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NONLINEAR OPTICAL PROPERTIES OF IONIC THERMOTROPIC AND LYOTROPIC LIQUID CRYSTALS

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Abstract**—***This work presents the analysis of experimental research on the optical and nonlinear optical properties of two different representatives of metal alkanoate-based liquid crystals. Namely, ionic lyotropic liquid crystals of potassium caprylate doped with electrochromic viologen additives, and anisotropic glasses of ionic thermotropic liquid crystals of cobalt alkanoates of the homologous series (n = 7, 9, 11) and their multicomponent mixtures. Prior to the nonlinear optical experiment, the optical absorption spectra of all samples were investigated. For proposed absorbing media, laser-induced dynamic grating recording under the action of nanosecond laser pulses has been realised, observed and analysed. The studied materials were found to exhibit cubic optical nonlinearity, with cubic nonlinear susceptibility* $\chi^{(3)}$ *and hyperpolarizability* γ *values comparable to the best organic dyes. The possible mechanism of nonlinear response in the systems studied was considered on the basis of the data obtained. The nonlinear response mechanism is related to the non-linear polarisation of the π-electrons in the field of the laser radiation.*

Index Terms—Ionic liquid crystals; viologen; spectroscopy; optical nonlinearity; dynamic gratings.

I. INTRODUCTION

Nowadays, liquid crystal (LC) materials and their compositions have plenty applications as electro-optical elements, since their optical properties (changes in absorption coefficient and refractive index) are controlled electrically. Finding new non-conventional types of LCs with outstanding optical, electro-optical and nonlinear characteristics circumvents challenges for application in advanced holographic components. It is well known that thermal and electrical studies of LC materials led to their successful application in low-cost techniques for large-area thermal mapping in avionics [1] and the design of the first liquid crystal displays (LCDs) developed and tested for use in aviation [2]. Active-Matrix Liquid-Crystal Display (AMLCD) – the type of display technology commonly used in aviation today [3] – emerged from the development of these technologies and further research into new non-conventional types of LCs. As a result, research into the properties and characteristics of liquid crystals has led to their wide application - not only in display technologies, but also in laser beam steering, non-destructive testing, the development of adaptive optical elements for space applications, the creation of tunable lenses for augmented reality, Bragg gratings for sensors, photonics and holographic recording [4], [5].

II. ANALYSIS OF RESEARCH AND PROBLEM STATEMENT

In particular, in the latter case, ionic liquid crystals based on metal alkanoates, which are capable of forming both lyotropic and thermotropic mesophases, have shown their effectiveness [6]. Ionic thermotropic liquid crystals (ITLCs) can be easily supercooled to form anisotropic smectic glasses and have intrinsic absorption in the visible wavelength range [7]. The absorption of ionic lyotropic liquid crystals (ILLCs) is caused by the introduction of dyes or photo- and electrosensitive impurities that do not disturb the liquid crystal structure of the matrix substance [8]. Due to their absorption in the region of laser irradiation, such liquid crystal materials are characterized by a large and fast nonlinear optical response and are promising media for holographic recording.

III. METHODS AND OBJECTS OF RESEARCH

Samples for optical and nonlinear optical studies were prepared as follows:

1) ILLC obtained by mixing potassium caprylate powder with water in a 1:1 weight ratio ($C_7H_{15}COO^-K^+$:H_2O), contained 2 wt. % of electrochromic impurities of two compounds from the class of viologens: N,N′-diheptyl-4,4′- dipyridyl dibromide ($HD^{2+}2Br^-$) та N,N'-di(2-carboxyethyl)-4,4'-dipyridyl dichloride ($CED^{2+}2Cl^-$), which differ in substituents at nitrogen atoms and counterions. This concentration of the impurity allows to preserve the LC structure [8] – [10], while ensuring the electrochromic properties of the samples, which consist in the ability to be coloured under the influence of an external electric field due to the reduction of viologen molecules.

2) Cobalt alkanoates in the form of polycrystalline powder were heated above the crystal-mesophase transition temperature and then rapidly cooled to room temperature to obtain smectic glasses. Such glasses of ionic thermotropic liquid crystals were obtained for the following compounds: three representatives of the homologous series of cobalt alkanoates $(C_nH_{2n+1}COO^-)_2Co^{2+}$ ($n = 7, 9, 11$); binary mixtures $Co,Li\,|\,(C_7H_{15}COO)_2$ (0,5:0,5 molar concentration) and $Co,K\,|\,(C_7H_{15}COO)_2$ (0.5:0.5 molar concentration); triple mixture $Co,Li,K\,|\,(C_7H_{15}COO)_2$ (0.5:0.25:0.25 molar concentration). In previous studies, it has been shown that all the substances under study form a liquid crystal of the smectic *A* type during melting [7], [11] and are capable of forming stable smectic glass.

All studies were performed at room temperature in glass sandwich cells, which in the case of ILRK-viologen samples were additionally coated on the inside with a layer of ITO electrodes to allow the application of voltage. The thickness of the samples was set using teflon spacers, and the cells themselves were sealed around the perimeter to prevent air from entering and to prevent sample deterioration.

The optical spectroscopy method was used to study the optical properties and the absorption spectra of the samples were obtained in the visible range.

To investigate the nonlinear optical properties and to determine the main holographic characteristics of the samples, we used the methods of dynamic holography and measurement of the nonlinear transmission of the medium. The holographic recording of the gratings was carried out on the basis of a two-beam scheme using the second harmonic irradiation of a pulsed Nd:YAP laser operating in the mode of *Q*-switched modulation (TEM_{00} mode, wavelength $\lambda = 539.8$ nm, duration of one pulse 20 ns, pulse frequency $\nu = 3$ Hz). The linearly polarised in the horizontal plane laser beam was split into two beams of approximately equal intensity, which were brought together at a given angle on the sample cell. We studied the efficiency of self-diffraction by varying the angle of beam convergence and the intensity of the laser radiation.

To study the lattice decay kinetics in the microsecond range, the recorded holographic lattices were read by a probe using unpolarised radiation from a continuous He-Ne laser (power $P = 2$ mW, $\lambda = 632.8$ nm).

IV. Experimental Data

A. Optical properties

The absorption of ILRK-viologen composites is caused by impurities of viologens, whose molecules are reduced under the influence of an external field and, depending on the applied voltage, can be in three forms: a dication of viologen (colorless), a blue radical cation, and a red dimer. When the viologen $HD^{2+}2Br^-$ is introduced into a lyotropic liquid crystal matrix, its reduction occurs in stages and is characterized by the formation of both cationic radicals and dimers.

The absorption spectrum of the ILLC-$HD^{2+}2Br^-$ composite can have bands with a maximum at ~ 605 nm ($U = 2$ V, blue) or ~ 520 nm ($U = 4$ V, red), depending on the applied voltage. Both reduced forms of viologen are formed simultaneously in the ILLC-$CED^{2+}2Cl^-$ composite and its spectrum is characterised by an absorption band with a main maximum at ~ 525 nm ($U = 3$–3.5 V, purple) (Fig. 1).

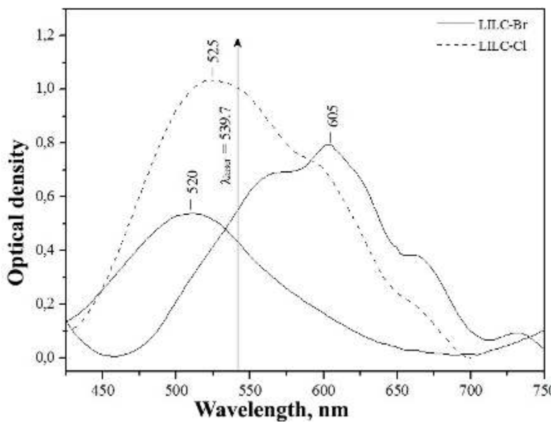

Fig. 1. Absorption spectra of the samples of ILLC-$HD^{2+}2Br^-$ (solid line) and ILLC -$CED^{2+}2Cl^-$ (dotted line)

All the smectic glasses of cobalt alkanoates that have been studied also absorb light in the visible region (Fig. 2). This absorption can be explained in terms of ligand field theory [12], [13] as a consequence of the excitation of octahedral (coordination number = 6) cobalt ion complexes, for which two characteristic electronic transitions have been recorded. The maximum at ~ 530 nm corresponds to the electronic transition $^4T_{1g}(^4F) \rightarrow {}^4T_{1g}(^4P)$, and maximum at ~ 565 nm – $^4T_{1g}(^4F) \rightarrow {}^4A_{2g}(^4F)$.

Bending of the absorption band around ~ 620 nm was observed in the mixtures ($^4A_2(^4F) \rightarrow {}^4T_1(^4P)$ transition). This indicates that, along with octahedral complexes, complexes with tetrahedral coordination are likely to exist in these mixtures. The absorption coefficient α of binary and ternary systems based on CoC_8 is 2–3 times higher than that of pure CoC_8.

B. Nonlinear optical properties

A single laser pulse was sufficient to record diffraction dynamic gratings in samples of ITLC glasses and ILLC-viologen composites, and the self-diffraction effect was observed for all samples. Several diffraction orders were usually observed. This fact, as well as the small value of the Cook-Klein parameter ($Q < 0.1$), indicate that the recorded gratings are thin. The diffraction gratings were recorded by modulating the complex refractive index in the interference field of two laser beams combined on the sample.

First, we evaluated the dependence of the optical density of the samples on the laser intensity to determine the nature of the recorded lattices. In the operating intensity range, the nonlinear absorption is insignificant, i.e., the recorded lattices in both cases were predominantly phase lattices. At the same time, the operating radiation intensity was $D = 0.45 - 0.46$, and for the ITLC samples – $D = 1.0 - 1.1$.

The diffraction efficiency η (I_1 / I_0 ratio) of ITLC glasses and ILLC-viologen composites was measured in the first order of self-diffraction and, for all the samples studied, it depended quadratically on the intensity of the recording laser radiation, indicating the presence of a cubic optical nonlinearity in the samples. At the same time, ILLC-$HD^{2+}2Br^-$ samples containing viologen dimers had twice the diffraction efficiency of the same samples containing cationic radicals, and smectic glasses of individual cobalt alkanoate homologs had higher diffraction efficiency values than mixtures. The experimental data obtained from a series of studies on a variety of samples showed that the repeatability of the results was satisfactory [11], [14]. A typical graph of the diffraction efficiency for the samples studied is shown in Fig. 3.

When studying the kinetics of decay of the recorded gratings, an exponential time dependence of the intensity in the first order diffraction was observed for all samples (Fig. 4). For ITLC smectic glasses, the decay constant τ for a lattice period of Λ = 16 μm was 60 μs, and for ILLC-viologen composites, the decay constant τ for a lattice period of Λ = 15 μm was 25 μs. This dependence is characteristic of the thermal lattice formed in samples when recording laser irradiation is absorbed. Due to the inertia of the photorecording system, lattice relaxation was monitored from 0.5 μs, i.e. the erasure of residual thermal lattices was observed in the microsecond range. However, a comparison of the diffraction efficiency of the thermal lattice η_T with the diffraction efficiency of the lattice recorded in the self-diffraction mode η shows that $\eta_T \ll \eta$ ($\eta / \eta_T > 100$), i.e., the diffraction lattice recorded in the self-diffraction mode is not caused by the thermal mechanism.

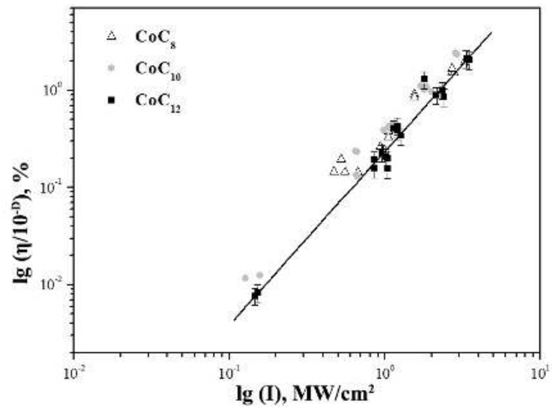

Fig. 3. Dependence of the diffractive efficiency (η) of the phase lattice on the laser radiation intensity (I) for smectic glasses of cobalt alkanoate homologs

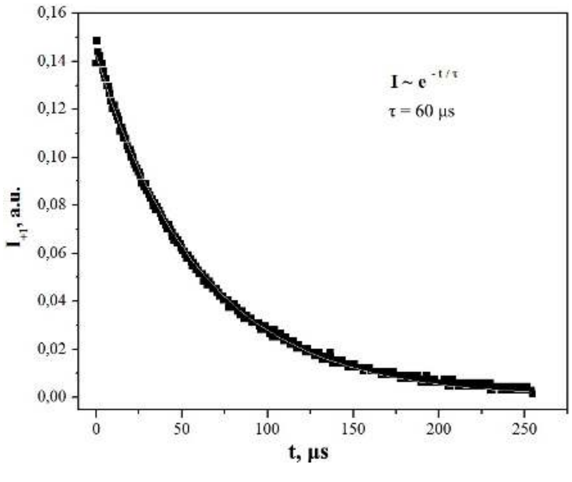

Fig. 4. The kinetics of the recorded lattice decay in the microsecond range for a sample of cobalt octanoate smectic glass

V. Results and Discussion

The main indicators of the cubic optical nonlinearity present in the samples studied are the nonlinear refractive index n_2 and the corresponding value of the cubic nonlinear susceptibility $\chi^{(3)}$. The nonlinear refractive index n_2 was determined by the experimental dependence $\eta = f(I^2)$ according to the expression:

$$|n_2| = \frac{\lambda}{\pi \cdot d \cdot m \cdot (I_1 + I_2)} \cdot \sqrt{\frac{\eta}{T}}, \quad (1)$$

where λ is the wavelength of laser radiation; d is the thickness at which the nonlinear phase progression occurred (cell thickness in the case of smectic glasses or photosensitive layer of viologen reduction products in the case of ILLC-viologen composites); m is the modulation depth; I_1, I_2 are intensities of laser beams that interfere on the sample under study; T is the transmission.

The values of hyperpolarizability γ and cubic nonlinear susceptibility $\chi^{(3)}$ were calculated using the following formulas:

$$\gamma = \frac{\chi^{(3)}}{N \cdot L^4}, \quad (2)$$

$$\chi^{(3)} = n_2 \cdot \varepsilon_0 \cdot c \cdot (n_0)^2 = \gamma \cdot N \cdot L^4, \quad (3)$$

where N is the concentration (of nonlinear optical centres in the case of smectic glasses or molecules in the case of ILLC-viologen composites); L is the local field factor; ε_0 is the dielectric constant; c is the speed of light; n_0 is the average refractive index.

The values of the nonlinear optical parameters n_2, $\chi^{(3)}$, and γ calculated from the experimental data are given in Table I. All data are presented in the CGS system, which is commonly used in nonlinear optics; an expression for the conversion from CGS to SI units is also given.

TABLE I. NONLINEAR OPTICAL PARAMETERS OF ILLC-VIOLOGEN COMPOSITES AND ITLC SMECTIC GLASSES

Sample	n_2 $cm^2 \cdot W^{-1}$	$\chi^{(3)}$ CGS	γ CGS
$CED^{2+}2Cl^-$ (dimers)	$1.8 \cdot 10^{-10}$	$0.8 \cdot 10^{-8}$	$1.2 \cdot 10^{-28}$
$HD^{2+}2Br^-$ (dimers)	$3 \cdot 10^{-10}$	$1.3 \cdot 10^{-8}$	$2.7 \cdot 10^{-28}$
$HD^{2+}2Br^-$ (radical cation)	$1.3 \cdot 10^{-10}$	$0.6 \cdot 10^{-8}$	$0.6 \cdot 10^{-28}$
CoC_8	$11.2 \cdot 10^{-10}$	$4.8 \cdot 10^{-8}$	$6.0 \cdot 10^{-30}$
CoC_{10}	$8.3 \cdot 10^{-10}$	$3.5 \cdot 10^{-8}$	$5.3 \cdot 10^{-30}$
CoC_{12}	$4.5 \cdot 10^{-10}$	$1.9 \cdot 10^{-8}$	$3.5 \cdot 10^{-30}$
Co, Li|C_8	$4.6 \cdot 10^{-10}$	$1.9 \cdot 10^{-8}$	$3.5 \cdot 10^{-30}$
Co, K|C_8	$5.3 \cdot 10^{-10}$	$2.2 \cdot 10^{-8}$	$4.2 \cdot 10^{-30}$
Co, Li, K|C_8	$4.6 \cdot 10^{-10}$	$1.9 \cdot 10^{-8}$	$3.6 \cdot 10^{-30}$

$$\chi^{(3)}(\text{СГС}) = \frac{9}{4 \cdot \pi} \cdot 10^8 \chi^{(3)}(\text{СІ})$$

The high values of nonlinear susceptibility and hyper-polarisability obtained for ILLC-viologen composites and ITLC smectic glasses are close in value to the best characteristics of organic dyes [10], [11].

VI. NONLINEAR OPTICAL RECORDING MECHANISM

It is known that the effect of nonlinear polarisation in an intense laser radiation field, where the polarisation value depends nonlinearly on the strength of the external electric field, can be exhibited by organic substances whose molecules contain delocalised π-electrons [15], [16].

The diffraction grating recording in ILLC-viologen samples occurs in the coloured layer of viologen reduction products formed on the cathode under the influence of an electric field. The initial (unreduced) molecules of viologen contain two dipyridylium rings on which weakly delocalised π-electrons are concentrated. After the molecules have been reduced to cationic radicals and dimers by the electric field, the degree of delocalisation of their π-electrons increases dramatically. While no holographic recording was observed for the original ILLC-viologen (transparent) samples, samples containing cationic radicals or dimers of viologen showed diffraction efficiency, which was higher in the latter case. Correspondingly, the values of the cubic nonlinear susceptibility ($\chi^{(3)}$) and hyperpolarisability (γ) were also higher in the case of dimers (Table I).

In ITLC samples, the main nonlinear optical centres are complexes formed by cobalt cations surrounded by oxygen atoms of carboxyl groups of alkanoate ligands, which contain delocalised π-electrons.

For the homologous series of cobalt alkanoates, the nonlinear susceptibility values increase with decreasing alkyl chain length, i.e. with increasing concentration of nonlinear optical centres. The oxygen atoms of the carboxyl groups coordinate several neighbouring cobalt cations simultaneously, which leads to the formation of a coordination network within the cation-anion layer. Since the cation-anion layer contains a large number of carboxyl groups with delocalised π-electrons, smectic glasses are capable of collective electronic polarisation, which leads to an enhanced nonlinear optical response of the medium.

Another important factor contributing to the increase in diffraction efficiency and hyperpolarisability is the wavelength of the excitation laser radiation (λ = 539.8 nm) that reaches the absorption band of the recording medium. According to the spectral data for ILLC-viologen composites (Fig. 1), the absorption maximum of the dimers is located near λ = 520–525 nm, and for the cobalt ITLCs studied, the absorption maxima are located in the region of $\lambda \sim 530$ nm and $\lambda \sim 560$ nm (see Fig. 2). Thus, for ILLC samples containing viologens in dimeric form and ITLC of cobalt alkanoates, the resonance enhancement effect plays a significant role.

The high values of $\chi^{(3)}$ and γ, as well as the small contribution of the thermal lattice to the nonlinear properties, distinguish ILLC-viologen composites and ITLC smectic glasses of cobalt alkanoates from other media. In particular, compared to traditional anisotropic materials such as nematic liquid crystals, the molecular reorientation effect was absent in the samples studied. All these factors make ILLC-viologen composites and ITLC cobalt alkanoate smectic glasses promising materials for holographic recording.

VII. Conclusions

Dynamic diffraction grating recordings on absorbing samples of ionic lyotropic liquid crystal (ILLC-viologen) and ionic thermotropic liquid crystal (ITLC) smectic glasses were obtained and the main experimental characteristics of the recordings, such as diffraction efficiency η and nonlinear refractive index n_2, were calculated.

Since viologen molecules in ILLC-viologen composites and alkanoate molecules in ITLC smectic glasses contain delocalised π-electrons, a mechanism of diffraction grating recording is proposed that is related to the effect of nonlinear polarisation in the field of intense laser radiation.

It was found that in the case of both ILLC-viologen composites and ITLC smectic glasses, the increase in the observed diffraction efficiency was facilitated by the wavelength of the excitation laser radiation getting into the absorption band of the recording medium.

The values of nonlinear susceptibility ($\chi^{(3)} \sim 10^{-8}$ esu) and hyperpolarisability ($\gamma \sim 10^{-28}$ esu) for ILLC-viologen composites and ITLC smectic glasses were estimated, which correspond to the best characteristics of organic compounds under resonant excitation in the order of magnitude.

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О. В. Грідякіна, Г. Б. Бордюг, Т. С. Лень. А. П. Поліщук. Нелінійно-оптичні властивості іонних термотропних та ліотропних рідких кристалів

У роботі представлено аналіз експериментальних досліджень оптичних і нелінійно-оптичних властивостей двох різних представників рідких кристалів на основі алканоатів металів. А саме – іонні ліотропні рідкі кристали каприлату калію, легованого електрохромними добавками віологену, та анізотропні скла іонних термотропних рідких кристалів гомологічного ряду алканоатів кобальту (n = 7, 9, 11) та їх багатокомпонентних сумішей. Перед нелінійно-оптичним експериментом були досліджені спектри оптичного поглинання всіх зразків. Для запропонованих поглинаючих середовищ було реалізовано та проаналізовано індукований лазером запис динамічної гратки під дією наносекундних лазерних імпульсів. Було виявлено, що досліджувані матеріали виявляють кубічну оптичну нелінійність із значеннями кубічної нелінійної сприйнятливості $\chi^{(3)}$ і гіперполяризовності γ, порівнянними з найкращими органічними барвниками. На основі отриманих даних розглянуто можливий механізм нелінійного відгуку в досліджуваних системах. Механізм нелінійного відгуку пов'язаний з нелінійною поляризацією π-електронів у полі лазерного випромінювання.

Ключові слова: іонні рідкі кристали; віологен; спектроскопія; оптична нелінійність; динамічні ґратки.

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