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Mechanical and Structural Properties of Enhanced Based on Carboxymethyl Cellulose Doped Ammonium Chloride Solid Biopolymer Electrolytes

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ABSTRACT

Solid Biopolymer Electrolytes (SBEs) systems based on carboxymethyl cellulose (CMC) doped ammonium chloride (AC) and varied amounts 4-20wt% of ethylene carbonate (EC) as a plasticizer were prepared via the solution casting technique. The electrical impedance spectroscopy (EIS) method was used to analyse the ionic conductivity of SBEs by room temperature (303K). The sample with 8wt% of EC exhibits the highest ionic conductivity at $4.43 \times 10^{-6} \text{ Sm}^{-1}$. In this study, mechanical, physical and structural of CMC-AC-EC were tested. Fourier Transform Infrared Spectroscopy (FTIR) demonstrated the composite nature and indicated the SBEs components formed a complex with each other, and this complexation was successfully confirmed by Gaussian software. The X-ray diffraction (XRD) revealed that the amorphous peak of CMC become broaden with addition of EC and the crystallinity of AC showed a peak. The UV-Vis spectroscopy tests the transparency value with transmittance at 600nm. The tensile strength (TS) and elongation at break (EAB) can be obtained from the stress-strain graph using the Universal Testing Machine. The morphological behaviour of electrolytes has been analysed by using Field Emission Scanning Electrolytes Spectroscopy (FESEM) to observe changes in CMC based on the solid biopolymer electrolytes when AC and EC are added to the system.

1. Introduction

The world has been using batteries as electrochemical devices to generate electricity from a chemical reaction. Battery consists of electrolyte and ions move between two terminals which are anode and cathode [18]. Basically, electrolytes are divided by three types, liquid electrolytes, gel electrolytes and solid electrolytes. Due to the increasing number of products of batteries that are not eco-friendly and high cost, the need for solid biopolymer electrolyte (SBEs) has become more prevalent.

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The researchers are making discoveries to minimize the impact on the environment by using the solid biopolymer electrolyte (SBEs) because it showed a positive side. It can provide various advantages such as high safety performance, light weight, biodegradable, low cost and flexibility [14] Cellulose is one of the most abundant materials on Earth, this being due to its ability to decompose and one of the bio-polymer used for this purpose. Carboxymethyl cellulose (CMC) has drawn attention in SBEs development and being chosen because of the outstanding advantages due to the lower cost, environmentally friendly manner, high solubility in water and can form a transparent film [19].

Thus, in this research CMC was selected as a polymer host. However, the ionic conductivity of the CMC based electrolyte is between 10^{-10} and 10^{-7} S/cm which is low conductivity. Cellulose based biopolymer electrolyte needs ionic donor to improve conductivity. One of the methods to enhance ionic conductivity is using ionic dopant.

Ammonium chloride (AC) is chosen as the ionic dopant for the polymer electrolyte [12]. The H^+ of the ammonium ion group NH_4^+ from ammonium salt helps to improve the ionic conductivity of the electrolyte system [15]. In addition, Ethylene carbonate (EC) is chosen as a plasticizer because it dissolves a large amount of electrolyte, higher dielectric constant $\epsilon = 89.1$ and higher boiling temperature ($248^\circ C$) compared to other plasticizers [11].

This work aims to develop the mechanical, physical and structural properties of the SBE system by using tensile strength test, UV-Visible Spectroscopy (UV-Vis), Fourier Transform Infrared (FTIR) comparable the computational method via Gaussian software, X-ray Diffraction (XRD) and Field Emission Scanning Electron Method (FESEM).

2. Methodology

2.1 Sample Preparation

2.0 g of carboxymethyl cellulose (CMC) was dissolved in distilled water and stirred until it is completely dissolved. Previously work done by Ahmad *et al.*, (2021), by using 0.381 g of Ammonium chloride (AC) was added into the CMC solution contained the highest ionic conductivity for unplasticized system [20]. This work was a continuity from the previous work. To prepare the CMC-AC-EC, the composition of the highest conductivity of CMC-AC (16wt%) SBEs was added with different weight percentages (4wt.%,8wt.%,12wt.%,16wt. & 20 wt.%) of plasticizer EC. Next, the mixture solution was cast into the petri dish and dried the sample in a desiccator with blue silica gel for film to form. The composition of the samples and their designations are tabulated in Table 1.

Table 1
Designation and compositions of SBEs samples

Designation	Formulation				
	CMC (g)	AC (wt.%)	AC (g)	EC (wt.%)	EC (g)
C0	2	0	0.000	0	0.0000
CA0	2	16	0.381	0	0.0000
CAE4	2	16	0.381	4	0.0159
CAE8	2	16	0.381	8	0.0331
CAE12	2	16	0.381	12	0.0520
CAE16	2	16	0.381	16	0.0726
CAE20	2	16	0.381	20	0.0953

2.2 Sample Characterization

2.2.1 Electrical Impedance spectroscopy (EIS)

The ionic conductivity plays a significant role in the electrical performance in the SBEs. The electrical impedance measurement has been run using HIOKI 3532-50 LCR Hi-Tester over the frequency range 50Hz to 1Mhz at room temperature (303K). The ionic conductivity can be calculated by using the formula [2] in Eq. (1):

$$\sigma = \frac{t}{R_b A} \quad (1)$$

where t (cm) is the thickness, R_b is the bulk resistance that determined from Cole-Cole plots and A is the area of electrolyte — electrolyte in cm^2 . The ionic conductivity (σ) of sample in $S\text{cm}^{-1}$.

2.2.2 Fourier Transform Infrared spectroscopy

Fourier Transform Infrared (FTIR) spectrometer was used to determine the complexation and to confirm the presence of the functional group between CMC-AC-EC salts by using Thermo Nicolet 380 FTIR spectroscopy equipment with Attenuated Total Reflection (ATR). The spectrum was run on the germanium crystal with frequency ranging between 4000 and 650 cm^{-1} with spectra resolution of 4 cm^{-1} .

2.2.3 Computational method

The vibrational properties, frequency calculations and performing geometry optimizations can be used Gaussian 09W program. These optimizations and frequency calculations were made at B3LYP method using 6-31G basis set. The vibration frequency was obtained at Gaussview animation used to support the experimental result from FTIR analysis.

2.2.4 X-ray Diffraction (XRD)

The structure and phase formation of the SBEs samples are determined by the X-ray diffraction (XRD), Rigaku with $\text{Cu K}\alpha$ radiation over the range 2θ about $5^\circ - 80^\circ$. The samples were cut with step size (2cm x 2cm) and placed in the sample holder for the XRD measurement.

2.2.5 Field Emission Scanning Electron Method (FESEM)

Field Emission Scanning Electron Method (FESEM) with Zeiss Supra 55VP microscope was used to capture the microstructure image of CMC-AC-EC solid polymer electrolyte using EHT 5kV. The magnification scale at $10\mu\text{m}$ in 500 magnification was determine the morphological structure of the solid biopolymer electrolyte.

2.2.6 UV-Visible spectroscopy

The UV-Visible of CMC-AC-EC SBEs were measured in the wavelength region of 200 - 1100 nm using a double beam spectrophotometer. The samples were cut into 2cm x 2cm measurements. UV3600Plus for model UV3600 (A12015400261) was used to measure the light transmittance and

absorbance of SBEs. Each transmittance at 600nm was used to calculate the value of samples using the equation transparency value = $-\log T_{600}/t$.

2.2.7 Universal testing machine

The tensile test was conducted by using a universal tensile machine to determine the tensile strength and elongation at break through the stress strain curve obtained from the software from the test. The CMC-AC-EC was cut into the specific dimension of 5cm length and 3.5cm width. Each sample was clamped between grips and force during extension at 20 mm/min.

3. Result and Discussion

3.1 Conductivity Studies

Figure 1 shows the plot of the ionic conductivity of the CMC-AC-EC SBEs system at room temperature. It shows that the CAE 8wt% was the highest ionic conductivity compared to the others due to the addition of concentration of EC. The highest conductivity measured was 4.43×10^{-6} S/cm at 8wt% of EC. This is due to the increasing the degree of salt dissociation which promotes to increment in the ion mobility [3]. The lowest ionic conductivity of EC is at 4wt% obtained at 1.80×10^{-6} S/cm. The increase in ionic conductivity by increasing the concentration of EC, is related to the increase number of mobile charge carrier while decrease in ionic conductivity due to decrease in number of free mobile charge carrier due to overcrowded ions [3].

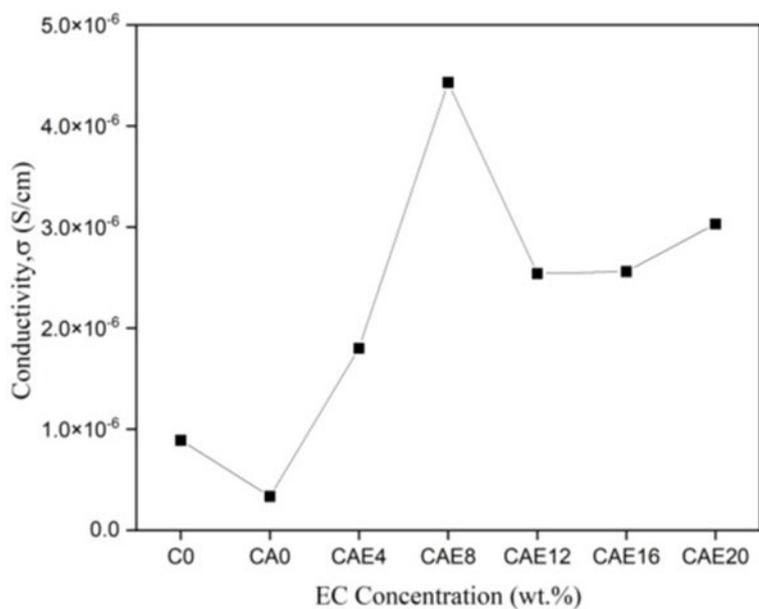


Fig. 1. Room Temperature conductivity for CMC-AC-EC SBEs

3.2 Infrared Spectra (IR) analyses

IR Analysis was performed to determine the functional group and molecular interaction of CMC-AC-EC SBEs. IR analysis was investigated using the computer simulation (Gaussian 09W Software) as computational and for experimental using FTIR technique. Figure 2 shows the C0, pure AC, CA0, pure EC and CAE8 solid biopolymer electrolyte (SBEs). The wavenumber at $3000 - 3500\text{cm}^{-1}$ show the broad absorption bands at 3320cm^{-1} , 3374cm^{-1} , and 3304cm^{-1} correspond to the O-H stretching

bond of water molecule [10]. While the C-H asymmetric stretching band is recorded at 2907cm^{-1} in Figure 2(a). The wavenumber at 1590cm^{-1} was assigned to C=O stretching of CMC structure [12]. 1415cm^{-1} , 1327cm^{-1} and 1055cm^{-1} were assigned to O-H scissoring CMC and C—H bending and C-O stretching vibration [12].

The addition of AC in the CMC SBEs in Figure 2(b) shows the shifting and changes in the intensity of the peak. The narrowing band shows O-H stretching and C-H stretching at 3374cm^{-1} and 2922cm^{-1} after addition of AC concentration. Figure 2(c) and (d) show the addition of EC plasticizer in the concentration of (4,8,12,16,20 wt.%). In contrast, [12] reported that the increasing concentration of EC SBEs, the peak shifted to the lower wavenumber and become broader.

The C=O stretching band was shifted to the lower wavenumber from C0 at 1590cm^{-1} to 1580cm^{-1} in CAE8 due to the interaction between CMC and EC indicates complex complexation [3]. The two peaks assigned at 1414cm^{-1} and 1319cm^{-1} assigned to the O-H stretching and symmetrical stretching of C-H band of CMC respectively. The addition of EC decreased the intensity of the peak. The band at 1056cm^{-1} at CAE8 present the C-O stretching shifted to the higher wavenumber at 1060cm^{-1} correspond to the C-O from HCOO of the EC salt.

The IR spectra experiment result was compared to the Gaussian 09W using scaling factor 0.95. the wavenumber from $300 - 1000\text{cm}^{-1}$ almost the same of infrared between experimental and computational result.

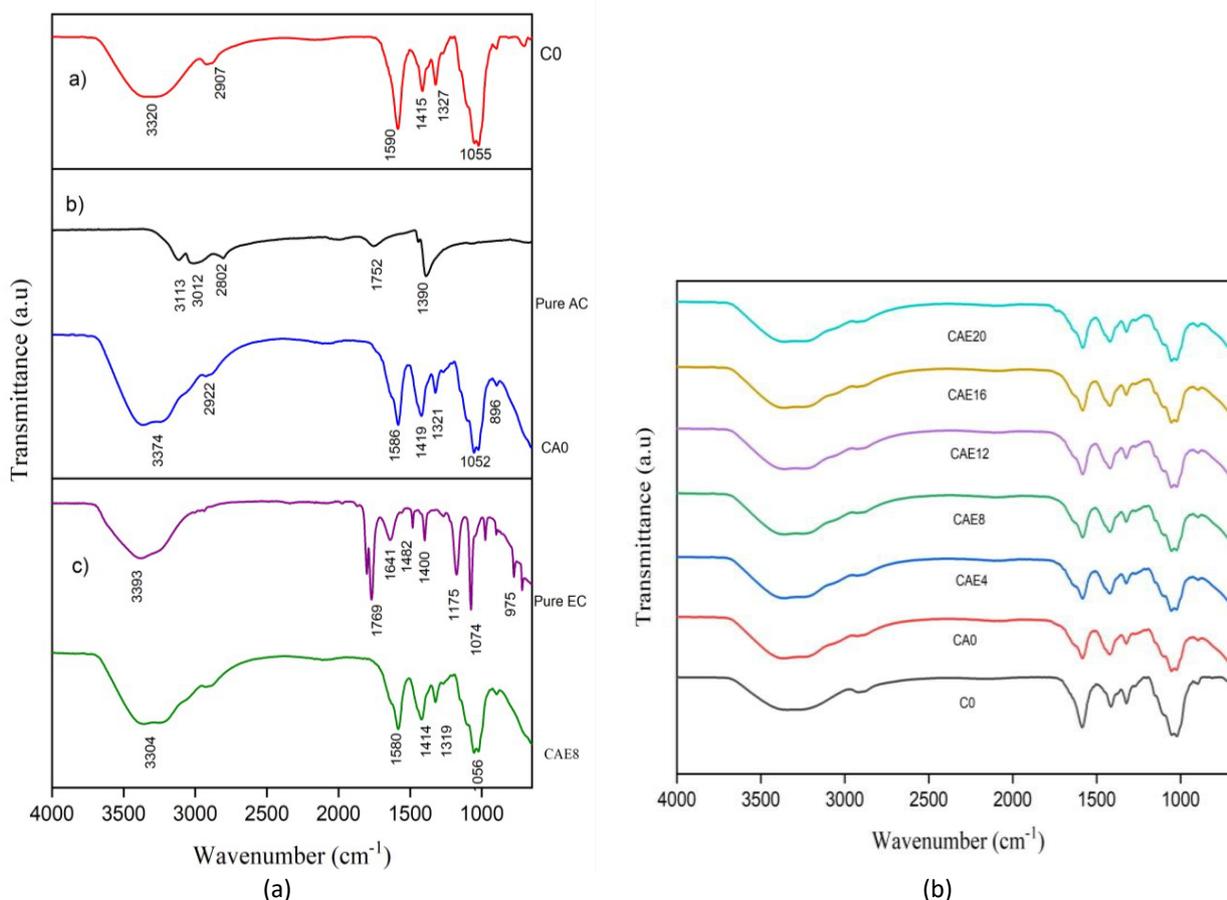


Fig. 2. FTIR Spectrum of (a) C0-pure (b) pure AC and CA0 and (c) pure EC and CAE8 (d) FTIR spectrum of C0, CA0 and CAE (4, 8, 12, 16 and 20)

3.3 X-ray Diffraction

Figure 3(a) showed the XRD pattern for C0 and pure AC. Figure 3(b) showed XRD spectra CMC-AC-EC SBEs with various amount of concentration of EC from 0 — 20 wt.%. The sharp peak at angle $2\theta = 22.92^\circ$ in Figure 4.18 for CAE4 show the crystalline peak from the AC salt appear. After the addition of EC concentration, the crystalline peak disappears and it's become broader until 20wt.% of EC. This clarifies the semi-crystalline structure of CMC. The twin peaks (30.56° (002) and 32.56° (101)) of semi-crystalline nature is due to the AC. It showed the broadened and shifted trend to the higher Bragg's angle. The addition of EC SBEs in this system may decrease the crystalline nature leading to the enhancement of amorphous nature of the film [3].

XRD analysis shows the semi-crystalline peak occur at CMC-AC-EC SBEs of EC concentration from 4 — 20 wt.%. The crystalline peaks were in line at the angle 44.28° is due to the present of C-H attributed to the increasing EC. The observation of crystalline peak increase with the increases of EC into the SBEs is due to the weaker interaction between EC in the CA0 [8]. Ethylene carbonate itself has a tendency to crystallize under certain conditions. When EC is added to CA0, it may form its own crystalline domains within the material. These crystalline domains can contribute to the appearance of semi-crystalline peaks in the XRD pattern. The semi-crystalline phase at CAE4 SBEs at the angle 44.44° and 47.43° show the H₂O chain due to the sample was sticky. The peak at the angle between 60° — 70° showed the broad peak and decrease in the crystallinity of SBEs. The amorphous region increased favorable the ion mobility also increased as strong plasticizing effect [5].

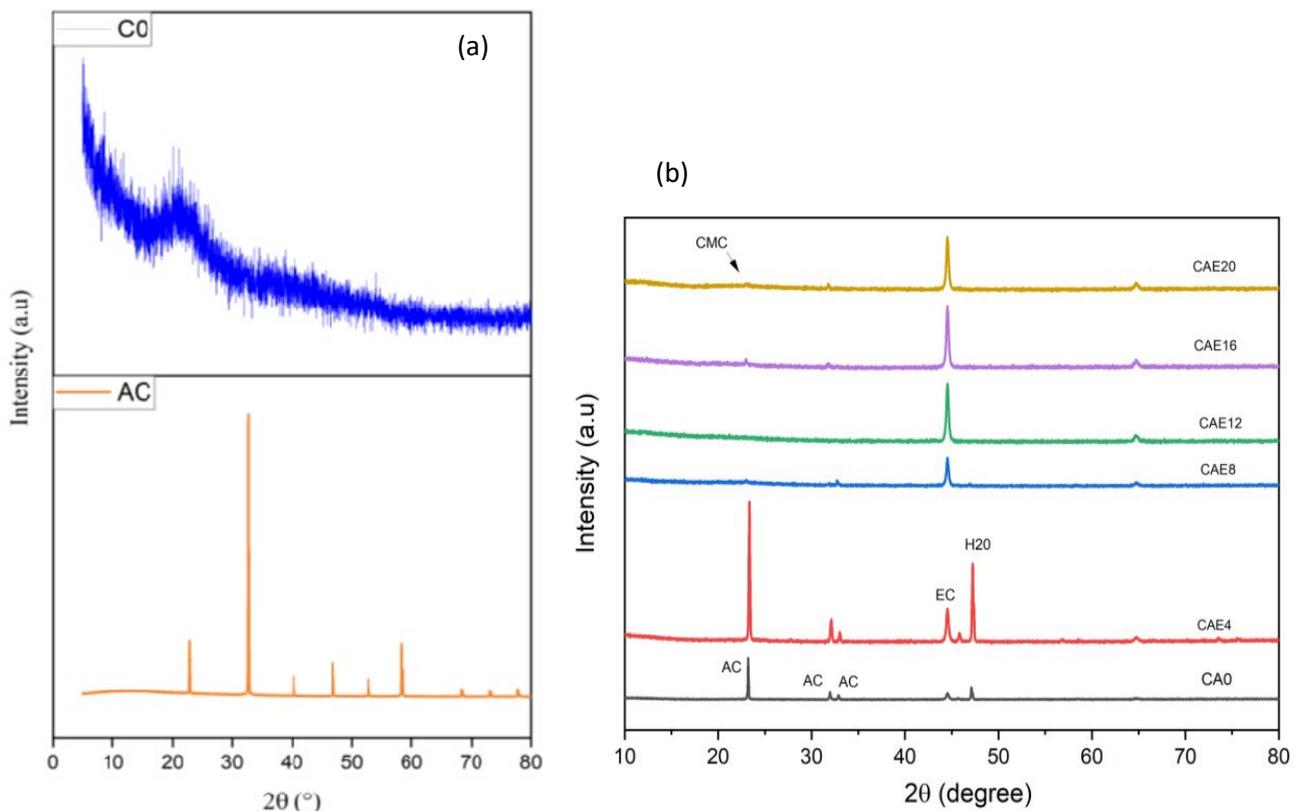


Fig. 3. XRD spectra for (a) C0 and pure AC (b) CAE (0, 4, 8, 12, 16 & 20 wt%)

3.4 Field Emission Scanning Electron Microscopy

Morphological studies were conducted by using Field emission scanning electron microscopy (FESEM) provided a higher resolution imaging capability in monitoring surface morphology. The image was obtained at $10\mu\text{m} \times 100$ magnification to support the XRD finding. Figure 4 represents the image of C0, CA0, CAE4 and CAE8 SBEs. Figure 4(a) is shown the micrograph of C0, it is obvious that the surface has a uniform and smooth surface which were supported to be homogenous [1]. This smooth surface confirms the amorphous nature of CMC and it proves with the XRD result [13]. The surface for the CA0 shows the crystalline polymer where the highly dense crystalline nature of electrolyte. The surface shows the rough surface and agglomerations in Figure 4 (b). The addition of the EC plasticized exhibit surface separation occurs between AC and EC [7]. The addition of EC in CA0 at CAE4 shows reduced roughness in surface and irregularities. Figure 4(d) of 8wt.% of EC show the smoothest and homogenous surface as compared to the 4wt% of EC as the fact that 8wt% EC exhibits a higher ionic conductivity. The EC plasticizer has become more obvious and creating more pathway for ion transportations that can enhanced the diffusion and mobility of AC ions in CMC-AC SBEs.

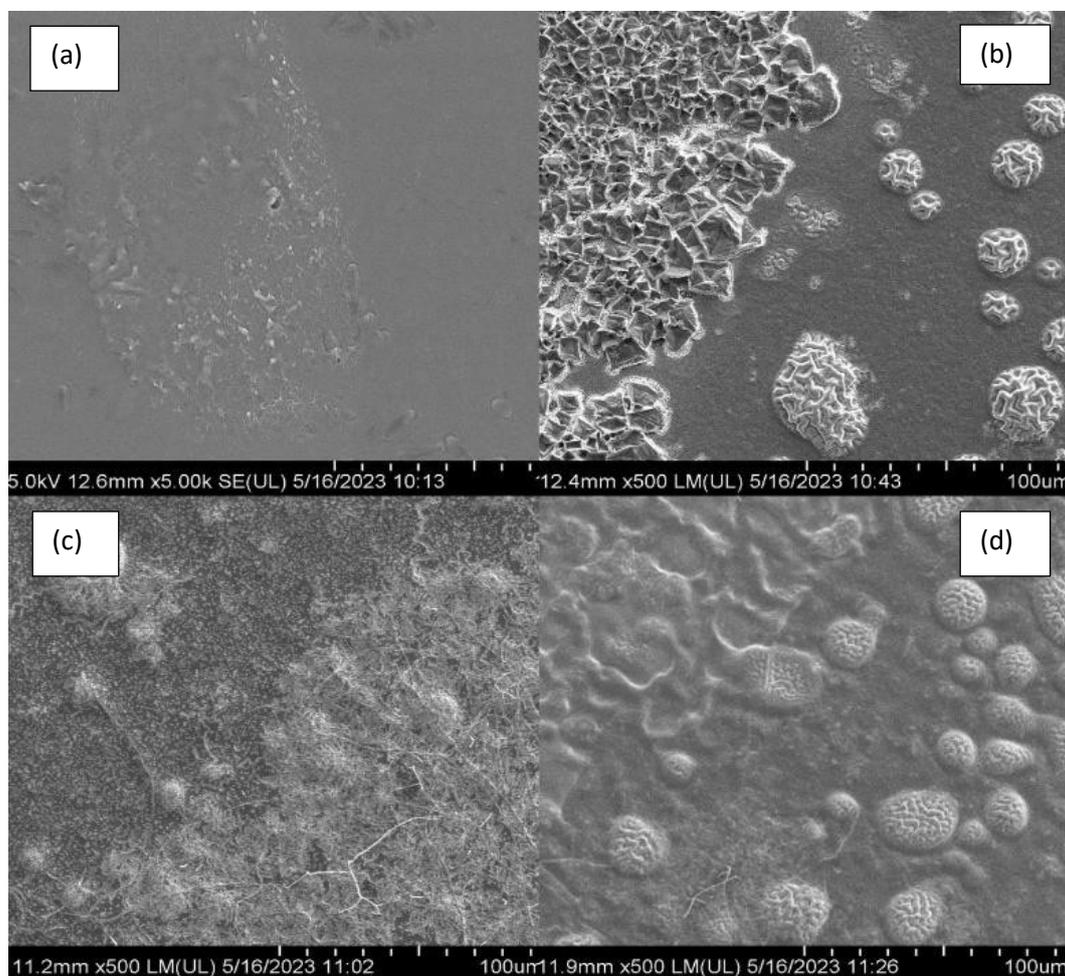


Fig. 4. The Surface Image of (a) C0 (b) CA0 (c) CAE4 and (d) CAE8 SBEs

3.5 Light Transmittance and Transparency

UV-Vis transparency value of all CMC-AC-EC SBEs was presented by Figure 5(b). The highest transparency value showed by C0 with 38.49 because of the smooth and clear surface that pass

through with minimal absorption. Lowest transparency value was observed for CAE20 SBEs with 13.57. The decrease transparency may due to phase separation within the CMC and AC that disrupt the film structure. Among the CMC-AC-EC SBEs that was added with EC, sample with 16wt% showed the highest transparency value with 18.30 due the low thickness. Furthermore, all SBEs samples presented high transmittance with lead to high transparency value as shown in Figure 5(a) and 5(b).

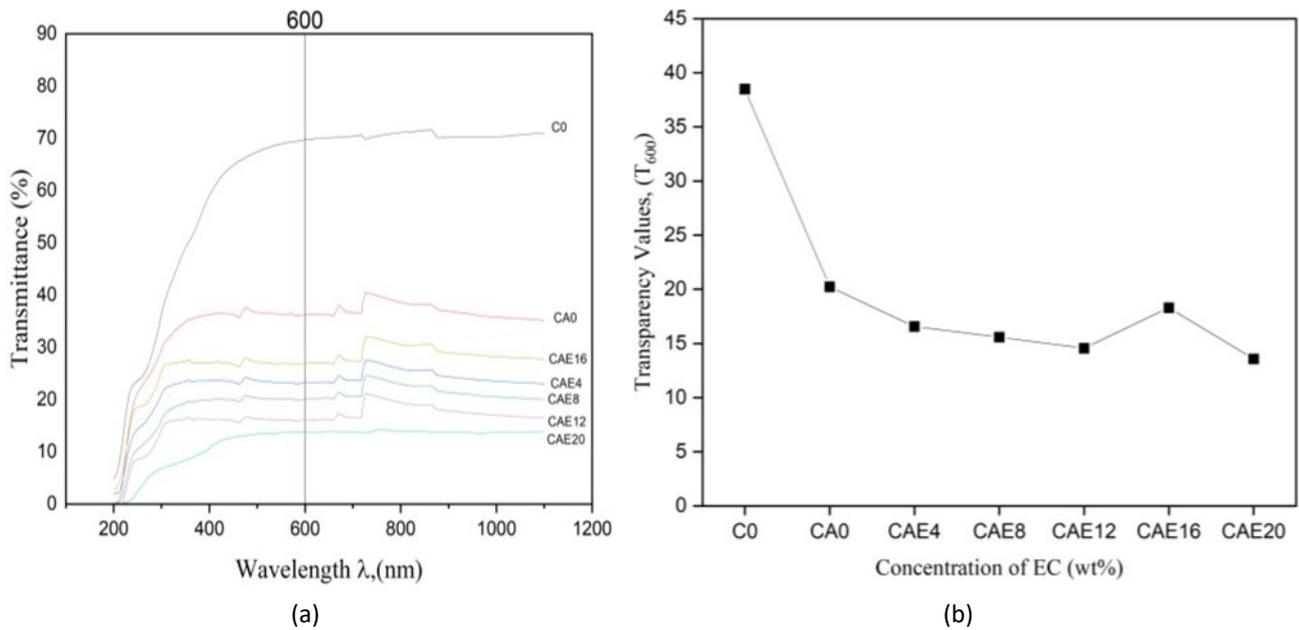


Fig. 5. (a) The transmittance of CMC-AC-EC SBEs (b) The transparency values of CMC-AC-EC SBEs

3.6 Tensile Test

3.6.1 Tensile strength

Mechanical properties of CMC-AC-EC SBEs were determined using the Universal Testing Machine through the Tensile Strength (TS) and elongation break (EAB) with C0, CA0 and different concentration of EC. The TS values for all samples obtained at 55.71 MPa, 1.94 MPa, 4.25 MPa, 1.82MPa, 0.73 MPa, 0.17 MPa and 0.40 MPa respectively. Figure 6 showed there were slightly decrease on TS values as increased of EC percentage from CAE4 to CAE16 due to the surrounding effect. SBEs with 16wt% of EC showed the lowest TS value in Figure 6 at 0.17 MPa. From the calculation in equation 4.5, sample C0 SBEs insert the figure was the highest tensile strength among the others at 55.71 MPa. Sample C0 exhibited higher values of TS due to the sample being brittle and stiff than the others.

3.6.2 Elongation at break

The EAB value has an opposite trend compared to the TS value where the lowest EAB value has the highest TS value in C0. The EAB values for CMC-AC-EC SBEs with different percentage of EC obtained in this study were 10.23%, 35.09%, 24.11%, 17%, 72.87%, 52.11% and 35.85% respectively. The EAB increased as the weight percentage of EC increased in CAE8 and CA0. CAE12 of EC gained the highest EAB value about 72.87% compared to the C0 with 10.23% due to the sample being flexible and highest elastic modulus. The good performance of CA12 might be influenced by the association of ion [6]. The EAB values were decreasing at CA0 and CAE12 show the sample was sticky and easily to break. The differences in EC concentrations could be due to the differences in the formulations of the film-forming and the method for development of SBEs.

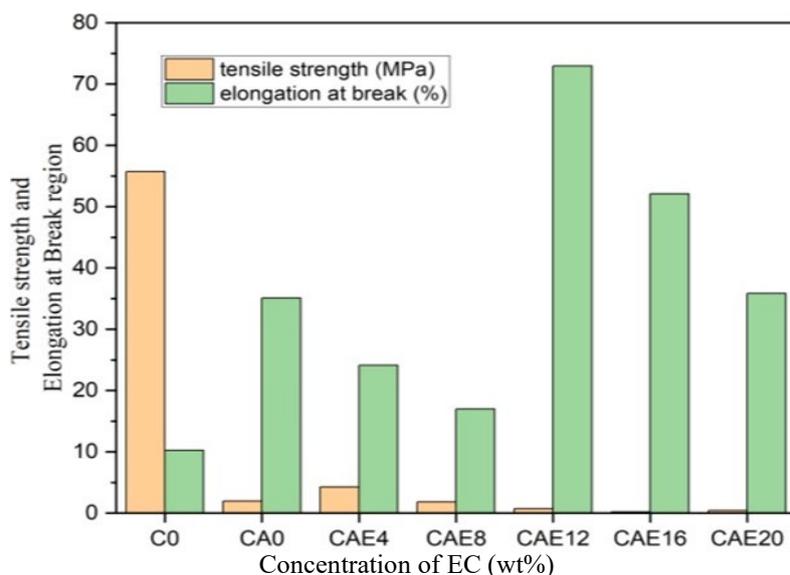


Fig. 6. The tensile strength and elongation at break of CMC-AC-EC SBEs

4. Conclusion

In this study, the CMC-AC-EC SBEs that consist of EC plasticizer with different weight percentages (0, 4, 8, 12, 16 and 20 wt.%) were successfully developed using solution casting technique. The SBEs were appearance in transparent and homogenous in physical apparent. The EIS revealed that the highest ionic conductivity at room temperature (303K) was at $4.43 \times 10^{-6} S cm^{-1}$ of CAE8 SBEs. It shows the addition of EC plasticizer to the CMC-AC system can increase the ionic conductivity and the number of mobile charge carrier. Based on the structural characterization by FTIR, the molecular interaction between the CMC-AC-EC have been studied comparable the experimental result with computational IR spectrum. The presence of O-H, C=O, N-H and C-H stretching peaks were observed and shifted to the lower wavenumber when EC is increased. The XRD measurement confirmed the amorphous region of C0, and some peaks are semi-crystalline. For morphological studies, FESEM image clearly shows the CMC SBEs were homogenous and started to be agglomeration when AC was added. The highest ionic conductivity surface creates a good pathway for ion transportation. For physical properties in optical characterization, the highest value of optical transmittance at 600nm and transparency of C0 is at 64.49 and 38.49. For mechanical characterization, SBEs with 12wt% of EC show the most elastic character due to higher value of elongation at break.

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