

## IONIC CONDUCTION STUDY OF 2 – HYDROXYETHYL CELLULOSE DOPED WITH DODECYLTRIMETHYL AMMONIUM BROMIDE SOLID BIOPOLYMER ELECTROLYTES

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**Abstract:** Green materials of the future 2 – Hydroxyethyl cellulose (2-HEC) doped with dodecyltrimethyl ammonium bromide (DTAB) have been prepared via solution cast technique. The typical XRD pattern shows this complexation has taken place in the amorphous phase. The system with 9wt. % DTAB presented the highest conductivity at room temperature,  $2.80 \times 10^{-5}$  S cm<sup>-1</sup> and ionic conductivity was observed to be influenced by the DTAB concentration. It was found that the conductivity measurement at elevated temperatures follow Arrhenius model and dielectric values were found to increase with increasing temperature, thus indicating the green solid biopolymer electrolytes (SBEs) system to be a non-Debye type. Jonscher's universal power law was employed to obtain the exponent *s* values. The results imply that the conduction mechanism in the most conductive green SBEs system can be represented by small polaron hopping (SPH) model.

KEYWORDS: DTAB, green cellulose, electrical impedance spectroscopy, Jonscher's power law, small polaron hopping.

### Introduction

For a long time, electrochemical devices were powered up using liquid electrolyte system (LES) due to its high conductivity (Sohaimy & Isa, 2015; Samsudin *et al.*, 2012; Saikia *et al.*, 2008). However, the problems continues with LES, e.g., poor electrochemical stability, leakage, and corrosion reactions with electrode (Sohaimy & Isa, 2015; Kim *et al.*, 2013). The probability of electronic conduction also has its own shortcomings. These drawbacks of LES make it less suitable for electrochemical devices. Production of new electrolytes is recommended to tackle the problem so that the new power sources for electrochemical devices can be more efficient (Bashirah & Isa, 2017; Sohaimy & Isa, 2015; Goriparti *et al.*, 2014). The discovery of organic and inorganic new solid materials with better conductivity values ( $10^{-3}$  –  $10^{-2}$  S cm<sup>-1</sup>) renewed interest in solid electrolytes (Hafiza & Isa (a), 2017; Samsudin *et al.*, 2013; Gauthier *et al.*, 1987; Shahi *et al.*, 1983).

Recent work, capability of green, sustainable and biodegradable polysaccharides like cellulose

series, chitosan (Chai & Isa, 2017; Navaratnam *et al.*, 2013; Winie *et al.*, 2013) and starch (Kadir *et al.*, 2014; Ramesh *et al.*, 2011) to be applied as polymer matrix have been renowned due to its richness and abundance in nature (Hafiza & Isa (b), 2017; Yusof *et al.*, 2014; Varshney & Gupta, 2011). In addition, the characteristics of polymer insulator and the low conductivity at ambient temperatures led researchers to find ways to make improvements. Adding dopants, such as ammonium salts or acid is believed to enhance the conductivity of polymer due to the mobile proton ion (H<sup>+</sup>) originated from salt/acid, which loosely bonds and proton exchange can occurs under the influence of an electric field (Kamarudin & Isa, 2015; Klaus-Dieter, 1996). Thus, lead biopolymers doped with salt/acid system is a green proton conductor. Conduction takes place when H<sup>+</sup> from salt/ acid hops via each coordinating site of the polymer host (Kamarudin & Isa, 2015; Shukur *et al.*, 2014; Majid & Arof, 2007).

Current research using cellulose has attracted more attention as a polymer host due to its mechanical stability and biodegradability

(Chai & Isa, 2016; Kamarudin & Isa, 2015; Winie *et al.*, 2013; Kadir *et al.*, 2014). 2 – HEC is a non-ionic rheology modifier derived from cellulose and widely used in cosmetics, coating, medicine, food, textiles etc. Our group is currently working to explore the potential of using 2 – HEC as a polymer host; it is hydrophilic, non – toxic has a low cost and other special properties (Hafiza & Isa (a), 2017; Kamarudin & Isa, 2015) and it also abundant in nature. In this paper, the present work aims to study the conduction mechanism of 2-HEC – DTAB green solid biopolymer electrolytes. The observed temperature and frequency dependence of complex impedance are investigated using the existing theoretical models proposed and the data have never been published elsewhere.

**Material and methods**

2 – Hydroxyethyl cellulose (2-HEC) (average MW = 90,000; DS = 0.7; purity > 99.9%) and dodecyltrimethyl ammonium bromide (DTAB) salt (purity ~ 99%) from Acros Organics and Sigma Aldrich respectively were dissolved with distilled water as a solvent. Green solid biopolymer electrolytes (SBEs) containing 0 – 15wt. % DTAB were prepared via solution cast technique. The desired ratios of polymer: salt were mixed and dissolved homogeneously in distilled water by stirring with magnetic stirrer and bar. The mixtures, then were poured into petri dishes and the solvent were allowed to evaporate in the oven till dry. Finally, green solid biopolymer electrolytes obtained in translucent and flexible. The composition of the samples and their designation are given in Table 1.

Table 1: The composition of sample 2 – HEC-DTAB based SBEs systems

DTAB composition (wt. %)	Designation
0	DTAB_0
3	DTAB_3
6	DTAB_6
9	DTAB_9
12	DTAB_12
15	DTAB_15

Characterization of ionic conductivity and electrical behaviour of SBEs were carried-out by using complex – impedance spectroscopy. Impedance analyser model HIOKI 3522 – 50 LCR Hi – Tester was interfaced to a personal computer in a frequency range between 50Hz to 1MHz. The SBEs were cut into suitable size and sandwiched between blocking electrode of sample holder made up from stainless steel in a MEMMERT oven as temperature controller.

The ionic conductivity was analysed based on frequency dependent electrical properties by using equation explained in previous studies (Bakar *et al.*, 2015; Kadir *et al.*, 2010). Dielectric constant or real part,  $\epsilon_r$ , and dielectric loss or imaginary part,  $\epsilon_i$  have been evaluated from the recorded complex impedance data for each temperature that related to the measured  $Z_p$ , real part of impedance and  $Z_i$ , imaginary part of the impedance for sample as follows:

$$\epsilon_r = \frac{Z_i}{\omega \epsilon_o (Z_r^2 + Z_i^2)} \tag{1}$$

$$\epsilon_i = \frac{Z_r}{\omega \epsilon_o (Z_r^2 + Z_i^2)} \tag{2}$$

where  $C_0 = \epsilon_0 A/t$ ,  $C_0$  is the capacitance of empty measuring cell of electrode area,  $\epsilon_0$  is the permittivity of the free space,  $A$  is the sample-electrode contact area and  $t$  is the thickness of the sample while  $\omega = 2\pi f$  = angular frequency in Hz.

## Results and Discussion

XRD patterns obtained from 2-HEC – DTAB SBEs with varied DTAB concentrations are shown in Figure 1. A broad hump centered at  $2\theta = 20^\circ$  is the amorphous nature of pure 2-HEC. Upon addition of DTAB salt, the broad peaks tend to broaden and slightly shift to  $2\theta$

=  $20.22^\circ$ . The change implies the conductivity increases in amorphous domain of the sample (Samsudin & Isa, 2012; Shuhaimi *et al.*, 2010). However, additional peaks obviously can be seen when DTAB more than 9wt. % at this stage correspond to the UN-dissociates DTAB have been observed and the sample has become more crystalline. Changes in amorphousness of the system contribute to the change of conductivity of samples. This amorphous nature is responsible for greater ionic diffusivity resulting in high ionic conductivity (Shuhaimi *et al.*, 2010). This observation confirms that complexation has taken place in the amorphous phase.

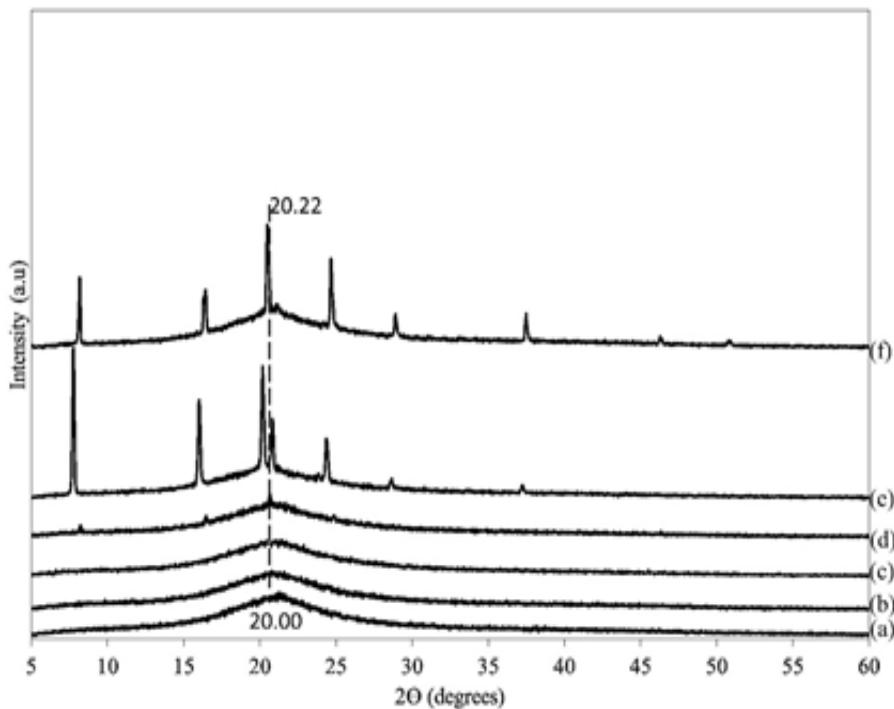


Figure 1: The XRD pattern of (a): 2-HEC with varied amount of DTAB salt concentration, (b) 2 HEC : 3wt. % DTAB, (c) 2-HEC : 6wt. % DTAB, (d) 2-HEC : 9wt. % DTAB, (e) 2-HEC : 12wt. % DTAB, (f) 2-HEC : 15wt. % DTAB.

### Ionic conductivity studies

Table 2 shows the variation of ionic conductivity with different compositions of DTAB at ambient temperature (303K) for SBEs system. The highest conductivity containing 2-HEC: DTAB was identified at  $2.80 \times 10^{-5}$  S/cm for

sample with 9wt. % because more protons ( $H^+$ ) are supplied in the system as composition of DTAB is added (Chai & Isa 2013; Kadir *et al.*, 2010; Winie *et al.*, 2006). It is believed that the function of DTAB in the SBEs system was to provide a new passageway of ions to hop more (Samsudin & Isa, 2015). After

the composition of DTAB increased (greater than 9wt. %), the ionic conductivity reduced gradually considered due to overcrowding of charge carriers, which obstructed the movement of mobile ions (Samsudin & Isa, 2014). The possibility of ion recombination to become a neutral ion pair increased when too much ions

are provided due to decrease the number of free mobile ions (Shukur & Kadir, 2014; Kadir *et al.*, 2010). Moreover, the development of ion aggregates deprive the movement of the free mobile ions (Shukur & Kadir, 2014; Li *et al.*, 2011). These phenomena to some extent reduce the conductivity.

Table 1: The composition of sample 2 – HEC-DTAB based SBEs systems

DTAB composition (wt. %)	Conductivity (Scm <sup>-1</sup> )
0	6.42 x 10 <sup>-7</sup>
3	3.50 x 10 <sup>-6</sup>
6	6.12 x 10 <sup>-6</sup>
9	2.80 x 10 <sup>-5</sup>
12	2.15 x 10 <sup>-6</sup>
15	7.61 x 10 <sup>-7</sup>

Figure 2 depicts the temperature dependence for ionic conductivity in various compositions. The plot are almost linear with regression values close to unity ( $R^2 \approx 1$ ) suggest the Arrhenius behaviour – thermally assisted. The increasing ionic conductivity for all composition in SBEs system with temperature is perhaps due to the availability and mobility of ions (Samsudin & Isa, 2014; McLean *et al.*, 2005) and also perhaps due to the segmental (i.e. polymer chain) motion resulting in an increase in the free volume of the system (Yusof *et al.*, 2014; Sit *et al.*, 2012).

**Conduction mechanism studies**

9 wt. % of DTAB was used for further studies due to the highest ionic conductivity among all samples. The frequency dependence of dielectric constant,  $\epsilon_r$  and loss,  $\epsilon_i$  at various temperatures is shown in Figure 3 refer eq. (1) and eq. (2) to calculate the real and imaginary

parts of the impedance. Dielectric study can give more understanding on the ionic transport phenomenon due to the contribution of interfacial effects and bulk material at the separable electrode. Observations made on  $\epsilon_r$  and  $\epsilon_i$  show the same array where all plots rise towards high temperature, but then decrease as the frequency increases.

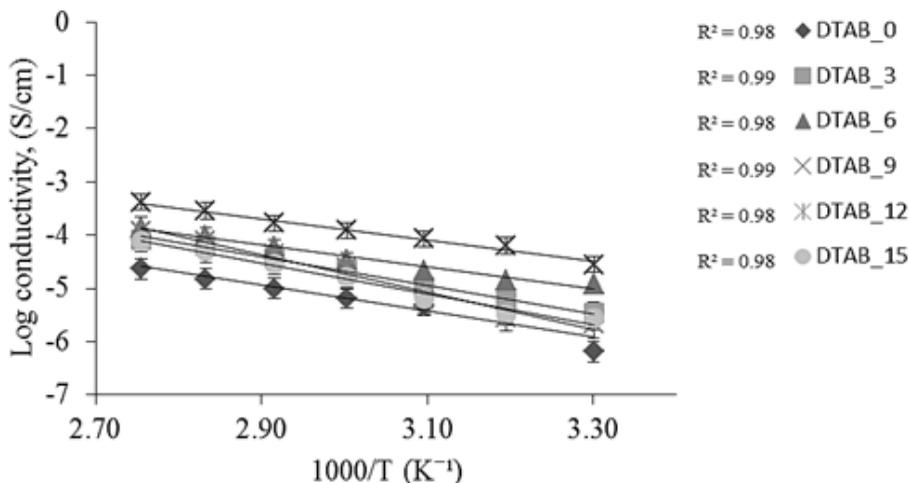


Figure 2: Temperature dependence for ionic conductivity of 2 – HEC – DTAB systems

At the low frequency,  $\epsilon_r$  and  $\epsilon_i$  values are highly probable due to the enhanced charge carrier density at the interface of electrode – electrolyte or equally means due to the charge species that accumulate at the electrode – electrolyte interface (Chai & Isa, 2012; Kufian *et al.*, 2007). These provide evidence that increase temperature will increase the dissociation degree of DTAB which increase the numbers of free ions, thus enhancing the conductivity of the system electrode polarization and space charge effects have occurred verifying non – Debye dependence (Chai & Isa, 2011; Khiar *et al.*, 2006) implying that conductivity exhibits relaxation that is non – exponential with time.

On the other hand, as frequency increased, the rate of reversal of the electric field increased, and so there was no time to charge to build up at the interface. Hence, the polarization due to charge accretion decreased that leading to the decrease in the value of dielectric loss (Khiar & Arof, 2010). No relaxation peaks are observed in both  $\epsilon_r$  and  $\epsilon_i$  signifying it possible used as the indicator of increasing in conductivity that mostly attributed to the increasing free mobile ions (Rashba, 1959).

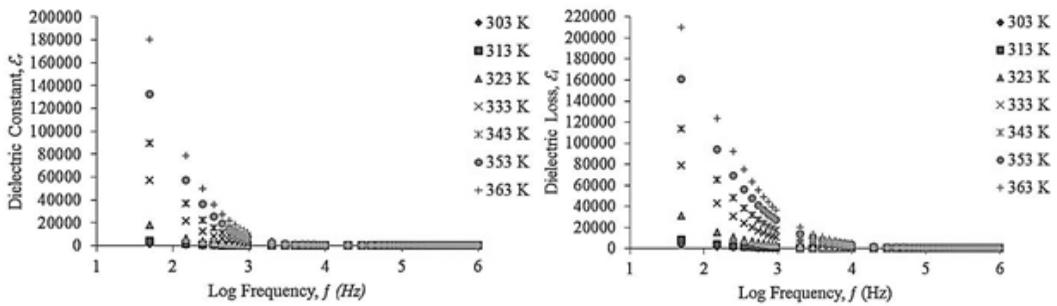


Figure 3: Dielectric constant and loss for 9 wt. % of DTAB at various temperatures

Plot of  $\log \epsilon_i$  versus  $\log \omega$  for 9wt. % of the green SBEs system at selected temperatures is shown in Figure 4.  $\log \epsilon_i$  was observed is experience high values at low frequencies ( $\log \omega$ ) that can be linked to charge accumulation. The increasing values of  $\epsilon_i$  as temperatures increase is probably due to the accretion of mobile ions causes by DTAB in SBEs system (Chai & Isa, 2012; Bonca *et al.*, 1999). Equation expressed

by Jonscher’s universal power law model (UPL) which explained an ion jump from one site to its neighbouring vacant site at lower frequency may contribute to the dc conductivity of SBEs and the probability to go back to its initial site is best described by other researchers (Hafiza & Isa (a), 2017; Kamarudin & Isa, 2015; Pradhan *et al.*, 2008).

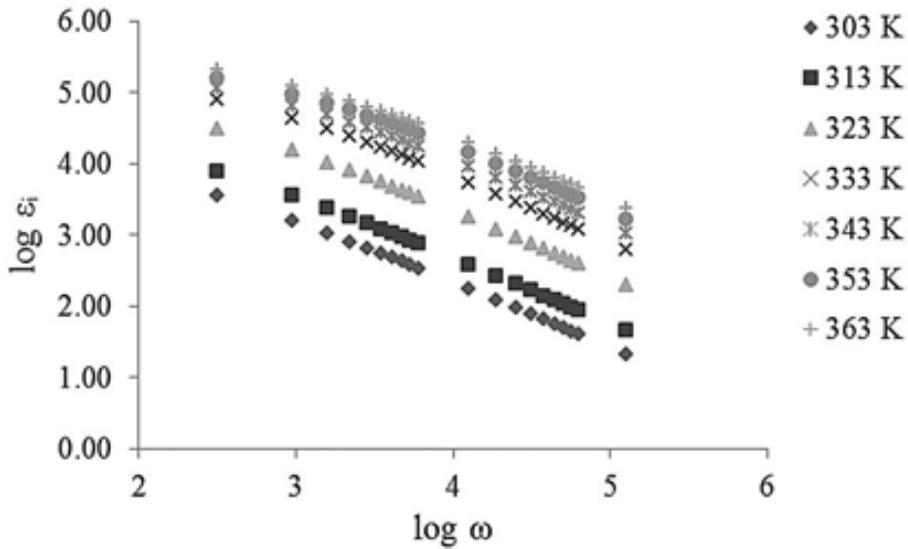


Figure 4: Plot of  $\log \epsilon_i$  versus  $\log \omega$  for sample 9 wt. % of DTAB

The a.c conductivity can be obtained from plot of dielectric loss at every frequency expressed by:

$$\sigma(\omega) = \sigma_{dc} + \sigma_{ac} \quad (3)$$

$$\sigma_{ac} = \epsilon_o \epsilon_i \omega \text{ by substituting } \epsilon_i = \epsilon_r \tan \delta \quad (4)$$

$$\sigma(\omega) = \sigma_{dc} + A\omega^s \quad (5)$$

$$\sigma_{ac} = A\omega^s \quad (6)$$

where  $\sigma(\omega)$  is total d.c and a.c conductivity,  $\sigma_{dc}$  is d.c. limit of conductivity,  $\sigma_{ac}$  is a.c. limit conductivity, A is a parameter dependent on

temperature and  $s$  is the power law exponent with values in the ranging from 0 to 1. From eq. (4) to eq. (6):

$$\log \epsilon_i = \log \frac{A}{\epsilon_o} + (s - 1) \log \omega \quad (7)$$

From the plot of  $\log \epsilon_i$  versus  $\log \omega$ , value of  $s$  can be attained via the slope at higher frequency area where there is no or minimal space charge acquired in SBEs system. The rudiment of  $s$  by temperature suggests the conduction mechanism

of the mobile species (Hafiza & Isa (b), 2017; Kadir *et al.*, 2010; Winie *et al.*, 2006). The variation of  $s$  with temperature is plotted in Figure 5.

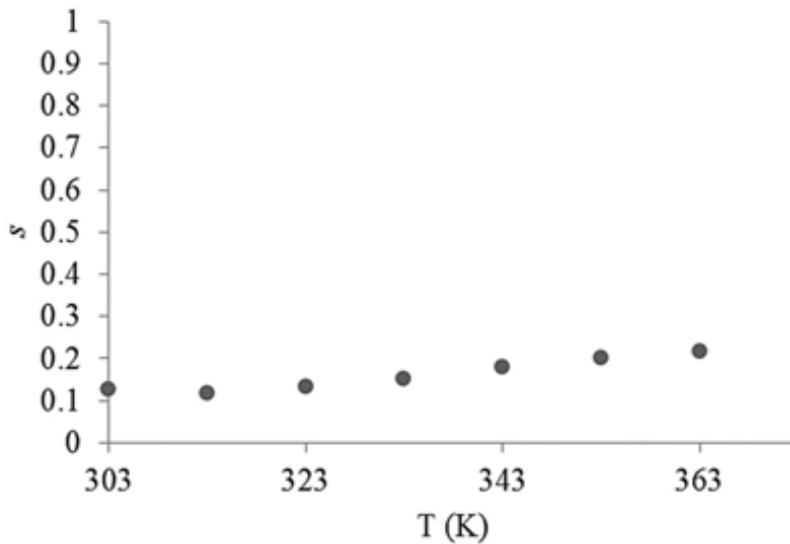


Figure 5: Variation of exponent  $s$  versus temperature

Figure 5 shows changes in exponent  $s$  determined from eq. (7) which increases with increasing temperature, suggesting the frequency dependence of system follow the small Polaron hopping (SPH) model which is caused by non-adiabatic mechanism. It can be observed that the exponent  $s$  values are almost 0.2 and independent of temperature. Polaron hopping refers to charge carriers that jump between localized sites due to the influence of atomic thermal vibration of molecules (Moiz *et al.*, 2012).

### Conclusion

2-HEC biodegradable cellulose series doped with DTAB green solid biopolymer electrolytes (SBEs) were successfully prepared via solution casting technique. XRD pattern shows that complexation has taken place in the amorphous phase. The highest ionic conductivity of 2-HEC-DTAB SBEs system found at 9wt. %,  $2.80 \times 10^{-5}$  S/cm. Temperature dependence of SBEs system unveils Arrhenius behavior which is the regression value nearly to 1 and exclusively influenced by temperature and wt. % of DTAB. The ac conductivity and conduction mechanism for this system could be best described by small polaron hopping (SPH) as the ideal hopping model through non-adiabatic mechanism.

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