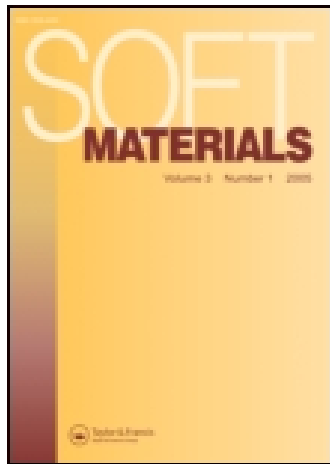


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Electrokinetic Study and Surface Conductance of Carbon Nanotubes in Liquid Crystal Medium

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The main objective of this study is to investigate the effect of dielectrophoresis and electrophoresis forces on charge and zeta potential values for colloidal carbon nanotubes in liquid crystal medium, which are investigated in homogeneously aligned in-plane field driven cell. It has been detected that carbon nanotubes suspended in liquid crystal medium oppose an applied electric field and behave like dielectric material. The dielectrophoresis force, electrophoresis force, electrophoretic velocity, zeta potential, charges on carbon nanotubes, Dukhin number, and surface conductance of colloidal carbon nanotubes in liquid crystal medium have been evaluated. The significant effect of dielectrophoretic force is found to be present in colloidal system and the value of surface conductance of suspending particles was of the order of $10^{-17}S$.

Keywords: Carbon nanotubes doped liquid crystal, Dielectrophoresis, Electrophoresis, Surface conductance, Zeta Potential

Introduction

The electric field induced forces of colloidal particle have attracted considerable scientific and technological interest in the area of biotechnology and genetic engineering (1), nanotechnology (2–5), and display devices (6). The potential use of electrokinetic techniques is to manipulate and separate micro/nanoparticles. Due to rapid development of this field, new technology has been developed through the application of micro-electronic methods to fabricate small electrode structures that can generate high electric fields from relatively small applied potentials (7). Basically, there are two types of electric field induced forces; dielectrophoresis (DEP) and electrophoresis (EP). The application of non-uniform fields that can induce the movement of polarizable particles is termed as DEP force. In an EP force, particles migrate in response to an applied voltage across electrodes, and the response of material is sensitive to the sign of applied voltage. Charged pigment particles move toward one electrode by EP forces.

The controlled manipulation of colloidal particles in liquid crystal (LC) medium has been the topic of research in recent

years (8–19). Fukuda et al. (8, 9) theoretically investigated the interactions of colloidal particles in nematic LC medium. The Quincke rotation for liquid crystalline phases has been reported by Liao et al. (10). Jakli et al. (11) have explained the translation of spinning particles by an elastic entrapment of spheres at tilted grain boundaries in meniscus region in smectic liquid crystalline phase. Nonlinear electrostatics of microparticles in LC medium have been first noted by Lavrentovich et al. (12, 13). The perpendicular component of velocity has also been noted by Lavrentovich group (12). Ryzhkova et al. (14) have reported nonlinear dependency of different electrokinetic parameters of microparticles in LC medium. They have also modeled the equation in order to estimate electrokinetic force (15). Pishnyak et al. (16) have demonstrated controlled out-of-equilibrium aggregation of spherical particles with long-range anisotropic interaction induced by nematic LC as dispersive medium. The translational electromigration of microparticles in LC medium have been explained by Dierking et al. (17).

In recent years, there are reports (14, 18–20) in which the unusual high value of zeta potential has been reported and it was believed (18–20) that this high value of zeta potential might be due to DEP force. However, the exact contribution of DEP force on colloidal particles could not be evaluated. In this paper, contribution of EP and DEP forces, electrophoretic velocity, zeta potential, charges on carbon nanotubes (CNTs), Dukhin number, and surface conductance of colloidal carbon nanotubes in LC medium have been investigated.

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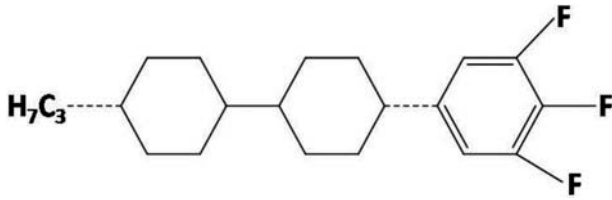


Fig. 1. Chemical structure of trifluorophenyl-2 (TFP2) liquid crystal.

Experimental

The super-fluorinated nematic LC mixture (MJ951160), which has a good reliability in thermal and ultraviolet stress, was purchased from Merck and used as received. Its typical trifluorophenyl-2 (TFP2) structure is shown in the Fig. 1. This molecule is composed of a hexagonal head part with three fluorine atoms, two cyclohexane rings, and a tail part alkyl chain. Its physical properties are listed as follows: dielectric anisotropy $\Delta\epsilon = +7.4$ at 1 kHz, flow viscosity $\eta = 18 \text{ mm}^2/\text{s}$ at 20°C and nematic phase between -40 and 87°C .

For a homogeneous alignment, driven by in-plane field, the interdigitated opaque electrodes made from aluminum were placed on the bottom substrate only. The electrodes have been separated at a distance of $40 \mu\text{m}$ with an electrode width of $10 \mu\text{m}$. For a cell fabrication, a homogenous alignment layer (AL-16139 from Japan Synthetic Rubber Co.) was first spin-coated to a thickness of 800 \AA on an electrode-patterned glass substrate. A rubbing process to the in-plane field direction was then performed on the substrate to align the nematic LC. The same alignment layer was coated on another glass substrate without electrode, and a similar rubbing process was then performed in antiparallel direction to the first one. The cell was assembled to give a cell gap (d) of $9 \mu\text{m}$, where the plastic balls were used to maintain the cell gap.

The HiPco single-walled carbon nanotubes (SWCNTs) (Carbon Nanotechnology Inc.) were used after nitric acid treatment to remove catalysts. The diameter ranged from 4 to 10 nm with a wide distribution of CNTs length from 0.1 to $0.5 \mu\text{m}$ (the most probable SWCNT length was 250 nm), which was observed from atomic force microscopy (AFM: Seiko SPA-400). The size distribution of SWCNTs in LC has been described elsewhere (21).

The SWCNTs in a powder type were not dispersed instantly in LC medium. In order to disperse SWCNTs in LC medium, 10^{-2} wt% of SWCNTs was dissolved into dichloroethane (DCE) solvent, followed by mixing with nematic LC. The amount of weight percentage of SWCNTs was determined by measuring the remaining CNTs on the filter and a nominal amount of CNTs was found to be altered after centrifugation. If the proper conditions, such as sonication time and power were not satisfied, SWCNTs would aggregate themselves during the dispersion process. After solvent evaporation, we measured the clearing point of LC mixture to confirm the evaporation of solvent perfectly. This gave rise to the same clearing temperature as for pure LC. The SWCNTs-dispersed LC mixture was thoroughly sonicated for an hour. Without further treatments, the cell was filled with mixture at room temperature by capillary action. It has been observed that CNTs agglomerate in LC when doping amount of CNTs exceeds the critical weight percent (21, 22).

The fabricated test cells were observed under optical polarizing microscopy (Nikon DXM1200) by applying an electric field of different magnitudes. The velocity (v) of CNTs was determined by monitoring their motion and textures at the rate of 30 frames per second.

Theoretical Aspect

Different approaches have been proposed in order to measure the zeta potential (ζ). A well-known Helmholtz-Smoluchowski equation (23) for zeta potential is based on the assumption that electrical double layer is behaving like a capacitor and $\kappa.r \gg 1$ where κ is the inverse of the Debye length (in m^{-1}) and r is the particle radius. The zeta potential can be calculated from electrophoretic mobility (μ) (23):

$$\zeta = \frac{\eta\mu}{\epsilon_1} \quad (1)$$

where ϵ_1 is the dielectric constant of LC. The other limiting case for $\kappa.r \ll 1$ and Reynolds number (Re) $\ll 1$, is provided by Hückel's formula (24):

$$\zeta = \frac{3.\eta.\mu}{2.\epsilon_1} \quad (2)$$

The zeta potential measured by Hückel-Onsager is increased by 3/2 from Helmholtz-Smoluchowski equation. In order to investigate the effect of DEP force in zeta potential measurement, we assumed that the stroked drag force is balanced by total electric field induced forces due to EP and DEP. When the effect of quadrupole and higher order multipoles are ignored, the electric force acting on the particle in an external electric field can be expressed as (25, 26).

$$6\pi\eta rv = -qE + 2\pi\epsilon_1 r^3 \text{Re}\{K(\epsilon_2^*, \epsilon_1^*)\} \nabla E^2 \quad (3)$$

where ϵ_2^* and ϵ_1^* are complex dielectric permittivities of the colloidal particles and the host dielectric medium, respectively. $K(\epsilon_2^*, \epsilon_1^*)$ represents the complex polarization factor, which is provided by:

$$K(\epsilon_2^*, \epsilon_1^*) = \frac{\epsilon_2^* - \epsilon_1^*}{\epsilon_2^* + 2\epsilon_1^*} \quad (4)$$

where $\epsilon^* = \epsilon - \frac{\sigma}{\omega}j$, and σ and ω stand for the conductivity and electric field frequency, respectively.

The Eq. (3) can be simplified as:

$$6\pi\eta rv = -qE + 4\pi\epsilon_1 r^3 E \text{Re}\{K(\epsilon_2^*, \epsilon_1^*)\} \nabla E \quad (5)$$

The zeta potential which is equal to the surface potential can be written as:

$$\zeta = \frac{q}{4\pi\epsilon_1 r} \quad (6)$$

From Eq. (5) and Eq. (6), the expressions for charge and zeta potential on the particles in terms of EP and DEP forces can be written as:

$$q = \frac{1}{E} [4\pi\epsilon_1 r^3 E \operatorname{Re}\{K(\epsilon_2^*, \epsilon_1^*)\} \nabla E - 6\pi\eta r v] \quad (7)$$

$$\zeta = r^2 \operatorname{Re}\{K(\epsilon_2^*, \epsilon_1^*)\} \nabla E - \frac{3\eta v}{2\epsilon_1 E} \quad (8)$$

Results and Discussion

Fig. 2 shows optical micrographs of dispersed CNTs in LC medium. In the absence of an external electric field, the CNT cluster in LC medium appears in form of black spot. When we apply an AC electric field, CNTs move back and forth between the electrodes whereas in case of DC electric field, CNTs move toward the negative electrode. Hence, according to DEP theory the CNTs are polarized opposite to the direction of applied electric field and behave like dielectric materials similar to diamagnetic materials being magnetized opposite to the direction of applied magnetic field. The term dielectricity in a dielectric material can be characterized by a negative dielectric susceptibility ($\chi_e = \epsilon_r - 1$; for vacuum $\chi_e = 0$) and a polarization opposite to the electric field. Therefore, the dielectric constant (ϵ_r) of the CNTs would be less than one or may be negative. Li et al. (27) have already reported that the dielectric constant of SWNTs is negative, which supports our experimental observation.

The Reynolds number is given by $Re = \rho v r \eta^{-1}$, where ρ is the internal fluid density. Here, we have $\rho = 1.0714 \times 10^3 \text{ kg/m}^3$, $r = 1.25 \times 10^{-6} \text{ m}$, and $\eta = 19.3 \times 10^{-3} \text{ kgm}^{-1}\text{s}^{-1}$ at room temperature (20°C). The value of Re at different applied electric fields is shown in Fig. 3, which was found to be in the order of 10^{-5} (far less than 1) and hence justifies Stokes law. Therefore, Eq. (7) and Eq. (8) can be used to determine the charge and zeta potential on the particles.

For present CNTs doped LC system, $\epsilon_1 = 5.4 \times 10^{-11} \text{ F/m}$ at room temperature (20°C). The experimentally observed velocity of CNTs at applied electric field of $0.5 \text{ V}/\mu\text{m}$ was found to

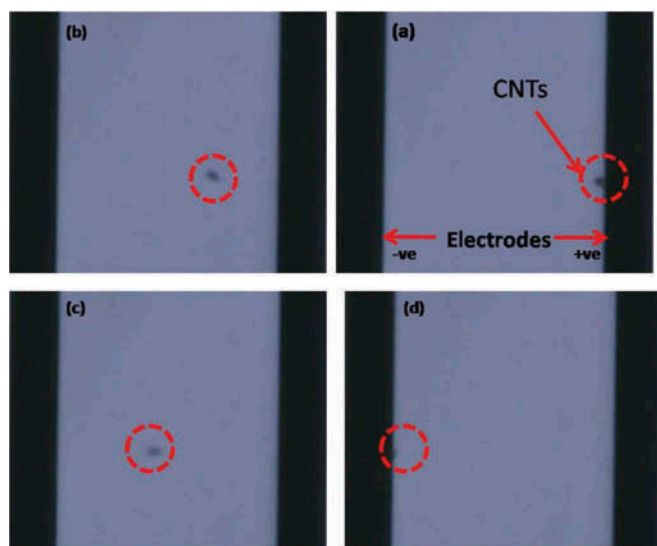


Fig. 2. Initial position of carbon nanotubes in liquid crystal medium (a). Position of carbon nanotubes after 266 ms (b), after 1166 ms (c), and after 1533 ms (d) at applied electric field of $0.5 \text{ V}/\mu\text{m}$.

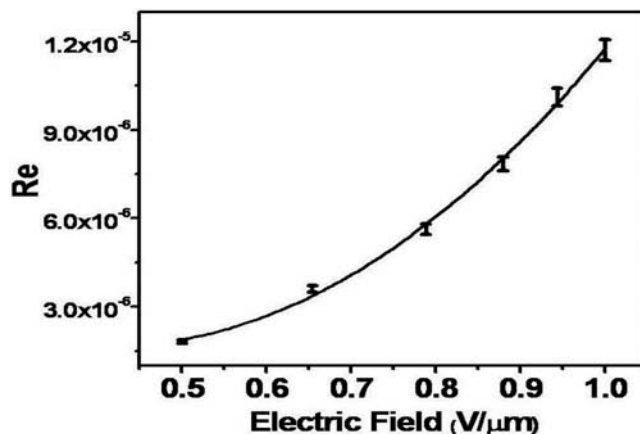


Fig. 3. Variation of Reynolds number (Re) with increasing electric field.

be $26.1 \mu\text{m}/\text{Sec}$. As the dielectric permittivity of CNTs is far greater than the dielectric permittivity of LC (25), the value of $\operatorname{Re}\{K(\epsilon_2^*, \epsilon_1^*)\}$ would be equal to one. To evaluate gradient of electric field in Eq. (7), the computer simulation at electric field of $0.5 \text{ V}/\mu\text{m}$ between two electrodes has been performed by using commercially available software “LCD Master” (Shintech, Japan). The LC molecular orientation and its spatial electric field distribution are shown in Fig. 4. The parameters assigned for simulation were the same as for LC Cell. As indicated in Fig. 4, the strong nonuniform electric field appears at the edge of electrode. However, the electric field between electrodes is almost constant. Therefore, the DEP force between electrodes of in-plane switching (IPS) cell is almost zero. However, at the edge of electrodes, there is strong DEP force effect. As the motion of particles starts from edge of electrode itself, therefore, strong DEP force impulse acting on the particles causes them to go to another electrode. The calculated gradient of electric field (∇E) value at the edge of electrode was $3.27 \times 10^{10} \text{ V/m}^2$. The magnitude of the DEP force was found to be 21.5 pN , whereas the contribution of EP force was only 11.6 pN . Hence, the value of DEP force is greater than that of EP in colloidal suspension. In reiteration, there are 65% of DEP force and only 35% of EP force acting on the colloidal CNTs. Therefore, the DEP force plays a major role in driving the colloidal particles. The contribution of charge and zeta potential due to EP and DEP force is given in Table 1 for comparison. The effective value of charge and zeta potential calculated from Eq. (7) and (8) come out to be $121 e^-$ and 23.0 mV , respectively. However, if we ignore the DEP force then the value of charge and zeta potential would be $148 e^-$ and 28.1 mV , respectively. According to Eq. (3), the applied electric field must be inhomogeneous in order to apply the DEP force. In the present case, the applied field is nonhomogeneous, because of the use of interdigitated electrode only at bottom substrate (22). The distance between bottom and top of the substrates is much larger than the height of bottom interdigitated electrodes, developing a strong nonuniform electric field especially at the aforementioned electrodes.

Variation of velocity of CNTs with the electric fields is shown in Fig. 5. It is evident from Fig. 5 that the velocity of CNTs increases linearly with a third of the power of the electric field strength $v \sim \mu^{(3)} E^3$, which is a similar trend as reported by Ryzhkova et al. (14). The term $\mu^{(3)}$ represents the nonlinear

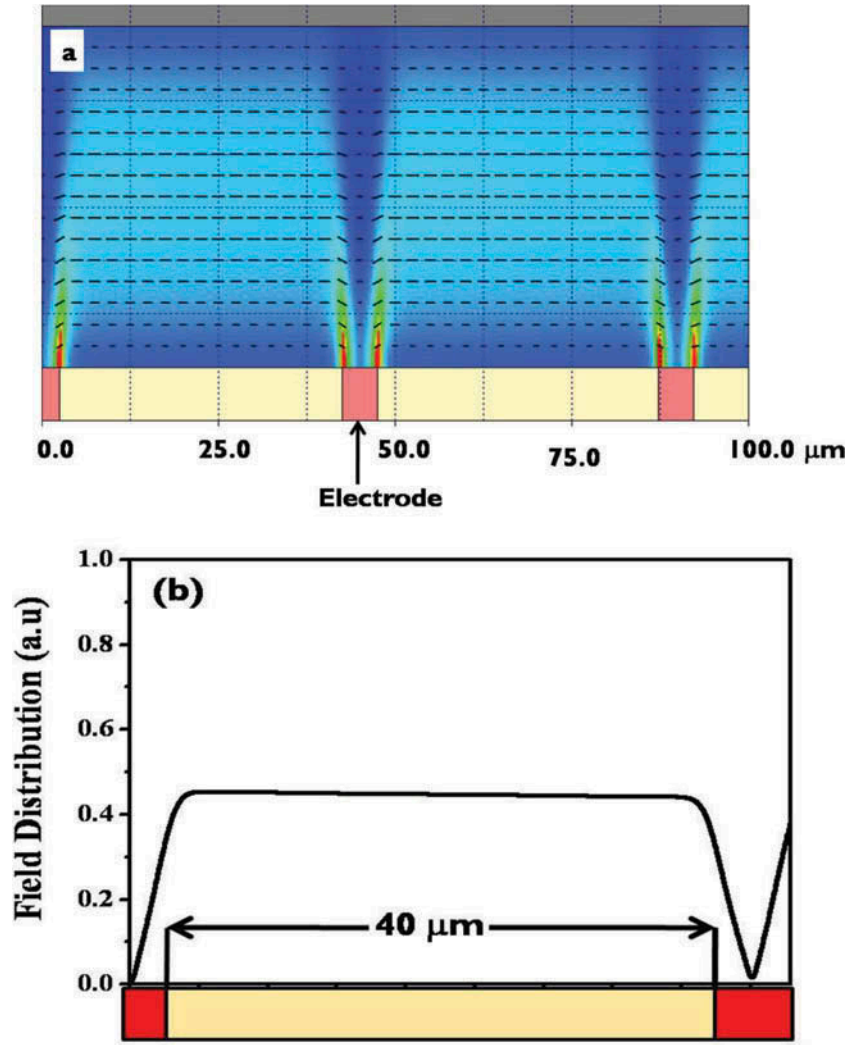


Fig. 4. Molecular distribution (a) and electric field pattern at the middle of the cell (b) at the applied electric field of $0.5V/\mu\text{m}$.

Table 1. Zeta potential and charge on the suspended particles due to EP and DEP force

	Force (pN)	Zeta Potential (mV)	Charge (C)
EP force	11.6	28.1	$148 e^-$
DEP force	21.9	51.1	$269 e^-$
Net force [Eqs. (7) and (8)]	9.6	23.0	$121 e^-$

electrophoretic mobility (14, 27). The coefficient $\mu^{(3)}$ calculated from Fig. 5 was equal to $2.0 \times 10^{-22} \text{ m}^4/V^3\text{s}$. Considering the velocity of colloidal CNTs in positive x - direction, we estimated the Dukhin number and, hence, the surface conductance of colloidal CNTs using the following relation (28, 29):

$$\mu^{(3)} = r^2 \frac{e}{k_B T} \frac{\varepsilon_1}{\eta} \frac{Du[23 + Du(269 + 464Du)]}{252(1 + 2Du)^3}, \quad (9)$$

where $Du = \frac{K^\sigma}{K_{LC}r}$ is the Dukhin's number, a dimensionless parameter that characterizes the role of polarization of thin

double layer in electric response of suspended particles, K^σ is the surface conductance of double layer and K_{LC} is the conductivity of the LC. The surface conductance K^σ is provided by:

$$K^\sigma = K_s^i + K_s^d, \quad (10)$$

where K_s^i is the conductance due to charge movement in the stern layer and K_s^d is the conductance due to charge movement in diffusion layer. The conductivity of LC was of the order of 10^{-9} S/m . The calculated values of Du from Eq. (9) were $+0.01$, -0.92 , and -0.10 . As there are one positive and two negative values and Dukhin number must be a unique number, therefore, unique positive value of $+0.01$ has been taken for surface conductance calculation. The value of surface conductance of double layer of colloidal CNTs for $Du = 0.01$ was found to be of the order of 10^{-17} S . However, most of the authors (30–34) have reported the value of surface conductance varying from 10^{-10} to 10^{-7} S for glass/electrolyte solution. The value of surface conductance reported by Morgan's group (30, 31) for Carboxy-functionalized fluorescent latex particles in KCL electrolyte was of the order of 10^{-9} S in which the conductivity of KCL electrolyte was of the

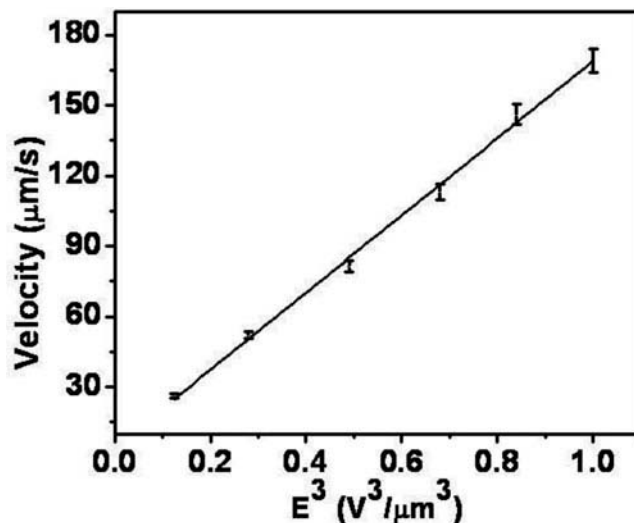


Fig. 5. Dependence of the CNTs velocity in liquid crystal medium on cubic power of electric field strength.

order of $10^{-3} S/m$ and the charge density of suspended particles was $\sim 1 \mu C/cm^2$. The value of surface conductance reported by Olphen and Waxman (32) for sodium bentonite in conductive water was $\sim 10^{-9} S$ in which the conductivity of water was $10^{-4} S/m$ and the charge density of suspended particles was $11.6 \mu C/cm^2$. The charge density in our case can be estimated from the charge given in Table 1 divided by surface area ($4\pi r^2$) and the value was $9.8 \times 10^{-5} \mu C/cm^2$. The value of conductivity of LC was of the order of $10^{-9} S/m$. Both the charge density of suspended particles and the conductivity of suspending medium are relatively small in our case. This may be the reason for getting such a low value of surface conductance. We also cross-checked the low value of surface conductance by relation given below for conductance of stern layer (K_{Stern}) (30, 31):

$$K_s^i = \sigma \mu \quad (11)$$

where σ is the charge density and μ is the mobility. In general, the conductance of diffusion layer is two orders of magnitude less than that of the Stern layer (30). Therefore, as far as the order of the conductance of electrical double layer is concerned, it can be assumed that $K_s^i \approx K^\sigma$. The mobility of the particles at the applied electric field of $0.5 V/\mu m$ was $5.2 \times 10^{-11} m^2 s^{-1} V^{-1}$. The value of surface conductance was found to be $5.1 \times 10^{-17} S$, which agrees well with the value estimated from Eq. (9).

Conclusions

We have investigated the effect of electrophoretic and dielectrophoretic forces and the related parameters such as charge and zeta potential of colloidal CNTs in LC medium. It has been observed that CNTs suspended in LC medium oppose the applied electric field and may have negative electrical susceptibility. The velocity of colloidal CNTs increases linearly with a third power of the applied electric field. There are 65% of DEP and 35% of EP forces acting in CNT-doped LC colloidal suspension. The Dukhin number and the surface conductance of electrical

double layer of CNTs in LC medium were 0.01 and $\sim 10^{-17} S$, respectively. The evaluated small value of surface conductance was due to low conductivity of suspending medium and the small charge density of suspended particles.

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