Electro-Optical Properties of a Liquid Crystalline Colloidal Solution of Rod Shaped V_2O_5 Nanoparticles and Carbon Nanotubes in an Alternating Current Electric Field

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We study the liquid crystalline phase behavior of the two-component aqueous colloidal suspensions of multiwalled carbon nanotubes (MWCNTs) and rod-like vanadium pentoxide (V_2O_5) nanoparticles. The phase diagram features a stable nematic phase in a wide range of concentration of solid components. The oriented nematic phase of the two-component suspension was exposed to the action of alternating current electric field. A variation in MWCNTs concentration within 0.01 – 0.51 wt.% demonstrates a significant increase in the optical response of the system to the applied electric field.

Key words: lyotropic liquid crystal, vanadium pentoxide, carbon nanotubes, electrooptical effects

1. Introduction

Over the past three decades, lyotropic aqueous and organic dispersions of inorganic compounds have been actively studied as a new trend in the field of liquid crystals (LCs) [1]. Since natural minerals usually contain dispersive phase, such systems in the physics of condensed systems are also commonly called mineral LCs, which form ordered phases, for example, with nano- and micro-sized particles [2,3]. These colloid materials which combine functionality of solid dispersed materials and advances of soft host solvent are very promising technology for a number of scientific and industrial applications. The dispersions of solid nanoparticles and consequent LC phases may point a promising way to design materials that can be aligned, reoriented and manipulated with moderate electric fields.

Applications on highly anisometric rigid rods with the Onsager mechanism of orientational ordering to build lyotropic LC systems result in LC materials with a significantly increased value of the excluded volume of particles and a higher order parameter than in LCs that consists of small molecules [4]. Also, orientational sensitivity in mineral LCs may be enhanced by the proper choice of dispersed materials with specific properties, like particle's anisometry ratio, magnetic or electrical sensitivity, that lead to a strong optical response to external stimuli [1,2].

Among such mineral LC systems, special attention is paid to dispersions based on vanadium pentoxide [5-9]. A number of experimental and theoretical publications on liquid crystalline systems based on V_2O_5 introduced their structural and phase properties in the LC state [4,10-12], and many studies analyzed various factors of interaction with external fields [8,13-18]. One more recent development of enhancing the functionality of liquid crystals through addition of second component to the solution was proposed in [18]. The authors showed that the addition of rod-like impurities sensitive to a magnetic field greatly increases a response of the system to an applied field, both in the isotropic and nematic phases. The physical reason for this increase was determined by a strong steric coupling between system components [19-21]: the field-sensitive component of the multicomponent suspension prompted another field-insensitive component [18,22-25]. At the same time, the study of electro-optical phenomena of multicomponent systems based on vanadium pentoxide with the addition of electrically sensitive components has not been explored yet. To improve the sensitivity of such systems to the action of external electric fields, we propose to use multi-walled carbon nanotubes (*MWCNTs*) as a second component. This choice of the system to study is justified by two factors. First, it is known that *MWCNTs* are strongly anisometric particles, the dispersions of which are similar to the Onsager mineral LC suspensions [26-28]. Second, numerous electro-optical studies of *MWCNTs* suspensions [29-33] show that such particles have a significant ability to polarize under the influence of an electric field.

Here we report on the liquid crystalline phase behavior of the two-component aqueous colloidal suspensions of rod-like $MWCNTs$ and vanadium pentoxide nanoparticles. The phase diagram featured a stable nematic phase in a wide range of concentration of solid components. Homogenously oriented samples were used to explore an electro-optical response of such liquid crystal composites in the nematic phase.

2. Materials and methods

Vanadium oxide gels were synthesized according to a previously published procedure via the acidification of an aqueous solution of sodium metavanadate $NaVO_3$ ($\sim 1\ mol\ L^{-1},\ pH = 9$) [4]. For the preparation of V_2O_5 aqueous suspension, the technique described in [18,34] was used. A number of suspensions with volume concentrations $C_{V_2O_5}$ were prepared in the range of $1.12\ wt.\% \leq C_{V_2O_5} \leq 2.24\ wt.\%$. Figures 1(a) and 1(b) show that the ordering degree of V_2O_5 rods in suspensions depends on their weight concentration: a pure suspension of V_2O_5 has two phases with the coexistence of an isotropic and nematic phase in the concentration range of $1.00\ wt.\% \leq C_{V_2O_5} \leq 1.80\ wt.\%$, and a completely nematic phase at the concentrations $C_{V_2O_5} > 1.80\ wt.\%$.

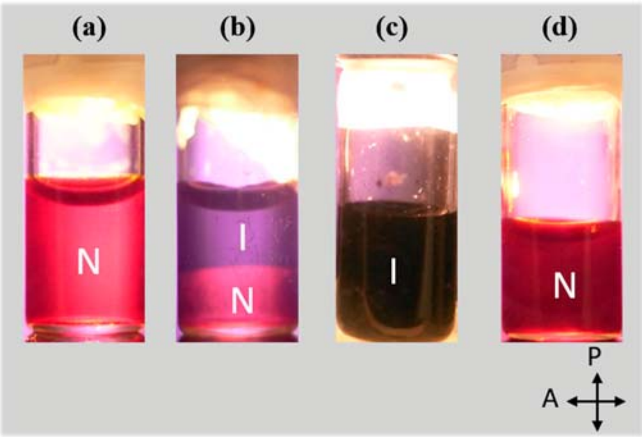

Figure 1. Aqueous inorganic colloids: $V_2O_5 - H_2O$ is in (a) nematic ($C_{V_2O_5} = 2.24\ wt.\%$) and (b) isotropic-nematic ($C_{V_2O_5} = 1.12\ wt.\%$) phases, respectively; (c) $MWCNTs - H_2O$ in isotropic phase ($C_{MWCNTs} = 2.33\ wt.\%$); (d) $V_2O_5 - MWCNTs - H_2O$ in nematic phase ($C_{V_2O_5} = 2.40\ wt.\%$, $C_{MWCNTs} = 0.04\ wt.\%$).

In our investigation we used commercially available (Cheap Tubes Inc., Cambridgeport, VT, USA) short multi-walled carbon nanotubes, prepared by chemical vapor deposition and purified using concentrated acid. The primary particles are rods with an average length of 2 $\mu m \pm 0.5\ \mu m$ and an outer diameter of $20\ nm \pm 2\ nm$, specific surface area is about $233\ m^2g^{-1}$, electrical conductivity is $> 100\ Scm^{-1}$, and density is $2.1\ gcm^{-3}$.

First of all, optically isotropic stable aqueous suspension, containing $MWCNTs$ were obtained by suggested earlier method of oxidation of the tubes surface. In this method $MWCNTs$ powder ($1\ g$) was mixed with a solution ($250\ mL$) of concentrated sulfuric acid (H_2SO_4: 100%) and nitric acid (HNO_3: 56%) in a ratio of 3: 1. Thereafter, the mixture was stirred using an ultrasonic bath at 60°C for 4 hours. The resulting suspension was diluted with $400\ mL$ of distilled water and further washed and filtered with colloid through a microporous $0.22\ \mu m$ hydrophilic polytetrafluoroethylene ($PTFE$) filters. This procedure was repeated until the acidity level pH of the water passing through a filter reached the level in the range from 6 to 7. $MWCNTs$ sediment was mixed with a distilled water to the required concentration ($C_{MWCNTs} = 0.10\ wt.\%$). As a result, such $MWCNTs$ aqueous dispersions are stable for a long time (up to six months) with value of $pH = 3 - 3.5$ at 24°C (Fig. 1(c)). The optical texture of the stable aqueous suspension is shown in Fig. 2(c).

A two-component suspension was prepared by directly mixing of aqueous suspensions V_2O_5 and $MWCNTs$ at certain weight concentrations of components $2.37\ wt.\% \leq C_{V_2O_5} \leq 2.46 wt.\%$ and $0.01\ wt.\% \leq C_{MWCNTs} \leq 0.51\ wt.\%$. The colloidal suspensions were mixed for 3 hours. The resulting suspensions were completely nematic (see Fig. 1(d)). The optical textures of the colloid are presented in Fig. 2(d). It should be noted that these colloids were stable and the particles do not aggregate in studied concentration range [34].

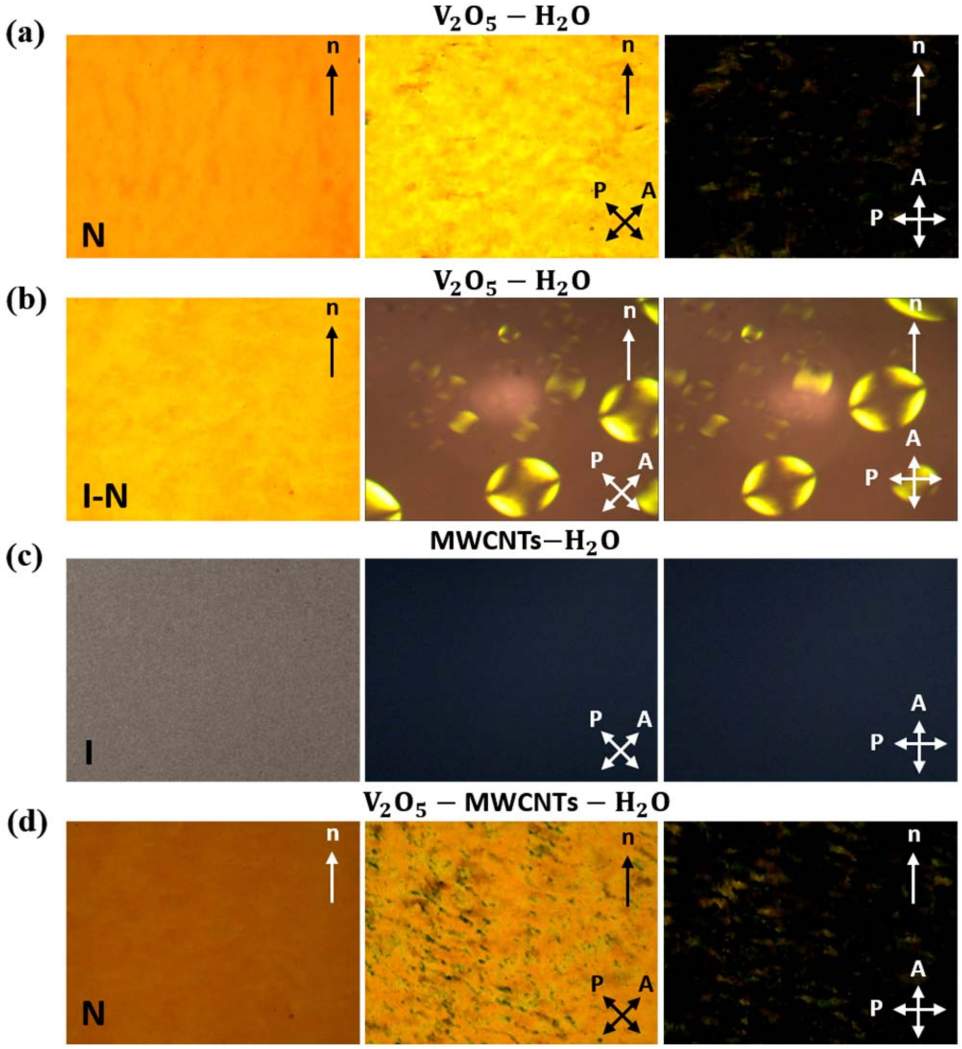

Figure 2. Textures of aqueous inorganic colloids (polarizing optical microscope pictures: left - without polarizers; in the middle - in crossed polarizers (director $\boldsymbol{n}$ of a liquid crystal oriented at the angle of 45^0 to the optical axis of the polarizer or analyzer); on right - in crossed polarizers (the director $\boldsymbol{n}$ of the liquid crystal oriented along the optical axis of the analyzer)). (a) and (b) $V_2O_5 - H_2O$ in nematic and isotropic-nematic phases correspondingly; (c) $MWCNTs - H_2O$ in isotropic phase; (d) $V_2O_5 - MWCNTs - H_2O$ in nematic phase. Corresponding concentrations are presented in the caption to Fig. 1.

Before characterization of the nanoparticles in suspensions, a series of scanning electron microscopy (SEM) studies were performed for solid films of V_2O_5, $MWCNTs$, $V_2O_5 - MWCNTs$, using field-emission scanning electron microscope (Zeiss, Oberkochen, Germany). The formation of thin layers of nanoparticles on the surface of the In_2O_3 occurred by dropping a small volume of suspensions of several types. The study was performed after complete evaporation of a solvent. Figure 3(a) shows a film of well-oriented V_2O_5 rods deposited from the nematic phase and the rods with chaotic orientation deposited from the isotropic phase (see insert in Fig. 3(a)). In Fig. 3(b) one can observe both the individual $MWCNTs$ and their aggregates. SEM image of films deposited from the $V_2O_5 - MWCNTs - H_2O$ suspension (Fig. 3(c)) revealed a layer of oriented V_2O_5 rods and $MWCNTs$ aggregates. We assume that aggregation occurs in the process of solvent evaporation.

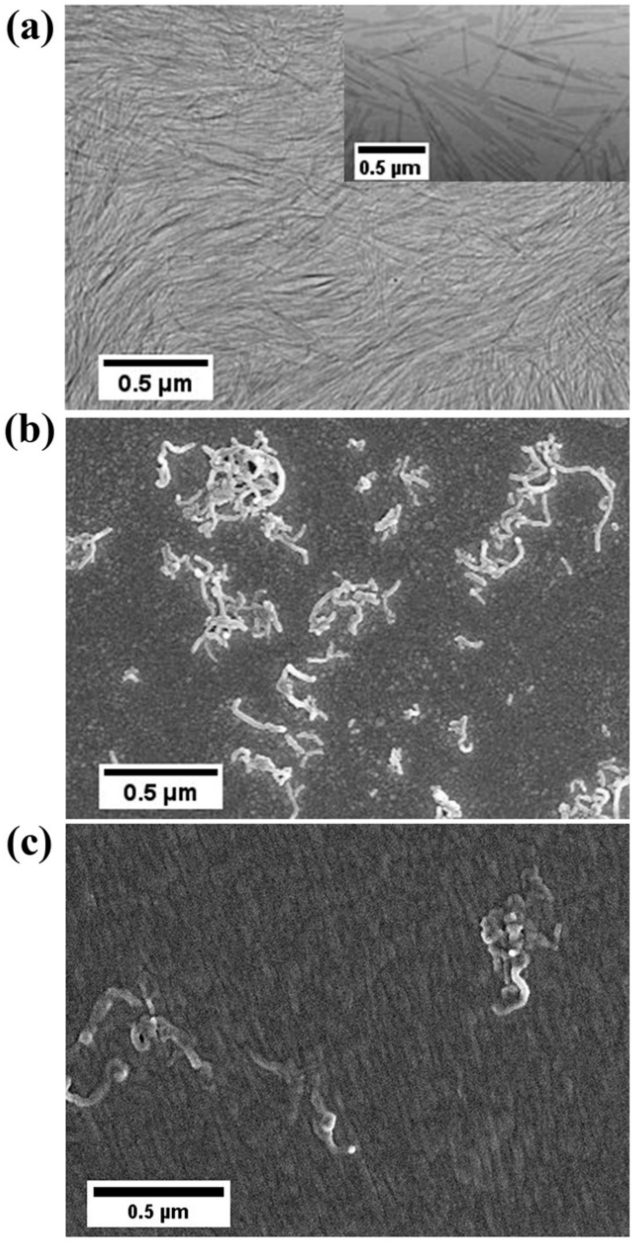

Figure 3. SEM images of inorganic water colloids: (a) $V_2O_5 - H_2O$ deposited from nematic phase ($C_{V_2O_5} = 2.0\ wt.\%$); the inset shows the same particles deposited from isotropic phase ($C_{V_2O_5} =$

0.5 $wt.\%$); (b) $MWCNTs - H_2O$ ($C_{MWCNTs} = 1.46\ wt.\%$); (c) $V_2O_5 - MWCNTs - H_2O$ deposited from nematic phase ($C_{V_2O_5} = 2.38\ wt.\%$, $C_{MWCNTs} = 0.02\ wt.\%$).

To study the electro-optical properties of aqueous suspensions of V_2O_5 and $MWCNTs$, the sandwich type cells were assembled from pairs of glass substrates coated with ITO electrodes [35]. A layer of nonionic surfactant was used to form the planar orientation of the director of LC cells ($C_nH_{2n+1}O(CH_2 - CH_2O)_mH$, where $n = 12 - 14$ and $m = 10$) [18]. The 1% surfactant solution was deposited on the surface of a glass substrate with the ITO electrode by centrifugation. The described procedure allows obtaining uniform films of surfactant molecules along the entire plane of the substrate. The cell thickness was determined by calibrated sphere polyester spacers with the diameter of 12 μm.

The cells were filled with colloidal suspensions ($V_2O_5 - H_2O$, $MWCNTs - H_2O$, and $V_2O_5 - MWCNTs - H_2O$) by capillary flow assisted by negative pressure applied to the open sides of the cell. Finally, the cell was promptly sealed after filling. The direction of flow which was parallel to the nonionic surfactant layers and the pressure gradient induce a homogeneous orientation of the LC director in the cells.

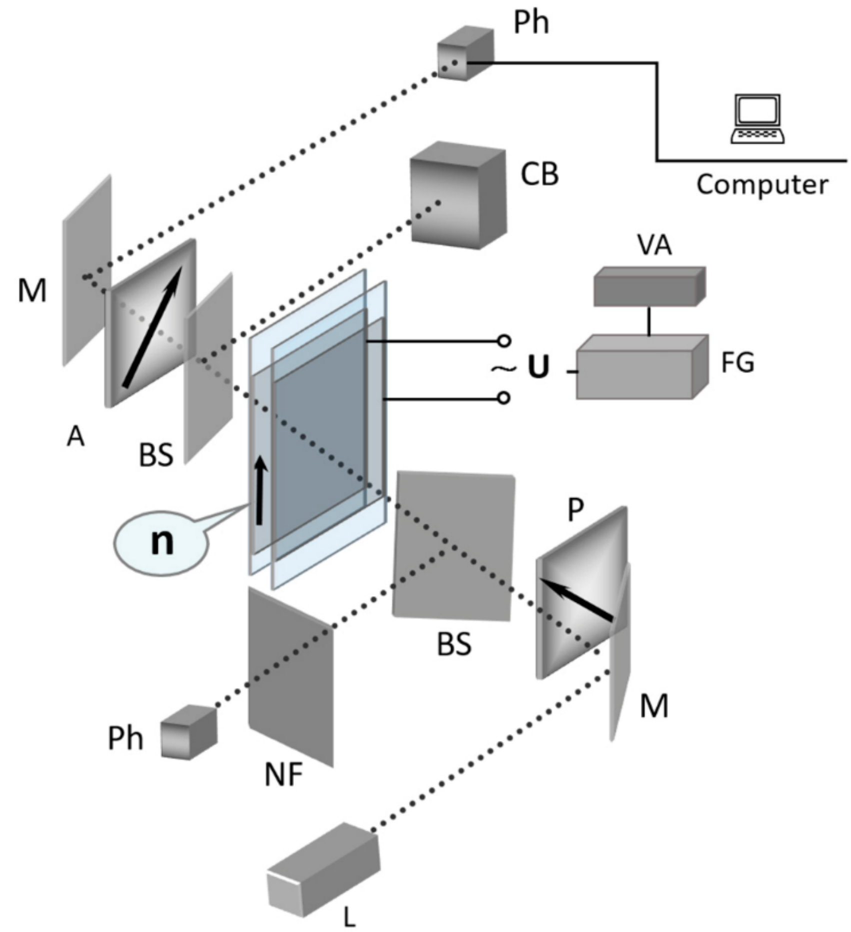

Figure 4. Scheme of the experimental electro-optical setup. **L − He-Ne** laser ($\lambda = 0.633\ \mu m$), **A** and **P -** polarizers, **M -** mirrors, **CB -** Berek phase-optical compensator, **BS -** beam splitter, **NF -** neutral filter, **FG** - electric signal generator (frequencies 30 MHz), **VA -** electric signal amplifier, **Ph** - reference photodiode.

We determined the optical path difference Δnd for LC suspensions by measuring the phase retardation $\Delta\varphi = 2\pi\Delta nd/\lambda$ between the extraordinary and ordinary waves (see Fig. 4), d is the cell thickness (μm), λ is the wavelength (μm) [35]. The phase retardation value was calculated by determination of the extrema positions of the sinusoidal light intensity curve using the relation $I = I_0\, sin^2(\,2\beta)\, sin^2(\,\Delta\varphi/2)$, where I is the intensity of light passing through the LC cell; I_0 is the intensity of the light that illuminated the LC cell; $\beta = 45^0$is the angle between the direction of the LC cell director and the optical axis of the polarizer. The method allows to measure the minimum value of birefringence and phase retardation as $\Delta n \sim 2 \times 10^{-6}$ and $\Delta\varphi \sim 10^{-2}\ rad$, respectively.

3. Results and discussions

Figure 5(a) shows the microscopic optical textures of the cell filled with the nematic suspension of $V_2O_5 - H_2O$. The cell has a homogeneous planar orientation, which was formed by a flow during cell filling. The application of an AC electric field to the cell ($12.5 \times 10^5\ Vm^{-1}$, $300\ kHz$) leads to a reorientation of the LC director along the field, visible as a change in the transmittance of the suspension between crossed polarizers. The threshold voltage of the director reorientation was found to be about $U_F \sim 15\ V$. The existence of a threshold voltage indicates low pretilt angle for LC director that correlates well with the experimentally measured angle, $\alpha = 3^0$.

In the optically isotropic phase of the $MWCNTs - H_2O$ colloid, the $MWCNTs$ particles were randomly dispersed in the matrix and the orientation of their long axis was chaotic. The application of AC electric field results in the orientation of the $MWCNTs$ particles along the electric field and in an appearance of electro-induced birefringence [36-39], which was measured using the Senarmon technique [40] for $\lambda = 0.633\ \mu m$. For the applied electric field of $5 \times 10^5\ Vm^{-1}$ and for $MWCNTs$ concentration of $C_{MWCNTs} = 2.33\ wt.\%$, the measured Δn is small and at the maximum demonstrates value of about $\Delta n = 2 \times 10^{-4}$, which is natural since the aqueous solution are highly diluted. For smaller concentration of the *MWCNT* in suspension, $C_{MWCNTs} = 0.08\ wt.\%$, a very low signal, probably caused by the buffed polyimide aligning layers, was registered [41].

Therefore, in both types of cells (V_2O_5 and $MWCNTs$ colloids), the textures responded to the application of the driving voltage. The intensity of this response like the driving voltage and the resulted induced optical retardation is rather low as compared to ordinary thermotropic nematic cells.

At the same time, the nematic phase of the two-component suspension $V_2O_5 - MWCNTs - H_2O$, that was exposed to an electric field, exhibits impressive behavior (Fig. 5(b)).

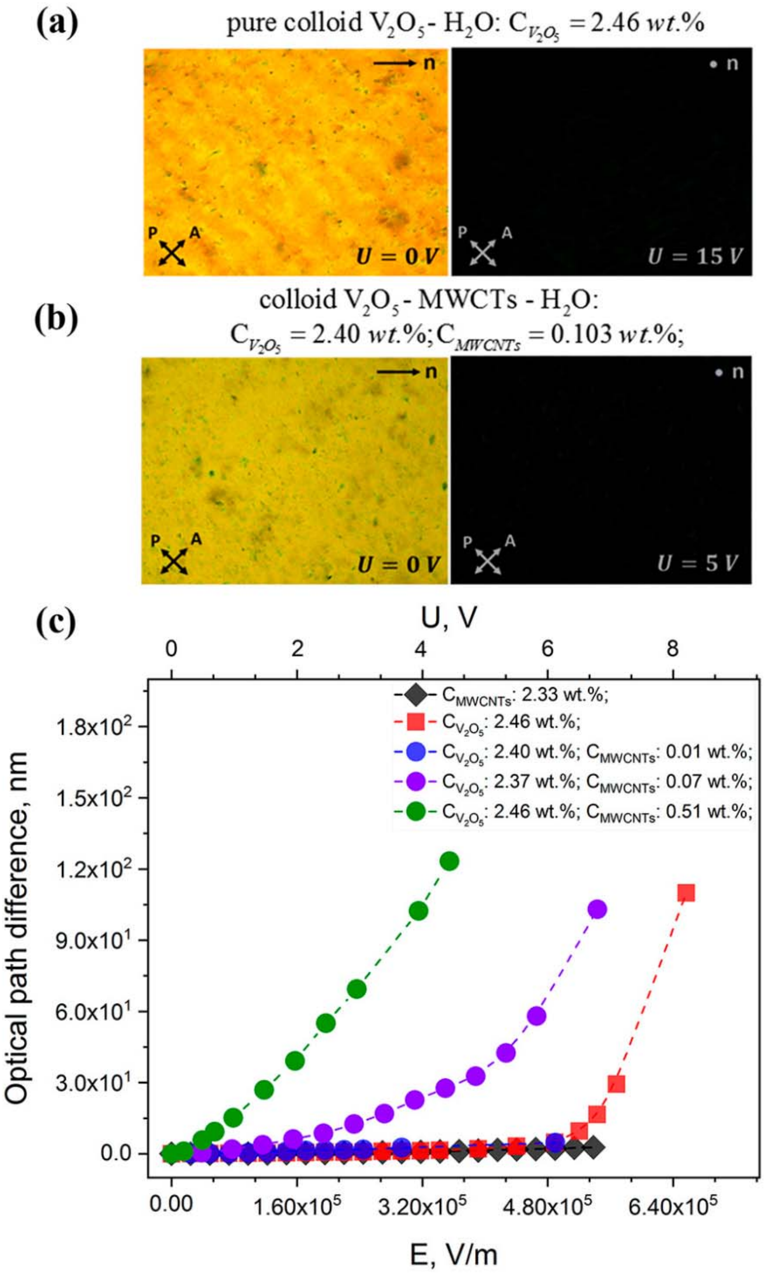

Figure 5. Electro-optical response of inorganic aqueous colloids: (a) $V_2O_5 - H_2O$ colloid textures in crossed polarizers at 0 and 15 V; (b) $V_2O_5 - MWCNTs - H_2O$ colloid textures in crossed polarizers at 0 and 5 V; (c) the dependence of optical path difference (Δnd) of colloids $V_2O_5 - H_2O$, $MWCNTs - H_2O$, $V_2O_5 - MWCNTs - H_2O$ on the external AC field strength.

A variation in $MWCNTs$ concentration within $0.01 - 0.51\ wt.\%$ range showed a significant increase in the response of the system $V_2O_5 - MWCNTs - H_2O$ to the applied electric field (Fig. 5(c)). The addition of a small amount of $MWCNTs$ to the nematic lyotropic suspension of $V_2O_5 - H_2O$ led to the appearance of a large electro-optical sensitivity of the mixture. The threshold voltage value for a complete reorientation of the LC director dropped down to the $U_F \sim 5\ V$. A significant decrease of the Fredericks threshold – from $5 \times 10^5\ Vm^{-1}$ to $5.5 \times 10^4\ Vm^{-1}$ was observed in comparison with a single V_2O_5 colloids.

We suppose that this effect can be described by a model in which $MWCNTs$ particles are considered as rigid rods sensitive to the electric field. Their field orientation leads to a reorientation of less electrically sensitive V_2O_5 rods due to steric interaction between particles. As a result a significant reorientation of the $V_2O_5 - MWCNTs - H_2O$ suspension director can be observed. A similar behavior in the form of the manifestation of a giant magneto-optical reorientation of the director was observed for two-component Onsager systems $V_2O_5 - Fe_3O_4$ in an external magnetic field [18].

Moreover, the two-component aqueous suspensions of $V_2O_5 - MWCNTs - H_2O$ showed unusual dynamic properties of the electro-optical switching under the action of alternating current electric field. Figure 6 shows the electro-optical response of two components colloid with concentrations of $C_{V_2O_5} = 2.46\ wt.\%$ and $C_{MWCNTs} = 0.51\ wt.\%$ respectively in the AC electric field of $1.6 \times 10^5\ Vm^{-1}$.

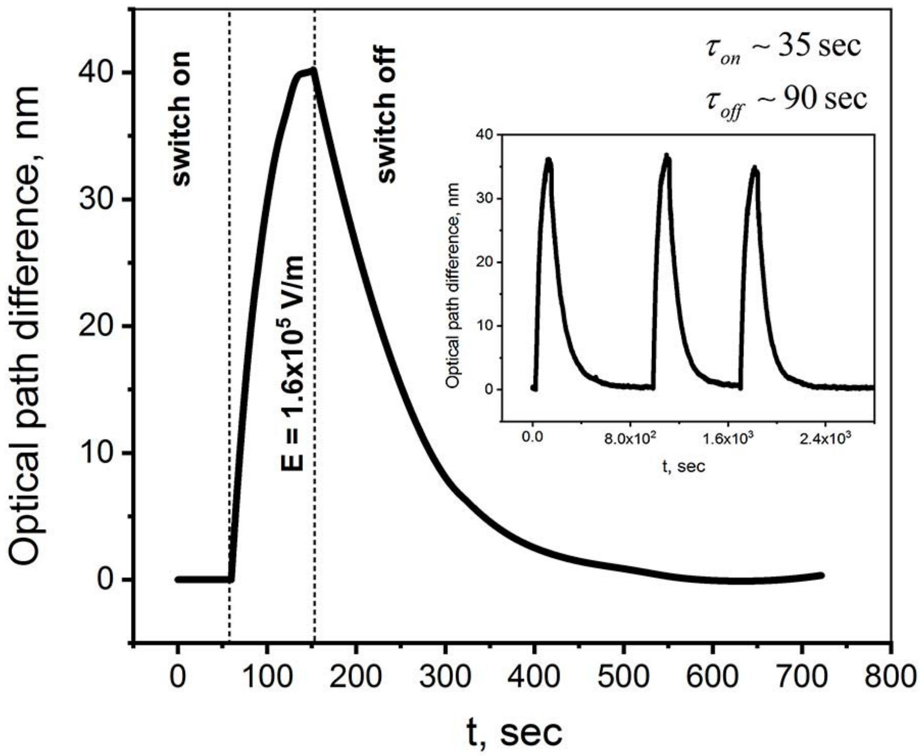

Figure 6. Electro-optical response of the nematic phase of the $V_2O_5 - MWCNTs - H_2O$ ($C_{V_2O_5} = 2.46\ wt.\%$, $C_{MWCNTs} = 0.51\ wt.\%$) colloid under application of AC electric field $E = 1.6 \times$

$10^5\ Vm^{-1}$. Cell thickness is $d_{cell} = 12\ \mu m$. The inset shows the sequence of switch *on* and *off* of the cell by electric field.

Application of the electric field leads to a reorientation of the LC director and to a change in the intensity of the light passing through the LC cell with the $V_2O_5 - MWCNTs - H_2O$ suspension. The characteristic switching-on time τ_{on} is relatively large compared to that in thermotropic liquid crystals [42,43]. Such a long-time response of the system to the electric field can be associated with the high viscosity of aqueous lyotropic V_2O_5 colloids [6,7,44]. The intriguing feature is the switching-off (relaxation) time τ_{off} of the system, that is almost two times larger than the turn-on time. It is well known, that the relaxation time τ_{off} of a LC is a quadratic function of the cell thickness D: $\tau_{off} = \gamma D^2/K\pi^2$ [45], where γ – rotation viscosity and K – appropriate elastic constant of the LC. In turn, the switching-on time τ_{on} also depends on the dielectric anisotropy $\Delta\varepsilon$ of the LC and applied voltage U. It results that a switching-on time τ_{on} is in general much faster than a relaxation time τ_{off}. For the $V_2O_5 - MWCNTs - H_2O$ suspension, the electric field sensitive component is $MWCNTs$ while induced Δnd originates mostly from the V_2O_5 component. The reorientation of the V_2O_5 chains does not significantly depend on applied voltage, but rather follows to the position of $MWCNTs$, that implies the mechanism which is similar to the mechanism of relaxation. The faster response to the electric field suggests a strong interaction between $MWCNTs$ and V_2O_5 chains in the suspension. Finally, the LC director of the suspension fully returns to the initial planar state when the electric field is turned off. The inset to Fig. 6 shows that periodic ripple of the quadratic field signal can be applied to the sample without loss of electro-optical parameters [14].

4. Conclusions

We investigated the electrooptical response of the primary liquid crystalline aqueous colloidal mixtures of V_2O_5 nanoparticles in AC electric fields as a function of the secondary added electric field sensitive component of $MWCNTs$. Our key findings are following: (1) The phase diagram of the two component mixture demonstrates a stable nematic phase in a wide range of concentration ($2.37\ wt.\% \leq C_{V_2O_5} \leq 2.46 wt.\%$ and $0.01\ wt.\% \leq C_{MWCNTs} \leq 0.51\ wt.\%$) of solid components. None of the concentrations showed any phase separation or particles aggregation for months. (2) In the nematic phase of the two-component system, a significant decrease in the Fredericks threshold was observed at the relatively small concentration of $MWCNTs$. The threshold dropped down from $5 \times 10^5\ Vm^{-1}$ for the pure solution of V_2O_5 to $5.5 \times 10^4\ Vm^{-1}$ for the solution with $0.51\ wt.\%$ of added $MWCNTs$.

The observed enhancement of the electro-optical response of two-component systems, the possibility of cyclic switching of the LC director together with the high resistance of the system to particle aggregation open prospects for utilization of the binary solution of $V_2O_5 - MWCNTs - H_2O$ for optical processing and display applications.

Acknowledgments

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