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The Effect of Metallomesogens on Electro-Optical Properties of a Flexible Polymer Dispersed Liquid Crystal

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Abstract: The electo-optical properties of flexible PDLC films doped with one mono-ligand alkyl/alkoxy/alkeneoxy-azobenzene with Pd metal-complex and three bi-ligands salicylaldiminates with Cu metal-complex metallomesogens (MOMs) as a function of MOM concentration under natural aging have been evaluated. The results indicate that, MOM-doped PDLC films exhibit improvements of their switching voltages and optical transmissions before and after natural aging.

Keywords: Metallomesogen, liquid crystal, flexible PDLC, electro-optics, voltage, transmission natural aging.

1 Introduction

Metallomesogens (MOMs) represent a class of inorganic liquid crystals, which incorporates metal complexes in organic mesogenic structures. In the past two decades, the chemistry of MOMs have been expanded enormously where many different chemical structures have been reported in the literature as potential materials for applications in electro-optical and display devices [1-6]. Some of the most notable developments in MOMs have been on the relationship between the shape and the type of metal complexes mesomorphism. According to these studies, some interesting correlations between the chemical and mesomorphic structures of metallomesogens have been found. Although some structure-property relations have been found for individual chemical structures of MOMs, they are not yet conclusive within the overall perspective and the exisitng materials, which are still far from being useful for possible application.

The presence of metal complexation chemistry of liquid crystals could provide additional physical and optical features not present in organic mesogenic material. These features include large electrical polarizability, large refractive index, high birefringence and order parameter, mesogenic stability, additional selective absorption and dichroic property. Therefore, by combining supra-molecular organic mesogenic and metal complexation, it is possible to provide an opportunity for utilizing MOMs in liquid crystal devices. In spite of extensive recent studies on MOM's chemical structures, only some attempts have made for their potential applications in the literature [7,8], including for photoluminescence [9-11], electroluminescence [12,13], magnetic [14,15] and electric [16-18] applications. Also, few scientific and patent literature have reported on some rod-like and discotic MOMs as dichroic dyes, non-linear optics, thermal recording, thermochromism, passive optical filters, photo-sensing, laser addressing, optical and thermal recording, polarizing flms, radiation absorbing films, ferroelectricity, ferromagneticity, electroconductivity, reaction catalysts, mesogenic intermediates, ink jet and security printing, medicinal and agricultural materials [19-27].

In spite of these proposed applications, until now no MOM material has not been yet appeared for even simple commercial guest-host liquid crystal application. Among the major draw backs of existing MOMs are their high transition temperatures, decomposition at high temperature, inaccesible mesophase range and low chemical stability. Inorder to overcome these drawbacks, in addition to proper molecular engineering and synthesis, an alternative approach for commercial application of MOMs will be the miscibility of these materials in liquid crystal devices.

In the present work, the phase diagrams through physical mixing of model rod-like MOM structures consisting of mono-ligand alkyl/alkoxy-azobenzene Pd complex and bi-ligand Cu, Ni and Pd complex salicylal-diaminate

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structures have been analysed. Accordingly, the mesogenic miscibility and eutectic behaviour in these binary model MOM materials have been evaluated. In addition, we carried out ternary phase diagrams consisting of a eutectic MOM-ligand mixture with two other MOMs and three commercial NLC materials to demonstrade potential use of MOMs in commercial applications.

2 Experimental

2.1 Materials:

A series of metallomesogen materials including bi-ligand *salicylaldiminates* with Cu metal complexes and a mono-ligand *alkyl/alkoxy/alkeneoxy-azobenzene* with Pd metal complex have been synthesized.

The general chemical formula of synthesized ligands and *mono-ligand* MOM is based on a common class of Palladium (Pd) metal complex and alkyl/alkoxy-azobenzene, as is presented in Figur-1. According to this formula, the chemical structures of utilized mono-ligand MOM in this study is obtained by a ligand incorporated in Pd-alkyl/alkoxy-azobenzene chemical structure. The details of the synthetic procedures of this class of MOMs have been entioned elsewhere [28-29].

Figure-1 mentioned the general structural variations of bi-ligand formula are obtained by changing the structure of the ligand's terminal groups R and R', which are different or the same having the same or different metal complex ion. The details of general synthetic procesdures of this class of bi-ligand MOMs chemistry, which has been part of an extensive industrial development project, have been reported elsewhere [30-35].

With reference to Figure-1, the following four MOM structures have been utilized:

Mono-ligand MOM:

- *L2-Pd*: R= -C_6H_{13}; R'= -$O(CH_2)_2CH=CH_2$

Bi-Ligand MOMs:

- *12-8NCu*: R= -O-(CH2)11-CH3; R'= -(CH2)7-CH3
- *LC1-Cu*: R= -O-(CH2)6-OOC-CH2-CH3; R'= -
- (CH2)7-CH3
- *A11O-8NPd*: R= -(CH2)7-CH3; R'= -O-(CH2)6-OOC-CH=CH2

The mono-ligand MOM component L2-Pd with *alkyl/alkoxy/alkeneoxy-azobenzene* metal complex structure exhibited a *monotropic nematic* phase and the overall mesomorphic stability of L2-Pd was within -35°C and +66°C temperature range. The bi-ligand *CL1-Cu* and 12-8NCu MOMs with *Salicylaldiminate* structure metal-complexes exhibited *enantiotropic nematic* phases and the mesophase stability of *CL1-Cu* and 12-8NCu were within 80-130°C temperature range. In Table-1, transition temperatures and mesomrphisms of utilized MOMs and TNO623 are tabulated on heating and cooling modes.

The utilized materials consisted of UV-curable pre-polymer resin NOA-65 (Norland Optical Adhesives), nematic liquid crystal mixture TNO623 (Hofmann LaRoche), Micro-Pearl micro-spacer (Sekisui), Irgacure819 photo-initiator (Sigma-Aldrich) and 200 □/□ ITO-PET film supports (Sheldahl). All materials were used without further purification.

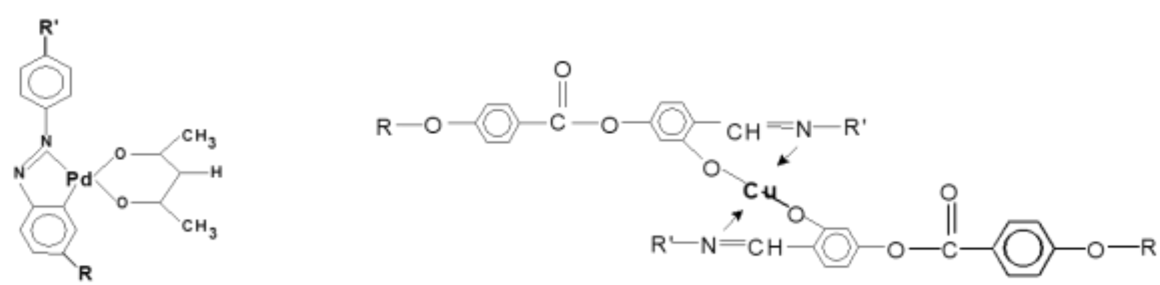

Fig.1: The formula of *alkyl/alkoxy/alkeneoxy-azobenzene* (left) and *Salicylaldiminate* (right) MOMs.

Table 1: Transition temperatures and mesomorphism of MOMs and TNO623 on heating (red) and cooling (blue) modes.

Compound	Transition Temperature (°C)				Mesophase
	Heating		Cooling		
	T_{cm}	T_{mi}	T_{im}	T_{mc}	
L2-Pd-acac	66.2	-	43.1	- 35	Monotropic Nematic
TNO623	-	104	102	(-35)	Enantiotropic Nematic
	T_{cm}		T_{mi}		
CL1-Cu	79		116		Enantitropic Nematic
12-8NCu	103.5		129		Enantitropic Nematic
A11-O-8NPd	112		144		Enantitropic Nematic

2.2 Sample Preperation:

As mentioned elsewhere, the PDLC preparation, was carried out with Polymerization Induced Phase Separation (PIPS) method by UV radiation [36-38]. The flexible PDLC films were prepared by a custom-made in-situ coating and lamination system of various homogeneous mixtures of NOA65 pre-polymer and TNO623 (50/50) including 1% Irgacure819 photo-initiator and pre-heated for 10 minutes at relevant temperature. The thickness homogeneity of PDLC layers were achieved by utilizing 0.2% of plastic micro-spacers. The uncured PDLC formulations were then poured between the vertical gap of two rolls of ITO-PET supports films on a custom-made plastic coater/laminator system. Under the coating rolls, the uncured PDLC films were passed through a pressure roll to insure the uniformity of the film by micro-spacers. The uncured PDLC films were then cut and transferred to a custom-built conveyor system and cured by a high-intensity UV lamp with corresponding radiation intensity range of I_{UV} = 10 and 15 mW/cm^2 at the line speed of 0.5 meter/minute at cure temperature range of T_c = 30-50 °C. The PDLC formulation was carried out with NOA65 and TNO623 at 50/50 %w/w concentrations and the PDLC layer thicknesses were within <d> = 10 μm. The experiments were carried out on three PDLC

Samples for each formulation and process parameters and the reported experimental results were the average values of three samples.

2.3 Methods

The phase transitions of the MOMs and TNO623 were measured by Nikon Eclipse-50i polarizing optical microscope (OM) equipped with a temperature-controlled Mettler FP5 microscopic hot-stage at heating (10°C/min) and cooling (5°C/min) rates. The transition temperatures of PDLC films, includinig crystal-nematic (T_{CN}) nematic-crystal (T_{NC}), nemati-isotropic (T_{NI}) and isotropic-nematic (T_{IN}) were determined by a Perkin Elmer DSC7 Differential Scanning Calorimeter (DSC) at heating (10°C/min) and cooling (5°C/min) modes.

The electro-optical properties of PDLC/MOM films included the threshold (V_{10}) and saturation (V_{90}) voltages; off-state (T_{off}) and on-state (T_{on}) transmissions were measured on the as-made and naturaly aged samples. All measurements were carried out with a specially constructed photometric system consisting of a white light source, sample chamber, a photometer, an amplifier, a function generator and electronic data acquisition network. The transmission-voltage curves of the PDLC samples were measured with VAC square wave at 100 Hz frequency.

3 Results and Discussion

We conducted experiments on flexible PDLC samples doped with one mono-ligand L2-Pd and three bi-ligand 12-8NCu, LC1-Cu and A11-O-8NPd MOMs materials, where we studied the effects of MOM concentration and natural aging on switching voltages and optical transmissions. The results are mentioned as follows:

3.1. Effect of L2-Pd on PDLC Voltage and Transmission:

In this section, we present the effect of mono-ligand MOM material L2-Pd on the switching voltages (V_{10} and V_{90}) and transmissions (T_{off} and T_{on}) as a function of L2-Pd concentration of as-made and naturally aged PDLC films. The results are tabulated in Table-2 and presented in Figure-2.

According to Figure-2, the effect of L2-Pd concentration on switching voltages of as-made PDLC films clearly indicates that, both V_{10} and V_{90} values of PDLC are *decreased* up to L2-Pd=1% and *increased* up to L2-Pd=2%. The effect of natural aging on the switching voltages also exhibit similar trends within the studied L2-Pd concentration range.

With respect to general trends of voltages as a function of L2-Pd concentration metioned in Table-2 and Figure-2, both as-made and aged PDLC films, both V_{10} and V_{90} values are reduced at 0.5-1% but are increased at 0.1-0.2% concentrations of L2-Pd. With reference to as-made PDLC films, the presence of L2-Pd shows a moderate voltage reductions in L2-Pd doped PDLC are around 3-4 volts, but they are distinctively at 150-days aged PDLC films. Namely, not only that the PDLC without MOM shows voltage *increase* by 10-14 volts after aging, but at L2-Pd=1% the PDLC voltages are reduced by 13 and 18 volts for V_{10} and V_{90}, respectivrly. Such difference in switching volages is important in practice, because the commercial PDLC products are usually aged after production and product quality control.

With regards to Table-2 and Figure-2, although T_{off} values of as-made and aged PDLC do not significantly change by the presence of MOM, however its T_{on} values first exhibits an increasing trend at 0.5-1% and decreasing trend at 0.1-0.2% concentration range of L2-Pd. Once more, the effect of L2-Pd, on V_{10}, V_{90} and T_{on} of naturaly aged PDLC samples is indication on potential utilization of MOMs to improve the electro-optical properties of PDLC film products.

Table 2: Effect of L2-Pd concentration on voltages & transmissions of as-made and aged PDLC.

L2-Pd (%wt)	**Natural Ageing** (day)	V_{10} (volt)	V_{90} (volt)	T_{off} (%)	T_{on} (%)
0	0	11	18	0.5	89.5
	150	21	32	0.5	92.1
0.5	**0**	11	17	0.5	91.1
	150	**13**	**20**	**0.5**	**92.8**
1	**0**	**8**	**14**	**1.1**	**91.4**
	150	**13**	**22**	**1.1**	**93.2**
2	**0**	11	19	0.6	89.3
	150	**17**	**27**	**0.5**	**89.9**

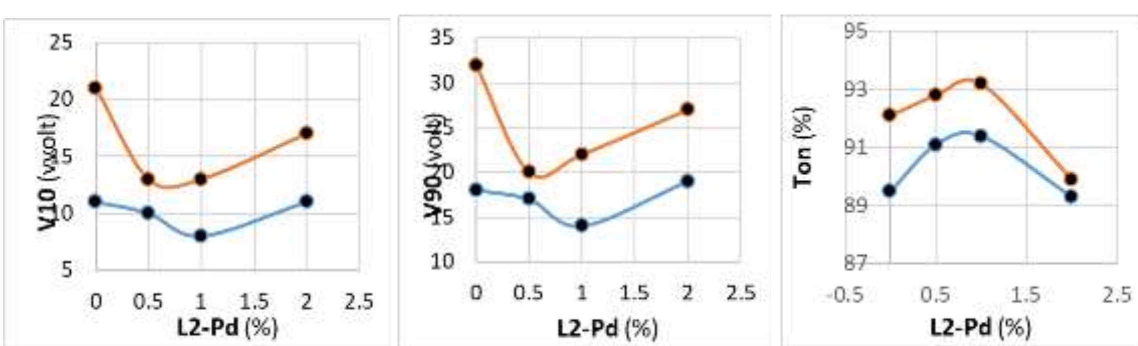

Fig.2: Effect of L2-Pd concentration on V_{10} (left), V_{90} (center) & T_{on} (right) of as-made PDLC (red) & aged PDLC (blue).

3.2. Effect of 12-8NCu on PDLC Voltage and Transmission:

In Table-3 and Figure-3, we present the effect of 12-8NCu concentration on switching voltages and optical transmissions of as-made and aged PDLC films. The results are in total agreement with those in L2-Pd PDLC system, indicating that both V_{10} and V_{90} exhibit a decreasing trends at lower MOM concentrations (1-2%) and moderate increasing trends at higher MOM concentration range (2-6.5%). We also observe similar trend of T_{on} with MOM concentration, meaning that it increases at 1-2% range and decreases at 2-6.5% range of 12-8NCu concentration.

With regards to further effect of aging in PDLC/12-8NCu films, in Table-4 and Figure-4 we provide the experimental study of as-made and naturally aged V_{90} in PDLC in comparison with PDLC/12-8NCu at 1%, 2% and 6.5% concentrations within 260 days natural aging. As shown more clearly in Figure-4, the effect of aging on V_{90} of PDLC films are significantly different in PDLC/12-8NCu films. More specifically, although all samples exhibit increasing trends of V_{90} by natural aging, the trend of PDLC without 12-8NCu shows a dramatic incease of V_{90}=22 volts in 260 days, while the increase in V_{90} values of PDLC/12-8NCu films are only within 6-8 volts. It should be noticed that, both PDLC films with L2-Pd and 12-8NCu exhibit the same trends of V_{10}, V_{90} and T_{on} values below 2% MOM concentration.

Table 3: Effect of 12-8NCu concentration on voltages and transmissions of PDLC before and after aging.

12-8NCu (wt%)	Natural Ageing (day)	V_{10} (volt)	V_{90} (volt)	T_{off} (%)	T_{on} (%)
0	0	16	24	0.3	82.6
1	**0**	**7**	**12**	1.9	**88.7**
2	**0**	**8**	**13**	0.7	**84.0**
6.5	**0**	**9**	**14**	1.3	**76.5**
0	**260**	**16**	**40**	**0.6**	**91.7**
1	**260**	**9**	**16**	3.5	**99.1**
2	**260**	**11**	**18**	3.3	**95.1**
6.5	**260**	**12**	**21**	3.9	**88..8**

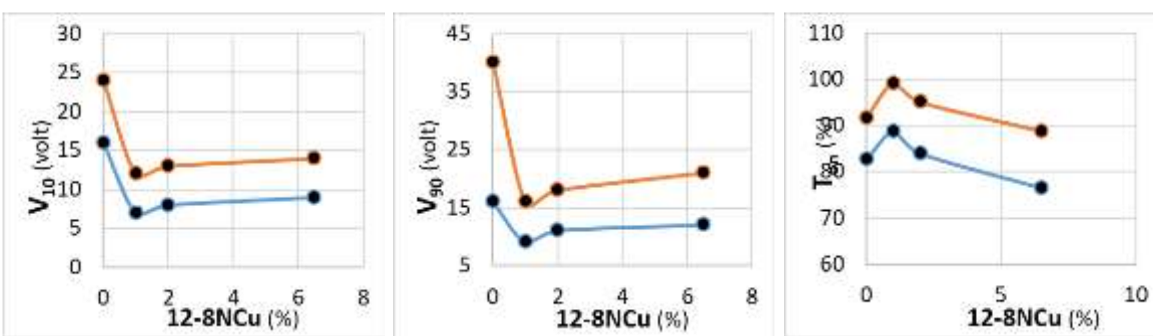

Fig.3: Effect of 12-8NCu on V_{10} (left), V_{90} (center) & T_{on} (right) of PDLC before (blue) & after (red) aging.

Table 4: Effect of natural ageing on V_{90} of PDLC films as a function of 12-8NCu concentration.

Natural Ageing	**12-8NCu** (%w)			
(day)	0	1	2	6,5
	V_{90} (volt)			
0	24	12	13	14
15	25	14	14	15
30	26	15	15	16
120	29	16	16	16
150	32	16	17	18
260	40	17	18	21

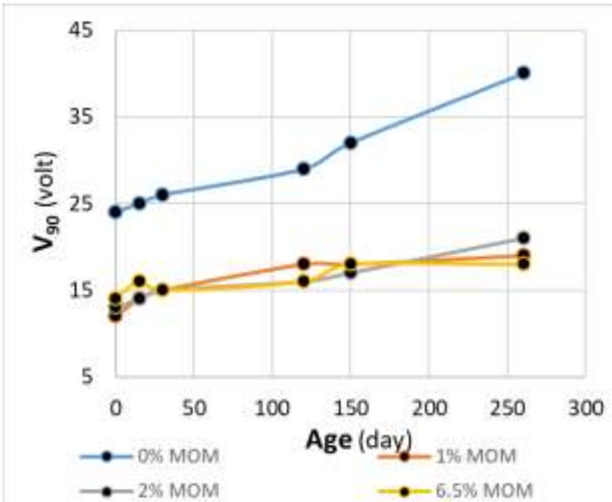

Fig. 4: Effect of natural aging on V_{90} of PDLC and PDLC/12-8NCu films.

In Table-5, we provide the effect of MOM CL1-Cu at 7.5 % on voltages and transmissions of as-made PDLC films at two UV curing intensities of I_{UV} = 10 and 15 mW/cm^2. The results clearly indicate that at I_{UV} = 10 mW/cm^2, although V_{10} does not change by the presence of CL1-Cu, but V_{10} increase by nearly 19 volts. With regardto transmission, T_{off} does not change but T_{on} decreases moderately, which seems to be due to index mis-matching of liquid crystal droplets with polymer matrix due to presence of Cli-Cu in droplets. At the UV intensity of I_{UV} = 15 mW/cm^2, again T_{off} does not change and T_{on} decreases moderately, however although V_{10} decreases by 7 volts, but V_{90} is substantialy reduced by 47 volts.

The overall results of this part of study indicates that, the presence of bi-ligand CL1-Cu MOM is important toimprove the electro-optical properties and particularly on switching voltages of PDLC films.

3.4. Effect of A11O-8NPd on PDLC Angular Transmission:

In this section, we present the effect of MOM material A11O-8NPd structure on the on-state transmission (T_{on}) of a PDLC film. Accordingly, in Table-6 and Figure-5, we tabulated the T_{on} values of PDLC at 0%, 2% and 6.5% concentrations of A11O-8NPd as a function of viewing angle.

The results clearly indicates that the presence of MOM increases the T_{on} values at higher viewing anlge. For example, the transmission at 60 degrees viewing angle in 2% A11O-8NPd increases by T_{on} = 10% and in 6.5% A11O-8NPd it increases by T_{on} = 20%. This effect is very interesting, because considering that PDLC usually suffers from an inherent on-state haze, such improvement in of-axis transmission is a promising application for reduction of on-state haze in PDLC films by MOMs.

Table 5: Effect of CL1-Cu on voltages & transmissions of PDLC at two UV curing Intensities.

I_{UV} (mW/cm^2)	**CL1-Cu** (%w/w)	V_{10} (volt)	V_{90} (volt)	T_{off} (%)	T_{on} (%)
10	0	7	33	5.6	83.5
	7.5	**7**	**14**	**5.6**	**78.2**
15	0	13	68	2.4	80.3
	7.5	**6**	**21**	**3.0**	**75.9**

Table 6: Effect of A11O-8NPd concentration on T_{on} values of PDLC films as a function of viewing angle.

Viewing Angle (degree)	**A11O-8NPd** (%w/w) **0**	**2**	**6.5**
	T_{on} (%)		
0	93.5	93.5	95.5
15	91.6	91.6	95.3
30	86.7	88.0	91.5
45	65.3	71.9	79.9
60	41.7	51.3	60.8

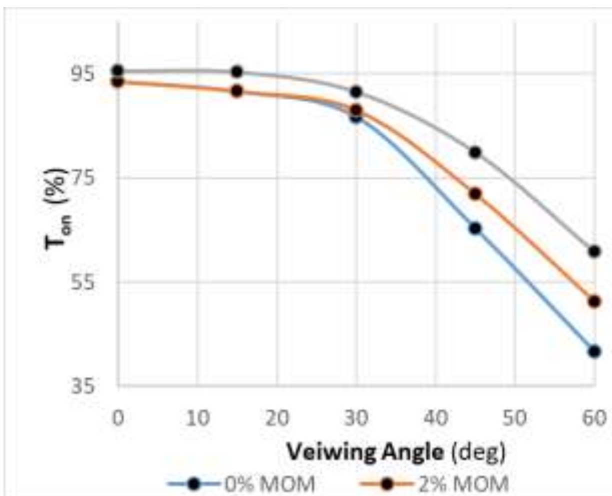

Fig.5: Effect of A11O-8NPd concentration on T_{on} values PDLC films as a function of viewing angle.

4 Conclusion

The effect of synthesized *mono-ligand* L2=Pd and *bi-ligand* 12-8NCu, CL1-Cu and A11O-8NPd MOMs on of V_{10}, V_{90}, T_{off} and T_{on} values in PDLC films as functions of MOM concentration and natural aging have been studied.

Regardless of the type of MOM, the outcome demonstrate that the presence of MOM significantly improves the electro-optical properties of PDLC films. Particularly at the lower concentration range of MOM, such improvements include, the reduction of switching voltages; increase of optical transmission at zero and wide viewing angle; enhancement of PDLC electro-optical properties after natural aging. The improvement of naturally aged PDLC properties doped with MOM is significant for commercial application of PDLC products. Consequently, the preliminary experimental results of this study suggest that, MOMs are potential materials for a number of applications in PDLC, guest-host liquid crystal materials and display devices.

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