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Study of Statistical Parameters through Image Analysis Technique of 16ABBA Liquid Crystal

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ABSTRACT:
Study of statistical parameters of azomethine ester, 4-hexadecyloxy benzylidene-4'-bromoaniline (16ABBA) liquid crystal with image analysis technique is reported in this paper. Textures of the sample are recorded with polarizing optical microscope attached with hot stage and colour camera as a function of temperature. The sample shows enantiotropic smectic A and monotropic smectic B phase. Statistical parameters such as Mean, Standard Deviation, Entropy, Energy, Contrast, and Homogeneity, are calculated using image analysis technique using Matlab software. The results obtained shows abrupt change at the phase transitions. So, temperature dependence of statistical parameters will be applied to identify mesophase transition temperatures.
Keywords: Image analysis, Statistical parameters, Phase transitions, Liquid crystals.

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I. INTRODUCTION

Research and development on liquid crystals has been growing steadily in last decades resulting better and cheaper displays by discovering new types of liquid crystals [1]. Liquid crystals exhibit rich variety of phase structures and phase transitions.. The structure of liquid crystal phases is easily influenced by external conditions e.g., temperature, pressure, electric field, magnetic field, etc. [2,3]. For the planar alignment of liquid crystals, the phases possess complex structures and properties which are studied in detail. Recently, homeotropic alignment of liquid crystals has got wide applications in liquid crystal displays such as high information display (LCD) devices, LCD TV's and digital display devices in medical field like medical imaging, etc. Still, the investigations for the properties of homeotropically aligned liquid crystals components are at a nascent stage, especially when more complex and different phase types are considered. Mesomorphic materials, either low molar mass or polymeric in nature, having an azo group in the mesogenic core, are often studied owing to their interesting oxygen

enriching ability, enhanced air-separation performance and optical properties, which enable applications in, for example, optical switching, holography and optical storage devices [4]. Dyes containing azo group are also being used in LCD devices for guest–host interaction [5]. However, the basic study of any liquid crystal compound involves the identification of phase transition temperatures. There are several techniques to identify the mesophases or phase transitions of liquid crystals, such as Polarizing Optical Microscope (POM), Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), X- RD, Raman spectroscopy etc [6,7].

The basic apparatus and most widely used technique in identification of phase transition temperatures is POM. DSC and DTA inform the presence of phase transitions in a material by detecting enthalpy and entropy changes that are associated with each phase transition. However, precise phase identification could not easily be made. The phase transitions causing small enthalpy and entropy changes are not identified in the procedures of DSC and DTA. Hence, DSC or DTA is used in conjunction with POM to determine the phase transitions temperatures of the samples as POM is a standard tool for liquid crystal compounds. In 1998 Sparavigna group proposed an image processing and analysis methodology in conjunction with POM to investigate the phase transitions of liquid crystals [8,9].

The optical intensities are very sensitive both in spatial and frequency domains for an image of any object or material. The material exhibits the apparent variations in radiated intensities when they undergo phase transitions either in the form and shape/orientation and even at the slightest transition occurs in the material form due to the reorientation of the atomic/molecular changes. The sensitive variations in the textural or transitional phases of the material in response to the temperature changes are measured or recorded using POM attached to the high resolution camera. The analysis of the recorded textural images basing on the radiation intensities in three primary spectral bands red, green and blue at the matrix of pixels has been used to identify the phase transition temperatures of the liquid crystals. Image analysis is the method used to extract the meaningful information from images by means of applying computational techniques and algorithms on image data. The computations of statistics and measurements in the image analysis method are done on considering the gray level intensities of image pixels. The statistical methods will analyze the spatial distribution of gray levels by computing the local and global features at each intensity value in the image and derive a set of statistical parameters from the distribution of these features [10,11]. A few researchers used the statistical parameters derived from the local features to identify the phase transitions temperatures of liquid crystals [12,13]. Recently, a novel approach [14] of statistical measure called image moment which is derived from the global features of the liquid crystal textures is applied to identify the phase transitions temperatures.

Image moments are weighted average intensities of the images and are calculated from the computational analysis of images. These will give meaningful information about image objects [15] However, the investigated phase transition results are compared with the data made by other standard techniques.

Statistical parametes through image analysis is reported on cholesterics [16], discotics [17], in nematics [18], in smectics [19], in cyano [20] liquid crystals. In this paper study of statistical parameters of azomithine ester 4-hexa-decyloxybenzylidene-4'-bromoanilene (16ABBA) liquid crystal using image analysis technique is reported.

II. EXPERIMENTAL

Liquid crystal material azomethine ester, 4-hexadecyloxy benzylidene -4'-bromoaniline (16ABBA) was synthesized [21] and the structure is shown below.

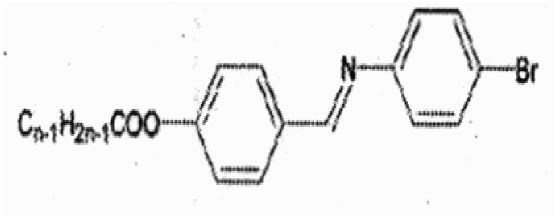


Fig. 1. *n*ABBA (where n=16)

An indium-tin oxide (ITO) coated homogeneous cell of dimensions 5 mm × 5 mm with 5µm spacing (tolerance ±0.2 µm), was obtained from Instec, USA. The mesogens were injected into liquid crystal cell by capillary action on heating the sample to the isotropic state. The different phase transitions were observed by eye in heating and cooling cycles, while good alignment was achieved by cooling the cell very slowly. The direction of flow of the liquid crystal during cell filling was found to influence the molecular alignment in the resultant liquid crystal layer. Better alignment was achieved when the liquid crystal was allowed to flow into the cell in the direction in which the surface was aligned.

Temperature-dependent textural studies were carried out using a Meopta POM provided with hot-stage, as described by Gray [22], and a high resolution Canon Digital Rebel XS EOS 1000D colour camera. This is a digital single-lens reflex camera with 10.10 megapixel image sensor and incorporates the EOS integrated self-cleaning sensor unit to eliminate dust on the images. The LCD monitor is manufactured at very high precision with over 99.99% effective pixels. With a 10.10 megapixel output, this camera gives true colour reproduction, low noise, a wide dynamic range and good spatial and spectral resolution. The maximum frame rate is high, at 3 frames per second. Live images are displayed on a monitor and are extremely simple and natural. The characteristic colours of microscope samples can be recorded faithfully with true colour reproduction. The ability to capture the colours observed with the microscope accurately enables colour analysis of high precision. Digital cameras tend to have difficulty balancing colours accurately; especially reds and yellows, and images often appear unnatural. To avoid this the camera is designed with a custom white balance, which calibrates the camera to the exact colour temperature of the light illuminating the object [23-26].Textures of the samples were recorded at a cooling rate of 1^oC min, under crossed polarizer's [27,28]. The texture images detected by the camera have a resolution of 2816 × 1880 pixels and the intensity values of the pixels ranges from0 to 255. The image dimensions were selected to be 256 × 256, and the images were stored in the computer for further processing. MATLAB software was used to compute the statistical parameters of the samples such as mean, standard deviation, entropy, energy, contrast and homogeneity [29].

2.1 Theoretical considerations:

Statistical image analysis combines the technique that computes the statistical parameters of the image based on the grey-level intensities of the image pixels. An image (*I*) isof size *m*-by-*n* and it is composed of *m* pixels in the vertical direction and *n* pixels in the horizontal direction; *i, j* are horizontal and vertical coordinates of the image, respectively. The total number of pixels in the image is $m*n= N$, $0 \le i \le m$, $0 \le j \le n$. The defined statistical parameters [30] are as follows and also given in literature [16-20]

(i) Mean:

Mean of an image is simply arithmetic average of the pixel values in the image. This can be obtained by summing up the all pixel values and divided by the total number of pixels.

Mean :

$$\mu = \frac{1}{N}\sum_{i=1}^{m}\sum_{j=1}^{n} I(i,j) \qquad (1)$$

(ii) Standard Deviation:

Standard Deviation of the image is defined as square root of the variance. Variance is nothing but the variation of intensity around the mean intensity value of the image. If the variance value is closer to mean, standard deviation is less. Standard Deviation:

$$S = \sqrt{\frac{\sum_{i=1}^{m}\sum_{j=1}^{n}(I(i,j)-\mu)^2}{N-1}} \qquad (2)$$

(ii) Entropy:

As a statistical measure, entropy is the measure of randomness of the grey levels in an image, that is, it refers to how randomly the grey levels are present in an image. The entropy of an image *I* is calculated by finding the probability *P* of a particular grey-level value found in that image.

Entropy:

$$-\sum_{i=1}^{m}\sum_{j=1}^{n} p_{(i,j)} \log(p_{(i,j)}) \qquad (3)$$

where $p_{(i\ \ j)}$ represents the number of occurrences of gray levels *i* and *j* in the given image.

(iii) Energy:

Like contrast, energy is also a second-order statistical parameter and it measures textural uniformity, that is, pixel pair repetitions. The maximum energy of the texture or image occurs when the grey-level distribution of a given image is either constant or a periodic uniform.

Energy of an image:

$$\sum_{i=1}^{m}\sum_{j=1}^{n} GLCM(i,j)^2 \qquad (4)$$

(iv) Contrast:

Contrast is a second - order statistical parameter and is calculated from the grey level co- occurrence matrix (GLCM) of the texture [31]. The elements of the GLCM represent the relationship between pixel intensity and its neighbouring pixels. The intensity contrast between a pixel and its neighbour is measured by the contrast of texture. The value of the contrast is low if the grey levels of each pixel pair are similar.

Contrast:

$$\sum_{i=1}^{m}\sum_{j=1}^{n}(i-j)^2 GLCM(i,j) \qquad (5)$$

(v) Homogeneity:

Homogeneity measures the closeness of the distribution of values in the GLCM. Homogeneous texture will contain only a limited range of gray levels, giving a GLCM with only a few values but relatively high probability (*P(i,j)*) for the GLCM values. Thus the sum of squares will be high. Energy and homogeneity are similar measures; the only difference is that energy considers the elements of the GLCM and homogeneity considers the probability of GLCM values.

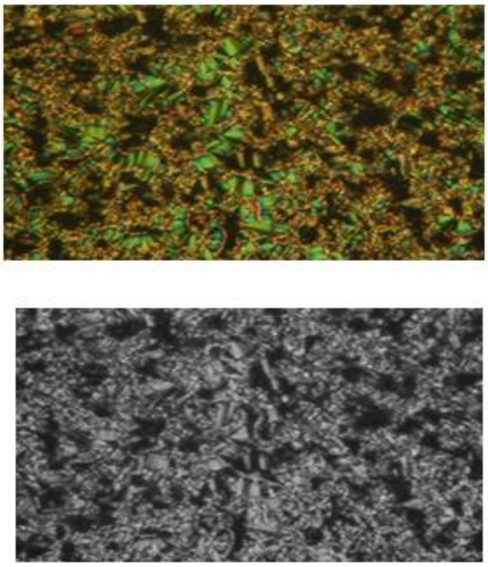
$$Homogeneity = \sum_{i=1}^{m}\sum_{j=1}^{n}(P(i,j))^2 \qquad (6)$$

In liquid crystals, the statistical parameters are assisted for the measurement of transition temperatures by means of statistical image analysis. These parameters are extremely sensitive to changes in the textures of liquid crystals as a function of temperature. MATLAB® software is used to compute the statistical parameters of the image, which is a high-level language and interactive environment that enables to perform computationally intensive tasks faster. Image Processing using MATLAB supports images generated by a wide range of devices, including digital cameras. The standard data and image formats include JPEG, JPEG-2000, TIFF, PNG, etc. MATLAB provides a comprehensive suit of reference-standard algorithms and tools for image analysis tasks, such as statistical analysis, feature extraction and property measurement.

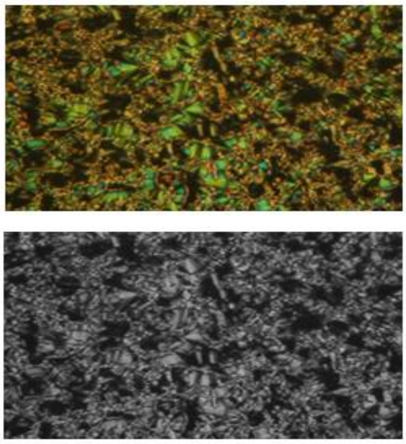
III. RESULTS AND DISCUSSION:

The present analysis consisting of azomethine ester, 4-hexadecyloxy benzyli
dene-4'-bromoaniline (16ABBA) possess-
ing even number of carbon atoms at the terminal chain ($C_{n-1}H_{2n-1}COO$- where n= 16) is characterized. Textures of 16ABBA liquid crystals are recorded using POM in arthroscopic mode. The recorded textures of the samples are stored in computer for further processing. MATLAB (implemented on P4 1.6GHz with 512MB RAM computer) is used to read and translate the image into gray scale version, and to compute the statistical parameters. The textures are captured as a function of temperature, till the sample temperatures reach to the isotropic state from solid. On heating, 16ABBA undergoes a phase transition from the crystalline state to the isotropic state via an intermediate Smectic A phase between 50 ^{o}C and 94.5 ^{o}C. The sample will becomes isotropic at 107.1 ^{o}C. while cooling the along with Smectic A phase at 84.1 ^{o}C one more phase i.e., Smectic B phase occurs at 70.7 ^{o}C.

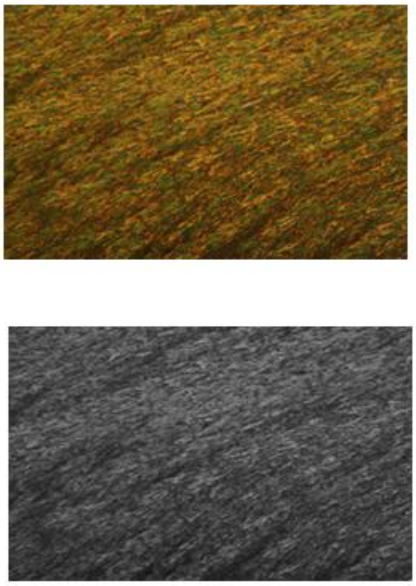
Textures of 16ABBA original and its translated version of gray colour at different temperatures are shown in Fig. 2.



Fig(2a)



Fig(2b)



Fig(2c)

Figure 2. (a) Smectic A texture of the 16ABBA original and its translated version (grey colour) at 80.4^{0}C.
(b) Smectic B texture of the 16ABBA original and its translated version (grey colour) at 70.7^{0}C.
(c) crystal texture of the 16ABBA original and its translated version (grey colour) at 49.4^{0}C.

The captured images or textures are stored in computer for further processing. Read the image from database, through preprocessing and it represents the image as a matrix where every element of the matrix has a value corresponding to bright/dark pixel at the corresponding position or feature of an image. The statistical parameters of the textures are computed using equations 1 to 6 with the help of MATLAB Software. Once the parameters are computed, the plots are drawn for parameters as a function of temperatures and are shown in **Figs.** 3(a) to.3(f).

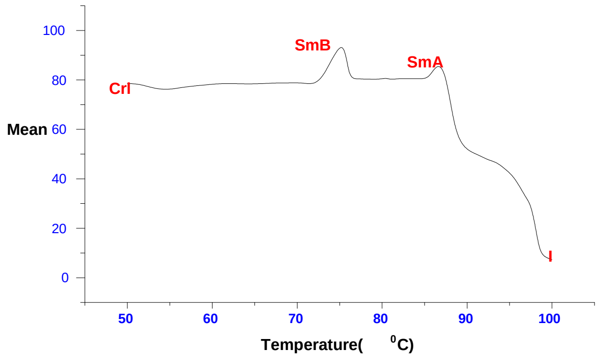
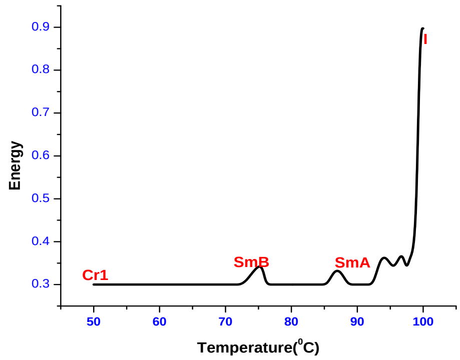
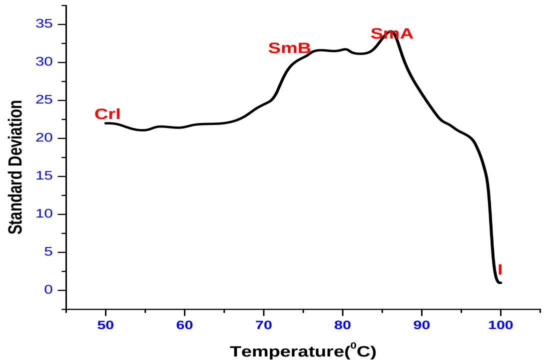


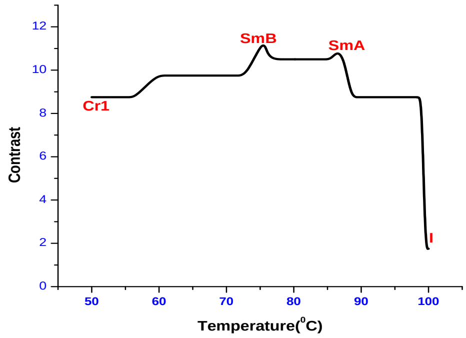
Fig (3a)



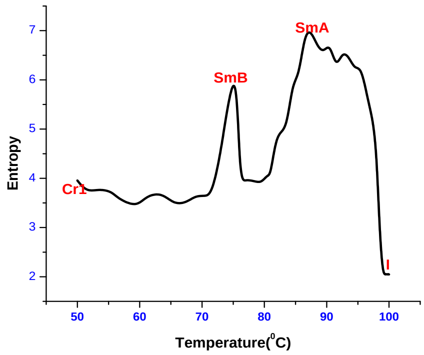
Fig(3d)



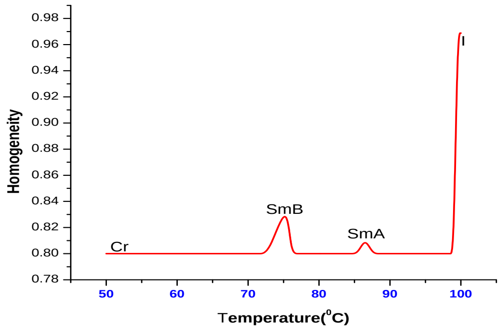
Fig(3b)



Fig(3e)



Fig(3c)



Fig(3f)

Fig. 3: Statistical parameters (3a)mean, (3b) standard deviation, (3c)entropy, (3d) energy, (3e) contrast, (3f) Homogeneity as a function of temperature where the phase transitions are indicated for *n*ABBA(where n=16) (Cr: crystal,SmB: SmecticB, SmA: smectic A, I: isotropic).

Table 1: Statistical parameters at existed phases in 16ABBA liquid crystal

n=16	Sm A	Sm B
Mean	87	99.6
Standard deviation	34.5	30.8
Entropy	6.9	6.7
Energy	0.33	0.36
Contrast	10.9	11.5
Homogeneity	0.8	0.54

The abrupt changes in the statistical parameter with temperature indicate the consequent changes in the features of the textures of samples which are due to the phase transition of material. The distinctive and abrupt changes observed in the curve at temperatures corresponding to the transition temperature of samples [32] make the proposed methodology a very sensitive method for the identification of phase transitions of liquid crystals. The behaviour of statistical parameters curves of thermotropic liquid crystals is different. Since energy is a measure of uniformity in the texture and entropy is measure of randomness of the texture, the entropy and energy of the compounds show the opposite behaviour (Fig.3). Fig.3a shows mean intensity levels of the present sample is varying linearly with rise of temperature except at phase transition, and the reason for such change in intensity occuring oscillations due to change in phase retardation. Standrd deviation (Fig. 3b) and entropy (Fig. 3c) are high at SmA phase compared to crystal phase and SmB as the order of the molecules are more. Entropy (Fig.3c) levels are high at Sm B and Sm A phases. Further comparing with Sm B, Sm A phase has more value due to decrease in polarizability and lower thermal stability because of very weak intermolecular inductive interactions. Entropy (Fig.3c), and energy (Fig.3d) shows opposite behavior while homogeneity (Fig. 3f). From Fig.2e, contrast levels are high at phase transitions Sm B and Sm A phase transitions due to dipole-dipole interactions.

The present sample under study, 4-hexadecyloxybenzylidene-4'-bromoaniline consists of Smectic A and Smectic B phases is due long aliphatic chains and amphiphilic interactions in the system. During heating endothermic reaction exists, while in cooling process exothermic reaction took place. The transitional entropy between the liquid crystalline state to isotropic state are related to the degree of internal order present in the system. Relatively small entropy change shows that the liquid crystalline states are associated with more subtle structural changes.

From Fig.1 if we observe the structure of the sample, there is an introduction of bromo compound in the para position of benzene ring which increases the dipole moment and mesophase thermo stability. As the sample contains bromine which is a halogen, bonds are strong, specific, and directional interactions that had given rise to well-defined structures. Polymorphism arised between the transformations of crystal to isotropic fluid due to weak intermolecular forces that imparts the equilibrium, results in well defined phase changes. The energy levels vary at phase transition due to intermolecular inductive interactions. Low thermal stability arises between SmA and isotropic phase due to decrease in polarizability [33] The present compound contains 4-alkoyloxy group hence thermal stability is high due to permanent dipolemoment [34] enhanced planarity of the molecule. Hence increased lateral forces favours smectic phase [35].

With the increase of temperature, transmission of the liquid crystal increases due to decrease in order parameter. This results in smectic phases in low transparency. As the temperature increases, intensity exhibits oscillations due to change in phase retardation. Entropy change determines the degree of order present in a system.[36]. Smectic A phases are the least ordered of all smectic phases found at the high temperature end of the smectic range. The molecules are arranged in layers and have their long axis on the average perpendicular to the layer plane [37,38]. In general, all smectic A phases studied are found to be optically uniaxial and frequently they show the pseudo isotropic or homeotropic texture in which the smectic layers are parallel to the supporting surface and the optic axis is perpendicular to it. The ordered arrange-ment of molecules appears to make the layers of smectic B much more rigid than smectic A [39] leading to frequent appearance of mosaic textures for these phases. It seems that smectic B phase is a soft solid with three dimensional ordering of finite range.

Anisotropic dispersion interactions, and consequently the anisotropy of polarizability, depending on the electron density distribution in the groups under consideration, also influence the packing and hence the stability of the mesophases but play a secondary role compared to the steric factors [40]. Other molecular aspects such as the association [42] or dipole-dipole attraction in polar liquid crystalline derivatives, which can influence the packing of the molecules, also affect the stability of the mesophases [35]. Particularly, in per bromoalkyl alkyloxy group [43-45] promotes microphase segregation and strong electrostatic interactions that can be responsible for the pronounced layer arrangements of molecules and phase formation.

DSC thermogram of 16ABBA liquid crystal is shown in Fig. 4 in both heating and cooling cycles. The transition temperatures are given in Table 2. To compare the phase transitions obtained from image analysis and DSC is also given Table 2. It is observed that the transition temperatures obtained in both the techniques is in good agreement.

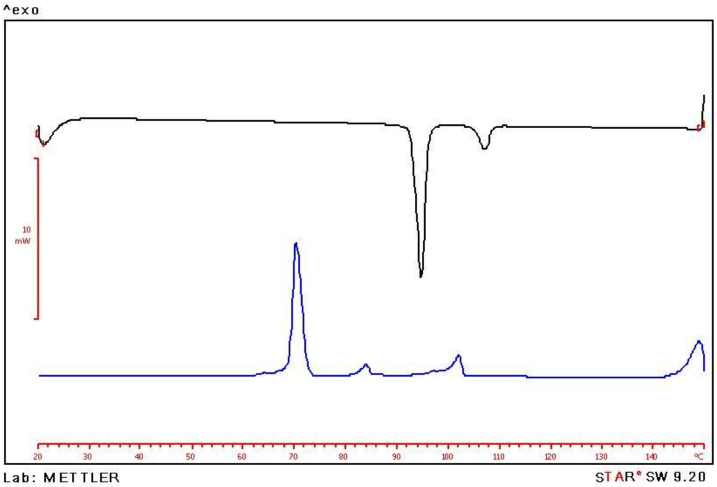


Fig. 4. DSC thermogram of 16ABBA

Table .2 Phase transition temperatures of 16ABBA

16 ABBA	SmA	SmB	Cr
POM (H)	107.1 °C	-	94.5°C
DSC (H)	107°C	-	94°C
POM (C)	84.1 °C	70.7°C	102.1°C
DSC (C)	84°C	70°C	102°C

H-Heating, c- Cooling

IV. CONCLUSION

The present study demonstrates the computation of statistical parameters through image analysis in conjunction with polarizing optical microscopy is a reliable and sensitive technique to identify the phase transition temperatures of thermotropic liquid crystals. Small changes in the textural features have brought variations in the transmitted intensity or gray values, which are useful to compute the desired parameters. Temperature corresponding to abrupt changes in the curve represents the transition temperature of sample.

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