Chapter 5

Electrowetting Approach for Breakdown Field in Teflon AF and Polystyrene

The dielectric breakdown study was carried out on Teflon AF and polystyrene insulators using planar electrode geometry electrowetting (EW) setup. The DC voltages were applied on various liquids such as Milli-Q water, aqueous KCl solution, aqueous Sodium-do-Decyle Sulfate (SDS) solution, ethylene glycol (EG), water with pH 4 and water with pH 9.2. It has been observed that the breakdown field depends on voltage polarity and is roughly proportional to the ionic sizes. However, aqueous SDS has low breakdown field despite the large ionic size of the SDS anion and for EG breakdown field is high in spite of relatively small ionic size. The results highlight that along with the ionic/molecular size, their interaction on polymer surface might be playing a role in lowering the breakdown field.
5.1 Introduction

Electrical power is mainly transmitted through the over-head and under-ground cables at a voltage difference of tens to hundreds of kilo-volt (kV). The underground cables are always in contact with ground water, humidity, sewage water etc. These liquids in contact with the underground cable form a capacitor where the metal conductor acts as one electrode while the cable insulation acts as a dielectric and water plays a role of another electrode which is always held at the ground potential. The cable insulation is well designed to withstand mechanical stress and thermal cycling due to Joule heating. Despite these common stresses, wetting of aqueous medium under external applied potential is another unseen stress, explored by Philips group. It is shown that water goes to more wetting state under the application of high potential. The wetting of water on polymer surface thus increases with the applied potential. When the applied electric field increases beyond the limiting value, current flows through the polymeric dielectric that leads to electrical breakdown. This effect has been studied and called as water treeing phenomenon in the cross-linked polyethylene cable insulation. On the other hand, cable insulation may have intrinsic defects or processing faults due to bending of cable so that it becomes susceptible to water tree growth. This partial discharge progresses through the stressed dielectric insulation and ultimately damages the insulation. The ions and macromolecules dissolved in water may have different effect on water treeing and breakdown phenomenon which demands systematic investigation.

The wetting on demand assisted by electrowetting (EW) has been extensively studied in the recent past. In EW a liquid droplet is placed on a polymeric dielectric deposited on conducting surfaces like ITO glass and the voltage is applied between liquid drop and underneath electrode which changes the wettability of liquid on dielectric surface. Figure 5.1 shows the schematic of EW set up used to study dielectric breakdown for both (a) positive and (b) negative voltage polarity. The contact angle (CA) changes as a function of applied voltage that can be expressed by Young-Lippmann equation 5.1:

\[ \cos \theta (U) = \cos \theta_Y + \frac{c}{2\gamma_{LV}} U^2 \quad \text{--- (5.1)} \]

where \( U \) is applied potential, \( \theta (U) \) is voltage dependent CA, \( \theta_Y \) is Young’s angle (zero voltage CA), \( \gamma_{LV} \) is interfacial tension of liquid with respect to the surrounding medium and \( c = \frac{\varepsilon_0 \varepsilon}{d} \) is capacitance per unit area with \( \varepsilon_0 \) as permittivity of free space, \( \varepsilon \)
being dielectric constant and \( d \) being thickness of the insulator material. The reversible CA change offered by EW has made it useful in many applications like liquid lenses,\textsuperscript{7-9} electronic display,\textsuperscript{10,11} and microfluidic devices.\textsuperscript{12}

Above discussion suggests that electrowetting offers a unique tool to understand wetting of liquid under external potential on dielectric surface. If a current is monitored in the circuit, it gives information on the dielectric breakdown in presence of liquid on dielectric surface. Also in EW based devices breakdown is needed to be avoided for repetitive use of the device. Hence systematic study of the dielectric breakdown can give knowledge to improve life of EW based devices.

\[ \text{Figure 5.1: The schematic of EW set up used to study dielectric breakdown for both (a) positive and (b) negative voltage polarity respectively.} \]

In the present study we examined dielectric breakdown using generic EW configuration for various liquid drops. Teflon AF, the most used dielectric for EW and another hydrophobic polymer polystyrene (PS) are used as test materials. The study carried out reveals that breakdown field is polarity and liquid dependent. The detailed information is given in the later section.

\section*{5.2 Experimental}

Dielectric layer of Teflon AF was deposited on conducting ITO glass (Präzisions Glas & Optik, Germany) using a standard protocol.\textsuperscript{13} Before depositing the dielectric layer, ITO glass was thoroughly cleaned successively in ultrasonic baths of diluted detergent, acetone and isopropanal. It was then dried in nitrogen jet and then utilized for dip-coating using 3 wt. \% solution of amorphous fluoropolymer Teflon AF 1600 (DuPont, USA) in perfluorinated solvent FC 40 keeping dipping/withdrawal speed at 15 cm min\(^{-1}\). The samples were heated at 110 °C for 10
min. and allowed to cool to room temperature and then the second layer was coated. They were finally heated at 160 °C for 10 min and 200 °C for 12 hrs. in a vacuum oven for complete removal of solvent. The thickness, as measured by profilometer was found to be 400 nm. The polymer PS was also deposited by dip-coating technique on ITO glass. The 10 wt % PS solution in toluene was used for dip-coating keeping dipping/withdrawal speed at 7 cm min$^{-1}$. The process was repeated thrice to achieve the desired thickness. The films were then annealed at 100 °C for 12 hrs. in a vacuum oven. The thickness of the films measured by profilometer was found to be 3 µm. After that the films of both the polymers, Teflon AF and PS, were subjected to EW breakdown study in ambient air.

EW response was studied on both the dielectrics using various liquids like Milli-Q water, aqueous solution of KCl, aqueous SDS, Ethylene glycol (EG), water with pH 4 and water with pH 9.2. The conductivity of Milli-Q water was increased to 1 mS cm$^{-1}$ by adding appropriate amount of KCl. A solution of 0.5 wt. % of SDS was prepared in Milli-Q water which is above critical micelle concentration (CMC) (CMC = 8.2 mM). The pH of water was adjusted to 4 and 9.2 by dissolving the commercial pH electrolyte (Merk, India). The conductivity of all the solutions was measured. The surface tensions of the liquids were measured using Pendant drop method. The electrowetting was studied using sessile drop of 5 µl in air ambient. Keithley 2400 source meter was employed with Lab Tracer (2.9) software for applying DC potential across drop and bottom ITO electrode and simultaneously the leakage current flowing through the circuit was recorded with the same source meter. All the voltages were measured with respect to the drop. The voltage was increased linearly with the rate of 2 V s$^{-1}$, at the same time contact angle was recorded using optical contact angle (OCA) goniometer and analyzed using software from DataPhysics, Germany.

5.3 Results and Discussion

5.3.1 Teflon AF dielectric

Figure 5.2 shows the graph of leakage current ($I$) flowing through the circuit during EW versus applied voltage ($U$) and also the corresponding electric field ($E$) for each liquid droplet is shown. The dielectric breakdown takes place when leakage current exceeds the value of 1 µA. The plot shows a large variation in breakdown
voltage for different liquids used. Also different breakdown voltage is noted for positive and negative polarity for the same liquid.

Figure 5.2: The variation of leakage current ($I$) with respect to the applied voltage ($U$) for Teflon AF dielectric. Also the corresponding electric field ($E$) is shown on upper X-axis.

In case of Milli-Q water dielectric failure current i.e. leakage current suddenly increases when the voltage exceeds 72 V and -62 V in positive and negative polarity regime respectively. The breakdown voltage for aqueous KCl is 51 V and -72 V. The dielectric breakdown voltages for aqueous SDS are 20 V and -3 V, which are considerably small as compared with other liquids. However, in case of EG dielectric failure was not observed giving leakage current value in the range of nA even when the applied voltage exceeds ±180 V. All these data along with other properties of liquids like surface tension and conductivity are summarized in Table 5.1. The earlier study carried out by Balaji Raj et al.\textsuperscript{14} suggests that the breakdown field is proportional to the ionic sizes in the aqueous solutions. The aqueous solutions in the present study contain ions like H$_3$O$^+$, OH$^-$, K$^+$, Cl$^-$, Na$^+$, C$_{12}$H$_{25}$SO$_4^-$ and monomer like OH-CH$_2$-CH$_2$-OH (EG). Thus in the present case high breakdown field is expected for aqueous SDS solution in the negative potential regime, which is not
observed. On the contrary considerably low breakdown field is observed for aqueous SDS in negative voltage regime. Also high breakdown field can be realized for EG which is having relatively small ionic size. So along with ionic size, ion-polymer interaction is also playing some role in defining breakdown field.

**Table 5.1:** The table lists surface tension ($\gamma_{LV}$), conductivity ($\sigma$), breakdown voltages ($U$) and electric fields ($E$) on Teflon AF for positive and negative voltage polarity for different liquids.

<table>
<thead>
<tr>
<th>Liquids</th>
<th>$\gamma_{LV}$ (mN/m)</th>
<th>$\sigma$ (mS/cm)</th>
<th>+U (V)</th>
<th>+E (V/µm)</th>
<th>-U (V)</th>
<th>-E (V/µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milli-Q water</td>
<td>71.8</td>
<td>0.0176</td>
<td>72</td>
<td>180</td>
<td>62</td>
<td>155</td>
</tr>
<tr>
<td>Saline KCl</td>
<td>71.7</td>
<td>1.0</td>
<td>51</td>
<td>127.5</td>
<td>72</td>
<td>180</td>
</tr>
<tr>
<td>Aqueous SDS</td>
<td>35</td>
<td>0.65</td>
<td>20</td>
<td>50</td>
<td>3</td>
<td>7.5</td>
</tr>
<tr>
<td>EG</td>
<td>48</td>
<td>0.0017</td>
<td>&gt;180</td>
<td>&gt;450</td>
<td>&gt;180</td>
<td>&gt;450</td>
</tr>
</tbody>
</table>

After carrying out EW we studied surface morphology of Teflon AF by using Atomic Force Microscopy (AFM). Figure 5.3 depicts AFM images for pristine and electrowetted surfaces using various liquids mentioned above. The nano-structurations were observed on Teflon AF after carrying out EW using aqueous SDS. The formation of nano-structurations might lead to decrease the breakdown field. The changes in surface morphology of Teflon AF are not seen in case of liquid contact like Milli Q water, aqueous KCl and EG droplets. However, exceptionally high breakdown field and no surface structurations are observed in case of EG which can not be justified based on the surface tension values and requires involved study.
5.3.2 Polystyrene

Similar study was carried out on polystyrene dielectric. Figure 5.4 shows the graph of leakage current versus applied voltage with corresponding electric field values. The graph shows that the voltage values corresponding to dielectric breakdown for all liquids are small except for pH 9.2 and EG in the negative voltage regime. All the liquid properties along with breakdown data are summarized in Table 5.2. Our earlier EW study carried out on PMMA-polystyrene bilayer shows that nano-structurations are observed on polystyrene in ambient air even when the applied voltage range was well below the rated breakdown field. Hence we didn’t study surface morphology after EW breakdown because it is evident that during EW, the nano-structurations will be observed on polystyrene via electro-hydrodynamic instability.
Figure 5.4: The variation of leakage current ($I$) with respect to applied voltage ($U$) for polystyrene dielectric. Also the corresponding electric field ($E$) is shown on upper X-axis.

Table 5.1: The table lists surface tension ($\gamma_{LV}$), conductivity ($\sigma$), breakdown voltages ($U$) and electric fields ($E$) on polystyrene for positive and negative voltage polarity for different liquids.

<table>
<thead>
<tr>
<th>Liquids</th>
<th>$\gamma_{LV}$ (mN/m)</th>
<th>$\sigma$ (mS/cm)</th>
<th>$+U$ (V)</th>
<th>$+E$ (V/µm)</th>
<th>$-U$ (V)</th>
<th>$-E$ (V/µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milli-Q water</td>
<td>71.8</td>
<td>0.0176</td>
<td>24.5</td>
<td>8.2</td>
<td>17.6</td>
<td>5.9</td>
</tr>
<tr>
<td>Aqueous KCl</td>
<td>71.7</td>
<td>1.0</td>
<td>6</td>
<td>2</td>
<td>22.2</td>
<td>7.4</td>
</tr>
<tr>
<td>pH 4</td>
<td>71.4</td>
<td>5.6</td>
<td>8.1</td>
<td>2.7</td>
<td>9.5</td>
<td>3.2</td>
</tr>
<tr>
<td>pH 9.2</td>
<td>71.1</td>
<td>4.4</td>
<td>5.6</td>
<td>1.9</td>
<td>101</td>
<td>33.7</td>
</tr>
<tr>
<td>Aqueous SDS</td>
<td>35</td>
<td>0.65</td>
<td>3.9</td>
<td>1.3</td>
<td>6.1</td>
<td>2</td>
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<tr>
<td>EG</td>
<td>48</td>
<td>0.0017</td>
<td>21.3</td>
<td>7.1</td>
<td>131.7</td>
<td>43.9</td>
</tr>
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</table>

The breakdown study carried out on Teflon AF and polystyrene dielectrics show different behaviour on both the polymers for same liquid e.g. EG. So along with
the ionic size ion-polymer interaction is likely playing an important role in deciding dielectric breakdown using EW based technique.

5.4 Conclusions

The breakdown is different for applied positive and negative voltages for the same liquid. EW based dielectric breakdown study carried out on Teflon AF suggests that aqueous EG has considerably high breakdown field. On the contrary considerably low breakdown field is observed for aqueous SDS. The nano-structurations were observed on Teflon AF after carrying out EW using aqueous SDS that might lead to decrease the breakdown field. The breakdown field observed on polystyrene is considerably low for all the liquids used in the study except for pH 9.2 and EG in the negative voltage regime. Thus it can be inferred that along with ionic size, ion-polymer interaction plays an important role in deciding breakdown field for the polymeric dielectric using EW based technique.
References


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