

Structural and Optical Characteristics of Carboxymethylcellulose / Polyacrylic Acid Polymer Blend

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ABSTRACT

The blending technology makes it possible to rebuild high molecular weights of partial polymers, thus to improve the product performance together with improvement of specific properties. Pure Carboxymethylcellulose (CMC) film and CMC (80%) doped Polyacrylic Acid (PAA) (20%) films were prepared by using a solution casting method. The influence of PAA polymer addition on the structural and optical properties of CMC polymer was investigated systematically. From X-ray diffraction patterns, adding a polymer PAA to a polymer CMC can lead to the appearance of peaks in polymer blend. FESEM image for CMC/ PAA film showed the formation of small spherical grains for PAA into CMC that insured the existence of some peaks in XRD pattern of PAA in CMC film which means the improvement of the microstructure of the polymer matrix. No chemical interactions were observed between CMC and PAA polymers from results of FTIR spectrum, so that incorporation of PAA into CMC polymer will results physical reaction only. The results of the UV spectrum have shown that the absorbance, absorption coefficient, extinction coefficient, refractive index, real and imaginary dielectric constants were increased in blend polymer compared with the pure polymer. The energy gap and transmittance of blend polymer compared with the pure polymer were reduced.

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الخصائص البصرية والتركيبية لمزيج بوليمر كاربوكسي ميثيل السليلوز / بولي أكريليك أسيد

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الخلاصة

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تتيح تقنية المزج إمكانية إعادة بناء أوزان جزيئية عالية من البوليمرات الجزيئية ، وبالتالي تحسين أداء المنتج جنباً إلى جنب مع تحسين خصائص محددة. تم تحضير غشاء كربوكسي ميثيل السليلوز النقي (CMC) وغشاء (CMC) 80 % مطعم ببولي أكريليك أسيد (PAA) (20) % باستخدام طريقة صب المحلول. تم دراسة تأثير إضافة بوليمر PAA على الخصائص التركيبية والبصرية لبوليمر CMC بشكل منهجي. من أنماط حيود الأشعة السينية ، يمكن أن يؤدي إضافة بوليمر PAA إلى بوليمر CMC إلى ظهور قمم في مزيج البوليمر. أظهرت صورة FESEM لغشاء CMC / PAA تكوين حبيبات كروية صغيرة لـ PAA في CMC والتي أكدت وجود بعض القمم في نمط XRD لـ PAA في غشاء CMC وهذا يدل على تحسين البنية المجهرية لمصفوفة البوليمر. لم يلاحظ أي تفاعلات كيميائية بين بوليمرات CMC و PAA من نتائج طيف FTIR ، لذلك فإن دمج PAA في بوليمر CMC سيؤدي إلى تفاعل فيزيائي فقط. أظهرت نتائج طيف الأشعة فوق البنفسجية أن الامتصاصية ، ومعامل الامتصاص ، ومعامل الخمود ، ومعامل الانكسار ، وثوابت العزل الحقيقي والخيالي تزداد في مزيج البوليمر مقارنة بالبوليمر النقي. تم تقليل فجوة الطاقة والنفاذية لمزيج البوليمر مقارنة بالبوليمر النقي .

1. Introduction

Polymer blends developed very significant focus for scientific investigation due to their expanding commercial acceptance, and they can exhibit unique property as a results of their combinations [1]. Polymer materials come in a variety of forms, and the polymer blends are one of them. Polymer blends are formed by physically mixing two (or more) distinct polymers (polymeric substance generated from more than one monomer). Blends are made by combining homopolymers or copolymers with various chemical structures. Depending on the number of polymeric components in the blend, it can be classified as binary, ternary, or quaternary [2, 3]. The characteristics of these materials typically vary depending on the properties of their constituents. Furthermore, in the case of a single component, their features can be complementary and difficult to find together. As a result, polymer blends are great methods to change and improve the properties of polymers [4, 5].

Carboxymethylcellulose (CMC) is a powder; white- to cream-colored, odorless and tasteless, an anionic polysaccharide, long-chain, linear, water-soluble and derived from natural cellulose. The chemical structure of CMC is

shown in Figure (1) [6, 7]. Because of its safety and non-toxicity, it has been widely employed in the pharmaceutical, food, and packaging industries [8, 9].

Polyacrylic acid (PAA) is a water-soluble, biodegradable polymer with a wide range of industrial applications. Polyacrylic acid is a type of polymer that is used to make oral solutions and bio adhesives. It is also used to gel, suspend, and stabilize emulsions [10, 11]. Its chemical formula $(C_3H_4O_2)_n$ and Figure (2) illustrates the chemical structure of PAA polymer. The radical polymerization of acrylic acid produces poly acrylic acid, an anionic muck sticky polymer. (PAA) and its derivatives are commonly utilized in the creation of highly sticky ocular drug delivery formulations, either as viscous gels or nanoparticles, and are frequently combined with other polymers [12, 13].

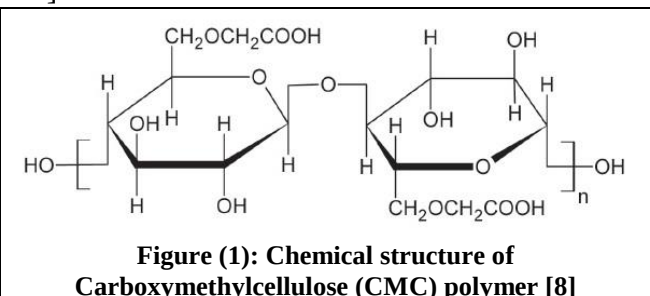


Figure (1): Chemical structure of Carboxymethylcellulose (CMC) polymer [8]

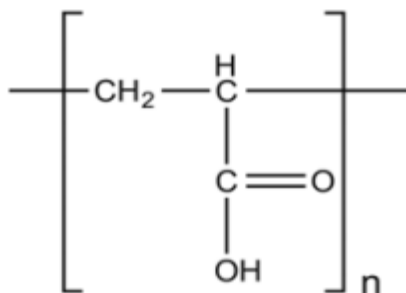


Figure (2): Chemical structure of polyacrylic acid (PAA) polymer [10].

Several investigations have been conducted in this area in order to improve the efficiency of the CMC/PAA blend polymer. For example, A. Abbas et al (2015) [14], studied the optical properties of (PVA-PAA-Ag) nanocomposites. The experimental results showed that the absorbance of polymers mixture, absorption coefficient, extinction coefficient, refractive index and real and imaginary dielectric constants were increasing with the increase of the weight percentages of silver nanoparticles, but the energy gap (E_g) of polymers was decreased with the increase of the silver nanoparticles concentrations. A. Hashim et al (2018) [15], investigated the fabrication of the polyvinyl alcohol - poly-acrylic acid/ niobium carbide (NbC) new bio-films as antibacterial applications. The (PVA-PAA-NbC) nanocomposites were tested as an antibacterial against staphylococcus aureus. The experimental results showed that the nanocomposites had good activity for antibacterial. S. K. Shetty et al 2020 [16], used different experimental techniques, the structural, optical and electrical properties of CMC/Sodium Bromide (NaBr) films were analyzed to understand the effect of sodium metal salt on the properties of the biopolymer CMC. A major difference in the metal salt concentration was seen in the optical bandgap energy and refractive index. Y. M. Jawad et al (2021) [17], considered the structural and optical properties of CMC/MgO nanocomposites films. The results showed that all optical properties of CMC polymer can be enhanced with an increase in concentrations of MgONPs except transmittance and the value of energy band gap will be decreased. The objective of this paper is to study influence of PAA additive on the structural and optical properties of CMC polymer.

2. Experimental part

2.1 Materials Used

The chemical formula of Carboxymethylcellulose (CMC) is $[-\text{CH}_2\text{COOH}]_n$ with molecular weight of 90,000 g/mole from Sigma-Aldrich Germany Company. After being dissolved in distilled water, it was utilized as a fine white creamy powder that becomes colorless. Poly Acrylic Acid (PAA) Polymer was used as a white crystal powder color transform to colorless after being dissolved in distilled water with molecular weight 250,000 g/mole, from Briture Co., Ltd./ China.

2.2 Preparation of CMC and CMC/PAA Films

Pure CMC film was prepared by employing a solution casting method [18]. Distilled water (50 ml) was used to dissolve (0.5 g) from (CMC). This solution has been magnetically stirred continuously for (4 hrs.) until the mixture becomes homogeneous viscous solution. Then it was poured into a glass petri dish with diameter (9 cm) and kept in room temperature of (23 – 28) °C for (7 - 14 days) to evaporate all the solvent slowly in order to obtain CMC film with thickness about (44 μm). The same method has been used to prepare CMC/PAA blend film by using different concentrations of CMC (80%) and PAA (20%), distilled water (50 ml) was used to dissolve the blend. The thickness of film was about (48 μm).

2.3 Characterization Techniques

Pattern of X-Ray Diffraction measurements were used to examine the structure intersystem of crystalline behavior of films at special condition. XRD – instrument type (XRD; X'Pert PRO, PANalytical, the Netherlands), with the following specifications: Target is $\text{CuK}\alpha$, Wavelength is (1.5406 Å), current is (40 mA) and voltage is (40 KV). The incident and reflected angles were scanned using 2 θ technique. The FESEM micrographs were characterized by a field emission scanning electron microscopy (FESEM) type (ZEISS SIGMA VP Field Emission Scanning Electron). Fourier transform infrared spectroscopy (FTIR) has been carried out using (Bruker-Tensor 27 with ATR unit). The Absorption spectrum of as-prepared samples are assessed with UV spectrophotometer sort (T80 Series UV/VIS spectrometer) that procedures within range

(200-1100) nm. The thickness of films was measured by digital micrometer type (Tesda (0.001) mm), the measurement exactness was within the scope of (0 – 150) mm, it made in Japan.

3. Results and discussion

3.1 XRD

The X-ray diffraction spectrum of the pure CMC polymer shown in Figure (3), and the polymer has a wide peak at 20.17° , as can be seen in this Figure. This suggests that it is completely amorphous in form, which is consistent with the findings of [19]

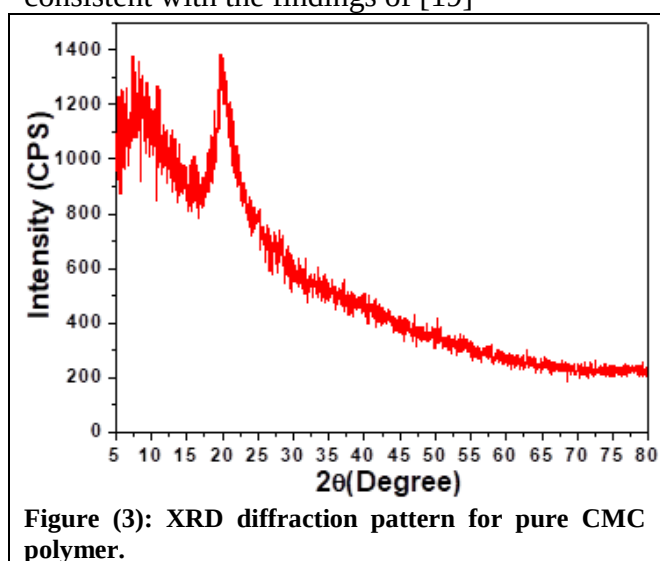
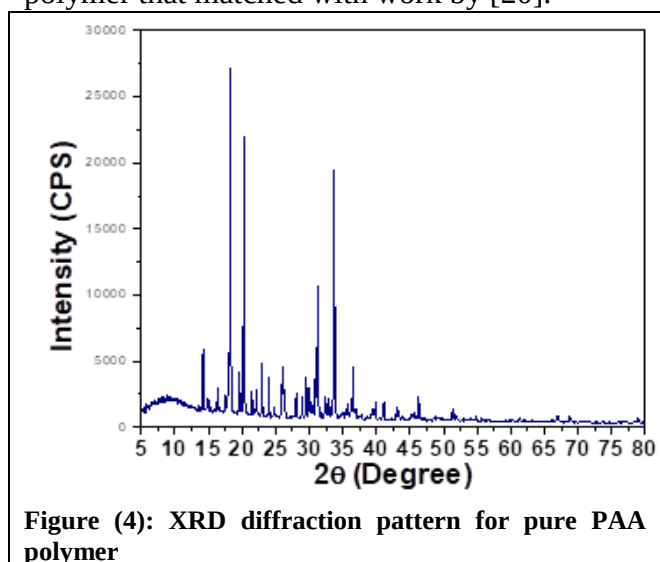


Figure (4) exhibits the X-ray diffraction spectrum of the pure PAA polymer, it can be seen from this Figure that the polymer comprises a number of sharp peaks that demonstrate the crystalline nature of the PAA polymer that matched with work by [20].



The X-ray diffraction spectrum of the pure CMC/PAA blend polymer film can be seen in Figure (5) with the disappearance of the PAA polymer peaks in the area of influence of the CMC polymer and the emergence of a wide peak representing the CMC polymer's XRD diffraction spectrum with the appearance of some PAA polymer peaks as shown in the Table (1).

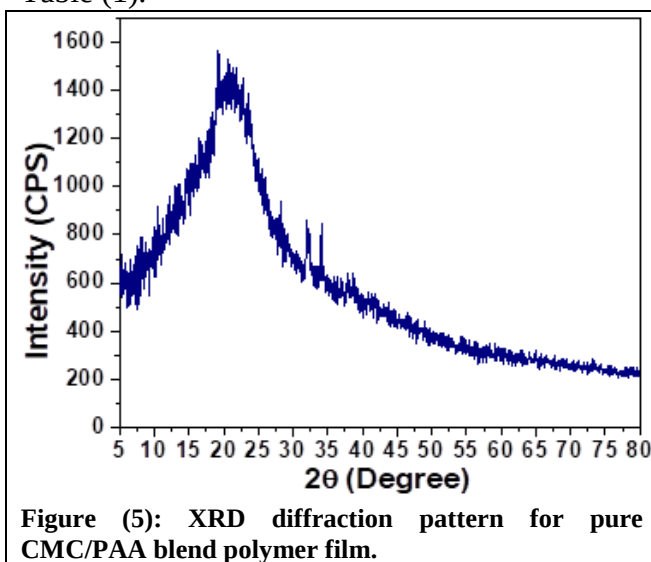


Table (1): XRD Parameters for pure CMC/PAA blend polymer film.

2θ(deg)	FWHM (deg)	Intensity (counts)	d (Å)
19.20	0.24	172	4.619
28.18	0.14	113	3.164
32.00	0.21	148	2.794
34.04	0.28	141	2.631
37.944	0.322	32.99	2.369
38.714	0.173	56.241	2.323

3.2 FESEM

FESEM was used to describe the powders morphology of CMC polymer, PAA polymer as shown in figure (6- A and B), respectively. The CMC polymer can be seen in image A because it is made up of large molecules. Image B describes a PAA polymer which consists of relatively small spherical crystals or particles.

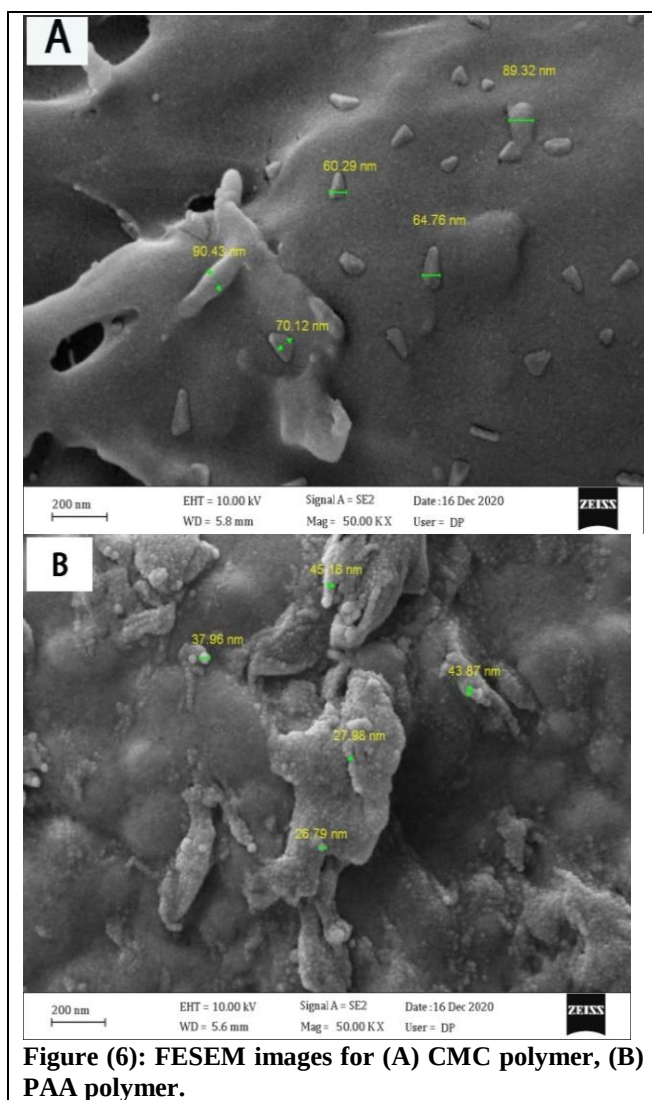
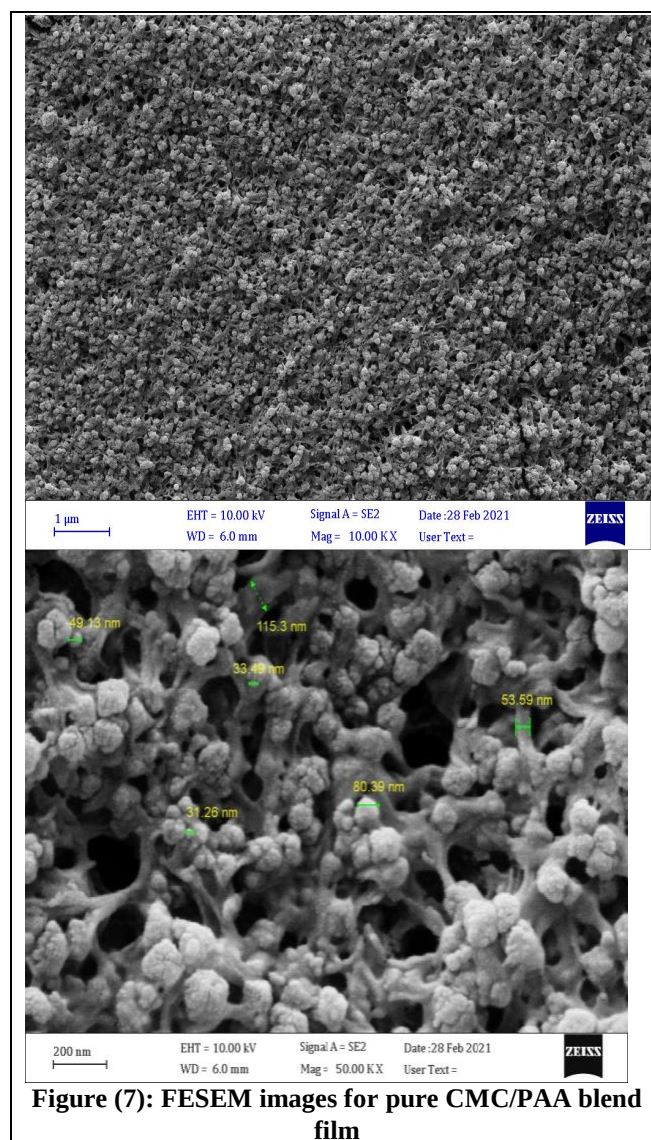


Figure (7) shows distinctive FESEM images of the pure CMC/PAA blend film. From the images, the film were observed as homogenous, cross-linked, coherent and more-soft, which coincidence with research [21]. When compared with FESEM image for CMC (Figure (6-A)), the formation of small spherical grains can be noticed that insured the existence of some peaks in XRD pattern of PAA in CMC film, Figure (5).



3.3 FTIR

The FTIR spectroscopy is important because it give the main characteristics of polymers peaks and nanoparticles. All FTIR spectra were measured in transmittance mode. FTIR spectrum was studied for pure CMC polymer as shown in Figure (8) compared with chemical structure of CMC polymer, (Figure (1)), in order to clarify the locations of the peaks that appeared with types of bonds may be seen in detail in Table (2).

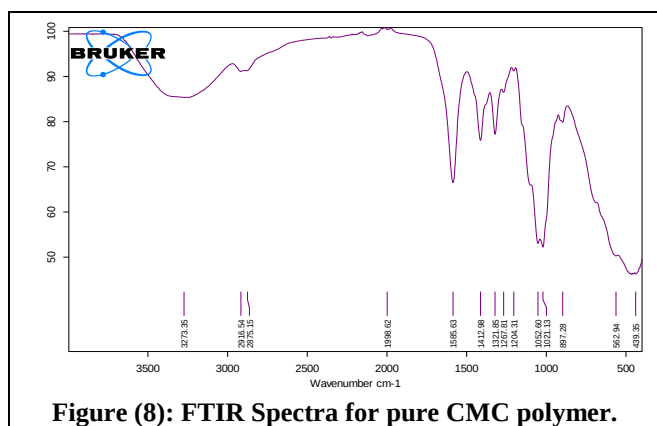


Figure (8): FTIR Spectra for pure CMC polymer.

Table (2): FTIR spectra data for pure CMC polymer

Wave number (cm ⁻¹)	Description of band	References
897.28	1,4-β Glycoside of cellulose	[23, 24]
1021.13	Bending C-O-C	[24]
1052.60	Bending C-O-C	[24]
1204.31	Stretching of ether linkage C-O	[23]
1267.81	Stretching of ether linkage C-O	[23]
1321.85	Bending -OH	[24]
1412.98	Scissoring -CH ₂	[24]
1585.63	Asymmetrical (stretching of carboxyl group) COO -	[25-27]
2875.15	C-H Stretching vibration of the CH, CH ₂ and CH ₃	[27]
2916.54	C-H Stretching vibration of the CH, CH ₂ and CH ₃	[27]
3273.35	Stretching -OH	[24]

To clarify the sites of the peaks that emerged with different types of bonding and according to chemical structure of PAA polymer, FTIR spectrum was analyzed for pure PAA polymer, as shown in Figure (9), and Table (3).

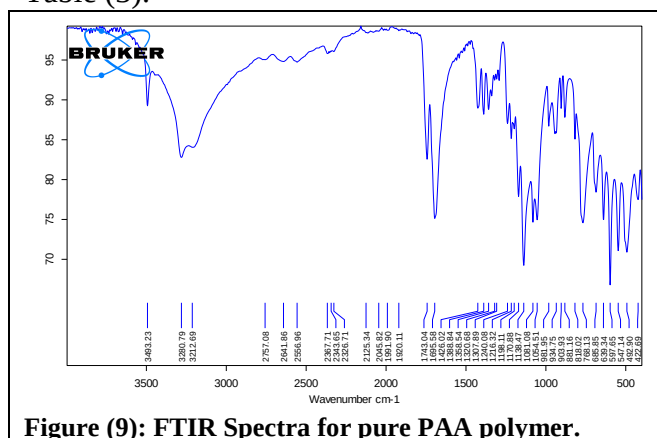


Figure (9): FTIR Spectra for pure PAA polymer.

Table (3): FTIR spectra data for pure PAA polymer

Wave number (cm ⁻¹)	Description of band	References
768.13	Bending C-H	-
818.02	Bending C=C	-
881.16	Bending C=C	-
903.93, 934.75, 981.95	C-O Symmetric stretching mode of PAA	[28]
1054.51, 1081.08	Bending C-O-C	[24]
1138.47, 1170.88	C-O-C Asymmetric bridge stretching	[24, 29]
1198.11, 1216.32, 1240.08	Stretching C-O	[24, 29]
1307.89, 1320.68, 1358.54, 1388.84	Bending -OH	[24, 25]
1426.02	Asymmetric stretching mode of CH ₂ group of PAA	[28]
1695.58	C=O Stretching of carboxylic group	[26, 30]
1743.04	C=O Stretching of acetyl or carboxylic acid	[24, 25]
1920.11, 1991.90	Bending C-H	-
2045.82, 2125.34	Stretching C≡C	-
2556.96, 2641.86, 2757.08, 3212.69, 3280.79, 3493.23	Stretching O-H	[25, 26, 28]

FTIR spectrum was plotted for pure CMC/PAA blend polymer film as shown in Figure (10) investigates the bonds and their positions that formed, see Table (4). It is possible to observe a strong binding ability between CMC and PAA resulting from the carboxyl groups, this matched with results obtained by research [30]. In addition, the presence of the carboxylic groups leads to the forming of hydrogen bonds for the cross-linked chains of polymers and this leads to the forming of an excellent binding ability [32, 33].

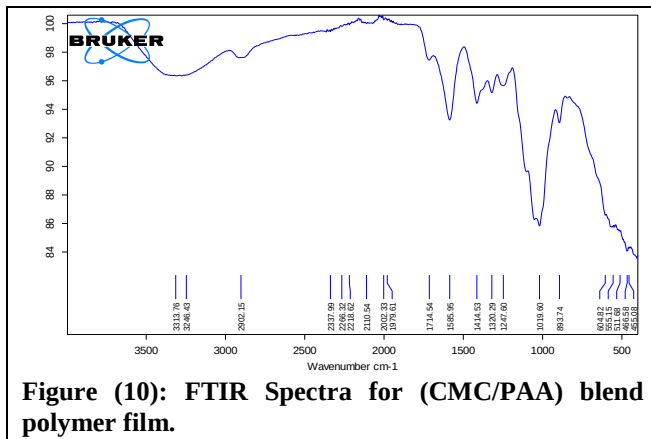


Figure (10): FTIR Spectra for (CMC/PAA) blend polymer film.

Table (4): FTIR spectra data for pure CMC/PAA blend polymer film.

Wave number (cm ⁻¹)	Description of band	References
893.74	1,4-β Glycoside of cellulose	[23, 24]
1019.60	Bending C-O-C	[24]
1247.60	Stretching of ether linkage C-O	[23]
1320.29	Bending -OH	[24]
1414.53	Scissoring -CH ₂	[24]
1585.95	Asymmetrical (stretching of carboxyl group) COO -	[25, 27]
1714.54	C=O Stretching of acetyl or carboxylic acid	[24, 25]
1979.61, 2002.33	Bending C-H	-
2110.54, 2218.62, 2266.32	Stretching C≡C	-
2902.15	C-H Stretching vibration of the CH, CH ₂ and CH ₃	[27]
3246.43, 3313.76	Stretching O-H	[24, 25, 28]

3.4 Optical properties

The absorption spectrum of pure CMC polymer and CMC/PAA blend polymer films can be seen in Figure (11). The absorbance in the blend polymer is higher than that of the pure polymer because of the increase of charge carriers resulted from the overlap of the polymeric chains with each other because of occur strong electrostatic interaction between the functional groups, intermolecular rearrangement and the change in the configuration of the main chain of the two polymer and this leads to the forming for the cross-linked chains of polymers [32– 34]. The increase in absorbance corresponds to a

decrease in transmittance of CMC/PAA blend polymer than pure CMC film, as shown in the figure (12). Due to the occurrence of the process of overlap and an increase in density, this increases the occurrence of the scattering process, which is the main process that affects the transmittance of light, and this leads to a decrease in transmittance [33]. Figure (13) shows the absorption coefficient (α) of pure polymer and blend polymer films, that calculated from eq. (1)[35].

$$\alpha = 2.303 (A/t) \quad (1)$$

Where (A): is the absorption of the material, (t): the sample thickness in (cm).

The absorption coefficient of blend polymer was improved when compared with the pure polymer, which is due to an increase in the number of carrying charges, which led the absorbance to increase[36, 37]. According to the data, the absorption coefficient is less than (10^4 cm^{-1}), indicating that it possesses an indirect energy band. The energy band gap is calculated from Eq. (2)[38].

$$\alpha h\nu = B(h\nu - E_g)^r \quad (2)$$

The photon energy is described as ($h\nu$), and (B) represents the constant that appears to be based on the kind of material, (E_g) act as the energy gap and the exponential constant (r) related to the type of transition. Figure (14) shows the energy band difference of pure polymer and blend polymer films, as shown in Table (5). The increases in the localized levels of forbidden energy band gap lead to an increase in the cases of overlaps between these levels, resulting in a decrease in the energy band gap value. The value of energy gap for CMC polymer is 5.69 eV that was matched with researches [16].

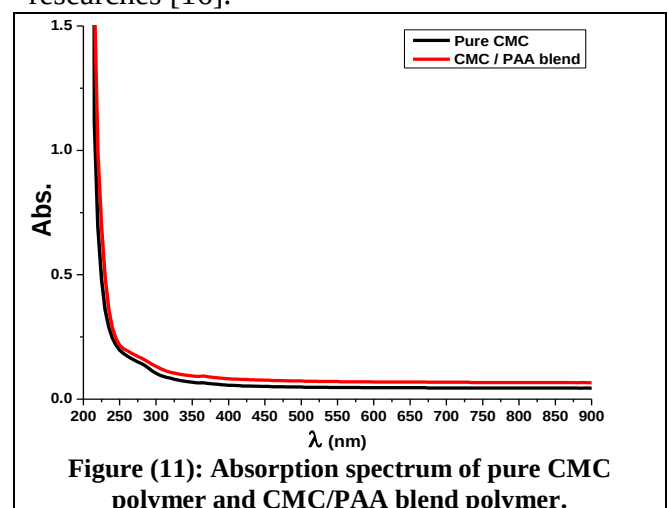


Figure (11): Absorption spectrum of pure CMC polymer and CMC/PAA blend polymer.

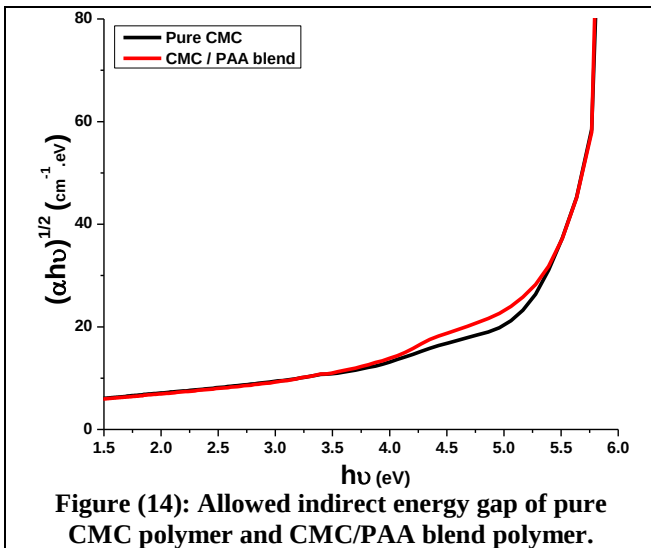
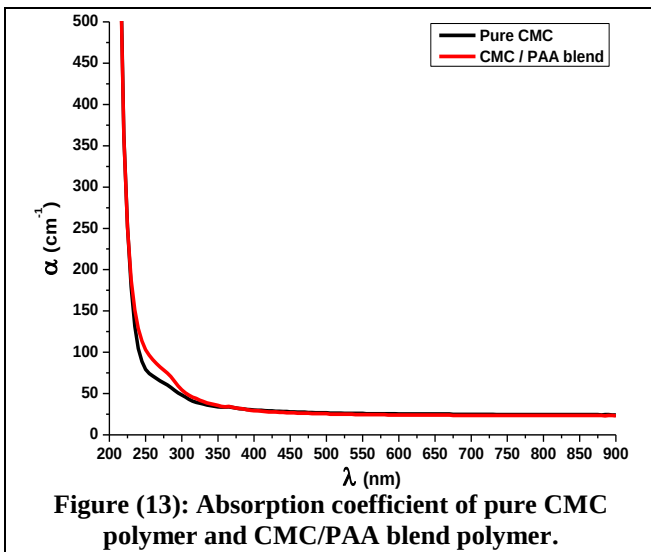
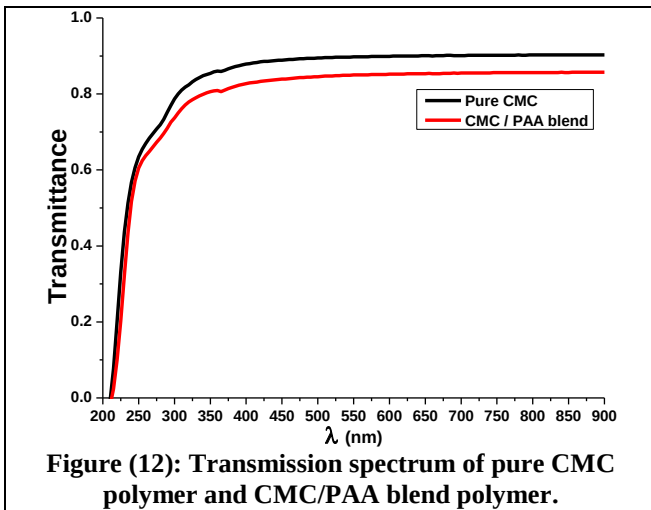


Table (5): Energy band gap value of pure CMC polymer and CMC/PAA blend polymer.

Samples	E_g (eV)
CMC pure	5.69
CMC/PAA blend polymer	5.61

Figures (4-15) to (4-18) show the relation between the extinction coefficient (K), refractive index (n), imaginary (ϵ_i) and real (ϵ_r) dielectric constants, respectively, of pure polymer and blend polymer films that calculated from eqs. (3-6), respectively [39, 40].

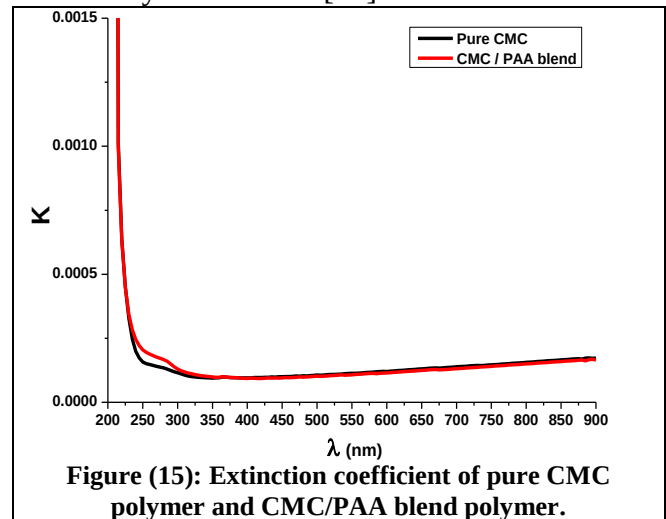
$$k = \frac{\alpha \lambda}{4\pi} \quad (3)$$

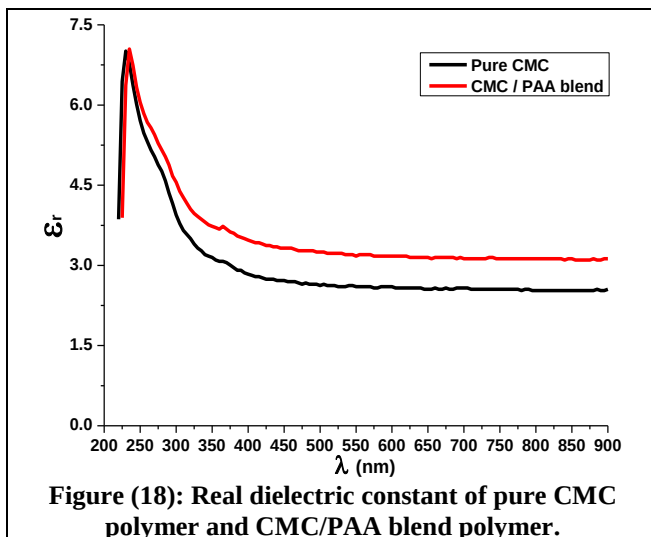
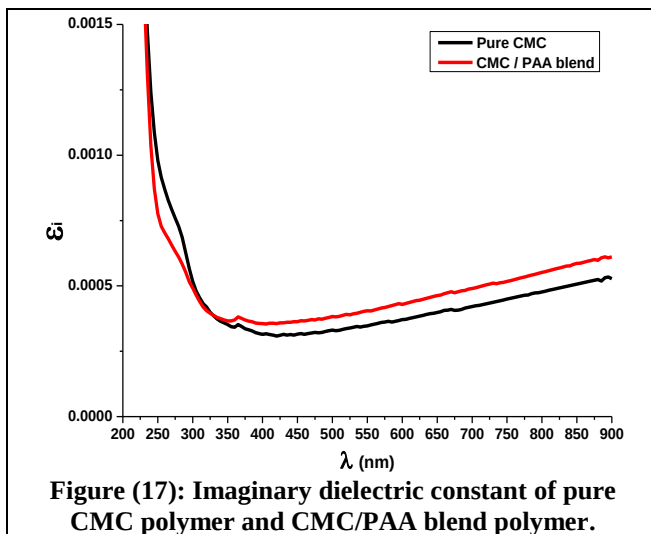
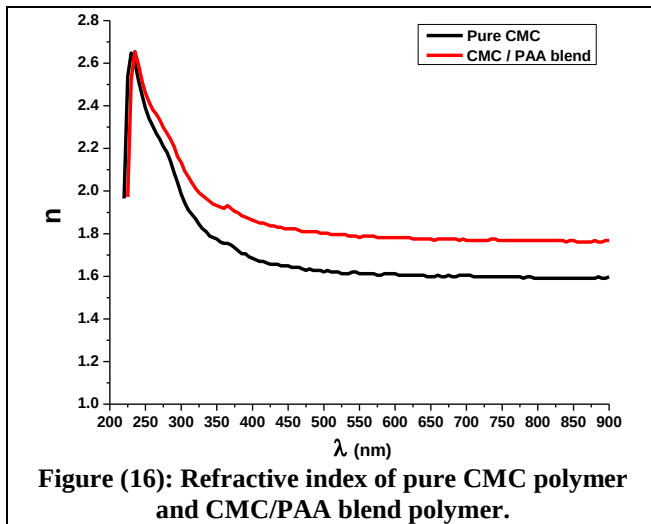
$$n = \frac{\left(1 + R^2\right)^{\frac{1}{2}}}{\left(1 - R^2\right)^{\frac{1}{2}}} \quad (4)$$

$$\epsilon_i = 2nk \quad (5)$$

$$\epsilon_r = n^2 - k^2 \quad (6)$$

Because of its dependence on the absorption coefficient, the extinction coefficient in the blend polymer was higher than that of the pure polymer. Also, the refractive index increases as a result of the increase in the overlap of the polymeric chains with each other and this led to an increase in the density [41]. The imaginary and real dielectric constants increase in blend polymer compared with the pure polymer due to the dependence of the imaginary dielectric constant on the extinction coefficient where with increasing wavelength the extinction coefficient increases while the refractive index is almost constant, while the real dielectric constant depends on the refractive index this is because the extinction coefficient has a very small effect [42].





4. Conclusion

A solution casting method was used to make the pure CMC and CMC (80%)/ PAA (20%) films. The results of XRD patterns indicating that appearance of some peaks of PAA crystalline structure into amorphous

structure of CMC polymer. FESEM image of CMC/PAA film can insure the XRD results by performed of small spherical grains (PAA) into structure of CMC polymer. FTIR spectrum indicated the physical interaction occurrence only. The absorption coefficient, absorbance, real and imaginary dielectric constant, extinction coefficient and refractive index were increased in blend polymer compared with the pure polymer. The energy gap and transmittance of blend polymer compared with the pure polymer were decreased.

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