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Hydrothermally Produced Cobalt Oxide Nanostructures at Different Temperatures and Effect on Phase Transition Temperature and Threshold Voltage of Nematic Liquid Crystal Host

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Abstract

In this study, cobalt oxide Co_3O_4 nanostructured material is synthesized by hydrothermal method by using different concentration of cobalt acetate salt at unique hydrothermal reaction time for five different hydrothermal reaction temperatures 105, 120, 140, 160, and 180°C. The obtained nanoparticles are annealed at 300°C for 5 h. Co_3O_4 nanostructures are determined by means of scanning electron microscopy (SEM), X-ray diffraction (XRD), and UV-vis spectroscopy. The hydrothermally produced samples were reannealed at 550°C, and morphological and structural properties were investigated deeply again. The effect of annealing temperature on morphologies and crystalline structure of cobalt oxide nanoparticle (NP) was also investigated. Nanopyramids and nanorods are two main morphologically obtained structures from the hydrothermal experiment. Nematic liquid crystal mixture E7 is doped with Co_3O_4 nanorod. Phase transition and threshold voltage of pure and Co_3O_4 NP-doped E7 LC are examined successfully. It reveals that for Co_3O_4 NP-doped E7 phase transition temperature and threshold voltage increased very slightly.

Keywords: cobalt oxide, hydrothermal synthesis, band gap, threshold voltage, thermal property

1. Introduction

“There is Plenty of Room at the Bottom” a titled lecture in 1959 was given by Richard Feynman, and this has opened a new era in the field of nanotechnology [1, 2], which can be

defined as the engineering of functional systems at the molecular level [3]. Nanoscience and nanotechnology have been growing rapidly due to the fact that nanomaterials are synthesized with new strategies and those synthesized materials are characterized and manipulated with new tools [4]. Nanostructures can be classified into three different categories according to their size and shapes: zero dimensional (0D), one dimensional (1D), and two dimensional (2D) [5, 6]. Nanodots-nanospheres, nanowires-nanorods, and nanosheets-nanoplates are some examples of different shapes and size nanomaterials for this classification. Nanomaterials, unlike to conventional materials, can be physically and chemically manipulated and used in different areas [7, 8] including biology and medicine [9–11], water treatment [12], electronics [13, 14], and optics [15–17].

Magnetic fields more or less impact a certain subclass of NP, which is called as magnetic nanoparticles (MNPs) [18, 19]. MNPs have their own special properties, such as superparamagnetism [20], high mass transference [21], and high field irreversibility [22]. The second most popular MNP, considering to application areas, is cobalt oxide (Co_3O_4) since these nanoparticles are used in various fields from micro-electronics to drug delivery [7]. Co_3O_4 is one of the significant transition metal oxides, and the direct optical band gap of bulk Co_3O_4 is 2.19 eV.

Dispersion of nanomaterials into liquid crystals (LCs) has been a topic of great interest in recent years. There are many distinguished researchers investigating liquid crystals, and some of whose works are focused on doping nanomaterials into liquid crystals [23–29]. Gold, silver, zinc oxide, carbon, and quantum dots nanoparticles are some of the examples of guest nanoparticles. Co_3O_4 nanomaterials doped nematic liquid crystals (NLC) have only been investigated for spherical morphological Co_3O_4 nanoparticles to our knowledge [30].

Researchers have focused on synthesizing Co_3O_4 nanoparticles not only with diverse morphology but also with different methods since any change in production methods, particle size, shape, and structure of Co_3O_4 nanomaterials could lead producing new nanomaterials for potential applications with unique properties. Up to now, Co_3O_4 nanoparticles with various morphologies such as nanospheres [31, 32], nanorods [33–36], nanowalls [37], nanoneedles [38], nanobelts [39], nanowires [40], nanoflowers [41], nanotubes [42, 43], nanofibers [44], nanodiscs [45], and nanochains [46] have been synthesized with different approaches, including urea precipitation [47], chemical vapor deposition [48], sol-gel [49], microwave-assisted process [50], wet chemical approach [51], and hydrothermal method [39, 52–54]. Hydrothermal methods are widely used to produce various different Co_3O_4 nanomaterials. Easily scaling up to industrial demand, requiring low temperature, no need for calcination, being inexpensive, and having a fairly uniform particle size and morphology are some of the advantages of this nanoparticle production method [55]. The term hydrothermal is used when water is used as a solvent, and solvothermal is used when organics are used as solvent [56]. In literature, the effect of hydrothermal reaction times and reaction temperature on morphologies of Co_3O_4 nanoparticles was examined [57–59].

In this study, new morphologies of Co_3O_4 have been synthesized by hydrothermal method using Cobalt(II) chloride hexahydrate precursor at different unique temperatures and hydrothermal reaction times. The influence of hydrothermal temperatures on crystal structures and particle morphologies was examined deeply. Five different hydrothermal temperature points were selected between 105 and 180°C, and the effect of annealing temperatures was also

investigated. The obtained particles were firstly annealed at 300°C for 5 h and then reannealed 500°C. The prepared Co_3O_4 NPs were characterized by XRD, SEM, and UV-vis. Moreover, we selected Co_3O_4 NPs obtained at 120°C hydrothermal reaction temperature to investigate how nanorod morphological Co_3O_4 NPs affect nematic liquid crystal phase transition and threshold voltages.

2. Materials and methods

2.1. Synthesis of Co_3O_4 NPs

All chemicals were used without further purification. A schematic diagram of the synthesis step is given in **Figure 1**. For the synthesis of the samples, 4.3983 g Cobalt(II) chloride hexahydrate

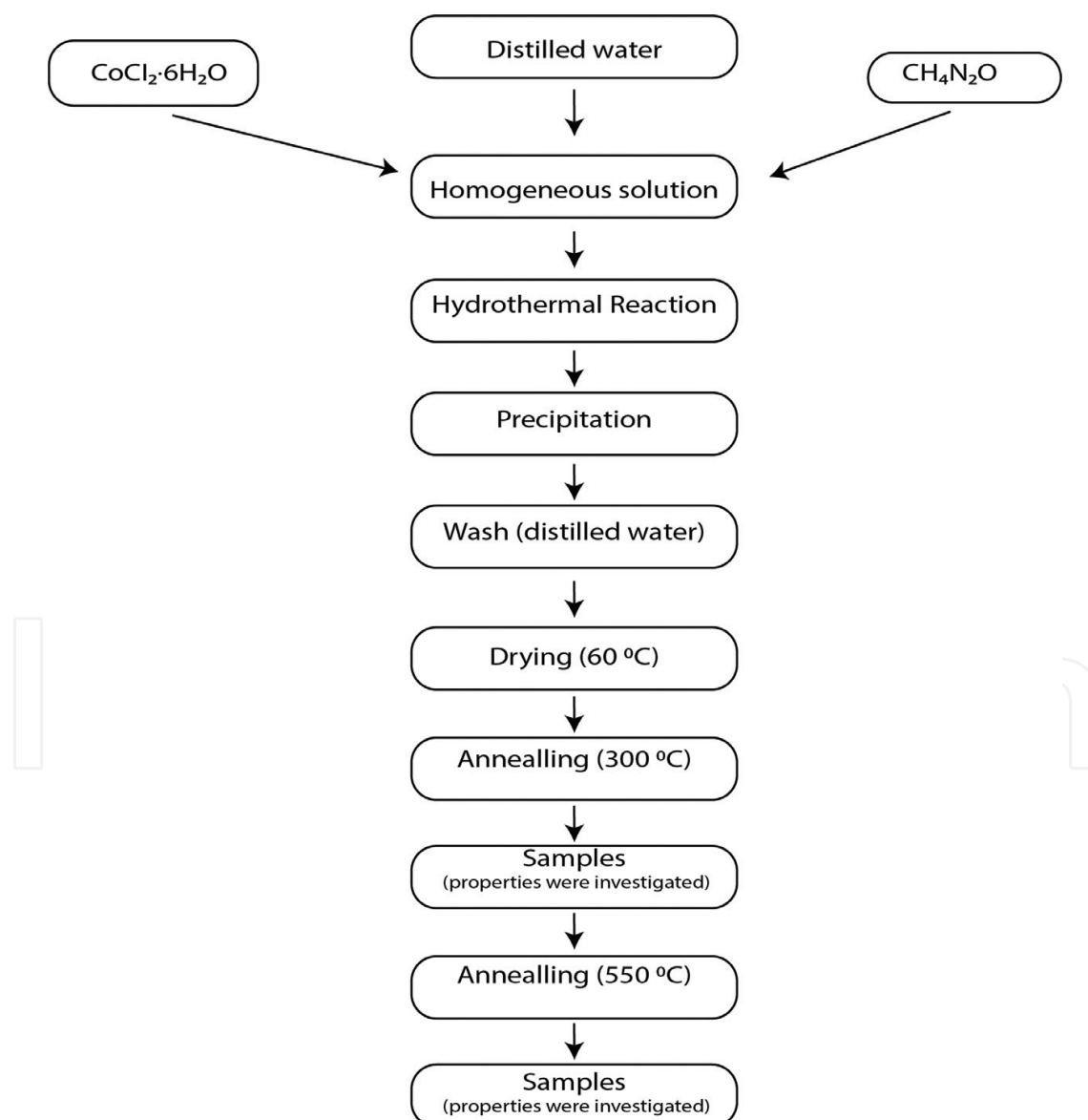


Figure 1. Schematic diagram for the synthesis procedure of Co_3O_4 .

and 0.12 g Urea ($\text{CH}_4\text{N}_2\text{O}$) solution were mixed with 50 ml distilled water. The prepared solution was placed in the autoclave, and it waited in the furnace at 105, 120, 140, 160, and 180°C for 6 h. It was precipitated and washed several times with distilled water and dried at 60°C for 10 h. The obtained particles were annealed for 5 h at 300°C. Moreover, all obtained samples were also annealed at 500°C for 1 h to compare the effects of different annealing temperatures on XRD and SEM results of obtained Co_3O_4 samples.

2.2. Liquid crystal experiment

The LC used was commercially available eutectic mixture E7 (SYNTHON Chemicals, Germany). The nematic to isotropic transition temperature for pure E7 is measured by using polarizing optical microscope integrated with hot stage, and it is found 58.6°C. The liquid crystal cells were made by indium-tin oxide-coated optical glass plates with a planar alignment layers. The thicknesses of cells were about 8 μm and an effective area of 1 cm^2 . The cells were purchased from Instec, Inc. Hydrothermally produced Co_3O_4 sample at 120°C was selected to mixed in host E7 since the morphology of this sample contains nanorods. E7 nematic LC mixture and Co_3O_4 nanoparticles were dissolved in isopropanol followed by the ultrasonic bath. The mixture was left for 48 h in the furnace at 50°C to fully evaporate isopropanol. 0.05% doped sample was filled to LC cell at 60°C by capillarity action [25].

2.3. Characterization

The hydrothermally obtained Co_3O_4 samples' crystalline structures were investigated by a Philips X' Pert Pro X-ray diffractometer (XRD) with $\text{Cu-K}\alpha$ radiation. The morphologies of the samples were examined by scanning electron microscope (Zeiss EVO 10LS). The optical

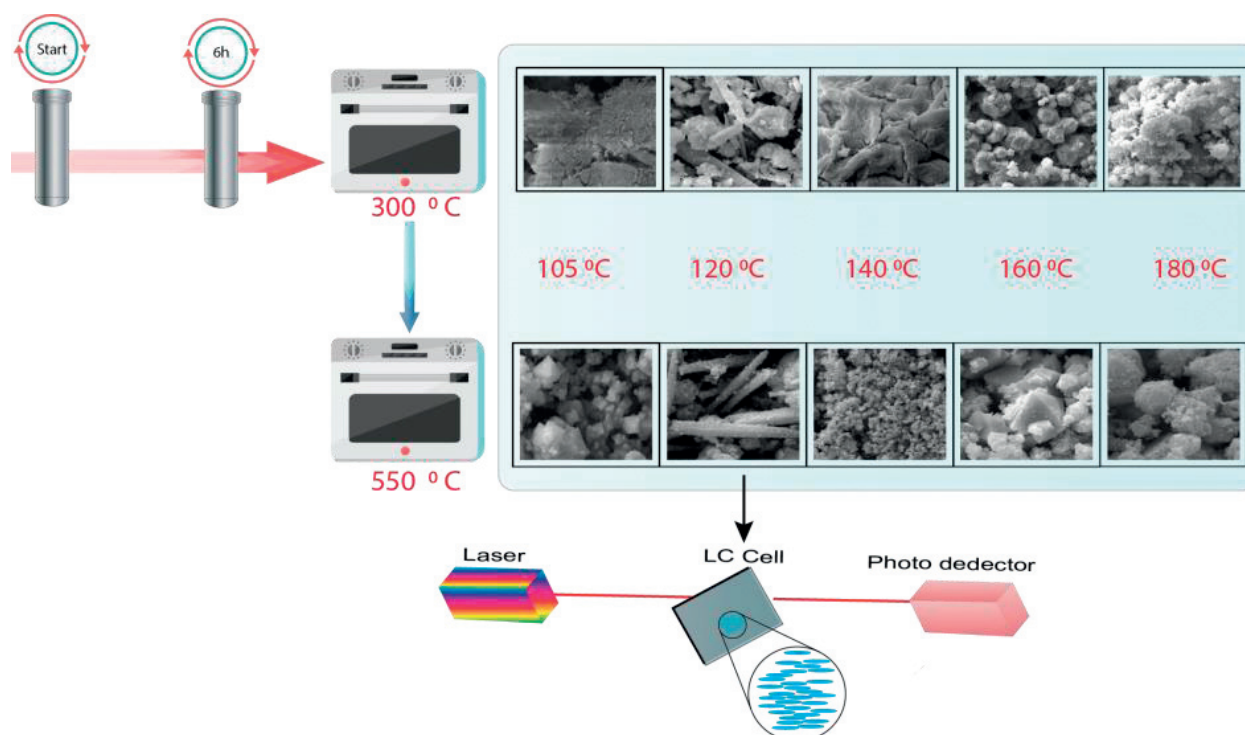


Figure 2. Synthesis of Co_3O_4 samples and basic setup of LC light transmittance experiment.

property samples were obtained by a Shimadzu UV-1800 ultraviolet visible spectroscopy (UV-vis) in the range of 200–900 nm using distilled water as a reference solvent. The samples were dispersed into distilled water solvent and ultrasonicated before the measurement. The textures of E7 and E7-doped sample were taken using polarizing optical microscope (Eclipse E200, Nikon, Japan) equipped with the digital camera. Temperature was controlled with heating stage (LTS 120, LinkamScientific Instruments, Ltd., England) with a temperature accuracy of $\pm 0.1^\circ\text{C}$ controlled with PE95 LinkPad. Optical transmittance experiment designed and performed by laser, polarizer, analyzer, and photodiode and experimental scheme is described in detail in the literature [60]. Experimental detail about synthesis of Co_3O_4 samples and basic setup of liquid crystal optical transmittance experimental is given in **Figure 2**.

3. Results and discussion

X-ray diffraction pattern of Co_3O_4 samples annealed at 300°C for 6 h is given in **Figure 3**. The diffraction peaks of hydrothermally produced cubic structured sample at 120°C are suitable with the values in the standard card (PDF-2, reference code: 01-074-1656) and the Co_3O_4 particles produced at 180°C are partly suitable to this reference code as shown in **Figure 3**. The others produced Co_3O_4 particles' apparent crystal structure patterns were not observed. It is supposed that this was originated from hydroxide structures and chemical contaminant.

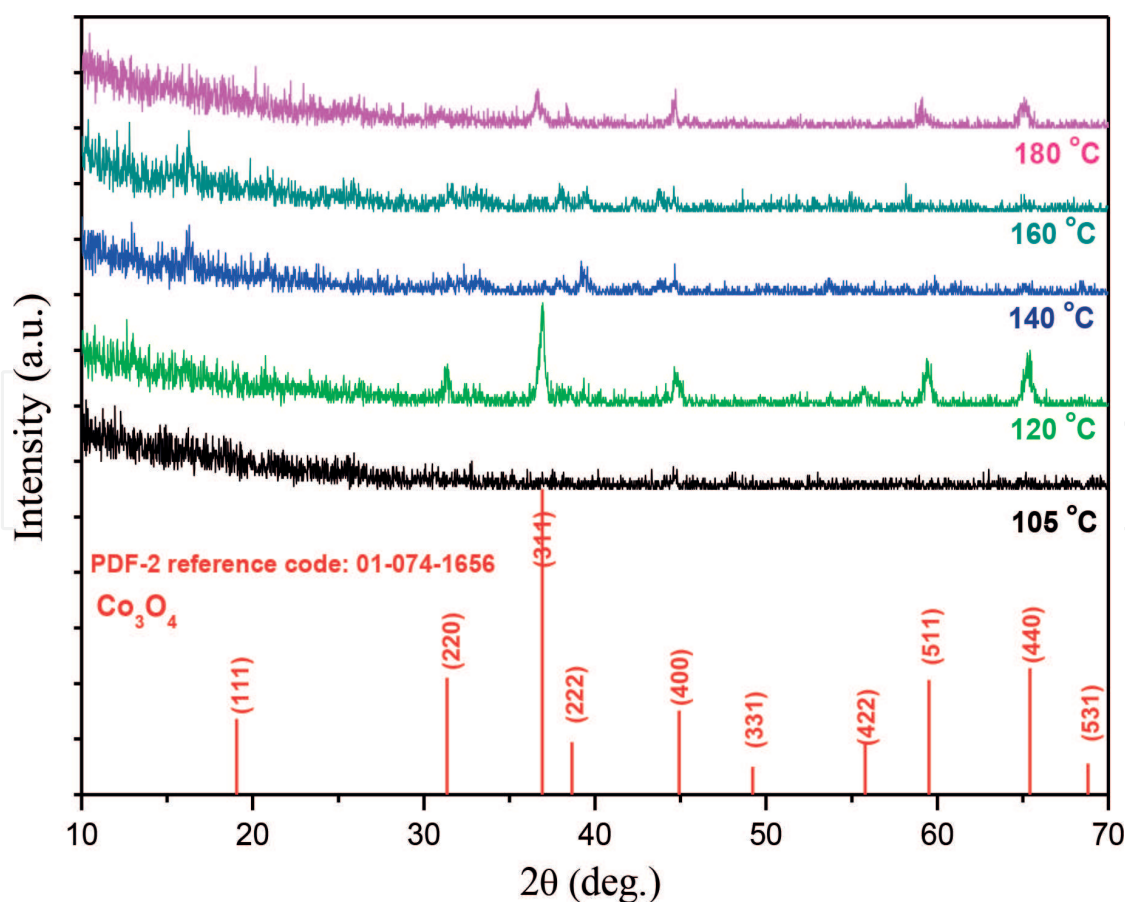


Figure 3. XRD graph of Co_3O_4 samples annealed at 300°C .

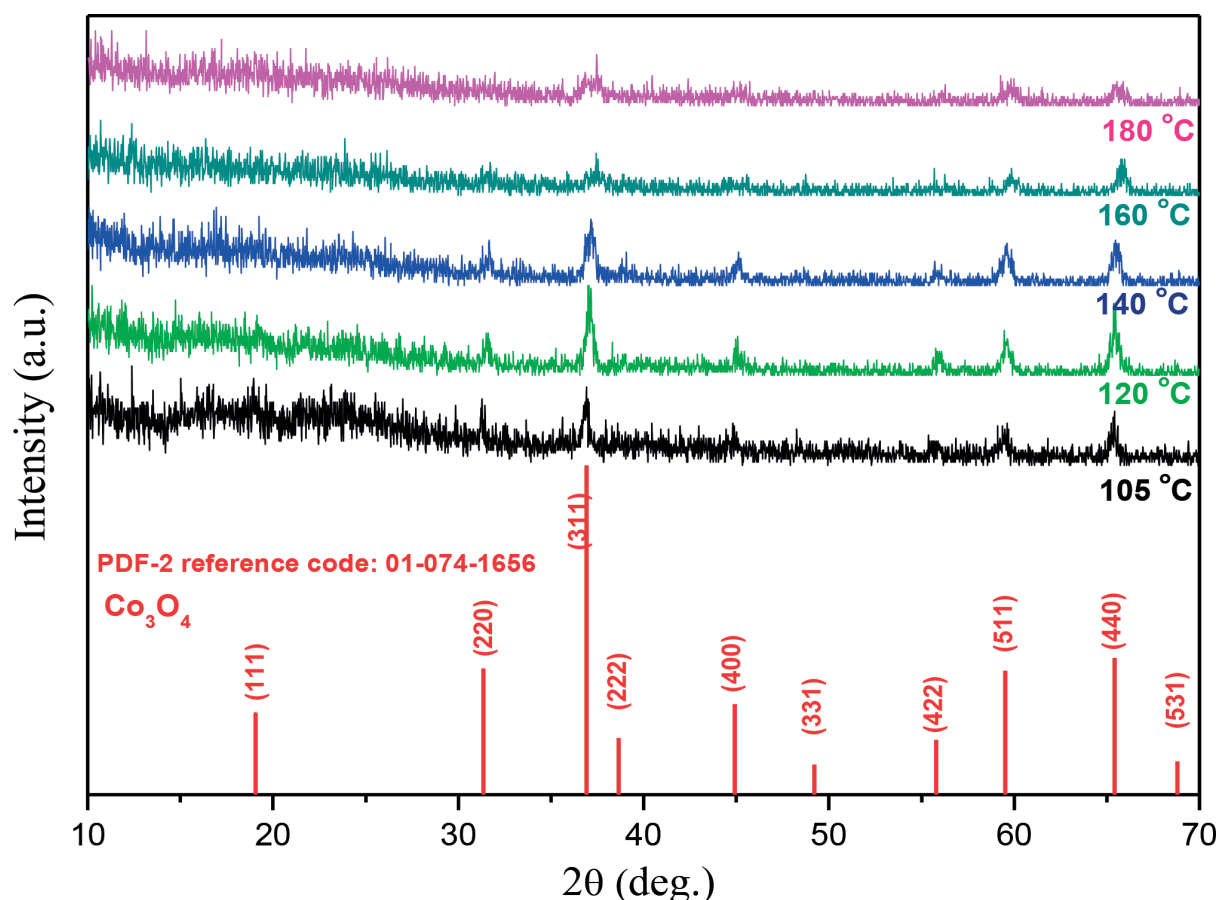


Figure 4. XRD graph of Co_3O_4 samples annealed at 550°C .

The obtained Co_3O_4 particles reannealed at 550°C for an hour and crystal structure of reannealed samples were investigated again. The effect of annealing temperature on the crystalline structure of obtained particles is shown in **Figure 4**. In this figure, XRD peaks of Co_3O_4 particles become clear, and all samples give at least some of the reference peaks of Co_3O_4 . Hydroxide structures disappeared with reannealing at 550°C , and XRD peaks of Co_3O_4 particles are seen more clearly than samples annealed at 300°C .

The morphologies of particles that are produced using hydrothermal method at various temperatures and annealed at 300°C for 5 h are seen in **Figure 5**. In this figure, hydroxide and chemical waste structures are observed. Especially in **Figure 5b**, nanorod structures are seen. Nanosheet structures as layers are seen in **Figure 5d**. However, remarkable structures have not been observed for the samples produced at 105, 140, and 180°C , which are corresponding to **Figure 5a, c, and e**.

To observe the effect of annealing temperature, SEM pictures belonging to particles annealed within 550°C for 1 h are given in **Figure 6**. It was observed that the hydroxide compounds and chemical wastes decreased with the effect of the annealing. In **Figure 6a and d**, octahedral microparticles have been observed clearly, and in **Figure 6b**, nano-/microrods have been shown. **Figure 6c** shows the octahedral particles in nano level. In **Figure 6e**, agglomerated nanoparticles are dominantly seen.

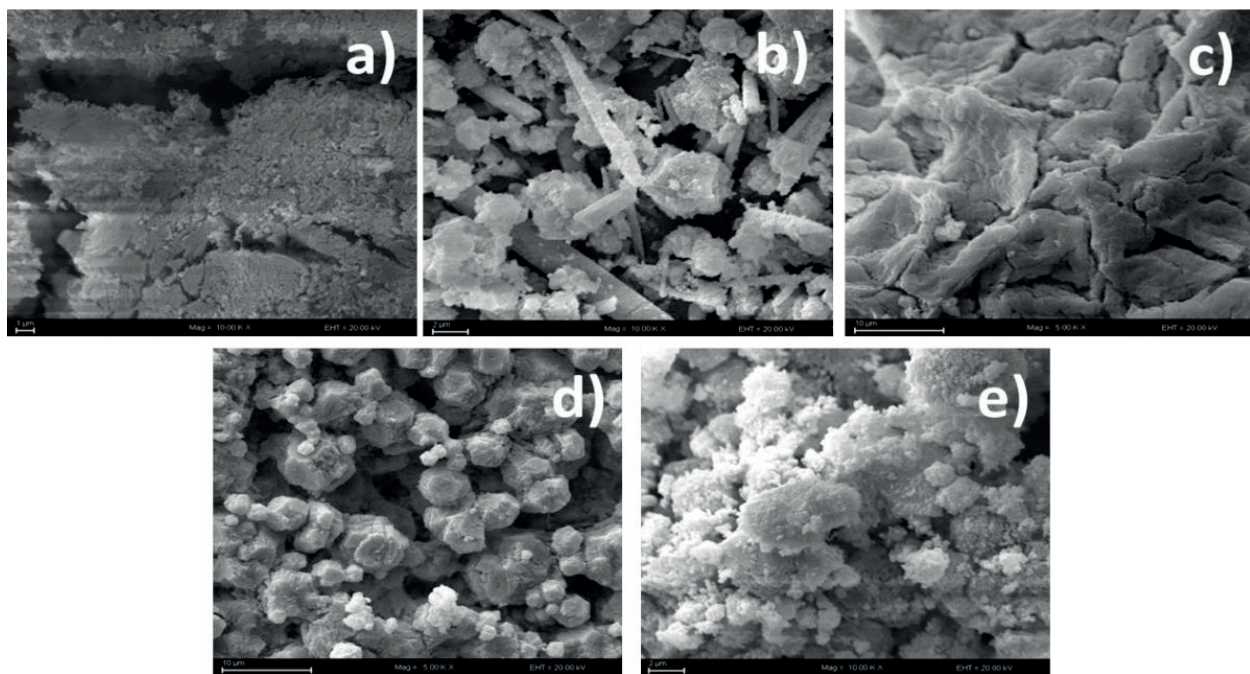


Figure 5. Morphologies of 300°C annealed samples produced at (a) 105°C, (b) 120°C, (c) 140°C, (d) 160°C, and (e) 180°C.

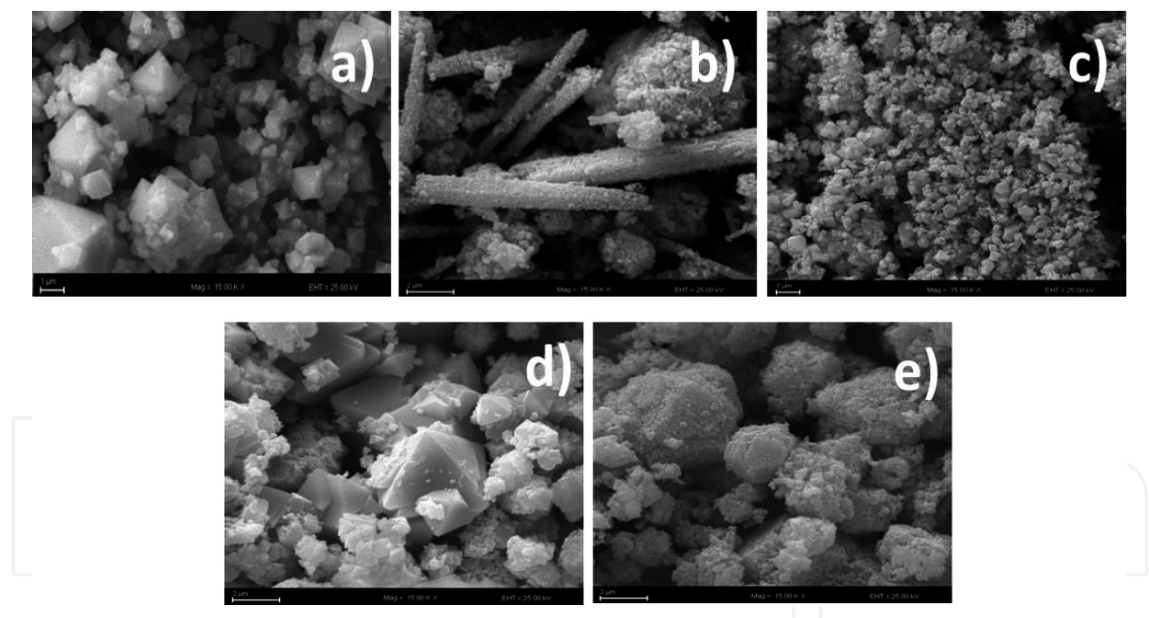


Figure 6. Morphologies of 550°C annealed samples produced at (a) 105°C, (b) 120°C, (c) 140°C, (d) 160°C, and (e) 180°C.

The optical band gap (E_g) for the direct transition was obtained using Tauc plot. Optical absorption graph of samples annealed at 300°C is given in **Figure 7**, left column and optical absorption of samples reannealed at 550°C is given in **Figure 7**, right column. The direct band gap of 300°C annealed Co_3O_4 NP changing between 3.1 and 3.5; on the other hand, reannealed samples band gaps were very close to 3.5 eV as interpreted in **Figure 7**. The measurement results of direct band gaps of reannealed nanoparticles were suitable with the literature [61, 62].

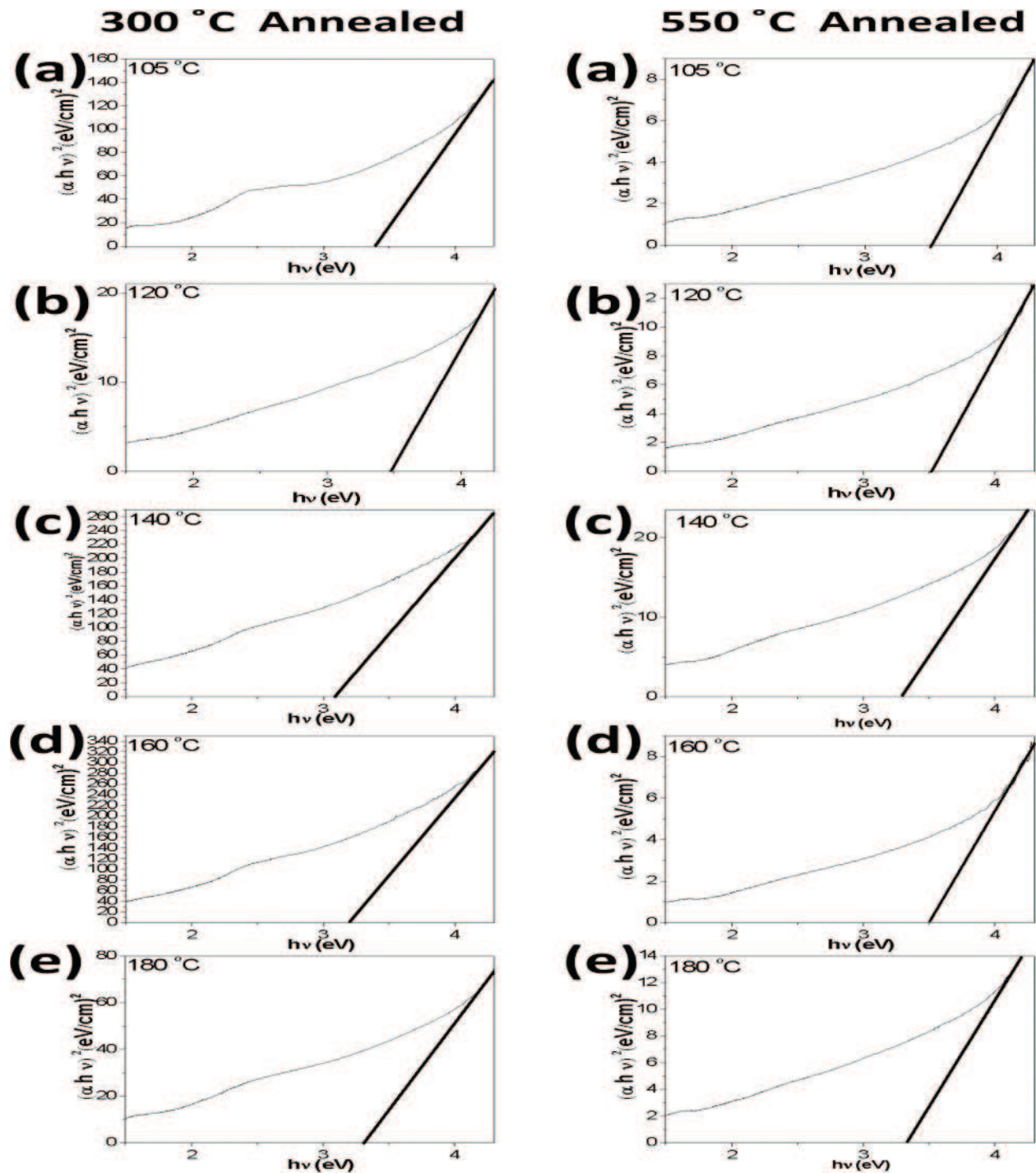


Figure 7. UV-vis spectra and band gaps of hydrothermally obtained particles annealed at 300 and 550 °C.

Polarizing optical microscope was used to investigate texture of pure and 0.05% nanoparticle-doped E7. The different magnification textures of pure E7 are given in Figure 8. The smallest magnification ratio of pure E7 is given in Figure 8a, and the most detailed texture of this LC is shown in Figure 8d.

Phase transitions of 0.05% cobalt oxide nanoparticle-doped E7 LC are illustrated in Figure 9a. The first drop of isotropic liquid appeared at 57.1 °C, T_N , and the last drop nematic disappeared

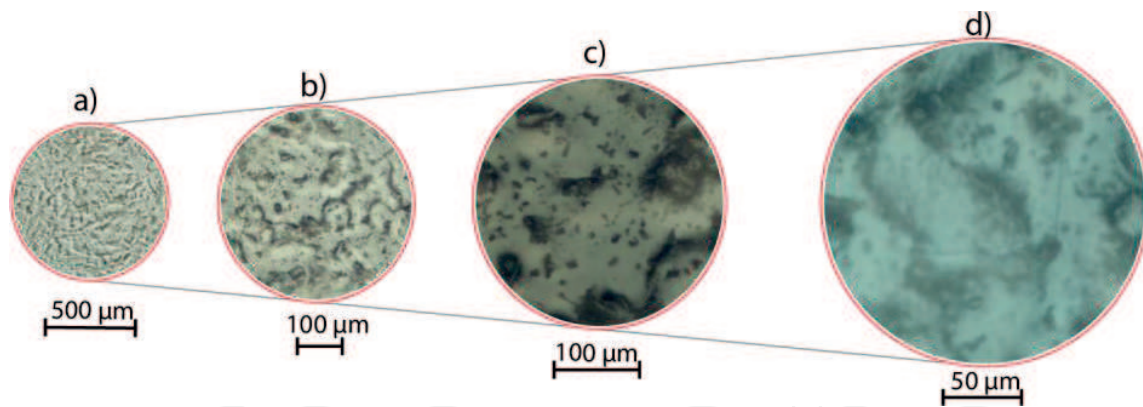


Figure 8. Optical polarizing image of E7 nematic LC, (a) 40× magnification, (b) 100× magnification, (c) 200× magnification, and (d) 400× magnification.

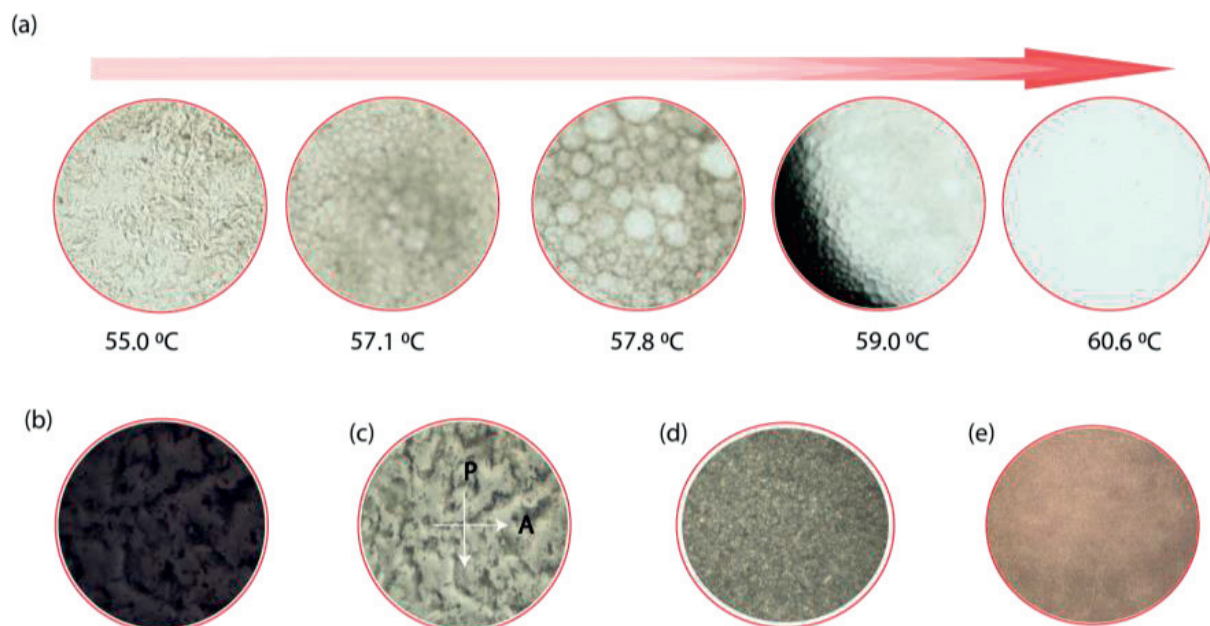


Figure 9. Texture of pure and NP-doped E7, (a) phase transition, (b) without cross polarizer, (c) with cross polarizer, and (d) E7 at 1 V, (e) 0.05% NP-doped E7 at 1 V.

at 60.6°C, T_I . The average temperature of nematic-isotropic transition of doped sample is calculated by using equation $T_{NI} = 0.5(T_N + T_I)$ [63], and found 58.85°C. The phase transition difference of pure E7 and NP-doped E7 is found as $\Delta T_{NI} = 0.2^\circ\text{C}$. **Figure 9b** and **c** shows the optical picture of E7 LC without and with cross polarizers, respectively. The texture of pure and doped LC under 1 V applied voltage is given in **Figure 9d** and **e**.

Transmission versus voltage graph of pure and Co_3O_4 NP-doped LC is illustrated in **Figure 10**. The wavelength of used laser light in transmission experiment was 650 nm. The prepared LC cells are placed between cross polarizers, and the angle between cross polarizers and LC cell is adjusted to 45° , and the output signal was detected by a photodiode detector. Transmission voltage behavior of pure and doped sample is not very different from each other, which

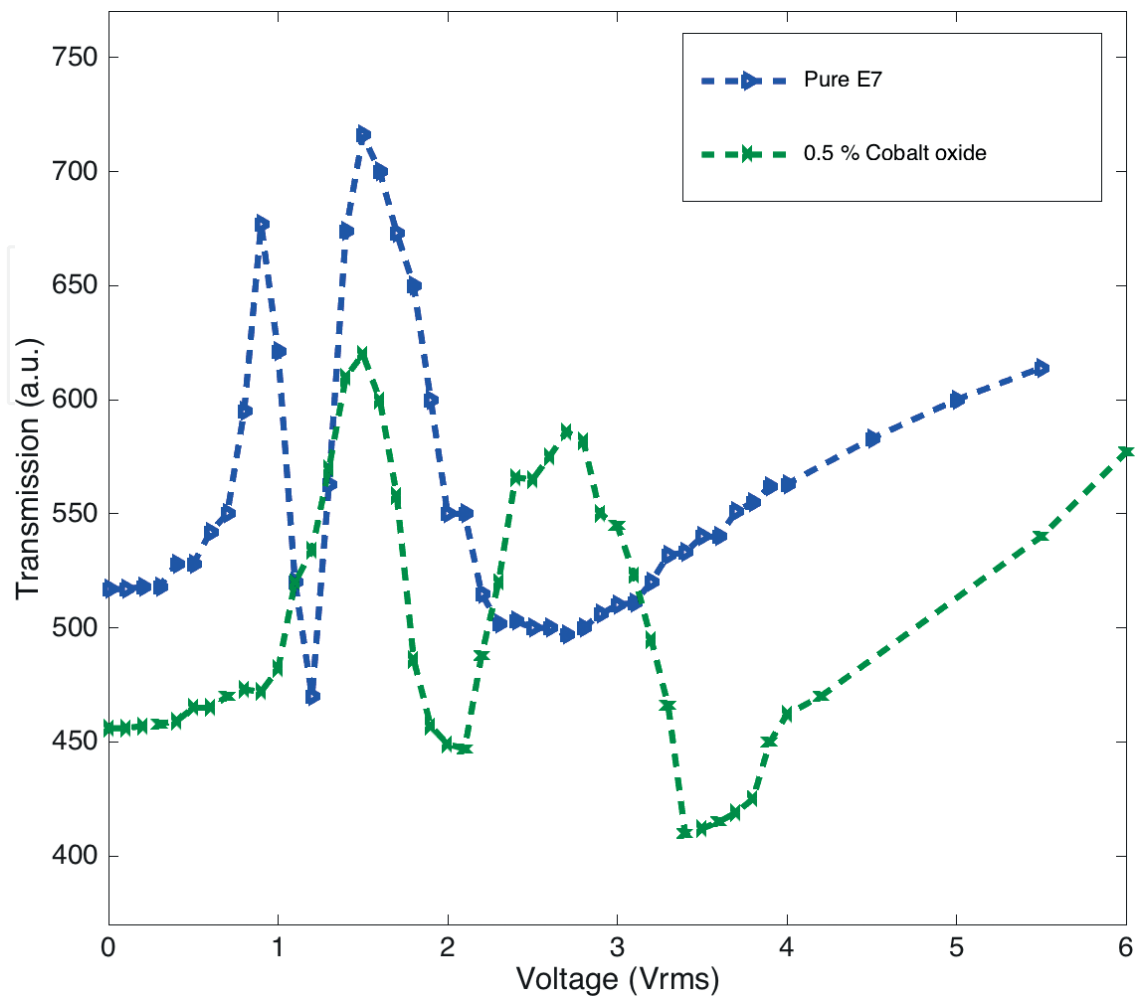


Figure 10. Transmission vs. voltage graph for pure and NP-doped E7.

implies that threshold voltage values are close to each other. The calculated threshold voltage for pure E7 and 0.05% Co_3O_4 NP-doped E7 are 0.8 V and 0.9 V sequentially.

4. Conclusion

In summary, Co_3O_4 nanostructures were synthesized using the hydrothermal method with unique hydrothermal reaction time at different temperatures. The crystalline structures, morphologies, and optical absorptions were investigated in detail with XRD, SEM, and UV-vis spectroscopy for two different annealed temperatures. The obtained samples were firstly investigated after being annealed at 300°C for 5 h, and the results of SEM and XRD separately indicated that cobalt hydroxide did not decompose fully to form cobalt oxide nanocrystalline. The samples were reannealed at 500°C for an hour to investigate deeply. The hydroxide compounds and chemical wastes are removed, and XRD peaks of Co_3O_4 particles become clear. Obtained morphologies of cobalt oxide nanostructures also changed with calcination. The particles, with a nanorod morphology, produced at 120°C hydrothermal reaction temperature and annealed at 500°C were selected to doped nematic LC mixture E7. Phase transition

and the threshold voltage of pure and Co_3O_4 NP-doped E7 LC were examined successfully. It reveals that for Co_3O_4 NP-doped E7 phase transition temperature and threshold voltage increased very slightly.

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