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Enhancing Structural and Optical Ordering in Liquid Crystals via ZnO Nanoparticle Doping for Sustainable Material Processing

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Abstract

The search for functional materials with adjustable structural and optical properties has become more intense due to the demand for precision-driven material processing and sustainable resource recovering. Liquid Crystalline (LC) materials are becoming interesting candidates for industrial refining and recovery processes because of their anisotropic character and sensitivity to outside stimuli. Still, it can be challenging to change their internal ordering and processing behavior. One possible path to improve the functional performance of LC systems is the integration of inorganic nanoparticles such as Zinc Oxide (ZnO). This work supports attempts to create material applications that are both efficient and environmentally friendly by addressing the need to understand how ZnO nanoparticles affect the structural and optical ordering of a binary LC mixture.

Major Findings: *The study investigates a binary mixture of p-(n-heptyl) benzoic acid (7ba) and p-(n-heptyloxy) benzoic acid (7oba), doped with ZnO nanoparticles (1-2.5 wt%), and uses SEM, XRD, and POM techniques for thorough characterization. The results confirm the retained crystalline phases, and consistent ZnO dispersion of the LC matrix. The optical order parameter, which is computed using the Kuczynski, Vuks, and Haller models at visible wavelengths (435-635 nm), shows that the addition of ZnO significantly improves the nematic ordering and texture regularity. These enhancements show that ZnO-doped LC systems provide finer control over alignment behavior, which qualifies them for resource-efficient recovery, advanced material processing, and environmentally friendly industrial operations.*

Keywords: *7ba+7oba Mixture, Order Parameter, POM, SEM, XRD, ZnO NPs*

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1.0 Introduction

Liquid Crystals (LCs) are related to soft matter, which has mobility and long-range order of molecules aligned in a specific direction known as director[1-3]. The molecules of LCs have Van der Wall forces due to the intermolecular attraction between the molecules[3]. The specific order of molecules is used for different optical devices like sensors, LC displays, and smart windows[4-6]. The incorporation of various Nanoparticles (NPs) like gold and others into the liquid crystals considerably changes different properties of displays, which are used in plasmonics[7,8]. Various methodologies are employed to achieve notable transformations in the self-organization of liquid crystals, utilizing the inherent order of the host material as a framework to impart structural arrangement onto dispersed nanomaterials[9,10]. The dispersion of ZnO NPs in small amounts, to the nematic LC increases the nematic phase range, enhances the electro-optical, and also improves the LC alignment[11-14].

The synthesis of oxide-based nanomaterials has been achieved through various methods, which are known for their flexibility, affordability, ease of use, and consistent reproducibility[15-18]. The research workers are interested in working on the prepared mixture of both p-(n-heptl) (7ba) and p-(n-heptyloxy) (7oba) benzoic acids of LC compounds because one (other) is related to alkoxy (alkyl) group. The mixture, 7ba+7oba LC contains Nematic (N) phase and smectic phases (smC) over a wide temperature range. The studies related to structural and order parameter calculations were estimated with the impact of ZnO NPs in various concentrations (1-2.5 wt%) in a mixture of 7ba+7oba LC compound and a few interesting observations of this study are presented in the ensuing sections.

2.0 Materials and Methods

The chemicals, such as p-n heptyl and heptyloxy benzoic acids,used in the present work were taken from the laboratories of Sigma Aldrich, USA. For the preparation of ZnO NPs, the high-pressure autoclave method was used. At first 3 g of ZnO dissolved in 10 ml of conc. HNO_3 solution and added with 10 ml of H_2O in RB flask (100 ml). Then the solution was stirred at room temperature for 1½ h. The reducing agent, urea was added to it and then add 10 mol% boric acid (flux). The complex was stirred about 1 h and kept the entire solution in to high pressure reactor maintained at the temperature of 120° C and pressure of 200 Kg/cm^2 for 45 minutes. As a result, the urea decomposes the entire solution in to ZnO NPs. The resultant powder was washed about 10 times with methanol and later dried in oven. Then pure ZnO NPs of size about 75 nm were obtained. The shape of the ZnO NPs is spherical and the size of the NPs was determined by SEM and XRD. While using XRD technique, the size of the ZnO NPs is found to be 69 nm[19-21].

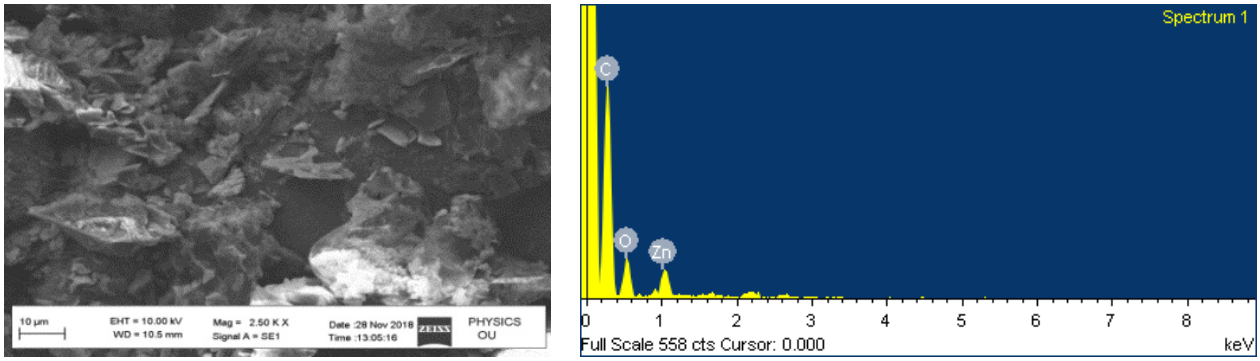
The ingredients of 100mg of benzoic acids p-n-heptyloxy and heptyl benzoic acid (7ba+7oba) were taken separately. At first, 100 mg of 7oba LC compound was taken in a beaker, and ethyl alcohol was mixed into the sample. After that, the sample of 7oba was stirred (700 revolutions per minute) continuously for about 1 h at an ambient temperature 60° C with the help of a magnetic stirrer. For the above sample, 100 mg of another LC named by 7ba compound was added to it and ethyl alcohol was added and stirred for 45 minutes at 60° C. Later, 1wt% of ZnO NPs was introduced in the mixture of both 7ba and 7oba LC compound and further, the temperature was raised to nearly 100° C, and then alcohol was evaporated. The homogenous mixture of LC composite of 7ba+7oba was prepared in an isotropic state. The same process was repeated for all other concentrations[22,23].

XRD analysis was done by Bruker X-ray diffractometer using Cu-Kα radiation for the mixture of 7ba+7oba dispersed with ZnO NPs. The scan rate of 0.01° to 100°/min (2θ), step size of 0.02°, and 2θ range of 5-80° was used in the XRD measurement to improve reproducibility. The surface topography was studied for the mixture of 7ba+7oba sample by scanning electron microscopy (Zeiss special addition-18 make). The chemicals present in the sample, 7ba+7oba with induced ZnO NPs were identified with the help of EDS data. The tool polarizing optical microscope (POM-SDTECHS make SDVPM2727) was used to observe the transition temperatures of the nematic phase of the LC mixture, 7ba+7oba.The order parameters of the mixture were determined by optical methods, like Kuczynski Vuks, and Haller in the stabilized nematic region at various wavelengths (460, 500, 570, and 635 nm), respectively.

3.0 Results and Discussion

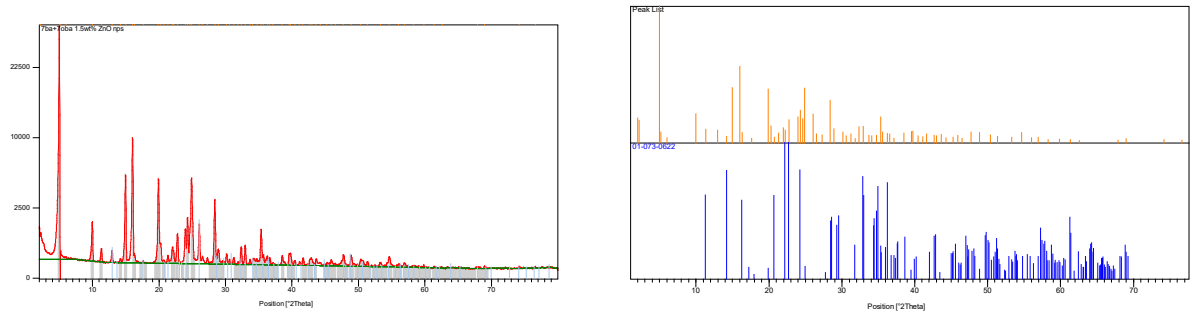
3.1 SEM Analysis

To determine size, shape, morphological, and structural information of the sample, the SEM analysis is used[24].



(a) **(b)**

Figure 1. **(a)** SEM image, **(b)** EDS data of 1.5 wt% dispersed ZnO NPs in the mixture of 7ba+7oba.



(a) **(b)**

Figure 2. **(a)** Complex XRD data, **(b)** XRD pattern of 1.5 wt% dispersed ZnO NPs in the mixture of 7ba+7oba

Figure 1(a) and (b) represent the SEM and EDS pattern of 1.5 wt% dispersed in ZnO NPs in the mixture of 7ba+7oba LC compound. The elemental analysis and weight ratios of chemicals are found with the help of EDS data. The elements present in the sample like Zn, O, and C are analyzed[22,23].

3.2 XRD Studies

XRD gives us information about the phase identification and unit cell dimension of the material. Figure 2 (a) and (b) show the complex XRD data and XRD pattern of 1.5 wt% dispersed ZnO NPs in 7ba+7oba LC mixture. The observed peaks are distinctly resolved and exhibit a strong correlation with the reference pattern provided in JCPDS card no. 01-073-0622.The XRD pattern shows various diffraction peaks at 2θ values, visually 14.32°,24.28°, 28.0°, 37.84°, 44.10°, 54.54°, and 64.44° indicating the presence of ZnO NPs[22,23]. The Scherrer's formula was used to determine crystallite size, which is given by,

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \qquad (1)$$

Where, D represents particle size, symbol β represents Full-Width of peak at Half Maximum (FWHM) while symbol λ is the wavelength of X-ray diffraction source, and θ is the angle of the diffraction. The average crystallite size of ZnO nanoparticle was found to be 62 nm[24].

3.3 POM Studies

The images obtained from the POM confirm that whether the crystal is optically isotropic or anisotropic, and also, explain the nature of the crystal (positive or negative). The pictures obtained from POM have two phases: One is nematic and the other is smectic-C with 1.5wt% dispersed ZnO NPs in the mixture of 7ba+7oba are presented in Figure 3. The POM contains heating block, which is always maintained with temperature accuracy ± 0.1° C and magnification 10X. The temperature variation of heating block is maintained with voltage variac. The LC nanocomposite textures are observed through CCD camera which is connected to the POM. The cooling scan rates of the LC sample are provided in Table 1[22,23].

Table 1. Cooling scan rates of LC mixture of 7ba+7oba with dispersed ZnO NPs of POM/DSC

S.No	Compound	DSC/POM	Scan Rate	Transition Temperatures (°C)			
				I-N	N-Smc	Smc-Solid 1	Solid 1 - Solid 2
1	pure 7ba +7oba	DSC	20°C/min ΔHJ/g	130.57-23.46		80.81-110.69	
		POM		130.5	116.5	85.8	72.1
2	7ba+7oba+1wt% ZnO NPs	DSC	20°C/min ΔHJ/g	128.3-26.49		79.11-143.67	
		POM		128.1	114.5	78.9	71.8
3	7ba+7oba+1.5wt% ZnO NPs	DSC	20°C/min ΔHJ/g	126.98-12.72		79.58-28.91	70.91-29.71
		POM		127.1	113.8	79.4	70.9
4	7ba+7oba+2wt% ZnO NPs	DSC	20°C/min ΔHJ/g	125.63-18.53		78.97-149.90	
		POM		124.6	112.6	78.9	70.5
5	7ba+7oba+2.5wt% ZnO NPs	DSC	20°C/min ΔHJ/g	125.40-16.53		77.22-119.49	72.45-25.87
		POM		125.4	110.5	77.0	71.5

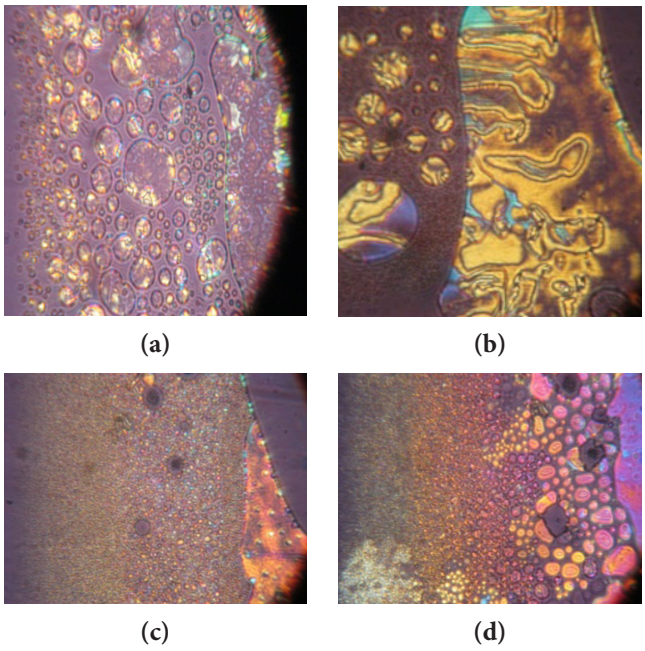


Figure 3. POM images of 1.5 wt% dispersed ZnO NPs in the mixture of 7ba+7oba, in which **(a)** Isotropic- nematic at 127.1°C, **(b)** Nematic at 124.6°C, **(c)** smectic-C at 113.8 °C, and **(d)** smectic -C to solid at 79.4°C.

3.4 Order Parameter

Order parameter (S) is nothing but the average direction which is always oriented about the optic axis in LC molecules. The values of S are measured from the values of RI data by various procedures, namely Kuczynski, Vuks, Haller, and also from effective geometry methods. The role of ZnO NPs in ordering the LC systems is used in photovoltaic applications. An improvement in the alignment of the molecules of LCs can be achieved due to the induced ZnO NPs in 7ba+ 7oba LC compound.

3.5 Kuczynski Model

For the measurement of S values, the birefringence (δn) values are taken in the nematic phase and not taken by the local field experienced by molecule of LCs. The way of finding of S values are so easy and this model was developed by Kuczynski. The birefringence is determined by simple equation and it is related to temperature $\delta n = \Delta n.\{1 -(T/T^*)\}^{\beta}$. For fitting these parameters, the value of δn is taken in log form and it can be written as:

$$\text{Log } \delta n = \log \Delta n + \beta \log \{1- (T/T^*)\} \quad (2)$$

The simplest equation of measurement of value of order parameter S is $S = \delta n/\Delta n$. In these terms having the special names like Δn is the birefringence in perfect order, $\delta n = (n_e - n_o)$, here the values are determined only in the nematic phase. The corresponding pictures are shown in Figure 4.

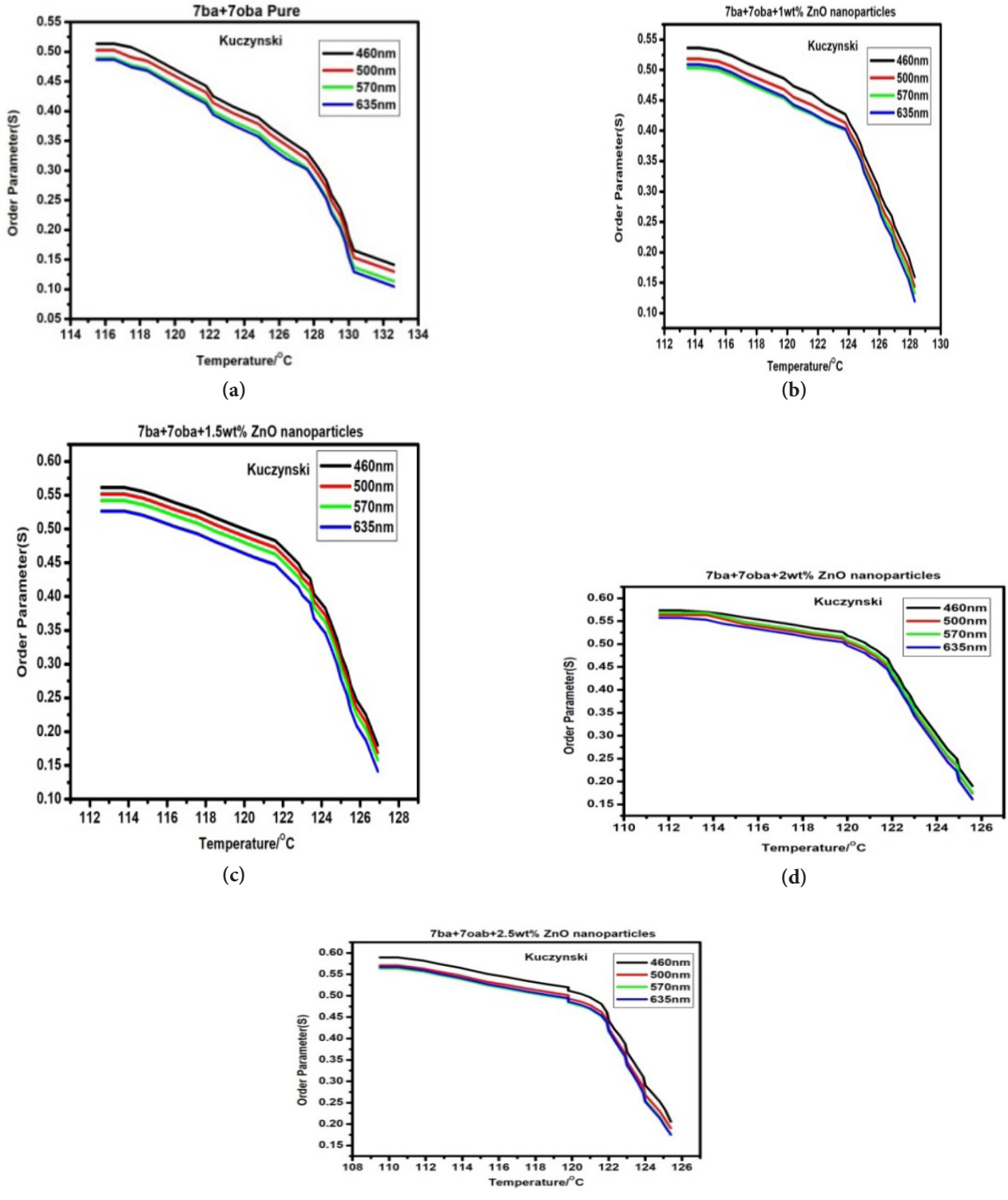


Figure 4. Temperature versus order parameter of **(a)** pure 7ba+7oba and with different concentrations, **(b)** 1 wt%, **(c)** 1.5 wt%, **(d)** 2 wt%, **(e)** 2.5 wt% at various wavelengths by Kuczynski model.

3.6 Vuks Isotropic Field Model

From the Equation (3), the values of order parameter can be calculated by Vuks' isotropic field model.

$$S_{vuks}\frac{\Delta\alpha}{\alpha}=\frac{n_e^2-n_o^2}{\langle n^2\rangle-1} \quad (3)$$

To find the value of Δα/α, a graph is drawn between Log $\frac{n_e^2-n_o^2}{\langle n^2\rangle-1}$ and log (1 – T/T_{NI}). The graph gives a linear relationship between the above two terms which is named as scaling factor by simply extrapolate this graph at absolute temperature T=0 K, the intercept value gives the value of scaling factor. Here, Δα and Δα ($\alpha_{\parallel}-\alpha_{\perp}$) are the mean value of molecular polarizabilities of LC molecules and α is the anisotropy, the order parameter can be determined at different temperatures. The corresponding pictures are shown in Figure 5.

The fitting parameters like Δn and β are provided in Table 2 and 3 for Kuczynski and Vuks models.

Table 2. Fitting parameters of LC mixture of 7ba+7oba with dispersed ZnO NPs in Kuczynski method

Compound	T^* = (T_{NI}+…)	β	Log(Δn)	Δn	R
At 460 nm					
7ba+7oba pure	417.713	0.2319	0.5587	0.2669	0.9569
7ba+7oba+1wt%	414.915	0.2227	0.5573	0.2748	0.9572
7ba+7oba+ 1.5wt%	413.005	0.1857	0.5460	0.2810	0.9476
7ba+7oba+2wt%	409.902	0.1791	0.5377	0.2935	0.9391
7ba+7oba+ 2.5wt%	409.203	0.1685	0.5288	0.3025	0.9307
At 500 nm					
7ba+7oba pure	417.713	0.2315	0.5684	0.2634	0.9555
7ba+7oba+1wt%	414.914	0.2330	0.5455	0.2730	0.9562
7ba+7oba+ 1.5wt%	413.005	0.1913	0.5412	0.2804	0.9467
7ba+7oba+2wt%	409.902	0.1869	0.5337	0.2916	0.9287
7ba+7oba+ 2.5wt%	409.203	0.1786	0.5296	0.2995	0.9275
At 570 nm					
7ba+7oba pure	417.710	0.2405	0.5658	0.2621	0.9592
7ba+7oba+1wt%	414.914	0.2407	0.5535	0.2723	0.9521
7ba+7oba+ 1.5wt%	413.004	0.1969	0.5426	0.2794	0.9432
7ba+7oba+2wt%	409.902	0.1938	0.5307	0.2887	0.9277
7ba+7oba+ 2.5wt%	409.202	0.1848	0.5275	0.2945	0.9235
At 635 nm					
7ba+7oba pure	417.708	0.2455	0.5743	0.2606	0.9580
7ba+7oba+1wt%	414.906	0.2440	0.5685	0.2707	0.9490
7ba+7oba+ 1.5wt%	413.004	0.2063	0.5343	0.2697	0.9427
7ba+7oba+2wt%	409.901	0.2015	0.5255	0.2736	0.9265
7ba+7oba+ 2.5wt%	409.202	0.1917	0.5285	0.2921	0.9226

Table 3. Fitting parameters of LC mixture of 7ba+7oba with dispersed ZnO NPs in Vuks method

Compound	T^* = (T_{NI}+…)	β	Log(Δn)	Δα/α	R
At 460 nm					
7ba+7oba pure	417.713	0.2194	0.1933	0.6405	0.9545
7ba+7oba+1wt%	414.915	0.2282	0.1870	0.6500	0.8688
7ba+7oba+ 1.5wt% wt460nm	413.005	0.1869	0.1729	0.6715	0.9273
7ba+7oba+2wt%	409.902	0.1762	0.1669	0.6808	0.8537
7ba+7oba+ 2.5wt%	409.202	0.1642	0.1600	0.6918	0.8775
At 500 nm					
7ba+7oba pure	417.711	0.2239	0.1943	0.6392	0.9512
7ba+7oba+1wt%	414.913	0.2357	0.1914	0.6435	0.8656
7ba+7oba+ 1.5wt%	413.004	0.1901	0.1735	0.6705	0.9242
7ba+7oba+2wt%	409.901	0.1748	0.1698	0.6765	0.8519
7ba+7oba+ 2.5wt%	409.201	0.1623	0.1640	0.6855	0.8748
At 570 nm					
7ba+7oba pure	417.708	0.2317	0.1971	0.6351	0.9460
7ba+7oba+1wt%	414.9125	0.2426	0.1939	0.6398	0.8673
7ba+7oba+ 1.5wt%	413.0038	0.1925	0.1759	0.6671	0.9200
7ba+7oba+2wt%	409.901 4409.9014	0.1870	0.1708	0.6747	0.8483
7ba+7oba+ 2.5wt%	409.201	0.1782	0.1677	0.6798	0.8686
At 635 nm					
7ba+7oba pure	417.707	0.2281	0.2042	0.6250	0.9428
7ba+7oba+1wt%	414.908	0.2495	0.1989	0.6325	0.8584
7ba+7oba+1.5wt%	413.002	0.1988	0.1773	0.6647	0.9162
7ba+7oba+2wt%	409.901	0.1951	0.1754	0.6677	0.8453
7ba+7oba+ 2.5wt%	409.201	0.1847	0.1684	0.6785	0.8657

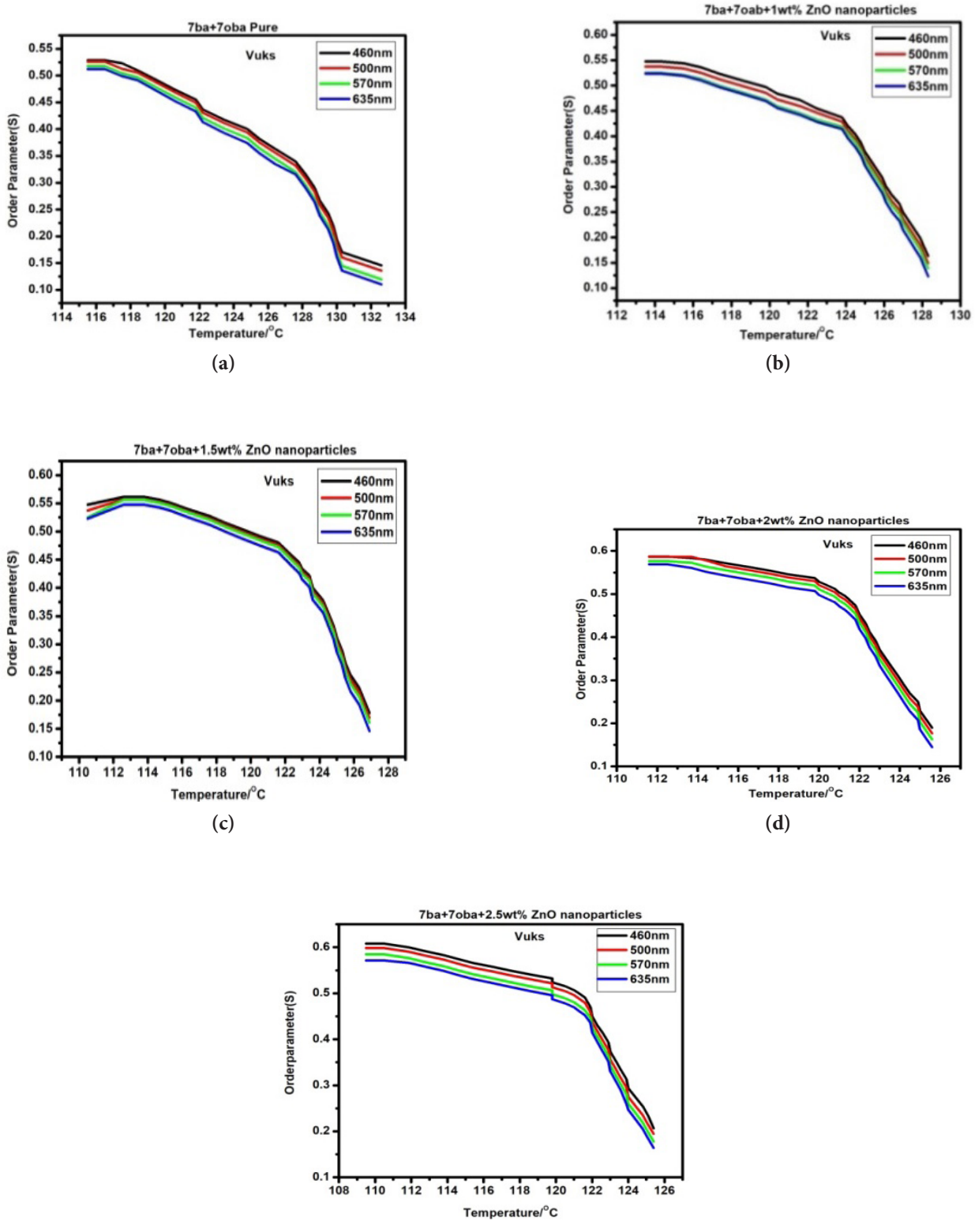


Figure 5. Temperature versus order parameter of (top left) pure 7ba+7oba and with different concentrations (top middle) 1 wt%, (top right) 1.5 wt%, (bottom left) 2 wt%, (bottom middle) 2.5 wt% at various wavelengths by Vuks model.

3.7 Haller Extrapolation Model

As the birefringence value increases, the order parameter is increased and it is in linear relationship. The values of S are calculated using Haller's model with the simple equation and it is temperature dependent. The equation is mentioned below: The pictures related to this model are presented in Figure 6.

$$S_{Haller} = \left(1 - \frac{T}{T_{NI}}\right)^{\beta} \quad (4)$$

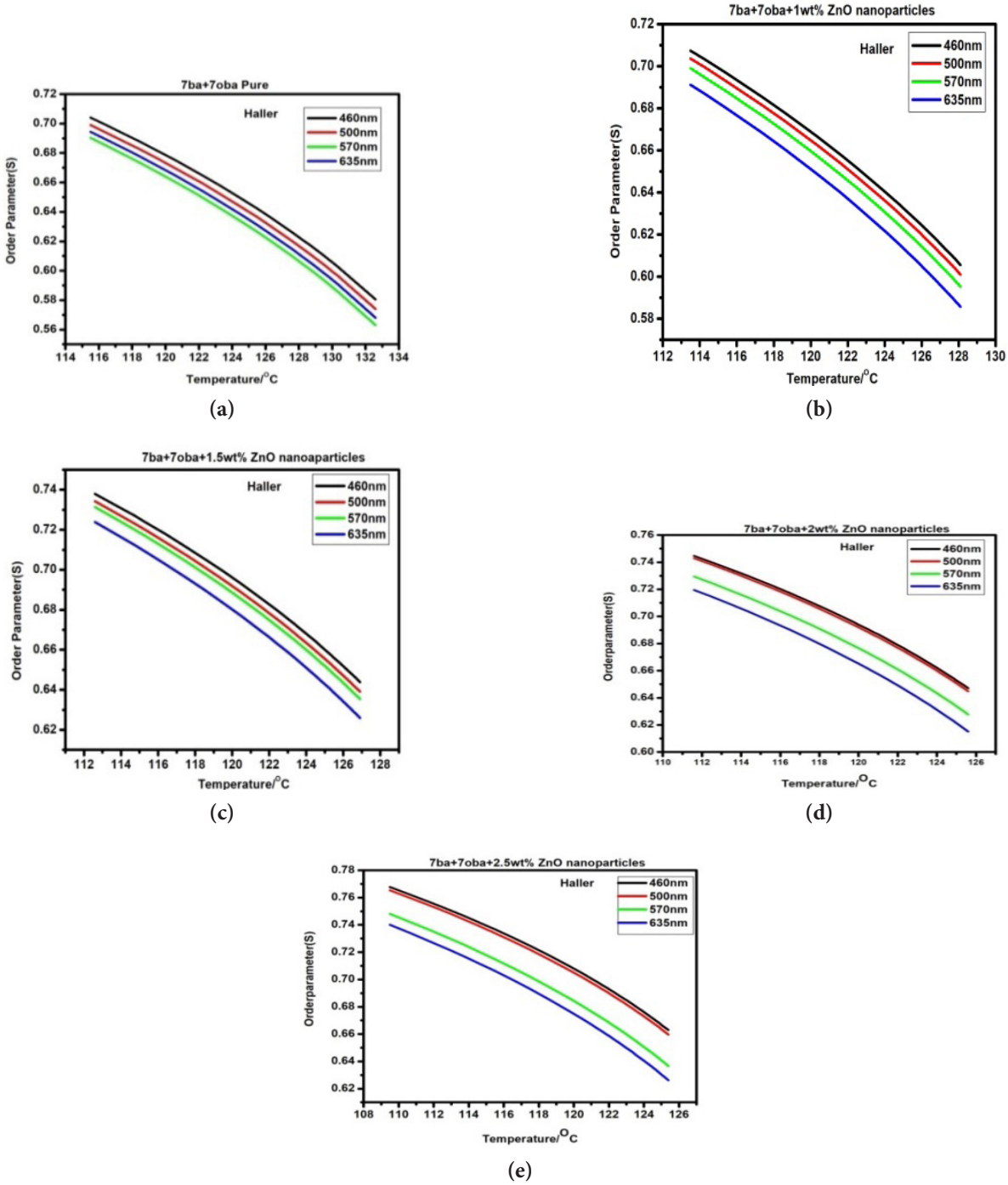


Figure 6. Temperature versus order parameter of **(a)** pure 7ba+7oba and with different concentrations **(b)** 1 wt% **(c)** 1.5 wt% **(d)** 2 wt% **(e)** 2.5 wt% at various wavelengths by Haller model.

Table 4. The order parameter of 7ba+7oba LC molecules with ZnO NP dispersions at varying wt% was analyzed using different field models

LC compound+ ZnO NPs	Wavelength (nm)	Values of Order Parameter, S by		
		Kuczynski	Vuks	Haller
7ba+7oba pure	460	0.5133	0.5288	0.7020
	500	0.5024	0.5257	0.6989
	570	0.4891	0.5176	0.6902
	635	0.4865	0.5124	0.6834
7ba+7oba+ 1 wt%	460	0.5360	0.5478	0.7040
	500	0.5184	0.5375	0.7036
	570	0.5037	0.5255	0.6989
	635	0.5091	0.5232	0.6912
7ba+7oba+ 1.5 wt%	460	0.5587	0.5612	0.7379
	500	0.5479	0.5576	0.7342
	570	0.5363	0.5559	0.7328
	635	0.5207	0.5479	0.7312
7ba+7oba+ 2 wt%	460	0.5739	0.5872	0.7468
	500	0.5631	0.5866	0.7444
	570	0.5684	0.5763	0.7420
	635	0.5570	0.5691	0.7400
7ba+7oba+ 2.5 wt%	460	0.5891	0.6079	0.7639
	500	0.5711	0.5986	0.7568
	570	0.5640	0.5849	0.7450
	635	0.5669	0.5712	0.7432

Where T_{NI} represents the clearing temperature of Isotropic-nematic phase and β is the slope. These values are meant for perfectly liquid crystalline state of LC materials of order S=1 which thermally influences the behavior of LC materials.

The order parameters of 7oba+7ba LC compound are increased with different weight concentrations of ZnO NPs. Further, the order parameter values are also improved from higher wavelength to lower wavelength in the visible region in the three internal field models[25]. From the above observations, since ZnO NPs are larger in size and these are having high dipole moments than nematic LC, which produces more torque than nematic LC[26-30]. This torque is used to orient the molecules of LCs enhances the birefringence and ordering of LC molecules. The ordering of LC system are calculated in the three internal field models also observed with induced of ZnO NPs (1-2.5 wt%), related data is presented in Table 4. Various researches are found the parameters like electrical conductivity, birefringence and photoluminescence are increased with the dispersion of ZnO NPs in LC material. The optical parameters are enhanced a significant role in display technology[31-34].

4.0 Conclusion

The mixture of of p-(n-heptyl) and p-(n-heptyloxy) benzoic acids with the homogeneous dispersion of ZnO NPs in different wt% were prepared. Different characterization techniques like SEM, XRD, and POM for 7ba+7oba LC nanocomposite were carried out. The topological studies and elements presented in the LC nanocomposite had been verified by SEM and EDS data. The crystalline size of ZnO nanoparticles were found with the help of XRD technique and it was 62 nm. Different textures like nematic and smectic C phases were observed through POM analysis. The variation of order parameter in the samples prepared revealed that they may be useful in optoelectronic gadget application for building LC display devices.

5.0 Acknowledgement

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6.0 Author Contributions

All authors have contributed significantly to this research. Dr. M.C. Rao and Dr. P. S. Brahmanandam conceptualization and supervised the research. All authors reviewed and approved the final manuscript.

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