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ABSTRACT

Glycolipids are components of cellular membranes comprising a hydrophobic lipid tail and one or more hydrophilic sugar heads, and are widely associated with the fields of life science and biochemistry. Due to the hygroscopic nature of sugar, the dry thermotropic phases of glycolipids have fewer studies. We report on the electric charge generation in anhydrous glucosides excited by the external high electric field (∼2 MV/m). This causes a large current in the smectic A phase, but not in the isotropic phase. Its intensity is about 100 times larger than the steady state current. The generation of the current was found to be irreversible by the repetition of the field application. The large electric carrier generation is originated in the smectic A phase, possibly due to an electron avalanche breakdown mechanism caused by the collisions of electrons through the impact ionization.

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I. INTRODUCTION

Glycolipids are widely associated with the fields of life science and biochemistry because of their amphiphilic nature. These are natural products and easily synthesized from renewable resources. Their excellent surface-active properties and biodegradable nature1 make them good candidates for the ecofriendly surfactant applications and as a medium to dissolve integral membrane protein.2 However, even though these are classified as sugar liquid crystals,3 their dry thermotropic phases have few studies4–6 and no known application to date.7–9 Naturally, the hygroscopic nature of sugar makes fundamental investigations of “dry” glycolipid more challenging because the completely dry state is difficult to achieve.10,11 However, despite the difficulty of controlling the hydration level, interest in the anhydrous state isreviving to exploit unique material properties for possible novel applications. Octyl-β-D-glucoside (βOG)12–14 used in many industrial applications has the dry thermotropic smectic A (SmA) within the range (∼69°C–107°C)12 in addition to the lyotropic phases. Recently, a deuterium NMR study of anhydrous βOG15 showed that the application of a high electric field induces a transition from the SmA phase to an isotropic phase. This work also reported that the phase change was accompanied by a transient current; also, the generation of air bubbles was observed by using the polarizing optical microscope (POM). This SmA-isotropic field-induced phase transition was rather unexpected, since a strong electric field usually induces ordering in the system as demonstrated by Ostapenko et al.14 The origin of this field induced phase change was unclear but Joule heating was ruled out. Instead it was thought that this is possibly due to the interaction between the field and the extensive hydrogen bonded network/cluster causing the hydrogen bond to weaken and eventually breaking the highly ordered structure to a lower ordered phase (isotropic). Such a phase transformation has been widely observed in water cluster systems, which have been subjected to an applied electric field of ∼5.14 × 106 MV/m.15 The study by Rai et al.16 further showed that increasing the field strength effectively reduces the HOMO-LUMO band-gap energy to zero at the break-point at which point the system becomes conducting. The band-gap energy at zero external electric field for these water clusters is about 6.74–7.79 eV. In the βOG lipidic system without water, the most likely
elementary process to occur in the presence of a strong electric field is the excitation of the hydrogen bond, which is equivalent to the thermal vibrational mode of an OH bond whose strength is typically in the range of 1–40 kcal/mol or 43–1700 meV. Thus, it is imperative to investigate in detail such phase transitions brought about by the electric properties. We note that there are numerous investigations of the electric field effect on biological membranes. For example, Stulen has studied a model membrane dimyristoyl phosphatidylcholine and potassium oleate subjected to a dc electric field of up to 10 MV/m. The conformation of the headgroup is affected by the electric field but not the chain structure or the dynamics. When the applied electric field is above 1 MV/m, a reversible structural change was observed, which was accompanied by a spontaneous current fluctuation. However, when electric field was increased to more than 10 MV/m, the bilayer structure was observed to break down irreversibly.

Taking a different approach, we used a simple bilayer model of dry glycolipids, i.e., the anhydrous βOG in the SmA phase to study the complex electrical phenomenon. These appear when an electric field is applied under the conditions where the anomalous transient current was observed. The current generation was investigated mainly using anhydrous βOG [see Fig. 1(a)]. βOG was supplied from Sigma Aldrich and used as received. The thermotropic phase transitions found are crystal → 69 °C SmA → 107 °C isotropic. For the purpose of comparison with βOG, dodecyl-β-D-glucoside (βDG) [see Fig. 1(b)] (also from Sigma Aldrich) was used. It has a longer chain than that of βOG and has thermotropic phase transitions (crystal → 80 °C SmA → 143 °C isotropic phase).

The layout of this paper is as follows: In Sec. II, we describe the transient current experiments. The results for the electric carrier generation and a possible mechanism for them are given in Sec. III. Our conclusions are outlined in Sec. IV.

II. EXPERIMENTAL

Special electric cells made of two conducting glass electrodes were constructed with the gap between the two electrodes varying from 5 to 200 μm. The surface of the glass electrodes was covered with a thin layer of indium tin oxide (ITO). A typical cell configuration consists of an electrode area, S = 50 mm². The cells were supplied from EHC Co., Ltd. The sample of the given glucoside was introduced into the open cells while it is in the isotropic phase. The cell was then sealed off by the use of an ultraviolet curing resin (SEKISUI Photolec A-720), which can be cured using UV radiation for a few minutes. During the UV radiation, the sample was masked to avoid the chemical damage and the temperature increase by UV. The cell containing the sample was then annealed before starting the measurements. That is, the sample was heated up from room temperature to 120 °C in the isotropic phase. It was kept in the isotropic phase for 20 min and then cooled gradually to the set temperature. This temperature control procedure was used to produce a homogeneous alignment in the smectic A phase.

The temperature was controlled with a heated air flow over the sample, giving a precision of ±0.2 °C, since recording the temperature of the sample directly is difficult. A dc or an ac sinusoidal electric field was applied to the sample. In a typical experiment after thermal equilibration, a voltage of a given strength and frequency is switched on and the current passing through the cell is time monitored for at least 200 s.

III. RESULTS AND DISCUSSION

In order to get a detailed picture of the effects on the transient current of electric field strength, frequency of the applied electric field, and temperature, we have investigated the behavior of a large number of cells. We start our discussion with the results shown in Fig. 2(a), which shows the effect of increasing the electric field strength from \( V_\text{RMS} = 80 \) to 100 V in a cell of 50 μm thickness at a fixed frequency of 3 kHz containing βOG at 85 °C. Red dotted curve in Fig. 2(a) shows the current with time after applying \( V_\text{RMS} = 80 \) V. There is a very small conduction current of 0.12 mA, whose value is multiplied by 10 times in the normal axis of Fig. 2(a) in order to make it visible. The current observed is constant with time and not a transient current. However, for \( V_\text{RMS} = 90 \) (blue dashed curve) and 100 V (black curve), an immediate increase in the current is seen, reaching a plateau of 14 mA and 18 mA, respectively. These transient currents for \( V_\text{RMS} = 90 \) and 100 V then decayed rapidly at ~110 s and 130 s, respectively, to a steady state value of 0.14 mA. These transient currents were about two orders of magnitude greater than the steady state current. These results clearly also show that there is a threshold field strength between \( V_\text{RMS} = 80 \) and 90 V, which causes the generation of the transient current. During the generation of the transient current, we observed the formation of bubbles. The origin of these bubbles is uncertain and remains an open question.

We have also carried out simultaneous optical transmission measurements during the application of the electric field passing through the cell set between the crossed polarizers, as shown in

FIG. 1. Anhydrous glycolipids used in the experiments: (a) octyl-β-D-glucoside and (b) dodecyl-β-o-glucoside. Glucose was also used for comparison.
Fig. 2(a) (right axis). Since the photocoupler was set in a high temperature atmosphere for measuring the optical transmittance, the output signal was unfortunately too noisy to estimate the absolute value of the transmittance correctly. For both \( V_{\text{RMS}} = 90 \) and \( 100 \) V, we observed a decrease in the light transmission through the cell during the generation of the transient current, which returned back to normal when the steady state of the conducting current is reached. We interpret this as a possible change in the state of the sample of \( \beta \text{OG} \) from its initial SmA state to one in which the order is lost during the period of the transient current occurrence and then back to the ordered SmA state when the current returns to the steady state. For \( V_{\text{RMS}} = 80 \) V, the light transmission did not suffer any change in intensity, confirming that the sample remained in the SmA phase during the time of the voltage application. Figure 2(b) shows the transient currents by the electric field of \( 2 \) MV/m (\( V_{\text{RMS}} = 100 \) V) for dc and various frequencies of \( 5 \) Hz \( \leq f \leq 10 \) kHz in the SmA phase. Almost immediately after the electric field was applied, the current rose to a peak of \( \sim 2 \) mA for the dc voltage and then decayed rapidly to 0 within \( \sim 20 \) s. Similar very rapid buildup of current was observed at 5 Hz to \( \sim 3 \) mA, which decayed slower than the dc case. As the frequency increased further, the peak current and peak time also increased. At \( f = 3 \) kHz, the transient current reached a plateau at \( \sim 18 \) mA and then suddenly dropped to zero after 130 s. However, the transient current at \( f = 10 \) kHz [red dashed curve in Fig. 2(b)] rose rapidly and then saturated at \( -I \approx 21 \) mA with time.

To gain a better insight of the state of the sample during the cycle at \( f = 10 \) kHz, \( V_{\text{RMS}} = 100 \) V, and \( T = 85 ^\circ \text{C} \), we measured the current (left axis) and the optical transmittance (right axis) simultaneously as shown in Fig. 2(c). When the current increased, the optical transmittance decreased and continued to decrease to its lowest value as the current saturates. When we turned off the voltage, the full transmittance was recovered eventually. We have repeated this cycle of simultaneous measurement of the current and transmittance upon the application of voltage and confirmed the pattern just described. The most likely explanation is that the liquid crystal sample is being heated by Joule heating induced by the electric field at \( V_{\text{RMS}} = 100 \) V and \( f = 10 \) kHz.

The frequency dependence of the conductivity for various temperatures in the crystal, SmA, and isotropic phases was investigated, and the results are shown in Fig. 3 for \( V_{\text{RMS}} = 30 \) V, which is low enough not to make the sample heat up and electric damages. As the frequency was increased in the range \( f = 100 \) Hz to 10 kHz, the conductivity changed from \( \sigma = 6 \times 10^{-7} \) to \( 5 \times 10^{-6} \) \( \Omega^{-1} \text{ m}^{-1} \) for the SmA phase at 85 °C. Higher temperatures gave higher conductivity under the same frequency. However, at \( f = 10 \) kHz, the conductivity was found almost temperature independent. The thermal activation energy at \( f = 10 \) kHz is small and is estimated to be \( E_a = 0.04 \) eV. This was obtained from the slope of the Arrhenius plots displaying the logarithm of a conductivity plotted against inverse temperature using the results shown in Fig. 3. The electric field application (\( f = 10 \) kHz) to \( \beta \text{OG} \) causes the phase transition from the SmA to the isotropic phase. Even though this would be initially considered as Joule heating, it is hard to explain the mechanism for the generation of large current by the change of the conductivity due to the change of the temperature from 85 °C to 110 °C. It should be noted that there is no difference in the conductivities measured with and without magnetic field of 7.05 T as used in Ref. 5.
To gain a better understanding of the conditions leading to the generation of large transient currents, the dependence of the current, I, on the electric field strength was investigated in details and the results are shown in Fig. 4. Three cells were used in the investigation in which the thickness was set at d = 10 μm and two frequencies were used, f = 1 kHz (black curve in Fig. 4) and f = 10 kHz (blue dashed curve in Fig. 4). The use of a cell with a thinner film thickness (d = 10 μm) makes the measurements of the current in a high electric field possible. Two temperatures were chosen for these measurements: T = 85 °C in the SmA phase and T = 110 °C in the isotropic phase (red dotted curve in Fig. 4). It was found that as the electric field strength increased, the current also increased linearly. However, as the electric field strength was further increased, the increase in current started to deviate from linearity and the Ohmic line. The black and blue dashed lines in Fig. 4 show the calculated Ohmic relationship between the current and voltage for the conductivities of σ = 1.6 × 10^{-8} Ω^{-1} m^{-1} and 7.0 × 10^{-8} Ω^{-1} m^{-1}, respectively. Although these values are about twice as large as those shown in Fig. 3, the black and dashed blue curves closely follow the Ohmic lines when E < 4 MV/m and E < 7 MV/m, respectively. The red dotted curve, recorded for f = 1 kHz at T = 110 °C, also follows the Ohmic blue line when E < 3 MV/m. In the region of high electric field, both for f = 1 kHz at T = 110 °C (red dotted curve) and f = 10 kHz at T = 85 °C (blue dashed curve), the current increases rapidly at E ~ 9 MV/m and an electrical breakdown occurs then. We found this to be an irreversible phenomenon, and we believe it is due to the injected electrons from the electrodes. Since the conductivities for both conditions are almost the same as shown in Fig. 3, the electrical breakdown occurs at the same electric field. On the other hand, there is no occurrence of the electrical breakdown for f = 1 kHz at T = 85 °C, where σ = 8 × 10^{-7} Ω^{-1} m^{-1}. As shown in Fig. 3, the conductivity of βOG increases with increasing frequency and temperature. The increase of conductivity leads to the electrical breakdown. Our experimental results suggest that the application of E = 2 MV/m to βOG, as shown in Fig. 2(b), does not cause electrical breakdown. The occurrence of electrical breakdown can be confirmed by observing the samples exposed to the high electric field under POM at room temperature without the electric field. Figures 5(a)–5(c) were recorded for samples at applied electric field strength of 2 MV/m at f = 1 and 10 kHz at T = 85 °C, and f = 1 kHz at T = 110 °C, respectively. The white crossed arrows show the directions of the polarizer and analyzer. It is clear from Fig. 5(a) for f = 1 kHz at T = 85 °C that the sample showed the focal conic texture and was not damaged so much by the electric field application. The sample in Fig. 5(b) at f = 10 kHz and T = 85 °C appears to have been partially damaged as displayed by the red arrows. On the other hand, the sample in Fig. 5(c) at f = 1 kHz and T = 110 °C was crushed by the electrical breakdown, whose crushed lines appeared clearly displayed by the red arrows. These results suggest that, when the electric field is smaller than 8 MV/m, an electrical breakdown does not occur under the conditions of frequency f = 1 kHz and T = 85 and 110 °C, and also at f = 10 kHz and T = 85 °C. That is, the electric field of 2 MV/m is too small to cause the electrical breakdown. The deviation of the current from the Ohmic line for f = 1 kHz at T = 85 °C and 110 °C at high electric field strength would be due to the electric carriers generated by the dissociation of ions from the βOG or from impurity molecules in the sample, and/or a thermal activation of mobile carriers in the sample.

As previously described for the sample with d = 50 μm, the transient current generated by the application of the electric field showed the occurrence of a threshold value for the electric field strength, E_{TH}. Figure 6 shows the dependence of the threshold electric field on the value of the sample thickness d. When d ≥ 25 μm, threshold electric field is almost constant at ~2 MV/m at f = 1 kHz and T = 85 °C. This suggests that a threshold field is needed to accelerate free electrons in βOG to an ionizing energy level. When the kinetic energy of the electrons exceeds the ionizing energy, these electrons then through collisions with the βOG accelerated charged molecules
generate more electrons. As a result, a large amount of electric charges is generated by these repeated collisions. This would lead to the so-called electron avalanche breakdown mechanism. When the generated carriers flow out to the electrodes, the impact of ionization is completed. Simultaneously, a large number of molecules are damaged by the collisions during the impact ionization, thereby weakening the molecular interaction stabilizing the SmA phase. This would result in the phase change from the SmA to the isotropic phase as observed in the experiments and is probably the reason why the generation of the transient current is irreversible.

The process of the impact ionization may be understood by estimating the transit distance, \( l \), of electrons during the half-period time \((1/2f)\). It is given by the relation, \( l = \nu/2f = \mu E_{TH}/2f \), where \( \nu \) and \( \mu \) are the electron velocity and mobility, respectively, assuming a uniform electric field \((E_{TH})\) in the sample. By using the values of \( \mu = 2 \times 10^{-8} \text{ m}^2/\text{V s} \) taken from Ref. 20, \( E_{TH} = 2 \text{ MV/m} \), and \( f = 1 \text{ kHz} \), the transit distance is estimated to be 20 \( \mu \text{m} \). When the accelerated electrons can transit a longer distance than 20 \( \mu \text{m} \), then the kinetic energy of these electrons is higher than the ionizing energy. In other words, when the transit distance is shorter than 20 \( \mu \text{m} \), the impact ionization does not occur at \( E_{TH} \approx 2 \text{ MV/m} \) and \( f = 1 \text{ kHz} \). This is the case of samples with \( d = 10 \) and 15 \( \mu \text{m} \) thickness. That is, when \( d < 20 \mu \text{m} \), the thinner the samples, higher electric field is needed to get the kinetic energy to go over the ionizing energy as shown in Fig. 6. Although the result for the sample with \( d = 5 \mu \text{m} \) thickness is not shown in Fig. 6, the transient current was not observed even by applying the high electric field of \( E = 11 \text{ MV/m} \). Another example is the result measured under the conditions of \( d = 50 \mu \text{m}, E_{TH} = 2 \text{ MV/m}, \) and \( f = 10 \text{ kHz} \) at \( T = 85 \text{°C} \) as shown in Fig. 2(b). In this case, the estimated transit distance is \( \sim 10 \mu \text{m} \), because \( f = 10 \text{ kHz} \). This is not long enough distance to accelerate the electrons and so results in the absence of the transient current. These provide further evidence to support the proposed simple mechanism of the threshold electric field, which causes the avalanche breakdown based on the impact ionization process. The relationship between the threshold electric field and the sample thickness is simply expressed by \( E_{TH} = A/\ln(B \cdot d) \), where \( A \) and \( B \) are physical parameters associated with the sample. Dashed line in Fig. 6 is calculated with \( E_{TH} = 5.3/\ln(0.2d) \) and follows fairly closely the experimental results. The physical parameters \( A \) and \( B \) are related to the collision relaxation time and the average kinetic energy of electron. From the present experimental results, a more detailed discussion on them is limited.

The generation of transient current was further tested for the related lipid \( \beta \text{OG} \) [Fig. 1(b)], which has a longer alkyl chain than \( \beta \text{DG} \). The measurements were performed using \( \beta \text{DG} \) with \( d = 25 \mu \text{m} \) thick cells and applying the electric field at \( E = 2.8 \text{ MV/m} \), in the frequency range of \( 100 \text{ Hz} \leq f \leq 3 \text{ kHz} \) and at \( T = 120 \text{°C} \),

![Graph showing the relationship between electric field and sample thickness for threshold electric field](https://example.com/graph.png)

**Fig. 6.** Dry octyl-\( \beta \)-o-glucoside sample thickness dependence of the threshold electric field, \( E_{TH} \), to cause a transient current. Dashed line is calculated by \( E_{TH} = 5.3/\ln(0.2d) \).

**Fig. 5.** After the application of the voltage, the samples were observed by POM at room temperature without the electric and magnetic fields. (a)–(c) were recorded for the samples (dry octyl-\( \beta \)-o-glucoside) measured under the conditions of \( f = 1 \text{ kHz} \) at \( T = 85 \text{°C} \), \( f = 10 \text{ kHz} \) at \( T = 85 \text{°C} \), and \( f = 1 \text{ kHz} \) at \( T = 110 \text{°C} \) (isotropic phase), respectively. White crossed arrows show the directions of the polarizer and analyzer. It is apparent from Fig. 5(a) that the director alignment remains after the voltage application.
which is 23°C lower than the SmA to the isotropic phase transition temperature for βOG. The conductivity of βDG was measured at $T = 120^\circ$C in the SmA phase and was smaller by one order of magnitude compared with βOG (shown in Fig. 3). Under these conditions, βDG also showed current and peak time decrease with the decrease in the frequency, similar to those shown for βOG in Fig. 2(b).

Finally, an electric field ($E = 4$ MV/m) was applied to a $d = 25 \mu$m thick cell containing just glucose at $T = 130^\circ$C. Since glucose has no liquid crystalline phase, no transient current was observed as expected.

IV. CONCLUSION

In summary, the electric charge generation in an anhydrous βOG and βDG was investigated. When an electric field ($E = 2$ MV/m) was applied to the sample, a large transient current was generated, whose value was about 100 times larger than the steady state current. The generation of the transient current was observed in the SmA phase, but not in the isotropic phase. The large transient current was accompanied by a phase change from the SmA to the isotropic phase and the generation of air bubbles. The generation of the transient current and the phase change were found to be irreversible by the repetition of the field application. These results suggest that the phase change is originated in the conformational change in the headgroup, but not in the chain structure, of the anhydrous βOG. This effect, we believe, is due to the accelerated electrons. The threshold electric field was also shown to depend on the sample thickness. This property is similar to the electron avalanche breakdown caused in a solid insulator. The avalanche breakdown due to the impact ionization in the SmA phase of anhydrous βOG was proposed as one possible mechanism for the generation of the transient current. This proposed mechanism does not result from an electrical breakdown since the electric field strength required to cause the electrical breakdown of the sample is about 5 times larger than the threshold electric field. We found no significant difference in the transient current measured with and without the magnetic field.

It should be noted that “electrical breakdown” is an electric phenomenon that occurs by injected electrons from electrodes to the sample, and it has no relation with phases of glycolipids and is triggered by injected carriers. On the other hand, “avalanche breakdown” reported in this paper is an electronic phenomenon initiated by carriers generated in the smectic phase of glycolipids, which is triggered by generated carriers in the sample.

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