"Alexandru Ioan Cuza" University
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CONTRIBUTIONS TO IMPLEMENTING SOME METHODS FOR CHARACTERIZING ANISOTROPIC MEDIA

Ph. D. Thesis Abstract

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Part I Study of literature

Chapter I

The present stage of research in the field of anisotropic media

Anisotropic media

The transparent natural crystals, stretch polymer films and the liquid crystals are media which present the property of optic anisotropy [1-7].

The polymer films become anisotropic by stretching under uniform heating, so polymer chains are ordered along the stretching direction. In this way the polymer foil becomes equivalent of an anisotropic crystalline layer [5].

The liquid crystals (LC) are intermediate phases between the isotropic liquids and the crystalline solids [6]. LC has several mechanic properties of the liquid state (fluidity, the capacity to form drops) and at the same time they are similar to crystals as their optical, mechanical, electrical and magnetic properties have anisotropy [7].

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Part II My contributions

Chapter II

Determining the main refractive indexes of some anisotropic media

1. Determining the main refractive indices of N-(4-metoxybenziliden)-4-butylaniline liquid crystal

The main refractive indexes of N-(4-metoxybenziliden)-4-butylaniline (MBBA) liquid crystal (LC) in pure state from the Merck Company at a temperature of 23°C without purification were measured in the absence as well as in the presence of an external electrostatic field using Rayleigh interferometer [1-4].

The extraordinary refractive index decreases together with the increase of the wavelength and slowly increases at the same time with the increase of the intensity of the electric field. The ordinary refractive index decreases at the same time with the increase of the wavelength of the incident radiation.

The birefringence of MBBA LC increases when an external electric field is applied. The highest birefringence (0.2488) is obtained for the incident radiation with the shortest wavelength (435.8 nm) in the presence of an electric field E=142.85 kV/m.
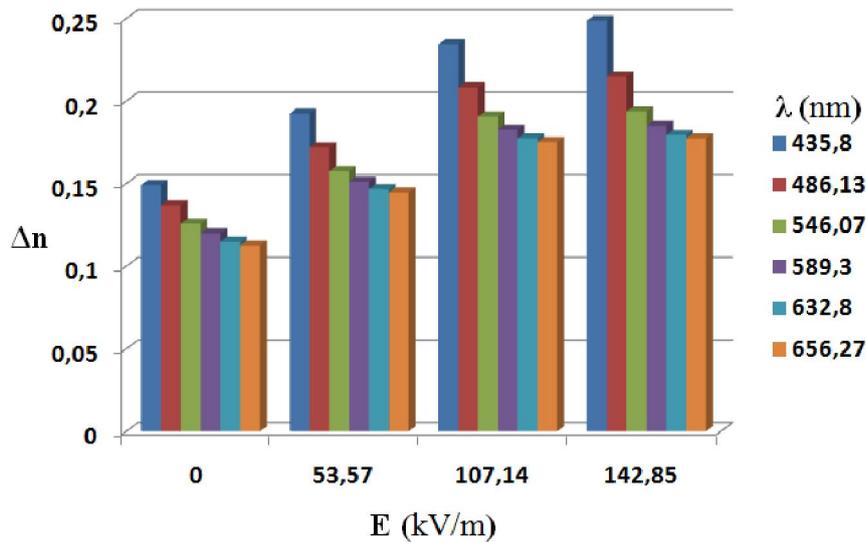

Fig. 1 Birefringence of MBBA LC vs. intensity of the electric field.

The birefringence of MBBA LC was also measured by using the method of the channeled spectrum. The data obtained through this method are the same with those obtained through the interferometric method.

By using the main refractive indexes, the birefringence of MBBA LC as well as the relations Cauchy and Sellmeier [5-9], there have been made various simulations which allow estimating the refractive indexes for each wavelength in the visible range.

The birefringence of MBBA LC increases at the same time with the intensity of the external electric field. One can notice that the birefringence increases less, tending towards saturation when the intensity of the external electric field is between (107.14-142.85) kV/m in the range (550-800) nm.
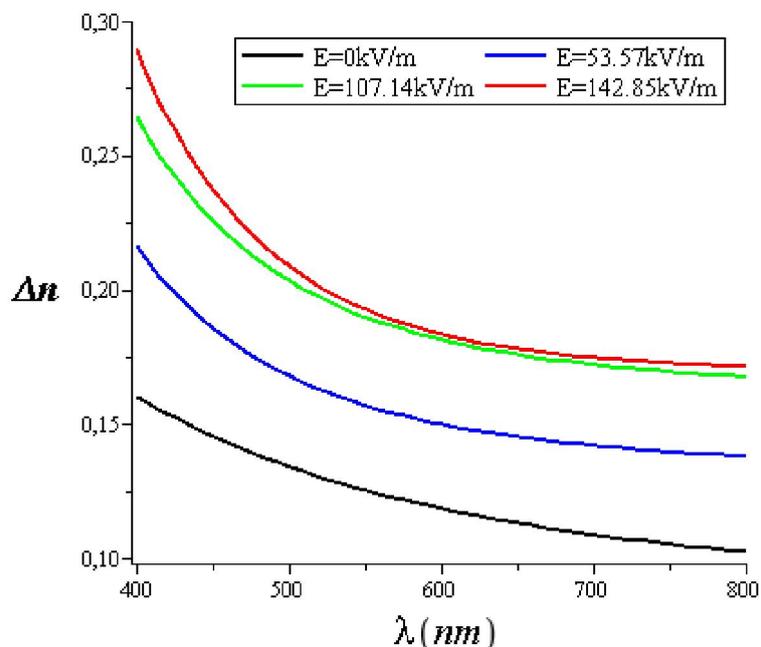
Fig. 2 Birefringence of MBBA LC vs. wavelength.

2. Determining the main refractive indexes of the ester of the alkolxy benzoic acid in tetrachlormethane liquid crystal

The dispersion of the birefringence in the visible range of liquid crystal (LC) layer from the polymer ester of the alkolxy benzoic acid (PEABA) in tetrachlormethane (TCM) was determined from the channeled spectrum recorded in the presence and absence of the external electrostatic field [10].

The polymeric ester of the alkolxy benzoic acid (Sigma – Aldrich Company) in tetrachlormethane has a lyotropic liquid crystal phase achieved by a parallel ordering of the secondary molecular chains owing to the collective dipole interactions [11-13].

The channeled spectra were recorded with a Specord UV VIS Carl Zeiss Jena spectrophotometer in the spectral range (14000 – 23000) cm^{-1} in the absence and presence of an electrostatic field. From the channeled spectra, the maximum and minimum positions were identified and the birefringence of PEABA LC in TCM was calculated.

The total dispersion of the birefringence in the spectral range (14000-23000) cm^{-1} is of 0.0014 in the absence of the electric field and becomes 0.0829 when the sample is under the influence of an external electric field.

3. Determining the main refractive indexes of some feldspar and mica crystals.

In the last part of the chapter an experimental method was refined for determining the main refractive indexes of a biaxial crystal by using the equation of the surface of the refractive indexes and there were made calculations in order to determine the thickness that the samples should have in order to be used as special plates [14].

In the first stage the main refractive indexes were measured with the Rayleigh interferometer n_a and n_c. After that the anisotropic crystal layer was inclined with $30°$, $45°$ or $60°$ and n_c as well as the secondary refractive index n_3 were measured. The third main refractive index n_b was determined by solving the equation of the surface of the refractive indexes [15-17]

$$(1) \quad (n^2 - n_b^2)[\alpha^2 \cdot n_a^2 \cdot (n^2 - n_c^2) + \gamma^2 \cdot n_c^2 \cdot (n^2 - n_a^2)] = 0$$

The solutions of this equation are:

$$(2) \quad n_{1,2} = \pm n_b$$

$$(3) \quad n_{3,4} = \pm \frac{n_a \cdot n_c}{\sqrt{(1-\gamma^2) \cdot n_a^2 + \gamma^2 n_c^2}}$$

Using the methods described above the main refractive indexes of some mica and feldspar samples coming from the area of Conțu-Negovanu (Lotru Cibin Mountains) were measured.

4. Conclusions

The extraordinary and ordinary refractive indexes of MBBA LC decrease at the same time with the increase of the wavelength. The extraordinary refractive index increases at the same time with the increase of the intensity of the electric field.

The birefringence increases when an external electric field is applied between the internal walls of the cell where the liquid crystal is because the molecules are oriented with the electric dipole moment in the direction of action of the electric field intensity.

The feldspar samples have an average birefringence of 0.011 while the mica samples have a birefringence of 0.044 for a wavelength of 589.3 nm.

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Chapter III

Interferential devices

1. Wood filters

The Wood interferential polarization filters are made of a transparent anisotropic material under the form of a layer having a constant thickness and placed between two polarizers [1-2]. The transmission sections of the polarizers can be parallel or perpendicular [3].

The transmission factor of these filters varies with wavelength of the incident optic radiations having alternating minimum and maximum values so that the spectrum of the transmitted radiations has a channeled aspect [4].

The transmission factor of a Wood filter was simulated depending on the wavelength and the birefringence CL MBBA ($L = 14 \ \mu m$).
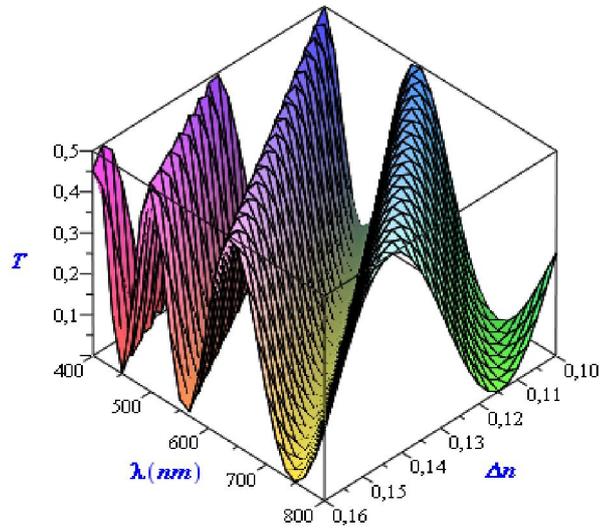

Fig. 1 Transmission factor of the Wood filter with

MBBA LC vs. wavelength vs. birefringence.

The number of bands in the channeled spectrum increases at the same time with the birefringence of the liquid crystal layer.

If more liquid crystal layers are added, one can see a decrease of the semi-width of the transmission bands in the channeled spectrum (Fig. 2, 3).
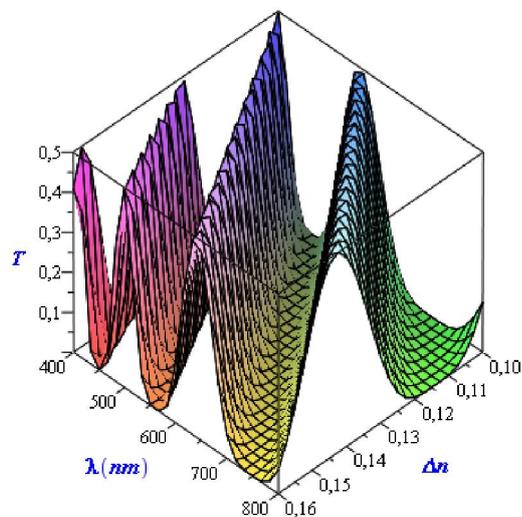

Fig. 2 Transmission factor of the Wood filter with 2 elements (MBBA LC)

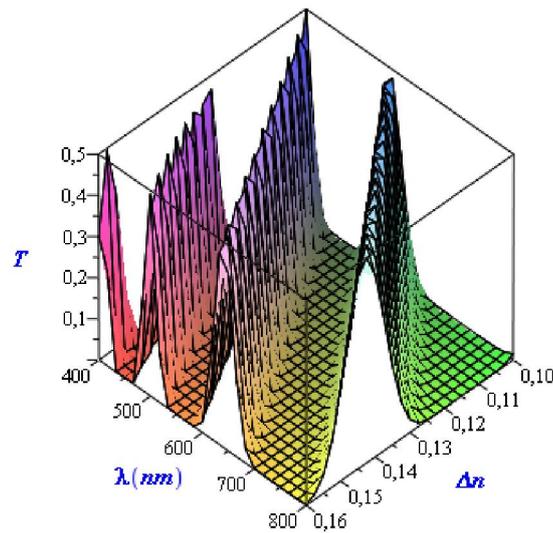*vs. wavelength vs. birefringence.*

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Fig. 3 Transmission factor of the Wood filter with 5 elements (MBBA LC)
vs. wavelength vs. birefringence.

In this chapter simulations of the Wood filter were made when the LC is situated in the electric field. Depending on the value of the intensity of the electric field applied to the sample, one may get in case that the transmission maximums in the spectrum are replaced by the transmission minimums.

2. Lyot filters

The Lyot is an optic filter containing one or more stages. A stage is made up of an anisotropic layer between two polarizers (with parallel or perpendicular directions) [5-7]. The anisotropic layer on the first stage has a thickness which is half of the thickness of the layer on the second stage [8]. The angle between the optic axis of the anisotropic layer and the transmission direction of the polarizer is 45°.

The transmission factor of a Lyot filter with one and two stages when the thickness of the layer is 7 µm and 14 µm is represented in Fig. 4.
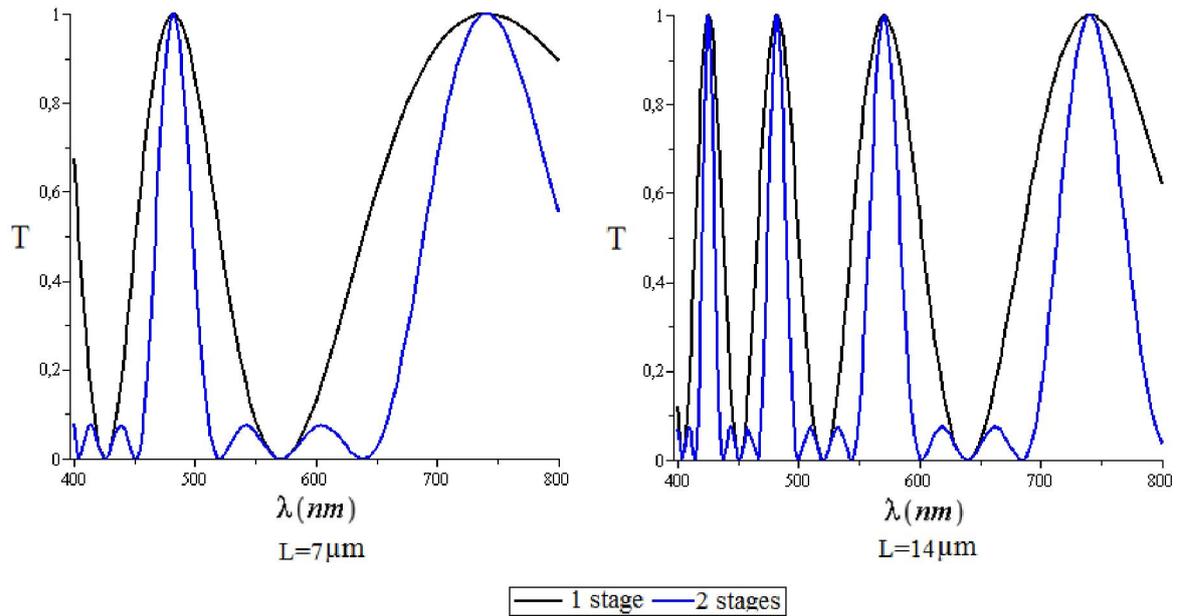
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Fig. 4 Transmission factor of the Lyot filter vs. wavelength.

In Fig. 4 one can see that the number of bands in the channeled spectrum increases at the same time with the increase of the number of stages.

3. Conclusions

When the main refractive indexes of a transparent anisotropic medium are known, different devices can be projected by changing the polarization state of the light. Using simulation programs, the variation of the transmission factor with wavelength for different interference filters can be represented.

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Chapter IV

Optical properties of the PVA films containing phthalazinium ylids

1. Introduction

In this chapter the analysis of the birefringence and the dichroism of the polyvinyl alcohol (PVA) films with phthalazinium ylids were followed with a view to emphasizing the ability of the dye molecules of arranging themselves along the polymer chains in the processes of stretching the PVA films [1]. The results of this study are important for establishing the conditions under which the PVA films containing ylide molecules become dichroic.

The chemical structure of the studied phthalazinium ylides [2] is presented in Fig. 1 and its substitutes in Table 1.
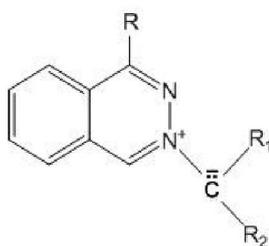

Fig. 1 Structure of the studied phthalazinium ylides.

Table 1 Subtitutes of the studied phthalazinium ylides.

Name	Notation	Substitutes	
Phthalazinium-di-benzoyl methylid	FY1	R	$-H$
		R_1	$-COC_6H_5$
		R_2	$-COC_6H_5$
6-methyl-phthalazinium-di-benzoyl-methylid	FY2	R	$-CH_3$
		R_1	$-COC_6H_5$
		R_2	$-COC_6H_5$
6-methyl-phthalazinium-cyano-carbethoxy-methylid	FY3	R	$-CH_3$
		R_1	$-CN$
		R_2	$-CO_2C_2H_5$

2. Characterizing phthalazinium ylide molecules using HyperChem program

The phthalazinium ylide molecules were analyzed and optimized by using a calculation program at a quantum level, HyperChem [3] in order to determining their physical chemical properties and of comparing them.

3. The study of the PVA films with phthalazinium ylides

The films were obtained from PVA (M_w=65000-85000) and distilled deionized water (concentration 20%). The mixture was heated at (80 - 90) $^{\circ}$C for 6 hours using a plate with mechanic stirrer. A part of the gel obtained was used for embedding the phthalazinium ylides in the PVA solution at room temperature.

The absorption spectra

The absorption spectra of the PVA films containing phthalazinium ylides were recorded by means of a Specord UV-VIS Carl Zeiss Jena spectrophotometer.

The electronic bands in the visible field of phthalazinium ylides under study are situated at 23600 cm^{-1} for FY1 ylide, 23200 cm^{-1} for FY2 şi 24400 cm^{-1} for FY3 ylide. The absorption bands of the ylides can be attributed to an intramolecular transfer of electronic charge from carbanion towards heterocycle [1].

The dichroism of PVA films with phthalazinium ylides

The dichroism of the polymer films was estimated by using formula [4]:

$$(1) \quad D = \frac{A_{\parallel} - A_{\perp}}{A_{\parallel} + A_{\perp}}$$

where $A_{\parallel}$ and $A_{\perp}$ are the maximum absorbencies in the spectra recorded with linearly polarized light having the electric field intensity parallel and perpendicular on the stretching direction.

The dichroism of PVA films with phthalazinium ylide molecules was calculated by using the absorbencies measured in the absorption spectra and formula (1).

In Fig. 2 one can notice the representation of the difference (ΔD) between the maximum dichroism and the dichroism obtained at a certain degree of stretching depending on the degree of stretching (γ).
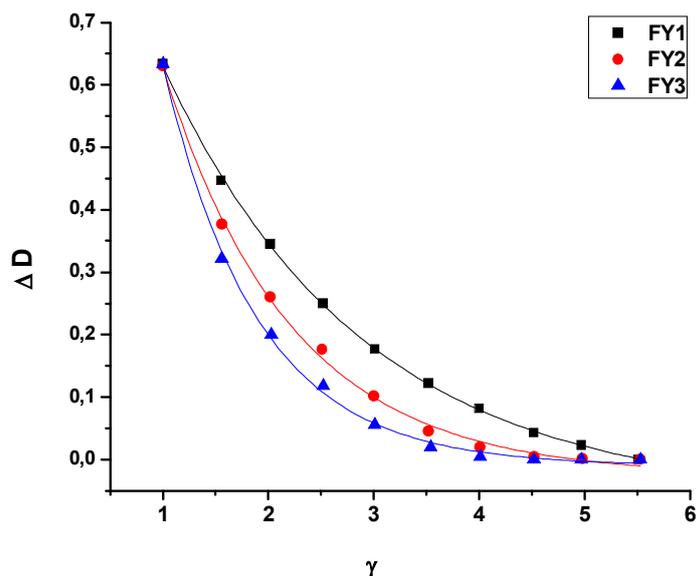
Fig. 2 ΔD vs. γ for films with phthalazinium ylides.

In Fig. 2 one can notice that ΔD decrease at the same time with the degree of stretch. When $\gamma > 4$ there is a saturation of the alignment phenomenon and ΔD tends towards 0 for the films containing FY2 and FY3. The highest value of the dichroism was obtained for the film containing FY3 ylide and the lowest for the film containing FY1 ylide.

The orientation degree of ylide molecules in PVA films

The orientation degree of phthalazinium ylide molecules was calculated by means of formula [1, 4]:

$$(2) \quad g = \frac{2D}{3 - D}$$

The difference (Δg) between the maximum degree of orientation and the orientation degree at a certain degree of stretch has the same decreasing tendency with degree of stretch (γ) like the dichroism.

Birefringence of PVA films

The birefringence of the films was measured using the Babinet compensator calibrated for the monochromatic radiation of a sodium lamp [5, 6].

The variation of the birefringence of pure PVA films and PVA films containing ylides depending on the degree of stretch is given in Fig. 3.
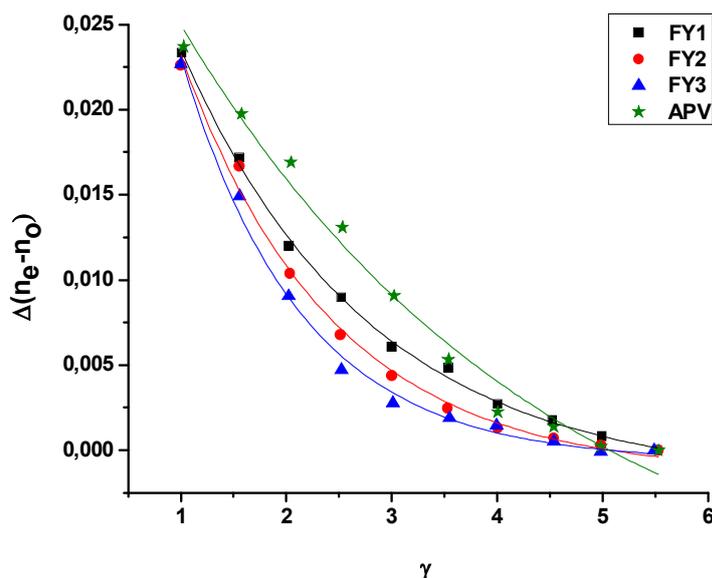
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Fig. 3 Δ(n_e-n_o) vs. γ of pure PVA films and of PVA films with ylides.

In Fig. 3 one can see that the birefringence of PVA films containing ylides is higher than pure PVA films up to the degree of stretch 3.5. For degree of stretch higher than 3.5 the birefringence of all samples is comparable. One can also notice a saturation of the birefringence for PVA films with FY3 ylide (the molecule with the largest dipole moment) and the lowest values for the films containing FY1 (the molecule has the shortest dipole moment).

The similarities of the dependencies described above suggest the fact that a formula such as:

$$(3) \qquad P(\gamma) = P_{max} + A \cdot \exp\left(-\frac{\gamma}{t}\right)$$

can describe the dependence of the anisotropic parameters (birefringence, dichroism, orientation degree) on the degree of stretch of the films. Formula (3) was used to calculate the P parameters characterizing the anisotropy of films with stretched ylides. There was found a linear dependence between the experimentally determined values and the calculated ones.

4. Conclusions

The phthalazinium molecules of the studied ylides have non-planar structures. The length of the ylide bond C(9) - N(2) has different values: 1.3827 for FY1, 1.3777 for FY2 and 1.3830 for FY3.

The studied molecules are polar and polarizable. The FY3 molecule has the largest dipole moment and the lowest polarizability. Molecules FY1 and FY2 au have large polarizability.

The most stable molecule is FY3 which has the highest module of the HOMO energy. This molecule has the highest electrophilicity index.

The highest dichroism belongs to the films with FY3 and the lowest to those with FY1. The orientation degree of the ylide molecules is the highest in case of the films with FY3 and the lowest in the films with FY2.

The highest birefringence can also be seen in the case of films with FY3 ylide. When the degree of stretching $\gamma > 4.5$ there takes place an alignment phenomenon of the polymer chains and the birefringence tends towards a constant value in the case of the films containing FY2 and FY3.

The birefringence, the dichroism, and the orientation degree of the PVA films containing phthalazinium ylides increase exponentially with the increase of the degree of stretch.

The PVA films containing phthalazinium ylides have higher birefringence than pure PVA films.

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Chapter V

Optical properties of the PVA films containing pyridazinium ylide

1. Introduction

In this chapter 3 pyridazinium ylide molecules were studied using the HyperChem program in order to find their electro-optic properties in order to explain the dichroism of the polymer foils containing ylides.

Pyridazinium ylides (PY) are amphionic compounds in which a nitrogen atom with positive charge hybridized sp^3 belonging to a pyridazinic cycle is covalently bond to a carbon atom with negative charge (carbanion) whose hibridization between sp^2 and sp^3 strongly depends on its attached substitutes (Fig. 1) [1-3].

The chemical structure of the studied carbanion pyridazinium ylides is presented in Fig. 1 and its substitutes in Table 1.

Fig. 1 Structure of the studied pyridazinium ylides.

Table 1 Substitutes of the studied pyridazinium ylides.

Name	Notation	Substitutes	
Pyridazinium p-methoxy –fenacylid	PY1	R	$-H$
		R_1	$-CO-C_6H_4-OCH_3$
Pyridazinium p-benzoy-fenacylid	PY2	R	$-H$
		R_1	$-CO-C_6H_4-C_6H_5$
Pyridazinium p-nitro-fenacylid	PY3	R	$-H$
		R_1	$-CO-C_6H_4-(NO_2)_p$

2. Characterizing pyridazinium ylide molecules using HyperChem program

The pyridazinium ylide molecules are polar. The molecule PY3 has the largest dipole moment and molecule PY2 has the highest polarizability.

The stability of the studied molecular structures is given by the value of the total energy which is negative. The module of the total energy is higher for molecules PY2 and PY3. Molecules PY2 and PY3 have high forming heating which indicate the fact that they are unstable from a thermodynamic point of view.

3. The study of the PVA films with pyridazinium ylide

The pure PVA films and those containing pyridazinium ylides were prepared according to the recipe described in the previous chapter.

The absorption spectra

The maximum electronic bands in the visible field of the studied pyridazinium ylides are situated at 21580 cm^{-1} for ylide PY1, 21500 cm^{-1} for PY2 and 21420 cm^{-1} for ylide PY3. The changes induced by stretching the foils in the normalized spectra of the PVA films are similar to those signaled in the previous chapter.

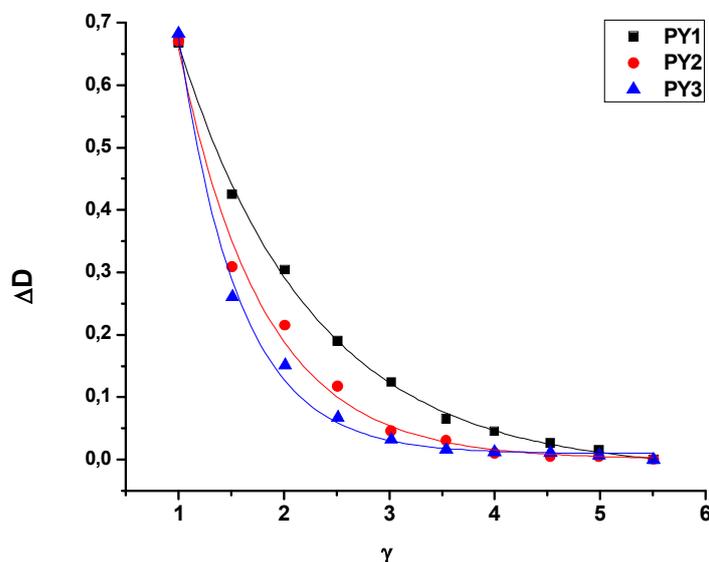**The dichroism of PVA films with pyridazinium ylide**

Fig. 2 ΔD vs. γ for films with pyridazinium ylides.

In Fig. 2 one can see the graphic representation of the difference (ΔD) between the maximum dichroism and the dichroism obtained at a certain degree of stretch depending on the degree of stretch (γ). From Fig. 2, one can see that ΔD decreases exponentially with the increase of the degree of stretch (γ). The saturation process takes place faster in the case of PVA films containing ylides PY2 and PY3. The highest value of the dichroism was obtained for films containing ylide PY3, and the lowest for films with PY1.

The orientation degree of ylide molecules in PVA films

The difference (Δg) between the maximum degree of orientation and the orientation degree at a certain degree of stretch decreases exponentially with the degree of stretch (Fig. 3) according to the formula:

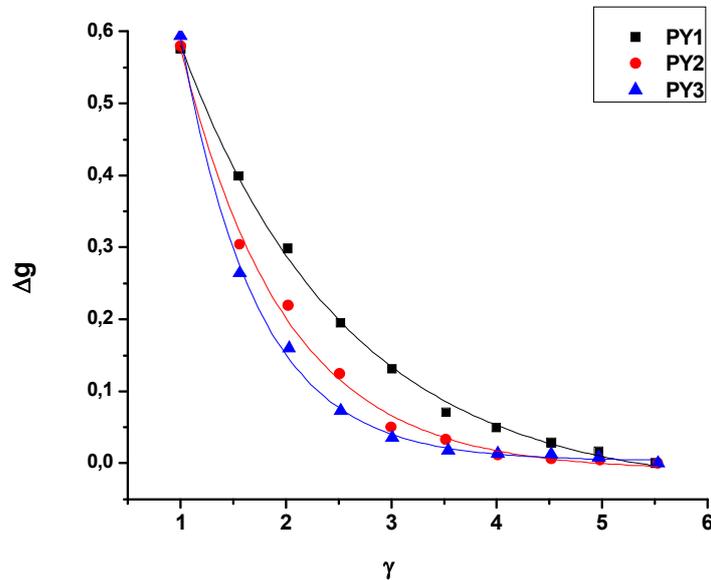(1) $$g_{max} - g(\gamma) = g_0 + A \cdot \exp\left(-\frac{\gamma}{t}\right)$$

Fig. 3 Δg vs. γ for films with pyridazinium ylides.

The difference (Δg) varies up to 0.6 when the degree of stretch is equal to the unity. For the same degree of stretch Δg is higher for films with PY1. For high degree of stretchs, one can notice there appears the saturation process.

Birefringence of PVA films

The difference between the maximum birefringence and the birefringence obtained at a certain degree of stretch depending on the degree of stretch is presented in Fig. 4.
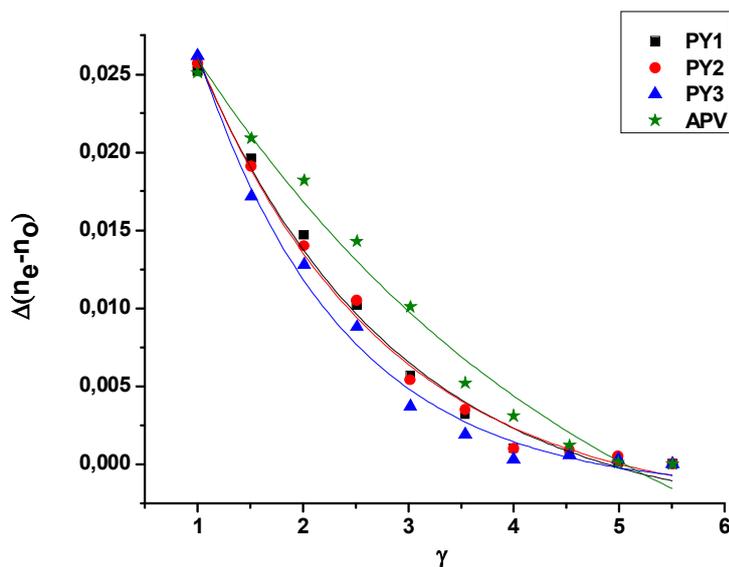
Fig. 3 $\Delta(n_e-n_o)$ vs. γ of pure PVA films and of PVA films with ylides.

The birefringence of the PVA films containing pyridazinium ylides is higher than the birefringence of pure PVA films. The highest birefringence was obtained in the case of films containing PY3 molecules (the molecules have the largest dipole moment).

4. Conclusions

The studied pyridazinium ylides are polar and polarizable. The ylide molecule PY3 has a plane structure, the largest dipole moment and the polarizability equal to that of the PY2 molecule.

The films containing PY3 ylide have the highest dichroism for all the degrees of stretch. The orientation degree of PY3 ylide molecules in the PVA is the highest. The highest birefringence can be noticed in the films containing the ylide PY3.

Like in the previous chapter, one can see that the birefringence, the dichroism and the orientation degree of the molecules in the PVA films containing ylides increase exponentially with the increase of the stretch degree.

References
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