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PAPER

Analysis of metal ion impurities in liquid crystals using high resolution inductively coupled plasma mass spectrometry

Huan-Yi Hung,[ab] Chien-Wei Lu,[ab] Cheng-Yu Lee,[a] Chain-Shu Hsu[a] and You-Zung Hsieh*[a]

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We have developed a method, using high resolution inductively coupled plasma mass spectrometry (HR-ICP-MS), to directly analyze metal ion impurities in liquid crystals (LCs). From measurements of resistivity, a commonly used technique for quality control of liquid crystal display (LCD) cells, we found that the resistivities of the LCs decreased significantly upon progressing through the different stages of the LCD cell manufacturing process. We determined the levels of metal ion impurities in the LCs through direct loading of diluted LC samples into the HR-ICP-MS system at a steady flow rate. Solutions featuring LC contents of 10% (v/v) were formed by dissolving each LC in isopropanol (IPA) containing acetone as a modifier. We quantified the trace metal ion impurities in the LC samples using a certified standard of metal ions diluted in IPA (external calibration) without the need for digestion pretreatment. Next, we compared the levels of metal ion impurities with the resistivities of the LCs at the different stages of the LCD cell manufacturing process. We confirmed that the resistivities of the LCs decreased as the total levels of metal ion impurities in the LCs increased. The total content of metal ion impurities added into the LC was higher in the siphoning process than in the one-drop filling process during the fabrication of LCD cells. The HR-ICP-MS method could, therefore, be used to directly measure metal ion impurities in LCs during the LCD cell manufacturing process, potentially replacing resistivity measurements of LCs as a means of quality control. We suggest setting 1400 ng L^{-1} as the limit of the total metal ion concentration in LCs used for LCD cell manufacturing. We used the HR-ICP-MS method to analyze a stained LCD panel to confirm that the content of metal ion impurities was indeed significantly greater in the stained area of the defected LCD. HR-ICP-MS appears to be a promising technique for the direct and effective analyses of metal ion impurities in LCs.

Introduction

Liquid crystal displays (LCDs) are the most common flat-panel displays in the world today. Although liquid crystals (LCs) were first discovered 100 years ago, only during the past 20 years they have become widely used. LCs are formulated materials that normally comprise a number of different types of molecules, typically featuring similar rigid groups (*e.g.*, cyclohexane, fluorinated benzene); as such, they are difficult matrices for metal ion analysis. The LCs within an LCD cell are driven by a voltage that switches the panel. The presence of metal ion impurities is one of the main factors influencing LCD quality through electric field shielding. Many studies have revealed that metal ion impurities in LCDs can result in image degradation, including image sticking, flickering, and staining in field shielding.[1–6] To avoid the introduction of excessive amounts of metal ion impurities, leading to LCD yield loss, the materials and surroundings that come into contact with the LC should be strictly controlled during the LCD cell manufacturing process.

The resistivity of LCs is conventionally measured to assess the levels of ion impurities in the LCs during the LCD production process.[6,7] The resistivity is tested for quality control of the LCs prior to their injection into the LCD cell. Although the resistivity of an LC is an indirect measurement of the level of metal ion impurities, no direct methods have been reported previously for the determination of metal ion impurities in LCs. Measurements of the voltage holding ratio (VHR),[8,9] transient current,[10,11] and residual direct current[11] have all been used during LCD production to measure metal ion impurities. These studies have indicated that the presence of metal ion impurities can have a dramatic effect on LCD performance. Nevertheless, these methods are indirect approaches toward the quantification of metal ion impurities in LCs. Ideally, measurement of the real content of metal ion impurities in LCs would benefit the routine analysis of LCD cells during the manufacturing process.

Inductively coupled plasma mass spectrometry (ICP-MS) can be employed to determine trace metals in various types of

[a]*Department of Applied Chemistry, National Chiao Tung University, Hsinchu, Taiwan. E-mail: yzhsieh@mail.nctu.edu.tw; Fax: +886-3-5723764; Tel: +886-3-5731785*
[b]*Industrial Technology Research Institute, Hsinchu, Taiwan*

samples.[12–14] The attractive features of ICP-MS include rapid multi-element analyses, high sensitivity, and low detection limits. Wet microwave digestion pretreatment of the sample, prior to ICP-MS analyses of trace metals in organic samples, eliminates interference caused by organic materials in the sample.[15–18] The pretreated sample is diluted with deionized water in order to minimize the effect of the acidic matrix and then subjected to ICP-MS measurement.

Organic samples can be introduced directly into ICP-MS systems for measurement of their trace metal ion contents.[19–22] Direct introduction of an organic sample will decrease the sample pretreatment time, but interference from the matrix will remain. Although non-spectral matrix interference can be resolved using the method of standard addition (MSA), it is a time-consuming process that is not applicable to large-scale sample analyses. The direct introduction of an LC sample into an ICP-MS system without using MSA would be a time-saving experimental process. The viscosity of a sample can negatively affect the sample flow rate and ICP-MS signal intensity.[22,23] The poor nebulization efficiency of a sample and incomplete combustion can also be deleterious when organic samples are introduced into ICP-MS systems. To solve these problems, a self-aspiration perfluoroalkoxy nebulizer (PFAN), operated at a flow rate of 50 μL min^{-1}, can allow smooth injection of the LC sample. At this low flow rate, complete combustion of the organic sample can be achieved by adding O_2 to avoid interference from the sample.[24–26] In this present study, we used isopropanol (IPA) solutions containing acetone to dilute the LCs; these diluted LC samples possessed the same viscosity as pure IPA.

Spectral interference in ICP-MS is often difficult to resolve, especially when the analyte concentration is low. Spectral interference arises mainly from the plasma, nebulizer gas, and matrix components in a sample. In this present study, we used high resolution inductively coupled plasma mass spectrometry (HR-ICP-MS) to separate the analyzed metal ion from the interfering ions by selecting an appropriate resolution that would eliminate any ambiguous identification of metal ion impurities.[12–14,22] We found that the HR-ICP-MS system, when used at an appropriate resolution, in conjunction with a PFAN operated at a low flow rate, did not suffer from spectral interference. Thus, with the use of a direct organic sampling HR-ICP-MS system and a low-flow-rate PFAN, we could effectively analyze metal ion impurities in the LC samples.

No data are available currently regarding the levels of metal ion impurities in LCs used in the manufacturing of LCD cells. Only the resistivity of LCs has been used previously as a quality control measure of ionic impurities in LCs prior to their use in the production of LCDs. In this study, we applied HR-ICP-MS in conjunction with low-flow PFAN to directly analyze metal ion impurities from three LC samples employed in different LCD cell manufacturing processes. We prepared solutions of LCs at a concentration of 10% (v/v) in IPA modified with acetone. We determined the metal ion concentration in each LC sample according to external calibration of metal ions in pure IPA. Because different LCs possess different viscosities, we varied the amount of acetone in the modified IPA solution for each LC. The viscosities of the diluted LC samples matched the viscosity of pure IPA. We established the trend in the total metal ion concentration in each LC with respect to the variation in resistivity of the LC. Finally, we used our HR-ICP-MS method to measure the levels of metal ion impurities in stained and normal regions of a defective 42″ LCD panel, obtaining direct evidence for metal ion impurities in LCs influencing LCD performance.

Experimental

Chemicals

Ultra-pure selected (UPS) grade IPA and acetone were obtained from BASF Taiwan (Taoyuan, Taiwan). Certified 1000 ppm standard metal ion solutions were purchased from Merck (Darmstadt, Germany) to establish the external calibration curves. Certified 10 ppm standard metal ion solutions were purchased from Ultra Scientific (North Kingstown, RI, USA) to verify the external calibration curves. Deionized water was obtained from a Milli-Q System (Millipore, Bedford, MA, USA). Standard solutions of metal ions in IPA were freshly prepared by diluting stock multi-element standard solutions. The various LCs in this study were provided by Chimei Innolux (Tainan, Taiwan), originally produced by Merck and Chisso.

Apparatus

A 6517A electrometer/high resistance meter (Keithley Instruments, Cleveland, OH, USA) and an RST-100 LC resistivity measurement system (Quatek, Taipei, Taiwan) were used for the resistivity tests. The working range of the LC test was from 1.00×10^9 to 1.00×10^{15} Ω cm. The clean room for the experiment was maintained at 22 ± 1 °C with 50% humidity in a class-1000 environment. HR-ICP-MS was performed using a Thermo Scientific Element 2 instrument (Thermo Scientific, Bremen, Germany). The sample introduction system, a PFA-50 nebulizer (ESI, Omaha, NE, USA), was operated at an optimal flow rate of 50 μL min^{-1} and fitted with an ES-2104 basic quartz water-cooled spray chamber (ESI, Omaha, NE, USA). The sampler cone and skimmer cone were made of Pt. The purities of Ar and O_2 exceeded 99.995%. The analytical resolution ($m/\Delta m$) of the HR-ICP-MS system was adjusted to 300 (low) for ^{7}Li and ^{23}Na; 4000 (medium) for ^{24}Mg, ^{27}Al, ^{40}Ca, ^{52}Cr, ^{56}Fe, ^{59}Co, ^{59}Ni, ^{64}Cu, and ^{65}Zn; and 10 000 (high) for ^{39}K. Table 1 lists the operating parameters of the HR-ICP-MS system for the LC samples.

Sample preparation

Three LCs used in a thin film transistor (TFT)-LCD were tested during the experiment: two were twist nematic (TN)-type used in a cell phone (LCTN-C) and a monitor (LCTN-M) and one was vertical alignment (VA)-type used in a television (LCVA-T). LCTN-M had a faster response time and a lower threshold voltage than LCTN-C. The siphoning process or one-drop filling (ODF) process was employed for LCD cell manufacturing. The LC sample was collected at three different stages of the LCD cell manufacturing process—namely, before, during, and after the LC was injected into the LCD cell. Seven LC samples were removed at each stage to examine their resistivities and levels of metal ion impurities. Sample preparation was performed at a class-1 booth in a class-1000 clean room to prevent sample contamination. The 6517A resistivity meter was calibrated, using

Table 1 Analytical parameters for the HR-ICP-MS system

Thermo Scientific Element 2 (Thermo Scientific)	Parameter
Nebulizer	Microflow
Nebulizer flow rate, μL min^{-1}	50
Spray chamber	Quartz, water-cooled
Injector	Quartz
Sampler cone	Pt
Skimmer cone	Pt
Peristalsis pump speed, rpm	5.00
RF power, W	1500
Plasma Ar flow rate, L min^{-1}	17.00
Auxiliary Ar flow rate, L min^{-1}	0.75
Sample Ar flow rate, L min^{-1}	0.645
O_2 flow rate, L min^{-1}	0.145
Extraction lens, V	−1970.0
Focus lens, V	−890.0
X-Deflection lens, V	2.32
Y-Deflection lens, V	3.48
Shape lens, V	125.00
Resolution ($m/\Delta m$)	300, 4000, 10 000 (low, medium, high)[a]

[a] The analytical resolution ($m/\Delta m$) of the HR-ICP-MS system was 300 for ^{7}Li and ^{23}Na; 4000 for ^{24}Mg, ^{27}Al, ^{40}Ca, ^{52}Cr, ^{56}Fe, ^{59}Co, ^{59}Ni, ^{64}Cu, and ^{65}Zn; and 10 000 for ^{39}K.

a standard, to 1.00×10^{13} Ω cm prior to measurement. A clean test cell was used to collect and carry a 1 mL sample of the LC; the LC resistivity was measured at 10 V. The test cell was cleaned with UPS-grade acetone and dried in a class-1 clean booth for 30 min prior to the next gauging to avoid any influence from residual acetone or ionic impurities.

The concentrations of metal ions in the LCs were determined by HR-ICP-MS based on external calibration curves of standard solutions of the elements analyzed at concentrations of 10, 30, 50, 100, 500, and 1000 ng L^{-1}, diluted in IPA. Each solution of LCs having a concentration of 10% (v/v) was diluted with a modified IPA solution prior to injection into the HR-ICP mass spectrometer. The viscosity of each LC sample matched that of pure IPA. The metal ion impurities in stained and normal regions (each area: 200 cm^2) of a defective 42″ LCD panel were analyzed using HR-ICP-MS. The LC within these regions (*ca.* 0.08 g) was extracted using IPA. The extraction process was performed at a class-1 booth in a class-1000 clean room to prevent sample contamination. The micro-pipe tip and polypropylene tube for extraction were soaked in 20% HNO_3 and cleaned with deionized water prior to use. The volume of extracted LC sample was adjusted to 4.0 mL prior to direct injection into the HR-ICP mass spectrometer.

Results and discussion

In this study, we tested three different LCs—LCTN-C, LCTN-M, and LCVA-T—as representative samples of most of the types of LCs used industrially. An LC is a formulated material that normally comprises approximately 10 different molecules, each featuring similar rigid groups (*e.g.*, cyclohexane, fluorinated benzene).[7] The resistivity of an LC is typically measured during the manufacture of LCD cells in order to evaluate its variation with respect to other LCs and different LCD cell manufacturing processes. Fluorinated LCs are very stable; these hydrophobic LC compounds are difficult to completely digest in a microwave digestion system. In a preliminary study, we placed 0.1 mL of an LC solution and 3.0 mL of HNO_3 into a microwave digestion bottle; in a microwave reactor with the power set at 540 W, we ramped the temperature from room temperature to 190 °C within 60 min and then held the system at that temperature for 30 min. The LCs were not digested fully when using this microwave pretreatment method to potentially eliminate organic interference. Therefore, we used modified IPA solutions to fully dissolve the tested LCs and then directly injected the diluted and dissolved LC samples into the HR-ICP mass spectrometer in order to determine their metal ion impurities, thereby avoiding potential contamination during sample pretreatment.

Variations in metal ion resistivity during the manufacture of LCD cells

The LC resistivity test is generally used as quality control to assess the content of trace metals when applying LCs in LCD production. The resistivity is an indicator of the quality of the LC. In this study, we selected three types of LCs frequently used in LCDs to examine the changes in LC resistivity that occur during the manufacture of LCD cells. For these three LCs, two types of injection processes are used. In the real manufacturing of LCD cells, LCTN-C is injected using a siphoning process, whereas both LCTN-M and LCVA-T are injected using an ODF process. The viscosities of LCTN-C, LCTN-M, and LCVA-T are 30, 11, and 20 cps, respectively. We collected the LC samples at three different stages of the LCD cell manufacturing process: before, during, and after the LCs were injected into the LCD cell. Fig. 1 displays the resistivity of each LC sample at the various stages of the LCD cell manufacturing process. Prior to injection into the LCD cell, each LC had an average resistivity of greater than 1.90×10^{14} Ω cm. This value decreased after the injection process, implying that the level of ionic impurities increased in each LC, regardless of the injection process.

The change in resistivity of LCTN-C (>174-fold) was the largest among the three tested LCs; those of LCTN-M and LCVA-T were 5.6- and 20.5-fold, respectively. These results

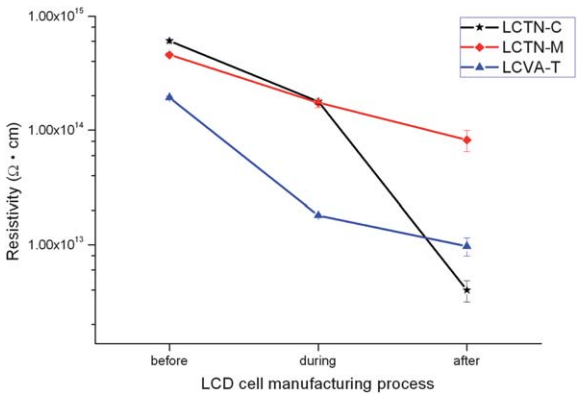

Fig. 1 Resistivities of LCs during the LCD cell manufacturing process. Three LCs were studied: LCTN-C, incorporated using a siphoning process, and LCTN-M and LCVA-T, incorporated using ODF processes. Seven LC samples were obtained from each of the three stages (before, during, after) of injection of the LCs in the LCD cell manufacturing process. Each data point represents the average from seven LC samples.

indicate that the changes in resistivity were significantly greater when the LCD cell was manufactured using the siphoning process than when the ODF process was applied, presumably because the siphoning process requires a longer processing time, leading to more direct contact of the LC with the manufacturing instrument. In addition, an LC with a large viscosity usually facilitates ion dissolution. Because the viscosity of LCTN-C was the largest among our three tested LCs, the effects of both siphoning and viscosity increase the risk of contaminating LCTN-C with ionic impurities. Relative to LCTN-C, the resistivities of LCTN-M and LCVA-T varied only moderately after performing the ODF process. Our experimental results indicate that the resistivity decrease of LCTN-M was less than that of LCVA-T. Because both LCs were subjected to the ODF process, we attributed their different changes in resistivity to their different original viscosities. LCVA-T, with its viscosity greater than that of LCTN-M, would be subjected to enhanced dissolution of ions during the manufacturing process.

The resistivity of LCs is currently used as a quality control specification during the manufacture of LCD cells. It has been suggested by the LC manufacturing company that an LC used in an LCD cell manufacturing process should have a resistivity of greater than 1.00×10^{13} Ω cm. If the resistivity of the LC is less than 1.00×10^{13} Ω cm, the concentration of ions in the LC would most likely be a negative influence on the production yield. Because the presence of metal ions in LCs can result in poor LCD performance, it would be preferable to directly determine the actual levels of contaminating metal ions in the LCs rather than infer them from the resistivity. In this study, we developed an HR-ICP-MS method to determine the concentrations of metal ions in LCs.

Analysis of metal ions using HR-ICP-MS

HR-ICP-MS can be used to determine the quantities of metal ions in an organic solvent. Here, we dissolved the standard metal ions in IPA and then diluted the solution to a predetermined concentration. We then injected the diluted standard sample directly into the HR-ICP mass spectrometer through a 50 μL PFAN for metal ion analysis; adding O_2 into the ICP torch facilitated the decomposition of the organic matrix, greatly minimizing the carbon residue in the sampler and skimmer cones. The left half of Table 2 lists the parameters of the calibration curves for the various metal ions dissolved in IPA and the corresponding coefficients of determination (R^2). The calibration curves of the various metal ions were established at concentrations between 10 and 1000 ng L^{-1}, with values of R^2 greater than 0.999. We also used the HR-ICP-MS method to evaluate the metal ion content of pure IPA. The metal ion with the highest concentration in pure IPA was Na^+ (*ca.* 10 ng L^{-1}); all other metal ions were present at less than 10 ng L^{-1}. Because the metal ion impurities in pure IPA were very low, it appeared to be an ideal solvent for subsequent analyses of the metal ion concentrations in the LC samples.

IPA solvent modified with acetone for LC dilution

The tested LCs could be dissolved in acetone at a concentration of 50% (v/v), but they could not be injected directly into the ICP system as such because acetone provided an unstable ICP. Because the LCs could not be dissolved completely in IPA at a concentration of 10% (v/v), we chose to modify IPA with acetone to ensure complete solubility. These LC solutions could be injected directly into the plasma because the modified IPA solution was stable in the ICP, even in the presence of acetone. Because the tested LCs had different viscosities, due to the various LC characteristics, we varied the amount of acetone that we added to the modified IPA solutions to obtain final LC samples with similar viscosities. This approach allowed us to use HR-ICP-MS to directly analyze the levels of metal ion impurities in the diluted LC samples through comparison with the externally calibrated metal ions in pure IPA. The viscosities of the diluted LC samples matched the viscosity of pure IPA, allowing us to maintain the same, steady sample uptake rate as that for IPA when directly injecting the samples into the HR-ICP mass spectrometer. Based on these considerations, we added 2.8% (v/v) of acetone in IPA for dilution of LCTN-C, 1.0% (v/v) of acetone in IPA for dilution of LCTN-M, and 2.0% (v/v) of acetone in IPA for dilution of LCVA-T; the resulting viscosity of each LC sample was 2.7 cps, the same as that of pure IPA. Adding acetone in IPA to adjust the viscosity of the LC sample minimized any interference arising from variations in the sample uptake rate in the ICP system. The right half of Table 2 lists the recoveries of the LCs spiked with metal ions at 500 ng L^{-1}; they ranged from 87 to 116%, suggesting that the repeatability of the HR-ICP-MS-based method was adequate and could be used to evaluate the levels of metal ion impurities in LCs.

The use of HR-ICP-MS for determination of the metal ion impurities in LCs

The resistivity of an LC decreases continually upon progressing through the LCD cell manufacturing process, but there has previously been no direct means of correlating the levels of metal ion impurities to the decrease in resistivity. Fig. 2 displays the results of our HR-ICP-MS analyses of the metal ion impurities in the three tested LCs. The major common metal ions in these LCs were Na^+, Al^{3+}, Ca^{2+}, and K^+. The concentrations of other metal ions, including $Fe^{2+/3+}$, Ni^{2+}, Cu^{2+}, and Zn^{2+}, varied depending on the type of LC, presumably because of the different structures of the LCs and/or the different injection processes used during the manufacture of the LCD cells. The LC samples analyzed here were the same samples we had used to obtain Fig. 1. The levels of the metal ion impurities increased during the process of injecting the LCs into the LCD cells. This trend continued until the injection process had been completed.

Fig. 2(A) reveals that the siphoning process led to a significant increase in the content of major metal ions. In Fig. 2(B) and (C), we find that the increases in the levels of metal ion impurities in LCTN-M were lower than those in LCVA-T; because the ODF process was used for injection of both these LCs, we suspect that the higher viscosity of LCVA-T was probably responsible for inducing greater amounts of metal ion impurities.

Prior to injection in the LCD cell manufacturing process, the total metal ion concentration was greater than 200 ng L^{-1} in each of the LCs (Fig. 2); this value increased to greater than 1100 ng L^{-1} after injection. The total content of metal ion impurities in LCTN-C was the highest (>3200 ng L^{-1}), followed by LCVA-T

Table 2 Calibration curves of standard metal ions in IPA and recoveries of metal ions spiked into LCs using the optimized HR-ICP-MS method

	Calibration curve[a] $y = ax + b$			Recovery[b] (%)			
Element	a	b	R^2	LCTN-C	LCTN-M	LCVA-T	LOD[c] (ng L^{-1})
Li	84.5	163	1.000	102 ± 7	113 ± 8	109 ± 8	0.59
Na	234	972	0.999	92 ± 7	95 ± 3	102 ± 4	0.67
Mg	12.1	139	0.999	116 ± 3	102 ± 6	100 ± 5	1.8
Al	17.0	48.4	0.999	105 ± 5	101 ± 6	95 ± 4	3.0
Ca	0.99	111	1.000	93 ± 7	106 ± 6	99 ± 6	2.8
Cr	23.4	−98.1	1.000	100 ± 6	96 ± 5	92 ± 5	0.68
Fe	26.0	−457	0.999	93 ± 9	87 ± 7	88 ± 4	1.4
Co	24.9	9.57	1.000	96 ± 5	93 ± 5	89 ± 3	0.66
Ni	16.4	194	0.999	98 ± 10	88 ± 4	88 ± 4	6.5
Cu	16.6	−85.1	0.999	92 ± 8	98 ± 6	95 ± 3	3.6
Zn	4.82	134	1.000	94 ± 9	102 ± 8	113 ± 7	2.8
K	8.74	155	1.000	104 ± 5	93 ± 6	103 ± 6	0.84

[a] The concentration range for the calibration curve ranged from 10 to 1000 ng L^{-1}. [b] 500 ng L^{-1} of each metal ion was spiked into the LC sample. The recovery was expressed as mean ± standard deviation ($n = 3$). [c] LOD was calculated according to 3σ criterion of the 10% (v/v) LCVA-T sample containing 10 ng L^{-1} of each metal standard ($n = 7$).

(>1500 ng L^{-1}), with LCTN-M having the lowest (>1100 ng L^{-1}). Thus, the direct analysis of metal ion impurities in LC through HR-ICP-MS clearly revealed different levels of total metal ion impurities in the different types of LCs; it was also relatively easy to compare the variations of the metal ion concentrations among the various manufacturing processes. Furthermore, the amount of LC sample required for HR-ICP-MS analysis was only 100 μL, compared with approximately 1 mL needed for resistivity measurements. If we consider the cost of analyzing over 500 LC samples per day in an LCD factory, the HR-ICP-MS method would be favored because the price of each LC is greater than US$5 per mL. The time required for the preparation and analysis for each LC sample (3 min) is another advantage that the HR-ICP-MS system has over the resistivity approach (*ca.* 35 min).

Metal ion impurity *versus* resistivity of LC

An LC exhibiting higher resistivity (Fig. 1) suggests that it contains a lower metal ion concentration (Fig. 2). Fig. 3 reveals the relationships between the total content of metal ion impurities and the resistivity of the tested LCs at three points (before, during, after) of the LCD cell manufacturing process. We observe that the resistivities decreased as the total metal ion content in the LCs increased. Fig. 3 also indicates that LCs containing the same total metal ion concentration could display different resistivities, presumably due to the different physical and chemical characteristics of these LCs. Because the different LCs exhibited different trends in the relationships between their total metal ion concentrations and resistivities, we derived three equations to fit these sets of data. The values of R^2 of these linear equations for LCTN-C, LCTN-M, and LCVA-T were 0.968, 0.965, and 0.972, respectively. Assuming a resistivity of 1.00×10^{13} Ω cm as a quality control limit for those equations, we obtained a value of 1400 ng L^{-1} as the lowest total metal ion concentration among these three LCs. To minimize the number of defective LCD products, we propose that 1400 ng L^{-1} should be the highest allowable total metal ion concentration in LCs used in the LCD cell manufacturing process. This number might not be optimal, but it could be adjusted in the LCD factory based on further studies of different LCs and manufacturing processes. Because the presence of metal ion impurities in LCD panels is a major factor affecting the performance of LCDs, quality control through direct analysis of metal ions in LCs, rather than through resistivity measurements, should benefit the manufacturing process.

HR-ICP-MS-based analysis of metal ion impurities in a stained LCD panel

Although previous studies have suggested that metal ion impurities affect LCD performance,[1–6] we are unaware of any direct evidence for metal ion impurities being present in defective LCDs. Resistivity studies can be used to reveal the ion content in 1 mL of an LC sample (*e.g.*, in the bottle), but not for the LCs used in LCD cells because of their limited amounts. Fig. 4 displays images of the stained and normal regions of a defective 42″ LCD panel when the voltage was on (the stained region consisted of a stained area, with an irregular shape, and a normal area). Table 3 lists the concentrations of metal ions in these two regions (each area: 200 cm^2). We dissolved the LC of the target region of the LCD panel in an IPA solution and then injected the LC sample directly into the HR-ICP mass spectrometer. The concentration of metal ion impurities in the normal region was approximately 7800 ng L^{-1}; in the stained region it was approximately 150 000 ng L^{-1}. Therefore, our HR-ICP-MS-based analytical approach provides direct evidence for a metal ion effect in defective LCDs; that is, metal ion impurities in LCs can influence the performance of LCDs under a driven electric field.

Conclusions

Metal ion impurities in the cells of an LCD can deteriorate the LCD performance. Here, we measured the resistivities of LCs as an indicator of the level of ionic impurities at different stages of the LCD cell manufacturing process; we found that the resistivity decreased significantly upon progressing through the manufacturing process. We developed a simple and rapid method, using HR-ICP-MS, for directly analyzing metal ion impurities in

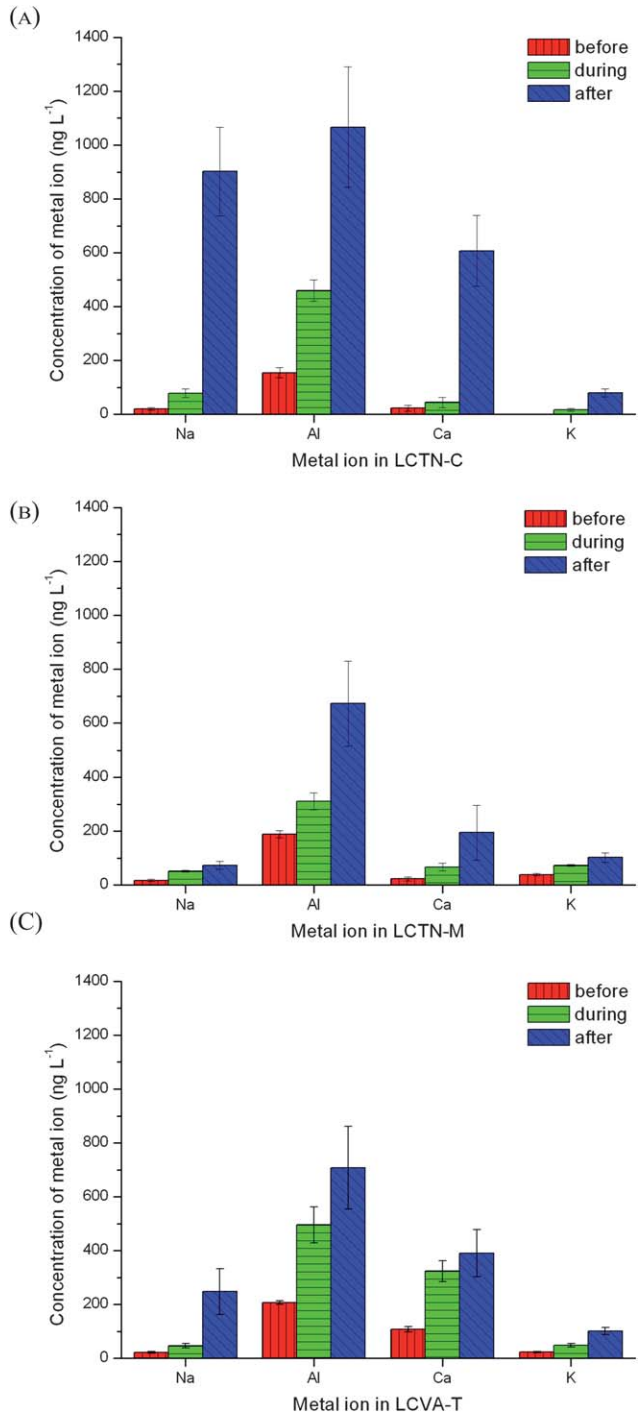

Fig. 2 Concentrations of major metal ion impurities in (A) LCTN-C, (B) LCTN-M, and (C) LCVA-T during the LCD cell manufacturing process. The LC samples were the same as those used to obtain Fig. 1; they were analyzed directly using HR-ICP-MS. Each concentration bar represents an average from seven LC samples.

LCs. In our approach, we used modified IPA solutions to dilute the LCs and then we injected them directly into an HR-ICP mass spectrometer, operated at different resolutions to eliminate spectral interference. Thus, we could directly determine the amounts of the metal ion impurities present in the LCs. This HR-ICP-MS method is more effective than conventional resistivity

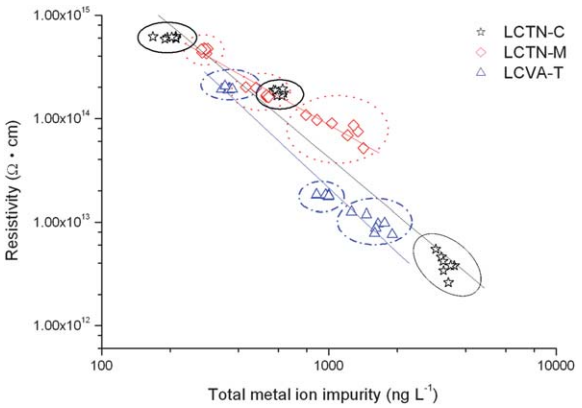

Fig. 3 Relationships between the total concentrations of metal ion impurities and the resistivities of the tested LCs.

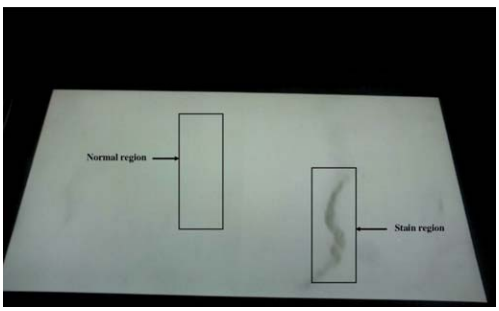

Fig. 4 Photograph of a defective LCD panel, highlighting the stained and normal regions.

assessments in terms of sample consumption and time; it can be used not only for routine and large-scale sample analyses but also to determine, in a factory setting, the changes occurring in the concentrations of metal ions in LCD cells throughout the manufacturing process. We suggest that 1400 ng L^{-1} should be set as the highest allowable total metal ion concentration in LCs used for the manufacturing of LCD cells. To test its applicability, we used this method to analyze a defective LCD cell; indeed, the level of metal ion impurities in the stained region was significantly greater than that in the normal region. The method we have developed in this study could be extended to analyses of metal ion

Table 3 Metal ion concentrations in normal and stained regions (each area: 200 cm^2) of a defective 42″ LCD panel

Element	Normal region (ng L^{-1})	Stained region (ng L^{-1})
Li	<30	<30
Na	2830	35 100
Mg	<90	13 600
Al	890	14 900
Ca	550	23 100
Cr	<30	<30
Fe	680	7300
Co	<30	<30
Ni	<330	4100
Cu	630	9900
Zn	1470	18 800
K	780	22 700
Total	7800	150 000

impurities in other organic materials, injected directly as organic solutions into HR-ICP-MS systems.

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