

# Comparison studies of SiO<sub>2</sub> and HCl-SiO<sub>2</sub> filler on the film formation and ionic conductivity of PMMA/ENR 50 electrolytes

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## ABSTRACT

In this study, polymer electrolytes derived from polymethyl methacrylate/50 % epoxidized natural rubber (PE) were successfully prepared. The effects of silicon dioxide (SiO<sub>2</sub>) (15 nm) and acid modified SiO<sub>2</sub> (HCl-SiO<sub>2</sub>) (15 nm) on the film formation and ionic conductivity of polymer electrolytes were investigated. In samples preparation, SiO<sub>2</sub> was used as received. Meanwhile, HCl-SiO<sub>2</sub> was prepared by reacting SiO<sub>2</sub> with 7 M of hydrochloric acid (HCl) solution. PE, PE filled with SiO<sub>2</sub> (PE-SiO<sub>2</sub>) and PE filled with HCl-SiO<sub>2</sub> (PE-HCl-SiO<sub>2</sub>) electrolytes were prepared by solvent casting method with tetrahydrofuran (THF) as solvent and lithium tetrafluoroborate (LiBF<sub>4</sub>) as doping salt. The smoothness surface, opacity and agglomeration of PE-HCl-SiO<sub>2</sub> film were found in between PE and PE-SiO<sub>2</sub>. CHNS analysis shows that the percentage of hydrogen in HCl-SiO<sub>2</sub> and SiO<sub>2</sub> are 0.88 and 2.07 %, respectively. Fourier transform infra-red (FT-IR) analysis confirm that HCl-SiO<sub>2</sub> has low number of silanol group (Si-OH) but high number of siloxane group (Si-O-Si) compared to SiO<sub>2</sub>. Field Emission Scanning Electron Microscopy (FESEM) analysis shows that HCl-SiO<sub>2</sub> has smaller size of agglomeration and porosity compared to SiO<sub>2</sub>. Meanwhile, electrochemical impedance spectroscopy (EIS) analysis shows that the ionic conductivity of PE-HCl-SiO<sub>2</sub> is higher than PE but slightly lower than PE-SiO<sub>2</sub>. Copyright © 2016 VBRI Press.

**Keywords:** Silicon dioxide; silanol group; polymethyl methacrylate; epoxidized natural rubber; polymer electrolyte.

## Introduction

Studies on development of polymer electrolytes have been given big attention by researchers since it was firstly discovered in 1970s by Wright [1]. Since few decades ago, the early period of development of polymer electrolytes was only focused on improvement of ionic conductivity. Lately, focus of development of polymer electrolytes became broader to obtain high energy density, good charge/discharge cyclic stability as well as high safety [2]. These align with the current requirement on designing Li ion batteries include excellence electrochemical performance and down-scale [3].

Polymer electrolytes consist of polymers implanted with inorganic salt in polymer matrix. Polymer electrolytes pleased its properties include free standing film, no leakage, safer, thermally and electrochemically stable [4].

Unfortunately, lower ionic conductivity at lower temperature became the major drawback to prepare a good ionic conductivity. Normally, the cell must be heated up to 100 °C in order to optimize its performance [5].

Addition of plasticizers was proposed to increase ionic conductivity of polymer electrolytes especially at lower temperature [6]. These plasticized polymer electrolytes known as gelled polymer electrolytes (GPEs). Frequently, plasticizers used were propylene carbonate (PC) [7], dimethyl carbonates (DMC) [8], poly(ethylene glycol) (PEG) [9] and ionic liquids [10]. Unfortunately, GPEs may have disadvantages such as non-free standing, leakage of harmful and high flammable chemical and corrosion to the body of cell [11 - 13].

Addition of inert ceramic fillers were suitable alternative to increase ionic conductivity [14, 15] by creating porosity for moving ions in the polymer electrolytes matrix [16]. This type of polymer electrolytes was classified as solid polymer electrolytes (SPEs). Besides, SPEs were physically stable, free standing and do not require external supporting structure [17]. Unfortunately, most of SPEs were suffer for filler agglomeration [18 - 20]. Usually, the surface of film becomes rough, brittle and opaque. Therefore, SPEs has less contact with electrodes and limits its performance [21].

In this study, for the first time, we presented the comparison effects of SiO<sub>2</sub> (15 nm) and HCl-SiO<sub>2</sub> (15 nm) filler on polymer electrolytes derived from PMMA and

ENR 50. This work was focus on the formation, appearance of free standing and ionic conductivity of polymer electrolytes. The polymer film properties were linked to the nature and functional group on the surface of SiO<sub>2</sub> and HCl-SiO<sub>2</sub>.

## Experimental

### Materials

All materials were used without prior purification. SiO<sub>2</sub> (15 nm), LiBF<sub>4</sub> and PMMA (120 000) were purchased from Aldrich Chemistry, USA. ENR 50 kindly supplied from Guthree Polymer. THF was supplied from J.T.Baker, USA and HCl was purchased from Merck, Germany.

### Material synthesis

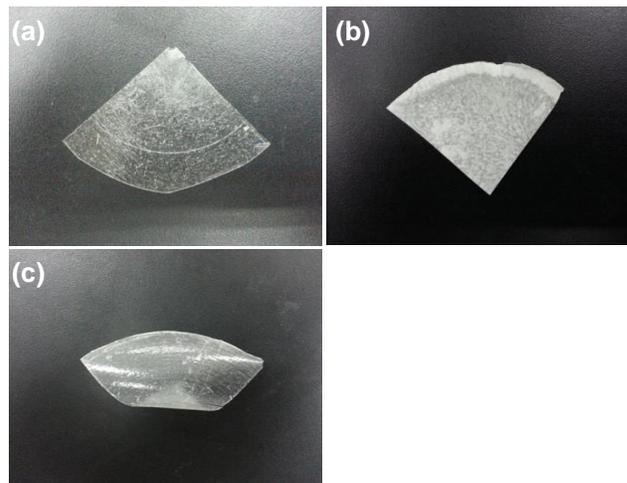
SiO<sub>2</sub> was reacted with 7 M of HCl solution under stirring condition at room temperature for an hour. HCl-SiO<sub>2</sub> slurry was then dried in an oven at temperature of 120 °C for 24 hours without calcination to remove water and excess HCl from the sample. PMMA, ENR 50, SiO<sub>2</sub> and HCl-SiO<sub>2</sub> were dissolved separately in THF (2, 1, 2 and 2% (w/v)) under continuous stirring using magnetic stirrers at room temperature. ENR 50-THF, and fillers-THF solutions were blended with PMMA-THF solution at composition of 10 and 5% (w/w). The mixtures were continuous stirred for 24 hours before LiBF<sub>4</sub> (40% (w/w)) was added. The mixtures were casted in Teflon petri disc under nitrogen flow for 12 hours. Polymer films was then peeled off and dried in an oven at 50 °C.

### Characterizations

Digital picture of samples film was captured using DSLR camera model Nikon D7200. The camera was equipped with Tamron 18-270 mm F/3.5-6.3 Di II VC PZD lens. Percentage of hydrogen in SiO<sub>2</sub> and HCl-SiO<sub>2</sub> were determined using Perkin Elmer Series 2 CHNS/O Analyzer 2400. In Tin samples pans, about 2 to 4 mg of samples were applied in combustion tube at temperature of 975 °C. Perkin Elmer Spectrum One FTIR Spectrophotometer was used for FTIR study. The spectra were recorded at room temperature. The samples were scanned at 400-4000 cm<sup>-1</sup> with resolution of 16 cm<sup>-1</sup> and 4 times scan rate. The FESEM micrographs of SiO<sub>2</sub> and HCl-SiO<sub>2</sub> were recorded using FESEM model Carl Zeiss Supra 40 VP. The samples images were taken under 1000 magnification without prior coated with gold. The impedance of thin film samples was measured using a Hioki 3532-01 LCR High Tester. The instrument was interfaced to a computer and set to measure the impedance in a frequency range of 100 Hz – 1 MHz from 298 up to 353 K. From the Cole-Cole plot obtained, the bulk resistance can be determined hence, the electrical conductivity of the sample can then be calculated using the following equation;

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

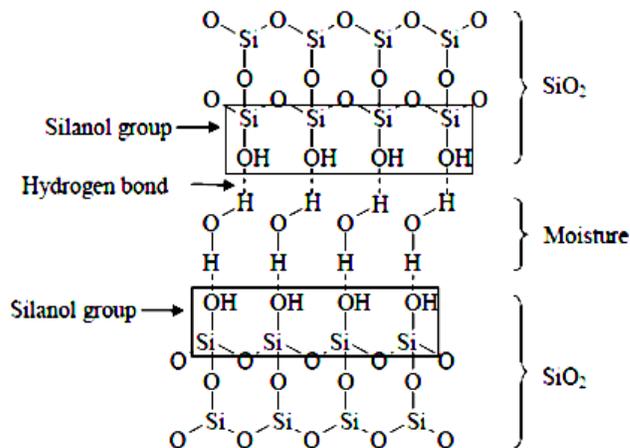
where,  $\sigma$  is the ionic conductivity,  $t$  is the thickness of the sample,  $A$  is the contact area between sample and electrode and  $R_b$  is bulk resistance.



**Fig. 1.** Free standing film of un-dope (a) PE and (b) PE-SiO<sub>2</sub> and (c) PE-HCl-SiO<sub>2</sub>.

## Results and discussion

The formation of PE, PE-SiO<sub>2</sub> and PE-HCl-SiO<sub>2</sub> films show in **Fig. 1**. The figure clearly shows that PE obtained was free standing film, transparence and mechanically flexible. Unfortunately, PE-SiO<sub>2</sub> film was found opaque, agglomerates and brittle when SiO<sub>2</sub> was added. The opacity of the films was due to the naturally white powder of SiO<sub>2</sub> [22]. Meanwhile, the agglomeration phenomenon is normally occurred in polymer filled nano size filler. The filler particles distributed in polymer matrix experience attraction forces between themselves and accumulated [23]. Interestingly, the smoothness surface, opacity and agglomeration of PE-HCl-SiO<sub>2</sub> film are in between PE and PE-SiO<sub>2</sub> indicated that HCl-SiO<sub>2</sub> has the ability to hinder formation of attraction force between filler particles.



**Fig. 2.** Formation of hydrogen bond between silicon dioxide and moisture [20].

Formation of SiO<sub>2</sub> agglomeration as seen as white spots on the surface of the films may due to tendency of oxygen atom of Si-OH on the surface of SiO<sub>2</sub> to form hydrogen bond with moisture in the polymer matrix (**Fig. 2**) [24, 25]. In other words, Si-OH may act like a glue to adhere SiO<sub>2</sub> particles (**Fig. 2**). This due to fact that the hydrogen bond between Si-OH with moisture is stronger than hydrogen bond among water molecules itself [21, 26].

The agglomeration of HCl-SiO<sub>2</sub> in PE-HCl-SiO<sub>2</sub> as expected was improved when the number of Si-OH on the surface of HCl-SiO<sub>2</sub> was reduced by the acid modification. The reduction number of Si-OH on the surface of HCl-SiO<sub>2</sub> after the acid modification was observed by comparing the percentage of hydrogen atom in HCl-SiO<sub>2</sub> and SiO<sub>2</sub> using CHNS analysis as shown in Fig. 3. It was found that the percentage of hydrogen in HCl-SiO<sub>2</sub> decreases compared to SiO<sub>2</sub>. This proved that the acid modification successfully reduces the number of Si-OH on the surface of HCl-SiO<sub>2</sub>.

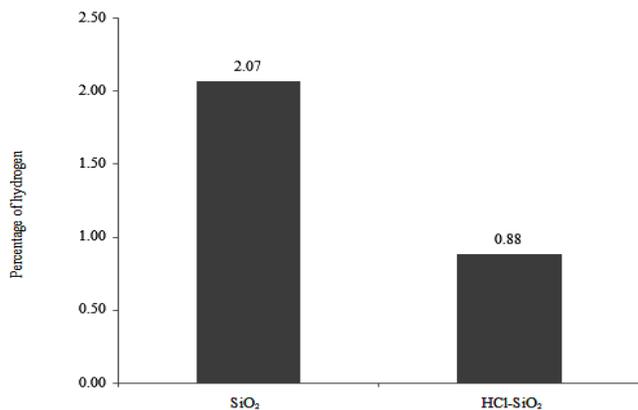


Fig. 3. CHNS analysis corresponding to SiO<sub>2</sub> and HCl-SiO<sub>2</sub>.

FT-IR analysis was used to further confirm the effect of acid modification on the number of Si-OH and Si-O-Si on the surface of SiO<sub>2</sub> and HCl-SiO<sub>2</sub>. The acid modification was proven by comparing the intensity of peaks related to Si-OH and Si-O-Si, including Si-O-Si asymmetric stretching at 1104 cm<sup>-1</sup>, Si-OH stretching at 970 cm<sup>-1</sup>, Si-O-Si bending at 817 cm<sup>-1</sup> and Si-O-Si rocking at 474 cm<sup>-1</sup> [27] (Fig. 4). From the result, it was found that SiO<sub>2</sub> is higher in Si-OH but lower in Si-O-Si group compared to HCl-SiO<sub>2</sub>. Contrary, after the acid modification of SiO<sub>2</sub>, it was observed that HCl-SiO<sub>2</sub> is lower in Si-OH but higher in Si-O-Si group compared to SiO<sub>2</sub>. This observation successfully confirmed that the number of Si-OH on the surface of SiO<sub>2</sub> was reduced by the acid modification.

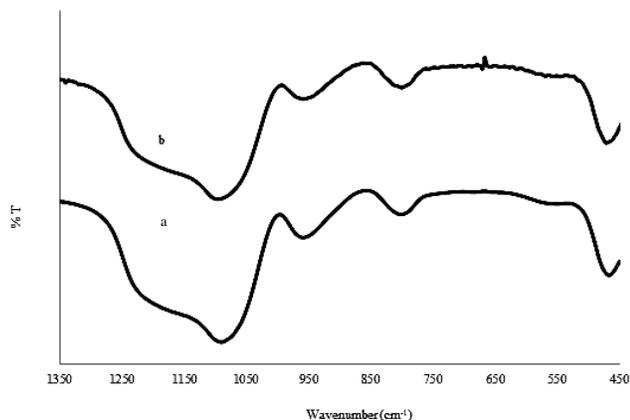


Fig. 4. FT-IR spectra corresponding to (a) SiO<sub>2</sub> and (b) HCl-SiO<sub>2</sub>.

SiO<sub>2</sub> and HCl-SiO<sub>2</sub> were examined under FESEM in order to investigate the effect of acid modification on the size of aggregation of the fillers (Fig. 5). In overall,

aggregation of HCl-SiO<sub>2</sub> in Fig. 5 (b) was observed to be smaller compared to SiO<sub>2</sub> in Fig. 5 (a).

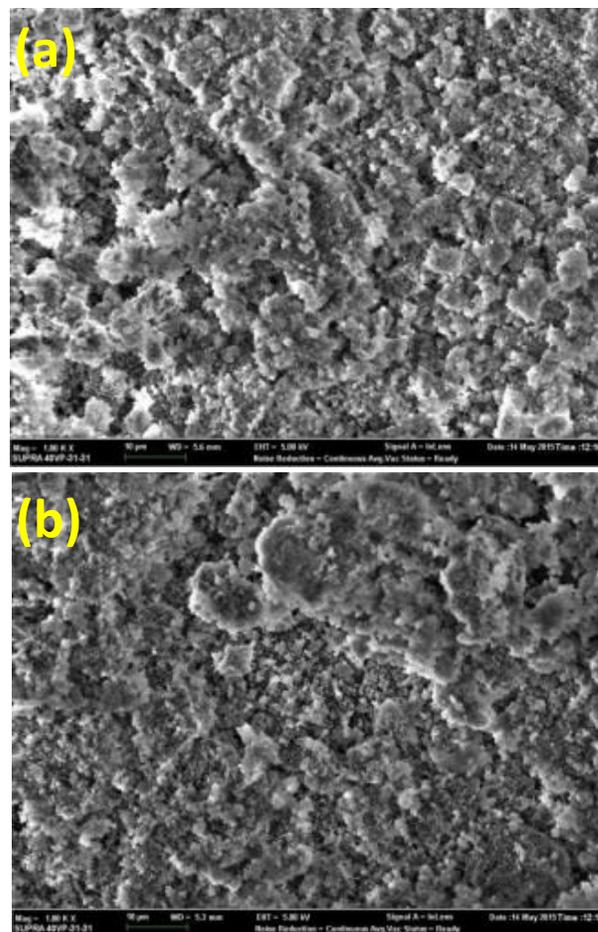


Fig. 5. FESEM micrograph corresponding to (a) SiO<sub>2</sub> and (b) HCl-SiO<sub>2</sub>.

In addition, the figure also shows that the intensity of porosity of HCl-SiO<sub>2</sub> is smaller in size compared to SiO<sub>2</sub>. As proposed by Shabanian *et al.* (2012) [28], the neighboring SiO<sub>2</sub> particles may attract each other due to the attraction forces between them. The attraction force attributed from condensed moisture from the surrounding gas on the surface of SiO<sub>2</sub> in between the two SiO<sub>2</sub> particles. In other words, the moisture may act as a bridge to connect the two SiO<sub>2</sub> particles [28]. Relatively, the attraction force between the two SiO<sub>2</sub> particles may be strongly influenced by the number of sites available, namely Si-OH on the surface of SiO<sub>2</sub>, for the attraction force. The more Si-OH on the surface of SiO<sub>2</sub>, the more attraction force takes place in between the two SiO<sub>2</sub> particles. Therefore, it can be concluded that the modification successfully reduces the number of Si-OH on the surface of SiO<sub>2</sub>, minimizing the attraction force between the two SiO<sub>2</sub> particles and reducing the SiO<sub>2</sub> agglomeration.

Reaction mechanism during the acid modification of SiO<sub>2</sub> is proposed in Fig. 6. The acid acts as a catalyst to reduce the number of Si-OH and increase the number of Si-O-Si groups on the surface of HCl-SiO<sub>2</sub>, simultaneously. The mechanism was suggested to involve 3 steps and be catalyzed by the acid. In the reaction, the acid may protonate the hydroxyl atom of Si-OH. The protonated hydroxyl group is

a good leaving group therefore the Si-OH<sub>2</sub><sup>+</sup> bond may dissociated to release water molecule [28] in the second step.

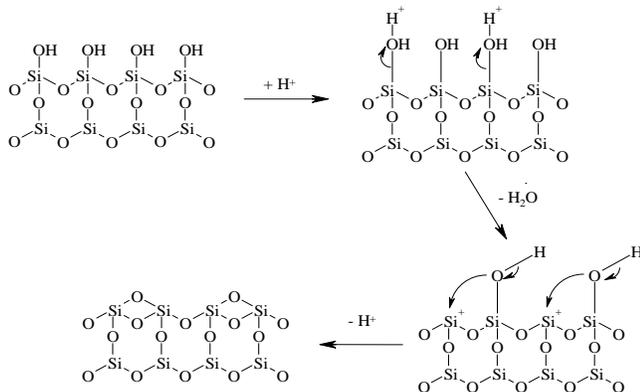


Fig. 6. Suggested mechanism of HCl-SiO<sub>2</sub> modification routes [20].

The lone pair electrons of oxygen atom at adjacent Si-OH may strongly attract to O-Si<sup>+</sup> ion. Therefore, a new bond formed between Si atoms with oxygen atom of adjacent Si-O and releases the acid back. The suggested reaction mechanism successfully demonstrated that the number of Si-OH reduced meanwhile the number of Si-O-Si increased due to the acid modification [29]. As result, the tendency formation of hydrogen bond between oxygen atoms of Si-OH on the surface of SiO<sub>2</sub> with hydrogen atom of trapped water molecule may successfully depress. The proposed mechanism may use to support CHNS, FT-IR and FESEM analysis result discussed previously in which after the acid modification HCl-SiO<sub>2</sub> compose low in Si-OH.

Room temperature ionic conductivity corresponding to PE, PE-SiO<sub>2</sub> and PE-HCl-SiO<sub>2</sub> electrolytes are shown in Fig. 7. The figure shows that ionic conductivity of PE-SiO<sub>2</sub> electrolytes was higher than PE electrolyte. According to Ravi *et al.* (2014), ionic conductivity of polymer electrolytes is depend on concentration of dopant salt and it's mobility [18]. Usually, polymer electrolytes with high porosity provide ion mobility and exhibit high ionic conductivity but may decrease its mechanical properties [30].

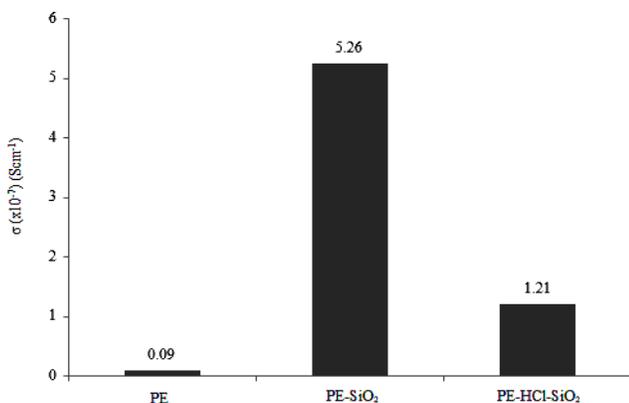


Fig. 7. Ionic conductivity of PE-SiO<sub>2</sub> and PE-HCl-SiO<sub>2</sub>.

In addition, the increase in ionic conductivity of PE-SiO<sub>2</sub> may attribute to increase the porosity in polymer matrix created by SiO<sub>2</sub> particles [31, 32]. The porosity may

provide easy pathway for Li<sup>+</sup> ion movement hence increases the ionic conductivity of PE-SiO<sub>2</sub>. Contrary, the ionic conductivity of PE-HCl-SiO<sub>2</sub> electrolyte was found decreases compared to PE-SiO<sub>2</sub> electrolyte but still higher than PE electrolyte. This may attributed to the ionic conduction in PE-HCl-SiO<sub>2</sub> are less due to HCl-SiO<sub>2</sub> exhibit smaller porosity compare to SiO<sub>2</sub> as shown in Fig. 5.

## Conclusion

As conclusion, PE, PE-SiO<sub>2</sub> and PE-HCl-SiO<sub>2</sub> were successfully prepared by solvent casting method. Addition of SiO<sub>2</sub> improved the ionic conductivity of PE-SiO<sub>2</sub> compared to PE. Unfortunately, PE-SiO<sub>2</sub> suffers for SiO<sub>2</sub> roughness surface, opacity, agglomeration and brittleness. Interestingly, addition of HCl-SiO<sub>2</sub> in PE-HCl-SiO<sub>2</sub> had improved the weaknesses found in PE-SiO<sub>2</sub>. Nevertheless, PE-HCl-SiO<sub>2</sub> shows slightly lower in ionic conductivity but still higher than PE.

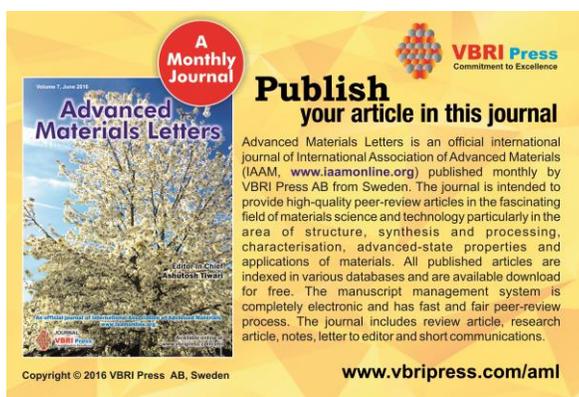
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