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The Role of Thermotropic Liquid Crystals in the Detection of Gases that Adversely Affect the Nervous System

Sinir Sistemine Olumsuz Etki Eden Gazların Belirlenmesinde Sıvı Kristallerin Rolü

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ABSTRACT

In this study, the aim was to develop an innovative and user-friendly sensor capable of quickly detecting the gases phenol, toluene, and 1,2-dichloropropane, which negatively affect brain functions when inhaled over extended periods. To achieve this, the thermotropic phase of liquid crystals, which possess thermotropic and lyotropic phases, was used, and a thermotropic cholesteric liquid crystal (CHLC) sample was formulized. The changes occurring in the presence of these gases were examined optically. In this context, the thermotropic ChLC sample, composed of E7 thermotropic liquid crystal and a chiral mixture made from chiral dopants CB15, R1011, and R811, was placed into a cell created using the sandwich method with glass surfaces modified with lecithin. The mentioned gases, evaporated at different temperatures, were introduced into the cells containing the liquid crystal sample over varying durations, and the resulting changes in the wavelength of the liquid crystal were analyzed. Thus, it was demonstrated that these gases, which have a detrimental impact on human health, can be detected at the lowest concentration levels using a sensor containing thermotropic liquid crystal.

Key Words

Gas sensors, liquid crystals, optical sensors.

ÖΖ

Bu çalışmada, uzun süre solunduğunda beyin fonksiyonlarına olumsuz etki eden fenol, toluen ve 1,2-dikloropropan gibi toksik gazları hızlı bir şekilde tespit edebilen yenilikçi ve kullanıcı dostu bir sensör geliştirilmesi amaçlanmıştır. Bu doğrultuda, termotropik ve liotropik fazlara sahip sıvı kristallerin termotropik fazı kullanılmış ve termotropik kolesterik sıvı kristal (ChLC) örneği formüle edilmiştir. Bu gazların varlığında, sıvı kristallerin yoneliminde meydana gelen değişimler optik olarak incelenmiştir. Bu bağlamda, E7 termotropik sıvı kristali ve kiral dopanlar CB15, R1011 ve R811'den oluşan bir kiral karışım kullanılarak hazırlanan termotropik ChLC örneği, lesitin ile modifiye edilmiş cam yüzeylerle sandviç yöntemiyle oluşturulan bir hücreye yerleştirilmiştir. Farklı sıcaklıklarda buharlaştırılan söz konusu gazlar, sıvı kristal örneği içeren hücrelere çeşitli süreler boyunca verilmiş ve sıvı kristalin dalga boyundaki değişimler analiz edilmiştir. Böylece insan sağlığı üzerinde zararlı etkisi olan bu gazların, termotropik sıvı kristal içeren bir sensör yardımıyla en düşük konsantrasyon seviyelerinde tespit edilebileceği gösterilmiştir.

Anahtar Kelimeler

Gaz sensörleri, sıvı kristaller, optik sensörler.

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INTRODUCTION

oxic gases such as ammonia (NH₂), hydrogen sulfide (H₂S), chlorine (Cl₂), acetone (C₂H₆O), nitrogen dioxide (NO₂), sulfur dioxide (SO₂), toluene (C₂H₂), dichloropropane $(C_3H_6C_{12})$, and phenol (C_6H_5OH) , as well as many organic solvents, are widely used in various industries such as the machinery-chemical industry, defense industry, agriculture and food industry, and electronics and technology industry. In industrial production, toluene, derived from fossil fuel sources, is preferred over benzene because it is more cost-effective. It is commonly used in the chemical industry for thinning paints, manufacturing rubber-based plastics, and producing explosive materials in the defense sector. Toluene is toxic to humans, and it irritates the eyes, respiratory system, and skin, and even at low doses when released as a toxic gas, whether through short-term or long-term exposure, it can suppress the central nervous system and impair memory. Prolonged exposure to high levels can cause serious damage to the central nervous system and may even be fatal. Dichloropropane, a chemical intermediate used in various industrial sectors, is utilized in the production of toner, paint, ink, and PVC adhesive. When it vaporizes, it can cause skin irritation. Phenol, widely used in different applications, releases toxic fumes that enter the body through the respiratory system and skin, and are eliminated through the kidneys and lungs. Phenol, another organic solvent, is commonly used in the electronics industry for manufacturing motherboards and in the furniture industry for producing adhesives and varnishes. The evaporation of these solvents depends on the temperature and humidity of the environment in which they are used. As mentioned earlier, since these solvents come into direct contact with humans, their evaporation can pose health risks. The release of phenol gas, especially in amounts of 1 gram or more, can have fatal consequences. When phenol contacts the skin, it is quickly absorbed, even though undamaged areas, and within minutes, it starts causing toxic effects in the brain.

Given the reasons mentioned above, it is crucial to carefully monitor the use of organic solvents and to promptly measure the levels permitted in the atmosphere. The methods used to identify the toxic gases produced by organic solvents in the air primarily involve gas chromatography-mass spectrometry [1-3] and Fourier transform infrared spectroscopy [4,5]. However, these techniques tend to be costly, time-consuming, and require complex sample preparation. Although cheaper and simpler alternatives, like polymer-based membrane surface tension sensors that can detect specific volatile organic compounds, have been developed [6], creating a variety of sensitive membranes for these sensors to detect different volatile organic compounds remains a complex and expensive task. Additionally, it necessitates the use of distinct membranes for measuring each type of toxic gas. Additionally, current methods for identifying toxic gases emitted by organic solvents rely on specific samples, costly equipment, and trained professionals who can utilize these devices. Taking these aspects into account, liquid crystals, which function as an intermediary phase between solids and liquids, represent one of the most effective sensing elements for detecting organic solvents. Because of their ordered structure, liquid crystal materials display solid-like behavior while also possessing fluid characteristics like liquids. The structures of liquid crystals, which represent the mesophase between solids and liquids, are significant due to their positional and orientational order. Liquid crystals create a distinct phase characterized by a three-dimensional arrangement, differing from both regular solids and entirely disordered liquids. Chemically, liquid crystal molecules are composed of rigid aromatic fragments linked by benzene rings and flexible hydrocarbon chains.

In recent literature studies, it has been observed that the easy alteration of the orientations of liquid crystals, which are generally composed of rod-like molecules, due to external stimuli such as temperature, electric, and magnetic fields, and the ability to convert these changes in orientations into easily observable optical outputs [7-10], plays a significant role in the use of liquid crystals in sensor applications. Due to their helical structures, these optical outputs are generated more rapidly, particularly within cholesteric and blue phase liquid crystal phases [8, 11-12]. Cholesteric liquid crystals are increasingly being explored in chemical and biological sensor applications due to their ability to sense and respond quickly. The implementation of liquid crystal sensors for the detection of hazardous organic gases has gained momentum in chemical applications, as these sensors enable visual result observation without the necessity for complex instrumentation or electrical power, unlike conventional analytical methods [13,14]. The optical detection of gases using liquid crystals is based on the principle that the gas molecule sent into the medium causes an increase in the helical pitch of

the cholesteric liquid crystal. Aromatic and halogenated solvents such as tetrachloroethylene at ppm (parts per million) levels as a function of the changing wavelength of liquid crystals were also identified [15]. In another study, to investigate the effect of molecular functionality on the sensor's ability to change color, samples created by adding trifluoroacetyl-functional molecules to cholesteric liquid crystals were used for the optical detection of amine vapors [16]. In another study, the color change of a cholesteric liquid crystal sample exposed to gases such as ethanol, acetone, benzene, pyridine, and hexane, using cholesterol nonanoate and cholesterol chloride chemical substances was investigated. They observed that the ethanol gas did not cause any optical changes in the liquid crystal sample, while the benzene and hexane gases transformed the liquid crystal sample from a colored state to a colorless state [17]. Chang et al. utilized the cholesteric liquid crystal sample for the determination of acetone and toluene solvents, which are commonly used in the chemical industry and are critically important for assessing their concentrations in the environment. A difference in the molecular polarities of acetone and toluene solvents, as well as their varying diffusion coefficients and lengths, has been found to enhance the rate of color change in the liquid crystal-based sensor created [8]. Thus, it has been observed that liquid crystal-based sensors can also be utilized for the identification of specific functional groups in polar and nonpolar organic molecules.

Han et al. have investigated the detection of CO₂ and O₂, which are important for smart packaging applications in food preservation, by introducing binaphthyl dithiol derivatives into cholesteric liquid crystals to study the formation of O₂. Due to the structural changes accompanying the oxidation of dithiol, it was observed that in the oxygen visualization, the cholesteric liquid crystal sensor shifted the wavelength from the green region to the red region. Additionally, it was noted that the carbamate formed during the reactions also affects the amount of CO₂ released, resulting in a shift of the wavelength from the red region to the blue region in the sensor [12]. Robinson et al. were able to detect target gases H₂S, NO₃, and NH₃ at a concentration level of 100 ppb (parts per billion) with a sensitivity of less than 60 seconds using their cholesteric liquid crystal samples. This advancement was achieved through the optimization of the surface density of the liquid crystal film samples that interact with the mentioned gases. Thus, it has paved the way for the development of high-precision,

small, lightweight, and low-power gas sensors for environmental monitoring and security applications [18]. Liquid crystal-based sensors have also been used in studies for the detection of organophosphorus chemical warfare agents, such as sarin, soman, and tabun, which exhibit high toxicity and contain a phosphoryl group, providing a rapid response time of approximately 30 seconds [10]. Kemiklioglu et.al. developed a lyotropic cholesteric liquid crystal (ChLC) sensor for the first time, capable of detecting both polar gases like toluene and phenol, and the nonpolar gas 1,2-dichloropropane. The ChLC sample was created by blending three different types of lyotropic ChLCs, incorporating oleyl carbonate, pelargonate, and benzoate groups. These gas vapors were then used to evaluate the sensor's detection capabilities. The gas vapors were introduced into the sample at various temperatures, which was placed on a glass cell coated with Dimethyloctadecyl [3-(trimethoxysilyl) propyl] ammonium chloride (DMOAP). The sensor's optical response was examined by observing changes in the twist angle of the lyotropic ChLC in the presence of these toxic gases [19].

In this study, the detection limits of toluene, phenol, and 1,2-dichloropropane gases were established by utilizing a different liquid crystal along with a chiral compound possessing lower helical twisting power. Moreover, lecithin was employed as the surface coating material instead of DMOAP. This approach allowed for an examination of how the surface coating material influences the initial alignment angles of molecules, as well as the effects of pitch, in relation to the performance of the gas sensor.

MATERIALS and METHODS

The nematic liquid crystal E7 was mixed with a blend of different chiral dopants to obtain a blue liquid crystal mixture. To achieve the desired pitch size of the blue liquid crystal helix, three different chiral substances, both right-handed and left-handed, were used. The prepared chiral material mixture consists of 20% by mass of the dopants of (S)-4-Cyano-4'-(2-methylbutyl)biphenyl (CB15) and (R)-2-Octyl 4-[4-(Hexyloxy)benzoyloxy]benzoate (R811), and 5% 1,2bis(4-(4-pentyl cyclohexyl) benzoate)-1-(R)-phenyl ethane (R1011)chiral materials. To ensure the homogeneous mixing of the sample containing 45% by mass of E7 solid substance and 55% of E7 liquid nematic liquid crystal, it was stirred in an ultrasonic bath at 70°C for 30 minutes in a vial. The cells

containing the liquid crystal sample were created by sandwiching glass surfaces coated with lecithin. Before lecithin coating, the glass surfaces were subjected to sonication for 2 hours in a 5% solution prepared with a detergent called Decon-90. They were then rinsed thoroughly with deionized water and subsequently dried for 15 minutes in an oven at 100°C. The cleaned glass surfaces were rinsed with isopropyl alcohol (IPA) and sonicated in ethanol for 15 minutes. Afterward, they were dried in a vacuum oven at 80°C for 20 minutes to prepare for lecithin coating. For the lecithin coating, a 1% ethanol solution containing dissolved lecithin was uniformly spread on the glass surface using a spin coater and then dried [20]. (Baek vd. 2015). Subsequently, a 10-micron thick spacer and UV-curable glue were placed between two glasses, and a cell was formed by sandwiching them through an ultraviolet (UV) curing process. The blue phase liquid crystal was filled into this cell through capillary action.

To identify the phase transition temperatures of the prepared samples, the cells filled with the samples were positioned in a Linkam programmable heater, which was situated between the crossed polarizers of a polarizing optical microscope (POM, Nikon Eclipse E200, Tokyo, Japan) equipped with a 10X magnification objective lens at a 90° angle. After heating to the isotropic temperature, the samples were cooled down to 25°C at a rate of 0.1°C per minute. During this cooling process, the structures formed were photographed, allowing for the identification of the phase transition temperatures. On the other hand, to generate toxic gas vapors, phenol, toluene, and 1,2-dichloropropane solvents were evaporated at different temperatures in separate beakers. During this process, the evaporated toxic gas vapors from the beakers were released into the system through a thin capillary tube. The reflective spectra of the liquid crystal sample exposed to the toxic gases were obtained using a spectrometer (USB4000, Ocean

Optics, USA). The differences in the wavelengths of the samples were analyzed before and after the release of toxic gas vapor using an Ocean Optics spectrophotometer (USB 2000+ (Ocean Optics) Deuterium Halogen Light source, Europe/USA version, Germany). To facilitate the introduction of toxic gas vapors into the cell, a cell was placed into a chamber with dimensions of 5x5x3.5 cm (height, width, and length). The design of the chamber was created using SOLIDWORKS® 2020 (Dassault Systems, USA), and the chamber was produced from Polylactic Acid (PLA) material using a Zaxe X2 3D printer (Zaxe Inc., Turkey).

RESULTS and DISCUSSION

The characteristic structures of the liquid crystal samples were observed through POM as a function of temperature, allowing for the determination of phase transition temperatures. The working temperatures of the liquid crystal sample were established by raising the temperature from the blue phase to the isotropic phase and then cooling it back to the blue phase at a rate of 0.1°C per minute. The POM images of textural structures of blue phase liquid crystal sample during the heating process before the gas vapor was introduced are presented in Figure 1. This sample exhibits a blue phase at room temperature and transitions to the isotropic phase at 40°C. The presence of the blue phase at room temperature offers a significant advantage for performing experimental studies. As can be seen in the POM images in Figure 1, the sample exhibits blue phase I up to 30.9°C, while it has a blue phase II structure between 30.9°C and 40°C. At lower temperatures, blue phase I is observed as a dark bright blue color, whereas blue phase II shows a shift in wavelength from blue to green. The sample remains stable in the blue phase I structure within the temperature range from room temperature to 30.9°C.

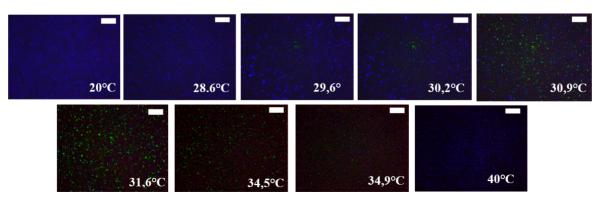


Figure 1. POM images of blue phase liquid crystal sample during the heating process. The white scalar bar corresponds to 20 µm.

After determining the working temperature range of the blue phase sample, the sample was placed in three different cells, and toxic gas vapors were separately introduced into each cell. For this purpose, phenol, toluene, and 1,2-dichloropropane solvents were heated in separate beakers to 90°C, 100°C, and 110°C, respectively, to vaporize them. The resulting gas vapors were then transferred into the cells which are in a closed chamber. Toluene, phenol, and 1,2-dichloropropane solvents were evaporated at different temperatures for 1 hour, and the amounts evaporated were determined. The mass ratio of the evaporated gases as a function of time at different temperatures was plotted in a graph, as shown in Figure 2. It was noted that the quantities of evaporating mass increased gradually in small increments during the first 30 minutes for each temperature applied; however, after 30 minutes, this increase became significant. Figure 2a illustrates that the amount of gas evaporating from toluene was greater at higher temperatures. In Figures 2b and 2c, it has been observed that the amounts of evaporating phenol and 1,2-dichloropropane gases significantly increased between the 30th

and 45th minutes. The amount of evaporating phenol gas was found to be higher than that of toluene and 1,2-dichloropropane.

After the amounts of evaporating gas were determined, these amounts were introduced into the cell containing blue phase liquid crystal sample. The changes caused by the gas amount at the wavelength of the blue phase liquid crystal sensor platform were detected using an Ocean Optics spectrophotometer, and the wavelength-Bragg reflection measurements were presented in Figure 3. Phenol, toluene, and 1,2-dichloropropane were evaporated at different temperatures between 40°C and 90°C for 60 minutes and introduced into three separate blue phase liquid crystal cells placed in three distinct chambers. It was observed that the wavelength changed depending on the alteration in the orientation of the liquid crystal caused by these gas vapors. The wavelengths of three different cells prepared for the treatment of toxic gas vapors of phenol, toluene, and 1,2-dichloropropane were determined to be 436 nm,

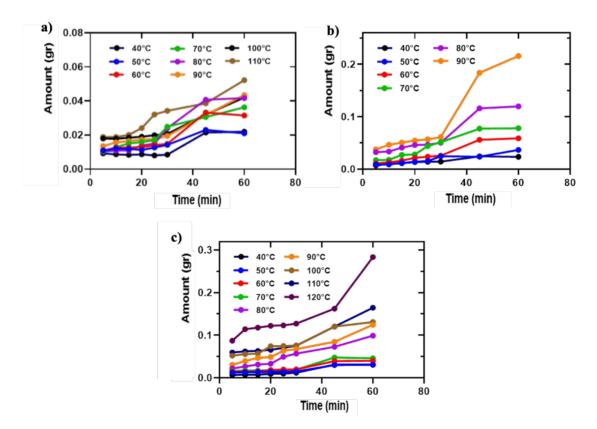


Figure 2. The mass evaporation rates of a) toluene, b) phenol, and c) 1,2-dichloropropane as a function of time at different temperatures.

440 nm. and 440 nm. respectively, before exposure to the gas. Figure 3 shows that increasing concentrations of phenol, 1,2-dichloropropane, and toluene gas vapors result in a shift of the blue phase liquid crystal's wavelength from green to blue. With the introduction of phenol gas vapor, the wavelength of the cell shifted from 436 nm to 425 nm, resulting in a wavelength change of 13 nm, as shown in Figure 3a. Similarly, when 1,2-dichloropropane was introduced, the wavelength of the cell shifted from 440 nm to 427 nm, resulting in a change of 10 nm, as shown in Figure 3b. As shown in Figure 3c, when toluene gas vapor is introduced to the blue phase liquid crystal, the wavelength shifts from 440 nm to 431 nm, indicating a 9 nm change. For all three toxic gas vapors, it is seen that with increasing gas vapor concentration, the wavelength of the blue phase liquid crystal shifts to higher wavelengths.

In this study, we showed that cholesteric liquid crystals (ChLCs) can serve as highly sensitive sensing materials for detecting gases such as toluene, phenol, and 1,2-dichloropropane. These substances are known to have toxic effects on the central nervous system and can cause damage to nerve cells, negatively impacting memory and concentration when exposure occurs. The diffusion of these gases disrupts the helical structure of the thermotropic ChLCs, and this alteration was examined using polarized optical microscopy (POM) and reflection spectra. When the ChLCs were subjected to vaporized gases at varying temperatures for 60 minutes, the wavelength shifted by 9, 11, and 13 nm for phenol, 1,2-dichloropropane, and toluene, respectively, as the exposure duration increased. Additionally, a distinct shift toward smaller wavelengths was noted with rising temperatures when the ChLCs were exposed gas vapors.

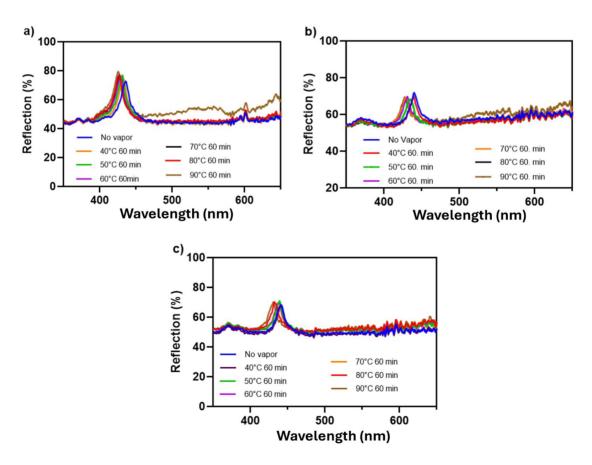


Figure 3. The wavelength-reflection graphs depicting the changes observed in the liquid crystal sample following exposure to a) Phenol, b) 1,2-dichloropropane, and c) Toluene toxic gases.

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