Thermal Paper and Time Temperature Integrators Made From a Structural Colored Polymer Crosslinked With Hydrogen Bonded Cyclohexanoic Acid Derivatives

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Hydrogen-bonded cyclohexanoic derivatives are applied in a structural colored cholesteric liquid crystal (CLC) helical polymer that demonstrates an extraordinary dual thermal response. Integration of the dynamic hydrogen-bonded cyclohexanoic acid derivatives in the CLC polymer as supramolecular crosslinks yields an immediate and irreversible loss of the green structural color upon exposure to temperatures above the cholesteric-to-isotropic transition temperature ($T_{NI}$) caused by the helical order loss. Exposure to temperatures below the $T_{NI}$ yields a gradual irreversible red color shift of the reflected wavelength over broad temperature and time ranges. Most likely due to polymerization confinement in combination with the dynamics of the carboxylic acid hydrogen bonds, helical unwinding takes place over time causing the red shift of the reflected wavelength. The temperature response can be programmed by altering the chemical composition of the liquid crystal mixture. Furthermore, the green, structural color of the polymer can also be easily changed making it possible to print different colored indicators combined with different respective temperature responses on a single substrate. The printed optical sensors can be easily delaminated by dissolving the non-covalent crosslinked polymer. Such optical polymer materials are attractive for easy processable time–temperature integrators and thermal paper.

1. Introduction

Irreversible thermal responsive optical materials know multiple practical applications in everyday life, including thermal paper and time temperature integrators (TTIs). Thermal paper is used in thermal printers where color changes from white to black, forming images or letters when exposed to heat. The current standard in thermal paper comprises a multilayer laminate with Bisphenol A as a color developer to interact with a thermochromic dye upon localized thermal exposure.[1] Bisphenol A has proven to cause adverse health effects upon dermal absorption through handling of such thermal paper.[2,3] In addition, multilayer laminates are difficult, if not impossible, to recycle. Alternatives, such as photonic paper, employ evaporation of a small molecule to induce a color contrast.[4]

Optical TTIs are an import class of indicators that track temperature exposure history to provide insight for quality and safety management in healthcare and nutrition industries, amongst others.[5–7] For the preparation of TTIs, extra procedures are often needed to activate the sensor and the operating temperature window is narrow as it is related to a phase transition temperature. So far, dual temperature responsive optical materials that both act as a TTI and thermal paper have never been reported.

Development of structural color-based polymer systems that function as temperature-responsive optical indicators offers a range of advantages including robustness, low-cost production, and easy readout.[8–10] Furthermore, such photonic polymer systems allow for a significant freedom of molecular programmability in color transition, response range, and sensitivity.[11,12] Cholesteric liquid crystal (CLC) polymers are a specific class of structural colored materials having a helical molecular organization. The molecular anisotropy in the refractive index parallel and perpendicular to the long axis of the liquid crystal molecule creates a periodic refractive index alternation through the depth of the helix, causing the reflection of a selective wavelength corresponding to the helical pitch.[13] Reactive CLCs can be photopolymerized to fix the structural colored state over a broad temperature window while preserving structural...
flexibility allowing the structural color to be altered upon changing the helical organization in the polymer by a stimulus.[15,36]

An irreversible color change can be induced by using shape memory CLC polymers.[17–19] First, a temporary structural colored state was programmed manually, usually by indentation. The restoration of the original structural colored state is temperature-dependent.[20–22] The optical response of these shape memory color shifting photonic polymers occurs in the limited temperature region around the glass transition temperature (T_g) of the polymer.

Recently, we eliminated the programming step by using CLC polymers that incorporate benzoic acid hydrogen-bonded dimers as crosslinks.[23–25] When this supramolecular network was heated above the CLC isotropic phase transition temperature (T_{NI}) at 105 °C, a gradual disappearance of the structural color was observed as result of the loss of helical order. The incorporation of hydrogen bonds in responsive supramolecular liquid crystal polymers is most often accomplished by using reactive benzoic acid derivatives. This hydrogen bonding motif typically features high temperatures in which the hydrogen bonds demonstrate dynamic and responsive behavior, limiting the tunability of the liquid crystal polymers and the scope of possible applications as sensors, actuators, and membranes.[26] Therefore, there is a need for other types of hydrogen bonding molecules with responsive behavior at lower temperatures.

This work presents a supramolecular crosslinked CLC polymer based on hydrogen bonded cyclohexanoic acid derivatives that allows for unprecedented optical responses at low temperatures and a tunability of the T_{NI} between 30 °C and 90 °C. These polymers demonstrate an immediate loss of structural color after exposure to temperatures above T_{NI} which can be exploited as photonic thermal paper. In contrast, exposure to a broad temperature range below the T_{NI} results in an irreversible time-temperature dependent, red-shifted color transition, in this case, from green to red. The transition occurs through helical unwinding in the polymer, caused by the photopolymerization step in combination with the dynamics of the hydrogen-bonded interactions between the cyclohexanoic acids. Hence, a time temperature responsive gradual color shift by a structural colored polymer material is introduced which does not require a phase transition or an activation procedure.

![Figure 1](image1.png)

**Figure 1.** a) Chemicals composition of the CLC inks and b) CLC nematic to isotropic transition temperatures determined by the fraction of M1a (squares) or M1b (circles) and M2 in different polymer compositions; the green square corresponds to the T_{NI} of polymer coating 1, the green circle to the T_{NI} of coating 2.

### 2. Results and Discussion

#### 2.1. Ink Formulation Based on New Reactive Mesogens

The printable ink to obtain CLC polymers with a lower T_{NI} required novel molecules M1a (4-hexyloxy-phenyl 4-(6-acryloyloxyhexyloxy)-2-methylenbenzoate) and M1b (4-methoxy-phenyl 4-(6-acryloyloxyhexyloxy)-2-methylenbenzoate) (Figure 1a). These molecules were prepared as described in the Supporting Information. The cyclohexanoic-acid derived monoacrylate (trans)-4-((6-acryloyloxyhexyloxy)cyclohexane-1-carboxylic acid (M2) provides hydrogen bond crosslinking with a reduced hydrogen bond strength due to the lack of resonance stabilization and excludes the π-π stacking interactions of traditional benzoic acid groups. Synthesis of M2 used (trans)-4-((6-hydroxyhexyloxy)cyclohexane-1-carboxylic acid as the starting compound[27]) that was converted to the acrylate derivative. The complete synthesis procedure and the full characterization of all synthesized monomers are described in the Supporting Information. Incorporation of M1a or M1b combined with M2 in a CLC polymer resulted in a significant reduction of the T_{NI}, with M1b resulting in an even lower T_{NI} than M1a at an identical concentration due to the shorter alkyl tail (Figure 1b).

Moreover, the ink contained a monoacrylate-based chiral dopant (M3) with a high helical twisting power.[23] 4-((6-acryloyloxyhexyloxy)phenyl 4-methylenbenzoate (M4) was added to obtain a broad cholesteric liquid crystal phase. The resulting CLC mixture contained solely monoacrylate components to exclude covalent crosslinks. The photoinitiator (M5) enabled photopolymerization of the acrylate groups to produce a CLC polymer system with supramolecular crosslinks in the form of hydrogen-bonded dimers.[26,28] For printing of the ink, all components were dissolved in cyclopetanone as a solvent, with 0.1 wt% of a commercial surfactant to support planar alignment at the CLC–air interface.

For preparing the structural color polymer film 1, a monomer mixture (12.5 wt% M1a, 21 wt% M2, 3.2 wt% M3, 62.5 wt% M4, and 0.8 wt% M5) was inkjet printed on flexible black biaxially-oriented polyethylene terephthalate (BOPET) foil which induces planar alignment. Before photopolymerization, the solvent was evaporated, and the printed coating was cooled down to the CLC phase in a nitrogen atmosphere box. In the cholesteric phase, the CLC monomer mixture displayed...
a reversible blue shift upon increasing temperature from 0 °C to 15 °C (monomer T_{NI}) (Figure S1, Supporting Information). After polymerization, a green reflective polymer coating with a thickness of ≈3 μm was obtained. The Fourier transform infrared spectroscopy (FT-IR) spectrum of polymer coating 1 showed the absence of the acrylate C=O peaks (1640, 1410, and 985 cm⁻¹), indicating successful photopolymerization (Figure S2, Supporting Information). Furthermore, an equilibrium existed between free carboxylic acids, indicated by the carbonyl vibration peak at ≈1730 cm⁻¹, and hydrogen-bonded carboxylic acid dimers acting as supramolecular crosslinks, indicated by the carbonyl vibration peak at ≈1700 cm⁻¹ (vide infra).²⁹,³⁰ Differential scanning calorimetry (DSC) (Figure S2, Supporting Information) shows a CLC to isotropic transition at T_{NI} = 68 °C (Figure 1b). The amount of M1a or M1b and M2 determines the T_{NI} of both monomer mixtures and polymer coatings (Figure 1b). The composition of the liquid crystal polymer allows tuning of the T_{NI} between 30 °C and 90 °C. A difference of ≈55 °C is noted between the T_{NI} of the polymer coatings and the T_{NI} of the monomer mixtures (Figure S3, Supporting Information).

### 2.2. Thermal Response

When the green-colored printed polymer coating 1 is heated above the T_{NI} (T = 70 °C) for a few seconds, the coating turns transparent (Figure 2). Upon cooling, the color does not return. Instead, a lightly scattering, translucent coating remains which appears black due to the BOPET substrate (Figure 2). The color loss is most likely due to the loss of cholesteric helical order when the non-covalently crosslinked coating is heated above the T_{NI} (vide infra).²² After the loss of the helical order above T_{NI}, the polymer is unable to re-organize again when cooled to the CLC phase. Experiments demonstrate that this green-colored coating can be used as photonic thermal paper in which features can be written with a hot pen or laser (Figure 2c). By modifying the amount of chiral dopant and chemical composition, the initial color and T_{NI} of the CLC polymer coatings can be altered for desired specifications. This allows to print different colored indicators combined with different response temperatures on a single substrate (Figure S4, Supporting Information).

Isothermal exposure below the T_{NI} (68 °C) of the green structural colored polymer coating 1 results in an irreversible redshift of the color over time. Over a broad temperature and time range below T_{NI}, the polymer coating demonstrates a time-temperature dependent visible color shift from green to red (Figure 3a). Analysis of the shift of the peak maximum over time is shown, where a time-temperature response of the selective reflection is expressed upon isothermal exposure to 60 °C, 40 °C, and 20 °C (for reflection spectra, see Figure S5, Supporting Information). During exposure to 60 °C, the coating undergoes a gradual shift of 80 nm from green (=540 nm) to red (=620 nm) reflection within 10 h (time lapse movie in Supporting Information). The reflection shift occurs mostly in the first 2 h of exposure. For exposure to 40 °C, the shift is significant on an hourly timescale and after almost two days (46 h), a shift of 70 nm is observed. At 20 °C, the coating seems stable on an hourly timescale but over the course of 20 days (480 h), a noticeable shift of 20 nm transpires. The time dependent color shift seems to be inversely proportional to the exposure temperature with the T_{NI} as an upper threshold.

To investigate if the time-dependent color shift is correlated to the T_{NI}, a new green colored polymer coating 2 was produced with a lower T_{NI} (48 °C) with M1b instead of M1a and with the same molar amount of cyclohexanoic acid derivative M2 to provide an equal degree of supramolecular crosslinking (Figure 1b). Coating 2 shows faster responses upon isothermal exposure to 40 °C and 20 °C, respectively (Figure 3b; Figure S6, Supporting Information). The reflection shift of 50 nm for exposure to 40 °C occurs mostly in the first 2 h. At 20 °C, the coating demonstrates a significant shift on an hourly time scale and a total shift of 40 nm over 8 days (192 h). These results indicate a correlation between the T_{NI} and the color response rate. Furthermore, the T_{g} of the polymer coating establishes the lower threshold for which a shift is possible. Polymer coating 2 (T_{NI} 48 °C), made with M1b, has a T_{g} around 8 °C and does not show any shift of selective reflection when stored in a refrigerator (6 °C) for over a month.

This peculiar temperature-dependent color shift effect was not observed in our previous research when a non-covalent

![Figure 2](image-url)  
**Figure 2.** a) Pictures before, during, and after thermal exposure above T_{NI} (68 °C) of green structural colored polymer coating 1. b) UV–vis reflection spectra before and after heating the green coating on a black BOPET substrate above its T_{NI}, demonstrating the complete loss of specific reflection. c) Photonic thermal paper: polymer coating 1 after 5 s exposure to a heated metal stamp pattern “1” at 80 °C.
crosslinked system with a benzoic acid as a hydrogen bonding moiety was exposed to a temperature (100 °C) just below \(T_{NI}(105 \,^{°}\text{C})\). In addition, a non-crosslinked polymer coating without cyclohexanoic acid moieties and with a \(T_{NI} = 68 \,^{°}\text{C}\) likewise does not display a color shift during isothermal exposure to 60 °C (Figure S7, Supporting Information). In addition, the color does not change before and after polymerization, suggesting that there is no influence of the polymerization on the helical twisting power of the chiral dopant. Therefore, the color shift must be ascribed to the hydrogen-bonded cyclohexanoic acid crosslinks in the CLC polymer.

To shed more light on the role of hydrogen-bonded cyclohexanoic acid crosslinks in the CLC polymer on the color shift, IR spectroscopy measurements were carried out. At 60 °C, the C=O stretch vibration at 1700 cm \(^{-1}\) shifts and broadens in time (Figure 4a). The full FT-IR spectrum (Figure S2, Supporting Information) demonstrates that an irreversible change happens throughout the entire polymer matrix as the peaks of the alkyl tails (720 cm \(^{-1}\)) and the cyclohexane ring (1100 cm \(^{-1}\)) vibrations become more pronounced. Interestingly, the rate of change for the reflection peak and FT-IR peak at 1700 cm \(^{-1}\) show similar time-dependent exponential behaviors (Figure 4b), indicating that the color change is related to the change in hydrogen bond interactions. Hydrogen-bonded cyclohexanoic acid dimers and free acids are present in the polymers immediately after polymerization (vide supra). However, due to polymerization confinement, a new thermodynamically favorable, more unwound, helical state is most likely formed. This new state results in a broad vibration band between 1730 and 1690 cm \(^{-1}\), which approaches the processes that determine food deterioration such as enzymic reactions (40–130 kJ mol \(^{-1}\)), vitamin degradation (85–130 kJ mol \(^{-1}\)), and microbial growth (85–250 kJ mol \(^{-1}\)).

3. Conclusion and Outlook

This work presents printable supramolecular crosslinked structural colored CLC polymer coatings based on novel hydrogen-bonded cyclohexanoic acid derivatives which enable an extraordinary twofold thermal response. Exposure of the CLC polymer coating to a temperature above the \(T_{NI}\) results in rapid, irreversible structural color fading which can be exploited as photonic thermal paper. Precise imprinting of the colored label can be done by local exposure to temperature with a hot stamp, for instance. Alternatively, exposure to temperatures below the \(T_{NI}\) demonstrates the application of these coatings as time-temperature...
integrators: over a broad temperature range below the $T_{NI}$, an irreversible time-temperature dependent color transition is manifested as a gradual shift of the reflected wavelength from green to red. The irreversible thermochromic response can be programmed by altering the $T_{NI}$ between 30 °C and 90 °C through changing the composition. The structural colored coating establishes a time-temperature dependent response with a tunable color shift and a programmable temperature response range. When cooled below $T_g$, the polymer system shows no response over time, allowing for storage of pristine coating. In addition, this technology is printable, which makes it scalable for industrial applications such as labels with images of choice for management of temperature-sensitive medicine and food products.

Interestingly, preliminary studies show that the printed supramolecular polymer coatings can be easily delaminated, possibly making them reusable (Figure S9, Supporting Information). In general, this work introduces the incorporation of cyclohexanoic acid-derived hydrogen bonds in responsive supramolecular liquid crystal polymers and allows for new properties and possibilities for responsive materials which are commonly used to produce sensors, actuators, and membranes.

4. Experimental Section

Materials: 4-hexyloxy-phenyl 4-(6-Acryloyloxyhexyloxy)-2-methylbenzoate (M1a), 4-methoxy-phenyl 4-(6-acryloyloxyhexyloxy)-2-methylbenzoate (M1b), and (trans)-4-(6-acryloyloxyhexyloxy)cyclohexanecarboxylic acid (M2) were synthesized according to procedures described in the Supporting Information. ((3R,3aS,6S,6aS)-6-((4-((4-((6-acryloyloxy)hexyl)oxy)benzoyl)oxy)benzoyl)oxy)hexahydrofuro[3,2-b]furan-3-yl 4-((4-hexyloxy)benzoyl)benzoate (M3) was synthesized according to the procedure described in previous work. 4-((6-acryloyloxy)hexyloxy)phenyl 4-methoxybenzoate (M4) was purchased from Synthon Chemicals. The photoinitiator Irgacure 369 (M5) was obtained from CIBA. The surfactant BYK-361 N was obtained from BYK. Cyclopentanone was purchased from Sigma–Aldrich.

Inkjet Printing: The inkjet printing was performed on a PXDRO LP50 printer (DMC printhead) using 10 pL droplet size cartridges. As ink, the CLC mixture was dissolved in cyclopentanone (1:2 solid:solvent ratio) and the resulting solutions were filtered through a 0.2 µm PTFE syringe filter. As substrate, flexible black biaxially oriented (BOPET) 36 µm thick foil (Tenolan OCN0003) was used. As print patterns, 15 × 15 mm² squares were set with a DPI of 1200 to achieve a dense layer. During printing, the PET foil was fixed on the printer table by air suction and heated to 35 °C. The printhead was heated to 25 °C and operated at 25 V. Deposited layers were in the isotropic phase after solvent evaporation.

Figure 4. a) FT-IR measurements over time during exposure to 60 °C represent the hydrogen bond mode transition through broadening and shift of the peak at 1700 cm⁻¹. b) UV–vis reflection peak shifts and FT-IR peak intensity (1700 cm⁻¹) changes, expressing similar behaviors over time. c) Schematic representation of the proposed time-temperature dependent unwinding effect in a cholesteric helix with insets of the hydrogen bond mode.
Polymerization: After printing, the coated BOPEt foil was transferred to an N2 environment and cooled to the cholesteric phase and photopolymerized for 5 min with 20 mW cm−2 UV light using an Exfo Omnicure S2000 lamp. After polymerization, the coatings were stored in the freezer until measurements could take place.

Characterization: Characterization of the phase transitions of the liquid crystalline monomer mixtures and polymer coatings were analyzed by differential scanning calorimetry using a TA Instruments Q2000 calorimeter with constant heating and cooling rates of 10 °C min−1. The reflection of the CLC coatings was measured through ultraviolet–visible spectroscopy by using a PerkinElmer LAMBDA 750 with a 150 nm integrating sphere over a range of 350–800 nm equipped with a hotplate to measure transmission spectra at specific temperatures. The temperature-dependent equilibrium of hydrogen bonding was monitored by infrared spectroscopy using a Varian FT-IR 670 equipped with a heating stage and a golden gate ATR accessory in a range of 4000–650 cm−1. NMR spectra were recorded on a 400 MHz Bruker Avance III HD spectrometer. Matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS) was performed on a Bruker Autoflex Speed MALDI-MS instrument.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Supporting Information
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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available in the supplementary material of this article.

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