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Molecular dynamics and alignment studies of silica-filled 4-pentyl-4'-cyanobiphenyl (5CB) liquid crystal

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A comprehensive study of the dielectric properties of 4-pentyl-4'-cyanobiphenyl (5CB) liquid crystal filled with silica particles (particle size 30–80 nm, concentration 2, 3, 5, 10 and 15 wt%). Dielectric spectroscopy in the frequency range 100 to 10^7 Hz was applied to investigate the influence of the filler on the dynamic behaviour of the liquid crystal molecules in both the nematic and isotropic phases. In this frequency range one relaxation process is observed (at $f > 10^6$ Hz). The dynamical behaviour of the 5CB liquid crystal is described by the Cole–Cole relaxation function. The temperature dependence of the relaxation time obeys the empirical Arrhenius equation. The activation energies are approximately 75 kJ mol^{-1} for the pure 5CB sample in the nematic phase and 50 kJ mol^{-1} for the 5 wt% silica-filled 5CB sample. These values are compared with the corresponding literature values. The reversible electro-mechanical response of these samples under the influence of an applied a.c. electric field is investigated.

1. Introduction

Liquid crystals containing dispersed inorganic particles (such as silica) of nano-size are important for both academic research and technological applications. From the fundamental point of view, it is interesting to study the effect of a random perturbation on ordered phases. In this case, the filler particles play the role of the perturber. On the other hand, filler particles at different concentrations are essential in the fabrication of liquid crystal displays (LCDs) with high bistability. The memory effect in LCDs is because the liquid crystal exhibits a transparent state after applying an electric field. This clear state remains after the removal of the applied field [1].

A variety of new properties and phenomena such as modification of phase transitions, orientational order, elastic properties, and director field, have been studied both experimentally and theoretically for liquid crystals (LCs) confined in a random porous network [2–15]. In the recent years, measurements of the dielectric constants of the n CB liquid crystal materials (with $n = 5$ and 8) have been reported [16–20]. In 1996 Rozanski *et al.* [16] employed broadband dielectric spectroscopy

up to 10^9 Hz to study the nematic 5CB liquid crystal in a porous membrane (having a diameter of $0.2 \mu\text{m}$ and length of $60 \mu\text{m}$). In this frequency range, they observed two relaxation processes. It was found that the relaxation rate in the isotropic phase of this sample is not influenced by the presence of the inner surfaces in the confined systems.

On the other hand, in 1998, Sinha and Aliev [17] reported results on the dielectric spectroscopy of 5CB and 8CB in different phases confined in random porous media (with mean sizes of 1000 and 100 \AA). Their measurements were performed in the frequency range 1 mHz to 1.5 GHz. In contrast to the previous results reported by Rozanski, it was found that confinement has a strong influence on the dielectric properties of these LC materials in the isotropic, nematic and smectic A (SmA) phases. In this case, they observed a slow relaxation process due to the presence of a surface layer at the solid pore–wall-LC interface, in which the dynamics of the molecular motion is different from that in the bulk. Moreover, a modified bulk-like relaxation process (due to the rotation of molecules around their short axes and due to the tumbling of molecules) was observed in the confined geometry. Also, it was mentioned that the main differences between the

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dynamic behaviour of confined LCs and that in the bulk, are due to finite size effects and the existence of the LC pore–wall interface.

In another investigation [18] of the dynamic properties of 8CB confined in an aerogel host, by dynamic light scattering, the observed dynamic behaviour was different from that in sintered porous silica [15]. In this report, the spin glass interpretation was compared with the random field interpretation given in [15]. Both interpretations are consistent if the geometrical differences between two matrices are taken into account. The orientational dynamics of 5CB confined in nanometer scale porous silica glass was investigated by the time – resolved transient grating optical Kerr effect [21] in the temperature range corresponding to the bulk deep isotropic phase. A non-exponential relaxation and a distribution of relaxation times were observed. The pore size dependence of the relaxation times was explained from the point of view of the Landau model applied to independent pore segments. However, this model and the distribution of the pore size do not describe the non-exponential form of the decay.

Moreover, Schönhals *et al.* [19], reported results on the dielectric behaviour of unaligned samples of 8CB smectic liquid crystal. Their measurements were performed at different temperatures in a large frequency range (10^{-2} to 10^9 Hz). They used Havriliak–Negami functions to evaluate the components of the complex dielectric permittivity and the corresponding dielectric shape parameters. They conclude that in this frequency range, the applicability of the well known Arrhenius equation is questionable.

In 1999, Urban *et al.* [20] used two different experimental techniques, time domain spectroscopy and impedance analyser techniques, to investigate the dielectric relaxation of chiral 5*CB in the liquid state up to 90 K above the melting point. They concluded that, although the chiral 5*CB and 5CB liquid crystals have very close molecular structure, they are completely different in phase behaviour.

In this paper we report a comprehensive study of the dielectric properties of 4-pentyl-4'-cyanobiphenyl (5CB) liquid crystal filled with silica particles (particle size 30–80 nm, concentration 2, 3, 5, 10 and 15 wt %). Dielectric spectroscopy in the frequency range 100 to 10^7 Hz was applied to investigate the influence of the filler on the dynamic behaviour of the LCs in both the nematic and isotropic phases. Investigation of the molecular alignment in the presence of a filler network of random domain distributions under the influence of an applied a.c. electric field has been performed for all samples under consideration.

2. Experimental

We used the 4-*n*-pentyl-4'-cyanobiphenyl (5CB) nematic liquid crystal without purification as obtained from E. Merk (Darmstadt, Germany). The filler used is a white amorphous silica powder, purchased from Degussa-Huels, Germany. It was produced by precipitation from aqueous solution, its density is 2.1 g cm^{-3} , and the primary particle size is 30–80 nm, as calculated from the specific surface area $175 \text{ m}^2 \text{ g}^{-1}$. The filled samples were prepared by gradually mixing a certain amount of silica particles (0.6 to 4.5 mg according to the desired concentration) on a metal plate with the 5CB liquid crystal (30 mg) in the nematic phase at room temperature. It was easy to obtain the formation of a LC-silica dispersion, by stirring with a rod. The material was surrounded by a polyimide spacer and covered by another metal plate, for the dielectric measurements.

The experimental set-up for the dielectric measurements from 100 to 10^7 Hz is shown in figure 1. The set-up consists of a sample holder, heat chamber, temperature regulator and Solartron frequency response analyser (model 1260) connected to a computer. The real and imaginary parts of the complex dielectric permittivity were studied as a function of frequency and temperature.

The complex dielectric constant ϵ^* at angular frequency ω is related to the capacitance C and conductance G of the sample as follows

$$\epsilon^* = \epsilon' - i\epsilon'' \quad (1)$$

where

$$\epsilon' = \frac{d C}{\epsilon_0 A} \quad (2)$$

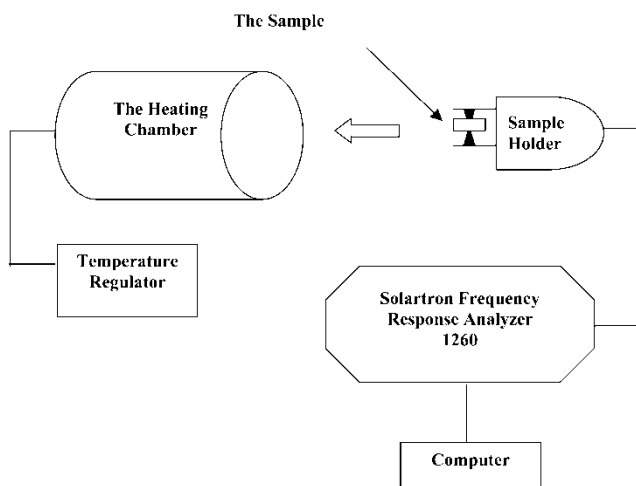


Figure 1. Schematic diagram of the dielectric experiment set-up.

and

$$\varepsilon'' = \frac{d G}{\varepsilon_0 A \omega} \quad (3)$$

In the above equations d is the thickness of the sample, A is the area of the electrode and ω is the angular frequency, where, $\omega = 2\pi f$, and f is the frequency. Also the a.c. conductivity σ , is given by the relation:

$$\sigma = \omega \varepsilon_0 \varepsilon'' \quad (4)$$

where ε_0 is the permittivity of free space.

In this experiment, two stainless steel circular plate electrodes, the upper plate of diameter 3 cm, are used. The two plates are separated by a circular polyimide spacer of 0.125 mm thickness (inner diameter 17 mm, outer diameter 21 mm). The cell is filled with the liquid crystal sample under consideration and held in the sample holder by two pin electrodes. The temperature-controlled chamber is made of copper with a relatively large thermal mass in order to stabilize the temperature. The sample holder inside the chamber is isolated in order to minimize the temperature gradient over the sample. The heating element is connected with a temperature regulator capable of maintaining the temperature within $\pm 0.01^\circ\text{C}$ in the range -190 to 160°C . A platinum thermistor (Pt100) is used in the regulator to sense the temperature, forming a feedback loop with the controller. The temperature of the sample is read out using another platinum resistor (Pt100) inserted very near to the sample.

The capacitance and conductance of the sample condenser were measured using Solartron frequency response analyser 1260 in the frequency range 100 to 10^7 Hz. The impedance measurements in this frequency range can be performed step by step. The applied signal amplitude is 1 V for all the dynamic measurements. From the measurements of the complex impedance, the analyser evaluates automatically the capacitance and conductance values. A computer collects these values at different frequencies from the analyser and the values of the dielectric permittivity and dielectric absorption are calculated instantaneously using equations (2) and (3), respectively. The accuracy of the measurements (especially the dielectric loss) has been checked by the measurement of a known low-loss teflon capacitor inside the chamber. The error in the measurement of the loss tangent is below 1.5% in the region below 1 MHz. Above 1 MHz, the experimental error greatly increases with increasing frequency, in the range 1 to 10 MHz.

The experimental error in the determination of the real part ε' is mainly determined by the geometry of

the sample condenser (thickness and diameter, including the influence of the spacer). The absolute accuracy for the real part ε' of the dielectric function is estimated to be 1%.

To study the molecular alignment of all samples under consideration, an a.c. electric field with frequency 50 Hz was applied for 1 min to the sample via a switch. At this frequency, the molecular dipoles are able to follow the a.c. electric field, and therefore the domains, formed by the silica particles network, tend to orient the director of the molecules perpendicular to the plane of the electrodes. The sample was controlled so that the maximum current (at 150 V) was $100 \mu\text{A}$, corresponding to a maximum of 15 mW of dissipated power in the sample, which does not affect the temperature of the sample considerably, since it is placed between stainless steel electrodes.

3. Results and discussion

In figures 2 and 3 the dielectric spectrum (up to 10 MHz) of the real ε' and imaginary ε'' parts of the complex permittivity ε^* in the temperature range 295–328 K are shown for the unfilled 5CB NLC sample.

From figure (2), in the nematic phase at $T=295$ K, ε' has a constant value of 14 in the frequency range 10^2 to 10^5 Hz. This value exceeds the corresponding value of $\varepsilon' = 10$ measured in the isotropic phase at $T=328$ K. The latter value is in agreement with previous measurements reported [22]. This means that the domains are oriented in the nematic phase with the director perpendicular to the electrode. According to the literature [22], the alignment of the domains in the nematic phase depends on the interaction with the surface of the

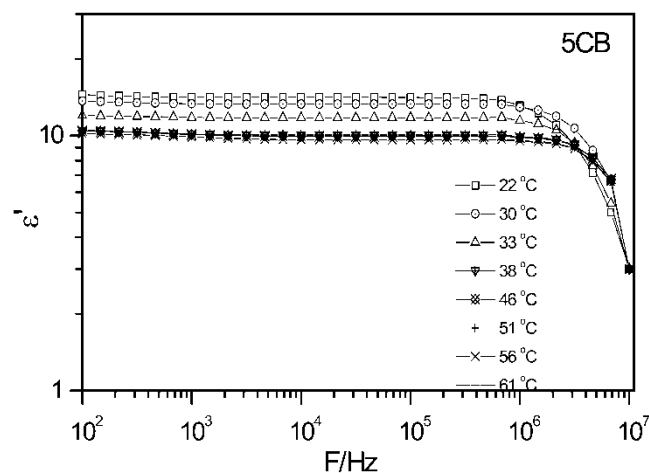


Figure 2. The real part ε' of the complex dielectric permittivity for pure 5CB in the nematic and isotropic phases within the frequency range 10^2 – 10^7 Hz.

substrate (here, the stainless steel electrodes) and may be changed by an external electric field.

The relation between $\log \epsilon''$ versus $\log f$ (Hz) at different temperatures in the nematic and isotropic phases is shown in figure 3, where one relaxation process is observed in the frequency range 10^2 – 10^7 Hz. It can be seen that the absorption process, which is assigned to rotational fluctuation of molecules around their short axes [17], changes at the phase transition temperature. Obviously, the relaxation rate f_p is shifted slightly towards higher frequency as the temperature increases. Here the dynamic behaviour of the liquid crystal is represented by the Cole-Cole type relaxation function given by [17]:

$$\epsilon^* - \epsilon_\infty = \frac{\Delta\epsilon}{[1 + (i\omega\tau)^{1-\alpha}]} \quad (5)$$

where ω is the characteristic angular frequency, and $\tau = 1/2\pi f_p$, ϵ_∞ describes the values of the real part ϵ' towards the high frequency, $\Delta\epsilon$ is the dielectric strength, α is a fractional form parameter ($\alpha < 1$) describing the deviation of this relaxation from ideal Debye behaviour. Dielectric strength $\Delta\epsilon$ is proportional to the square of the effective dipole moment and to the number of relaxation dipoles. Cole–Cole representation of permittivity ϵ' against dielectric loss ϵ'' in liquid crystal and isotropic state were shown generally in the form of Cole–Cole plots. Figures 4(a) and 4(b) show Cole–Cole plots for the 5CB pure sample in the nematic phase at $T=295$ K and in the isotropic phase at $T=328$ K. From figure 4, the values of α , and $\Delta\epsilon$ are extracted for all the samples under consideration and collected in the table. In comparison, the increment $\Delta\epsilon$ in the nematic phase is found to be 11.1. This value

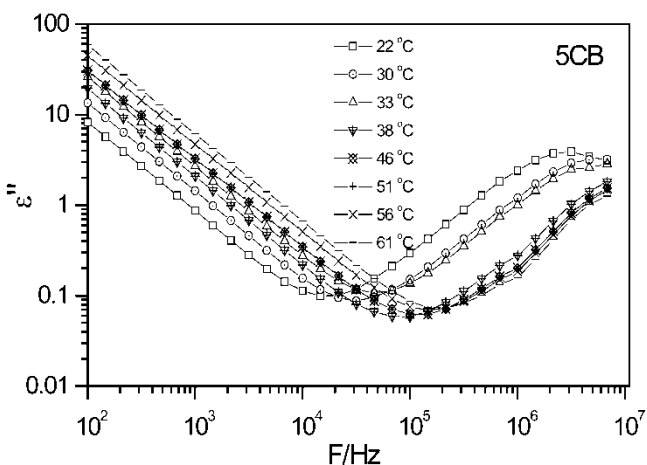


Figure 3. The imaginary part ϵ'' of the complex dielectric permittivity for pure 5CB in the nematic and isotropic phases within the frequency range 10^2 – 10^7 Hz.

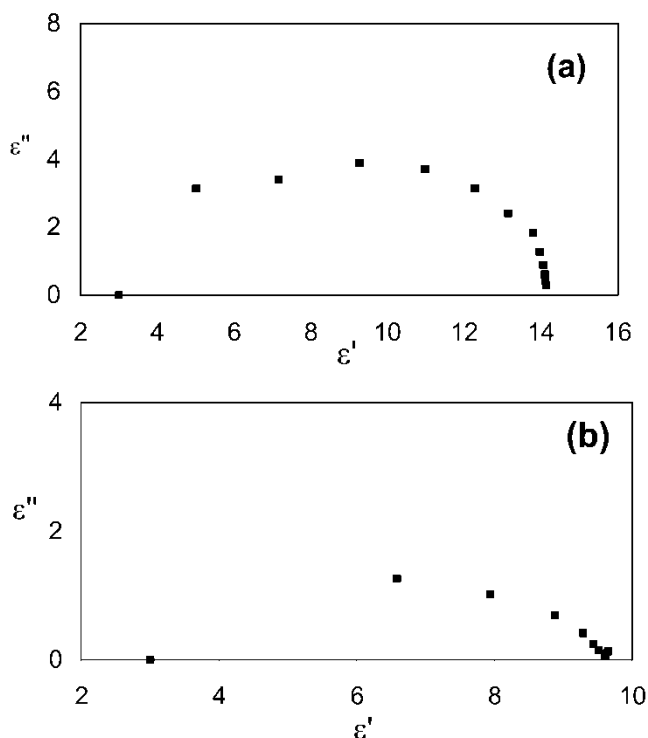


Figure 4. The Cole–Cole plot for the pure 5CB in: (a) the nematic phase, (b) the isotropic phase.

exceeds the corresponding value extracted in the isotropic phase by 40%. This is mainly due to the alignment of the strong dipole moment connected with the (–CN) cyano group present in 5CB molecules.

The results of the dielectric measurements of the real part ϵ' and imaginary part ϵ'' of the complex permittivity of the 5CB samples filled with silica particles with concentrations 2, 3, 5, 10 and 15 wt% are illustrated in figure 5(a–e) and 6(a–e), respectively, as a function of frequency in the range 10^2 – 10^7 Hz. From figure 5, it is seen that in the whole spectral range 10^2 – 10^7 Hz, the value of ϵ' in the nematic phase ($T=295$ K) is less than the corresponding value obtained in the isotropic phase ($T=328$ K). For

Table. The value of ϵ' at 10^5 Hz, the dielectric strength $\Delta\epsilon$, the form parameter α for all samples in the nematic ($T=295$ K) and isotropic ($T=328$ K) phases.

| Sample | Nematic Phase | | | Isotropic phase | | |
|-----------------------------|---------------|------------------|----------|-----------------|------------------|----------|
| | ϵ' | $\Delta\epsilon$ | α | ϵ' | $\Delta\epsilon$ | α |
| 5CB | 14.10 | 11.10 | 0.28 | 9.60 | 6.60 | 0.60 |
| 5CB+2 wt% SiO ₂ | 6.84 | 3.84 | 0.53 | 9.68 | 6.68 | 0.77 |
| 5CB+3 wt% SiO ₂ | 7.26 | 4.26 | 0.46 | 9.47 | 6.47 | 0.75 |
| 5CB+5 wt% SiO ₂ | 7.50 | 4.50 | 0.42 | 9.49 | 6.49 | 0.72 |
| 5CB+10 wt% SiO ₂ | 7.75 | 4.75 | 0.50 | 9.37 | 6.37 | 0.75 |
| 5CB+15 wt% SiO ₂ | 7.40 | 4.40 | 0.36 | 8.83 | 5.83 | 0.72 |

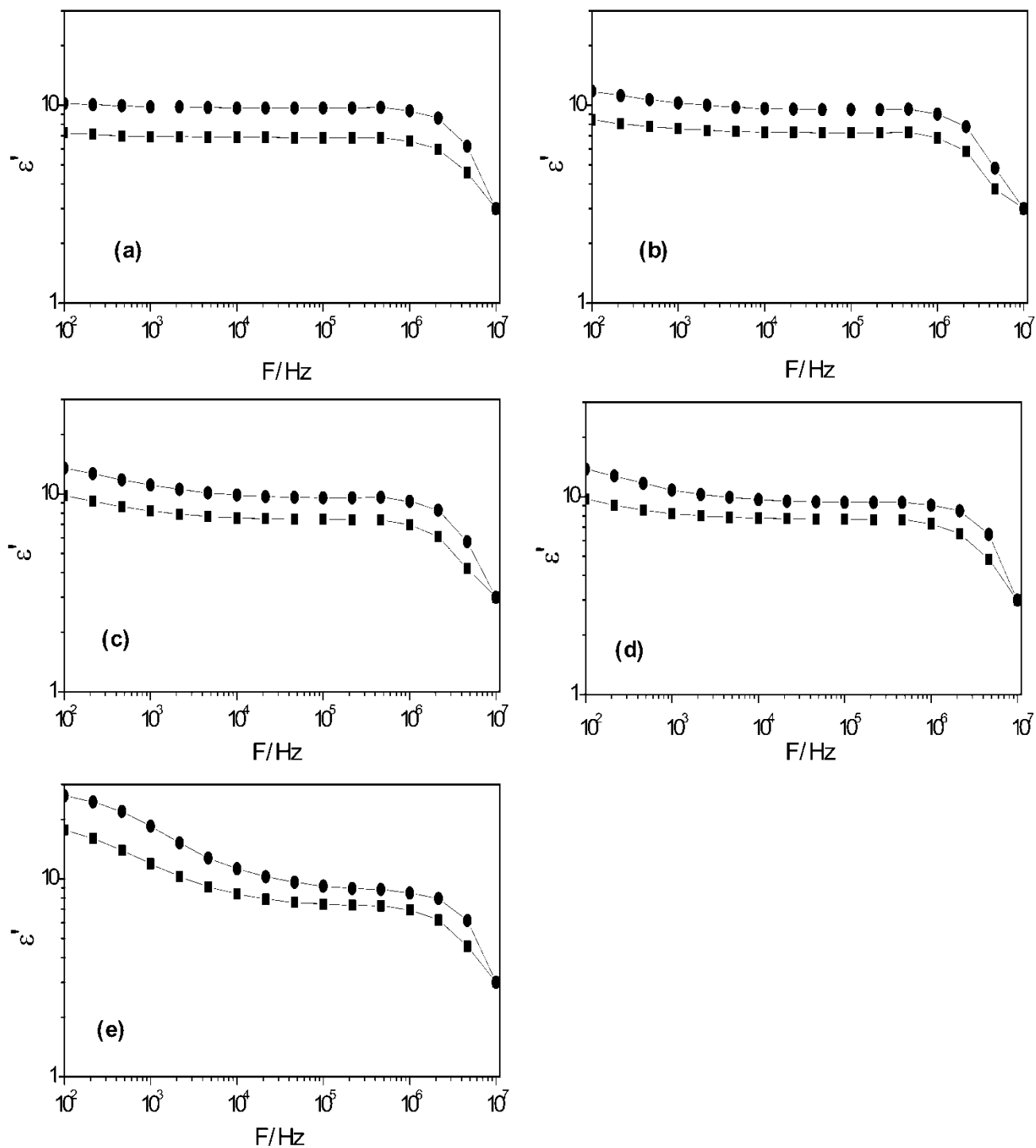


Figure 5. The real part ϵ' of the complex dielectric permittivity for the 5CB Samples containing: (a) 2, (b) 3, (c) 5, (d) 10, (e) 15 wt% silica, in the nematic (\blacksquare) and isotropic (\bullet) phases.

example, the value of ϵ' is found to be 8 for the sample containing 2 wt% silica in the nematic phase. This value is less than the corresponding value obtained for the unfilled sample as discussed above. As shown in figure 5, the value of ϵ' in the nematic phase decreases

with increasing filler content. This behaviour is attributed to the influence of the filler particles on the orientation order of the domains in the nematic phase. This means that the molecules at the surface of the filler are not free to rotate.

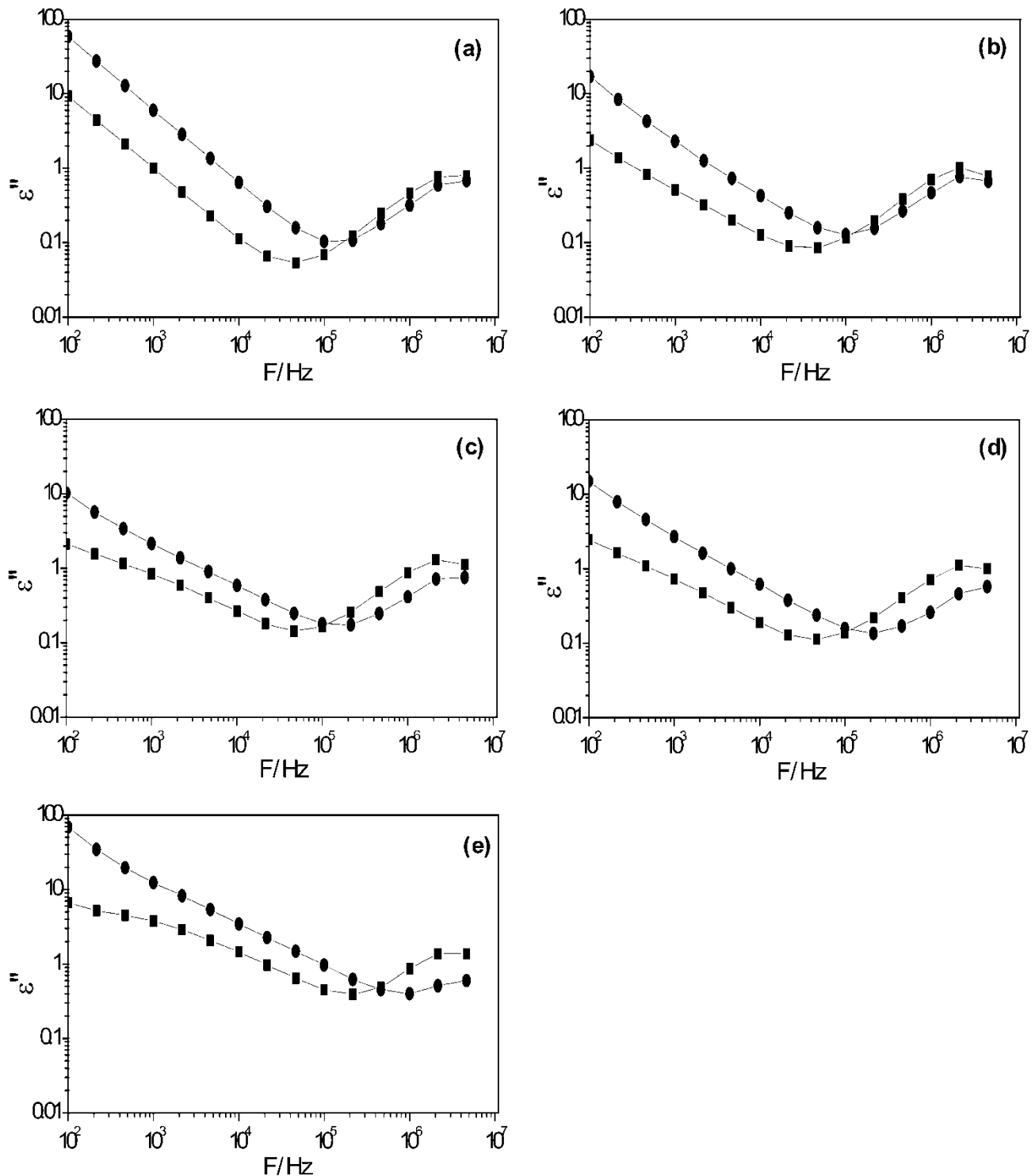


Figure 6. The imaginary part ϵ'' of the complex dielectric permittivity for 5CB samples containing: (a) 2, (b) 3, (c) 5 (d) 10, (e) 15 wt% silica, in the nematic (■) and isotropic (●) phases.

In all filled samples the high value of ϵ'' at frequencies lower than 10^4 Hz is assigned to the motion of the free charge carriers. These may be due to ionic impurities of the 5CB liquid crystal. Furthermore, in this low

frequency range we observed different characteristics for different filler contents, where the slope of the dielectric loss decreases on increasing the amount of the filler. These changes are attributed to the interfaces

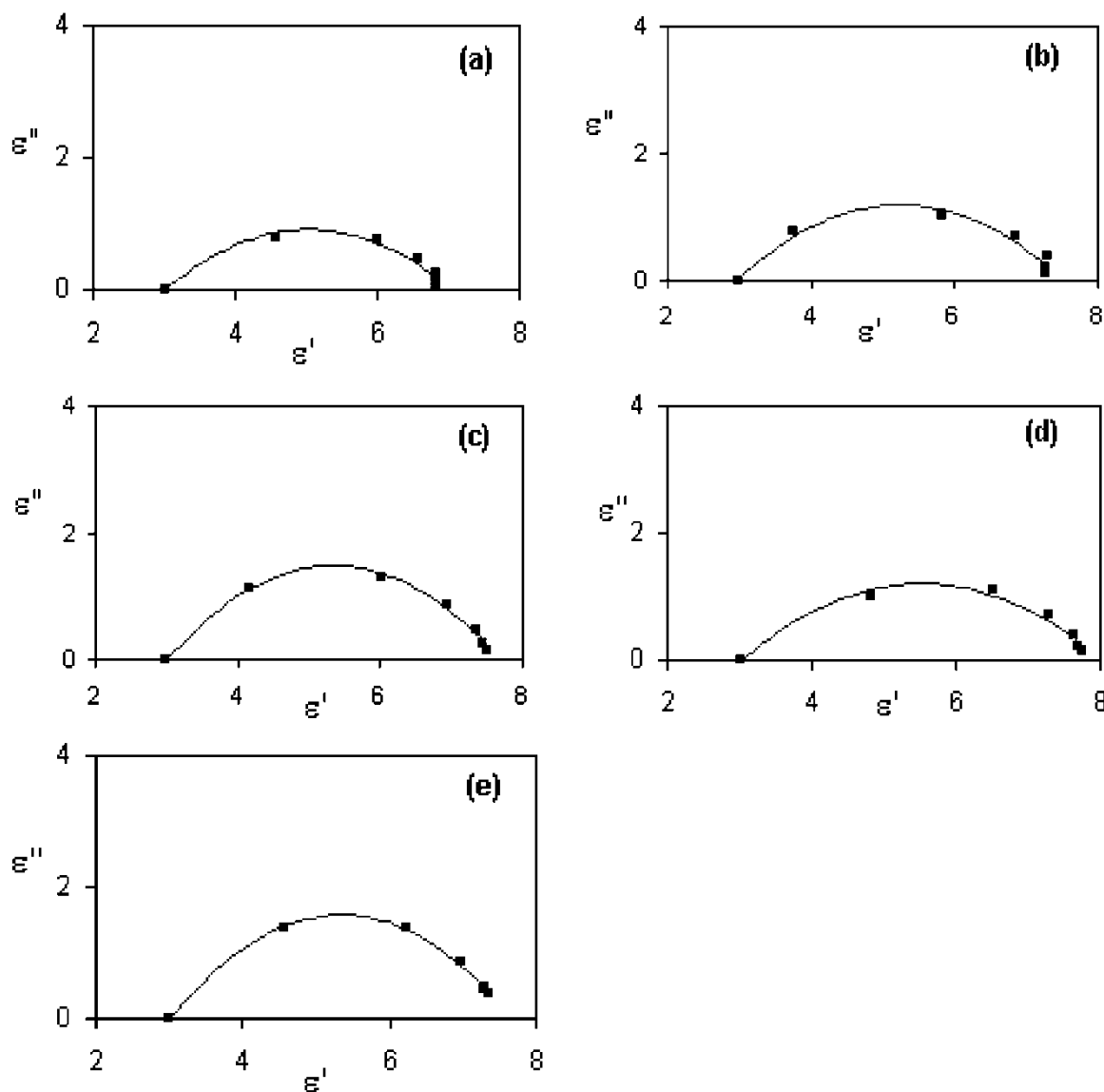


Figure 7. The Cole-Cole plot in the nematic phase for samples containing: (a) 2, (b) 3, (c) 5, (d) 10, (e) 15 wt% silica.

between the filler and the liquid crystal material, where the motion of the free charge carrier is hindered. Similar results are reported for measurement of the orientational order and mobility of the nematic liquid crystal in random nanometer confinement [16–20].

The characteristic curves of all the sample illustrated in figure 6(a–e) for the dielectric loss ϵ'' of the filled samples are similar to the characteristic curves obtained for the unfilled 5CB samples, figure 3. In both the nematic ($T=295$ K) and isotropic ($T=328$ K) phases one relaxation process has been identified in the frequency range 10^2 – 10^7 Hz. Cole-Cole plots of all filled samples (which illustrate the relaxation between ϵ'' versus ϵ') are shown in figure 7(a–e) and figure 8(a–e)

for nematic ($T=295$ K) and isotropic ($T=328$ K), respectively. From figures 7 and 8, the parameter α and the dielectric strength $\Delta\epsilon$ is extracted for all samples. The results are presented in the table for both nematic and isotropic phases.

The activation energy E_a for the observed relaxation process was determined by the Arrhenius equation [23]

$$\tau = \tau_0 \exp(E_a/RT) \quad (6)$$

where τ_0 is the value of the relaxation time at infinite temperature ($= 3.07 \times 10^{-21}$ s) and R is the gas constant equal to $8.3 \text{ J mol}^{-1} \text{ K}^{-1}$. From figure 3 we extracted an activation energy of value 75 kJ mol^{-1} for the pure 5CB sample in the nematic phase. This value is compared with the corresponding value of 65 kJ mol^{-1} determined

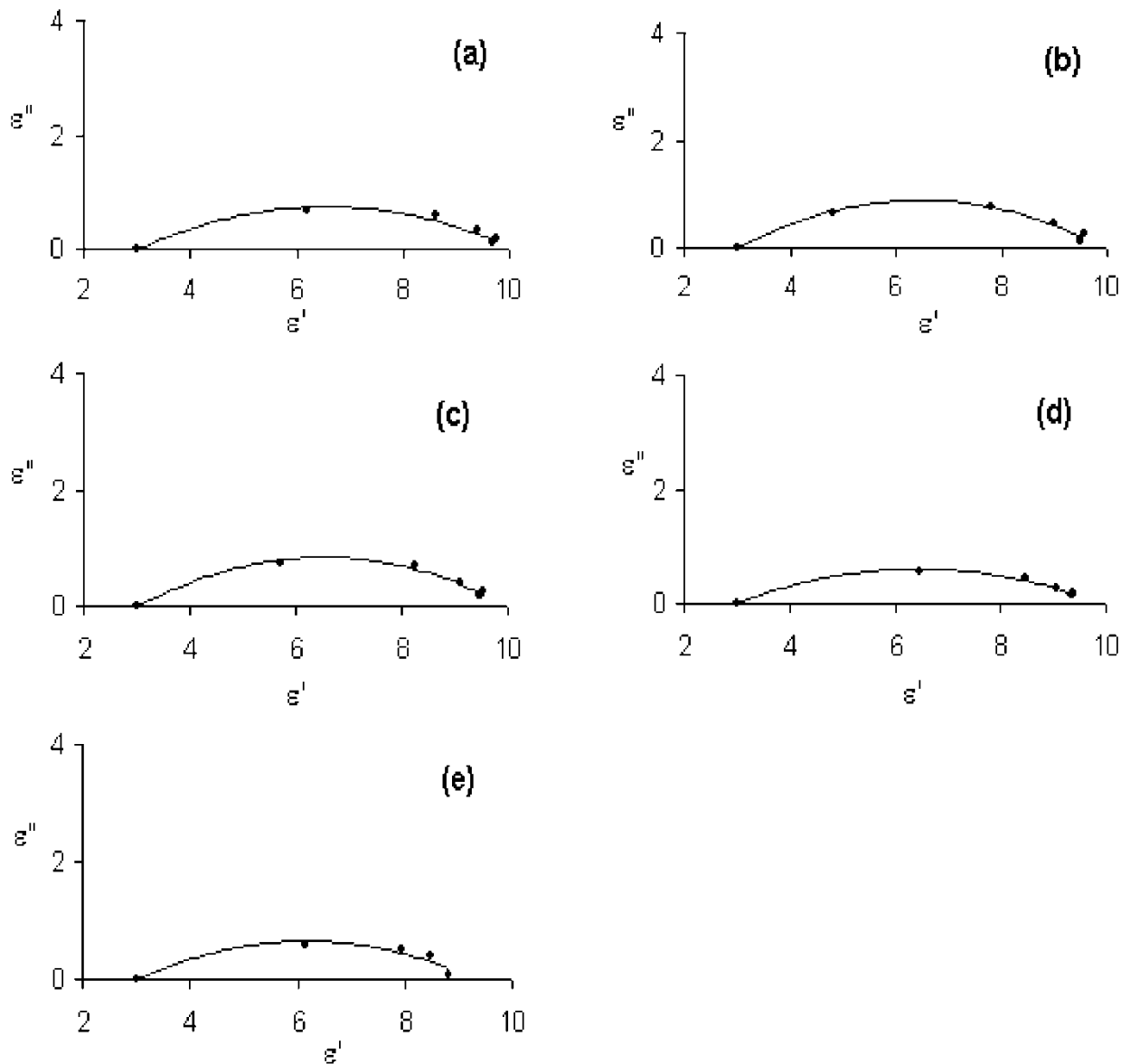


Figure 8. The Cole–Cole plot in the isotropic phase for samples containing: (a) 2, (b) 3, (c) 5, (d) 10, (e) 15 wt% silica.

by Rozanski *et al.* [16]. For the filled sample we determined activation energy of 50 kJ mol^{-1} for the 5 wt% silica-filled 5CB sample. This result is compared with 23 kJ mol^{-1} , obtained by Sinhal and Aliev [17], for a 5CB sample in a 1000 \AA porous media. In the isotropic phase the activation energy is found to be 20 kJ mol^{-1} . This is lower than the corresponding value obtained for the unfilled sample. The difference in the activation energies from nematic to isotropic reflects the role of molecular rotational dynamics. A decrease of the activation energy from nematic to isotropic phase has also been reported [17, 24].

The effect of an applied electric field on the

molecular alignment of silica-filled 5CB nematic liquid crystal has been studied. It is worth mentioning that for the unfilled 5CB sample the molecules are aligned homotropically in the nematic phase (see figure 2) on the untreated metal electrodes. In this case the value of the ϵ' is higher than the corresponding value in the isotropic phase.

The value of the real part ϵ' of the dielectric permittivity of the samples under consideration is measured at frequency 10^5 Hz in both nematic and isotropic phases. This value is used to probe the molecular alignments and the breaking of the molecular ordering. The value of ϵ' is measured before

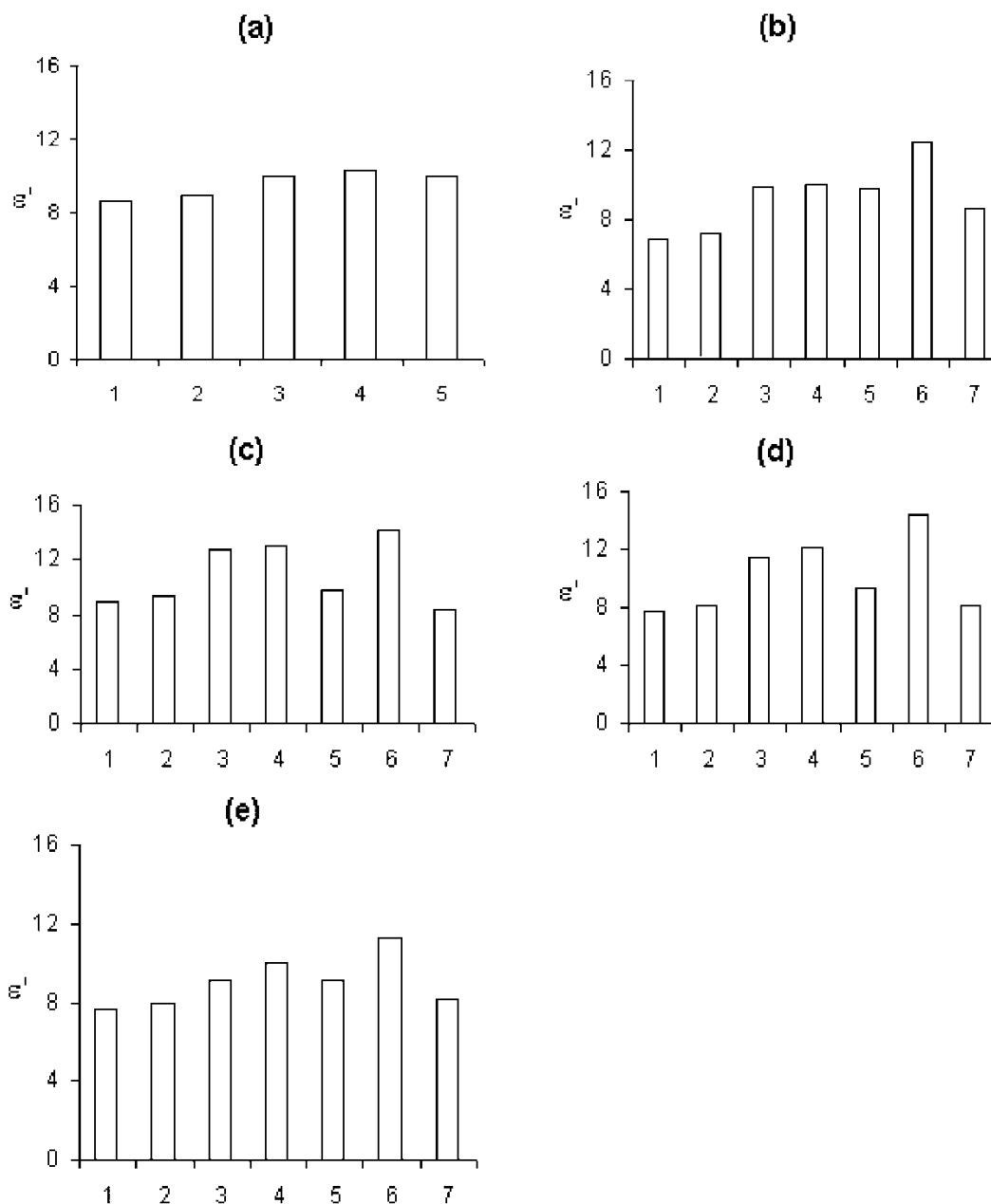


Figure 9. The values of ϵ' under the influence of an applied electric field for samples containing (a) 0, (b) 3, (c) 5, (d) 10, (e) 15 wt% silica, where the values of the applied electric field strength in the nematic phase are: (1) 0, (2) 0.04, (3) 0.2, (4) 1.2, (5) $0 \text{ V } \mu\text{m}^{-1}$ (in the isotropic phase); (6) in order to obtain alignment the sample was cooled gradually with a cooling rate 0.5 K min^{-1} while an electric field of strength $1.2 \text{ V } \mu\text{m}^{-1}$ was applied—after reaching room temperature at $T=295 \text{ K}$, the field was removed; (7) after applying a small displacement.

applying the external electric field in the nematic phase at $T=295 \text{ K}$. Afterwards, an external electric field is applied to each sample. In this experiment an alternating electric field with frequency 50 Hz is used. The advantage of the a.c. electric field is that no chemical reaction at the electrode takes place. Nevertheless the dipole moment of the molecules can follow the direction of the applied field, since the relaxation

frequency is greater than 10^5 Hz . After 1 min the field is switched off and the value of ϵ' is remeasured.

The results are presented in the histograms given in figure 9(a–e) for the 5CB nematic liquid crystal sample and samples filled with 3, 5, 10 and 15 wt% of silica. It is obvious from the histogram shown in figure 9(c), for the 5CB sample filled with 5 wt% silica, that the value of the real part of the permittivity increased after

applying the electric field. This is because the filled sample at the beginning of the experiment does not show homeotropic orientation with respect to the metal electrode. After application of an electric field of strength $0.2 \text{ V } \mu\text{m}^{-1}$ homeotropic alignment of the molecules is established. The value of ϵ' reaches a saturation, with no further increase at higher fields. After heating to the isotropic phase at $T=328 \text{ K}$, the value of ϵ' again dropped to about 10, as measured before in the isotropic state. In order to obtain alignment the sample was cooled gradually with at 0.5 K min^{-1} while an electric field of strength $1.2 \text{ V } \mu\text{m}^{-1}$ was applied. After reaching room temperature at $T=295 \text{ K}$, the field was removed and a value of 14.1 for ϵ' measured.

Considering the technical applications of this effect, we found that by a small displacement of the stainless steel electrode the alignment of the molecules is destroyed. For this experiment the position of the upper electrode was shifted slightly back and forth. We found that it does not matter how much and how often this displacement was applied. Thus it is sufficient to change the arrangement of the filler particles inside the liquid crystal matrix to destroy the alignment.

From our results we found that the value of ϵ' is strongly dependent on the amount of silica. For example 3 wt% silica is not sufficient to stabilize the orientation of the 5CB molecules after applying the electric field in comparison with the samples containing 5 and 10 wt% silica; on the other hand, very high filler content reduces the value of ϵ' as shown in figure 9(e).

At this point we may state that the molecular alignment of the filled samples is controlled by the surface of the filler particle while in the unfilled sample it is controlled by the metal surface of the electrodes. This feature opens up new fields of applications such as developing a liquid crystal–solid particles interface that makes the role of the liquid crystal surface layers important in the determination of the material properties.

4. Conclusion

The dielectric properties of nematic liquid crystal 5CB filled with silica particles of size 30–80 nm have been measured in the frequency range 10^2 to 10^7 Hz at different temperatures in the nematic and isotropic phases. It has been shown that the inorganic filler particles provide a convenient tool for the orientation of the molecules in a dielectric experiment. In this frequency range one relaxation process have been observed. The relaxation frequency is slightly shifted toward higher frequency by increasing the content of the filler in the sample. Activation energies of 75 and 50 kJ mol^{-1} are obtained for the bulk 5CB and the 5 wt% silica-filled sample, respectively. The director

alignment of the molecules was found to be sensitive to the strength of the applied electric field, the silica content in the samples, and the applied displacement.

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