Volume 18, No. 3, 2024

ISSN: 1750-9548

# Cold Plasma Surface and Linear/Nonlinear Optical Properties of Poly (Acrylic Acid-Co-Methyl Methacrylate) Hydrogel Film

Marwan Manhal Awad <sup>1</sup>, M. T. Ahmadi <sup>2</sup>, Hisham Mohammed Ali Hasan <sup>3</sup>

<sup>1</sup> Marwan Manhal Awad, PhD student in the Physics department, Faculty of Science, Urmia University, Urmia, Iran.

aljanaby965@gmail.com

<sup>2</sup> Professor. Dr. M. T. Ahmadi, Nanoelectronic Research Group Physics Department, Urmia University, Iran,

mt.ahmadi@urmia.ac.ir

<sup>3</sup> Professor, Hisham Mohammed Ali Hasan, Physics Department, College of Education, University of Al-Qadisiyah, Diwaniyah, Iraq,

hisham.hasan@qu.edu.iq

#### Abstract

In this current study cold plasma-treated poly (acrylic acid-co-methyl methacrylate) hydrogel P(AA-co-MMA) films for application in optoelectronics devices. The Attenuated total reflectance (ATR) and X-ray photoelectron spectroscopy (XPS) methods were used to document the impact of hydrogel on the structures, morphologies, and functional groups of the cold plasma, respectively. The optical characteristics of the hydrogel specimens were analysed using UV-Vis spectroscopy. The absorption edge and Urbach energies of films exposed to pure and cold plasma under  $O_2$ ,  $N_2$ , and  $CO_2$  have been calculated using Tauck's formula. As hydrogel surface cold plasma was treated, the Urbach tail decreased from 0.69 eV to 0.53 eV and in lear optical properties the absorbance via the wavelength show n to  $\sigma^*$  and also to  $\pi^*$  orbital, the nonlinear dispersion characteristics and optical susceptibilities of pure and plasma-treated materials were recorded. The processed samples showed improved structural and linear/nonlinear optical properties, rendering them appropriate for use in optoelectronics.

Keywords: hydrogel; cold plasma; optical properties; ATR analysis. XPS.

#### 1. Introduction

Plasma treatment involves exposing the polymer surface to a low-temperature, ionised gas (plasma). The plasma contains a variety of reactive species such as ions, electrons, free radicals, and UV photons. When the plasma comes into contact with the polymer surface, it can induce a range of chemical and physical modifications. One of the commonly used plasma sources for polymer surface modification is atmospheric pressure plasma jets (APPJs). These plasma sources generate a non-thermal plasma at atmospheric pressure, allowing easy integration into existing manufacturing processes[1,2]. APPJs can be used to deposit thin film hydrogels onto polymer surfaces through a process called plasma polymerization or plasma polymer grafting [3,4].

Plasma polymerization involves introducing a precursor gas containing monomers into the plasma environment. The plasma dissociates the monomers into highly reactive species, which then polymerize and form a thin film on the polymer surface. By carefully selecting Plasma polymerization involves introducing a precursor gas containing monomers into the plasma environment. The plasma dissociates the monomers into highly reactive species, which then polymerize and form a thin film on the polymer surface, by carefully selecting the precursor gas and process conditions, since the precursor gases such as nitrogen, oxygen or carbon

Volume 18, No. 3, 2024

ISSN: 1750-9548

dioxide affect the surface properties (functional groups) by chemical modifications on the treated surface with varying influence [5,6,7].

The hydrogel film application fields based on optical properties include light emission and detection, solar cells, contact lenses, photoelectronic, waveguide, and biosensing. Owing to the significance of altering the properties of hydrogel surfaces, various surface treatments have been employed to improve the surface properties, a wide part of these treatments include chemical and mechanical treatments which are not very effective, and the use of chemical treatments involving solvents, strong acids, or bases is becoming less and less acceptable due to health and environmental concerns [8]. Because of the importance of hydrogel surfaces for the development an effective diagnostic optical properties, here we report the synthesis and fabrication of poly (acrylic acid-co-methyl methacrylate) hydrogel film with cold plasma surface treatment under the effect of N2, O2, and CO2 gases, to overcome those serious limitations, and for development of advanced hydrogels with controllable surface properties such as surface chemistry and morphology by perspective, a generation of functionalities such as hydroxyl (–OH), amine (–NH2) and carboxyl (–COOH) groups at the surface of hydrogels. In this work, change in optical properties is studied as the surface concentration of functional groups affected by cold plasma treatment [9,10].

Overall, the plasma polymerization of thin film hydrogels offers precise control over the film properties, enabling improvements in transparency, refractive index control, surface modification, and the incorporation of optically active compounds. These benefits make plasma-polymerized hydrogels promising materials for various optical applications[11,12].

#### 2. Material and methods.

## **2.1** Materials and experimental details.

Hydrogel was synthesized in three necks bottom flask by mixing the following substances with certain ratios of (75ml) of methyl acrylate (CH2=CHCOOCH3), (2ml) of Hexanediol diacrylate C12H18O4, (1gm) of initiator, the manufacturer of these substances is (sigma -Aldrich/American chemical), and (25ml) acrylic acid (CH2=CHCOOH) from Himedia (India). After 30 minutes of purging with nitrogen, the reaction mixture was stirred with an overhead stirrer set to 1000 rpm ,under nitrogen, the mixture was allowed to react at 70 °C for 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 μm thickness were surface-treated by an argon atmospheric pressure cold plasma jet and exposed by low-pressure gases O2, N2 and CO2 respectively, the hydrogel film has an angle of 450 to the gas flow when exposed.

# 2.2 Characterization.

The X-ray photoelectron spectroscopy (XPS) results were acquired using a Fi Kratos Axis Ultra HAS instrument, using a monochromatic X-ray source Al at a voltage of 15 kV (90 W). The morphology of produced hydrogel sheets was analyzed using scanning electron microscopy (SEM). Images were acquired using an FEI Quanta 400FEG ESEM/EDAX Genesis X4M. The FTIR spectra of the synthesized hydrogel samples were obtained using the FTIR spectrophotometer Bomem Hartmann and Braun MB-100 series, covering wave numbers ranging from 4000 to 400 cm-1. The Z-scan closed aperture methods were used to compute the nonlinear refractive index. The Z-scan closed aperture system is used to analyze a laser beam produced by a continuous wave diode laser operating at a wavelength of 650 nm and a power output of 50 mW.

#### 3. Surface Characterization.

#### 3.1 Attenuated total reflectance analysis.

The ATR spectrum of The hydrogel films of Poly (Acrylic acid-co-methyl methacrylate) are shown in Figure 1. The absorption band near 2955.65 cm<sup>-1</sup> was assigned to the C-H stretching was observed for the AA–MMA copolymeric which agree with Ref [13,14].

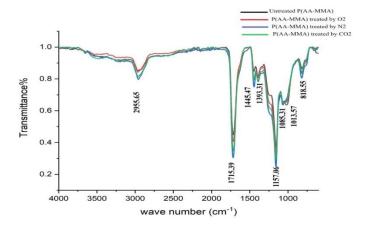


Fig. 1. The IR spectrum of untreated and treated films made of poly (acrylic acid-co-methyl methacrylate) hydrogel.

The peak at 1715.39 cm<sup>-1</sup> was assigned to the C=O stretching of methyl ester in MMA units. A distinct change was observed in the absorption band near 1445.47 and 1393.31 cm<sup>-1</sup> related to C=N stretching of the primary amide, while the three bands at 1157.06, 1058.31 and 1013.57 cm<sup>-1</sup> to aromatic C-N stretching absorption band. The band at 818.55 cm<sup>-1</sup> belongs to the MMA-triazine ring as in references [15,16,17].

### **3.2** X-ray photoelectron spectroscopy.

The spectrum in Figure 2, a,b,c and d, comprises a series of peaks labelled C, N, and O KLL, these peaks centred at binding energies 1200, 990 and 951 eV respectively correspond to the energy levels of electrons ejected from the 1s states (K shell) Caused by an electron from the L shell being coupled with the ejection of another electron from a L shell. Figure 2 displays the X-ray photoelectron spectroscopy (XPS) C 1s, N 1s, and O 1s narrow scans of poly (acrylic acid-co-methyl methacrylate) hydrogel films and the structure of one unit of the polymer[18,19]. The C 1s peak corresponds to the methyl group,  $-CH_3$  and  $-CH_2$  in chains of the structure, from Table.1 These carbon atoms correspond to the peak at 285eV for an untreated hydrogel made of P(AA-MMA) and, 286eV for treated P(AA-MMA) hydrogel by  $O_2$ ,  $O_2$  and  $O_3$  respectively, have an increase in atomic percentage (AT%) and full-width half maximum (FWHM) it implies a change in the local chemical environment around the carbon atom as in references [20,21].

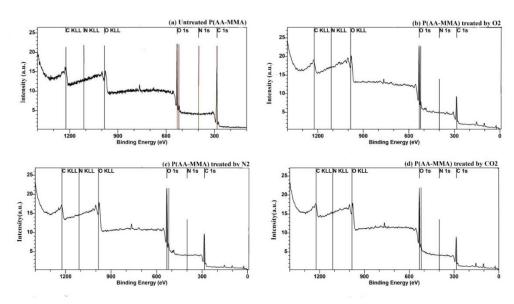


Fig. 2. XPS spectra of poly (acrylic acid-co-methyl methacrylate) hydrogel structure showing the presence of core level spectra of O1s, N1s, and C1s.

Table 1 Binding energy, full width at half maximum (FWHM) and % of H1s, O1s, N1s and C1s core peaks for P(AA-MMA) hydrogel films.

	544	3.07.13		D(1.1.3.0.61)			D(1.1.3.D(1))			7(1.1.3.0.61)		
	P(AA-MMA) untreated			P(AA-MMA) treated by			P(AA-MMA) treated by			P(AA-MMA)		
				$O_2$			$N_2$			treated by CO <sub>2</sub>		
	B.E	FWHM	AT%	B.E	FWH	AT%	B.E	FWHM	AT%	B.E	FW	AT%
	(eV)			(eV)	M		(eV)			(eV)	HM	
H 1s	8	6.77	98.33	15	3.46	97.09	15	4.45	97.21	12	4.81	97.6
O 1s	532	5.21	0.27	533	5.16	0,92	532	5.17	0.77	533	5.17	0.72
	485	4.89	0.16	491	4.52	0.18	490	4.75	0.16	491	4.57	0.12
N 1s	400	4.05	0.01	400	4.68	0.02	398	0.87	0.01	401	3.07	0.02
C 1s	285	5.59	1.23	286	5.7	1.78	286	5.86	1.86	286	5.71	1.55

The methyl methacrylate (C<sub>4</sub>H<sub>8</sub>O<sub>2</sub>) in P(AA-MMA) hydrogel consists of a methyl group (CH<sub>3</sub>), a methacrylate group, and an ester functional group, ester groups contain one C=O bond and two C-O bonds, the carbon atom is often considered electron-poor or oxidized. When carbon and oxygen bond together in the ester group, the electronegative difference between them causes carbon to lose electron density to oxygen, carboxyl carbon, shows the highest narrow binding energy in the spectrum, and the O 1s peaks show two peaks, 532 eV corresponding to C=O, shifted by 1 eV as hydrogel treated by O<sub>2</sub> and CO<sub>2</sub>, while the second peak at 485eV related to C-O, shift by 6 eV in binding energy as hydrogel treated by O<sub>2</sub> and CO<sub>2</sub>, and 5eV as treated by N2 [22,23].

The small peak for XPS N 1s is identified at 400 eV, the shift in binding energy indicates that each carbon-nitrogen in amide groups varies the binding energy of a nitrogen 1s electron (chemically shifts it) by 1.0 eV (23.06 kcal/mol), as the hydrogel treated by cold plasma in N<sub>2</sub> or CO<sub>2</sub>, while the binding energy didn't change as treated in O<sub>2</sub>[24]. The chemical shifts seen in these spectra may be attributed to the chemical composition of the materials and are indicative of changes in the binding energy of the atom, which in turn correspond to a decrease in the electron density of the atom. Plasma treatment forms chemical bonds on the surface of the specimen, and the introduction of oxygen-containing functional groups such as C-O bonds or changes like carbon-carbon bonds could lead to a chemical shift in the XPS spectrum, additionally, exposure to reactive species like oxygen radicals is a common part of the cold plasma effect. This can lead to the oxidation of carbon-containing species on the surface, resulting in the formation of C-O bonds and causing a chemical shift in the XPS spectrum[25,26].

#### 3.3 Ultraviolet-Visible-Near Infrared Spectroscopy.

#### 3.3.1 Optical Properties.

Examining the optical properties of materials is a helpful approach to study both the band structure and the density of electronic states [27]. The optical characteristics of Poly (Acrylic acid-co-methyl methacrylate) hydrogel film often change when treated with cold plasma in the presence of  $N_2$ , $O_2$ , $CO_2$  gasses [28].

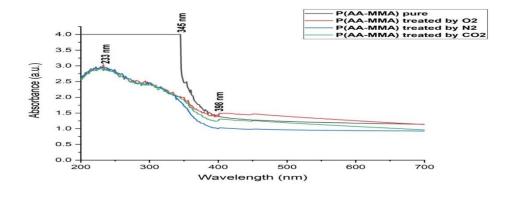


Fig. 3. UV absorption spectra of poly (acrylic acid-co-methyl methacrylate) hydrogel films.

Figure 3, shows the highest absorbance for Optical absorbance values in untreated P(AA-MMA) are unchanged in the UV region (200 to 345 nm) as a result of Chromophores conjugated pi-electrons in inter-chain hydrogen R(CH=CH)nR undergoing  $\pi \to \pi^*$  transition, for P(AA-MMA) treated with O2, N2, and CO2 in the UV region, the peak at 233 nm is due to strong absorption by additional  $\mathbf{n} \to \pi^*$  transition, depending on the type of molecule and the nature of the chemical bonds that the molecule contains [29,30].

The absorbance decreases as the wavelength increases, and the peak at 398nm undergoes  $\mathbf{n} \to \boldsymbol{\sigma}^*$  due to the presence of electron-withdrawing p-nitro substituent, it means that one or more of the ethylene units in the polymer have a p-nitro group (-NO<sub>2</sub>) attached to them[31]. The Urbach energy is used to characterize the structural and electronic properties of materials that can arise from impurities, defects, or disorders in the structure. For untreated P(AA-MMA) Urbach energy is 0.68 eV, and 0.69, 0.55, and 0.53 eV for P(AA-MMA) treated by N<sub>2</sub>, O<sub>2</sub>, and CO<sub>2</sub> respectively, as Urbach energy reduces, the surface modification affected the molecular structure and reducing the disorder and improving the structural order of the hydrogel film[32]. In treated hydrogel films the reason for the increase in the amount of optical absorption at the wavelength 233nm, is that the incident photons with an energy greater than the value of the energy gap increase the practical value of the absorption coefficient, this indicates to possibility assure of electronic transitions between the valence and conduction band at these energies [33,34].

# 3.3.2 Z-scan technique.

This method is a very effective and precise way to measure the nonlinear refractive indexes and nonlinear absorption of nonlinear optical materials. It is known for its ability to quickly spread and generate a strong response. The nonlinear refractive index may be readily determined by analyzing the graph of the nonlinear transmission through an aperture in the far field [35]. The typical Z-scan plots of untreated and treated hydrogels films are shown in Fig. 4. The peak-valley configurations of the transmittance open and closed-aperture curves indicate the negative sign of nonlinear refractive indexes,  $n_2$  exhibited large index, of the order of  $10^{-9}$  cm<sup>2</sup>/W indicates a strong intensity-dependent change in the refractive index of a hydrogel films, this is crucial for applications in nonlinear optics, such as in the design of optical switches, modulators, and certain types of lasers. The unsymmetrical curves show a minimum transmittance at z=0, is due to Kerr effect in agreement with the two-photon absorption process.

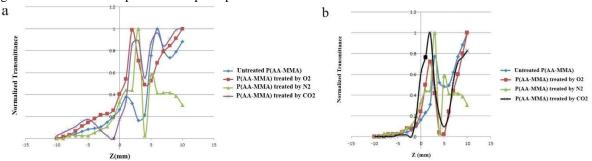


Fig. 4. Z-scan data at of CW diode laser 650 nm with a power of 50mW for poly (acrylic acid-co-methyl methacrylate) hydrogel films: (a) open z-scan and (b) close z-scan.

This approach is a sensitive and contagious single-beam methodology for determining the nonlinear absorption and nonlinear refractive index of nonlinear optical materials. The nonlinear refractive index may be readily estimated from the plot obtained from the nonlinear transmission through an aperture in the far field, it is usual for the nonlinear optical curves to converge at their lowest, which can be caused by absorption mechanisms, experimental variables, inherent material features, or a mix of these [36].

Table 2. Values of nonlinear refractive index,  $n_2$  and nonlinear absorption coefficient, b, of P(MMA-AA) hydrogel films.

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Sample	$\Delta T_{PV}$	n <sub>2</sub> x10 <sup>-9</sup> (cm <sup>2</sup> /mW)	β x10 <sup>-4</sup> (cm/mW)				
untreated P(AA-MMA)	0.292	0.0861	4.706				
P(AA-MMA) treated by O <sub>2</sub>	1.9807	0.4353	23.819				
P(AA-MMA) treated by N <sub>2</sub>	0.9706	0.1601	7.7615				
P(AA-MMA) treated by CO <sub>2</sub>	0.9034	0.0833	4.5578				

According to the transmission spectrum, Figure 4 presents the obtained refractive index values and absorption coefficient of hydrogel films produced from poly(acrylic acid-co-methyl methacrylate) under closed and open aperture circumstances. Table 2 demonstrates that the P(AA-MMA) hydrogel films demonstrated self-focusing. The refractive index was altered by the gas treatment of the hydrogel sheet. The absorption coefficient was calculated from the transmission spectrum using the open aperture z-scan method, as shown in Table 1. Figure 4 displays the outcomes of the non-linear absorption coefficient and saturation absorption for each concentration [37].

#### 4. Conclusion

Poly (acrylic acid-co-methyl methacrylate) hydrogel have shown varying optical properties for different surface modification by cold plasma treatment in presence of one of oxygen, nitrogen, or carbon dioxide gas compared with non-treated hydrogel film provided new ideas on changing hydrogel optical properties. The absorbance verses the wavelengths in UV region show deferent responses between treated and untreated hydrogel films due to deferent electronic transitions, this was diagnosed by FTIR spectra which the main functional groups of the hydrogel network unaltered with plasma treatment, XPS also shows a chemical shifts in the spectra of samples can be related to the chemical structures of the hydrogels at the surface. Only a minor change in the transmittance of the functional groups is found in the direct treatment which also involves UV-Vis radiation. Nonlinear optical properties, the sign and magnitude of nonlinear refraction of hydrogel samples were determined using the Z-scan method. The nonlinear refractive index (n<sub>2</sub>) is of order 10<sup>-6</sup> this indicates a relatively small but significant change in the refractive index as the intensity of the light increases, and nonlinear absorption coefficient ( $\beta$ ) are determined by measuring their values using He-Ne laser light. All of the examined samples exhibited a negative nonlinearity and a significant nonlinear refractive index on the scale of 10<sup>-9</sup> cm<sup>2</sup>/mW indicates that the material has a very small nonlinear response with respect to light intensity. This means that the change in absorption due to changes in light intensity is relatively weak, these materials can be important in fields like nonlinear optics, laser technology, and optical communications.

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International Journal of Multiphysics

Volume 18, No. 3, 2024

ISSN: 1750-9548

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