

Electro-Optical Performances of Flexible Polymer Dispersed Liquid Crystals as A Function of Liquid Crystal Concentration



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Abstract

We investigated the effect of liquid crystal concentration on some electro-optical performances, such as optical transmissions, switching voltages and response times of flexible PDLC formulations of by UV polymerization induced phase separation (UV-PIPS) technique. Depending on the type of liquid crystal and formulation, thickness and process conditions, we found various electro-optical property dependences on liquid crystal concentration.

Keywords: Flexible PDLC; Liquid Crystal; Phase Separation; Transmission; Voltage; Response Time.

Abbreviations: PDLC: Polymer Dispersed Liquid Crystal; PIPS: Polymerization Induced Phase Separation; NOA65: Norland Optical Adhesives; CLC: Crystal Concentration

Introduction

The Polymer Dispersed Liquid Crystal (PDLC) technology has been the subject of many academic research and industrial production in the past five decades. The chemical and physical nature of raw materials, thickness, type of phase separation and curing process parameters are important factors influencing the morphology and electro-optical properties of PDLC films. The majority of published literature in the field are academic studies, dealing with the effect of materials, process conditions and the nature of phase separation methods, including the formulation, composition, thickness, curing radiation intensity and temperature, pre-curing and curing time on morphology and electro-optical properties on glass-based PDLC devices. On the other hand, most industrial developments work, which have been rarely published in the open literature, are based on development and production manufacturing of PDLC products by Polymerization Induced Phase Separation (PIPS) method on flexible supports. At the industrial development scale and manufacturing, the correlation between materials and process conditions with morphology and electro-optical properties of PDLC is essential. In this respect, the chemistry and physical parameters of raw materials, phase separation and curing kinetics

are important factors on the performance of flexible PDLC products. According to recent studies on thermally and UV cured PDLC films, it has been shown that their morphology and electro-optical properties have been strongly influenced by material compositions, formulation type, thickness, cure temperature, UV radiation and thermal intensities, cure temperature and time [1-10] These factors have been demonstrated to affect the micro-structure, size, shape and number density of liquid crystal domains, degree of phase separated and plasticized liquid crystal, as well as their anchoring within droplets. The morphology that affected the PDLC performances itself depends on a number of factors, including the nature of liquid crystal and pre-polymers, as well as the mechanism of phase separation and curing kinetics. These factors have been influenced the overall micro-structure of polymer matrix, the size and the shapes of liquid crystal domains and their anchoring within micro-droplets. [11-15] In this work, we studied the effect of liquid crystal concentration (C_{LC}) on the electro-optical properties of five flexible UV-cured PDLC formulations by Polymer Induced Phase Separation (PIPS) method. This study has been part of industrial R&D program to establish direct empirical relations between curing parameters and electro-optics of PDLC products for subsequent scale-up and

manufacturing. Such relations are essential for direct quantitative correlations between process parameters and electro-optical performances during manufacturing of PDLC products.

Materials and Methods

Materials

The utilized materials in this study were UV-curable NOA65 pre-polymer resin (Norland Optical Adhesives) and five commercial

nematic liquid crystal mixtures, including TN0623 and TN0403 (Hoffmann-La Roche), E43 and E7 (Merck Ind. Chem.) and YM55 (Valiant Fine Chem.); Irgacure819 photo-initiator (Sigma-Aldrich); 100 Ω / ITO-PET film supports (Sheldahl) and 10-20 Ω m thick plastic Micro-Pearl micro-spacers (Sekisui). All utilized materials were used without further purification. The optical and thermal properties of commercial liquid crystal mixtures are tabulated in (Table1).

Table 1: Optical and thermal parameters of liquid crystals.

Liquid Crystal	n_o	Δn	T_m (°C)
TN0623	1.507	0.198	105
TN0403	1.524	0.258	82
E7	1.521	0.225	61
E43	1.525	0.238	83
YM55	1.526	0.249	92

Sample Preparation

As presented in (Figure 1), the flexible PDLC samples were prepared with Polymerization Induced Phase Separation method by UV radiation (UV-PIPS) through a custom-made vertical in-situ film coating / lamination system of homogeneous mixtures of liquid crystals and NOA65 pre-polymer at corresponding liquid crystal concentration ranges of 30-80 weight %, 1 % of Irgacure819 vs total formulation and pre-heated temperature of

50°C for 10 minutes. The thickness homogeneity of PDLC layers were achieved by utilizing 0.2% plastic micro-spacers with respect to total formulation. Also, according to (Figure 1), the uncured PDLC formulations including micro-spacers were poured between the vertical gap of two ITO-PET film support rolls of plastic coater/laminator system and the uncured PDLC sandwich under the coating rolls were passed through a pressure roll to insure the uniformity of the film.



Figure 1: PDLC coater/laminator (left), UV curing conveyor (middle) and electro-optical system (right).

The uncured PDLC film sandwiches were then cut and transferred to a custom-built conveyor machine (Figure 1) and cured by a high-intensity UV lamp with corresponding radiation intensity ranges of 1-60 mW/cm² at the line speed of 0.5 meter/minutes and relevant cure temperatures of 22-80°C. The specific processing and curing conditions of the studied PDLC

formulations are tabulated in (Table 2). The experiments for each formulation were carried out on three PDLC samples and the cure temperatures were measured with a thermocouple during the curing process. The reported results were the average values of the three samples.

Table 2: Processing and parameters of the PDLC formulation.

PDLC	Thickness (μm)	UV Intensity (mW/cm^2)	Cure Temperature (C)
TNO623	10	60	80
TNO403	10, 15	20	55
E43	10	8	30
E7	20	1	25
YM55	20	1	22

Methods and Measurements

As presented in (Figure 1), the electro-optical 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 electro-optical properties of PDLC films were measured on the as-made PDLC samples. The transmission-voltage curves of the PDLC samples were measured with VAC square wave at 100 Hz frequency. The switching times were carried out at peak-to-peak voltage of $V_{\text{RMS}} = 80$ volts at the frequency of 100 Hz and pulse life time of 1/8 second. The nematic-isotropic transition temperatures of liquid crystals were measured under Nikon Eclipse-50i polarizing optical microscope equipped with Linkam LTS120 microscopic hot-stage. The normal haze in some PDLC films were measured by Gardner model XL835 colorimeter by a white light source.

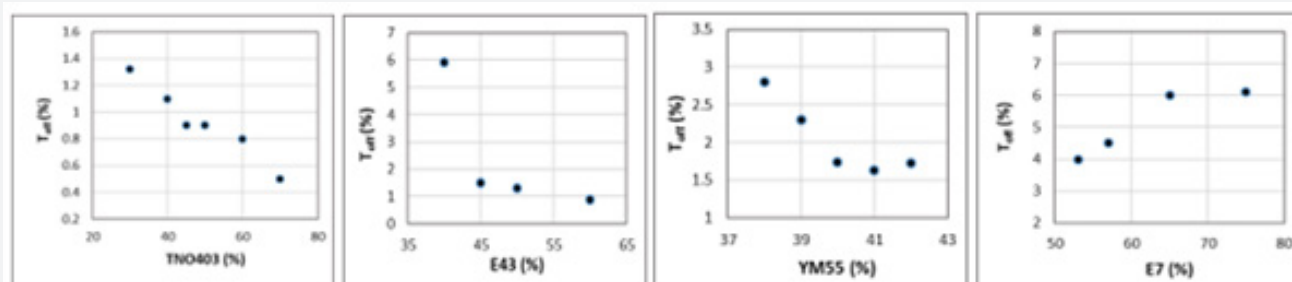
Results

We studied the electro-optical properties of PDLC formulations utilizing the five commercial liquid crystals TNO623, TNO403, YM55, E43 and E7 mixtures as a function of liquid crystal

concentration (C_{LC}). The measured electro-optical parameters include: the optical transmissions off- state (T_{off}), on-state (T_{on}); the threshold (V_{10}) and saturation (V_{90}) voltages; as well as the rise (τ_{on}) and decay (τ_{off}) response times. The results of these experimental studies are provided in the following sections:

Optical Transmissions

The effect of liquid crystal concentration (C_{LC}) on the off-state transmission (T_{off}) or opacity of four PDLC-TNO403, PDLC-E43, PDLC-YM55 and PDLC-E7 formulations are presented in (Figure 2). According to (Figure 2), whereas the T_{off} (opacity) of PDLC-TNO623, PDLC-E43 and PDLC-YM55 samples exhibit linear and exponential decreasing trends of T_{off} (opacity) with C_{LC} . The decreasing trend of T_{off} with C_{LC} is in agreement with that reported on PDLC-E7 film cured by electron beam [16] On the other hand, this result is different than in present PDLC-E7 (Figure 2), which shows an unexpected increasing trend of T_{off} with C_{LC} . In (Figure 3), we present the effect of C_{LC} on-state transmission (T_{on}) or transparency in three PDLC- E43, PDLC- YM55 and PDLC-E7 formulations. The results indicate that T_{on} in E43-PDLC and YM55-PDLC show apparent linear increasing trends, whereas E7-PDLC exhibits a nonlinear decreasing trend with C_{LC} , which is similar to its T_{off} trends (Figure 2).

**Figure 2:** Effect of liquid crystal concentration on off-state transmission (T_{off}) of four PDLC formulations.

Switching Voltages

In (Figure 4), we present the effect of liquid crystal concentration on the threshold (V_{10}) and saturation (V_{90}) voltages in the five flexible PDLC-TNO623, PDLC-TNO403, PDLC-E43, PDLC-

E7 and PDLC-YM55 formulations. As tabulated in (Figure 4), the switching voltages of all five PDLC film formulations, indicate that both “threshold voltage” (V_{10}) and “saturation voltage” (V_{90}) exhibit decreasing trends with liquid crystal concentration. It

should be noted that, the decreasing trend of V_{10} and V_{90} with C_{LC} in the five PDLC formulations is also in agreement with the other literature studies including the study on PDLC-E7 film cured by electron beam [16].

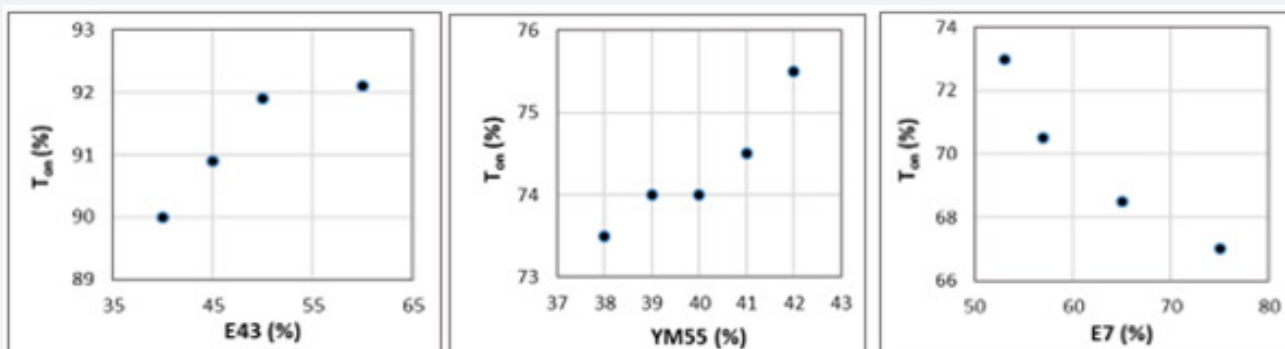


Figure 3: Effect of liquid crystal concentration on on-state transmission (T_{on}) of three PDLC formulations.

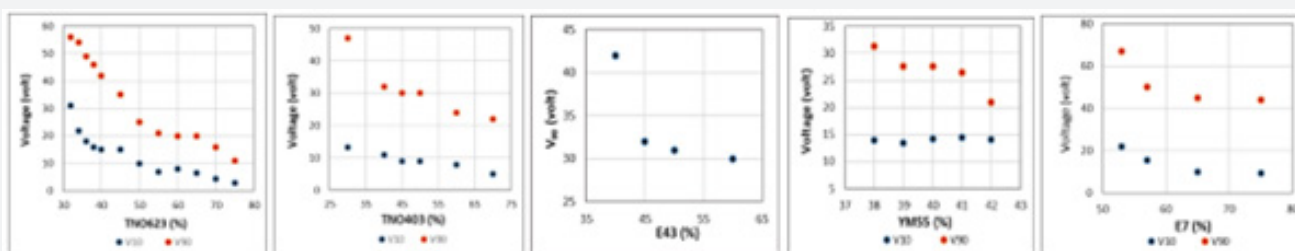


Figure 4: Effect of liquid crystal concentration on switching voltages of five PDLC formulations.

Response Times: The effect of liquid crystal concentration on the rise (τ_{on}) and decay (τ_{off}) response times in two PDLC-E43 and PDLC-YM55 formulations are presented (Figure 5). The overall outcome of the present results is that whereas the τ_{on} decreases and τ_{off} increases in PDLC-E43, but in PDLC- YM55 τ_{on} increases and τ_{off} decreases as a function of liquid crystal concentration (C_{LC}). Also, according to (Figure 5), the τ_{off} of PDLC-YM55 film is more than one order of magnitude larger than that in PDLC-E43 film.

Discussions

Optical Transmissions

According to (Figure 2), with respect to the effect of liquid crystal concentration on T_{off} (opacity) in the four formulations, considering that materials, thicknesses and process conditions are quite different (Table 2), except the PDLC-E7 which exhibits an increasing T_{off} trend, the decreasing trends of T_{off} with C_{LC} in PDLC-TN0403, PDLC-E43 and PDLC-YM55 formulations could be correlated according to the following equation,

$$T = T^o \cdot \exp^{-(\eta \cdot \delta \cdot d)} \quad (1)$$

where T is the off-state transmitted light intensity, T^o is the incident light intensity, η is the droplet number density, δ is the “scattering cross-section” of a single droplet and d is the sample thickness. According to Equation-1, only the increase of scattering cross-section by C_{LC} increase could explain the observed decreasing trends in these three PDLC samples. On the other hand, due to the lack of morphological information, in E7-PDLC the increasing trend of T_{off} or decreasing opacity with C_{LC} could not be properly explained in the present study. Also, according to (Figure 3), with respect to decreasing trends of T_{on} with C_{LC} in three PDLC-E43, PDLC-YM55 and PDLC-E7 formulations, in addition to other material and process differences, one may explain the unexpected increasing trend of T_{on} with C_{LC} in PDLC-E7 in the absence of micro-droplet scattering to be due to additional index miss-matching in this PDLC formulation. This behaviour may explain the reason for linear increasing of T_{on} with C_{LC} in PDLC-E7 system, which could be the initial step to achieve an empirical relationship to explain the bulk and surface contributions to morphology (micro-droplet size, shape and number density) and electro-optics of PDLC films.

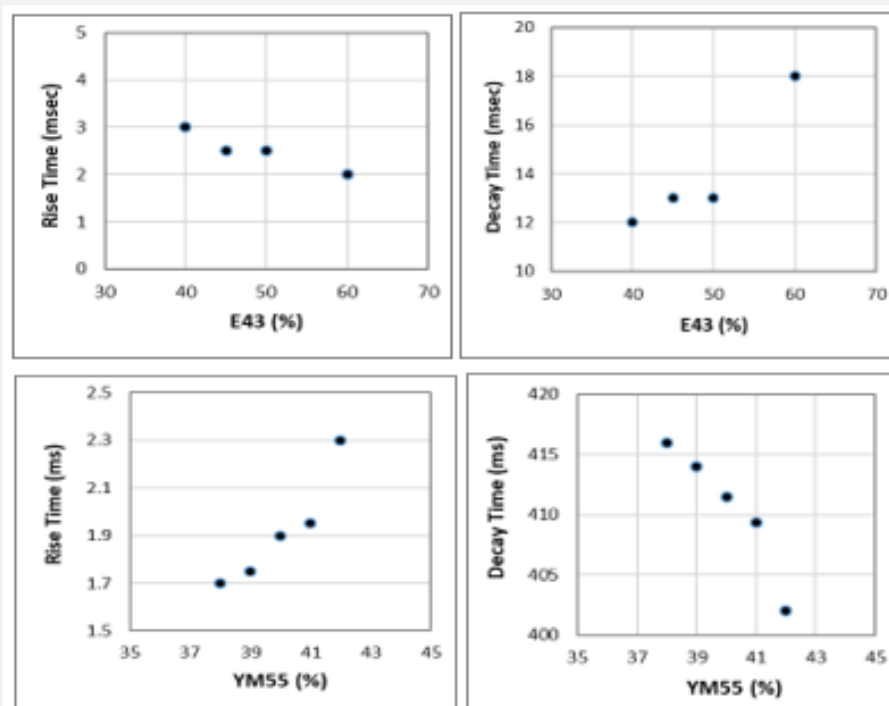


Figure-5: Effect of liquid crystal concentration on rise (T_{on}) and decay (T_{off}) times of E43-PDLC (top) and YM55-PDLC (bottom).

These unexpected behaviours of T_{on} and T_{off} in PDLC-E7 sample could be attributed to different curing condition and thickness with respect to those in E43-PDLC and YM55-PDLC (Table 2). In contrast to this study, other UV-cured PDLC and thermoset (epoxy) PDLC films claims that, only in agreement with T_{off} of PDLC-E7, the T_{off} increases modestly with C_{LC} within 35-50% concentration range, whereas the T_{on} is almost independent of C_{LC} [17,18]. In contrast to other formulations, the different trends of T_{off} and T_{on} in PDLC-E7 could be due to preferential higher solubility of E7 in the polymer matrix, resulting to a significant amount of uncured pre-polymer in micro-droplets and highly plasticized E7 in the matrix. These factors cause a significant refractive index mis-matching and, consequently, increases T_{off} and decreases T_{on} of PDLC-E7 films with liquid crystal concentration [19]. Although rather speculative, the different trends of T_{off} and T_{on} of PDLC-E7 with other formulations could be also explained as a result of the influence of surface phenomenon from substrate and matrix on phase separation and polymer curing kinetics at higher C_{LC} of this sample in comparison to those of PDLC-E43 and PDLC-YM55 films. Another words, at higher C_{LC} the contribution of surface effect diminishes in favour of the bulk, affecting the phase separation and polymer curing kinetics and morphology of PDLC film as a result of changes in temperature distribution and shear stress at the surface-bulk boundary. Because, if the morphology is influenced due to surface phenomenon, then both T_{off} and T_{on}

values should be also affected.

In addition to morphology, the geometric boundary conditions that is different at the surface and the bulk. could be another parameter influencing the phase separation and matrix curing kinetics. Also, with regards to the haze, we only explored its behaviour in PDLC-TNO403 formulation at 10 μm and 15 μm thicknesses. The results indicated that haze increased with C_{LC} at both thicknesses, which is a known phenomenon in PDLC literature where we have also reported that the haze of PDLC-E7 as a function liquid crystal concentration [20]

Switching Voltages

With respect to the effect of liquid crystal concentration on threshold (V_{10}) and saturation (V_{90}) voltages of the studied PDLC formulations, it is known that liquid crystal composition is strongly coupled parameter with PDLC switching voltages, where both V_{10} and V_{90} decrease by increase of liquid crystal composition (CLC). These decreasing trends of switching voltages with C_{LC} (Figure 4) in various UV-cured PDLC systems are also in agreement with previous literature [8,21&22] which directly correlate with the increasing of micro-droplet dimensions by increasing of C_{LC} . For example, an approximate relation between threshold voltage (V_{10}) and micro-droplet dimension has been expressed by the following relation [18]

$$V_{10} = d / \langle R \rangle \cdot [(\langle K \rangle - 1)(\epsilon_{11} \Delta \epsilon)]^{1/2} \quad (2)$$

where it is film thickness, $\langle R \rangle$ is average droplet radius, K is average elastic constant, ϵ_{11} is parallel electric susceptibility and $\Delta \epsilon$ is dielectric anisotropy of LC, respectively. According to Equation-2, V_{10} should decrease with increase of PDLC droplet radius, which is in agreement with increasing of droplet dimension with liquid crystal concentration [8]. Regardless of other different parameters that contribute to different values of V_{10} in the four PDLC formulations (Figure 4), Equation-2 clearly indicate the effect of LC droplet size ($\langle R \rangle$) increase on decreasing trend of V_{10} values. According to (Figures 4), it is also noticed that in the studied PDLC systems the order of magnitude of switching voltages V_{10} and V_{90} are correlated with the liquid crystal droplet morphologies, where it is lowest is in YM55-PDLC and highest in PDLC-TNO623 and PDLC-E7 formulations. For example, at $C_{LC} = 40\%$, the decreasing trend of V_{10} values are around 25volts in PDLC-E7, 15volts in PDLC-TNO623, 14volts in PDLC-YM55 and 11volts in PDLC-TNO403 samples. Similarly, the V_{90} values are >70volts in PDLC-E7, 42volts in PDLC-TNO623, 37volts in PDLC-E43, 32volts in PDLC-TNO403 and 27volts in PDLC-YM55, respectively. These results indicate that the decreasing trend of switching voltages in these five PDLC formulations, as well as in other reported UV-cured and thermoset PDLC studies, [18,23] is mainly due to increasing of droplet dimensions and decrease in number densities as a function of liquid crystal concentration.

In these PDLC formulations, the quantitative differences in C_{LC} dependencies of operating voltages are mainly due to differences in their material and process parameters (Table 2), which result to qualitative variations in the mechanisms of phase separation and polymer curing kinetics. It is known that the phase separation, morphology and electro-optical properties of PDLC films are also the outcome of other inter-dependent factors, such as the type, chemical, physical and optical properties of materials; solubility temperature of liquid crystal and pre-polymer; degree of cross-linking and matrix plasticization by liquid crystal and curing parameters. In short, due to different chemical, optical and thermal properties of commercial liquid crystal mixtures (Table 1) and their different PDLC process parameters (Table 2), a direct comparison between the switching voltages of the studied PDLC formulations is difficult for the time being.

Response Times

According to (Figure 5), the differences in response times τ_{on} and τ_{off} in PDLC-E43 and PDLC-YM55 formulations could be due to their different morphologies, thicknesses and UV intensities as tabulated in (Table 2). Namely, while PDLC-E43 thickness is 10mm and UV intensity is 8 mW/cm², these parameters in PDLC-YM55 are 20mm and 1 mW/cm², respectively. Also, according (Table 1), the difference of around 30°C of nematic-isotropic transition temperatures (T_{ni}) of the two formulations would be another

factor resulting to different morphologies and consequently their different trends of response times with C_{LC} . We also noticed that, in both PDLC systems the response times exhibit predominantly linear trends within the studied liquid crystal concentration ranges. In general, as the molecular orientation, instead of voltage, depends on the electric field, then the trends of τ_{on} and τ_{off} with liquid crystal concentration would not be necessarily linear. However, this is not a satisfactory explanation, because other important above-mentioned parameters are not taken into account. Furthermore, the different effect of C_{LC} on τ_{on} and τ_{off} of PDLC-E43 and PDLC-YM55 systems (Figure 5) should be verified by taking account of other different parameters, such as chemical and physical properties, anchoring energy, as well as surface and bulk effects. Also, according to (Figure 5), the trends of response times in PDLC-E43 are qualitatively similar to and in agreement with the reported literature in other UV-cured and thermoset PDLC, where τ_{on} is almost independent and τ_{off} increases with C_{LC} [24,25]

Conclusion

In the present work, we investigated the effect of liquid crystal concentration (C_{LC}) on the electro-optical performances (T_{off} & T_{on}) of few flexible PDLC film formulations, which has been part of industrial R&D program for eventual manufacturing of high-quality PDLC products. These experimental studies were carried out by phase separation UV-PIPS technique. The results indicated that, regardless of material, formulation and process parameters, the studied optical transmissions (T_{off} & T_{on}), switching voltages (V_{10} & V_{90}) and response times (τ_{on} & τ_{off}) mainly exhibited significant and expected dependencies on the liquid crystal concentration. However, in few unexpected cases the differences were explained to be the result of different in materials, formulations, process conditions, thickness and morphology of the studied PDLC films. Aside from quantitative effect of C_{LC} on the electro-optics of studied PDLC formulations, the results were also due to subtle effects in the mechanism of phase separation and polymer curing kinetics. In addition, the phase separation of liquid crystal from matrix and curing efficiency were also governed by other inter-dependent factors, such as solubility, cure temperature, UV cure intensity and matrix plasticization by liquid crystal, which all require further systematic investigations. It is obvious that further detail investigations would be necessary to provide a deeper understanding of the electro-optical performances of flexible PDLC films. Once the structure-property relations between material and process parameters are quantified, it will be possible to develop meaningful empirical models to control and improve the electro-optical properties for reliable and quality-controlled industrial manufacturing of high-quality PDLC products.

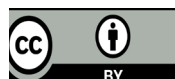
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