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RECYCLING OF LIQUID CRYSTALS FROM E-WASTE

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ABSTRACT

For several decades, Liquid Crystal Displays (LCDs) have been widely used in televisions, laptops, mobile phones, and other devices. Nowadays, liquid crystals (LCs) represent an important economic value of the recycling system of LCDs. The reuse of these organic molecules could become a profitable basis since it permits to preserve the value of these materials. In this context, the general objective of this work focuses on the recovery of LCs as well as on other valuable materials present in end-of-life LCDs. An orderly, manual LCD dismantling line is put into operation for differentiated recycling of electronic boards, cold cathode lamps that may contain mercury, polymers, metals, and other valuable materials. There is also an extraction line where LCD panels are opened and exposed to an ultrasonically activated organic solvent bath to recover LCs. The resulting solution contains LCs, solvent, organic and inorganic impurities. The LCs mixtures were purified and then characterized mainly by spectroscopic, chromatographic, and thermal techniques. A study of the influence of adding diamond nanoparticles at 0.05, 0.1 and 0.2 wt% to recycled LCs was also performed using dielectric spectroscopy. Dielectric properties of LCs were measured at room temperature, using an impedance analyzer in the frequency range from 0.1 to 10⁶ Hz.


1. INTRODUCTION

Electrical and electronic equipment (EEE) has become an essential part of our daily lives. Much of the world's population enjoys a higher standard of living, thanks to their availability, widespread use, and easy access. The passion to keep up with the latest technology of millions of consumers, prematurely throwing away their cell phones, tablets, computers, televisions, etc., as well as the programmed obsolescence of most devices, are two of the main causes of the increasing generation of Waste Electrical and Electronic Equipment (WEEE) in the world (Centre Européen de la Consommation, 2013). According to the United Nations (UN), in 2019, the world generated over 50 million metric tons (Mt) of WEEE and only 20% of these devices were properly recycled. The remaining 80% are not accounted for and most end up buried (Platform for accelerating the circular economy (PACE), 2019).

In Europe, despite having the most advanced waste legislation in the world, only 42.5% of WEEE was properly collected and recycled in 2020 from a total of 12 million tons (Forti et al., 2020; United Nations News, 2019). If no measures are taken now, the amount of waste will more

than double by 2050, to about 120 million tons per year. Because of its often hazardous materials, WEEE can cause environmental and health damage if they are not properly managed. It is important to understand that all WEEE are not biodegradable and the lack of proper recycling is also aggravated by the fact that devices are becoming more and more complex. Effective recovery of their valuable materials is an expensive process that requires sophisticated technologies. In this regard, the main objective of our research focuses on the valorization of the different materials present in end-of-life LCDs.

An LCD panel is mainly composed of ~85 wt% glass and ~15 wt% organic materials (LCs, polarizing filters,...) and metals (indium, tin, aluminum,...) (Goodship et al., 2019). Since 2010, the economic importance and supply of indium has forced the European authorities to classify this metal as one of 30 critical raw materials (European Commission, 2020). This makes indium one of the most attractive material for LCD-recycling (D'Adamo et al., 2019). In Europe and on a worldwide scale, a large number of end-of-life LCD recycling projects have been developed recently, with the main goal of indium recovery. Most of the meth-

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odologies used are based on manual and semi-automatic dismantling, with size reduction, thermal pre-treatments allowing the removal of organic materials (plastics and liquid crystals), and hydro-metallurgical processes (EIT Raw-Materials, 2020; Fontana et al., 2020; ReVolv, 2021; Song et al., 2020).

Industrial LC recovery was made possible in a Taiwanese pilot plant. This process, developed by Industrial Technology Research Institute (ITRI) researchers, allows the recovery of LC mixtures with high purity (< 1 ppb) and ready for new use (in LCD or smart windows) (ITRI, 2018). Recently, research groups are using GC-MS technique to identify LC molecules released into the atmosphere during LCD dismantling. This gas particles partitioning is considering as a class of emerging chemical pollutants (Cheng et al., 2022; Shen et al., 2022; Su et al., 2022; Zhu et al., 2021).

The remarkable point of our approach, unique in Europe, is the revalorization of LCs. The reuse of these organic molecules could make these materials profitable. LCs are organic molecules that exhibit intermediate properties between the solid state and the isotropic liquid state (Singh, 2002). Depending on the order of orientation, position and chirality of the LCs molecules, three main mesophases are found: nematic, smectic and cholesteric (Collings & Hird, 2017; Yang & Wu, 2014). In this study, the recovered LCs mixtures exhibit a nematic phase at room temperature. This phase can be characterized by an uniaxial orientation of the long axes of the LC molecules. This singular direction is represented by a vector «n», the director of the nematic phase (Oswald & Pieranski, 2005).

This study will be based on the research of innovative ways of purification allowing the reconditioning of these LCs mixtures. One of the challenges is to detect and neutralize impurities in order to ensure the quality of the mixture and its conformity for a possible recycling. Some unwanted effects are introduced by inorganic impurities present in LC blends; for example, they tend to increase the electrical conductivity, which can lead to alter their optical and electro-optical properties (Garbovskiy, 2016). One of the most promising and widely studied ways to capture ionic impurities is the addition of nanomaterials as ion adsorbing materials to LCs (Osipov & Gorkunov, 2016). In this work, diamond nanoparticules (DNP) were chosen to study their impact on inorganic impurities present in the recycled LCs using dielectric spectroscopy.

2. MATERIALS AND METHODS

2.1 Materials

End-of-life LCDs were provided by the French recycling company ENVIE²E. In this company, a manual and orderly dismantling line of LCD screens is installed in order to separate their different components: batteries, electronic boards, capacitors, scrap metal, plastic foils, speakers, lamps, LCD panels, ABS and PMMA plastics and plasma glass. Among all these components, we will focus specifically on LCD panels from which the LCs mixtures will be extracted (Figure 1a-c). End-of-life LCD panels are opened and exposed to a bath of an ultrasonic activated organic solvent in order to extract LCs mixtures. The details of the

extraction process are reported in a patent developed by Maschke et al. (Maschke et al., 2015).

In this report, three different LC mixtures were extracted, corresponding each to a working period at ENVIE²E of 4 months during one year, i.e.: NP-1 (1-4 months), NP-2 (5-8 months), and NP-3 (8-12 months).

The extraction of the LC mixtures was carried out from large numbers of end-of-life LCD displays (65700 panels) of heterogeneous nature: diversified screens from TVs, computers and tablets of completely different types, sizes, brands and years of production. Each native LC mixture used in one single LCD screen is composed of about 20 or more LC molecules (mainly nematic ones), together with a certain number of additives. Each manufacturer uses a specific native LC mixture according to the type of screen and the technology to be developed. Since this information is not available (trade secret), the LC composition of the corresponding native LC mixture is unknown. This means that it is not appropriate to study specific native LC mixtures in order to compare their dielectric properties with those from the collected end-of-life LC mixtures.

The extracted solutions contain LCs molecules, organic solvent, as well as organic and inorganic impurities, including ions. In terms of optical aspect, they present a black coloration that is not typical of LCs (Figure 1d). It is evident that during the industrial recovery procedure performed to extract the LCs, several sources of contamination appeared. The purification of these mixtures is therefore a necessary step for a possible reutilization of the LCs (Figure 1e). This purification leads to a nematic mesophase as evidenced by the Schlieren texture obtained by Polarizing Optical Microscopy (POM) (Figure 1f).

2.2 Purification of LCs Mixtures

First, an evaporation of the organic solvent is performed using a pressure-controlled rotavapor (BÜCHI R-100). The extracted solution was placed in a distillation flask which is rotated in a heating bath (BÜCHI B-100) by increasing the temperature up to 65°C. A vacuum pump (VARIO PC 300) was placed on the entire circuit to create the vacuum ($P = 27$ kPa) and lower the boiling point of the organic solvent. At the end of the process, the content of distillation flask (LC molecules + impurities) is recovered. The distilled organic solvent is collected and will be reused in the industrial dismantling LCD-line at ENVIE²E.

After the distillation step, the presence of solid particles is observed in the solution. Using a funnel and filter paper (Whatman®, qualitative filter paper, Grade 287 1/2, diameter 185 mm), the solid impurities were filtered out. To achieve higher purity of LCs mixtures, column chromatography technique is used to remove organic impurities. Column chromatography is a very useful technique that allows separation and purification of a compound in a mixture by adsorption. Two phases are involved in this method: a stationary phase and a mobile phase. The LC mixture (5 g) was introduced into the upper part of a chromatographic column (length 45 cm, interior diameter 4 cm) loaded with an adsorbent (stationary phase). Then each component crosses the column at different retention times depending on its affinity with the adsorbent and the solvent (mobile

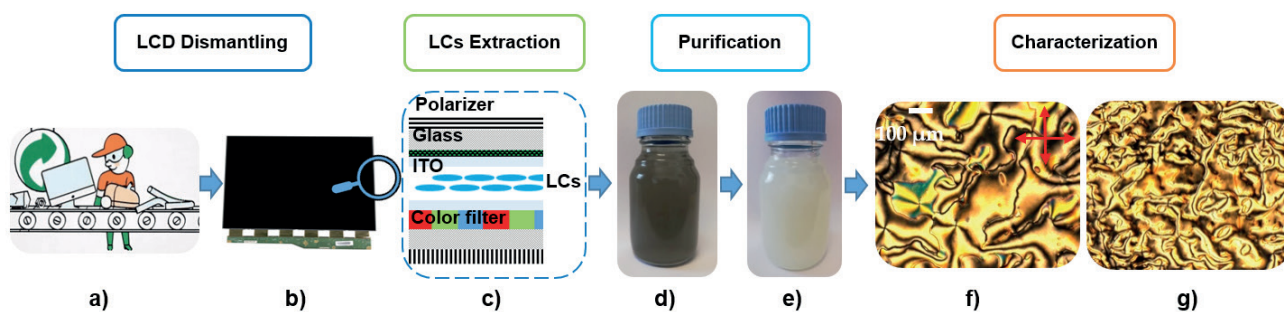


FIGURE 1: a) LCD dismantling line, b) LCD display, c) LCD display composition, d) Non-purified and e) purified (before doping) LCs mixtures, and texture of f) purified and g) doped (0.2 wt% DNP) LCs mixtures observed under Polarizing Optical Microscope (POM) exhibiting nematic Schlieren texture at room temperature. Adapted from (Barrera et al., 2021).

phase). Silica 60 M (SiO_2 , 0.04-0.063 mm, Macherey-Nagel) and petroleum ether (40-60°C, VWR) were used as stationary and mobile phases, respectively. The evaporation of the petroleum ether is then carried out and the purified LCs mixtures are obtained.

An additional purification step is performed to capture ions (inorganic impurities) by addition of nanoparticles to LCs. Diamond nanoparticles (DNP) were added to the recycled LCs at 3 different concentrations: 0.05, 0.1 and 0.2 wt%. Mixtures were placed in an ultrasonic bath to ensure good dispersion. After the purification steps, a spectroscopical characterization is performed to determine the dielectric properties of the recycled LCs mixtures. No formation of a colloidal system and no phase separation effects were observed on a microscopic scale within the time scale applied for the dielectric experiments. In order to validate these findings, Figure 1g presents the morphology of a LC sample doped with 0.2 wt%, showing no significant difference compared to Figure 1f.

All dielectric measurements presented in this report were conducted at room temperature where the LC mixtures were in the nematic state. Figure 2 presents thermo-

grams of non-purified, purified, and doped LC mixtures, exhibiting a single nematic-isotropic transition temperature (T_{NI}) around 70°C. An increase of T_{NI} of four degrees was observed when comparing non-purified with purified LC samples, as a consequence of the purification process. On the other hand, T_{NI} decreases of about two degrees, when 0.2 wt% DNPs were added to the purified LC mixture.

2.3 Dielectric measurements

Dielectric measurements were realized on non-purified, purified and DNP-doped LCs mixtures. The samples were inserted into commercial cells by capillary action. These cells have a thickness of 20 μm and possess either a homogeneous (HG cell) or a homeotropic (HT cell) alignment. The relative real and imaginary (ϵ' and ϵ'') components of the complex relative dielectric permittivity (ϵ^*) were measured using a ModuLab-MTS impedance analyzer (Solartron Analytical, Ametek) in the frequency range from 0.1 Hz to 10^6 Hz.

The interest of the dielectric study was mainly focused on the determination of the dielectric anisotropy ($\Delta\epsilon$) as well as the electrical conductivity (σ') of LCs mixtures.

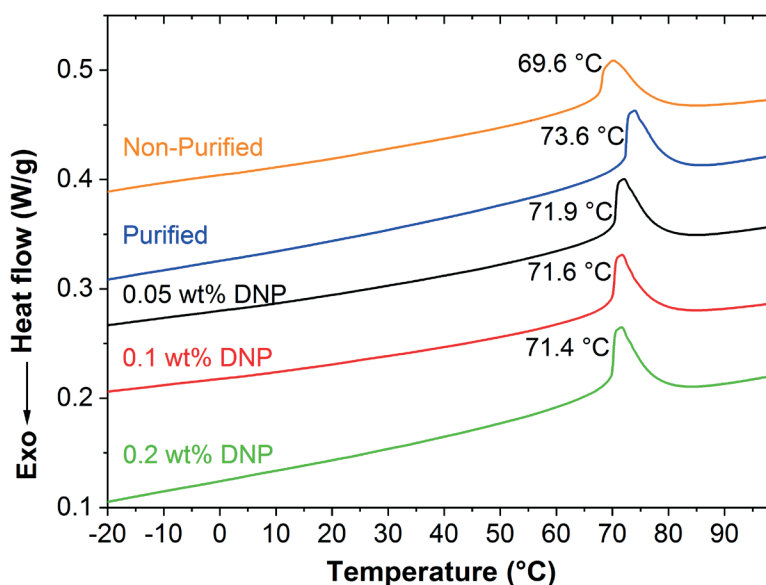


FIGURE 2: Thermograms obtained during the heating cycle (10°C/min) of non-purified, purified and DNP-doped LC mixtures at three concentrations (0.05; 0.1 and 0.2 wt%) in the temperature range between -20 and 100°C.

These are the most appropriate parameters to consider when determining the type of LCD display device in which the LCs will be used.

Dielectric anisotropy characterizes the ability of LCs to orient themselves in response to an external electric field. $\Delta\epsilon$ is obtained from the difference between the dielectric permittivity when the electric field is parallel to the direction vector "n" ($\epsilon'_{//}$, obtained with HT cell) and when the field is perpendicular to "n" (ϵ'_{\perp} , obtained with HG cell).

The complex electrical conductivity is an alternative and complementary representation to the dielectric properties, it allows to better understand the phenomena of charge transport (ions, electrons,...). σ^* is related to the complex relative dielectric permittivity ($\sigma^* = \epsilon_0 \epsilon^*$), and it also has a real part (σ') and an imaginary part (σ'').

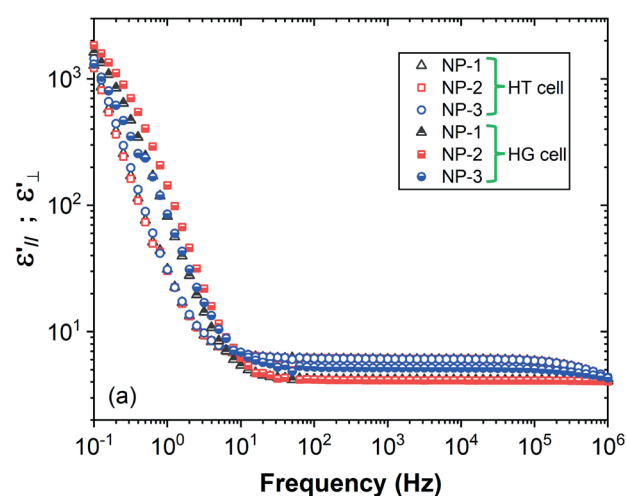
3. RESULTS

3.1 Dielectric properties of non-purified LCs

Figure 3 presents: a) the real part of the relative complex permittivity and b) the dielectric anisotropy of three non-purified LCs mixtures (NP-1, NP-2, and NP-3) for homogeneous and homeotropic alignments. In Figure 3a two behaviors are distinguished depending on the frequency range:

- 1) **0.1 - ~10 Hz:** As the frequency decreases, a significant increase of the values of $\epsilon'_{//}$ and ϵ'_{\perp} is noticed, reaching values above 10^3 . The presence of ionic impurities in the non-purified samples affects electrode polarization and electrical conductivity.
- 2) **10 - 10^6 Hz:** The permittivity for each LCs mixture is independent on the frequency. In this range, the ionic impurities are not able to follow the periodic inversion of the electric field.

The Figure 3b shows an extended view of $\Delta\epsilon$ in the frequency range between 10^2 and 10^5 Hz to highlight the different values of dielectric anisotropy. This frequency range corresponds to the plateau of the real permittivity. The obtained values at 10^3 Hz are: 1.87, 1.99, and 0.90 for the NP-1, NP-2, and NP-3 materials, respectively.



A fundamental aspect for the reuse of a recycled product is the reproducibility of its properties. Indeed, both chemical and physical parameters of the different mixtures must have similar values in order to be eligible for a future reuse. Here, a difference in the dielectric anisotropy results has been observed. The presence of impurities in these mixtures could explain the non-reproducibility of the values. Consequently, the purification of these mixtures is an essential step for the reuse of the recycled LCs. For this purpose, several distillations and chromatographic techniques were employed to purify the recycled LCs mixtures.

3.2 Dielectric anisotropy of purified and doped LCs

Figure 4a shows the relative permittivities of three purified LCs mixtures (P-1, P-2, and P-3) as a function of frequency. After purification, all LCs mixtures present comparable dielectric anisotropy values at 1 kHz: 3.16, 3.51, and 3.36 for P-1, P-2 and P-3, respectively. These values were found to be in the range of corresponding data for commercial LC-mixtures. Dielectric anisotropy data are given in Merck data sheets, gathering physico-chemical properties of commercial LC mixtures applied for a variety of electro-optical applications, such as TN and STN LCDs (Kelly & O'Neill, 2001; Merck, 1988; 1997 and 2022). As already mentioned above, a large number of LC molecules in an annual deposit of end-of-life LCD screens were considered in this report. Since each LC molecule present a distinct value of the dielectric anisotropy ($\Delta\epsilon$), which could be either positive ($\Delta\epsilon > 0$) or negative ($\Delta\epsilon < 0$), the overall average value was determined around $\Delta\epsilon = 3$. It is worth mentioning that these values are higher than those found for non-purified mixtures due to the significant reduction of the amount of impurities. Consequently, it was decided to present the results of doped sample for only one of these LCs mixtures.

Figure 4b illustrates the dielectric anisotropy of a representative mixture of purified (undoped) and doped LCs for three concentrations of DNP: 0.05 wt% DNP, 0.1 wt% DNP and 0.2 wt% DNP. A decrease of ~30% of the value of the dielectric anisotropy of the purified sample was observed by adding DNP. A weak dependence between

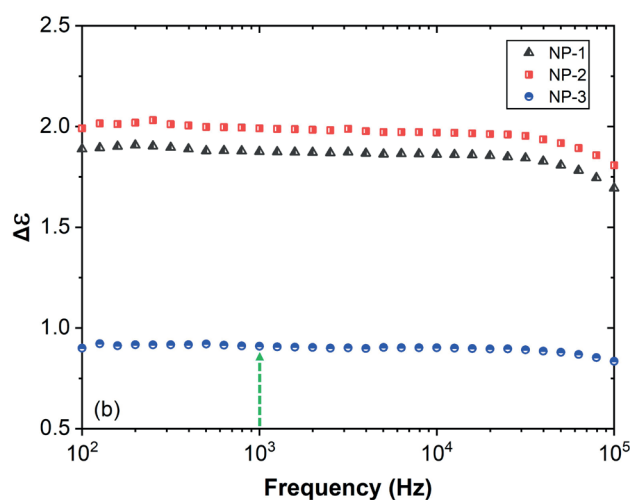


FIGURE 3: a) Relative real permittivity and b) dielectric anisotropy of non-purified LCs mixtures as function of frequency in homogeneous and homeotropic alignments. NP-1, 2 and 3 stands for three non-purified LCs mixtures.

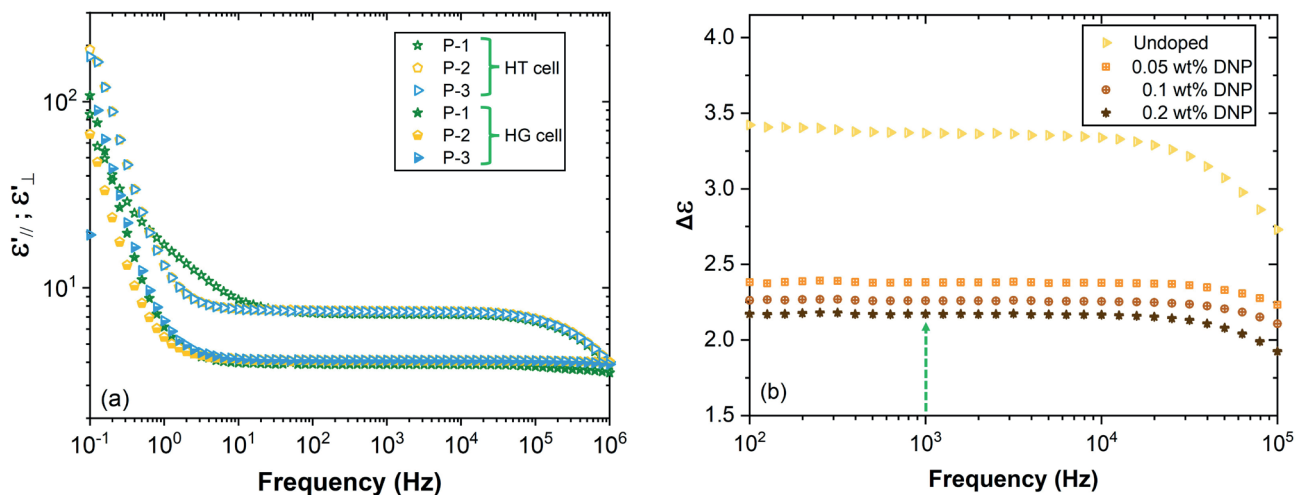


FIGURE 4: a) Relative permittivity of three purified LCs mixtures as function of frequency in homogeneous and homeotropic alignments. P-1, 2 and 3 represents the 3 purified LCs mixtures. b) Dielectric anisotropy of purified and doped LCs mixtures. 0.05 wt%, 0.1 wt% and 0.2 wt% correspond to purified LCs mixtures doped with diamond nanoparticles.

the amount of DNP present in the LCs mixtures and the decrease in dielectric anisotropy is also noticed. Thus, at a frequency of 1 kHz, $\Delta\epsilon$ shows the following values: 2.38, 2.25 and 2.17 for DNP concentrations of 0.05, 0.1 and 0.2 wt%, respectively.

3.3 Real conductivity of non-purified, undoped and doped LCs

Conductivity spectra are studied by Almond-West formalism which is derived from Jonscher's universal power law (Jonscher, 1977). This model is widely used to analyze the frequency dependence of the real part of the complex conductivity. The equation can be expressed as follows:

$$\sigma' = \sigma_{DC} \left(1 + \left(\frac{f}{f_c} \right)^n \right) \quad (1)$$

where σ_{DC} , f_c and n represent the DC conductivity, the characteristic frequency and the degree of interaction between the mobile ions and their environment, respectively.

The $\sigma'(f)$ spectra of non-purified, purified and doped (0.1 wt% DNP) LCs mixtures in homogeneous and homeotropic alignment are reported in Figure 5. In this figure, two behaviors can be appreciated:

- 1) $\sim 1 - 100$ Hz: A plateau almost independent of frequency is found, from which the DC conductivity values can be determined applying the Almond-West formalism.
- 2) > 1 kHz: A very significant increase in σ' values is detected with increasing frequency. Relaxation effects, arising from mobile charge carriers, might be responsible for this dependence.

The solid fitted curves in Figure 5 illustrate the good correlation between experimental data and used formalism. As expected, the non-purified mixture has the highest conductivity compared to the other samples. It should be noted that the conductivity

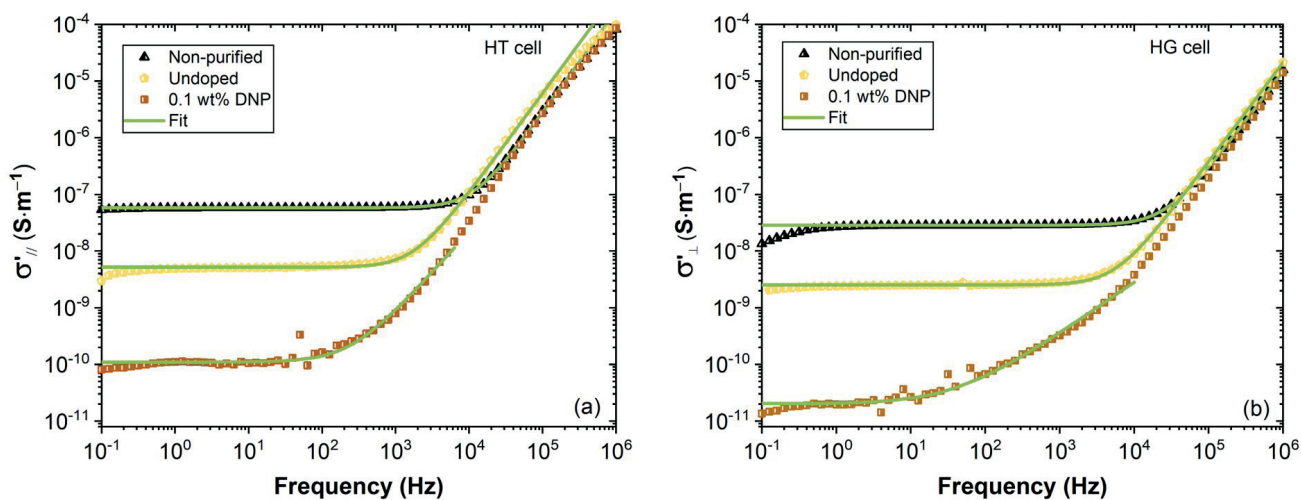


FIGURE 5: Real part of the complex conductivity in: a) homeotropic and b) homogeneous alignment as a function of frequency for non-purified, undoped and doped (0.1 wt% DNP) LCs mixtures. Solid lines represent the curves obtained using Almond-West formalism.

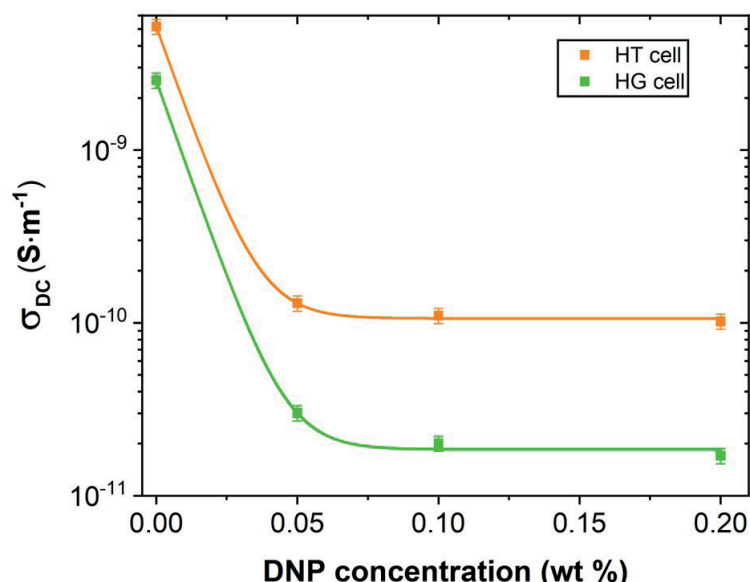


FIGURE 6: DC conductivity values as a function of DNP concentration. The curves represented by solid lines were obtained applying a decreasing exponential fit.

values of the doped samples are low in the order of 10^{-10} and 10^{-11} $S \cdot m^{-1}$ and close to the sensitivity of the impedance analyzer.

3.3.1 Conductivity DC of undoped and doped LCs

In Figure 6, DC conductivity values as a function of DNP concentration are reported.

Figure 6 shows that 0.05 wt% of DNP is enough to reduce significantly the DC conductivity values of recycled LCs mixtures in both alignments. Impurities (anions and cations) are adsorbed on the surface of the spherical DNP. All σ_{DC} values are found within the range of commercial nematic LCs mixtures (de la Fuente & Dunmur, 2014; Garbovskiy & Glushchenko, 2015; Viciosa et al., 2002).

4. CONCLUSIONS

In this study, the results concerning the dielectric characterization of non-purified, purified and DNP-doped LCs mixtures have been presented. An increase in dielectric anisotropy values at 1 kHz was found for the purified LCs mixtures compared to the non-purified ones. For the non-purified samples, the orientation effects were disturbed by the presence of impurities. In fact, the dispersivity values of 1.47, 1.99 and 0.90 for the three non-purified samples was reduced to an average value of ~ 3.5 after purification. Since the LCs mixtures have the same characteristics (optical, dielectric, etc.) after purification, only one mixture was used to achieve the DNP doping. Regarding the electrical conductivity of the purified LCs, it decreases significantly with the addition of DNP for both alignments. All the LC mixtures studied in this work have conductivity values within the range of values of conventional LCs.

Three batches of end-of-life LCD screens were analyzed separately. These batches correspond to consecutive work sequences of LC recovery at the industrial line

level, for a total period of 12 months; i.e. batch 1 corresponds to 25739 screens (4 months), batch 2 refers to 22580 screens (4 months), and batch 3 corresponds to 17381 screens (4 months). It should be noted that during these periods, a strong heterogeneity of the deposit was observed. The individual analysis of these three batches led to fairly similar results in terms of dielectric response. Multiple studies were performed to assess the reproducibility of these measurements. These results could be explained by the large number of LC mixtures collected and mixed together, leading to average dielectric properties. If one considers only small numbers of EOL LCD screens, rather strong deviations of the dielectric data might be expected.

Additional research and development work will be conducted, especially investigations of specific LC properties, such as evaluation of structures, topological defects, morphologies, elastic constants, refractive indices, and viscosity. One of the first steps of a potential economic development consists to prove if it is possible, on the basis of extended laboratory research, to fulfill a desired product specification in order to valorize the recycled LC blends.

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