Characterisation and mesomorphic behaviour of liquid crystals with dispersed PdCl₂ nanoparticles

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Abstract

The synthesis and characterisation are carried out on liquid crystalline (LC) p-dodecylxylo benzoic acid (12OBA) with 1 and 2 wt% for PdCl₂ nanoparticles dispersion. Further, characterizations are carried out by different spectroscopic techniques like X-ray diffraction spectrometry studies, scanning electron microscopy, Fourier transform infrared and differential scanning calorimetry (DSC). Textural determinations of the synthesised compounds are recorded by using polarising optical microscope (POM) attached with a hot stage and camera. The results show that the dispersion of PdCl₂ nanoparticles in 12OBA exhibits Nematic phases as same as the pure 12OBA with reduced clearing temperature as expected. Further, the nematic thermal ranges are quenched and the smectic C thermal range has been increased while performing both DSC and POM with the dispersion of 1 wt% PdCl₂ nanoparticles. Size dependence on bonding nature with LC compounds is established.

1. Introduction

Liquid crystals (LCs) are self-assembled functional soft materials which possess both order and mobility at molecular, supra-molecular and macroscopic levels [1–3]. LCs received much attention in the recent years, since they show unique properties, such as long-range order, cooperative effects and an anisotropic nature in optical and electronic properties, based on a self-organising nature in a certain temperature range with fluidity [4–10]. Nanoparticles hold promise for use as innovative materials with new electronic, magnetic, optical and thermal properties [11–25]. They have also been applied to the catalyst in, for example, hydrogenation of olefins and dienes [26], hydration of acrylonitrile to acrylamide [27], photogeneration of hydrogen from water [28] and reduction of carbon dioxide [29]. LCs are now playing a very significant role in nanoscience and nanotechnology [30–32]. Nanoscale particles do not induce significant distortion of LC phases. Therefore, different nanomaterials are dispersed and studied in LC media to enhance the physical properties of LCs [30]. Moreover, alignment and self-assembly of nanomaterials themselves can be achieved in LC phases [31]. The key point for all the possible applications is the alignment of LC molecules (i.e. the director) on the substrate [33,34]. LCs act as tuneable solvents for the dispersion of nanomaterials and LCs being anisotropic media, they provide a very good support for the self-assembly of nanomaterials in to large organised structures in multiple dimensions. Hence LC-mediated self-assembly can be efficiently used to organise different kinds of nanomaterials in to soft and well-defined functional superstructures. Homeotropic alignment of LCs has applications in liquid crystal display (LCD) technology, such as high information display devices, large-area LCD TVs and digital display devices used in the medical field like digital medical imaging [35]. Recently, it was reported that palladium(II) complexes with organic LC materials containing the azobenzene or Schiff base frames, bonded through a metal–carbons bond and having a metal–metal chloride bridge, display mesophases [36]. Moreover, LCs will degrade over time and generate ions which affect quality issues. To solve this problem, researchers merged LCs and nanotechnology and obtained the good results in reducing the ion concentrations by trapping ions in LCs embedded with nano-objects and enriches enhanced electro-optical responses. In this

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work, the changes in the nematic ranges were studied with the dispersion of palladium nanoparticles into the LC compounds.

2. Material and experimental details

LC compounds such as 12OBA and PdCl$_2$ nanoparticles are brought from Sigma-Aldrich laboratories (St. Louis, MO, USA) and used as such. For uniform dispersion of nanoparticles in 12OBA, the nanoparticles are first dissolved in ethyl alcohol, stirred well for about 45 min and later introduced in the isotropic state of mesogenic material in the ratio of 1 and 2 wt% concentration separately. After cooling, the nano-composite 12OBA is subjected to study of the textural and phase transition temperatures using a POM (SDTECHS make) with a hot stage in which the substance was filled in planar arrangement in 4-µm cells and these could be placed along with the thermometer, as described by Gray [37]. Textural and phase transition temperatures are studied after preparation of the sample and observations are made again to understand the stability of PdCl$_2$ nanoparticles. A differential scanning calorimeter (DSC) (PerkinElmer Diamond DSC, Waltham, MA, USA) is used to obtain the transition temperatures and the enthalpy values. Fourier transform infrared (FTIR) is a powerful tool for identifying different types of chemical bonds in a molecule by producing an infrared (IR) absorption spectrum and useful for identifying chemicals that are either organic or inorganic. X-ray Diffraction (XRD) technique is used to determine the grain size of the PdCl$_2$ nanoparticles which are dispersed in 12OBA. The presence of PdCl$_2$ nanoparticles in 12OBA is studied by scanning electron microscopy (SEM) data and existence.

The molecular structure of 12OBA is given below:

\[
\text{C}_{12}\text{H}_{25} \begin{array}{c} \text{C} \\ \text{OH} \end{array}
\]

3. Results and discussion

Phase transition temperatures and phase sequence of the compounds have been presented by polarising optical microscope (POM) and confirmed by DSC. The existence of dispersed nanoparticles and their size is determined by FTIR, XRD and SEM techniques. Doping concentrations of nanoparticles are limited to 1 and 2 wt% only. Studies on LCs dispersed with nanoparticles reveal that only 1 wt% is sufficient for uniform coverage of substrate [38]. Values above that concentration may not affect the properties and leads to either contamination or no change conditions due to aggregation.

The phase variants, transition temperatures, enthalpy values of 12OBA pure and with dispersed 1 and 2 wt% PdCl$_2$ nanoparticles are given in Table 1.

3.1. Polarising microscope

The transition temperatures and textures observed by POM in pure 12OBA are shown in Figure 1(a–d) while that of 12OBA with dispersed 1 and 2 wt% of PdCl$_2$ nanoparticles shown in Figures 2(a–d) and 3(a–d), respectively. The thermal ranges of nematic phase is quenched by 3° with 1 wt% while slightly increased with the dispersion of 2 wt% and the thermal range of SmC phase increases in both the dispersions and the textures of the phases changes by the self-assembly of nanoparticles. DSC (PerkinElmer Diamond DSC) is used to obtain the transition temperatures and the enthalpy values before and after synthesis in exothermic as well as endothermic regimes. DSC thermograms (in cooling) of 12OBA and with dispersed 1 and 2 wt% PdCl$_2$ are shown in Figures 4 and 5, respectively.

The results are showing and supporting that the suspensions of dispersed nanoparticles may generate a considerable structural defects in the LC phase and break the continuous rotational symmetry. This type of modification in LCs permits much easier fabrication than conventional chemical synthetic methods for special electro-optical features [4]. As a result, LCs doped with nanomaterials show great potential for future LCD technology development.

### Table 1. Phase variants, transition temperatures, enthalpy values of 12OBA pure and with dispersed 1 and 2 wt% PdCl$_2$ nanoparticles.

| S. No | Compound | DSC/POM | Scan rate | Transition temperatures (°C) | Thermal range |
|-------|----------|---------|-----------|-----------------------------|---------------|
|       |          |         | I-N       | N-SmC | SmC-SolidI | SolidI-Solid II | ΔN | ΔSmC |
| 1     | 12OBA PURE | DSC     | 2°C/min   | 138.8 | 132.8 | 97.3 | 6 | 35.5 |
| 2     | 12OBA+1% PdCl$_2$ | DSC | 2°C/min | 129.09 | 125.78 | 86.23 | 63.01 | 3.31 | 39.55 |
|       |          | POM     | ΔH (J/g)  | 3.5161 | 0.8275 | 28.6874 | 3.2 | 41.3 |
| 3     | 12OBA+2% PdCl$_2$ | DSC | 2°C/min | 134.69 | 128.08 | 86.21 | 62.44 | 6.61 | 41.87 |
|       |          | POM     | ΔH (J/g)  | 4.4552 | 2.6644 | 22.6602 | 7.4 | 44.1 |
3.2. FTIR analysis

As synthesised PdCl$_2$ nanoparticles dispersed in 12OBA compound is analysed by using FTIR at room temperature. The compound is stable at room temperature; the IR frequencies in solid state are correlated with the pure bond 12OBA. The assigned bonds corresponding to the resultant frequencies from the spectra are tabulated. Due to the excitation of both molecular vibrations and rotational absorptions, electromagnetic radiation causes the formation of absorption bands in the IR spectra which are useful to explain the bonding interaction of the molecules. Both spectra exhibit a strong electromagnetic absorption at

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**Figure 1.** Texture of pure 12OBA. (a) Nematic droplets at 137.3°C; (b) nematic phase at 132.4°C; (c) nematic to smectic C at 130.7°C; (d) solid at 85°C.

**Figure 2.** Textures 12OBA with 1 wt% PdCl$_2$. (a) Isotropic to nematic droplets at 132.8°C; (b) nematic to smectic C at 129.6°C; (c) smectic C at 98.3°C; (d) solid at 78°C.

**Figure 3.** Textures of 12OBA with 2 wt% PdCl$_2$. (a) Isotropic to nematic at 134.4°C; (b) nematic to smectic C at 127.0°C; (c) smectic C at 100.2°C; (d) solid at 65.2°C.

**Figure 4.** DSC thermogram of 12OBA with 1 wt%PdCl$_2$.

**Figure 5.** DSC thermogram of 12OBA with 2 wt%PdCl$_2$. 
1595.51, 1601.96 and 1249.29, 1258.67 cm\(^{-1}\) corresponding to aromatic ring stretching. The absorption bands at 2916.44 and 2909.98 cm\(^{-1}\) are corresponding to OH bond. The existence of OH bond vibration at 648.97 and 645.45 cm\(^{-1}\) also represents the benzoic acids moiety due to their strong intensity and strongly supports the existence of 12OBA. The bonds at 842.62 and 839.69 cm\(^{-1}\) are assigned to stretching ring vibration at the out of plane. While dispersing the PdCl\(_2\) nanoparticles, the intensity of the peaks are found to be increased as shown in the Figure 6. The FTIR of 12OBA pure and with dispersed PdCl\(_2\) nanoparticles.

Table 2. Functional group intensities for 12OBA pure, and with dispersed PdCl\(_2\) nanoparticles across the following wavelengths.

| S. No. | Wavelength (cm\(^{-1}\)) | Intensity for pure LC | Intensity for LC with dispersed PdCl\(_2\) nanoparticles | Functional group |
|--------|---------------------------|-----------------------|--------------------------------------------------------|-----------------|
| 1      | 2916.44                   | 0.8055                | 0.8402                                                 | OH bond         |
| 2      | 1674.73                   | 0.7885                | 0.8140                                                 | Benzoic acid    |
| 3      | 1595.51                   | 0.7911                | 0.8190                                                 | Ring stretching |
| 4      | 1573.21                   | 0.9071                | 0.9241                                                 |                |
| 5      | 1515.7                    | 0.9156                | 0.9342                                                 |                |
| 6      | 1471.69                   | 0.8834                | 0.9055                                                 |                |
| 7      | 1293.88                   | 0.7971                | 0.8241                                                 | Dimer           |
| 8      | 1249.29                   | 0.6963                | 0.7318                                                 | Aromatic ring structure |
| 9      | 1166.54                   | 0.8241                | 0.8597                                                 | CHOH bending vibration |
| 10     | 842.62                    | 0.8428                | 0.8817                                                 | Ring out of plane |
| 11     | 772.79                    | 0.8156                | 0.8487                                                 | Aromatic ring stretching |
| 12     | 712.35                    | 0.9080                | 0.9257                                                 | CH out of plane |
| 13     | 690.05                    | 0.9122                | 0.9299                                                 | C=O bending     |
| 14     | 648.97                    | 0.8207                | 0.8436                                                 | OH bond         |

Table 3. SEM analysis of the compounds.

| S. No. | Element | Weight% | Atomic% |
|--------|---------|---------|---------|
| 1      | C       | 76.97   | 84.71   |
| 2      | O       | 17.45   | 14.42   |
| 3      | Cl      | 1.25    | 0.46    |
| 4      | Pd      | 2.09    | 0.26    |

3.3. **SEM analysis**

SEM is one of the most popular and widely used techniques for the characterisation of nanomaterials. The SEM provides the investigator with a highly magnified image of the surface of a material as the present sample contains electrons which are needed for getting SEM image. SEM can be effectively used to characterised specimens down to a resolution of a few nanometres, with image magnification achievable in the range from ~10 to over 3,00,000. The SEM images of the grown sample are shown in Figure 8 and the analysis resultant weights and atomicpercentages are presented in Table 3. From EDS related data of Figure 7, the presence of PdCl\(_2\) nanoparticles in the 12OBA compound is well established.

3.4. **XRD analysis**

The XRD data of 12OBA pure and with 1 wt% PdCl\(_2\) nanoparticles are shown in Figure 9. In comparison to JCPDF data, peaks were well resolved and are matched with JCPDF card number 010228 which is clearly evidenced by the existence of palladium nanoparticles. By using Scherrer’s formula, \( t = \frac{k\lambda}{\beta \cos \theta} \), the particle size is found to be 61 nm, \( \Lambda = 1.54 \) Å, \( \beta = \text{FWHM} \), peaks at 40° resembles the existence of PdCl\(_2\) nanoparticles.

The diffraction peak at around 40° corresponds to the \((1 1 1)\) plane of the fcc lattice. In the XRD pattern, diffraction peaks broaden as the crystallite size decreases [39]. So, there is a definite change and is confirmed with these nanoparticle dispersed in 12OBA LC compound. XRD related data is shown in Table 4.

4. **Conclusions**

From these studies on 12OBA LC compound with dispersed PdCl\(_2\) nanoparticles, transition temperatures with image magnification achievable in the range from ~10 to over 3,00,000. The SEM images of the grown sample are shown in Figure 8 and the analysis resultant weights and atomicpercentages are presented in Table 3. From EDS related data of Figure 7, the presence of PdCl\(_2\) nanoparticles in the 12OBA compound is well established.

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4. **Conclusions**

From these studies on 12OBA LC compound with dispersed PdCl\(_2\) nanoparticles, transition temperatures
obtained from POM attached with the hot stage are in good agreement with those obtained from DSC. The slight differences can be attributed due to the different experimental conditions. The transition temperatures of nematic as well as smectic C phases have been reduced while dispersing the PdCl$_2$ nanoparticles. The nematic thermal ranges for the dispersion of 1 wt% of PdCl$_2$ nanoparticles have been reduced by 3°C while these increased slightly in the case of 2 wt% dispersion, and the smectic C thermal ranges are increased. From FTIR analysis, the intensities of 12OBA with dispersed PdCl$_2$ nanoparticles are found to be increased. This related to the change in dipole that occurs during the vibration. The vibrations that produce small change in dipole moment of a molecule result in a less intense absorption than those that result in a relatively modest change in dipole. The presence of PdCl$_2$ nanoparticles is shown from SEM and XRD analysis. From these studies, it is clear that orientation changes with the nanoparticle dispersion will modify the transition temperature as well as textural changes which are useful for the display applications at low temperatures.

Table 4. XRD related data.

| Pos. (°2Th.) | Height (cts) | FWHM (°2Th.) | d-Spacing (Å) | Rel. Int. (%) |
|--------------|--------------|--------------|---------------|---------------|
| 10.7215      | 17.02        | 0.7085       | 8.25179       | 6.17          |
| 13.9904      | 140.63       | 0.3542       | 6.33025       | 50.98         |
| 16.5800      | 254.13       | 0.3542       | 5.34693       | 92.12         |
| 19.4558      | 239.52       | 0.3542       | 4.56258       | 86.82         |
| 22.2817      | 128.79       | 0.3542       | 3.98990       | 46.68         |
| 23.9794      | 275.88       | 0.4723       | 3.71114       | 100.00        |
| 26.6115      | 202.66       | 0.3542       | 3.34976       | 73.46         |
| 29.4359      | 41.58        | 0.4723       | 3.03446       | 15.07         |
| 33.4565      | 18.73        | 0.3542       | 2.67842       | 6.79          |
| 39.7383      | 42.00        | 0.3542       | 2.26831       | 15.22         |
| 42.6696      | 34.45        | 0.3542       | 2.11903       | 12.49         |
| 46.4722      | 9.03         | 0.9446       | 1.95411       | 3.27          |
| 49.7226      | 8.71         | 0.7085       | 1.83372       | 3.16          |
| 52.4975      | 12.47        | 0.5904       | 1.74314       | 4.52          |
| 67.7707      | 7.77         | 0.8640       | 1.38162       | 2.82          |

Figure 7. EDS related data.

Figure 8. SEM photograph.

Table 4. XRD related data.

Figure 9. XRD of 12OBA and 12OBA with PdCl$_2$ nanoparticles.
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Disclosure statement

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