h^{-1}\cdot bar^{-1}$. At a concentration of 1.5 wt%, the flux decreased to 4.201 $L\cdot m^{-2}\cdot h^{-1}\cdot bar^{-1}$, and further decreased at a concentration of 2 wt% with a flux value of 3.613 $L\cdot m^{-2}\cdot h^{-1}\cdot bar^{-1}$. Tensile testing was performed using the ALIYIQI AMF-20 tensile testing machine based on ASTM D 638 Type IV standards. The test results showed that an increase in the concentration of silver nitrate ($AgNO_3$) in the membrane had a significant effect on reducing the tensile strength value. Among the three samples tested, the PES@$AgNO_3$ membrane with a concentration of 1 wt% exhibited the highest average tensile strength value, which was 5,0009 MPa. This indicates that the addition of excessive amounts of $AgNO_3$ can lead to a decrease in the membrane's mechanical strength due to increased agglomeration, which disrupts the uniformity of the material's structure.

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