Characterization of negative corona plasma discharge reactor using point-to-plane electrode configuration in atmospheric pressure and its application in the treatment of woven natural silk

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Abstract. This research is aimed at obtaining the characteristics of negative corona plasma discharge with the presence and absence of woven natural silk and to figure out morphological alteration of woven natural silk by negative corona plasma discharge and water absorption characteristics of irradiated woven natural silk. The plasma reactor used employed multi point-to-plane electrode configuration using 100 electrodes. The reactor is connected to a high DC power supply with the point electrodes with spacing and the plane electrode varies from 0.6 to 3.0 cm. The silk sample has a dimension of (10 x 10) cm\textsuperscript{2}. Plasma irradiations were performed with a varied duration that ranged from 5 to 30 minutes. Sample testing was performed using SEM and water drop test. SEM testing results also show damages on the fabric surface compared to non-irradiated fabric. Meanwhile, the water drop test results reveal that optimum absorption for woven natural silk is at a distance of 2.4 cm for 30-minute irradiation. These settings resulted in an average absorption time of 10.28 seconds, which is way faster than absorption for non-irradiated fabric, at 48.88 seconds.

1. Introduction
Textile is produced from thread weaving or knitting in the form of a textile sheet, weave and knitwork \cite{1}. Indonesia has many featured industries including food, pharmacies, cosmetics, and health, as well as textiles. Textile belongs to the featured industry as it has an international competitive advantage. Among the weaknesses of our national textile industry is a dependency on imported raw material which still amounts to 90\%. This is due to a lack of mastery of the skills required to obtain proper raw material using relevant technology \cite{1}.

In the textile industry, the wet process is performed full circle, from preparation, dyeing, printing, and refinement\cite{2}. According to Nugraha (2019), the dyeing method using chemical substances still belongs to conventional methods. The conventional wet process requires a huge amount of water and energy, and it results in environmental pollution from the use of dangerous chemical substances. This is where the potential of plasma technology comes to play, with its efficient energy usage and less real waste produced.
Plasma is ionized gas and is known as the fourth state of matter after solid, liquid, and gas. The plasma phase is a mix of positively charged ions and negatively charged electrons with completely different nature to other gases. Plasma can be generated in the atmospheric condition in the form of corona discharge. Corona discharge is the discharge generated by the non-uniform strong electric field between two electrodes[4]. Corona discharge is generated in atmospheric pressure using low frequency and high voltage between a pair of electrodes[5]. In the point-to-plane electrode configuration, corona plasma discharge is generated from some point electrodes.

Generated corona plasma discharge can either be positive corona or negative corona, depending on the polarity of the voltage supplied. Positive corona plasma discharge can be generated by attaching positive polarity to point electrodes. Electrons moving from the anode to the cathode ionize gas atoms and molecules between the electrodes. Negative corona plasma discharge can be generated by attaching negative polarity to point electrodes. Ions flowing through the current zone are negatively charged ions formed in the air that contains electronegative molecule (O2), which easily captures electrons.

In 2000, Kaelani conducted research that modified the surface of polyester fabric using glow discharge plasma. Results from this research show that glow discharge plasma increases color intensity from dyeing, which saves colorants and provides a smooth effect on the fabric[6]. Similar research was carried out by Untung Prayudie and Eva Novarini in 2015. They also modified the surface of polyester fabric using corona discharge. Results from SEM imaging revealed the effects of etching that increase roughness on the fabric’s surface. Etching effect creates space for water molecules to fill in that in turn, increases absorption by polyester fabric[7]. In 2018, Muhlisin et al. also performed research investigating the effects of irradiation by negative corona plasma discharge on cotton and grey polyester, which revealed less absorption time for both cotton and grey polyester.

Plasma irradiation can also be applied for silk. In 2016, Dayioglu conducted research on silk dyeing using plasma effect [8]. In 2019, Nugraha et al. also carried out irradiation on silk. The discharge generated was in the form of positive corona in atmospheric conditions. Irradiated silk showed improvement in its hygroscopic properties [3].

This research attempts to irradiate woven natural silk produced by silkworm (Bombyx mori) with negative corona plasma discharge. The effects of the presence of woven natural silk against negative corona plasma discharge will also be examined. Other than those, the effects of negative corona discharge on woven natural silk itself will also be investigated.

2. Method
This research investigated negative corona plasma irradiation on woven natural silk in atmospheric conditions. Plasma discharge was generated using a point-to-plane electrode configuration connected to a high voltage DC power supply. Point electrodes served as the active electrode, while the plane electrode served as the passive electrode. Point electrodes were 100 in number and were distributed in a square, each separated 1.3 cm apart, whereas the plane electrode was a copper plate. Point electrodes were connected to the negative pole, while the plane electrode was connected to the positive pole of a high voltage DC power supply.

Prior to the irradiation of woven natural silk, the instrument was assembled as depicted in Figure 1. In order to measure voltage, the circuit was connected to a high voltage probe of model no: PD-28 and serial number 01605733, and the output of the probe was connected to a SANWA digital multimeter of type CD771. The high voltage probe was used to convert voltage value from kilovolt to volt. Meanwhile, in order to measure discharge current, a SANWA YX360TRF multimeter was used. It was connected in series to the negative pole of the source.

Once the circuit was assembled, the characterization of plasma discharge in atmospheric conditions ensued. The anticipated characteristic is a variation of generated voltage against generated current discharge. Discharge characterization was performed by varying the spacing between point and plane electrodes. Other than that, characterization was facilitated by the presence of a woven natural silk sample on the discharge plane.
Once characterization was finished, the next step was negative plasma irradiation treatment against a silk sample of (10 x 10) cm$^2$ dimension. This treatment involved varied treatment periods. Irradiated silk samples had to also undergo a water drop test. This treatment was performed by dropping 1 ml of water on to the silk and measured the water absorption time. Afterward, the same samples were tested using SEM (Scanning Electron Microscope) to obtain relevant microscopic images.

![Figure 1. The layout of the research instrument](image)

### 3. Result and discussion

Current and voltage characterization was conducted with the sample at electrode spacing from 0.6 cm to 3.0 cm, with 0.3 cm increment. Current flows from the negative electrode to the positive electrode. The current surcharge in the plasma formation chamber is directly proportional to the voltage supplied to the electrodes.

Figure 2 shows I-V characterization without a sample. At the shortest spacing of 0.6 cm at 1.345 kV, the result is a current of 0.001 mA. At a voltage of 7.09 kV, the generated current reaches 5 mA. The current for voltage below 1.345 kV is not detectable by analog multimeter as it has not flowed to the electrodes. At a spacing of 1.5 cm at 1.079 kV, the result is current of 0.001 mA, and as the voltage reaches 17.36 kV, the generated current reaches 4 mA. At a spacing of 2.4 cm at a minimum voltage of 0.986 kV, the result is current of 0.001 mA, and as the voltage reaches a maximum of 23.34 kV, the generated current reaches 1.5 mA.
A research carried out by Damayanti [9] revealed that the characteristics of current (I) as a function of voltage (V) show that greater voltage results in the greater current. At a spacing of 0.6 cm to 1.2 cm, the plasma generated still appears in faded purple. It is also the same for the spacing of 2.7 cm to 3.0, which results in a fading plasma color. Optimum spacing for plasma generation is between 1.5 cm and 2.4 cm. At this distance, the plasma is evenly generated as indicated by the purplish color at each electrode point. Results of visual observation for characteristic relationships between current (I) and voltage (V) from point-to-plane electrode configuration corona plasma discharge without sample are shown in Figure 3.

Figure 3. Corona discharge from without sample in multi point-to-plane electrode configuration at 0.9 cm spacing

Current and voltage characterization was conducted with the sample at electrode spacing from 0.6 cm to 3.0 cm, with 0.3 cm increment. Results of current and voltage characterization with the sample are shown in Figure 4.
Figure 4. Current-voltage characteristics with sample

Figure 4 shows I-V characterization with the sample. At a spacing of 0.6 cm, a minimum voltage of 0.796 kV generates a current of 0.001 mA, while at a maximum voltage of 5.29 kV, it generates current at 4.5 mA. At a spacing of 1.5 cm, a minimum voltage of 0.847 kV generates a current of 0.001 mA, whereas at maximum voltage of 12.69 kV, it generates current at 5 mA. At a spacing of 2.4 cm, current of 0.001 mA is generated from a minimum voltage of 0.859 kV, while at a maximum voltage of 19.22 kV; it generates current at 1.5 mA. Those three spacing reveal surcharge in current and voltage with increasing electrode spacing. Results of visual observation for characteristic relationships between current (I) and voltage (V) from point-to-plane electrode configuration corona plasma discharge with the sample are shown in Figure 5. Figure 6. Graph showing characteristic current (I) and voltage (V); with and without sample.

Figure 5. Corona discharge form with the sample in multi point-to-plane electrode configuration at 0.9 cm spacing
In can be seen in Figure 7, that current increases significantly with increasing voltage supplied. This is also the case for corona discharge. Corona discharge is generated at 3.211 kV without sample, and at 3.064 kV with the sample. During characterization with the sample, generated current and voltage tend to be higher, whereas during characterization without sample, generated current and voltage tend to be lower. This is in line with the findings by Damayanti [9] which mentioned that characterization with sample shows greater current and voltage than without sample. Other than characterization results, values of the mean charge carrier in the reactor as a function of electrode spacing were also obtained, as shown in Figure 7.

**Figure 6.** Current-voltage characteristics with and without sample

**Figure 7.** Mean mobility of charge carrier in a plasma reactor as a function of electrode spacing

Figure 7 shows that mobility for both with and without sample decreases with increasing electrode spacing. This is because the greater distance between electrodes slows down the movement of electrons, ions, and radical species. Figure 7 shows that the lowest mobility without a sample is at 0.000011 cm²/V.s with a spacing of 3.0 cm, while the highest mobility is at 0.000694 cm²/V.s with a spacing of
0.6 cm. Meanwhile, the lowest mobility with the sample is at 0.000033 cm$^2$/V.s with a spacing of 3.0 cm, whereas the highest mobility is at 0.000824 cm$^2$/V.s with a spacing of 0.6 cm.

Scanning Electron Microscope imaging in this research is aimed at understanding morphological changes on the surface of woven natural silk. Figure 8. shows the results of SEM imaging on the woven natural silk with and without irradiation of negative corona plasma discharge. Irradiations were conducted for 5, 15 and 30 minutes at an electrode spacing of 2.4 cm.

Figure 8. Results of SEM imaging at 1000 magnification at 2.4 cm spacing (a) for 0 minutes without plasma irradiation, (b) for 5 minutes of plasma irradiation, (c) for 15 minutes of plasma irradiation, and (d) for 30 minutes of plasma irradiation.

SEM testing shows that, as depicted in Figure 8 (a), the non-irradiated sample shows softer threads and that there is no damage observed. On the other hand, as depicted in Figure 4.7 (b-d), the irradiated sample reveals damage to the threads that make up the fabric. Damage comes in the form of blisters and etching on threads that are even greater and more extensive with longer irradiation. These damages are due to collisions of charge-carrying ions produced from the plasma process with the surface of the woven natural silk. These results are in line with those of research by Damayanti [9] on reduced weight polyester, which revealed that non-irradiated fabric has a softer surface, while the irradiated fabric has a coarser fabric.

The mechanical tear test in this research is aimed at knowing the strength of woven natural fabric both with and without irradiation with negative corona plasma discharge. Figure 9 shows the results of the mechanical tear test on woven natural silk at electrode spacing of 2.4 cm with negative corona plasma discharge irradiation of 5, 15, 30 minutes, and that of woven natural silk with 0-minute irradiation as control.
Figure 9. Results of mechanical testing on silk

Figure 9 shows an increase and decrease in pressure along with the plasma irradiation period. It takes a pressure of 778.7 kPa to tear the control fabric that does not undergo irradiation. In the meantime, a sample with 5-minute irradiation takes 0.8 kPa more to tear, while samples irradiated for 15 and 30 minutes require 29.3 kPa and 71.4 kPa less pressure to tear, respectively.

Increasing pressure required to tear the fabric after 5-minute irradiation proves the effect of plasma treatment. This is in line with the result from Damayanti [9], which revealed that pressure increase takes place at initial plasma treatment. Plasma irradiation increases bond within the fabric early in the treatment, and hence, stronger fabric. However, longer irradiation period causes weaker fabric that in turn, allows for less pressure to tear it. This is a result blister on the fabric’s surface that weakens bonds within the fabric. A similar result is also obtained by Dayioglu [8], which found less silk strength, compared to samples without irradiation.

Therefore, it is found that the highest pressure is for 5-minute irradiation, at 779.5 kPa. Figure 9 shows that longer irradiation with negative corona plasma discharge results in less pressure required to tear the fabric, as its strength is already weakened.

In the meantime, the water drop test in this research is aimed at figuring out the rate of water absorption by the fabric. Figure 10 shows the results of a water drop test on woven natural silk with a varied electrode spacing of 0.6, 1.5, 2.4, and 3.0 cm. Plasma irradiation was conducted for 5, 10, 15, 20, 25, and 30 minutes, and with the help of a sample with 0 minutes (without irradiation) as control.
Figure 10. Results of water drop testing on silk

Figure 10. shows slower absorption time with increasing electrode spacing and plasma irradiation period. At a distance of 1.5 cm, the sample irradiated for 5 minutes has a 46.94 seconds absorption rate, while the sample irradiated for 30 minutes has a 10.91 seconds absorption rate. The water absorption rate lowers as electrode spacing increases. At a distance of 2.4 cm, the sample irradiated for 5 minutes has a ~46.94 seconds absorption rate, while the sample irradiated for 30 minutes has a 10.28 seconds absorption rate.

This is in contrast to the non-irradiated sample which absorbs water for a lengthy 48.88 seconds. In relation to SEM imaging, non-irradiated samples do not suffer any damage to their surface. Etching effect creates roughness on the fabric’s surface that in turn, provides space for water molecules to fill in, which increases absorption by polyester fabric [7]. This is proven as testing with water dropped vertically results in the slow absorption process. A study by Prayudie [7] revealed that irradiated polyester fabric has a greater absorption rate, compared with its non-irradiated counterpart.

It can be seen in Figure 10 that longer irradiation on woven natural silk results in a greater water absorption rate. It is found in this research that the speediest absorption rate is at 10.28 seconds that results from 30-minute irradiation at an electrode spacing of 2.4 cm.
4. Conclusion

Results from this research lead to the following conclusions that the current and voltage characterization from negative corona plasma discharge shows that greater voltage supplied results in greater discharge current, either with or without woven natural silk. The presence of fabric in the negative corona plasma discharge chamber increases discharge mobility. Other than that, further electrode spacing affects the mobility of charged particles in the plasma chamber. Irradiation with negative corona plasma discharge alters the surface of woven natural silk. Irradiation woven natural silk has increased absorption. Moreover, the fabric is also stronger for a short irradiation time. However, longer irradiation period results in reduced fabric strength.

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