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An electro-rheological study of the nematic liquid crystal 4-*n*-heptyl-4′-cyanobiphenyl

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An experimental and theoretical study of the electro-rheological effects observed in the nematic phase of 4-*n*-heptyl-4′-cyanobiphenyl has been conducted. This liquid crystal appears to be a model system, in which the observed rheological behaviour can be interpreted by the Leslie–Ericksen continuum theory for low molecular weight liquid crystals. Flow curves are illustrated at different temperatures and under the influence of an external electric field ranging from 0 to 3 kV mm^{-1}, applied perpendicular to the direction of flow. Also presented is the apparent viscosity as a function of temperature, over similar values of electric field, obtained at different shear rates. A master flow curve has been constructed for each temperature by dividing the shear rate by the square of the electric field and multiplying by the square of a reference value of electric field. In a log–log plot, two Newtonian plateaux are found to appear at low and high shear rates, connected by a shear-thinning region. We have applied the Leslie–Ericksen continuum theory, in which the director alignment angle is a function of the electric field and the flow field boundary conditions are neglected, to determine viscoelastic parameters and the dielectric anisotropy.

Keywords: electrorheology; liquid crystals; 7CB; continuum theory

1. Introduction

The electro-rheological (ER) effect can be considered as the change in the apparent viscosity of a fluid in the presence of an external electric field. This can sometimes have dramatic consequences, inducing fluid solidification [1, 2]. The ER effect is an important phenomenon, leading to a broad spectrum of new technological applications, ranging from electro-optical devices to automobile technology – for example, shock absorbers derived from tunable vibration damping systems.

The first observation of the ER effect was reported by Winslow [3], for a suspension of polarisable microparticles in water. In this case the variation in apparent viscosity was caused by the alignment of the microparticles, either in chains or columnar structures oriented in the direction of the electric field, increasing the viscosity by one order of magnitude.

Liquid crystalline (LC) materials are known to exhibit an ER effect, since they are susceptible to orientation by external electric and magnetic fields in their anisotropic phases [4].

Over the past two decades a number of research publications have considered the experimental characterisation of the ER effect in various LC systems, and some of these have proposed a theoretical quantitative interpretation of the results.

The ER effect has been studied for lyotropic LC polymers such as poly(*n*-hexyl isocyanate) in *p*-xylene [5], for poly(γ-benzyl-L-glutamate) (PBLG) in 1,4-dioxane [6, 7] and for acetoxypropylcellulose (APC) in dimethylacetamide (DMAc) [7]. A significant increase in viscosity, by a factor of four for an electric field of 2 kV mm^{-1}, was observed for solutions of PBLG in 1,4-dioxane. On the other hand, a decrease in viscosity was seen with solutions of APC in DMAc, since this system has negative dielectric susceptibility.

The ER effect has also been reported in thermotropic LC compounds, such as polysiloxane-based LC polymers [8–10]. In this case a significant increase in viscosity (about one order of magnitude) has been observed, caused by the orientational effect of mesogenic side-chain groups attached to the polymeric backbone.

A number of low molecular weight LC systems have also been studied, principally by Negita [11–14]. Starting with the liquid crystal, *N*-(4-methoxybenzylidene)-4-butylaniline (MBBA), the ER effect was studied in the nematic phase. In this system a small decrease in viscosity was observed in response to the applied electric field, supported by the negative dielectric susceptibility of MBBA [11]. The influence of different LC phases – namely, smectic A and nematic phases – on the ER effect, was also considered in experiments performed with 4-*n*-octyl-4′-cyanobiphenyl (8CB) [13] and 4-*n*-octyloxy-4-cyanobiphenyl (80CB) [14]. From these studies it is clear that in the nematic phase the presence of an

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electric field causes a significant increase in viscosity, and the opposite effect is found for the smectic A phase. In these systems the electric field reorients the smectic layers and the nematic director, causing the ER effect. A deeper understanding of the ER effect in the nematic phase was found with 4-*n*-pentyl-4′-cyanobiphenyl (5CB), in which important features were clearly identified [12]:

- the ER effect – in this case, a viscosity increase in response to the electric field, observed in the nematic but not in the isotropic phase;
- the flow changes its character from Newtonian to non-Newtonian, and back to Newtonian, as the electric field is increased;
- the viscosity increase reaches a limit at sufficiently high electric fields; and
- the ER effect is dependent on both shear rate and temperature.

It was also established by Negita that in LC systems the ER effect in the nematic phase is caused by the reorientation of the nematic director, which in turn is determined by the balance between the flow field and the electric field. When the applied electric field is perpendicular to the direction of flow, the apparent viscosity increases when the system has a positive dielectric anisotropy (meaning the molecules preferentially align along the electric field). When the dielectric anisotropy of the system is negative, however, the opposite behaviour is induced, and the apparent viscosity decreases.

The physical properties of the liquid crystal, 4-*n*-heptyl-4′-cyanobiphenyl (7CB), have been reported previously in the literature, involving a range of experimental techniques. The dielectric anisotropy (ϵ_a) has been determined experimentally in a number of studies [15–19]. The rotational viscosity (γ_1), also known as twist viscosity, has also been determined experimentally by electric field-dependent dynamic light scattering [19], and by measurement of the Zvetkow twist viscosity coefficient [20]. Finally, in the work of Liu and Jamieson [19], a combination of a number of experimental techniques (Freedericksz transition, rheology, electro-rheology and electric field-dependent dynamic light scattering) has allowed the determination of Miesowicz viscosity and the Leslie viscosity coefficients, α_1, α_2 and α_3. From the values obtained for these parameters it can be concluded that the LC, 7CB, is a flow-aligning material.

In the present article, we present an experimental characterisation of the apparent viscosity of the liquid crystal, 7CB, as a function of shear rate, temperature and applied electric field, using electro-rheology. To estimate the physical parameters of the system, and to interpret the behaviour observed, we have applied the Leslie–Ericksen (L–E) continuum theory, which is valid for low molecular weight nematic liquid crystals [21, 22].

It is our belief that a *model* system has been identified, in that all the experimental rheological behaviour is accounted for by the L–E theory. In particular, we show that flow curves obtained for different intensity values, with and without the application of an electric field, describes the total expected behaviour by construction of a viscosity master curve. Such a master curve has not been reported previously.

We also demonstrate the dependence of the apparent viscosity on temperature, but we have observed that this behaviour is continuous, even close to the nematic–isotropic transition temperature (T_{NI}), and is independent of the shear rate and applied electric field. As expected, the ER effect vanishes above T_{NI}, where the nematic order is no longer defined.

2. Experimental

The liquid crystal used in this study, 7CB, is known commercially as K21 (Merck), and its molecular structure is illustrated in Figure 1. It exhibits a nematic phase between 30 and 42.8°C.

The electro-rheological properties of 7CB were determined using a Bohlin Gemini HRnano rotational rheometer coupled to a Bohlin electro-rheological cell. Rotating parallel plates were used, 40 mm in diameter, with a gap of 500 μm. After temperature equilibration and before starting the measurements, the samples were subjected to a pre-shearing stage, at 10 s^{-1} for 60 s, followed by an equilibration period of 120 s.

The apparent viscosity as a function of shear rate was determined at different temperatures in the nematic phase, with and without the application of an external electric field perpendicular to the direction of flow, for electrical fields in the range 0–3 kV mm^{-1}. Also measured was the apparent viscosity as a function of temperature, covering the nematic and the isotropic phase, at different shear rates and a range of values of electric field. The electric field was generated by applying a high voltage to the gap (500 μm)

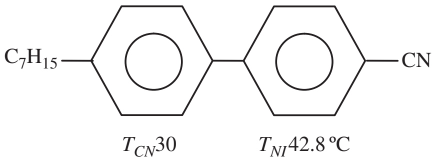


Figure 1. Molecular formula for 4-*n*-heptyl-4′-cyanobiphenyl (7CB) and its transition temperatures.

between the plates using a 50 Hz AC power source (Spellman SL150).

3. Experimental results

3.1 Flow curves – ER effect

Figure 2 presents the flow curves of 7CB for different electric fields applied, for example, at $T = 32$ and 36°C. Curves for other temperatures were also produced and showed similar trends, even though differences in the absolute values of the apparent viscosity were observed.

Examination of Figure 2 shows that in the absence of an electric field the LC presented a Newtonian behaviour over the shear rate range concerned. The application of an electric field increased the apparent viscosity at low and intermediate shear rates, but at higher shear rates the curves obtained when the sample was under an electric field tended to converge with the curve obtained without the applied electric field, unless the electric fields were sufficiently strong, when no convergence was observed.

The shape of the flow curve is due to competition between the electric and flow fields. In fact, the molecular orientation of a liquid crystal of positive dielectric anisotropy, which is the case with 7CB, will follow the direction of the applied electric field. As shown in Figure 3, when the electric field is perpendicular to the flow field the molecules will reorient themselves in the direction of the electric field, or perpendicular to the flow field, as long as the flow field is not strong enough to avoid it, thus increasing the apparent viscosity of the sample. At high flow field strengths, which means high shear rates, the flow field is known to orient the molecules of a liquid crystal in a direction that makes a relatively small angle with the flow field, known as the flow alignment angle, θ_0 [2]. This is in fact what took place with our sample. At sufficiently high flow field strengths the flow field became dominant over the electric field, and the molecules were oriented in such a direction that the apparent viscosity decreased and all the curves tended to the curve obtained in the absence of an applied electric field. Naturally, the shear rate for which a strong decrease in apparent viscosity was observed depended on the electric field strength, meaning that at higher electric fields, higher shear rates were necessary to overcome the effect of the electric field. In fact, for sufficiently high electric fields it seemed that the flow field was no longer able to compete with the electric field, and no significant decrease in the apparent viscosity was therefore observed, at least within the range of shear rates considered. This fact has previously been reported by Negita [12] and Liu and Jamieson [19], for different but related systems. At intermediate shear rates there is competition between the two fields, and the molecules become oriented at an intermediate angle θ ($\pi/2 < \theta < \theta_0$) with the flow field, the angle increasing with electric field strength.

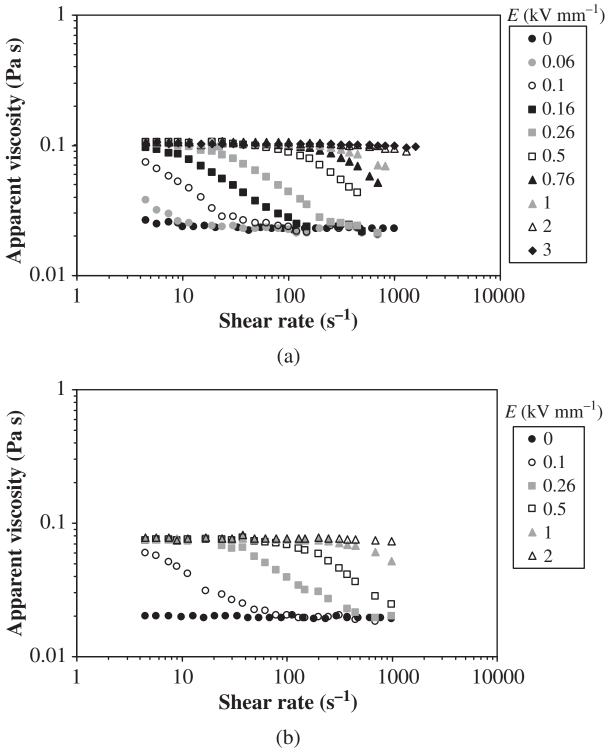


Figure 2. Flow curves (apparent viscosity *vs* shear rate) for different values of electric field at (a) 32°C and (b) 36°C.

Figure 4 illustrates the dependence of the apparent viscosity on electric field strength at low, intermediate and high shear ranges, at $T = 32$ and 36°C. A similar pattern is seen in the nematic range at other temperatures.

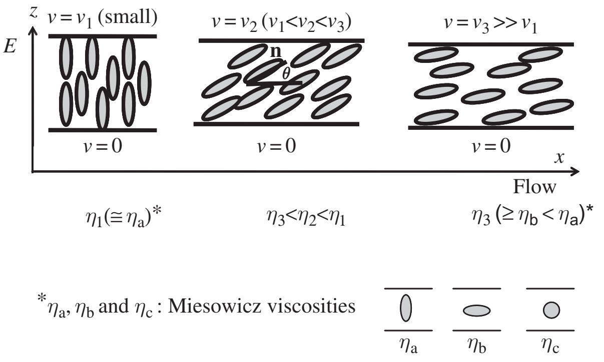
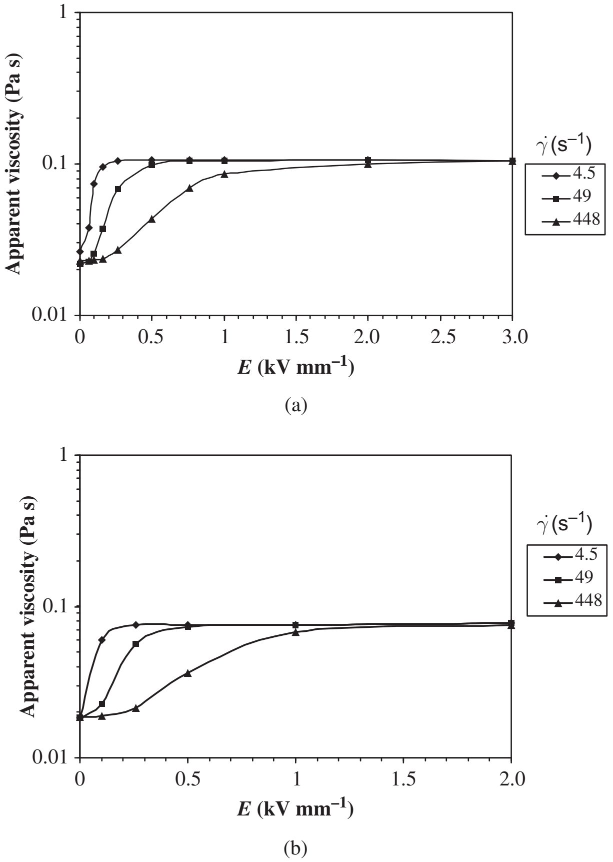


Figure 3. Schematic representation of molecular orientation at different flow field strengths.



(a)

(b)

Figure 4. Dependence of viscosity on electric field strength at three different shear rates, (a) at 32°C and (b) 36°C.

Figure 4 shows that the electric field value which saturates the viscosity increases with increasing shear rate.

3.2 *Flow master curve*

Figure 5 presents a master curve of the apparent viscosity *vs* shear rate at $T = 32°C$. This was obtained by dividing the shear rate by the square of the electric field and multiplying it by the square of a reference electric field. An equivalent master curve was obtained for each temperature studied in the nematic range.

3.3 *Apparent viscosity* vs *temperature (the ER effect)*

Figure 6 shows the influence of temperature on the apparent viscosity of 7CB, with and without applied electric fields up to 3 kV mm^{-1}, at shear rates of 10 s^{-1} and 100 s^{-1}. Similar curves were obtained at shear rates of 50 s^{-1} and 500 s^{-1}, apart from the absolute values of the apparent viscosity.

Figure 6 shows that the isotropic phase is not influenced by the electric field, which is also as expected.

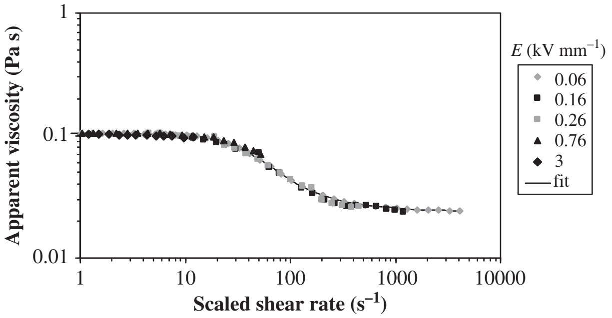
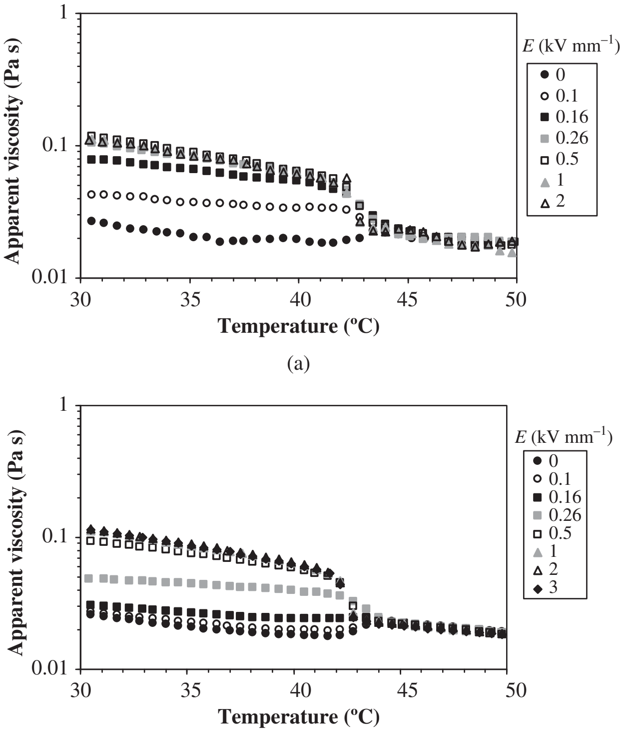


Figure 5. Master curve of the apparent viscosity *vs* shear rate at 32°C. The normalisation was performed by dividing the shear rate by the square of the electric field and multiplying by the square of the reference value of 0.26 kV mm^{-1}. The fit was calculated using the parameters given in Table 1.



(a)

(b)

Figure 6. Apparent viscosity as a function of temperature (at different values of the electric field) for shear rate values of (a) 10 s^{-1} and (b) 100 s^{-1}.

This is the reason why all the curves converge into a single curve at 43 °C, the curve without applied electric field. In the isotropic region the curve shows a slight decrease in apparent viscosity with increasing temperature.

We also observed a slight decrease in the apparent viscosity with increase in temperature in the nematic phase, distant from the clarification temperature. In the vicinity of the nematic–isotropic transition temperature, however, a different behaviour was observed, depending on the value of the electric field. Without an electric field, or at very low values of the electric field, a sudden increase in the apparent viscosity was observed at the transition, as is usual with liquid crystals. This is due to the fact that in the nematic phase the molecules are oriented in a manner (characterised by a positive order parameter) which favours the flow, whereas in the isotropic phase the order parameter is zero. However, at higher values of the applied electric field, and where there is already a sufficiently high apparent viscosity, this behaviour becomes completely different and, at the transition from the nematic to the isotropic phase, a decrease in apparent viscosity is observed. Once again, it is competition between the electric and flow fields that determines the behaviour, and the temperature at which the slope of the $\eta(T)$ curve increases significantly depends on the electric field strength.

4. Theoretical results

The Leslie–Ericksen theory describes the dynamic behaviour of a low-molecular nematic LC. It generalises the Navier–Stokes theory of simple fluids, coupling the velocity field of the molecules, $\boldsymbol{v}$, with their average orientation, which is given by the nematic director field, $\boldsymbol{n}$.

In the present study the rheometer performed a simple horizontal Couette flow, $\mathbf{v} = (v, 0, 0)$, with an applied perpendicular electric field, $\mathbf{E} = (0, 0, E)$ (see Figure 3). The thickness of the cell was large enough to allow the effect of the nematic anchoring conditions at the surfaces to be neglected. It was verified that the wall effect becomes significant only for gaps of less than 50 μm. The bulk orientation of the nematic phase is then constant, $\mathbf{n} = (\cos\theta, 0, \sin\theta)$, independent of space and time, and the velocity gradient tensor will have only one component, the constant shear rate, $\dot{\gamma} = \partial_z v_x$. With these assumptions, the dynamic equations lead only to one condition, Equation (1), already obtained in previous studies [for example, 11, 24],

$$(\alpha_3 \cos^2\theta - \alpha_2 \sin^2\theta)\dot{\gamma} - \frac{1}{2}\epsilon_a E^2 \sin 2\theta = 0, \quad (1)$$

in which α_2 and α_3 are two of the six L–E viscosity coefficients ($\alpha_1, \ldots, \alpha_6$) although only 5 are independent. The tendency of the molecules to align parallel (or perpendicular) to the electric field, $\mathbf{E}$, is given by the positive (or negative) sign of the dielectric anisotropy, ϵ_a. Equation (1) determines the value of the angle θ as a function of $y = \alpha_2\dot{\gamma}/(\epsilon_a E^2)$, for a given ratio of fluid viscosities, α_3/α_2. This demonstrates why all the flow curves, for different applied electric fields, may be plotted on the same master curve, only redefining the scale of the shear rate by $\dot{\gamma}E_{\mathrm{ref}}^2/E^2$, where E_{ref} is a reference electric field (see Figure 5).

The apparent viscosity can be obtained from the stress tensor, $\eta = \sigma_{zx}/\dot{\gamma}$, and depends on the angle θ and the L–E viscosity coefficients of the fluid:

$$\eta = \eta_b + \alpha_1 \sin^2\theta \cos^2\theta - (\alpha_2 + \alpha_3)\sin^2\theta, \quad (2)$$

where $\eta_b = (\alpha_3 + \alpha_4 + \alpha_6)/2$ corresponds to a Miesowicz viscosity (see Figure 3).

Equation (1) may be solved analytically, yielding a somewhat complicated expression for the viscosity. However, some insight may be gained if we explore some limiting situations. For sufficiently large E, the solution of Equation (1) is $\theta_{\max} = \pi/2$, and the director will be parallel to the electric field if $\epsilon_a > 0$. In this case, the apparent viscosity, $\eta_{\max} = \eta_b - (\alpha_2 + \alpha_3) = \eta_a$. When no electric field is applied, the solution of Equation (1) becomes $\theta_{\min} = \arctan\sqrt{\alpha_3/\alpha_2}$, and the viscosity $\eta_{\min} = \eta_b + \alpha_1\alpha_2\alpha_3/(\alpha_2 + \alpha_3)^2 - \alpha_3$.

To understand the behaviour of the apparent viscosity for intermediate values of the applied electric fields and shear rates, it is theoretically useful to consider small values of α_3/α_2 and α_1/α_2, since α_2 is generally large and negative. Taking $\theta = -\arctan(1/y)$, the apparent viscosity yields

$$\eta(x = \ln y) \approx \eta_b + \frac{\alpha_1 e^{2x}}{(1 + e^{2x})^2} - \frac{\alpha_2}{1 + e^{2x}} + \frac{\alpha_3(1 + e^{2x} + 2e^{4x})}{(1 + e^{2x})^2}, \quad (3)$$

to linear order in α_1 and α_3. It is interesting to observe that Equation (3) corresponds exactly to a sigmoid if $\alpha_1 = \alpha_3 = 0$. Indeed, this function is well in line with the experimental data. Any deviation from this behaviour is thus caused by a non-zero situation, but probably small values of α_1 or α_3. The approximate sigmoid shape of the data allows an easy geometrical 4-parameter fit (4-fit), associated with the upper-leg, lower-leg, mid-point and breadth of change regions. For linear order in α_1 and α_3, we may write $\eta_{\min} = \eta_b - \alpha_3$, and the mid-point of the viscosity is given by

$$y^* = \alpha_2\dot{\gamma}^*/(\epsilon_a E^2) = 1 - \alpha_1/2\alpha_2, \quad (4)$$

and the slope of the viscosity at the mid-point (which is associated with the breadth of change region) is given by

$$\left.\frac{d\eta}{d(\ln\dot{\gamma})}\right|_{y^*} = \frac{\alpha_2 - \alpha_3}{2}. \quad (5)$$

From these relationships, we can estimate that $\alpha_2 = \eta_{\min} - \eta_{\max}$, α_3 (if the slope is sufficiently well defined), η_b, and the relationship between $\epsilon_a$and α_1. If α_1/α_2 is negligible, it becomes possible to estimate the dielectric anisotropy directly, $\epsilon_a \approx \alpha_2\dot{\gamma}^*/E^2$. For non-negligible values of α_1 this formula underestimates the dielectric anisotropy.

We have performed the geometrical 4-fit for the flow curves, η *vs* $\ln\dot{\gamma}$, at temperatures of 32, 34, 36 and 38°C and an applied field of 0.26 kV mm^{-1}, in which both plateaux can be observed and there is also a well defined mid-point slope. We have added to each curve a new point, corresponding to the value of the apparent viscosity without electric field, or alternatively for a very high shear rate. For comparison and confirmation of our first fits, we have used the Levenberg–Marquardt method [23] to fit the same flow curves, but without any approximations. For each value of $\dot{\gamma}$ we have obtained θ from Equation (1); the apparent viscosity was then calculated using Equation (2), with a total of five non-linear fitting parameters (5-fit): α_1, α_2, α_3, η_b, and ϵ_a (Table 1).

We have observed that our fits were very sensitive to experimental accuracy. The error associated with α_2, corresponding to a difference between minimum and maximum viscosities, may readily be estimated to ± 2 mPa s. We thus deduce that the errors in the other viscosity parameters, α_1, α_3 and η_b, will be larger. The dielectric anisotropy depends on α_2, $\dot{\gamma}$ and E^2. The last of these in our case has a large relative error. The error for ϵ_a is then estimated to be $\pm\, 0.5\, \epsilon_0$.

The values obtained for α_2 compare well with those reported elsewhere. For example, the work of Liu and Jamieson [19] indicates $\alpha_2 = -59.7$ mPa s ± 2% at 34°C. Also, their values at this temperature for α_1 and α_3 are 8.4 mPa s ± 6% and ≈0, respectively, which although different from those we obtained are of the same order of magnitude. A more significant discrepancy between our results and those in the literature exists in the value obtained for the dielectric anisotropy, around $6.8 \pm 0.5\, \epsilon_0$, in which our result is quite different from the values reported elsewhere [15–19], around 8–12 ϵ_0; the latter are for a similar temperature range – but adopting different experimental techniques. An alternative explanation for the discrepancies might be a difference in chemical purity, or the degradation of the samples, as has in the past been pointed out for the 5CB liquid crystal [25, 26].

Table 1. Each cell gives the results for the geometrical 4-fit or 5-fit without approximations.

T (°C)	α_1 (mPa s)	α_2 (mPa s)	α_3 (mPa s)	η_b (mPa s)	$\epsilon_a(\epsilon_0)$
32	n/1	−81/−81	−15/−10	8/13	6.6/6.7
34	n/−3	−63/−63	−9/−7	12/14	7.1/7.2
36	n/1	−56/−56	−10/−9	9/10	6.6/6.6
8	n/−4	−49/−48	−6/−6	12/12	6.6/6.9

Note: ϵ_0 is the permittivity of a vacuum, n indicates neglected.

From an analysis of our results it is possible to see that at 32°C $\alpha_2 \approx -80$ mPa s; it then increases rapidly and almost linearly to zero as the nematic–isotropic temperature is approached. In addition, $\eta_{\min} = \eta_b - \alpha_3 \approx 23$ mPa s at 32°C, and decreases slowly to a constant value, 18 mPa s, at the transition. The L–E theory indeed predicts that α_2 and α_3 vanish at the nematic–isotropic transition, and that all Miesowicz viscosities tend to the same isotropic viscosity. The dielectric anisotropy, ϵ_a, remains approximately constant over the temperature range studied.

5. Conclusions

We have studied the influence of the application of an electric field, perpendicular to the flow field, in the rheological behaviour of the LC, 4-*n*-heptyl-4′-cyanobiphenyl (7CB). The following conclusions can be drawn:

(i) 7CB presents an ER effect, with an increase of about five times the apparent viscosity for small shear rates and moderate electric field strengths;

(ii) For each of the temperatures examined, a master flow curve can be created by dividing the shear rate by the square of the electric field and multiplying by the square of a reference electric field. In a log–log plot two Newtonian plateaux appear, at low and high shear rate values, connected by a shear-thinning region. Theoretical interpretation of the behaviour observed, in the framework of the continuum theory, allows a number of viscoelastic parameters to be extracted in addition to the dielectric anisotropy. The values obtained for the viscoelastic parameters are consistent with those reported previously. However, the estimates obtained for the dielectric anisotropy are smaller than the values reported elsewhere. In spite of this difference, we believe that our system based on 7CB may be considered to be a model for the L–E theory, and well describes its global rheological behaviour;

(iii) Regarding the influence of temperature on the rheological behaviour of 7CB, it has been observed that in the nematic phase, distant from the nematic–isotropic transition, the apparent viscosity slightly decreases with temperature, at a constant value of the electric field. After the transition, we have also observed a small decrease in the apparent viscosity (which is now independent of the electric field) with temperature. Near the nematic–isotropic transition temperature, two different behaviours can be distinguished. For small electric fields, a continuous increase in the apparent viscosity is observed with temperature, but at higher electric fields a rapid decrease in the apparent viscosity is seen. In all cases the transition from nematic to isotropic phase appears to be a continuous process.

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