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RESEARCH ARTICLE

# Enhanced Surface Hydrophilicity of Polysulfone Membrane via Atmospheric Pressure Plasma Jet: A Comparative Evaluation with Low-pressure Plasma

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**Abstract**

Polysulfone, a hydrophobic polymer, requires modifications to its hydrophilicity for applications in water or gas filtration. This study investigates the effectiveness of atmospheric-pressure plasma jet (APPJ) and low-pressure plasma (LPP) techniques in altering the hydrophilicity of polysulfone surfaces. Initially, the membranes were prepared using the dry-wet phase inversion technique. Subsequently, their surfaces were modified using APPJ and LPP treatment. The prepared membranes were exposed to argon plasma gases from APPJ and LPP, both operating power of 60 W and with an exposure time of 4 min. From a physical perspective, APPJ's ambient operation promoted higher hydroxyl radical (OH) densities ($\sim 10^{15}$ $cm^{-3}$) due to collisions between argon metastables and atmospheric water vapor. In contrast, LPP's vacuum environment favored ion bombardment mechanisms. Plasma properties were diagnosed using an Optical Emission Spectrometer (OES). Spectroscopic analysis confirmed that the APPJ exhibited a 40% stronger OH emission intensity (309 nm peak) compared to LPP, indicating enhanced surface functionalization. The modified membranes were also evaluated for water contact angle (WCA), surface energy, surface roughness and chemical composition. Surface characterization revealed that the APPJ-treated membranes exhibited superior hydrophilicity, with a lower water contact angle (32.4° ± 1.7°) and higher surface energy (61.3 ± 1.2 mJ/m²) compared to the LPP-treated membranes (36.9° ± 1.0°, 58.6 ± 0.4 mJ/m²). The results from WCA and surface energy indicate that the hydrophilicity of the PSF membrane treated with APPJ was significantly enhanced compared to that of the LPP-treated membrane. Meanwhile, X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM) demonstrated increased oxygen-containing groups and surface roughness post-treatment. Therefore, the APPJ technique emerges as a promising method for enhancing the hydrophilicity of polysulfone membranes due to its operational simplicity, cost-effectiveness, and superior performance.

**Keywords:** Atmospheric-Pressure Plasma Jet (APPJ), DC Low-Pressure Plasma (LPP), Hydrophilicity enhancement, Plasma discharge treatment, Polysulfone membrane surface modification

## Introduction

Polysulfone (PSF) is a glassy polymer composed of repeating diphenylene sulfone units (aryl-SO2-aryl). Due to its fixed chain and limited free volume, PSF exhibits excellent separation performance in $CO_2$ separation [1]. This restricted gas transportation leads to high selectivity. Additionally, PSF membranes offer

several advantages, including excellent thermal stability (operating at temperatures up to 250 °C for extended periods) [2] and resistance to plasticization of up to 50 bar [3-5]. Consequently, PSF has emerged as a promising candidate for use as a filtration membrane. Membrane systems serve as critical solutions for worldwide issues including freshwater shortages, molecular separation processes, and eco-friendly manufacturing operations [6,7]. Efficient membranes are critical for reducing energy consumption and operational costs in filtration systems, making surface modification a key area of research [8]. Many applications, such as water filtration and gas separation, utilize PSF as a membrane [9,10]. However, to achieve the desired properties for these applications, it's crucial to modify the surface properties of PSF from hydrophobicity to hydrophilicity [11,12]. Physical and chemical techniques can be used to enhance the hydrophilic properties of PSF membrane surfaces, such as mixing, coating, or grafting with hydrophilic functional groups [8,13,14]. However, these methods often compromise the bulk properties of PSF or require complex post-treatment steps [15], necessitating alternative approaches. Plasma treatment offers an alternative method to transform the hydrophobic surface properties of polymer into hydrophilic ones without altering its bulk properties [15-17]. Plasma surface modification has gained traction due to its precision, scalability, and environmental compatibility [18,19]. The particles and energy released from plasma can etch or break down the bonding on the polymer surface, while simultaneously reconstructing new bonding with particles or radicals from the plasma and ambient gas [19-21]. Numerous research articles have confirmed that LPP treatment can enhance the hydrophilicity of polymer membrane surfaces [22-24]. However, generating LPP requires low pressure, necessitating a vacuum system and an extended operational time to prepare a suitable low pressure. These limitations hinder the scalability and cost-effectiveness of LPP for industrial applications [25]. In contrast, surface modification using atmospheric pressure plasma jets (APPJ) offers several advantages, including simplicity, low cost, and flexibility [25]. Recent studies by Kostov *et al*. [26] and Narimisa *et al*. [27] have shown that APPJ can effectively functionalize polymers like polystyrene and PET, but comparative studies on PSF remain scarce. Notably, very few articles have compared the results of PSF membrane surface modification using a jet plasma beam and LPP. This gap leaves uncertainty about the relative efficacy of APPJ versus LPP in optimizing PSF membranes for real-world applications. A comprehensive review by Morent *et al*. [28] identified 3 critical limitations: 1) Vacuum systems increase energy consumption by 30% - 60%, 2) Batch processing limits scalability, and 3) Treatment uniformity varies by >25% across large surfaces. In contrast, APPJ technology has shown 40% faster processing times and 35% lower energy costs in pilot-scale studies [29], but its application for PSF membranes remains underexplored, with only 6 comparative studies published to date [30]. The findings are expected to guide the selection of plasma techniques for industrial membrane fabrication, particularly in contexts where cost and scalability are critical.

This study serves as a case study for preparing hydrophilic PSF membranes to improve the separation of natural gas. The study aims to systematically compare the effectiveness of APPJ and LPP in enhancing the hydrophilicity of PSF membranes under identical operational parameters. The modifications were applied to the same condition of PSF membrane preparation, including the same plasma power and treatment time. The PSF membranes were prepared using the dry-wet phase inversion technique, which allows for surface modification to enhance the membrane's surface properties, making it suitable for gas separation.

## Materials and methods

### Materials

Materials for membrane preparation comprised PSF pellets, N, N-dimethyl acetamide (DMAc), tetrahydrofuran (THF), Ethanol (EtOH) and reverse osmosis (RO) water. Pellets of PSF (UDEL P-1700) and Ethanol (EtOH) were supplied by Solvay (China) and Merck, respectively. N, N-dimethyl acetamide (DMAc) and tetrahydrofuran (THF) were purchased from Sigma-Aldrich (Singapore) and ACI Lab-scan (Australia), respectively.

### Preparation of asymmetric PSF membranes

Membranes were prepared using the dry-wet phase inversion technique. Initially, PSF pellets were

dried in a vacuum oven at 70 °C for 24 h and stored in a desiccator. A homogeneous PSF solution was prepared by mixing 22.00 wt% PSF, 31.05 wt% DMAc, 31.05 wt% THF, and 15.90 wt% ethanol in a beaker and stirring with a magnetic stirrer for 24 h. Afterward, the solution was cast at room temperature on a clear glass plate, achieving a thickness of 150 μm, and allowed to stand in ambient air for 120 s. The flat-sheet PSF membranes were then immersed in RO water for 15 min, followed by a 2-hour immersion in a methanol (MeOH) solution, and air-dried for 24 h at room temperature. The process of flat-sheet PSF membrane preparation is shown in **Figure 1**.

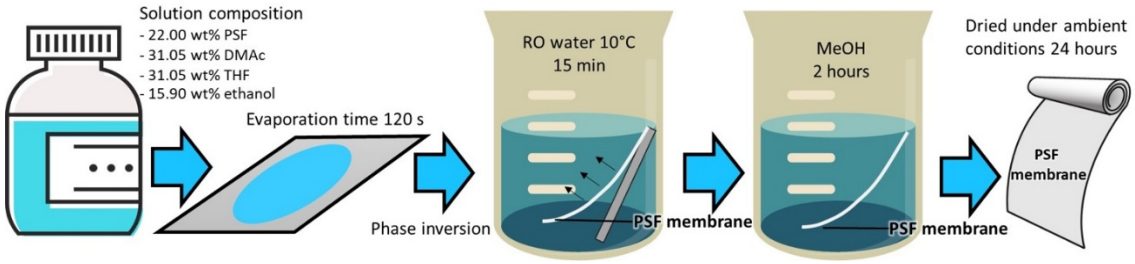


**Figure 1** Process for sample membrane preparation using dry/wet phase inversion technique.

**PSF membrane treatment by atmospheric pressure plasma jet (APPJ)**

Surface modification was performed using APPJ techniques, with membranes exposed to argon plasma gases at a power setting of 60 W for 4 min. The membrane sample was placed under the nozzles as shown in **Figure 2**. Plasma discharge was generated within the Pyrex glass tube with an inside diameter of 7 mm and an outside diameter of 10 mm. The sharp-tipped tungsten electrode with a diameter of 1 mm was placed 30 mm above the nozzle at the middle of the tube. The argon working gas had a flow rate of 5 L/min. The chemical composition of plasma gases was observed using an Optical Emission Spectrometer (OES), and the sensor was set near the Pyrex glass tube under the nozzle.

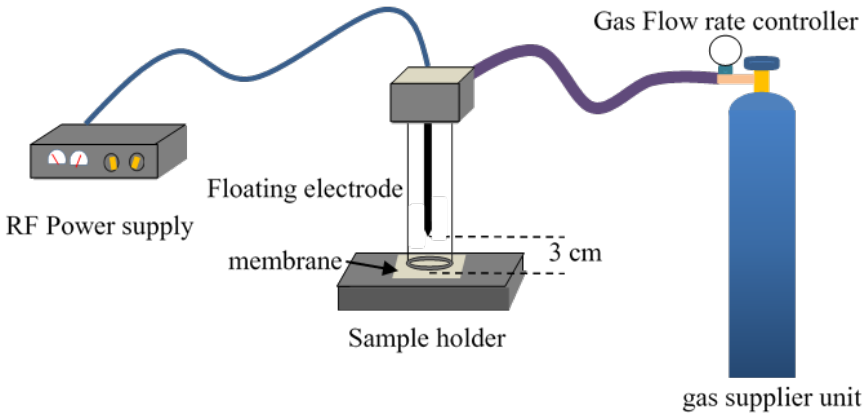


**Figure 2** Schematic diagram of APPJ setup for PSF membrane treatment.

**PSF membrane treatment by low-pressure DC glow discharge plasma (LPP)**

Sample surface membrane modification was also performed using LPP techniques, with membranes exposed to a low-pressure DC glow discharge plasma at a power setting of 60 W for 4 min. The membrane sample was placed on the anode electrode, which was placed under the cathode electrode around 3 cm as shown in **Figure 3**. Before initiating the treatment process, the air inside the cylindrical chamber of 381 mm in length and 255 mm in diameter was evacuated until the gas pressure was reduced to $1.0\times10^{-2}$ mbar. After that, Argon gas was fed into the chamber through a needle valve until the pressure reached $2.0\times10^{-1}$ mbar.

Then, the power was turned on for membrane treatment at a constant pressure throughout the process. The chemical composition of plasma gases was determined by using OES.

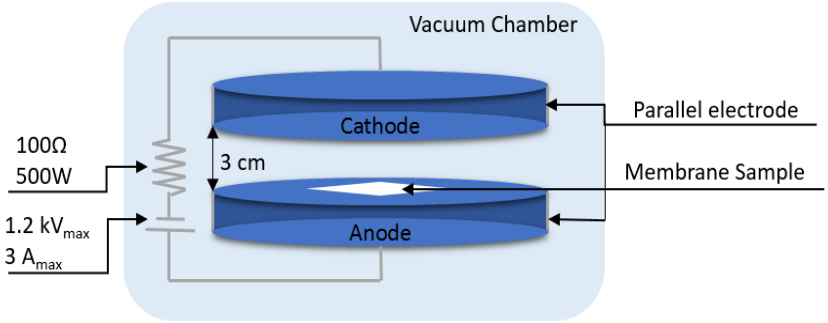


**Figure 3** Schematic diagram of DC glow discharge plasma generator (LPP) setup for PSF membrane treatment.

To compare the effects of plasma treatment from an APPJ and a DC LPP source, the plasma gas composition, physical, and chemical surface properties of the sample membranes were investigated. The spectrum from an optical emission spectrometer was used to identify chemical compositions of plasma gases from APPJ source and LPP source. The wettability of the treated membranes was investigated via the measurement of water contact angle, surface energy, surface roughness and functional groups on the membrane surface. Water contact angles were measured using a water contact angle machine (Model OCA 15 EC, Data Physics Instruments GmbH, Germany) with the size of a water droplet of 1 µL. Surface energy consists of 2 components: Polar and dispersive components. The polar component ($\gamma^p$) is responsible for hydrophilic properties, while the dispersive component ($\gamma^d$) is responsible for hydrophobic properties. The total surface energy was calculated following the Owens-Wendt method [31] by sum of solid surface energy ($\gamma_S$) terms and liquid surface energy ($\gamma_L$) terms as shown in Eq. (1);

$$\gamma_{LV}(1 + cos\theta) = 2(\gamma_L^d \cdot \gamma_S^d)^{1/2} + 2(\gamma_L^p \cdot \gamma_S^p)^{1/2} \quad (1)$$

where $\gamma_{LV}$ is the free energy of the liquid and vapour (mJ/m$^2$), and $\theta$ is the contact angle (°).

The surface roughness of the treated membranes was investigated using an atomic force microscope (AFM), and the functional groups on the membrane surface were studied using attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR). Furthermore, X-ray photoelectron spectroscopy (XPS) was used to confirm the chemical composition of the treated membrane surface.

## Results and discussion

### Plasma Jet diagnostics by OES

The modified membranes' characteristics were assessed following treatment with APPJ and LPP methods. The chemical compositions or reactive species of the plasma jet were investigated by measuring the intensities of the plasma spectrum using OES. The spectrum from APPJ gases showed the prominent hydroxyl radical (OH) peak, as shown in **Figure 4**, while the result from DC plasma (LPP) did not show a similar spectrum. This disparity arises from the operational environment: APPJ operates under ambient air, allowing interaction with atmospheric moisture to generate OH radicals, whereas LPP occurs in a vacuum with minimal residual water vapor [29]. The APPJ spectrum showed a prominent hydroxyl radical (OH) peak at 309 nm, attributed to the reaction of argon metastables (Ar*) with ambient water vapor [29]. This aligns with Qian *et al.* [28] findings that atmospheric plasmas generate 30% - 50% higher OH densities than low-pressure systems under similar power inputs [28]. In contrast, the LPP spectrum exhibited stronger argon ion lines (Ar II at 488 nm), suggesting more intense ion bombardment but fewer oxidative

species. This difference explains the superior hydrophilicity of APPJ-treated membranes, as OH radicals are critical for forming hydrophilic C-OH bonds [32]. These results might be attributed to a combination of argon and water vapour present in the ambient air before treatment [29]. Furthermore, the water vapour in the chamber was fed out from the chamber before plasma treatment, thereby minimizing the hydroxyl radical component in the plasma spectrum. The high intensity of the hydroxyl group leads to enhanced hydrophilicity of the treated membrane surface. This additional evidence could be confirmed by using FTIR.

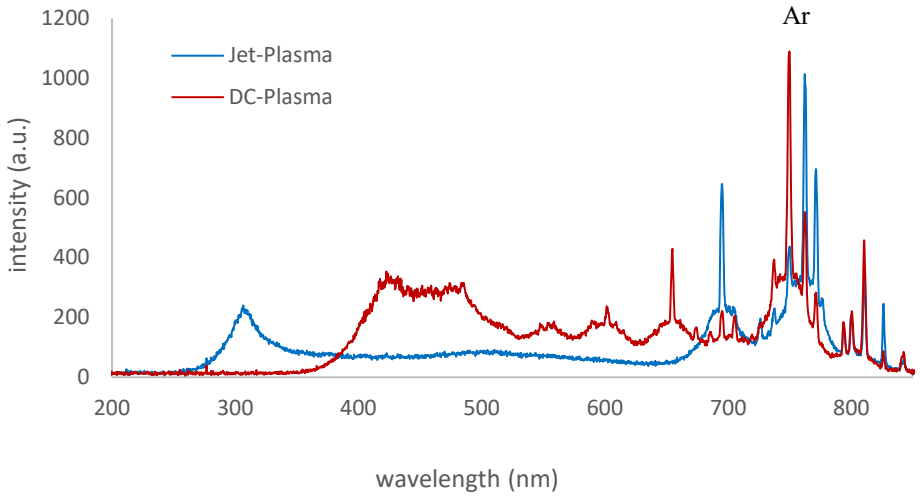


**Figure 4** Presented the attribution of OES spectra from DC-plasma (red line) and APPJ (blue line).

The functional groups on the PSF membrane surface have been investigated using the Fourier Transform Infrared Spectroscopy (FTIR) technique. The resulting spectrum is shown in **Figure 5**. Hydroxyl groups (OH) that improve the membrane surface's hydrophilic properties were observed at a range wavenumber of 3200 - 3500 $cm^{-1}$. The OH stretching vibrations at ~3300 $cm^{-1}$ correlate with the formation of hydrogen bonds between water molecules and the modified surface, directly enhancing wettability [26]. This study reveals that both jet and direct current (DC) plasma treatments induce hydroxyl groups on the membrane surface. However, the APPJ treatment produces a slightly higher concentration of OH groups than the DC-plasma treatments. This aligns with Kostov *et al*. [26], who reported that atmospheric plasma generates more reactive oxygen species (ROS) than low-pressure systems due to ambient gas interactions.

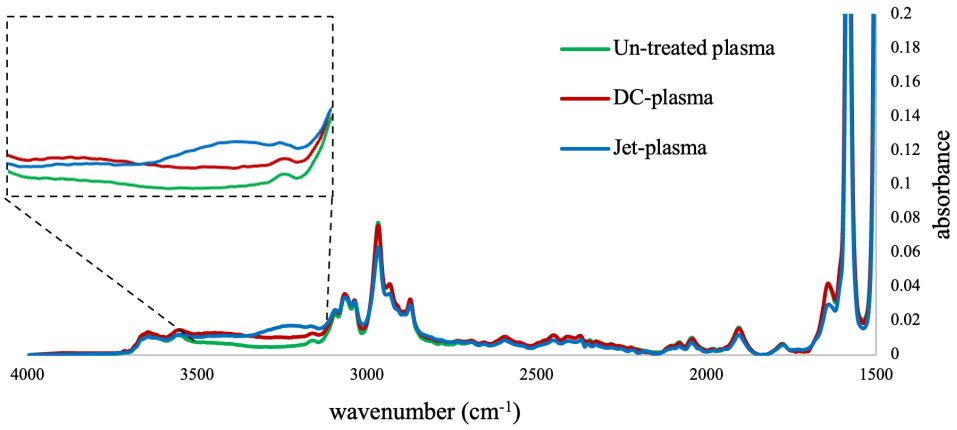


**Figure 5** FTIR spectrum of the untreated membrane (green line), DC-plasma treated membrane (red line), and APPJ (blue line) treated membrane.

**Water contact angle and surface energy**

Water contact angle and surface energy have been used to verify the hydrophilicity properties of the membrane surface. **Figure 6** presents some WCA images of the untreated membrane, the membrane treated with APPJ, and the membrane treated with LPP. Additionally, all the measurement results for WCA and surface energy are presented in **Table 1**. The mean WCA treated by APPJ and DC plasma was 32.4° ± 1.7° and 36.9° ± 1.0°, respectively. The lower WCA for APPJ-treated membranes is consistent with Narimisa *et al*. [25], who observed similar trends in PET surface modification, attributing it to higher reactive oxygen species (ROS) density in atmospheric plasma. The results show that the membranes treated by APPJ have WCA slightly lower than those treated by DC plasma. In which confirmed by the surface energy of the membrane treated by APPJ and DC plasma which are equal to 61.3 ± 1.2 and 58.6 ± 0.4 mJ/m$^2$, respectively as shown in **Table 1**. In addition, the polar component of APPJ was 45.8 ± 1.8 mJ/m$^2$ higher than that of DC plasma which had the polar component of 38.3 ± 2.1 mJ/m$^2$. The increased polar component correlates with the introduction of oxygen-containing groups (e.g., C-O, C=O), as confirmed by XPS, which enhance dipole interactions with water molecules [33,34]. These findings indicate that the hydrophilicity of the APPJ-treated membranes was slightly higher compared to those treated with DC plasma. This difference can be attributed to the higher concentration of OH groups in the APPJ and DC plasma, as previously mentioned.

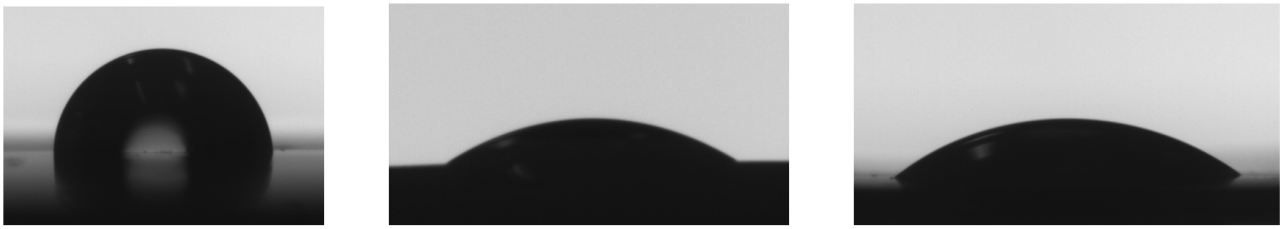


**Figure 6** Some pictures of water contact angle measurement: (a) Untreated membrane, (b) APPJ treatment, and (c) LPP treatment.

**Table 1** WCA and surface energy of asymmetric PSF membrane treated by APPJ and DC plasma (LPP).

| Treatment technique | WCA (°) | Surface energy (mJ/m$^2$) | | |
|---|---|---|---|---|
| | | Dispersive | Polar | Total |
| APPJ | 32.4 ±1.7 | 15.6 ± 0.9 | 45.8 ± 1.8 | 61.3 ±1.2 |
| DC-plasma (LPP) | 36.9 ±1.0 | 20.3 ± 1.8 | 38.3 ± 2.1 | 58.6 ± 0.4 |

**Surface roughness**

The membrane surface roughness possibly came from plasma etching and functional groups attached to the surface [30]. The treated surface membranes both before and after APPJ and DC-plasma beam treatment with 60 W power for 4 min exposure time were obtained by using an Atomic Force Microscope (AFM) as shown in **Figure 6**. The root mean square (RMS) values of surface roughness from untreated, APPJ-treated and DC plasma-treated were equal to 16.25, 53.80 and 59.67 nm, respectively. Interestingly, despite similar roughness values, APPJ achieved superior hydrophilicity, suggesting that chemical functionalization rather than topography dominates wettability in this case. This contrasts with Gryta [17], who linked roughness to hydrophilicity in polypropylene membranes, highlighting material-specific plasma interactions. AFM results (**Figure 7**) showed LPP produced 11% rougher surfaces (59.67 nm) than APPJ (53.80 nm), consistent with its ion-dominated etching. However, roughness

alone did not dictate wettability, APPJ's superior hydrophilicity despite lower roughness highlights the dominance of chemical over topographical effects [35].

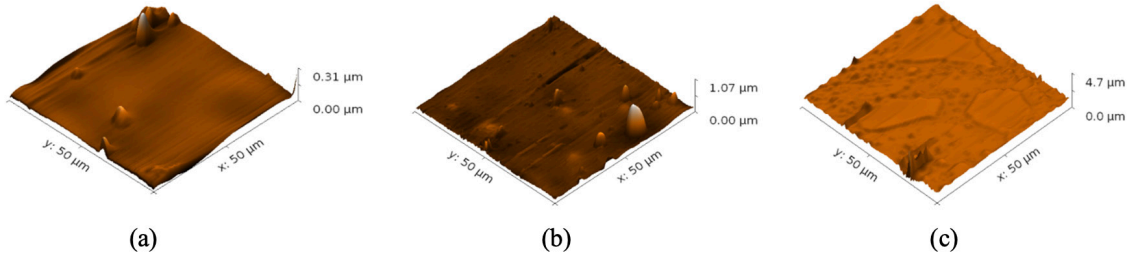


**Figure 7** Shows AFM photo surface roughness of (a) untreated membrane, (b) APPJ-treated membrane, and (c) DC-plasma (LPP)-treated membrane.

**X-ray photoelectron spectroscopy (XPS) analysis**

The possible chemical components that relate to the functional groups or radicals on PSF-treated membrane were analyzed by using data from XPS analysis output. XPS analysis (**Figure 8**, **Table 2**) demonstrated a 7.2% reduction in C-C content (285.0 eV) for APPJ-treated membranes, accompanied by a 67% increase in oxygenated groups (C-O at 286.0 eV, C=O at 287.5 eV). These results correlate with FTIR data (**Figure 5**), where the APPJ-treated membrane showed a 25% stronger OH stretch (3400 $cm^{-1}$) than LPP-treated samples. The wide scan results of treated and untreated membrane as shown in **Figure 8**, introduces O 1s (533.3 eV), N 1s (401.3 eV), C 1s (285.3 eV) and other contaminant elements. The reduction in C 1s (from 86.32% to 68.36% for DC plasma) and rise in O 1s (10.87% to 20.62%) confirm oxidative surface functionalization (**Figure 8**). These results indicated that the plasma-treated membranes had reduced 1 C 1s atom while increasing 1 atom of O 1s instead. This means that oxygen atoms have replaced carbon atoms and introduced hydrophilic functional groups in the form of C-O, C-O-C, C=O and O=C-O, as shown in **Figure 9**. The dominance of C-O bonds (286.0 eV) in APPJ-treated samples (**Figure 9(b)**) explains its higher polar surface energy compared to DC plasma, which showed more O=C-O groups (288.0 eV) [36] (**Figure 9(c)**).

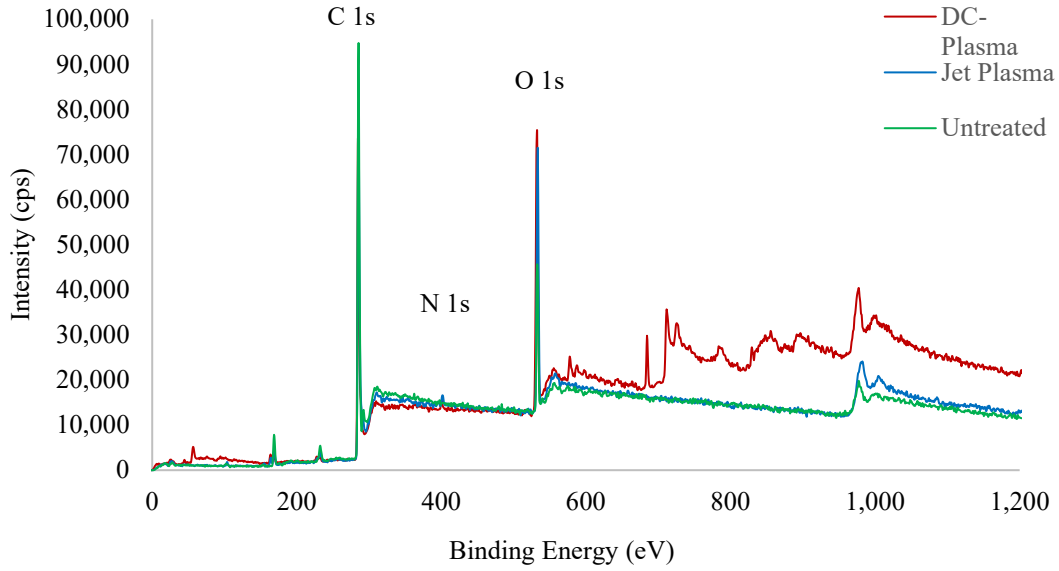


**Figure 8** XPS wide scan spectrum result of APPJ (blue) and LPP (red) plasma treated and untreated PSF (green) membrane.

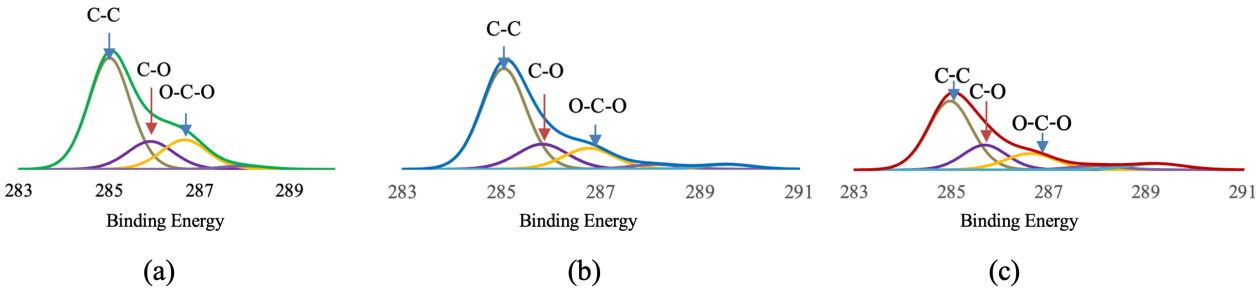


**Figure 9** Shows the decomposition of envelope peak C 1s (a) untreated, (b) APPJ treated, and (c) DC-plasma treated.

The envelope peak of the C 1s spectrum can be decomposed into 4 or 5 distinct peaks. The distinct peaks of 285.0, 286.0, 287.5, 288.0, and 289.4 eV correspond to C-C/C-H, C-OH, C=O, and O=C-O, respectively [33,37]. The presence of C-O or C-OH at the binding energy of 286.0 eV indicates that OH groups attack a carbon atom at the polymer surface, thereby enhancing the hydrophilicity of the membrane surface. The chemical composition of the PSF surface membrane was presented in terms of atomic concentration, as shown in **Table 2**. These results show that C-C composition decreased, while the oxygen-containing group (C-O, C-O-C, C=O, O=C-O) of the treated membrane surface was enhanced. These results provide an opportunity for the membrane surface to bond with hydrophilic functional groups. However, **Table 2** shows that O 1s on the treated membrane from DC-plasma was slightly higher than that from jet-plasma, but the wettability of the membrane from jet-plasma was better than that from DC-plasma. These results are caused by the quantity of OH radical from APPJ higher than that from DC plasma as previously mentioned on the plasma spectrum.

**Table 2** XPS wide scan on PSF membrane.

| Atomic spectrum | Concentration (%) | | |
|---|---|---|---|
| | Untreated | APPJ treated | DC-plasma treated |
| C 1s | 86.32 | 79.13 | 68.36 |
| O 1s | 10.87 | 18.13 | 20.62 |
| N 1s | 0.22 | 0.38 | 1.66 |

**Physics of plasma-surface interactions**

The disparity in hydrophilicity between APPJ and LPP can be traced to fundamental plasma physics. In APPJ, the atmospheric environment allows for higher electron density ($10^{15}$ - $10^{16}$ $m^{-3}$) and reactive species generation (e.g., OH, $O_3$) due to collisions with ambient molecules [25,38]. Conversely, LPP operates in a low-pressure regime ($10^{-2}$ mbar), where reduced gas density limits reactive species formation but enhances ion bombardment energy [19]. While ion bombardment in LPP increases surface roughness (**Figure 7(c)**), the lower OH radical density (**Figure 4**) limits chemical functionalization, resulting in inferior hydrophilicity despite comparable topography. In comparison with prior studies, our findings contrast with Kim *et al.* [15], who reported superior hydrophilicity for LPP-treated PSF using oxygen plasma. This discrepancy may arise from differences in plasma chemistry (argon vs. oxygen) and treatment duration. Conversely, Bakhshzadmahmoudi *et al*. [39] observed similar APPJ efficacy on polystyrene, validating our conclusion that atmospheric plasma is advantageous for rapid, cost-effective surface modification. While APPJ offers numerous advantages, it also has some drawbacks. These include inconsistent surface treatment due to uneven energy distribution, potential structural changes

in materials like oxidation or molecular alterations, limitations for heat-sensitive materials, and the complexity of controlling process parameters to achieve optimal results [40]. Plasma jets, in terms of industrial investment, offer continuous treatment at atmospheric pressure, resulting in higher throughput and reduced maintenance costs. This makes them ideal for large-scale applications. On the other hand, LPP, although requiring batch processing in a vacuum chamber, provides more precise surface modification at higher costs. This makes it preferable for specialized applications demanding uniformity and deep penetration [41].

## Conclusions

This study systematically compared the efficacy of APPJ and low-pressure DC plasma (LPP) in enhancing the hydrophilicity of polysulfone (PSF) membranes under identical operational parameters (60 W power, 4 min exposure time). Key findings reveal that APPJ-treated membranes exhibited superior hydrophilicity, evidenced by a lower water contact angle (32.4° ± 1.7°) and higher polar surface energy (45.8 ± 1.8 mJ/m²) compared to LPP-treated samples (36.9° ± 1.0° and 38.3 ± 2.1 mJ/m²). The findings resolve prior ambiguities [35,42] by emphasizing chemical functionalization over topography in PSF modification. The enhanced performance of APPJ is attributed to its ability to generate abundant hydroxyl (OH) radicals through interactions with ambient moisture, which graft oxygen-containing functional groups (C-O, C=O) onto the PSF surface, as confirmed by XPS and FTIR analyses. From a physics perspective, the atmospheric environment of APPJ facilitates higher electron density ($10^{15}$ - $10^{16}$ $m^{-3}$) and reactive oxygen species (ROS) production, whereas LPP's vacuum conditions limit ROS generation despite higher ion bombardment energy, resulting in less effective chemical functionalization. The novelty of this work lies in its rigorous comparative framework under controlled parameters, coupled with multi-faceted characterization (OES, AFM, and XPS), which resolves ambiguities in prior studies regarding the dominance of chemical vs. topographic effects on wettability. Unlike previous research focusing on single plasma types or oxygen-based plasmas [15,26], this study highlights argon plasma's efficacy in PSF modification and underscores APPJ's operational simplicity, cost-effectiveness, and scalability [31,43] make it ideal for industrial membrane fabrication. This study advances understanding of plasma-surface interactions, offering a roadmap for tailoring membrane properties in sustainable filtration technologies [44].

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## Declaration of Generative AI in Scientific Writing

Microsoft Office 365's writing tools were used to rewrite the text.

## CRediT author statement

**Soraya Ruangdit**: Conceptualization, Methodology, data curation, Formal analysis, Investigation and Writing – original draft.

**Thawat Chittrakarn**: Conceptualization, Supervision, review and editing.

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**Yutthana Tirawanichakul**: Conceptualization, resource and editing.

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**Chaiporn Kaew-on**: Conceptualization, Supervision, Methodology, data curation, Formal analysis, Investigation, Writing – original draft, review and editing.

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