

Colorimetry characterization of molecular reorientation transition in thin nematic cells

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ABSTRACT

The characterization of equilibria and their transition is fundamental in dynamic systems. Experimentally, the characterization of transitions is complex due to time scales separation, the effect of thermal fluctuations, and inherent experimental imperfections. Liquid crystal devices are derived from the manipulation of the molecular reorientation and transition between them by employing external electrical and magnetic fields. Here, we investigate and determine the Fréedericksz transition using hue measurements of the transmitted light in thin nematic liquid crystal cells. Based on birefringent retardation experienced by transmitted light due to molecular reorientation, the color adjustment of the nematic liquid crystal cells under white light illumination is characterized. By monitoring the hue of the transmitted light, the bifurcation diagram is determined. As a function of the voltage frequency, the critical transition voltage is characterized. The critical voltage increases with the applied frequency.

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Materials formed by molecules with rod-like structure can exhibit states of matter that are locally oriented, liquid crystal state that can be reoriented by external electric and magnetic fields. Molecular reorientation changes the optical and elastic properties of the material. The understanding of molecular reorientation dynamics and transitions in liquid crystal cells is relevant for optoelectronic, electronic, and photonic applications and devices. Thin films of liquid crystals exhibit color adjustment as a consequence of molecular reorientation. By characterizing the color adjustment of liquid crystal cells illuminated with white light, we can characterize the molecular reorientation transition and determination of the critical transition voltage as a function of the frequency of the applied voltage. Then, the color adjustment allows characterizing various dynamic properties.

I. INTRODUCTION

Liquid crystals (LC) are a state of matter where the molecules have a preferential orientation and can or cannot have a positional organization.¹⁻⁴ This state of matter shares properties of solids and liquids. For example, fluidity and birefringence are characteristic properties of liquid crystals. Nematic liquid crystals are composed of rod-like organic molecules.¹⁻⁴ As a result of intermolecular interaction, for specific temperature ranges, these molecules are arranged to have a similar molecular orientation. This results in a strong anisotropy of all their physical properties, especially elastic and optical characteristics. The configuration of the lowest energy is reached when all rod-like molecules are aligned along one averaged direction denoted by a vector \mathbf{n} , which is nominated as the director.¹⁻⁴ The molecules are oriented in a given direction, but which does not have

a privileged sense, that is, any description must include the reflection symmetry $\mathbf{n} \leftrightarrow -\mathbf{n}$. When a liquid crystal is introduced inside a cell—it is sandwiched between two confining layers—the molecules are oriented according to how layers induce their respective anchoring conditions.^{3,5} Using surface treatment, it is possible to design how the molecules are anchored in the walls. One of the most used configurations is planar cells,⁵ where the molecules are anchored tangentially to the walls and the process of *rubbing* over the treated surfaces determines the orientation of the nematic director.

By using transparent electrodes adhered to the glass layers, an electric field can be applied orthogonally to the cell. For large enough applied voltage, in the case of positive (negative) dielectric anisotropic susceptibility, the molecules try to reorient parallel (orthogonal) to the applied electric field. For small voltages, the molecules are not reoriented due to the interaction induced by the surface anchored molecules. Only when the voltage exceeds a critical value V_{FT} , the molecular reorientation begins. This phenomenon is known as the *Fréedericksz transition* (FT).⁶ Indeed, this transition corresponds to an exchange of balance between the electrical and elastic force. Theoretically, this transition is characterized by the fact that the average or maximum angle of molecular reorientation exhibits a supercritical transition with mirror symmetry,¹⁻⁴ which corresponds to a pitchfork bifurcation in the instabilities theory of dynamic systems.⁷ Direct measurements of the average or maximum angle of molecular reorientation are not accessible experimentally. However, experimental observations of this transition based on interferometry,⁸ dielectric,⁹ capacitive,^{10,11} transmittance,¹² and refractive effect¹³ method are consistent with bifurcation diagrams without mirror symmetry. Indeed, all these methods depend on the angle module reorientation. In addition, these bifurcation diagrams are imperfect as a consequence of inherent fluctuations (temperature) and experimental imperfection in boundary conditions (pretilt). Harnessing the light intensity is crucial to all optoelectronic, electronic, and photonic devices based on liquid crystals and using the birefringence and molecular reorientation.¹⁴ The determination of the Fréedericksz transition (FT) voltage is fundamental for all technological applications. In particular, the control of molecular orientation is relevant for displays,¹⁵ tunable filters,¹⁶ polarization sensors,¹⁷ optical switches,¹⁸ lenses,¹⁹ spatial light modulators,²⁰ optical computing, and holographic optical tweezers, to mention a few. Recently, interest has emerged in molecular reorientation when working at high-frequency, in specific switching beam steering,²¹ terahertz, and sub-terahertz on-off switch of photonic crystals,^{22,23} and switchable photonic nanojet.²⁴

Optical methods are more accessible and straightforward than dielectric techniques to determine the Fréedericksz transition, which, in addition, are noisier and more challenging to establish the critical voltage.²⁵ As a consequence of the constant improvement of camera technologies, imaging has allowed the qualitative and quantitative characterization of different phenomena. The optical characterization of the Fréedericksz transition usually uses the intensity of the light transmitted through a liquid crystal cell subjected to a driving alternating voltage V_0 and inserted in between two crossed polarizers (see, e.g., Ref. 26). The light transmission of cells between two crossed polarizers depends on the thickness of the cell, the light wavelength and the refractive index, which, in turn, depends on temperature. For thin cells, the effect of color-changing

during molecular reorientation has a slope sufficiently small to distinguish of different wavelengths. Hence, one can associate a color with the applied voltage. The physical origin of the above effect is due to the fact that the molecular reorientation induced by the electric field changes the birefringent retardation experienced by transmitted light.²⁷ Colorimetry-based retardation measurement has been used to characterize elastic deformations²⁸ and gas density or temperature gradients.²⁹

A natural representation of color is based on hue, saturation, and lightness (HSL system). The hue correlates the light wavelength with a value in degree. For small voltage, the transmission light is unaltered because the electric force does not overcome the elastic one. Hence, when the voltage exceeds a critical value, $V_0 > V_{FT}$, the intensity of the transmitted light begins to change. The critical voltage V_{FT} is determined by using elastic and electric energy analysis.^{1,2} This critical voltage depends on the elastic and dielectric constants at a given temperature and assuming that the electric field is static. Most liquid crystal optoelectronic devices operate at high-frequencies (kHz) compared to the reorientation dynamics of the liquid crystal, then it is usually considered that the effective electric field is static. In general, the dielectric anisotropy can be modified with the voltage frequency. For example, E7 liquid crystal exhibits a constant regime until 10 kHz.^{30,31} Likewise, the effects of inherent fluctuations (thermal and electrical) are ignored in the reorientation transition. However, it is well-known that fluctuations anticipate and deform the bifurcation diagrams.^{32,33} This phenomenon is called as a *precursor*,³⁴ which hinders the determination of the bifurcation point.

This paper aims to investigate the molecular reorientation transition in nematic liquid crystal thin cells by characterizing their color adjustment under white light illumination. By monitoring the hue of the transmitted light, we can determine the bifurcation diagram, transition voltage, and intensity level of the random orientational fluctuations. As a function of the frequency of the applied voltage, the critical transition voltage and the level of fluctuations are characterized. We observe that the critical voltage changes with the frequency.

II. EXPERIMENTAL SETUP

To characterize the molecular reorientation, we consider two setups as illustrated in Fig. 1. A liquid crystal E7 is inserted in the cells, which is in the nematic phase with positive anisotropic dielectric constant ϵ_a at room temperature. The liquid crystal cells are composed of two glass layers separated by the liquid crystal film of thickness d . The inner surfaces of the commercial cells have a surface treatment so that the molecules are anchored parallel to the glass substrates in a given direction, *planar anchoring*. The liquid crystal cells are classified into two types. Twisted-nematic (TN) and planar-nematic (PN) cells, in which the anchoring of the molecules is orthogonal and parallel in the opposite glass layers, respectively (see Fig. 1). Besides, transparent electrodes have been deposited over the inner surfaces of the glass substrates in order to apply an external alternating voltage across the liquid crystal film. Planar-nematic and twisted-nematic cells have thickness of $d = 3.2 \pm 0.3 \mu\text{m}$ and $d = 4.0 \pm 0.4 \mu\text{m}$, electrode area of 25 mm^2 and 100 mm^2 of ITO with a thickness of $0.023 \mu\text{m}$ and $0.08 \mu\text{m}$, a resistance of 100Ω and

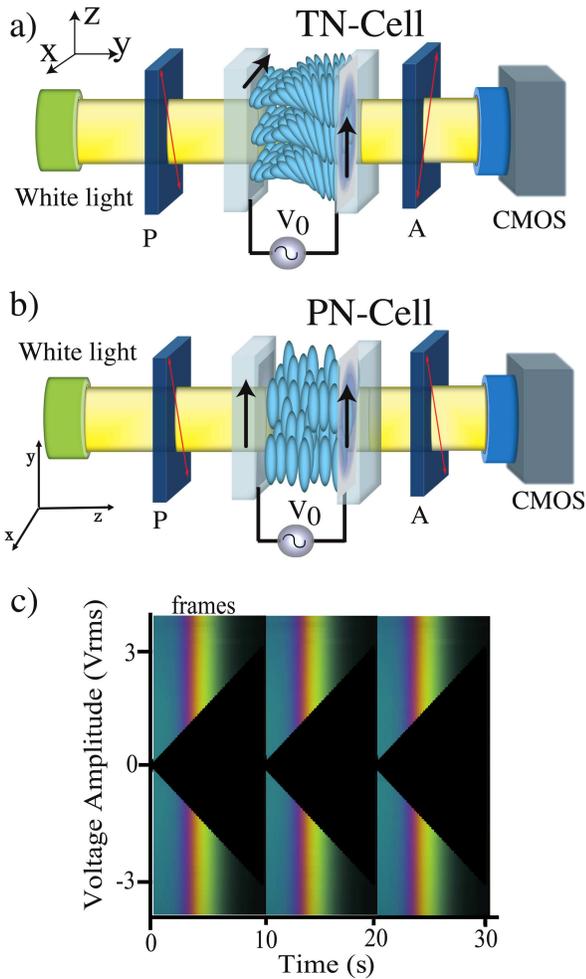


FIG. 1. Schematic representation of experimental setups. The cells are inserted in between two polarizers, the symbols P and A account for the first and second polarizer, respectively. An alternating current voltage of strength V_0 and ω frequency is applied to the cells, thanks to transparent electrodes (ITO). The samples are illuminated with white light, and a CMOS camera monitors transmitted the light. Black arrows account for the respective anchoring of the molecules in the cell walls. (a) The twisted-nematic configuration of the liquid crystal cells (TN cells). The molecules on the surfaces of the glass layers are orthogonally anchored in the respective layers. The polarizer and analyzer are orthogonally oriented, cross polarizers. (b) The parallel-nematic configuration of the liquid crystal cells (PN-cell). The molecules on the surfaces of the glass layers are parallelly anchored onto the layers. The polarizer and analyzer are oriented parallel one to another. (c) The up-ramp waveform applied as a function of the time to the TN cell; the background shows the frames obtained from the CMOS camera.

25 Ω , and the same polyimide thickness of 0.06 μm , respectively. Both cells have a homogeneous alignment layer with a 1° to 3° pretilt angle from the plane of the substrate.

The voltage is controlled by a function generator and registered in an oscilloscope. The liquid crystal samples are in between two

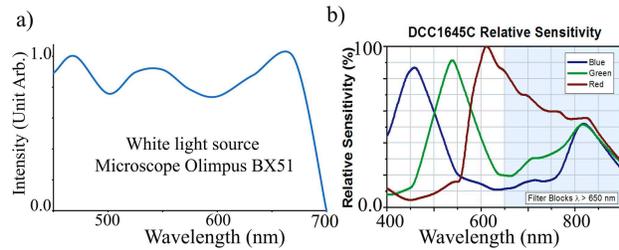


FIG. 2. (a) The spectrum of the white light source (Halogen lamp) of microscope Olympus BX51. (b) Relative spectrum sensitivity of the CMOS camera (DCC 1645C, Thorlabs).

polarizers (see Fig. 1). The first allows us to orient the light in a given direction, which we call by the symbol P . The second polarizer that receives the light transmitted by the liquid crystal sample is called an analyzer and represented by the symbol A . By changing the orientation of the polarizer and analyzer, the birefringence property of the liquid crystal is analyzed. The angle of the polarizer and the analyzer with respect to the vertical are $\pm\pi/4$ for the TN cell and $\pi/4$ for the PN-cell, corresponding to orthogonal and parallel polarizer-analyzer orientations, respectively [cf. Figs. 1(a) and 1(b)]. Namely, the angles of analysis between polarizer and analyzer for the TN cell are $\pi/2$ or 0, respectively. The system is illuminated by a white light (Halogen lamp). The area of the liquid crystal sample that we have analyzed is $X \text{ mm}^2$. Figure 2 shows the spectrum of the white light. A CMOS camera records the transmitted light. An optical power and energy meter console (PM400, Thorlabs) with a detector of Silicon Power Head (S121C), with a range between 400 and 1100 nm, and 500 mW was used for comparison. Our experiments were carried out in a microscope Olympus BX51 with an objective of magnification 10. Snapshots and videos (120–140 fps) were recollected in a CMOS camera model Thorlabs-DCC1645C. Figure 2 shows the relative spectrum sensitivity of the CMOS camera. The voltage was acquired in a waveform modulator (Agilent 33500B series) with the up-ramp waveform (fast frequency: 1 kHz–1400 kHz, slow frequency: 100 mHz) and square waveform (fast frequency: 0.1 Hz and 1 kHz, slow frequency: 100 mHz–800 mHz). An example of images recorded by driving the TN cell is shown in Fig. 1(c). The observed change of colors reflects the changing of the molecular orientation during the Fréedericksz transition. First, the uniform green corresponds to the unaltered orientation, with the voltage below the FT threshold, then, the color starts to change according to the change of the molecular reorientation. Finally, the transmitted color becomes practically black, corresponding to saturation of the reorientation effect, when most of the molecules (except small boundary layers close to the substrates) are aligned orthogonally to the direction of the applied field and the cell becomes practically equivalent to a homeotropic cell.

III. COLOR CHARACTERIZATION OF THIN NEMATIC CELLS

As an example of color characterization, Fig. 3(a) shows the response of a thin twisted-nematic cell with different polarizer

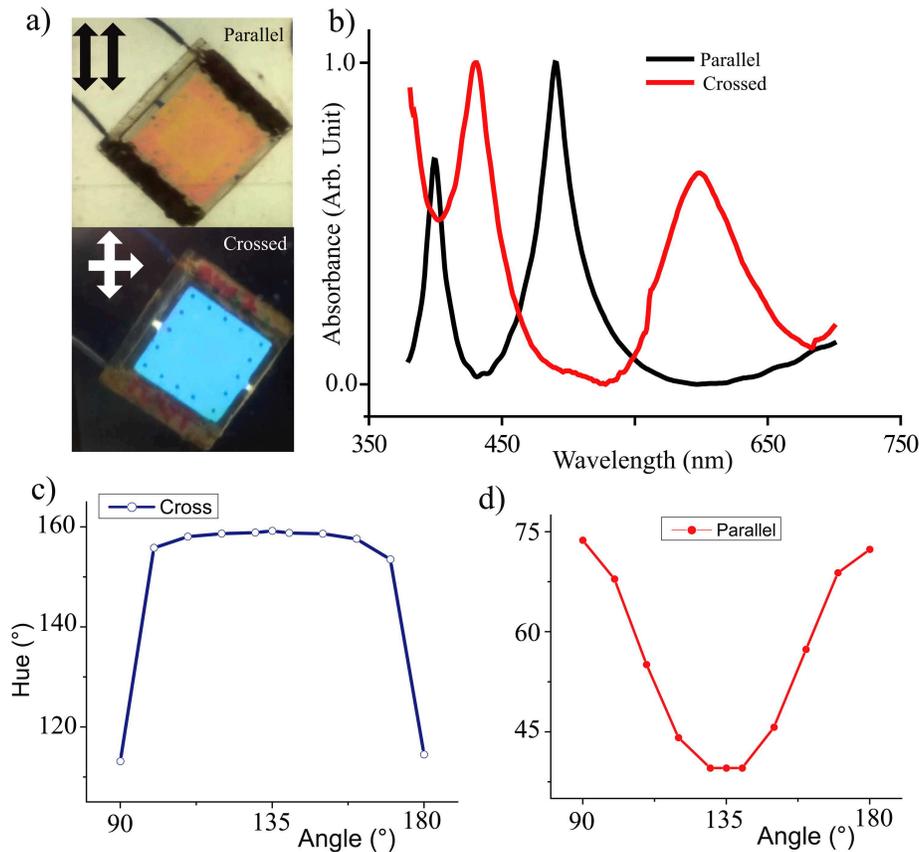


FIG. 3. Absorption spectra of cells with different polarizers configurations, parallel and cross polarizers. (a) Twisted-nematic cell oriented at 45° in between parallel and crossed polarized and illuminated with white light. (b) Absorption spectra obtained in the case of parallel and cross polarizers. The hue of the light transmitted as a function of the angle with respect to the horizontal axis \hat{x} for the twisted-nematic (c) and parallel-nematic (d) liquid crystal cells.

configurations and under white light polarized light illumination. The TN-cell is illuminated from an LCD computer screen with a white background. Figure 3(b) shows the absorbance spectra when the cell is oriented at 45° in between cross and parallel polarizers, respectively.

The CMOS camera has a spectrum in the visible light range with a filter after 650 nm and absorbance with three maximum (455 nm, 540 nm, and 610 nm, approximately, cf. Fig. 2). The white light of the microscope also has a spectrum with three maximum wavelengths (465 nm, 550 nm, and 670 nm, approximately, see Fig. 2) and an intensity decay after 690 nm. The software of the camera works with three filters that provide information about the intensity of primary colors, G green, R red, and B blue. The hue is taken from intensities of the transmitted light and defined as $\text{hue} = \arctan[\sqrt{3}(G - B)/(2R - G - B)]$. Figure 3 shows the hue of the light transmitted as a function of the angle of the sample with respect to the first polarizer and the second crossed (c) and parallel (d) analyzer. Note that in the case of crossed polarizers, the hue remains almost flat in a long region of parameters and exhibits a maximum. On the other hand, in the case of parallel polarizers, the hue is more sensitive and exhibits a minimum.

A. Fréedericksz transition

Let us consider a liquid crystal cell when it is illuminated with a white light at a fixed alternating current voltage of strength V_0 and frequency ω . Hence, a hue(V_0, ω) of a given color is observed. When sweeping with different values of voltage intensity a bar of different colors is found. Figure 4 shows the change of the hue of the transmitted light as a function of the voltage strength for liquid crystal cells with twisted-nematic and planar-nematic configurations. For small voltages, below the deterministically expected threshold of the FT, V_{FT} , we observe that the hue changes slightly, leading to an *imperfect bifurcation*. This type of variations in color is a consequence of the imperfection of the system such as inherent fluctuations of the macroscopic systems (thermal and electronic) and pretilt due to flaw of the anchoring of the molecules in the walls.^{32,33} Fluctuations cause directors to reorient randomly in all directions, and on the other hand, the anchoring in cell walls tends to align the director with these. The balance between these two processes generates an effective director orientation on average. This phenomenon of balance between fluctuations and the relaxation process is well-known as a precursor.³⁴ As a system approaches a bifurcation this effect increases.

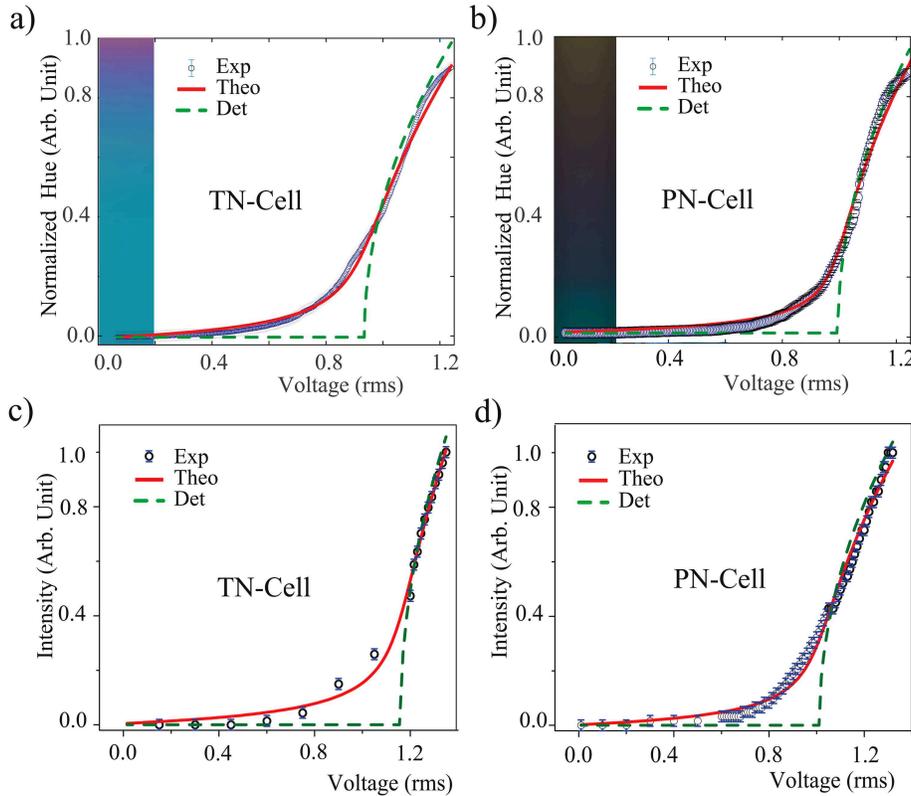


FIG. 4. Bifurcation diagram of a twisted-nematic [(a) and (c)] and planar-nematic [(b) and (d)] liquid crystal cells, respectively. Results were obtained with a sinusoidal waveform of type up-ramp, frequency 1 kHz. The top panels show the normalized hue of the transmitted white light as a function of the voltage strength (rms). The vertical color bars illustrate the color observed for the respective hue. The bottom panels show the normalized intensity obtained from the transmittance of white light and power meter calibrated to 532 nm, as a function of the voltage strength (rms). The hue and transmitted intensity are normalized in arbitrary units. The black open circle symbols are the data obtained experimentally and blue bar is its respective error, the continuous red curves are obtained by using formula (1), with $\alpha = 1.99$, $V_{FT} = 0.9501$, $\eta = 0.0049$, and $hue_0 = -0.098$ (a) and $\alpha = 2.20$, $V_{FT} = 0.9986$, $\eta = 0.0015$, and $hue_0 = -0.060$ (b). Likewise, we have used the same formula (1) to fit the intensity curves with $\alpha = 2.50$, $V_{FT} = 1.1660$, $\eta = 0.0033$, and $hue_0 = -0.089$ (c) and $\alpha = 1.87$, $V_{FT} = 1.0160$, $\eta = 0.0039$, and $hue_0 = -0.080$ (d). The green dashed lines account for the deterministic bifurcation diagram by using formula (1) for the hue and intensity of the light with $\eta = 0$ and $hue_0 = 0$.

If the fluctuations and imperfections were not considered, one expects below the Fréedericksz transition voltage, the hue of the transmitted light should be constant, and when the critical voltage is surpassed it should begin to change the color smoothly (supercritical transition). Hence, the presence of inherent fluctuations and imperfections transform this bifurcation into an imperfect one, as it is illustrated in Fig. 4. Note that similar shape for the bifurcation diagram is obtained by other experimental methods such as interferometry,⁸ dielectric,⁹ capacitive,^{10,11} transmittance,¹² and refractive effects.¹³ Experimentally, only one branch of the supercritical bifurcation is observed because the transmitted light is related to effective birefringence, which is a function of the average director orientation and does not depend on the sign of the angle between the director and glass layers.

B. Colorimetry characterization of molecular reorientation transition

To characterize the noisy bifurcation, we can use the universal shape law of stochastic supercritical bifurcation,^{32,33}

$$hue(V_0) = \alpha \sqrt{\frac{\Delta V + \sqrt{\Delta V^2 + 2\eta}}{2}} + hue_0, \tag{1}$$

where $\Delta V \equiv V_0 - V_{FT}$ is the bifurcation parameter that accounts for voltage displacement with respect to the Fréedericksz transition

voltage V_{FT} , α is a conversion factor between voltage and hue, η is the level of noise intensity of the orientational fluctuations originated by the defective pretilt, thermal, and electronic fluctuations, and hue_0 accounts for the hue of the transmitted light at zero voltage, which is regulated by the pretilt. This formula has been used to characterize the hue of the transmitted light close to the Fréedericksz transition for different liquid crystal cells. Top panels of Fig. 4 show a comparison of this formula with the experimental data showing a quite fair agreement. Then, from the recording of the transmitted light, we can determine with a good approximation the Fréedericksz transition voltage. For example, for a twisted-nematic cell of a thickness of $4.0 \mu\text{m}$ at room temperature, we obtain a critical voltage of $V_{FT} = 0.9501$, which is close to the value provided by the manufacturer of E7 (Merck $V_{FT} = 1.05 \text{ V}$). Likewise, the intensity level of the fluctuations η for the director reorientation is determined from the data. When fluctuations are ignored in formula (1), one retrieves the deterministic bifurcation diagram, i.e., $hue(V_0) = 0$ for $V_0 \leq V_{FT}$ and $hue = \alpha \sqrt{V_0 - V_{FT}}$ for $V_0 > V_{FT}$. The dashed (online green) curves in Fig. 4 illustrate the deterministic curves.

C. Characterization of the molecular reorientation transition by light transmittance intensity

We have measured the transmittance intensity of a white light source using a power meter (Thorlabs PM400, with a detector of

silicon powerhead, S121C). The instrument is calibrated to detect the transmittance intensity of the wavelength of 532 nm. This transmittance intensity can be transformed into a normalized phase.³⁵ The bottom panels of Fig. 4 show the bifurcation diagrams found for the different configurations. Note that both methods lead to similar qualitative and quantitative results; the bifurcation point is shifted because of imperfections and fluctuations intrinsically present in the system. Observe that bifurcation diagrams of the hue and the normalized intensity are similar; however, the critical voltages are

slightly different ($V_{FT} = 1.13$ and 1.00 , respectively, for the twisted-nematic and planar-nematic cells). Note that we have not detected hydrodynamic effects on the range of frequency and voltage under study. The sample maintains homogeneous behavior (no backflow, rolls, or other patterns are observed) and impurity movements are not detected. From the previous analysis, we can determine the bifurcation diagram of the molecular reorientation transition as a function of different frequencies of the applied voltage. Figure 5 summarizes how the bifurcation diagram, critical Fréedericksz transition voltage, and the level of intensity of the fluctuations are changed as a function of the voltage frequency. From this analysis, we infer that the critical voltage as a function of the frequency of the applied voltage increases if the voltage frequency rises. This result may be a consequence of the modification of the dielectric susceptibility as a function of the driving voltage frequency. Characterization of the origin of this critical voltage behavior is in progress.

IV. CONCLUSIONS

The understanding of molecular reorientation dynamics in liquid crystal cells induced by external fields is a problem of fundamental relevance for optoelectronic and electronic devices. We have studied the molecular reorientation transition of twisted-nematic and planar-nematic cells by analyzing their color adjustment under white light illumination. By monitoring the hue of the transmitted light, the bifurcation diagram, transition voltage, and intensity level of the random orientational fluctuations were analyzed. The colorimetry method is validated by comparing the obtained bifurcation diagrams with those obtained for the same cells by using the conventional method of light intensity. The qualitative and quantitative behaviors are confirmed. The critical transition voltage as a function of the frequency of the applied voltage is determined.

The characterization of the reorientation transition of the molecules by using colorimetry is a flexible and precise technique. Likewise, easily implemented in a microscope, this technique of characterization of molecular reorientation can become a standard of characterization of liquid crystal cells, in particular, in laboratories oriented to chemistry or biology or routinely quality control. Hence, colorimetry complemented with the bifurcation theory opens a manageable way to identify transition points and dynamical features with minimal resources.

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DATA AVAILABILITY

The data that support the findings of this study are available within the article.

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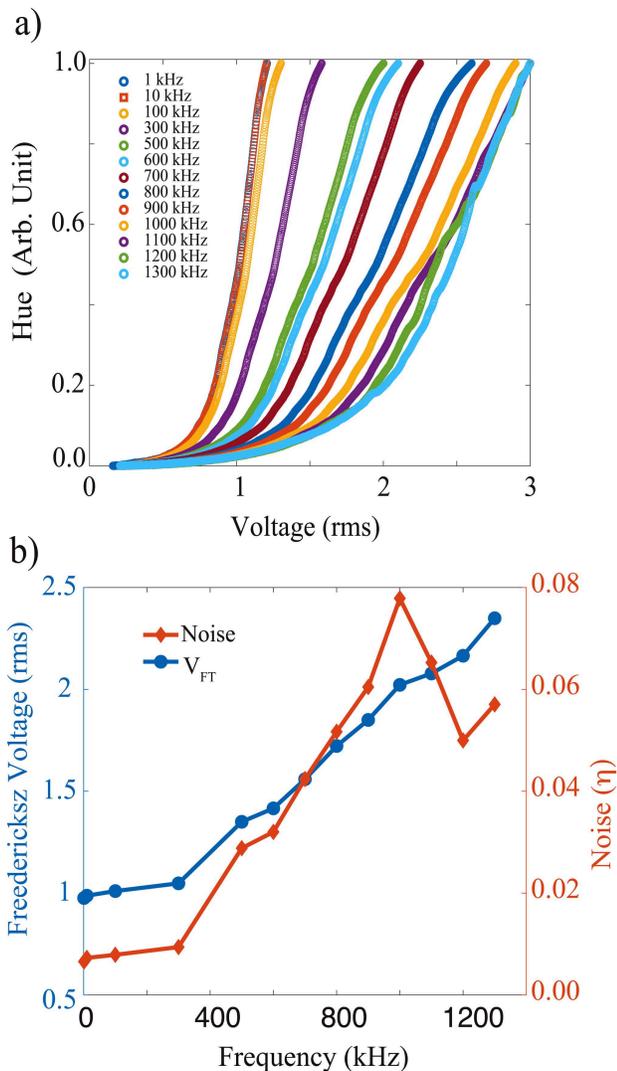


FIG. 5. Critical voltage as a function of the applied voltage frequency. (a) Bifurcation diagram of liquid crystal cells for different frequencies of the applied voltage. The hue of the transmitted light is plotted as a function of the voltage strength (rms); the color curves account for different frequencies. (b) Critical voltage and noise level intensity as a function of the applied voltage frequency.

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