

# COLD PLASMA GAS TYPE TREATMENT ON SURFACE CHARACTERISTIC OF POLY (ACRYLIC ACID-CO-METHYL METHACRYLATE) HYDROGEL

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## ABSTRACT

The objective of this study was to investigate the impact of cold plasma treatment on the surface characteristics of hydrogel films made from poly (acrylic acid-co-methyl methacrylate), these films were carried out by using four different gases, which are O<sub>2</sub>, N<sub>2</sub>, Co<sub>2</sub>, and untreated. Surface properties have been evaluated using photoelectron spectra (XPS), contact angles, attenuated total reflection (ATR), and scanning electron microscopy (SEM). The SEM images showed morphological changes, while the XPS results revealed the chemical composition of the surface treated with argon plasma. Consequently, the contact angle measurements of the hydrogel to water indicated a notable reduction in surface free energy after the argon plasma treatment. The surface free energy exhibited a positive correlation with the level of argon plasma treatment using various gases. This rise may be mostly attributed to the augmentation of polar constituents. The presence of oxygen-containing functional groups on the surface which was confirmed by XPS data had a crucial role in enhancing the hydrophilic characteristics of the hydrogel during the plasma oxidation process.



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## 1. INTRODUCTION

Cold plasma hydrogel superwetting surfaces involve the combination of cold plasma treatment, hydrogel materials, and superwetting characteristics to create surfaces with unique properties without significantly changing their bulk characteristics by introducing various functional groups and altering the surface energy of materials (Kyung et al., 2013; Begam et al., 2004). Polymer hydrogels have indeed proven to be highly

suitable materials for a wide range of demanding applications due to their unique properties (Bashir et al., 2020; Tyliczszak et al., 2009). Three-dimensional hydrophilic polymeric networks known as hydrogels can absorb and retain large amounts of water or biological fluids (Katime et al., 2005). Poly (Acrylic acid-co-methyl methacrylate) hydrogel P(AA-MMA) is a copolymer consisting of acrylic acid and methyl methacrylate, which forms hydrophilic polymer chains that create three-dimensional networks. These networks can absorb and

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hold water (Bialik-Waś et al., 2015; Leadley & Watts, 1997). The goal of the treatment is to preserve important characteristics of the immobilised polymer including the mobility and swelling of polymer chains as well as the presence of functional groups (Geng et al., 2013; Bialik-Waś & Pielichowski, 2018; Moritz et al., 2020). Treatment in the presence of different gases such as oxygen, nitrogen, and carbon dioxide leads to an increase in the number of polar functional groups on the surface oxygen plasma treatment can introduce oxygen-containing functional groups such as hydroxyl (-OH) and carbonyl (C=O) groups onto the surface of the hydrogel (Inphonlek et al., 2023; Cui et al., 2020). Nitrogen plasma treatment may introduce nitrogen-containing functional groups, such as amino (-NH<sub>2</sub>) or nitrile (-CN) groups, depending on the specific conditions (Seddiki & Aliouche, 2013). These groups can also contribute to increased surface polarity. CO<sub>2</sub> plasma treatment can contribute to the introduction of carboxyl (-COOH) groups on the surface. Carboxyl groups are polar and can significantly increase the hydrophilicity of the surface (Begam et al., 2004; Micutz et al., 2020). Cold plasmas, or non-thermal plasmas, are a distinct kind of matter that may be found at low temperatures. Thermal plasmas, in contrast to classic high-temperature plasmas, are often created at very elevated temperatures (Nehra et al., 2008; Boulos, 1991). Cold plasmas can be generated at or near ambient temperature this attribute renders them especially advantageous for a variety of applications in disciplines including environmental remediation, medicine, materials science, and surface modification (Domonkos et al., 2021). Atmospheric pressure plasmas, electrical discharges, radiofrequency fields, and microwave stimulation are some of the ways that cold plasmas may be produced. By supplying energy to the gas, these methods enable the electrons to achieve sufficient energy to escape from their parent atoms or molecules, this process leads to the creation of ions and electrons inside the plasma (Conrads & Schmidt, 2000). Within a cold plasma, only a small portion of the particles possess high levels of energy, while the majority maintain temperatures similar to that of the surrounding environment this unique distribution of energy is achieved by introducing an electric field to a gas or plasma medium, resulting in the separation of charges and the formation of a plasma with a diverse range of electron energies (Nehra et al., 2008). Physicists have observed that cold plasmas possess unique characteristics that set them apart from thermal plasmas. One notable difference is the significant disparity between the electron temperature and the gas temperature. While the ions maintain a lower temperature, the electrons reach much higher temperatures. This discrepancy arises due to the low collision rates between the electrons and the heavier, slower ions (Conrads & Schmidt, 2000). Additionally, cold plasmas tend to have a lower density compared to thermal plasmas, resulting in a more diffuse nature. Another notable feature of cold plasmas is their lack of thermodynamic equilibrium. This results in the electron temperature often being much higher than the

gas temperature, creating a state of non-equilibrium. With a high electron temperature, a wide range of chemical reactions can take place, resulting in the formation of reactive species like radicals, ions, and excited molecules (Domonkos et al., 2021; Bruggeman et al., 2014).

Surface properties encompass the various qualities and actions of a material's outermost layer as it engages with its environment. These properties are incredibly important in a wide range of scientific, engineering, and industrial applications, while it's impossible to list all of the surface properties due to their vast variety, i can give you a general overview of some commonly observed ones (Gardner et al., 2008). Surface roughness is the term used to describe the small irregularities and deviations on the surface of a material at a microscopic level. These imperfections have an impact on various surface interactions such as friction, adhesion, and optical properties (Bhushan & Ko, 2003). Surface roughness can be measured and characterized using techniques like profilometry and atomic force microscopy (AFM). Surface energy refers to the additional energy found on the surface of a material in comparison to its interior. This energy has a significant impact on wetting behavior, adhesion, and surface tension. Measuring contact angles of liquids on the surface allows for the quantification of surface energy. Surface tension is the force that occurs along the surface of a liquid due to the cohesive forces between its molecules. Understanding the behaviour of liquids at interfaces is crucial for studying wetting, spreading, and capillary action (Tholt et al., 2006; Israelachvili, 2011; Adamson & Gast, 1967). Understanding surface charge is crucial for studying the effects of electrical charges on the surface of a material or particle, it plays a significant role in influencing electrostatic interactions, adsorption phenomena, and colloidal stability, the surface charge can have a positive or negative value, and its strength determines the intensity of electrostatic interactions. Understanding surface wettability involves examining how liquids interact with solid surfaces, one way to measure this is by looking at the contact angle formed between a liquid droplet and the surface. Surface wettability is affected by factors such as surface roughness, surface energy, and chemical composition, reflectivity, also referred to as optical reflectance is the capacity of a surface to bounce back light. It is influenced by various factors including surface texture, roughness, and the optical properties of the material. Understanding reflectivity is crucial for assessing the visual and optical qualities of objects (Hunter et al., 1987; Quéré, 2008; Jenkins & White, 1957). Surface hardness is an important characteristic for materials used in applications that demand wear resistance and durability, it measures the material's ability to resist indentation, scratching, and deformation, surface hardness can be measured using techniques such as Vickers hardness or Rockwell hardness tests (Jones & Ashby, 2012). Surface porosity is the existence of empty spaces or pores on the material's surface, It can have an

effect on properties like permeability, adsorption capacity, and surface reactivity, Surface porosity can be characterized using techniques such as scanning electron microscopy (SEM) and mercury intrusion, porosimetry is crucial for examining surface properties and exploring measurement techniques, influencing factors, and their impact on various applications (Lowell et al., 2006). Cold plasma sources used to modify the surface of polymers, we will now discuss the primary and significant categories of cold plasma and methods of generating it for polymer surface modification. The most notable types of cold plasma can be categorized into four or five types, one of the most prevalent forms is glow discharge plasma which is produced by applying voltage between two electrodes in a chamber filled with gas, resulting in the formation of a visible glow (Moon et al., 2004). Glow discharge plasma is used in many industrial applications, including thin-film deposition and surface modification. The second type is known as "dielectric barrier discharge" (DBD) plasma in this type a high-frequency electric field is applied between a pair of electrodes that are separated by a dielectric material, the presence of the dielectric barrier prevents a direct discharge between the electrodes, resulting in the creation of a diffuse plasma glow, DBD plasmas are often used for surface treatment, including cleaning, sterilization, and surface activation. The third kind of cold plasma is referred to as the "atmospheric pressure plasma Jet (APPJ)" The APPJ device produces a plasma jet by directing a gas flow, usually helium or argon, via a tiny nozzle, the gas is ionized by placing a high-voltage electrode close to the nozzle, resulting in a stream of cold plasma that may be aimed at the target surface. APPJ, or atmospheric pressure plasma jet, is used in several fields such as surface cleaning, wound healing, and material processing (Foest et al., 2005; Corke et al., 2009; Liu et al., 2004). The fourth category is corona discharge plasma corona discharge plasma is generated by subjecting a gas-filled environment to a high voltage applied to a pointed electrode, the intense electric field at the tip of the electrode induces ionization of the gas molecules, resulting in the formation of a plasma (Chang et al., 1991). Corona discharge plasma is used in several applications, including air filtration, ozone formation, and electrostatic precipitation (Dorsey & Davidson, 1994). The final form of cold plasma is known as inductive coupled plasma (ICP). ICP is produced by exposing a gas to a high-frequency electromagnetic field inside a reactor chamber. The high-frequency field induces an electric current in a coil that surrounds the chamber, resulting in the creation of a powerful magnetic field. This magnetic field, in turn, generates the plasma (Soltanpour et al., 1996). ICP, or inductively coupled plasma, is widely used in the semiconductor industry for many purposes such as plasma etching, deposition, and material characterization these applications represent just a few instances of the extensive research and utilization of cold plasma each variety has unique qualities and is appropriate for certain applications, determined by criteria such as the desired attributes of the plasma, the

material being targeted, and the needs of the process (Tinck & Bogaerts, 2012). Cold plasma is used in the process of treating material surfaces, cold plasma has the capacity to alter the surface characteristics of different materials, resulting in improved adhesion, enhanced wettability, and increased bonding strength, this effect may be seen in materials such as plastics, metals, and ceramics. this is applicable in sectors such as automotive, electronics, and packaging (Carrino et al., 2004). The use of cold plasma for water sterilization and decontamination is very successful in eradicating bacteria, viruses, and organic contaminants from water, without the need for chemical substances, cold plasma has the potential to be used in several areas such as wastewater treatment, drinking water purification, and water recycling, these examples demonstrate the broad range of possibilities for this technology (Gururani et al., 2021). Chemical composition alterations through the addition of new functional groups or the removal of contaminants, plasma treatment has the ability to modify the chemical composition of a surface. This may lead to enhancements in the surface energy and reactivity of the material. Polymers may be improved in terms of wettability and adhesion by introducing oxygen-containing groups by plasma treatment. Plasma treatment enhances the biocompatibility of materials used in biomedical applications. It improves cell attachment, proliferation, and differentiation by increasing surface hydrophilicity and promoting protein adsorption. This is particularly important for implants and tissue engineering applications, where the interaction between materials and tissues is crucial for success. Plasma surface treatment is a powerful method that modifies the surface properties of various materials to meet particular application requirements, leading to enhanced performance and utility in several industries (Ujino et al., 2019; Mozetič, 2019).

The purpose of this work was to show that the plasma treatment process could be used to change poly (acrylic acid-co-methyl methacrylate) hydrogel polymer surfaces properties under extremely varied gas conditions. For that purpose the objectives of the present work are to perform a complete understanding of the properties and behaviour of hydrogel, contact angle, X-ray photoelectron spectroscopy (XPS), and other techniques to provide valuable information about the surface and chemical composition of hydrogel surface (Khan et al., 2022; Sun et al., 2023).

## **2. HYDROGEL PREPARATION AND PLASMA TREATMENT**

Hydrogel was synthesized in three necks bottom flask by mixing the following substances with certain ratios of (75ml) of methyl acrylate ( $\text{CH}_2=\text{CHCOOCH}_3$ ), (2ml) of Hexanediol diacrylate  $\text{C}_{12}\text{H}_{18}\text{O}_4$ , (1gm) of initiator, the manufacturer of these substances is (sigma - Aldrich/American chemical), and (25ml) acrylic acid ( $\text{CH}_2=\text{CHCOOH}$ ) from Himedia (India), while agitating

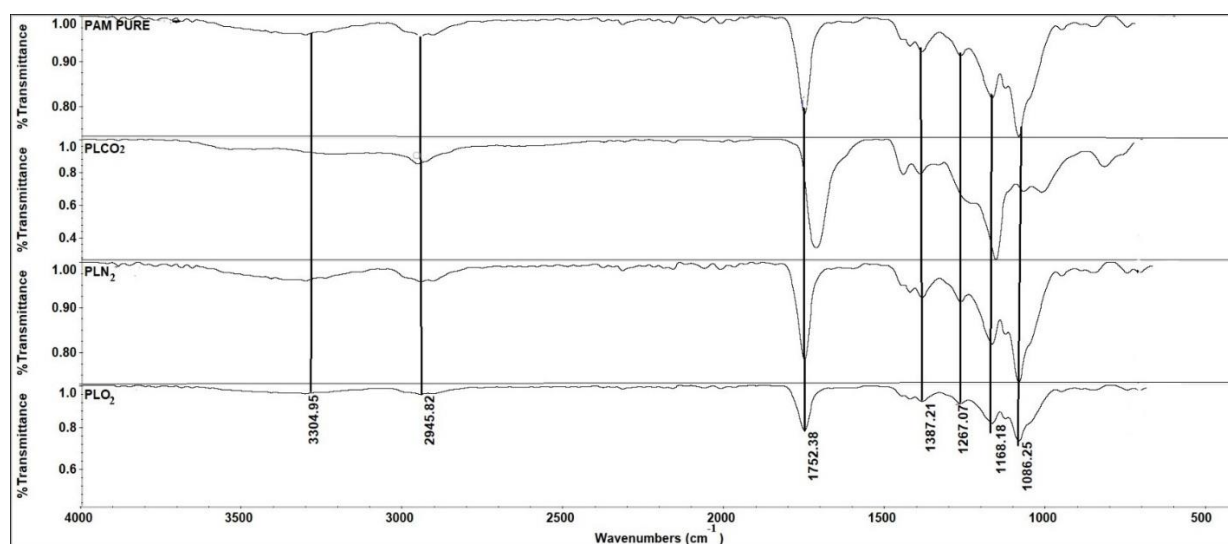
with an overhead stirrer at a speed of 1000 revolutions per minute, the reaction mixture was flushed with nitrogen for a duration of 30 minutes and then allowed to react at a temperature of 70 degrees celsius in a nitrogen atmosphere for a period of 5 hours. Nitrogen gas has also been used as an auxiliary coefficient for interaction as it does not react rapidly with many chemical compounds and is therefore used to reduce the oxidation effect in specific reactions. Nitrogen gas is added to protect sensitive compounds from damage caused by their interaction with oxygen in the air. To obtain a photo-crosslinked hydrogel 100 L of the sample was irradiated using the optical to obtain hydrogel by an ultraviolet lamp, a pure hydrogel film and three films of samples of 350  $\mu\text{m}$  thickness were surface-treated by an argon atmospheric pressure cold plasma jet and exposed by low-pressure gases  $\text{O}_2$ ,  $\text{N}_2$  and  $\text{CO}_2$  respectively, the hydrogel film has an angle of  $45^\circ$  to the gas flow when exposed. The experiment used a VERTEX 70 spectrometer fitted with a PIKE single reflection

MIRacle ATR component. The XPS spectra of the plasma-treated samples and the control were obtained using a SPECS SAGE spectrometer equipped with a monochromatic Mg-K alpha radiation source (1253.6 eV) operating at a power of 200 W. The scanning electron microscope (JEOL JSM-6400, NORAN Instruments, Tokyo, Japan) was used to analyze the physical characteristics of the produced samples.

### 3. RESULTS AND DISCUSSION

#### 3.1 ATR Spectra

The Attenuated total reflection (ATR) spectrum of poly (acrylic acid-co-methyl methacrylate) can provide information about the functional groups present at the surface of the copolymer. The characteristic peaks and corresponding functional groups you would typically observe in the ATR spectrum of this copolymer Figure 1.



**Figure 1.** Attenuated total reflectance (ATR) The infrared spectra of the sample were converted using fourier analysis.of pure P(AA-MMA) hydrogel and hydrogel plasma treated by  $\text{CO}_2$ ,  $\text{N}_2$  and  $\text{O}_2$

A broadband for untreated P(AA-MMA) hydrogel is observed around  $3304.95\text{ cm}^{-1}$  corresponding to hydroxyl stretching groups from AA, these bands are affected as P(AA-MMA) is exposed to  $\text{CO}_2$  and  $\text{O}_2$ . The peak at  $2945.85$  for the  $(\text{CH}_3\text{-CH}_2)$  group, because the Poly (Acrylic acid-co-Methyl methacrylate) structure has both methyl and methylene groups, the peaks observed at  $1752.38\text{ cm}^{-1}$  is for the stretching band in  $\text{C=O}$  stretching of ester group and have a significant shift towards lower wave number side as the hydrogel exposed to  $\text{CO}_2$  indicated that the strength of the bond between C and O is affected by the molecular structure, peaks at  $1387.21$  and  $1267.07\text{ cm}^{-1}$  where assigned to the vibration bands of  $-\text{CH}_2$  and  $-\text{CH}_3$  respectively, these peaks show a different decrease in the intensity as the hydrogel exposed, the peak  $1168.18\text{ cm}^{-1}$  for  $\text{N-O}$  stretching, the peak shift toward lower wave number as the hydrogel exposed to  $\text{CO}_2$ , the disappearance of  $\text{C-O}$  stretch peak at  $1086.25\text{ cm}^{-1}$  in hydrogel sample as exposed to  $\text{CO}_2$

show a chemical interaction between hydrogen and carbon dioxide at the surface (Lejnięks et al., 2010; Gyurova et al., 2017).

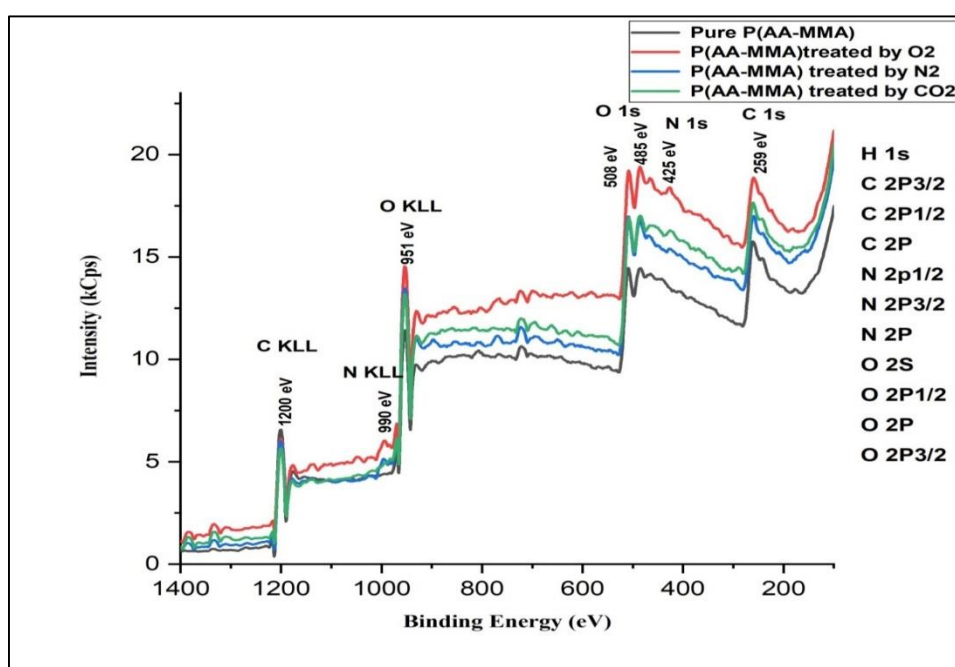
#### 3.2 X-ray photoelectric spectroscopy (XPS)

Surface composition alteration of the samples was investigated through x-ray photoelectron spectroscopy (XPS) measurements, which were conducted to analyze the effects of plasma treatment. Figure 2 a,b,c,d display the survey scans of the samples. The samples consist of peaks H, O, N, and C that originate from 1s.

The synthesized hydrogels were studied by X-ray Photoelectron Spectroscopy (XPS) hydrogel before and after hydrogels exposed to cold plasma under various gases to determine the chemical states of the surface functional groups, Figure 2 and Table 1 show the data have been reported, the spectrum in figure 2 includes a

sequence of peaks labelled O, N, C KLL. The peaks in the spectrum correspond to the energy levels of the electrons that are released from the atoms when the O, N, C 1s state (K shell) is filled by an electron from the L shell, resulting in the ejection of an electron from a L shell. The C, N, O 2p peaks are made up of two main components., namely S 2p3/2 and S 2p1/2, arising from the spin-orbit splitting in the p-type orbitals. The spectrum for samples contains a single peak with O, N, and C KLL centred at binding energies 1200, 990 and 951

eV respectively. Poly (acrylic acid-co-methyl methacrylate) hydrogel films, peak for electrons originating from O, N, and C 1s levels The peaks located at 508 eV (atomic percentage:0.43) and 485 eV (atomic percentage:0.61) for O 1s related to C=O and C-O bonds at the hydrogel surface, while the peak at 425eV (atomic percentage:0.01) for N1s can be related for N-H, and C 1s at 259 eV (atomic percentage:1.23) due to C-C bound. (Martínez et al., 2023; Tokuda et al., 2015).



**Figure 2.** XPS Spectra for pristine and treated P(AA-MMA) hydrogel samples with cold plasma in O<sub>2</sub>, N<sub>2</sub> and CO<sub>2</sub> gases

**Table 1.** Binding energy, full width at half maximum (FWHM), and percentage of H1s, O1s, N1s, and C1s core peaks are being analyzed for P(AA-MMA) samples

	P(AA-MMA) untreated			P(AA-MMA) treated by O <sub>2</sub>			P(AA-MMA) treated by N <sub>2</sub>			P(AA-MMA) treated by CO <sub>2</sub>		
	B.E (eV)	FWHM	AT%	B.E (eV)	FWHM	AT%	B.E (eV)	FWHM	AT%	B.E (eV)	FWHM	AT%
O 1s	508	5.21	0.43	533	5.16	1.1	532	4.45	0.93	533	5.17	0.84
	485	4.89	0.61	491	4.52	0.68	490	4.75	0.73	491	4.57	0.69
N 1s	425	4.05	0.01	424	4.68	0.04	425	5.17	0.02	423	3.07	0.02
C 1s	259	5.59	1.23	260	5.7	1.78	260	0.87	1.86	258	5.71	1.55

### 3.3 Surface hydrophilicity

The hydrophilicity of the surface of P(AA-MMA) hydrogels was determined through contact angle measurements, the contact angle is a quantitative indicator of the degree to which a liquid may spread on a solid surface. It is often determined by measuring the angle created between the liquid droplet and the solid surface at their point of contact. It provides information about the hydrophilicity or hydrophobicity of the surface. Poly (Acrylic acid-co-methyl methacrylate) hydrogels are typically considered to be hydrophilic due to the presence of the acrylic acid component, which contains polar functional groups (Guo et al., 2015; Li et al., 2012).

Figure 3 displays images of the contact angle of water droplets on the surface of a hydrogel film made of pure Poly(acrylic acid-co-methyl methacrylate). The surface films were subjected to oxygen, nitrogen, and carbon dioxide at atmospheric pressure.

However, the specific contact angle of the hydrogel will depend on the exact composition and structure of the copolymer, as well as any modifications or surface treatments applied to it. Figure 3 and Table 2, show poly(methacrylic acid-co-methyl methacrylate) is hydrophobic hydrogel as the hydrogel is exposed to cold plasma under oxygen, nitrogen and carbon dioxide in atmospheric pressure. The results of contact angles,

wetting and adhesion interactions are gradual changes from hydrophobic to hydrophilic. The shifting and varying amounts of carbonal, methylene and nitrogen-containing groups function groups. Figure 1 affected the degree of crosslinking and was found to have varying contact angles.

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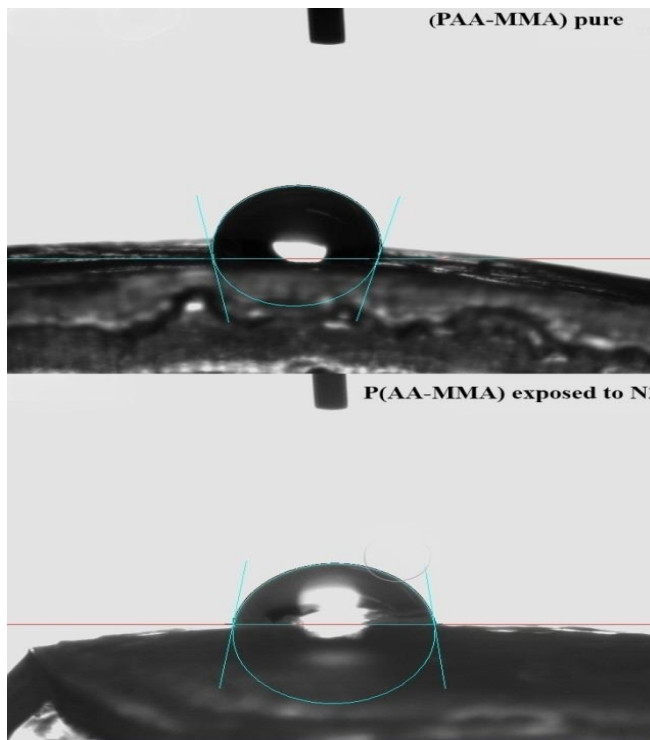


Figure 3 displays images of the contact angle of water droplets on the surface of a hydrogel film made of pure Poly(acrylic acid-co-methyl methacrylate).

Table 2. The contact angles for the sessile drop of water

Sample	Left angle	Right angle
P(AA-MMA) Pure	102.1 <sup>0</sup>	103.4 <sup>0</sup>
P(AA-MMA) exposed to O <sub>2</sub>	56.9 <sup>0</sup>	53 <sup>0</sup>
P(AA-MMA) exposed to N <sub>2</sub>	78.8 <sup>0</sup>	83.32 <sup>0</sup>
P(AA-MMA) exposed to CO <sub>2</sub>	60.1 <sup>0</sup>	59.7 <sup>0</sup>

### 3.4 SEM Characterization

Figure 4 displays the scanning electron microscope (SEM) pictures depicting the surface of poly (acrylic acid-co-methyl methacrylate) hydrogel films before and after cold plasma treatment at different gas treatments, correspondingly. The films subjected to plasma treatment exhibited a rough surface, following plasma treatment it was discovered that the surfaces had several concave areas. Figures 4b, c, and d show that the hydrogel degraded as a consequence of plasma treatment.

The surface films were subjected to oxygen, nitrogen, and carbon dioxide at atmospheric pressure

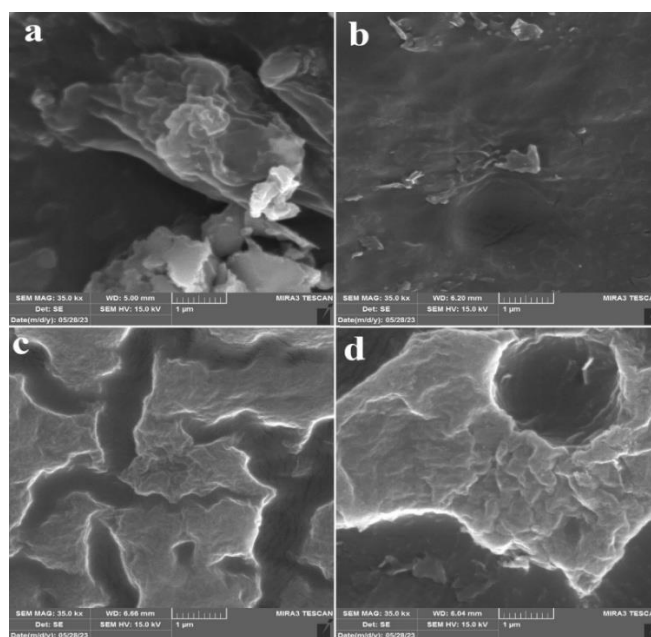


Figure 4. SEM micrograph of P(AA-MMA) hydrogel films different cold plasma treatments (a) untreated, (b) Nitrogen gas, (c) oxygen gas, (d) carbon dioxide gas

When polymers are subjected to cold plasma radiation, they undergo weight loss and the outermost layers of hydrogel films are eroded [53]. Plasma functionalization provides a versatile way to modify the surface properties of materials. By using different gases or organic precursors, it's possible to achieve a wide range of surface functionalities, such as increased hydrophilicity, enhanced adhesion, or improved wettability. Plasma functionalization is an appealing option that provides a wide range of possibilities for modifying surface properties. It often involves the use of ecologically friendly substances like gases, such as oxygen, nitrogen, and carbon dioxide, as well as very small quantities of organic precursors. Since the modified surfaces are only a few monolayers thick, these processes are extremely rapid and low in cost. Figures 4 a,b,c, and d show cold plasma allows for the creation of a new family of surface chemistries, enabling the development of tailored surfaces with specific properties.

The formation of a heterogeneous structure can be attributed to the presence of bubbles formed during polymerization, potentially caused by the use of nitrogen to remove impurities from the reactants. Additionally, the heterogeneity may be a result of the partial compatibility between the acrylic acid crosslinker and the Methyl methacrylate monomer.

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#### 4. CONCLUSION

Cold plasma interacting with hydrogel surface has been investigated, in this study four different poly (acrylic acid-co-methyl methacrylate) hydrogel films were prepared, three of them treated by cold plasma under oxygen, nitrogen and carbon dioxide gases. The hydrogel samples were analyzed using attenuated total reflection (ATR) spectroscopy, contact angle and XPS to study the chemical and physical structures. ATR identifies all functional groups of P(AA-MMA) hydrogel, and well-defined crosslinking and hydrogen bonding. The contact angle data provided in this work demonstrate a reduction in all cases after cold plasma treatment, depending on the kind of gas used. This indicates an improvement in the surface hydrophilicity of the hydrogel surfaces. The XPS analysis revealed an increase in the quantity of oxygen atoms in these samples, as well as the inclusion of contact angle measurement. The analysis indicated that the proportion of C=O double bond is the primary determinant of the contact angle and hydrophilicity of hydrogel surfaces. Combining SEM findings of the P(AA-MMA) surface with contact angle data, we discover that cold plasma treatment of the surface at the micron scale may increase hydrophilicity, in addition to the contribution of the C=O bond increasing.

*Awad et al., Cold plasma gas type treatment on surface characteristic of poly (acrylic acid-co-methyl methacrylate) hydrogel*

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