HISTORICAL DEVELOPMENT OF LIQUID CRYSTALS INVESTIGATIONS IN THE POLYTECHNIC SCHOOL OF BUCHAREST

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Abstract. This paper refers to the historical development of liquid crystals investigations in polytechnic school of Bucharest, which started four decades ago. The work has four parts: Introduction (presents the history of liquid crystals researchs in the world); liquid crystal investigations in the polytechnic school of Bucharest (from the folowing periods: 1972 to 1979; 1980 to 1989; 1990 to 1999 and 2000 to 2009); a set of conclusions (reffering to the researches made in the polytechnic school of Bucharest and dissemination of obtained results). All references used represents the work and results in the liquid crystal laboratory from Politechnica University of Bucharest, Romania, from 1972 to 2009 period.

1. INTRODUCTION

Liquid crystals were discovered in the 19th Century. Different investigations performed by scientists working in different fields of science reported an annomalous behavior of some substances. In 1888 the botanist Reinitzer noticed that cholesteryl benzoate displayed a turbid state at a temperature below the clearing point. His rezult has been examined by Lehmann who pointed out that a new type of substance, intermediate between liquid and solid crystal, has been discovered; he named it "Liquid Crystal". As it was shown later, Reinitzer discovered a cholesteric liquid crystal. It is interesting to notice that this new state was predicted by Edgar Allen Poe who in the story „The adventures of Arthur Gordon Pym" described a puzlling liquid exhibiting an unexpected behaviour similar to the one belonging to cholesteric liquid crystals. In 1922 Fridel published the article „Les états mesomorphes de la matière" in which he introduced the first classification of liquid crystals into nematics cholesterics and smectics. Today we recognize the existence of several smectic forms, whereas Friedel described only one (today's smectic A, SmA), exhibiting a layered structure.

The period from 1922 to about 1960 was characterized by a general interest in synthesizing mesomorphis compounds (Vorlander and his group in Halle). The Faraday Meeting of 1933 was of great importance for the development of liquid crystal investigations. It brought together brilliant scientists, presenting important news in this field, as for example Freedericksz and Zolina (forces causing orientation of an anisotropic liquid), Zocher (magnetic field effect on nematics), Lawrence and Oswald (lyotropic liquid crystals) and Ossen (whose elastic theory was later developed by Frank as a continuum theory of liquid crystals).

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From 1960 to the present time important progress in theoretical and experimental liquid crystal researches were pointed out.

Applications in electro-optical displays were established. In 1970 patents of Schadt and Helfrich and of Fergason on the twisted nematic liquid crystal electro-optic display have appeared. Two years later at the International Liquid Crystal Conference in Stockholm the authors presented different display types based on dynamic scattering, Freedericksz transition, cholesteric memory and cholesteric nematic bistability. After the discovery, by Meyer, of ferroelectric liquid crystals a new electro-optical device was proposed by Clark and Lagerwall. This is a surface stabilized liquid crystal device, with fast switching capability, based on a ferroelectric chiral smectic C liquid crystal material.

There was also much activity in the field of lyotropic liquid crystals.The importance of lyotropic LC in relation to biological systems or in medicine has been also intensively studied. Another apllication of these lyotropic phases is in the oil, detergent and soap industry as it is necesary to know the exact behaviour of amphiphile/water/oil systems and the role played by micelar phases in the extraction processes from natural rocks or cultivated plants.

In recent years polymer dispersed liquid crystals (PDLC) have attracted much attention. They consist in liquid crystal droplets (usually nematic droplets) embedded in an amorphous polymer matrix in which the orientation of nematic droplets can be changed by electric fields, allowing the appearace of a dark or light state. They can be used as large, flat panel electro-optical displays.

Polymeric liquid crystals were also discovered and intensively studied due to their apllications in electro-optical devices.

New phases and new materials have also been discovered in the last decade. It is worth mentioning antiferroelectric or ferrielectric liquid crystals displayed by chiral smectic phases in which, sometimes, additives were introduced.

2. LIQUID CRYSTAL INVESTIGATIONS IN THE POLYTECHNIC SCHOOL

In the Polytechnic Institute (now „Politehnica" University Bucharest) the investigations in the field of liquid crystals were started in 1972. They were related to electronic device applications, such as light shutters, transducers, filters and so on. It was only in 1974 when more systematic studies were accomplished due to the organization of a research group, leaded by Prof. Cornelia Moțoc, the head of the Solid State Physics Laboratory of the Department of Physics. First, there were only a small number of participants to this group: assistents (eng. Emil Sofron, physicists – Iuliana Cuculescu and Maria Honciuc) and students (Constantin Roşu and Mihai Răzvan Mitroi). We take advantage when two chemists (dr. Ion Baciu and Octavian Savin) accepted to participate to our researches and synthesised new mesomorphic compounds. During ages the number of participants to our "Liquid

Crystal Group" was increased and, as a result of this, it was possible to extend our investigations to new and interesting topics in the field of liquid crystals.

The scientific results obtained by this research group will be presented in chronological order and the following contributions will be pointed out:

- Scientific papers published in periodicals;
- Papers presented at National and International Conferences;
- Books;
- PhD thesis elaborated by the members of this group;
- Patents (Inventions or innovations);
- Grants.

The researches performed during the period 1972-2009 covered a large aria of subjects, such as:

– methods initiated for physical characterization of liquid crystals (LC);

– electro-optical devices using nematic liquid crystals (NLC), cholesteric liquid crystals (CLC) and smectic liquid crystals (SLC);

– modeling and simulation of LC devices;

– applications of LC non-destructive techniques of characterization silicon wafers and electronic devices;

– electric and dielectric properties of CLC and CLC – SLC mixtures;

– Freedericksz transitions in nematic and ferronematic materials;

– relaxation phenomena and viscosity of LC;

– magnetooptic properties of NLC with azo-dyes additives and induced birefringence;

– studies of polymer-dispersed NLC;

– induced photovoltaic and piroelectric effects by different additives in NLC;

– thermal stimulated polarization currents.

It is a great responsibility for me to relate the history of the development of liquid crystal investigations in the „Politehnica" University of Bucharest. All the same I will try to do my best. Due to the overwhelming number of papers in this field, it may be posible that some contributions to be missing and I appologize for that.

In the following all scientific results will be presented, considering four periods.

THE FIRST PERIOD FROM 1972 TO 1979

Scientific investigations elaborated during 1972-1979 were concerned first with nematic liquid crystals (NLC). Morphological properties and changes arising under electric fields and laser irradiations were followed (Moțoc C., 1976; Sterian P., 1976). Patents related to different applications of NLC such as colour systems for broadcasting TV (Florea C., 1976), transducers (Popescu M. I.,1975; Sofron E., 1977; Teodorescu, N. H., 1978; Luca E., 1979) and light shutters (Honciuc M., 1979; Cuculescu I., 1979; Popescu M. I.,1979; Sofron E., 1977), relays (Sofron E., 1977), stroboscopes (Sofron E., 1977) and pressure indicators (Sofron E., 1977) were elaborated.

Then the investigations have been obtained to cholesteric liquid crystal (CLC) and smectic liquid crystals (SLC). Binary mixtures of cholesteryl esters exhibiting positive or negative dielectric anisotropies have been obtained and their morphological and physical properties were investigated (Moțoc C., 1976; Sterian P., 1976; Cuculescu I., 1977).

The cholesteric-nematic transition in binary mixtures was examined both theoretically (Cuculescu, 1977) and experimentally (Moțoc C., 1976, 1977). A new depolarized light method was proposed to determine the temperature dependence of the critical field for cholesteric-nematic transition (Moțoc C., 1978). A lot of physical parameters, such as electric conductivity (Honciuc M., 1978; Moțoc C., 1978, 1979), dielectric anisotropy (Honciuc M., 1978, 1979) and activation energies (Cuculescu I., 1979) were determined as function of temperature. Light scattering phenomena of monochromatic light in mixtures exhibiting several mesophases (Moțoc C., 1978, 1979) were followed.

New mesomorphic compounds (sitosteryl esters) were synthetized and morphological changes when heating or cooling were determined (Moțoc C., 1979).

Applications of CLC mixtures as light shutters (Honciuc M., 1979; Cuculescu I., 1979), bistable and memory devices were examined (Cuculescu I., 1977; Honciuc M., 1977; Moțoc C., 1977, 1978). These results were included into the monograph (Sofron E., 1976) devoted to applications of liquid crystals in electronic devices. Morphological changes occurring under d.c.or a.c. electric fields were examined in some nematic phases and the possibility of using laser light modulation were discussed (Moțoc C., 1976; Sofron E., 1977).

Relying on these studies three PhD theses were elaborated (Sofron E., 1977; Cuculescu I., 1977; Honciuc M., 1977; Savin O., 1983; Roșu C., 1988; Socaciu M., 1985; Mitroi R., 1986; Bena R., 1986; Iacobescu G., 1995; Ionescu D., 1997; Dascălu C., 1997; Mănăilă-Maximean D., 1997; Popescu O., 1998; Petrescu E., 1998; Tudoran Cr., 2000; Puică M., 2001; Păun L. A., 2008).

THE SECOND PERIOD FROM 1980 TO1989

During this period new techniques were introduced in order to characterize mesomorphism intervals or to determine new properties (Cuculescu I., 1977; Sofron E., 1977; Moțoc C., 1979).

The mechanism of isotropic liquidus-cholesteric phase transition was investigated by determining the growth kinetic of choesteric spherulites of cholesteryl laurate-cholesteryl caprilate mixture (50–50% wt.). It was shown that the nucleation was bidimensional and the growth rate was decreased under d.c. electric fields (Popescu M. I., 1979). It was found that the growth was determined by the orientational relaxation of the molecules near the interface and that d.c. fields change some coefficients associated with dissipation effects during relaxation and the supersaturation. Under magnetic fields the growth kinetics of cholesteric spherulites was increased (Honciuc M., 1977).

Different techniques were used for determining phase transitions in mixtures of cholesteryl/sitosteryl esters such as optical microscopy, depolarizing light intensity during heating and cooling cycles, small angle light scattering under monochromatic light, changes in activation energies obtained from Arrhenius plots, etc. Some less common techniques were used. One of these consists in registering the electric current appearing when heating respectively, cooling a liquid crystal cell. A thermodielectric effect occurs when a phase transition is taking place (Moțoc C., 1978; Honciuc M., 1978; Socaciu M., 1985). A spectroscopic method was also used for revealing phase transition in binary systems of mesogenic compounds. It consists in determining the optical densities using visible light as significand optical absorption changes are noticed when a phase transitions takes place (Socaciu M., 1985, 1986).

Optical and electro-optical properties were devoted to new mixture of cholesteryl and sitosteryl esters.In most of them dyes were indroduced. Colour filters were obtained (Bena R., 1981) and useful optical properties, such as transmitance, absorption as function of temperature and applied electric voltage were determined.In some of these mixtures electrohydrodynamical instabilities were noticed. These consisted of pulsing instabilities of 0.02-0.05 Hz and were explained by using the theoretical model elaborated by Brand and Pleiner (Cuculescu I., 1983). A temperature dependence of these instabilities was also noticed (Cuculescu I., 1984). The anisotropies of electric and dielectric properties of cholesteryl; laureate-cholesteryl caprilate mixtures as function of temperature and applied electric field were determined (Moțoc C., 1984) and field effects were noticed when phase transitions were involved (Teodorescu N. H., 1981).

Photovoltaic effects were revealed in nematic 7A, in smectic sitosteryl undecilenate or crotonate and in cholesteryl chloride-cholesteryl laurate/crotonate mixtures, containing different additives, such as anthracene, Rhodamine B or sulphur. The effects are dependent on previous polarizing treatments and on the asymmetric configuration of tin oxide electrodes (Moțoc C., 1983, 1984; Roşu C., 1988).

It was also demonstrated that small additives of nonmesogenic compounds, exhibiting ferroelectric or pyroelectric properties may induce in cholesteric, smectic or nematic phases important ferroelectric properties. To exemplify, by adding KNO_3 to compensated mixtures of cholesteyl chloride-cholesteryl miristate/laurate, a rather high spontaneous polarization is induced and by adding biphenyl, anthracene and sulphur to smectic sitosteryl undecilenate/octanoate, the mixtures exhibited an electret behaviour (Moțoc C., 1983, 1984, 1987; Honciuc M., 1978; Roşu C., 1988).

A nondestructive technique for testing some electronic components was developed; nematic and cholesteric mesophases were used. In silicon and germanium wafers pinholes and mobile contaminants were revealed (Moțoc C., 1983); their evolution was followed in time and under the action of electric fields (Moțoc C., 1983; Mitroi R., 1986; Sofron E., 1987). Nematics were used to determine field distribution in transistors or integrated circuits (Sofron E., 1987; Moțoc C., 1988) and cholesterics to obtain the thermal maps of these electronic components (Moțoc C., 1982; Mitroi R., 1986; Sofron E., 1987).

A new book containing structural properties of thermotropic liquid crystals, physical properties of nematic and cholesteric phase and liquid crystal theories was elaborated by C. Moțoc and I. Muşcutariu (Moțoc C., 1986).

THE THIRD PERIOD FROM 1990 TO 1999

During this period our liquid crystal studies covered a large variety of topics involving thermotropic, lyotropic and polymeric liquid crystals.

Electro-optical and non-linear optical properties of some ferroelectric liquid crystals with high spontaneous polarization were investigated (Dascălu C., 1996); some of these displayed a chiral SmC sequence (Dascălu C., 1996). A method for obtaining free-standing ferroelectric liquid crystal films was applied and the temperature dependence of their spontaneous ring structure as well as their viscoelastic properties were determined (Dascălu C., 1997). The results are included into the PhD thesis of C. Dascălu (Dascălu C., 1997). A theoretical approach, relaying on thermodynamics of irreversible processes, was proposed to elucidate the origin of transient currents in ferroelectric liquid crystals (Roşu C., 1997). The polymorphism of the binary smectic mixture TBBA-cholesteryl miristate and the possibilities of using its specific thermo-electro-optical effect were examined (Socaciu M., 1991).

A lot of experimental data were obtained when nematic or cholesteric mixtures were subjected to laser fields.

Optical nonlinearities such as diffraction rings and self-focussing effects were examined as function of laser intensity in nematics and ferronematics (Petrescu E., 1998). For the last ones the effects are smaller. A theoretical approach was elaborated which proved to be in good agreement with the experimental data (Moțoc C., 1998). Laser radiation induced cholesteric-nematic transition was examined both theoretically and experimentally (Bena R., 1991). Laser – induced non-linear optical effects and electric properties in mixtures of fatty acid and cholesterol were also reported (Dumitru M., 1992; Honciuc M., 1992).

With reference to lyotropic liquid crystals reseaches were related to determining some electric parameters of binary mixtures exhibiting binary layers and considered as artificial membranes and also some binary solutions of lecithine or potassium laureate-decanol and lecitine solutions, some of them acted by magnetic fields (Popescu O., 1997, 1998).

New polymeric liquid crystals were investigated and dielectric and nonlinear optical properties were investigated (Moţoc C., 1996; Ionescu D., 1997). Complex spectroscopic and electrooptic studies were obtained and included into the PhD thesis of D. Ionescu (Ionescu D., 1997).

During this period studies of polymer dispersed liquid crystals were initiated by D. Mănăilă-Maximean and described in her PhD thesis [(Mănăilă-Maximean D., 1997). Electro-optical and conductive properties of new systems were investigated (Mănăilă-Maximean D., 1998) and a Patent referring to some technology of obtaining these films was obtained (Mănăilă-Maximean D., 1997). Bistability and multistability was noticed in some new otained PDLC and phase transitions were determined (Mănăilă-Maximean D., 1997, 1999).

THE FORTH PERIOD FROM 2000 TO THE PRESENT TIME

The main subjects investigated during this period are concerned with nematic or cholesteric phases containing different kinds of nonmesogenic additions (ferronematics, ferrocholesterics or azo-dye doped nematics) subjected to external fields and composite materials, such as DPLC.

Different kinds of nematics embedded with magnetic particles (ferronematics) were obtained and then subjected to magnetic fields or laser irradiation. Critial fields for magnetic Freedereicksz transition were determined (Moţoc C., 2003; Bena R., 2003), laser-induced effects (Petrescu E., 2004) and surface effects were also estimated Păun L. A (Păun L. A., 2008). The behaviour of a planar oriented ferronematic with positive dielectric and magnetic anisotropies, simultaneously subjected to an electric (E) and magnetic (B) field, was investigated. It was found that the transition from a planar to a homeotropic alignment is described into a plane [E, B] by an elipse having as halfaxes the critical fields for electric, respectively, magnetic Freedericksz transitions (Păun L. A., 2008). Theoretical studies on ferronematics with soft particle anchoring subjected both to magnetic and laser field were examined and the critical fields for magnetic and laser-induced Freedericks transitions were also determined (Bena R., 2002). In such systems the influence of surface effects was also considered (Bena R., 2002; Petrescu E., 2007).

The influence of external fields on the orientational behaviour of ferrocholesterics was theoretically examined. In case of ferrocholesterics with positive magnetic anisotropy it was found that the critical fields for the cholesteric ↔ nematic transitions are increased when compared to those intervening in pure nematics (Păun L. A., 2008). The theoretical and experimental results, obtained by

E. Petrescu, C. Moțoc and R. Bena, relating magnetic and laser field – induced effects in ferronematics or ferrocholesterics are summarized in the monograph.

The effects induced by magnetic field into a nematic mixtures doped with different kinds of azo-dyes were intensively studied (Moțoc C., 2005, 2006). It was found that a small amount (2% by weight) of azo-dye induced optical activity into the mixture. The linear polarized light, of a He-Ne laser, passing through the sample acted by a magnetic field (having an intensity higher than the critical one for magnetic Freedericksz transition) become elliptically polarized. The light varied quasiperiodically when increasing/decreasing the magnetic field. The effects of UV light were revealed (photoisomerization); ellipticity and magnetic-induced bifrefringence were estimated (Moțoc C., 2006). A static and dynamic investigation in azo-dye doped nematics has also been performed. It resulted that the critical field was decreased in samples containing the „trans" isomer and increased in those containing the "cis" isomer. A theoretical model was elaboraed to explain this phenomena and applications are suggested (Iacobescu G., 2008). Similar results were obtained when NLC was doped with Prussian blue (Roşu C., 2009). Optical, electro-optical, and thermal properties of new synthetized azo-dyes compounds were investigated (Tudoran S. C., 2000).

An important number of papers in the field of PDLC were elaborated. A synthezis of these studies was included by into a momography (Mănăilă-Maximean D., 2008).

Thermally stimulated depolarization currents and optical transmission currents were used to characterize new composites (Roşu C., 2002, 2003; Mănăilă-Maximean D., 2004). The influence of electric field on phase transition temperatures and on electrical and optical properties were followed (Mănăilă-Maximean D., 2001, Klosowicz J. S., 2004). Studies on colloidal silica particles in NLC copolymer particles in NLC were performed (Mănăilă-Maximean D., 2004, 2007). Applications of PDLC films, such as infrared filters were proposed (Mănăilă-Maximean D., 2004).

A book containing general information with reference to liquid crystal structures, properties and applications were elaborated by Moțoc C. and Iacobescu G. (Moțoc C., 2004).

3. CONCLUSIONS

It may be considered that liquid crystal researches in the Polytechnic School of Bucharest have been succesfull. More books (Sofron E., 1976; Moțoc C., 1986; Moțoc C., 2004; Mănăilă-Maximean D., 2008; Petrescu E., 2008) and a great number of scientific papers have been published in well known periodicals; these are: Molecular Crystals and Liquid Crystals (Moțoc C., 1978, 1979; Honciuc M., 1983; Dumitru M., 1992; Roşu C., 2000, 2002; Mănăilă-Maximean D., 2001, 2004; Petrescu E., 2004; Gili M. J., 2004; Klosowicz J. S., 2004; Cîrcu V., 2009), Journal of Magnetism and Magnetic Materials (Bena R., 2002, 2003; Moţoc C., 2006; Petrescu E., 2008; Iacobescu G., 2008), Modern Physics Letters B (Cuculescu I., 1995; Mănăilă-Maximean D., 1997, 1999; Roşu C., 1997, 2002), Journal of Optoelectronics and Applied Materials (Moţoc, 2005; Roşu C., 2009), Phase Transitions (Roşu C., 1983; Moţoc C., 1984; Socaciu M., 1985, 1986) and in other periodicals (Sofron E., 1977, 1979, 1983; Cuculescu I., 1983; Sterian P., 1986; Petrescu E., 2000; Roşu C., 2003; Moţoc C., 2006).

A lot of papers were published in the periodicals of the Romanian Academy of Sciences (Iliescu A., 1974; Moţoc C., 1976, 1977, 1981, 1988, 1996, 1998; Honciuc . M., 1978, 1979, 1984; Enache A., 1984; Socaciu M., 1991; Dascălu C., 1996, 1997; Petrescu E., 1998), of Universites (Popescu M. I., 1979; Luca E., 1979; Honciuc M., 1978, 1980; Teodorescu N. H., 1980; Cuculescu I., 1977, 1981, 1983; Bena R., 1981, 1991; Moţoc C., 1977, 1978, 1979, 1980, 1981, 1983, 1984, 1995, 1999, 2003; Mitroi R., 1987; Roşu C., 1995; Dascălu C., 1996; Covaci D., 1997; Petrescu E., 2006) or were included in the Proceedings of International Conferences (Popescu M. I., 1975; Teodorescu N. H., 1978, 1981, 1982; Roşu C., 1997; Mănăilă-Maximean D., 1997, 1998; Dumitru M., 1997; Moţoc C., 1983, 1994, 1997, 1998) or National Conferences (Moţoc C., 1976, 1979, 1981, 1982, 1983; 1986; Honciuc M., 1979, 1986; Cuculescu I., 1979; Teodorescu N. H., 1980, 1984; Sofron E., 1977, 1983, 1985, 1988; Roşu C., 1982).

From 1972 up to the present time over 15 PhD theses were elaborated by young assistents and specialists (Sofron E., 1977; Cuculescu I., 1977; Honciuc M., 1977; Savin O., 1983; Roşu C., 1988; Socaciu M., 1985; Mitroi R., 1986; Bena R., 1986; Iacobescu G., 1995; Ionescu D., 1997; Dascălu C., 1997; Mănăilă-Maximean D., 1997; Popescu O., 1998; Petrescu E., 1998; Tudoran Cr., 2000; Puică M., 2001; Păun L. A., 2008).

Many applications were sustained by different grants were obtained (with the followimg themes: *Achievement of visible light sensitive electro-optical devices*; *Electric and electro-optic studies of some organic substances for applications in aeronautic, electrotechnic and telecommunication industries, zootechny and medicine*; *Biological implications in some mechanisms of biological membranes*; *The establishing of biological implications of some membrane mechanisms*;*Order-disorder phenomena in condensed matter physics*; *Elucidation and setting biological implications of some membrane mechanisms*; *Electro-optice and magneto-optic effects in composite materials – liquid crystals-organomagnetic-polimers*; *Effects induced by electric and magnetic fields and surface phenomena in thermotropic and polymeric liquid crystal devices*).

By through research carried out has also taken the protection of technical solutions with patents (Tărîcă St., 1974; Sofron E., 1977; Sofron E., 1980; Sofron E., 1981; Socaciu M., 1988; Mănăilă-Maximean D., 1997) and innovations (Sofron E., 1987; Sofron E., 1988).

In 1983, dr. Iuliana Cuculescu, Rodica Eleonora Bena, Ana Enache and in 1984 Prof. Dr. Cornelia Moţoc, dr. Maria Honciuc, Constantin Roşu, Răzvan Mitroi were awarded by the Romanian Academy of Sciences with the "Constantin Miculescu" Prize for contributions into the liquid crystals physics and their industrial applications.

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