Orientational coupling enhancement in a carbon nanotube dispersed liquid crystal

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We present a detailed study of a dilute suspension of carbon nanotubes (CNTs) in a pentylcyanobiphenyl (5CB) liquid crystal (LC) by probing the dielectric properties as a function of applied ac voltage and frequency. In principle, to minimize the elastic distortion in the nematic matrix, the monodispersed CNTs follow the nematic director without disturbing the director field significantly. A strong anchoring energy due to π - π electron stacking between LC-CNT molecules results in an increase in the dielectric anisotropy for the hybrid system, indicating a significant enhancement in the orientational order parameter. The frequency-dependent dielectric anisotropy for the composite system reveals the intrinsic frequency response of the LC-CNT anchoring mechanism. As a matter of consequence, the extracted value of splay elastic constant suggests that LC-CNT anchoring has an impact on the structural modification of the hybrid LC+CNT system. This strong anchoring energy stabilizes local *pseudonematic domains*, giving rise to a nonzero dielectric anisotropy in the isotropic phase that also shows an intrinsic frequency response.

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Nematic liquid crystals (LCs) have gained great research interest in recent years for transferring their orientational order onto dispersed nanomaterials [1-7]. Experiments have shown that a low concentration of carbon nanotubes (CNTs) sample can be organized in a nematic matrix over macroscopic dimensions by dispersing them in LCs [1,2]. This hybrid LC-CNT represents a versatile composite system that has drawn a great deal of interest in fundamental and applied physics by exhibiting remarkable physical phenomena, such as, improvement in electro-optic response of LCs [4], electromechanical memory effect [8,9], and superelongation of CNT-clusters in the nematic matrix [10]. A dilute suspension of CNTs in an LC platform is a unique assemblage of an anisotropic dispersion in an anisotropic media, which makes it an important and active area of research for realizing the LC-CNT interactions and the principles governing CNT assembly through a nematic mediated platform. In general, the host particles introduce disorder in a nematic matrix, decreasing the nematic order parameter [11]. Here, we show that a dilute suspension of CNTs in a nematic LC results in a significant enhancement in the dielectric anisotropy of the system. Furthermore, the increment in the dielectric anisotropy strongly depends on the probing frequency of an applied ac field. As the dielectric anisotropy, $\Delta \varepsilon$ is proportional to the scalar order parameter S, an enhancement in the dielectric anisotropy indicates an improvement in the net orientational order of the LC. In this paper, we report the fieldinduced dielectric $(\bar{\epsilon})$ response as a function of probing frequency (1-100 kHz) for multiwall carbon nanotubes (MWCNTs) dispersed in 4-cyano-4'-pentylbiphenyl (5CB) LC.

The nematic phase of 5CB LC shows dielectric anisotropy due to the anisotropic nature of the molecules where ε_{\parallel} and ε_{\perp} are the components parallel and perpendicular to the molecular long axis, respectively. 5CB being a positive dielec-

tric anisotropic LC, $\varepsilon_{\parallel} > \varepsilon_{\perp}$, and so, the *director field* (average direction of LC molecules) reorients parallel to an applied electric field. In a uniform homogeneously aligned parallel-plate cell configuration, the nematic director is aligned perpendicular to the applied electric field due to surface anchoring, but the director can reorient parallel to the applied field if the field magnitude is above some critical threshold. This is the essence of a Fréedericksz transition and an ac-capacitive measurement of the $\overline{\varepsilon}$ reveals ε_{\perp} below and ε_{\parallel} above this switching, the exact values depending on frequency. Having very high-aspect ratio, CNTs also exhibit dielectric anisotropy [12].

The CNT alignment mechanism in an nematic matrix is driven by the coupling of the unperturbed director field to the anisotropic interfacial tension of the CNTs in the nematic LC matrix, as individual CNTs (not in bundle) are much thinner than the elastic penetration length [13]. Thus, a dilute concentration of CNTs in an LC is very important for this alignment process as monodispersion without any agglomerates is needed. The LC-CNT composite was prepared by dispersing 0.007 wt % of MWCNT sample in 5CB (nematic to isotropic transition at T_{NI} =35 °C) host. The MWCNT sample used for these experiments contains nanotubes <8 nm in diameter and $0.5-2 \ \mu m$ in length. The mixture then was ultrasonicated for 6-8 h to reach monodispersion of CNTs. Soon after ultrasonication, the mixture was degassed under a vacuum at 40 °C for 2 h. Homogeneous LC cells with 5×5 mm² indium tin oxide (ITO) coated area and 20 μ m spacing [14] were used for our dielectric studies. These cells were filled by capillary action with LC and CNT doped LC separately at an elevated temperature T > 35 °C. The typical amount of the LC or LC+CNT sample filled into the cell is around 0.5 mg. The cell spacing filters out any nanotube aggregates larger than the spacing dimension. Before performing any dielectric measurements, the CNT-doped LC cell was studied under a cross polarized microscope. The optical micrographs revealed a uniform nematic texture, like a pure LC cell, indicating a uniform nematic director field. There were no indications of phase separation or agglomer-

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FIG. 1. (Color online) (a) The dielectric constant $\bar{\varepsilon}$ as a function of applied ac voltage for 5CB and 5CB+CNT in the nematic phase $(T=25 \ ^{\circ}\text{C})$ at frequency 1 kHz; $\Delta \varepsilon_{\text{LC}}=+10.5$ and $\Delta \varepsilon_{\text{LC+CNT}}=+13.8$; (b) the dielectric constant $\bar{\varepsilon}$ as a function of applied ac voltage for 5CB and 5CB+CNT in the nematic phase $(T=25 \ ^{\circ}\text{C})$ at frequency 100 kHz; $\Delta \varepsilon_{\text{LC}}=+10.1$ and $\Delta \varepsilon_{\text{LC+CNT}}=+11.2$.

ates at any temperature. Thus, at least on the length scales probed by visible light, the structure of CNT-aggregates (if any) must be small enough that they do not perturb the director field due to their low concentration and monodispersion.

The dielectric ($\overline{\epsilon}$) measurements as a function of applied ac voltage and frequency (1–100 kHz) were performed by the ac capacitance bridge technique [15–17]. The reason for applying the ac voltage (not dc) is to avoid the affect of ion migration on the dielectric measurements. The LC 5CB does not exhibit any tumbling mode [18] and MWCNTs show no space charge or dipole orientation dynamics [17,19] at the probing frequencies. Thus, the observed increase in $\overline{\epsilon}$ as a function of applied ac voltage should be driven mainly by a mechanical rotation of the nematic director field.

Figures 1(a) and 1(b) show the dielectric constant ($\overline{\varepsilon}$) as a function of applied ac voltage for 5CB and 5CB+MWCNT sample in the nematic phase (T=25 °C) at the two extreme probing frequencies 1 and 100 kHz, respectively, in the frequency range studied. As seen in the Fig. 1, both the LC and the mixture undergo planner (ε_{\perp}) to homeotropic $(\varepsilon_{\parallel})$ orientational transition, as the applied voltage increases. The dielectric anisotropy ($\Delta \varepsilon = \varepsilon_{\parallel} - \varepsilon_{\perp}$) for pure 5CB is around +10 [20], as also experimentally observed $10.5 \ge \Delta \varepsilon_{LC} \ge 10.1$ in the probing frequency range 1-100 kHz, shown in Fig. 2(a). Figures 1 and 2(a) depict that the dilute concentration of CNTs in the LC results in a dramatic increase in the dielectric anisotropy of the system. At 1 kHz, $\Delta \varepsilon_{\text{LC+CNT}}$ =+13.8, that gradually decreases to, $\Delta \varepsilon_{\text{LC+CNT}}$ =+11.2 at 100 kHz. Everywhere in the frequency range studied, $\Delta \varepsilon_{\rm LC+CNT}$ $>\Delta \varepsilon_{\rm LC}$. This indicates an improvement in the orientational order in the nematic matrix as $\Delta \varepsilon \propto S$ [21]. It is important to point out that this dramatic increase occurred due to the addition of only 0.007 wt % MWCNT sample to the LC.

The nanotubes used for this work are not ferroelectric in nature. Thus, an additional ordering effect observed must not



FIG. 2. (Color online) (a) The dielectric anisotropy $\Delta \varepsilon$ as a function of frequency for 5CB and 5CB+CNT in the nematic phase (*T*=25 °C); (b) the dielectric anisotropy $\Delta \varepsilon$ as a function of frequency for 5CB and 5CB+CNT in isotropic phase (*T*=42 °C).

be due to the electronic coupling of any permanent dipole moments with the LC dielectric anisotropy that generally occurs in ferroelectric nanoparticle suspensions in the LCs [22]. A low-concentration CNT suspension in an LC is stable because dispersed CNTs, without large agglomerates, does not disturb the director field significantly. Importantly, the suspended CNTs share their intrinsic properties with the LC matrix, such as conductivity [2] or dielectric anisotropy in this case, due to the alignment with the LC molecules. The suspension, therefore, has been characterized by an effective dielectric anisotropy $\Delta \varepsilon_{LC+CNT}$ as if the CNTs acted as a molecular additive in the LC media. As CNT concentration is very low (0.007 wt %), the CNT-CNT interaction is insignificant, and therefore, the effective dielectric anisotropy can be written as

$$\Delta \varepsilon_{\rm LC+CNT} = \rho_{\rm LC} \Delta \varepsilon_{\rm LC} + \rho_{\rm CNT} \Delta \varepsilon_{\rm CNT}, \qquad (1)$$

where $\rho_{\rm LC}$ and $\rho_{\rm CNT}$ are volume fractions of LC and CNTs, respectively, in the mixture. Substituting $\rho_{\rm LC}$ =0.999 95, $\rho_{\rm CNT}$ =5×10⁻⁵, $\Delta \varepsilon_{\rm LC}$ (1 kHz)=10.5, and $\Delta \varepsilon_{\rm CNT} \approx 10^4$ [23] in equation-1 gives $\Delta \varepsilon_{\rm LC+CNT}$ (1 kHz)=10.999. This predicted result does not agree with the experimental value, $\Delta \varepsilon_{\rm LC+CNT}$ (1 kHz)=13.8, suggesting a required modification in Eq. (1) for LC-CNT composite.

Recent theoretical calculations predict that a strong interaction, mainly due to surface anchoring with a binding energy of about $U_{anchoring}=-2$ eV for π - π electron stacking between LC-CNT molecules [4,24], is associated with the CNT alignment mechanism in the nematic state. In our model, this anchoring energy induces local short-range orientation order of LC molecules surrounding the CNT wall having *local director* along the tube axis, shown schematically in Fig. 3. These local *pseudonematic domains* (PNDs) have different anisotropy than the bulk LC depending on the radius, length, and chirality of the CNTs. The mean induced short-range orientation order increases the net polarizability



FIG. 3. (Color online) Schematic diagrams; (a) the presence of a CNT (cylindrical) in an nematic LC media and formation of the local short range orientational order by LC molecules on the surface of the CNT due to the LC-CNT anchoring energy (π - π stacking). The thin arrow represents the long-range nematic director and the thick arrow on the CNT represents the short range *local director*; (b) the isotropic phase of LC; (c) the presence of a CNT (cylindrical) in an isotropic LC media and formation of the local short range orientational order by LC molecules.

of the system. In the nematic matrix a CNT long axis is coupled to the nematic director [1,2], and so, the nematic state gains an additional orientational order for this anchoring energy due to the presence of CNTs. Thus, one can visualize the suspended CNTs in the nematic media as *local anchoring fields* along the nematic director that collectively amplify the orientational order in the matrix. This enhancement in orientational order causes for an increase in $\Delta \varepsilon$ in the hybrid system. So, the formula for $\Delta \varepsilon_{LC+CNT}$ can be modified as

$$\Delta \varepsilon_{\rm LC+CNT} = \rho_{\rm LC} \Delta \varepsilon_{\rm LC} + \rho_{\rm CNT} \Delta \varepsilon_{\rm CNT} + \Sigma \Delta \varepsilon_{\rm PND}, \qquad (2)$$

where $\Delta \varepsilon_{PND}$ is the dielectric anisotropy of a local PND formed by the LCs. Clearly, $\Delta \varepsilon_{PND}$ arises from the LC-CNT interaction and, all the domains have cumulative effect on $\Delta \varepsilon_{\text{LC+CNT}}$. Simple calculation reveals that $\Sigma \Delta \varepsilon_{\text{PND}}$ $(=\Delta \varepsilon_{\rm LC+CNT} - \rho_{\rm LC} \Delta \varepsilon_{\rm LC} - \rho_{\rm CNT} \Delta \varepsilon_{\rm CNT} = 2.8)$ contributes as much as 20% to the total value of $\Delta \epsilon_{\text{LC+CNT}}$. Additional measurements with slightly lower CNT concentration (0.0045 wt %) yielded lower value of $\Delta \varepsilon_{\text{LC+CNT}}$ =+13.1 at 1 kHz. This indicates that lower CNT concentration reduces the value of $\Sigma \Delta \varepsilon_{\text{PND}}$, resulting in a decrease in the effective anisotropy. Recently, it has been shown experimentally that the nematic to isotropic phase transition temperature of a liquid crystal is enhanced by incorporating of small amount of MWNT sample [25] that indicates the significant increase in the degree of LC order, supporting our results. However, the transition temperature should not always depend upon the CNT concentration but also on how well CNTs are dispersed.

Figure 2(a) reveals the significant impact of the applied probing frequency on $\Delta \varepsilon$ for the hybrid LC-CNT system. A 4% increase (10.1–10.5) in the $\Delta \varepsilon$ for 5CB in the low-frequency regime might be attributed to the ionic conduction/relaxation mechanism [26]. But, a 24% increase (11.2–13.8) in the $\Delta \varepsilon$ for the composite in the low-frequency regime should not be caused only due to ionic conduction/ relaxation mechanism as only 0.007 wt % CNTs were added to the LC. Also, some recent work shows that the presence of



FIG. 4. (Color online) The splay elastic constant K_{11} (in pN) as a function of applied frequency for 5CB and 5CB+CNT in the nematic phase (T=25 °C). K_{11} has been extracted from the formula $V_{\text{th}}=\pi\sqrt{(K_{11}/\varepsilon_o\Delta\varepsilon)}$.

the CNTs reduces the free-ion concentration in an LC media by ion trapping process [4,27]. This suggests that the ions do not play a significant role in the dramatic increase in $\Delta\varepsilon$ for the composite system in the low-frequency regime (1–10 kHz). It is possible that the anchoring mechanism by π - π electron stacking between LC-CNT molecules is affected by the applied probing frequency as the LC molecules have an intrinsic frequency response, and therefore, $\Delta\varepsilon_{\rm PND}$ is a frequency-dependent quantity. The dramatic increase in $\Delta\varepsilon_{\rm LC+CNT}$ with decreasing frequency suggests that the anchoring mechanism favors the low-frequency regime (1–10 kHz), showing the intrinsic frequency response of the *local directors* formed by LC-CNT anchoring.

The nematic phase (T=25 °C) of pure 5CB exhibits $S \approx 0.6$ [28]. As $\Delta \varepsilon \infty S$, the order parameter *S* can be scaled from our measured dielectric anisotropy $\Delta \varepsilon$ in the nematic phase at T=25 °C. Thus, a 31.43% increase in the dielectric anisotropy (10.5–13.8) at 1 kHz due to incorporating CNTs implies the same 31.43% enhancement in the orientational order parameter *S* (0.6–0.78). This significant amplification in the orientational coupling is attributed to the LC-CNT interaction (i.e., formation of PND) and CNT alignment with the LC.

The splay elastic constant K_{11} for the bent distortion for the LC and the hybrid system has been extracted from the well-known formula,

$$V_{\rm th} = \pi \sqrt{(K_{11}/\varepsilon_o \Delta \varepsilon)},\tag{3}$$

where V_{th} is the reorientation threshold voltage for the Fréedericksz transition [21]. The estimated K_{11} , by using the observed experimental values of V_{th} and $\Delta \varepsilon$, is shown in the Fig. 4 as a function of the probing frequency. Clearly, $K_{11}^{\text{LC}+\text{CNT}} > K_{11}^{\text{LC}}$. It has been observed that V_{th} does not quite change with increasing frequency, and so, increasing $\Delta \varepsilon$ increases K_{11} with decreasing frequency for the hybrid system. The strong elastic interaction between LC-CNT due to the surface anchoring may increase the elastic energy of the hybrid system and therefore can be attributed to the increase in K_{11} in the LC+CNT system; which is essentially a structural modification in the nematic phase due to the addition of a small amount of CNTs. The dramatic increase in K_{11} for the hybrid system in the low-frequency regime (same as $\Delta \varepsilon$) is attributed to the enhancement in the anchoring mechanism in that frequency range.

The LC-CNT anchoring mechanism becomes more prominent in the isotropic phase. Due to the absence of elastic interactions in the isotropic phase, the LC molecules no longer maintain long-range orientation order and act as an isotropic liquid. Isotropic phase of 5CB, as expected, shows $\Delta \varepsilon = 0$, confirming no long range orientational order; S = 0. See Fig. 2(b). But a dramatic change in the field-induced dielectric constant has been observed in the isotropic phase for the hybrid system. The composite system shows an increment in $\overline{\varepsilon}$ with increasing applied voltage without saturating at the highest voltage applied. Also, no distinct $V_{\rm th}$ has been observed in the isotropic phase. So, the dielectric anisotropy in the isotropic phase for the LC+CNT system cannot be defined as $\Delta \varepsilon = \varepsilon_{\parallel} - \varepsilon_{\perp}$. Rather, the dielectric anisotropy for the composite system in the isotropic phase is defined as $\Delta \varepsilon = \overline{\varepsilon}_{max} - \overline{\varepsilon}_{min}$, where $\overline{\varepsilon}_{max}$ and $\overline{\varepsilon}_{min}$ are determined from an " $\overline{\varepsilon}$ vs applied voltage" graph (not shown in this letter) at a temperature above $T_{NI}=35$ °C. The frequency dependence of $\Delta \varepsilon$ in the isotropic phase is shown in Fig. 2(b).

As stated earlier, the energy associated with LC-CNT anchoring mechanisms is $U_{\text{anchoring}} = -2 \text{ eV}$. In the deep isotropic state at T=42 °C=315 K, the thermal energy $U_{\text{thermal}} \sim k_{\text{B}}T=2.71 \times 10^{-2} \text{ eV}$. So, the thermal energy is not even close enough to eliminate the anchoring mechanisms in the isotropic phase. Thus, local short-range orientation order of LC molecules surrounding the CNT due to the anchoring, discussed earlier, still exists in the isotropic media. This can be visualized as the presence of isolated pseudonematic domains in an isotropic media, as schematically shown in Fig. 3(c). This indicates that in the isotropic phase CNTs, surrounded by few layers of LCs, stabilize nematiclike shortrange order in the isotropic phase, giving rise to a nonzero order parameter. This *isotropic order parameter* would depend on the concentration and distribution symmetry of the CNTs in the LC media. As these local anisotropic pseudonematic domains have polarization, their short-range director field interact with external electric fields, resulting in a nonzero value of $\Delta \varepsilon$ in the isotropic phase. The intrinsic frequency response of the anchoring mechanism in the isotropic phase behaves in a very different way than it does in the nematic phase, clearly observed in Fig. 2. However, the composite possesses higher anisotropy ($\Delta \varepsilon = 3.4$) at 1 kHz that gradually decreases ($\Delta \varepsilon = 1.5$) at 100 kHz, such as the nematic state.

In conclusion, we have observed that a dilute suspension of CNTs in a nematic LC, results in a dramatic improvement in the nematic ordering, showing enhanced $\Delta \varepsilon$. Even though the CNT sample may contain some impurities, they are not significant enough to alter the $\Delta \varepsilon$. The increase in $\Delta \varepsilon$ is solely attributed to the enhancement in orientational order parameter S. If the enhancement in $\Delta \varepsilon$ in the hybrid system in the nematic phase were to occur due to the impurities or ionic conduction and not due the PND, then the value of $\Delta \varepsilon$ in the isotropic phase would have been zero, such as pure LC. The strong frequency dependence of the dielectric anisotropy suggests that the LC-CNT anchoring mechanism may be perturbed by the probing frequency of the applied field. However, more experimental and theoretical studies are needed to fully understand the frequency response of the anchoring mechanism. The presence of local anisotropic pseudonematic domains in the isotropic phase causes a net residual order in the hybrid system, exhibiting nonzero value of $\Delta \varepsilon$ that is also strongly influenced by the applied probing frequency. Future work involves light scattering studies on concentration-dependent CNT suspensions in LC media to investigate more on the frequency-dependent anchoring effect in both the nematic and isotropic phases.

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