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Rheological characterizations of ionic liquid-based gel polymer electrolytes and fumed silica-based composite polymer electrolytes

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Abstract

In this work, two different polymeric systems were investigated. Ionic liquid-based gel polymer electrolytes (IL-GPEs) are designated as the first system and composite polymer electrolytes (CPEs) with addition of nano-sized fumed silica (SiO_2) are assigned as the second system. From amplitude sweep and oscillatory shear sweep tests, it was found out that the values of storage modulus (G') are much higher than loss modulus (G''), indicating the elastic behavior of these samples. No behavioral transition of samples was observed in the tests. Linear visco-elastic range (LVE) of samples becomes wider with increasing content of ionic liquid and SiO_2. Long-term structural stability was enhanced upon addition of ionic liquid. In the frequency sweep test, the absence of G'' further confirmed the solid properties of samples. The inclusion of SiO_2 did not show any improvement in the frequency sweep test. The zero-shear viscosities were reduced upon inclusion of ionic liquid and SiO_2. In the viscosity curve, the decrement of viscosity was shown as increase in shear rate due to the structural changes.

Keywords: Rheological behavior; BmImTFSI; SiO_2

1. Introduction

In recent years, many researchers have shown keen interest in ionic liquid-based gel polymer electrolytes. In general, ionic liquids are composed of bulky organic cations and inorganic anions [1]. These room temperature molten salts have low melting point with some attractive characteristics. These inherent properties are wide electrochemical potential window, relatively high ionic conductivity, non-volatility, non-flammability, negligible vapor pressure, excellent thermal and chemical stabilities as well as high ability to solubilize both organic and inorganic compounds [2–4]. However, the mechanical stability of ionic liquid-based polymer electrolytes may not reach the desirable level. Therefore, inorganic reinforcement filler is introduced to enhance the mechanical strength. This results the formation of composite polymer electrolytes (CPEs). In this work, fumed silica (SiO_2) is embedded into the polymer matrix to improve mechanical properties of polymer electrolytes. Fumed silica is a hydrophilic material prepared through vapor phase hydrolysis of silicon tetrachloride ($SiCl_4$) through thermal combustion in a hydrogen–oxygen flame atmosphere. Formation of three-dimensional network is originated by its native surface group on the fumed silica through hydrogen bonding and Van der Waals attractive forces. This native surface group is so-called as silanol (Si–OH) [5,6].

Upon addition of ionic liquid, the polymer electrolytes show the gel appearance. The viscosity of these gel polymeric systems would be decreased as ionic liquid manifests low viscosity. As aforementioned, fumed silica forms hydrogen bonds and Van der Waals of native Si–OH on the surface of fumed silica. However, these interactive bonds may be partially replaced by other functional group. An internal network structure which may result from physical bonds (known as physical gel) is formed through hydrogen bonding and/or Van der Waals attraction of native or functional surface groups when fumed silica is incorporated in the polymer matrix. This type of gel is able to flow under shear by disrupting the physical bonds between the fumed silica and polymer matrix. However, the physical bonds reform when the shear is removed [5]. Therefore, the physical properties of polymer electrolytes are the main features in this work.

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Among all the physical tests, rheology is a reliable and unique study on the physical behavior of polymer electrolytes. Chen et al. synthesized 1-butyl-3-methylimidazolium bis {(trifluoromethyl)sulfonyl} imide, [C$_4$mIm] [NTF$_2$] ionic liquid [7]. The shear viscosity of ionic liquid is decreased with increasing temperature over a temperature range of 20–90 °C. The linear relationships between shear rate and shear stress are also obtained at 25 °C and 40 °C. It is indicative of Newtonian behavior of ionic liquid [7]. Another approach by Ahmad et al. [8] studied the effect of dispersion of fumed silica concentration in PMMA–LiClO$_4$–PVC gel polymer electrolytes. These composite polymer electrolytes were found to show higher thermal stability with excellent rheological properties.

2. Experimental

2.1. Materials

In this study, poly (methyl methacrylate) (PMMA) with an average molecular weight of 350,000 g mol^{-1} (Aldrich), high molecular weight of poly (vinyl chloride) (PVC) (Fluka), inorganic dopant salt lithium bis(trifluoromethylsulfonyl imide) (LiTFSI) (Fluka), ionic liquid 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl imide) (BmImTFSI) (Sigma–Aldrich), nano-sized inorganic filler, SiO$_2$ with the particle size 7 nm (Sigma–Aldrich) and solvent tetrahydrofuran (THF) (J.T. Baker) were used without further purification.

2.2. Sample preparation

Prior to the preparation of the polymer electrolytes, LiTFSI was dried at 100 °C for 1 h to eliminate trace amounts of water in the material. Appropriate amounts of PMMA, PVC, LiTFSI and BmImTFSI or SiO$_2$ were dissolved in THF. The weight ratio is expressed as weight percentage. Two polymeric systems are employed in this study. Table 1 shows the designations of BmImTFSI-based gel polymer electrolytes in the first system. Different weight fraction of SiO$_2$ was added in the second system and tabulated in Table 2. The solution was continuously stirred for 24 h to obtain a homogenous mixture at room temperature.

2.3. Characterizations

Rheological measurements were performed on Anton-Paar Physica MCR 301 rheometer. The geometry was a cone plate with diameter of 60 mm and gap height of 0.056 mm. All the rheological measurements were carried out at ambient temperature with a fresh sample each time. All the parameters were in logarithm scale. Amplitude sweep studies were examined in a log strain ramp from 0.001% to 150% with 5 points per decade at angular frequency of 10 s^{-1}. For time setting, 30 measuring points with 5 s duration had been set in the whole measurement. All the data acquisitions for frequency sweep tests were carried out over a frequency range between 0.01 s^{-1} and 0.1 s^{-1} with 20 measuring points per 5 s duration. A fixed shear rate parameter, ranging from 0.1 s^{-1} to 100 s^{-1}, with 32 measuring points per 5 s duration was recorded as the profile of viscosity curves.

The sample was sandwiched between stainless steel cone plate geometry and a stationary bottom plate and excessive sample is trimmed thereafter. Solvent trap was used to slow down the evaporation process of solvent in the sample. Before starting the test, about 1 min of rest period was allowed, in order to stabilize the normal forces between the polymer electrolytes and the cone plate.

3. Results and discussions

3.1. Amplitude sweep

Amplitude sweep (AS) is the major part of the oscillation test in rheological study. It is used to determine the visco-elastic behavior and establish the linear visco-elastic (LVE) range throughout the imposed deformation of sample without interruption of internal structures [9]. When a shear is applied onto the sample, the polymer system is being deformed. This produces a strain which is defined as the change in the dimensions of the sample. The visco-elastic properties are governed by two important features viz., storage modulus (G') and loss modulus (G''). G' is a measure of elastic energy, whereas G'' is the dissipation of viscous energy. In other words, the solid and liquid properties of polymer electrolytes are measured by G' and G'', respectively. Both storage and loss

Table 1
Weight compositions of PMMA–PVC–LiTFSI and BmImTFSI for first polymer electrolyte system and their designations.

Designation	Composition of PMMA–PVC–LiTFSI: BmImTFSI					
	PMMA–PVC–LiTFSI				BmImTFSI	
	Weight (g)			Weight percentage (wt%)	Weight (g)	Weight percentage (wt%)
	PMMA	PVC	LiTFSI			
IL 0	0.490	0.210	0.300	100	–	–
IL 2	0.392	0.168	0.240	80	0.20	20
IL 5	0.245	0.105	0.150	50	0.50	50
IL 6	0.196	0.084	0.120	40	0.60	60

Table 2
Weight compositions of PMMA–PVC–LiTFSI–BmImTFSI and SiO_2 for second polymer electrolyte system and their designations.

Designation	Composition of PMMA–PVC–LiTFSI–BmImTFSI: SiO_2						
	PMMA–PVC–LiTFSI–BmImTFSI					SiO_2	
	Weight (g)				Weight percentage (wt%)	Weight (g)	Weight percentage (wt%)
	PMMA	PVC	LiTFSI	BmImTFSI			
CPE 1	0.1921	0.0823	0.1176	0.5880	98	0.02	2
CPE 3	0.1842	0.0790	0.1128	0.5640	94	0.06	6
CPE 4	0.1803	0.0773	0.1104	0.5520	92	0.08	8

moduli are expressed as:

$$G\prime = \left(\frac{\tau_0}{\gamma_0}\right)\cos\delta = G^*\cos\delta \tag{1}$$

$$G\prime\prime = \left(\frac{\tau_0}{\gamma_0}\right)\sin\delta = G^*\sin\delta \tag{2}$$

where τ_0 is stress amplitude; γ_0 is strain amplitude; δ is the phase angle between the stress and strain (or known as loss angle) within $0^\circ < \theta < 90^\circ$ and $G*$ is complex modulus which defined as the resistance for deformation process of a sample.

3.1.1. First system

Fig. 1 shows a typical plot of G' and G''as a function of strain for GPEs with different proportion of ionic liquid. The values of G' are much greater than G'' for all the samples over the range of strain. This indicates the solid properties of all the samples within the range. For all the samples, the crossover point of G' and G'' (usually known as yield point or flow point) is not visible. This implies the absence of behavioral transition of the sample upon the applied shear. Similar trends are observed for all the samples. Two distinct regions are attained within the range. Initially, a constant range known as LVE range is observed. This LVE range is defined as the stability range of the sample. Beyond this range, the values of G' and G'' decrease again due to the breakdown of the physical bonds within the polymer matrix upon the deformation, resulting the reduction of mechanical properties of polymer electrolytes.

Upon addition of ionic liquid, the values of G' and G'' are decreased. It suggest that the ionic liquid favors to weaken the interactions between the polymer matrices by forming the hydrogen bonding of TFSI anions with polymer blends (represented by dashed line), as shown in Fig. 2. At 0.001% of strain, IL 0 and IL 2 exhibit comparable G' and G'' values. It is mainly attributed to the lesser amount of ionic liquid and therefore the effect being not so apparent. These values decrease further with increasing BmImTFSI content. This thus confirms the interruption of the transient coordination bonds within the polymer matrix with increase in BmImTFSI concentration, asserting the lower mechanical stability of polymeric system. Another perspective is the plasticizing effect of ionic liquid. This effect softens the polymer backbone and thus weakens the interactions between the polymer systems. Even though the mechanical properties of polymer electrolytes are lower, however, it still discloses the elastic behavior of samples. The gel appearance of samples possesses advantage in electrochemical device designing for providing a good contact between electrode and electrolyte. As shown in Fig. 1, LVE range increases with BmImTFSI loadings. This indicates that the stability of polymer electrolytes increases with ionic liquid

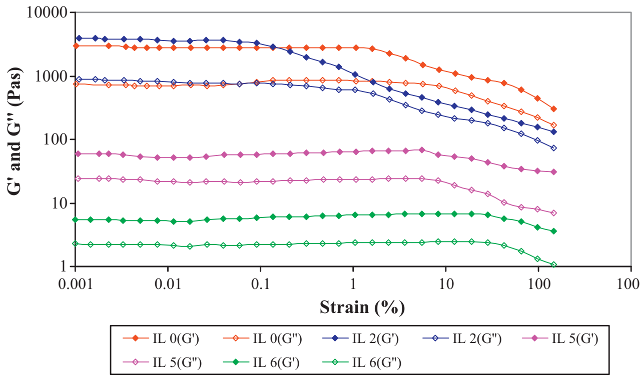

Fig. 1. Amplitude sweep for first system.

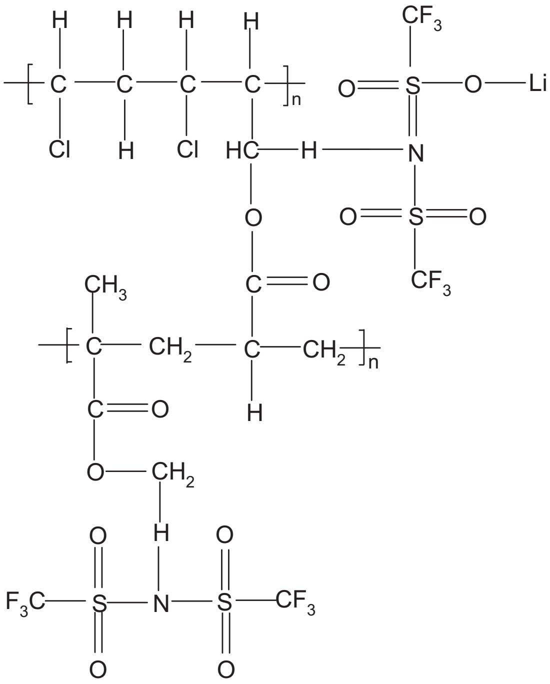

Fig. 2. The interaction between TFSI anions from BmImTFSI and polymer matrix through formation of hydrogen bonding.

compositions. Since IL 6 illustrates the widest LVE range, it can be concluded that IL 6 is the most stable polymer electrolyte.

3.1.2. Second system

The amplitude sweep of second system is shown in Fig. 3. For all the samples, the G' values are higher than G'' in the strain regime, inferring the solid properties of all the samples. The behavioral transition of the polymer electrolytes is not observed within the range as the crossover point of G' and $G''$is absent. The trend of plots is similar as the first polymer system. A constant LVE range is observed in the initial region followed by an abrupt decrease at high imposed deformation. As can be seen, CPE 1 shows the narrowest LVE range among all the composite polymer electrolytes. This indicates the least stable of CPE 1 compared to other samples.

At 0.001% of strain, the values of G' and G'' are substantially increased upon addition of fumed silica. It is suggestive of establishment of three-dimensional network among the SiO_2 particles. This three-dimensional network arises from the formation of silanol (Si–OH) linkages on the surface of silicon atom of SiO_2 through Van der Waals attractive force and hydrogen bonding as it is a hydrophilic compound [5,6]. More physical reticulate structures would be formed with increase in SiO_2 composition, which manifests higher G' and G'' values theoretically. However, the result is against with the theory. This observation might be due to the formation of space charge layers as a result of repulsion forces between the SiO_2 particles. At high SiO_2 content, these space charge layers induce to the longer distances between the SiO_2 grains and further weaken the physical networks. As shown in Fig. 4, the TFSI anions prefer to form hydrogen bonding (represented by dashed line) with the network between SiO_2 grains and thus weaken the interaction among the aggregates. Among all the composite polymer electrolytes, CPE 4 exhibits the lowest values in both moduli and hence denotes the lowest viscosity of CPE 4.

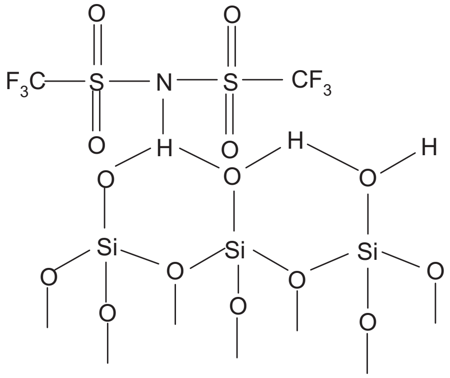

Fig. 4. Formation of hydrogen bonding between TFSI anions and SiO_2.

3.2. Oscillatory stress sweep

3.2.1. First system

Fig. 5 depicts the variation of G' and G'' as a function of shear stress. Again, the values of G' are higher than G'' in the range and this discloses the elastic behavior of the samples. The crossover of G' and G'' is absent in all the samples and signifies that there is no behavioral transition in the polymer electrolytes within the range. Again, the same pattern of plots is observed. As shown in Fig. 5, the starting shear stress of IL 6 is the lowest and this implies that IL 6 sample has the lowest mechanical properties with lowest viscosity. It is in good agreement with Section 3.1.1.

On the contrary, IL 2 manifests the highest value of shear stress in comparison to all the ionic liquid-based GPEs. IL 2 requires more stress for deformation and thus discloses the higher mechanical stability of IL 2. This is strongly correlated to the interruption of coordination bonds within polymer matrix by BmImTFSI, as explained in Section 3.1.1. The weakening of the bonds reveals the lower values of G' and G''. In other words, the physical and chemical bonds within the polymer matrix is more easily deformed with increasing ionic liquid mass fraction as ionic liquid weakens the entanglements in the polymer system. At high shear stress, there is an abrupt decrease in G' and G'' for IL 2. This decrement divulges the unstable properties of IL 2 at high shear stress. Although the mechanical integrity of IL 6 is the lowest, however, it shows higher stability at high deformation by demonstrating the small changes in G' and G''.

3.2.2. Second system

The oscillatory shear curves of second system are exemplified in Fig. 6. The higher values of G' than G'' propose the solid nature of the samples within the range of shear stress. The behavioral transition does not occur as the flow point is absent in all the samples. Two distinct stages are observed and

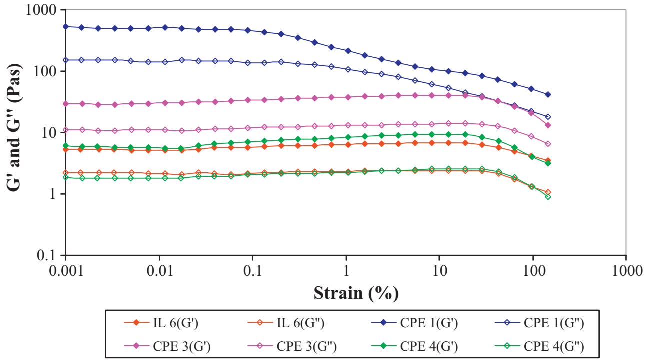

Fig. 3. Amplitude sweep for second system.

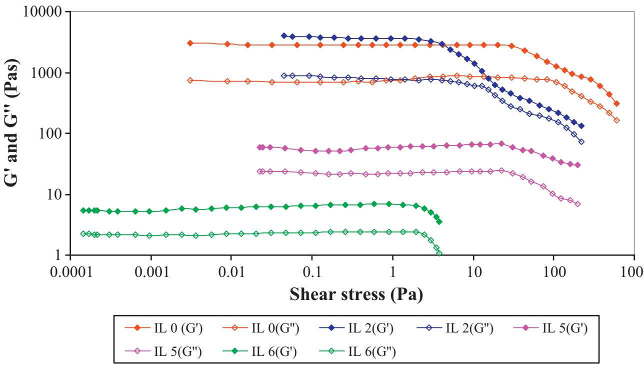

Fig. 5. Oscillatory shear sweep for first system.

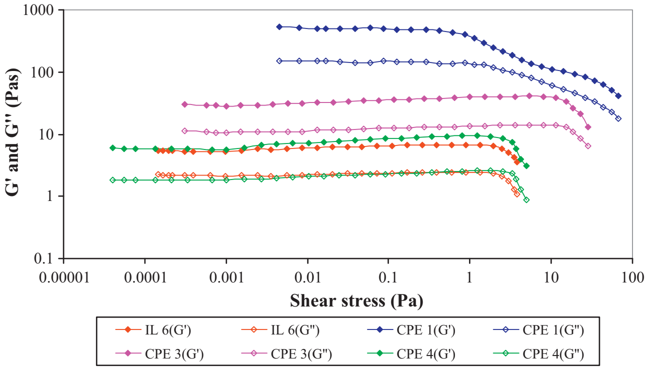

Fig. 6. Oscillatory shear sweep for second system.

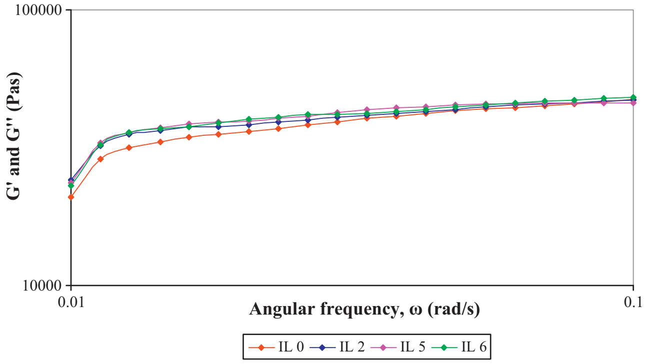

Fig. 7. Frequency sweep for first system.

the trend of plots was discussed in Sections 3.1.1 and 3.1.2. Upon incorporation of SiO_2, the polymer electrolytes normally use larger shear stress to disrupt the bonds and linkages due to the formation of three-dimensional network. As proven in the figure, CPE 1 and CPE 3 are not easily to be distorted as they need higher shear stress to rupture the polymeric systems. The main attributor of this observation is the silanol linkages among the silica particles, favoring the three-dimensional network in the complex. As a result, more energy is required to destroy the network or entanglement.

Among all the CPEs, the increase sequence in the required amount of shear stress for the initial deformation is in this order: CPE 4 < CPE 3 < CPE 1. The apparent decrease in initial shear stress is attained as increase the SiO_2 loadings. Excessive nano-SiO_2 particles cause the repulsion among the aggregates and hence further weaken the interactions within the polymer matrix, which in turn lowers values of G' and G''. In addition, CPE 1 imparts lower stability at high shear stress by showing a rapid decrease in G' and G''. In contrast, CPE 3 and CPE 4 exhibit higher stability even though they depict lower values of both moduli which denotes as lower mechanical properties. It leads to the conclusion that the solid-like behavior of polymer electrolytes can be improved with doping more SiO_2 concentration at high deformation.

3.3. Oscillatory frequency sweep

Frequency sweep (FS) is defined as time dependent study on the structural strength in a wide frequency range. In other words, it is used to examine the extent of long-term structural stability of polymer electrolytes. The frequency sweep is further performed with the proposed strain amplitude obtained from the amplitude sweep.

3.3.1. First system

Fig. 7 shows the storage (G') and loss (G'') moduli with respect to frequency for first system. The absence of G'' within the frequency regime is the main feature of FS study. This characteristic reveals the gel nature of ionic liquid-based GPEs. As shown in Fig. 7, G' is frequency dependent. At low frequency, the deformation of samples takes place for a longer time and G' shows the lower value. This is mainly attributed to the longer relaxation time whereby the polymer chain has enough time to prevent the externally imposed deformation by relaxing to an energetically more favorable state [9]. The slippage of the entanglements of the entwined polymer chains contributes to the relaxation process, leading to the increase in segmental motion. Consequently, it reduces the solid properties of polymer electrolytes and hence indicates the lower structural stability at low frequency.

As shown in Fig. 7, the value of G' increases with impregnation of BmImTFSI. It is suggestive of formation of hydrogen bonding of TFSI anions with the polymer chain, which in turn to the difficulty of slippage of the entanglements or cross-linkage in the polymer matrix as hydrogen bond is well known as the strongest covalent bond. As a result, the elastic properties of polymer electrolytes decline slowly. On the other hand, the value of G' is increased with an upward shift in frequency. This indicates the excellent stability of polymer electrolytes at shorter time scale of deformation, arising from the decrease in relaxation time. At this stage, the polymer chains no longer slip past one another and therefore the entanglements within the polymer matrix acts like fixed network junctions [9]. This temporary polymer network favors to store the temporarily imposed energy and eventually increases the elastic properties. At higher frequency range, the almost flat region implies the strong gel behavior of the polymer electrolytes [10]. Since the G' values for ionic liquid-free polymer electrolytes and GPEs are similar, it can thus be concluded that addition of ionic liquid does not possesses any effect on the structural stability at high frequency when the deformation takes places for shorter time.

3.3.2. Second system

The typical frequency sweep of second system is shown in Fig. 8. The absence of G'' profile confirms once again the elastic solid-like properties of CPEs in the frequency range. The effect of incorporation with SiO_2 is not apparently observed as the G' values are almost the same in the frequency regime. It might be due to the low doping amount of SiO_2 which induces the futile effect on the structural stability of polymer electrolytes. For CPE 0 and all CPEs, there is an abrupt decrease in G' at low frequency, as illustrated in Fig. 8. When the deformation occurs for longer time at low frequency, the relaxation time becomes longer and contributes to the slippage of entanglements. This slippage reduces the ability for storing the imposed energy and

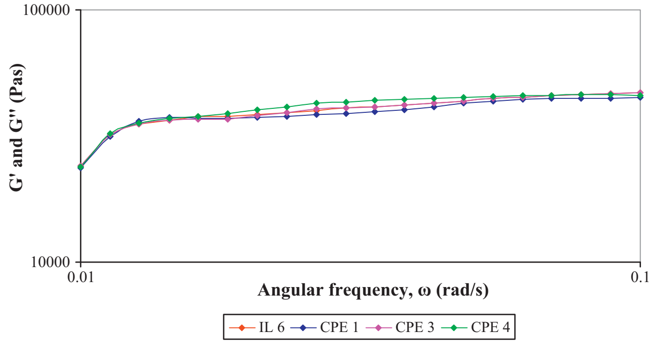

Fig. 8. Frequency sweep for second system.

hence decreases the solid behavior of sample, indicating the lower structural stability of the polymer system at low frequency. In contrast, at higher frequency, the structural stability of polymer electrolytes is improved as higher value of G' is shown in Fig. 8. Again, it is primarily attributed to the low relaxation time which then leads to insufficient time for slippage mechanism. Therefore, the structural strength of polymer electrolytes at high frequency is better than at low frequency. Since all the plots show the comparable values within the range, it can therefore be concluded that the addition of SiO_2 is not a promising candidate to improve the structural stability of polymer electrolytes.

3.4. Viscosity

3.4.1. First system

The comparison of viscosity of polymer electrolytes with or without ionic liquid as a function of shear rate is depicted in Fig. 9. As can be seen, the viscosity follows a linear relationship with respect to the shear rate, revealing the non-Newtonian properties of the polymer electrolytes. Under shear, the viscosity becomes lesser and it is a typical shear thinning behavior [10]. At low shear rate, the sample still remains its solid-like behavior with a high viscosity value when the deformation is lesser. On the contrary, the viscosity is decreased at high shear rate. It is due to the disturbance and breakdown of the interactive bonds in the complex by applying the shear on the polymer electrolyte. Another important feature is observed at high shear rate, i.e. the analogous of the viscosity and shear thinning rate of all the polymer electrolytes. This arises from the preferential orientation of the entanglements of polymer electrolyte parallel to flow direction [6].

Since ionic liquid is a low viscosity material, thus it is expected that the ionic liquid-based gel polymer electrolytes have lower viscosity. At 0.1 s^{-1} of shear rate, the viscosity of GPEs is declined upon addition of BmImTFSI, as shown in Fig. 9. The viscosity decreases further with increasing BmImTFSI loadings due to the plasticizing effect. The entanglements of polymer matrix such transient cross-linkage can be disordered easily as ionic liquid favors to weaken the physical and chemical interactions within the polymer system. The low viscosity of polymer electrolyte enhances the ionic hopping process by providing more amorphous region.

3.4.2. Second system

Fig. 10 shows the typical viscosity profile as a function of shear rate with various mass fraction of fumed silica. Non-Newtonian nature has been shown in CPE 0 and CPEs as the viscosity exhibits the linear relationship with respect to shear rate. When the sample is less deformed, the value of viscosity is still higher at lower shear rate. This is due to the initial deformation whereby the sample still maintains its elastic properties by forming the polymer network among the aggregates via silanol linkage. However, the viscosity decreases monotonically with increasing the shear rate and reveals the shear thinning properties. It is because the network among the SiO_2 particles is being destroyed by shear. At high shear rate, the viscosity and shear thinning rate for CPE 0 and CPEs are quite similar due to the preferential orientation of the silanol linkage parallel to the flow direction [6].

In principle, the viscosity of sample should be increases with shear rate due to the formation of three-dimensional network among the aggregates through the silanol linkage, as explained in previous amplitude sweep and oscillatory shear studies. However, at low shear rate (at 0.1 s^{-1}), the viscosity is reduced upon addition of fumed silica. The perspective on this result is because of the presence of ionic liquid. The plasticizing effect of ionic liquid weakens the intertwined network linkages among the aggregates. The viscosity of CPEs decreases with

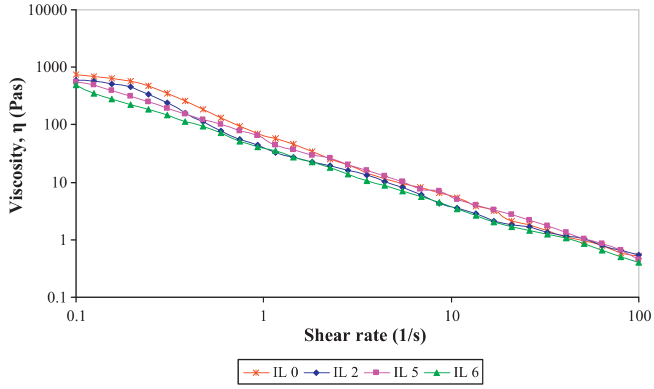

Fig. 9. Typical viscosity curve of first system.

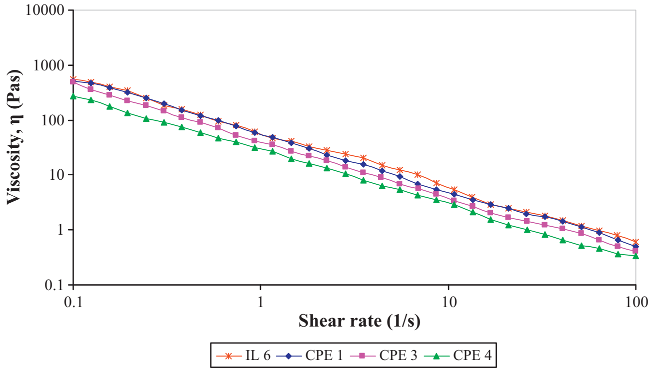

Fig. 10. Typical viscosity curve of second system.

increasing the content of SiO_2. At high SiO_2 composition, SiO_2 nano-particles are in excess mode. Excessive SiO_2 causes the repulsion forces between the aggregates and therefore weakens the interaction between three-dimensional networks by producing longer distance between the aggregates. As a result, CPEs imparts lower viscosity.

4. Conclusion

Two different types of polymer electrolyte systems were prepared viz. GPEs and CPEs. The elastic solid-like properties of GPEs and CPEs had been observed in the amplitude sweep and oscillatory shear sweep studies, as the value of G' was higher than G''. In amplitude sweep, both G' and G'' decreased with ionic liquid and fumed silica compositions, revealing the gel-like appearance of polymer electrolytes with low mechanical stability. LVE range became wider with increasing ionic liquid and fumed silica contents. It indicated better stability of these polymer electrolytes. In addition, the lower mechanical property of the samples had been illustrated in oscillatory shear sweep by showing the lower value of starting point of shear stress. In frequency sweep, G'' was absent and further implied the solid nature of the GPEs and CPEs. At low frequency, G' shows lower value due to the longer relaxation time. Therefore, the structural stability was reduced at low frequency. In contrast, the structural stability was improved at the short-term of deformation. It is mainly attributed to the declined relaxation time. Shear thinning properties of GPEs and CPEs were observed in viscosity curve with respect to the shear rate. Under shear, the viscosity was decreased due to interruption of the weak physical and chemical bonds within the polymer matrix.

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