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Analysis of Dielectric Barrier Discharge (DBD) Plasma with Variation Flow Rate to Increase Wettability Polytetrafluoroethylene (PTFE)

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Abstract: Polytetrafluoroethylene (PTFE) is a widely used fluoropolymer with excellent thermal stability and chemical resistance. However, its hydrophobic nature makes it challenging to process and manage as waste. Plasma treatment is an effective method for modifying PTFE surface properties to improve wettability. This study investigates the effect of Dielectric Barrier Discharge (DBD) plasma treatment on PTFE with variations in oxygen gas flow rates. The plasma reactor was fabricated using a pyrex glass chamber, aluminum tape electrodes, and a stepper motor-driven stirring mechanism to ensure uniform exposure. PTFE samples were treated with DBD plasma at different oxygen flow rates (20–50 mL/min) for three minutes. Wettability changes were analyzed using Contact Angle Measurement (CAM), while plasma characterization was performed using Optical Emission Spectroscopy (OES). The results indicate that plasma treatment reduces the PTFE contact angle, improving its wettability. However, at higher flow rates, some samples adhered to the reactor walls, leading to uneven plasma exposure. The study concludes that plasma treatment effectively enhances PTFE wettability, but optimizing flow rate parameters is essential to ensure uniform surface modification.

Keywords: Dielectric Barrier Discharge (DBD); PTFE; Wettability; Surface Modification.

Introduction

Polytetrafluoroethylene (PTFE) is a plastic material with a well-known trade name, Teflon. PTFE belongs to the fluoropolymer category. It can be used as a coating for parts or machine components exposed to heat, as a coating for laboratory equipment requiring corrosion resistance, and as a coating for cookware. Additionally, PTFE is widely used as an electrical insulator, seal, gasket, bushing, and anti-friction material in the chemical, electrical, and textile industries. The final products made from PTFE as the base material will inevitably generate waste. The waste produced from the use of PTFE usually appears as chips, fibers, or shavings depending on the process involved. This waste can create environmental problems if not addressed promptly (Aryanta et al., 2017).

PTFE as a type of fluoropolymer, possesses unique properties such as high thermal stability, low surface tension, and resistance to chemical reactions with various chemicals. These unique properties make PTFE one of the most widely used materials in industrial applications (Baumgärtner et al., 2001). However, the use of PTFE in various industrial components results in several problems, including waste. PTFE waste is difficult to decompose due to its hydrophobic nature. After use, PTFE waste cannot be incinerated because it

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releases hydrogen chloride and other toxic substances, posing environmental hazards. On the other hand, burying PTFE waste in soil can lead to its degradation into toxic trifluoroacetate (TFA), which is harmful to plants. Therefore, special treatments are required to address the waste produced.

One method to address PTFE waste is by using plasma treatment. Plasma treatment on PTFE aims to modify its surface properties. By exposing the PTFE surface to ionized gas in a plasma system, changes in the PTFE surface can be achieved (Selim et al., 2007). Common gases used in plasma treatment include Ar, N_2, O_2, NH_3, and CF, each with different physical and chemical characteristics to modify PTFE surfaces. Plasma can occur naturally or be artificially created. Currently, plasma is widely used in various industries, with plasma being generated between two electrodes under pressures ranging from 10 to 1000 Pascals, which is lower than atmospheric pressure at 101,325 Pascals. This type of plasma is known as low-pressure plasma (Nasution et al., 2016).

Given the problems arising from the use of PTFE as an industrial material and the effects of plasma treatment on polymer surfaces, the author attempts to analyze a plasma system that can be used to modify the PTFE surface and improve its wettability under low-pressure plasma treatment. This approach aims to manage PTFE waste effectively by turning it into a mixture for other materials, such as composites and others. Based previous research, a plasma reactor system with a dielectric barrier discharge (DBD) was developed, incorporating a rotating mechanism and gas input to optimize plasma treatment on surfaces. However, in practice, the plasma system still faced challenges, such as the frequent ejection of samples from the reactor tube, necessitating improvements to ensure the plasma system functions optimally.

Method

The research began with the fabrication of a plasma reactor using heat-resistant pyrex glass, designed to operate in a closed system. The reactor cover was equipped with several holes for inserting oxygen gas from a gas cylinder, installing a stirrer rod, and connecting electrodes. The stirrer rod was driven by a NEMA 17 stepper motor, which converted electrical energy into kinetic energy to rotate the PTFE sample. This ensured that the sample was evenly exposed to plasma during treatment. The electrodes were made from aluminum tape. The active electrode was designed with a striped pattern to focus the plasma in specific areas, while the passive electrode was a simple rectangular shape placed outside the pyrex glass. The dielectric barrier of the reactor was provided by the pyrex glass itself.

During plasma treatment, PTFE samples were exposed to plasma for 3 minutes with variations in oxygen gas flow rates ranging from 20 to 50 mL/min, adjusted using a flowmeter. Once the flow rate was stable, the power supply was activated to generate plasma. After the treatment, the samples were tested to evaluate their wettability using a contact angle measurement (CAM) tool by dripping 20 μL of distilled water on their surface. The contact angle was analyzed to observe changes in surface properties. Additionally, plasma characterization was carried out using Optical Emission Spectroscopy (OES) to identify the reactive species generated during the plasma treatment. Variations in oxygen gas flow rates were also analyzed for their influence on plasma characteristics and the sample's surface morphology.

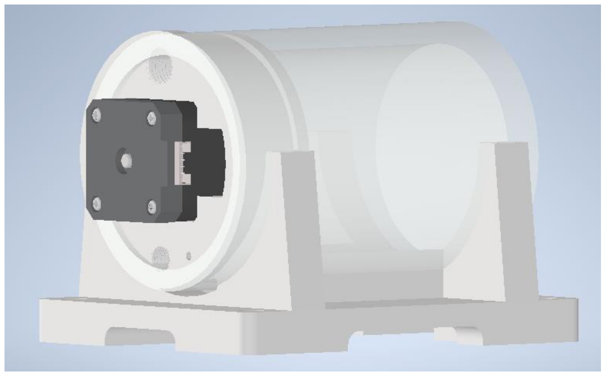


Figure 1. Design Plasma Reactor

Result and Discussion

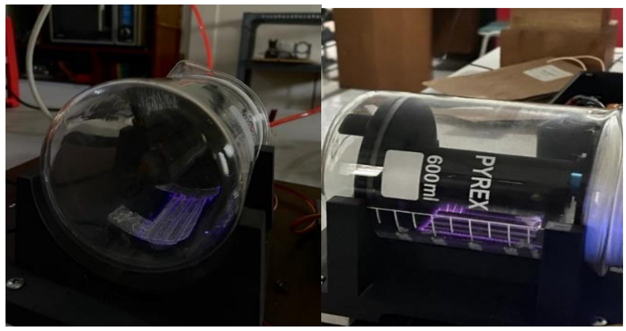
The plasma reactor tube uses a pyrex glass tube with a volume of 600 mL. The use of a pyrex glass reactor tube is because pyrex glass is included in good dielectric materials and is heat resistant so that it is not easily cracked when exposed to heat from plasma emissions. A cover is provided on the open part of the reactor tube so that during treatment the sample does not scatter out. In addition, this cover also functions as a place to place the stepper motor as a driver. The function of the stepper motor in the lid is to move the stirring blade inside the reactor tube. There are also several holes on the reactor lid. The first hole is a hole to insert the oxygen gas hose into the reactor tube. Then there is a hole for the output hose from inside the reactor. The function of the output hose is to help spread ozone as a by-product into the open air.

In addition, there is also a hole to place the stepper motor complete with its stirring blade and a small hole to connect the connecting cable to the inner electrode. This plasma reactor is placed on a support with a fan at the bottom. The use of supports and the addition of fans under the plasma reactor is so that during plasma treatment, the plasma reactor can be placed in a strategic position so that it is easy for the reactor to be used for treatment. The addition of fans in the support section is to help cool the plasma reactor whose temperature increases due to the electric voltage provided and to reduce the risk of cracks in the plasma reactor tube due to increased temperatures.

Figure 2. Dielectric Barrier Discharge Plasma Reactor

Plasma can be generated by providing high voltage, with 3 kV AC voltage where this voltage has been amplified using a ZVS circuit. The voltage input also needs to consider the area of the electrode used. This experiment uses electrodes with an initial size of 10 x 4 cm. This is done so that the length of the tube from top to bottom can generate plasma. Then, a voltage of 3 kV AC was given but plasma could not appear. The second experiment was carried out by reducing the size of the electrode used, namely 8 x 2.5 cm and when given 3 kV AC voltage, plasma could appear.

This study used variations in flow rates ranging from 20 mL/min to 50 mL/min with oxygen as the input gas and an increase in each flow rate of 5 mL/min. The given flow rate affects the plasma flame produced and the intensity of the species of the resulting plasma. The greater the given flow rate, the brighter the resulting plasma flame will be. This is due to the increase in the number of gas particles flowing into the reactor tube. If the gas particles flowing are more, this will cause the collision process between particles to also be greater. The following are the differences in plasma flames produced at a flow rate of 20 mL/min and a flow rate of 50 mL/min



(a) (b)

Figure 3. Plasma at Flow Rate (a) 20 mL/menit, (b) 50 mL/menit

One way to improve the wettability of polymers is by giving plasma treatment for three minutes with variations in oxygen gas flow rates and then observing the changes in wettability through contact angle measurements. This contact angle measurement was carried out by dripping 20 µL of distilled water on the surface of PTFE that had been given plasma treatment and the size had been adjusted to the size of the preparation table and then the contact angle was observed using Contact Angle Measurement (CAM).

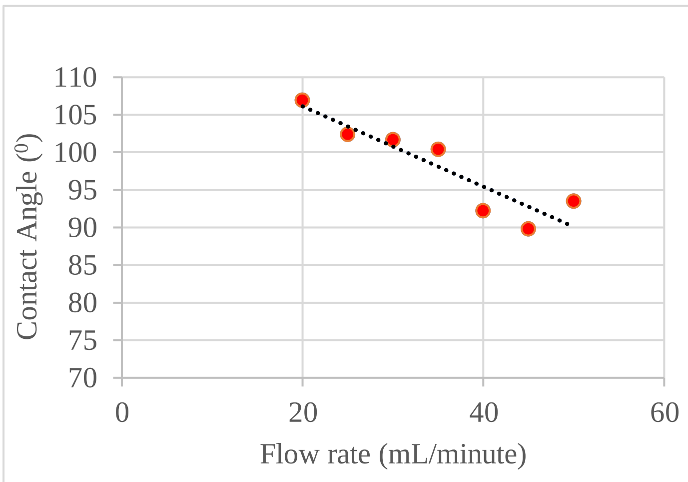
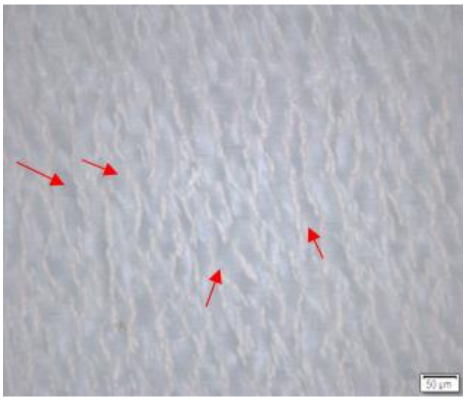
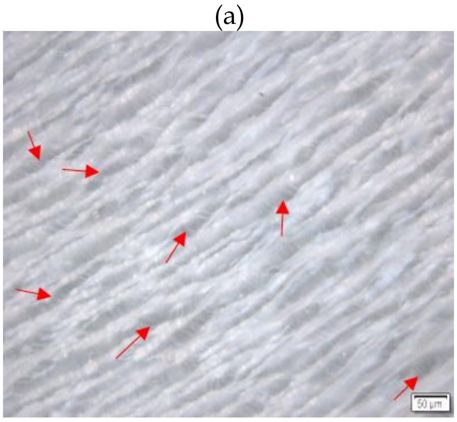


Figure 4. Relationship between Composition Variation and Composite Hydrophobicity

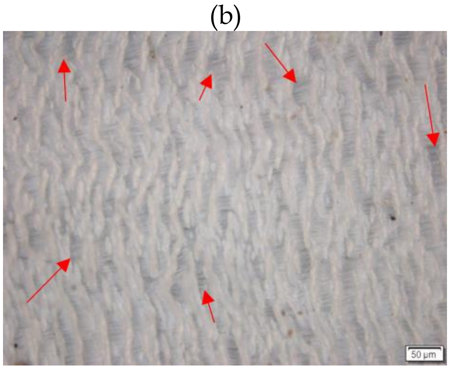
The contact angle observed on the sample surface after being given plasma treatment decreased along with the increase in flow rate. Indeed, it is not very significant for the decrease in contact angle, even when given a flow rate of 50 mL/min the observed contact angle actually shows an increase in contact angle. This is influenced by the increasing flow rate which will cause the sample in the reactor tube to scatter in the reactor tube and some samples stick to the walls of the reactor tube. This is because the mass of the sample is light enough so that it is easily scattered by the flow rate of oxygen gas given. Thus, this causes the plasma treatment of the sample to not occur evenly even though it is assisted by the stirring spoon in the reactor tube. This decrease in contact angle is caused by the interaction between the PTFE waste sample and the plasma that appears. The plasma that appears contains positive ions, electrons, free radicals, and other reactive elements which then bombard the surface of the PTFE with each energy in the plasma element. This interaction will make the PTFE surface more reactive. The reactive PTFE surface will create polar functional groups. Polar functional groups will make the surface bond on PTFE with water stronger so that the water droplets formed will be flatter and wider on the PTFE surface.



(a)



(b)



(c)

Figure 5. PTFE Surface During Optical Microscope Observation at 20x Magnification (a) Without Plasma Treatment, (b) Given Plasma Treatment with Oxygen Gas Flow Rate of 20 mL/min, (c) Given Plasma Treatment with Oxygen Gas Flow Rate of 50 mL/min

The surface of the PTFE sample exposed to plasma will be etched which causes the surface structure to change. This structural change can change the wettability of the sample. However, there are several factors that influence the uneven exposure of the plasma to the sample. One of them is the influence of the flow rate of oxygen gas given. A flow rate that is too high also causes the sample to stick to the reactor wall because the sample mass is very light. Thus, there are some samples that when treated are not evenly exposed to plasma. A high flow rate is not always good in plasma treatment. This is because it can cause the sample in it to scatter and stick to the walls of the plasma reactor, making the treatment uneven. At a higher flow rate, the plasma flame tends to be brighter because the more gas particles are introduced, the more the particles collide, so that the possibility of the PTFE sample being exposed to a maximum.

Conclusion

The Dielectric Barrier Discharge (DBD) plasma system with the addition of a lid on the top of the tube and the addition of a stirring spoon with additional modifications of the seal door brush is able to effectively provide treatment to the sample by rotating the sample in the reactor tube so that it is exposed to plasma evenly. Plasma treatment can improve the wettability of polytetrafluoroethylene (PTFE) as seen by the decrease in the contact angle and changes in the surface of the sample due to plasma exposure.

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