

Qualification of liquid crystal mixtures by bulk-state transient current analysis

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Abstract

Transient current (TC) on bulk-state liquid crystal mixtures was measured. We found that TC is very sensitive to impurities and the features of TC curves depend on the type of contamination, from which the quality of materials can be definitely evaluated and the type of impurities can also be revealed.

1. Introduction

Nowadays liquid crystal displays (LCDs) have been the dominating technology among flat panel displays. Due to the development of advanced materials and display modes, the optical performance of LCD (e.g., response time, color saturation, contrast ratio and view angle) has been improved greatly in these years. In such circumstances, therefore, the long-term reliability of LCDs becomes more and more important and often turns to be the key parameter to qualify panels.

Deterioration of display quality by the appearance of surface-type and line-shape image sticking defects [1,2] is often observed in long-term operated LCDs, and the time for these defects to show up determines the reliability of the display. It has been recognized that the main cause of these defects is the presence of ionic/ionizable impurities in liquid crystal (LC) mixtures [3]. Since such impurities facilitate electrical conduction through LC mixtures, the specific resistivity (ρ_s) measurement of LC mixtures has become a routine procedure to evaluate the quality of LC mixtures.

Though ρ_s can be used to qualify LC materials, information revealed by this index is nonetheless very limited. Moreover, electrical conduction in dielectric liquids is actually time-dependent [4, 5], which means

the qualification of LC samples by ρ_s could be misleading if detailed measurement parameters are not specified.

Therefore, we developed a transient current (TC) measurement system to monitor the time-dependent current passage in bulk-state LC mixtures. We found that TC is very sensitive to the presence of impurities, from which the quality of LC samples can be definitely discriminated. Besides, inorganic and organic contaminants present different TC features, which can help to track the impurities in LC samples.

2. Experimental

The LC mixture investigated here is a commercial negative-type LC. The LC sample saturated with NaCl is prepared as follows. Crystals of dry NaCl were added into the LC (ca 0.1g/20mL) and sonicated in a carefully sealed vial for 24 hours. Excess and undissolved salts were then expelled from the sample by 0.45- μ m micro-filters. The Na⁺ content of this salt-saturated sample was measured to be 21 ppb by an inductively-coupled-plasma mass spectrometer (ICP-MS), while that of the clean LC is found below the detection limit. Then these two samples were blended in different ratios to prepare other samples with an apparent Na⁺ content of ca. 14, 10, 7 and 5 ppb, respectively. The UV-dosed LC sample was prepared by exposing the clean LC mixture to a 95-mW/cm² 365-nm UV lamp for 30 minutes (dosage: 171 J/cm²). The humidified LC sample was prepared by 72-hr exposure of the clean LC mixture to saturated water vapor in a close environment. The water content of LC increases by ca 50 ppm after the humidification, which was determined by a Karl-Fisher coulometer.

Transient currents of LC samples in the test module (gap: 1 mm) were then taken at room temperature by a software-controlled high-resistance meter. Before each measurement, the LC test module was carefully cleaned and dried. And each sample was measured repeatedly to guarantee the reproducibility of the results. As a comparison, the ion density of test cells (gap: 3.8um and PI: JSR AL60101L) were measured for the investigated LC samples by the TOYO 6254 LC material characteristics measurement system.

3. Results and discussion

The effect of the sampling timing on resistivity in a routine measurement is shown in figure 1. The measurement was carried out at 10V in a 1-mm gap test module. The time dependence of current and resistivity is clearly revealed. The specific resistivity (ρ_s) of a LC mixture is normally obtained by one sampling from a stable region of curves in figure 1, say, a point somewhere after 1 min. However, such a sampling may cause confusion since in this region the ρ_s of clean LC and that of NaCl-contaminated LC are still on the same order and the small difference between samples may be misunderstood as an experimental error. Therefore, it would be very difficult to be aware of the presence of trace impurities by just comparing the ρ_s of two samples of a LC mixture. Moreover, if samplings are not specified at the same time, qualification of samples by ρ_s would even become misleading.

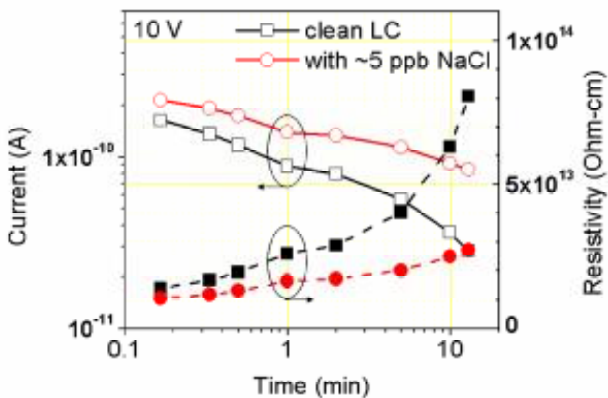


Fig. 1. The electrical current and the corresponding resistivity of a commercial VA-LC mixtures in a routine resistivity measurement (gap=1 mm, voltage=10V DC). The diagram is a log-log plot.

To avoid the ambiguity in the routine resistivity test as mentioned above, we set up a transient current (TC) measurement system to monitor the time-dependent current passage in “bulk-state” liquid crystal mixtures. From this system, TC curves of LC (the same one as in figure 1) with different extents of NaCl content were recorded as Figure 2(A). A bipolar-double-rectangular pulses scheme (shown in figure 2(B)) was applied for the TC measurement. The bipolarity of pulses is to enhance the influence of impurities so that the detection of impurities can be more sensitive and definite [6]. The voltage of the pulses used for TC measurement is +/-500V to produce an electric field (0.5 MV/m) close to that in real panels. It clearly demonstrates that TC increases with the NaCl content. Besides, additional TC peaks (marked by arrows in figure 2 (A)) show up, which is definitely due to the increase of NaCl content in LC.

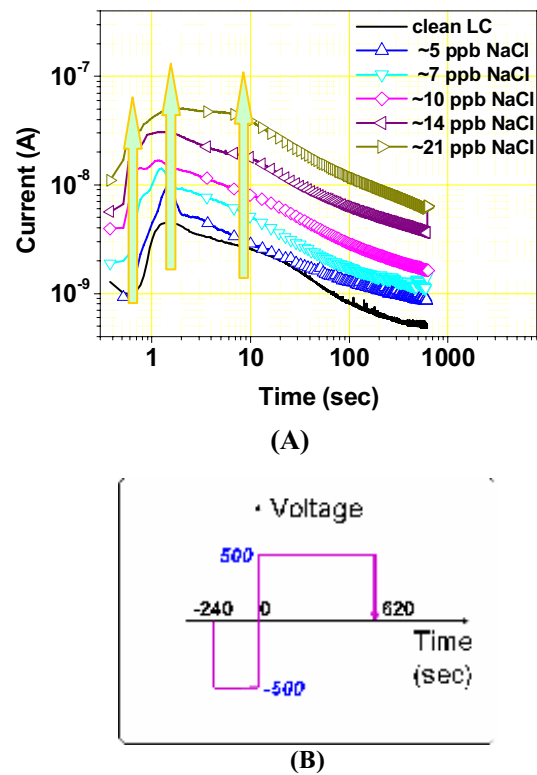


Fig. 2. (A) Transient current curves of LC mixtures (the same as that in figure 1) with different extent of NaCl content; (B) the driving scheme for the measurement. The diagram (A) is a log-log plot.

The integrated area of curves in figure 2(A) is the charge conducted by TC and can be an index to indicate the extent of contamination in LC, as shown

in figure 3. The charge increases linearly with NaCl content. (Since samples containing NaCl are prepared by diluting the NaCl-saturated LC with the clean sample, the deviation from linearity in the low NaCl content region should be mainly caused by the impurities already present in the clean LC). Therefore, samples of this LC can be qualified by checking the “effective NaCl content” from figure 3.

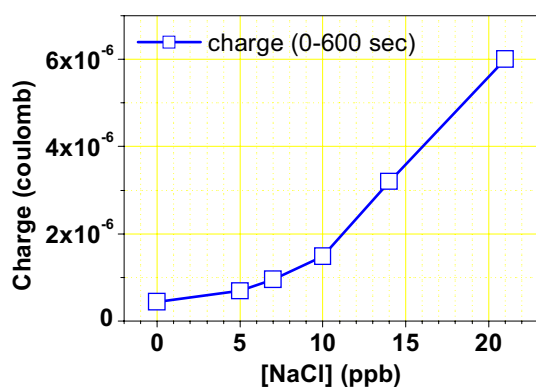


Fig. 3. The integrated area (charge) of TC curves in figure 2(A) for LC samples with different apparent content of NaCl.

In the bipolar-driving procedure for TC measurement, ions are drawn near to electrodes by the first negative pulse and ions are then swept in the opposite direction by the second positive pulse. Since during the second pulse the motion of ions is measured as current, both the rate and the dispersiveness of ion motions can affect the strength of current and the features of TC curve. From this point of view, the TC measurement adopted here is somewhat similar to the time-of-flight (TOF) technique [7], from which the motion of charge carriers (electrons and holes) in solid can be characterized.

In addition to the LC samples containing NaCl, we have also measured TC curves for UV-exposed as well as humidified LC samples. The results are shown in figure 4. The most attractive phenomenon is that each type of contaminants in LC has its own TC features:

i) Inorganic impurities (e.g., Na⁺ and Cl⁻) introduce multiple peaks in the short time region (< 10 sec, as shown in figure 2(A)), indicating the relatively faster motion of these kind of contamination;

ii) Deteriorated organic molecules result in a relatively broad TC plateau (up to 100 sec), indicating a dispersive drift of impurities and a relatively slower mobility;

iii) The influence of an increase in the water content by tens of ppm on TC feature is very limited

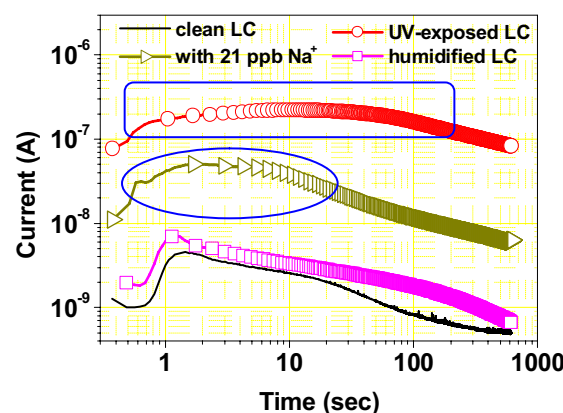


Fig. 4. Transient current curves of LC mixtures (the same as that in figure 1) with different type of contaminations: NaCl-containing LC, 171 J/cm² UV-dosed LC and LC with humidity 50 ppm higher than the clean sample. The diagram is a log-log plot.

As shown in figure 4, inorganic and organic contaminants in LC mixtures present different features in their TC curves. This result is novel but reasonable since inorganic ions are usually smaller and have higher charge density than ionic/ionizable organic molecules. Therefore, the motion of organic impurities in LC mixtures is slower and probably more dispersive than inorganic ions, making TC curves broader and flatter. From figure 4, the mobility of inorganic ions (from NaCl) and ionizable organics (the deteriorated LC molecules) in the investigated samples can be roughly estimated to be on the order of 10⁻¹⁰ and 10⁻¹¹ m²/Vs, respectively.

Test cells of the investigated LC samples were also measured for the ion density, which is an index for the cleanness of materials. As shown in figure 5, test cells of all LC samples have an impurity peak, which could originate from LC itself, other cell materials and process. For the bulk-state TC analysis method, the cleanness of samples can be evaluated by the charge conducted by TC curves. To compare these two methods, the charge densities of LC samples (nC/μL) are calculated from figures 4 and 5, and the result is shown in figure 6. Both show that the content of impurities is in this order: UV-exposed LC > salty LC > clean LC. However, discrimination between samples by orders of magnitude is observed in the

bulk-state TC measurement, while the difference between samples in the ion density test is distinguishable but very limited.

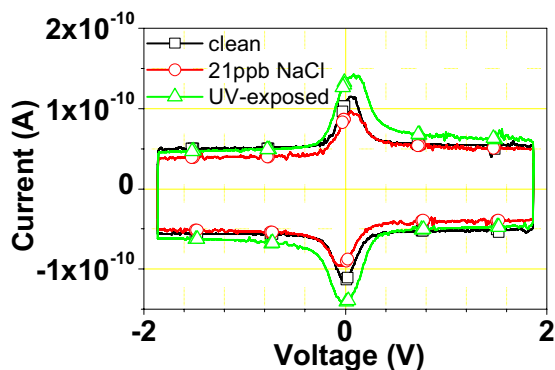
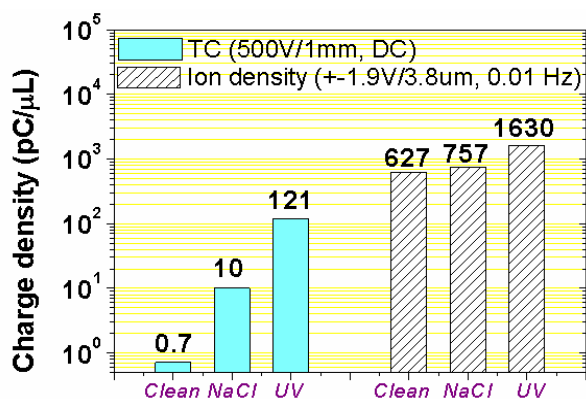


Fig. 5. Impurity peak from the ion density measurement of test cells (+/-1.9V, 0.01 Hz).



TC of bulk-state LC Ion density of test cells
Fig. 6. The charge density of LC samples calculated from transient current curves (figure 4) and ion density measurements of test cells (figure 5).

The results above clearly demonstrate that transient current measurement on bulk-state LC can qualify samples very definitely, and the distinguishability of this method is better than the common evaluation procedures, such as the specific resistivity test of bulk-state samples and the ion density measurement of test cells.

4. Summary

Transient current (TC) measurement was conducted on “bulk-state” liquid crystal mixtures. We found that this analysis technique can qualify LC mixtures very definitely and provides better distinguishability than

common evaluation methods such as the specific resistivity test of bulk-state samples and the ion density measurement of test cells. And contamination in samples of a liquid crystal mixture is revealed both qualitatively and quantitatively by this measurement. Therefore, the content and the type of impurities in LC can be estimated by the integrated area and the features of TC curves, respectively.

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