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Chameleon-Inspired Color-Changeable Colloidal Photonic Crystal Films Sensitive to Human Body Temperature

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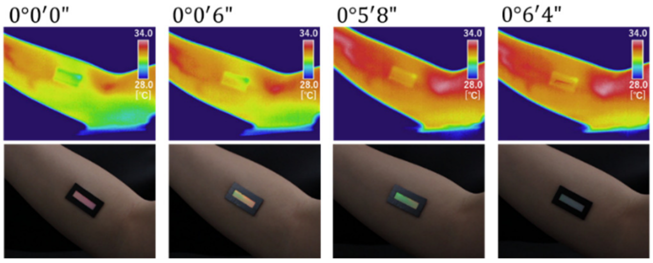
Received: 6 February 2025; Revised: 21 March 2025; Accepted: 24 March 2025; Published: 24 March 2025



Abstract: Artificial chameleon skins have been developed using advanced materials, such as photonic crystals, for camouflage and thermoregulation. However, to respond to subtle changes in human body temperature, the thermosensitivity, quality, and color response of these biomimetic films need to be improved. We report the development of chameleon-inspired color-changeable films with enhanced sensitivity to changes in the human body temperature. Non-close-packed colloidal photonic crystals were immobilized in a thermosensitive poly(*N*-isopropylacrylamide) (PNIPAM) hydrogel film and simultaneously attached to a flexible polyethylene terephthalate (PET) sheet by photopolymerization. The attachment to the PET sheet ensured high thermosensitivity and film quality besides ease of use. The film displayed full color spectrum from red to violet within a small range (~3 °C) of human body temperature without any change in the film area and film distortion. The temperature range of the full color spectrum was easily tuned by adding a poor solvent, ethylene glycol, to PNIPAM. The film attached to a human arm exhibited color changes from red to yellow, light green, and blue in response to changes in the body temperature without external heat. This study could contribute to the basic research and practical applications of artificial chameleon skins.

Keywords: structural color; colloidal photonic crystal; tunable color; chameleon; thermosensitive hydrogel

1. Introduction

Panther chameleons change their body color for camouflage, communication, and thermoregulation [1–5]. Their unique changeable color originates from the periodic nanostructure of guanine particles present in skin cells. Although guanine particles are colorless, the periodic nanostructure generates color by reflecting or scattering a specific wavelength of light depending on the spacing of the guanine particles. Chameleons can change the spacing of guanine particles, resulting in a color change. The color generated by the periodic nanostructure is called structural color [6–12]. Color-changeable films that mimic chameleon skins have been studied for applications such as artificial camouflage and sensors that detect changes in environmental conditions based on color changes. An important example is the elastomer-immobilized colloidal photonic crystal film, which comprises a non-close-packed particle array of submicron-sized monodisperse colloids embedded in an elastic polymer film [13–17]. When the film is attached to a human elbow and the elbow is bent, the film color changes according to the change in the particle spacing of the colloidal photonic crystals in the stretched elastomer [13]. However, this type of stress-responsive color change is limited to nearby joints because the film must be elongated or contracted to change the particle spacing.

Thermoresponsive hydrogel-immobilized colloidal photonic crystal films are promising color-changeable films that mimic chameleon skins [18–20]. The color changes are based on the change in the particle spacing of the non-close-packed colloidal photonic crystals, which is caused by the volume change of the surrounding hydrogel in response to a temperature change. In particular, the poly(*N*-isopropylacrylamide) (PNIPAM) hydrogel undergoes significant volume change near its phase transition temperature of 32 °C [21]; i.e., the PNIPAM hydrogel-immobilized colloidal photonic crystal film changes color within a temperature range that encompasses human body temperature. However, a significant color change in response to temperature changes (i.e., higher thermosensitivity) is required because changes in the human body temperature are small. In addition, the film quality must be preserved after the color change. The generation of various colors involves a significant volume change in the hydrogel, which generally results in a significant change in the film area and film distortion. Furthermore, easy color customization is indispensable because the human body temperature differs between individuals and body parts [22]. Although color tuning of thermoresponsive colloidal photonic crystal film is possible by varying the particle diameter, particle volume fraction, and thermosensitive monomer concentration, this approach is impractical because the laborious and time-consuming preparation must be repeated for the individual.

We previously found that the linear thermosensitivity of a PNIPAM-poly(*N*-methylolacrylamide) hydrogel-immobilized colloidal photonic crystal film can be increased 1.6-fold by bonding the film to a glass substrate using a silane coupling agent [23]. Furthermore, the film area does not change; no waving or warping occurs during the temperature change owing to the attachment of the film to the glass substrate. Herein, we report the preparation of chameleon-inspired color-changeable films that respond to small changes in the surface temperature of the human body without any change in the film area or distortion.

2. Materials and Methods

2.1. Materials

An ion-exchange resin (AG501-X8(D), Bio-Rad, Hercules, CA, USA) was added to an aqueous suspension of monodisperse polystyrene particles with a diameter of 200 nm (5020 B, Thermo Fisher Scientific, Waltham, MA, USA) and gently stirred to form charge-stabilized colloidal photonic crystals by deionization. The obtained colloidal photonic crystals were concentrated using a centrifuge (5702 RH, Eppendorf Corporate, Hamburg, Germany). Thereafter, a gelation reagent comprising *N*-isopropylacrylamide (NIPAM) monomer (FUJIFILM Wako Pure Chemical Corp., Tokyo, Japan), *N*,*N′*-methylene*bis*-acrylamide (BIS) crosslinker (FUJIFILM Wako Pure Chemical Corp., Tokyo, Japan), and 2,2′-azo*bis*[2-methyl-*N*-(2-hydroxyethyl)propionamide] (VA) photopolymerization initiator (FUJIFILM Wako Pure Chemical Corp., Tokyo, Japan) were added. Ultrapure water (Milli-Q system, Merck KGaA, Darmstadt, Germany) was added to adjust the concentrations of the polystyrene particles, NIPAM, BIS, and VA to 10 vol.%, 800 mM, 40 mM, and 0.4 mM, respectively.

2.2. Immobilization and Photopolymerization of the Film

A PET sheet (Lumirror T60, thickness: 250 µm, Toray Industries, Inc., Tokyo, Japan) was cut into a rectangular piece (7.5 cm × 2.5 cm), and the surface was treated with a plasma ion bombarder (PIB-20, Vacuum Device, Mito, Japan) for 90 s to attach a PNIPAM hydrogel film to the PET sheet by graft polymerization [24]. The PET sheet was sandwiched between a quartz substrate with a flow channel (channel height: 0.1 mm; width: 9 mm; length: 50 mm) and a plain quartz substrate to assemble the cell (Figure 1). The cells were fixed between metal frames and screws. The charge-stabilized colloidal photonic crystals with a gelation reagent were bubbled with Ar gas for 5 min and subsequently shear-flowed into the cell to form a single-crystalline structure [25,26]. The top and bottom of the cell were irradiated with ultraviolet (UV) light (MBRL-CUV7530, MORITEX Corporation, Saitama, Japan) for 90 min to photopolymerize the gelation reagent. The cells were then disassembled to obtain the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film. A 500-µm thick silicone spacer was placed on the PET sheet to surround the film. Thereafter, ethylene glycol aqueous solution with different ethylene glycol concentrations, C_{EG}, was added to the film as the swelling solvent and covered with a 250-µm thick PET sheet (Lumirror T60, Toray Industries, Inc., Tokyo, Japan) or a glass slide. For comparison, a PNIPAM hydrogel-immobilized colloidal photonic crystal film without the PET sheet was prepared. The film was cut into a 3-mm diameter disk and sandwiched between 250-µm thick PET sheets (Lumirror T60, Toray Industries, Inc., Tokyo, Japan) along with ultrapure water using a 500-µm thick silicone spacer.

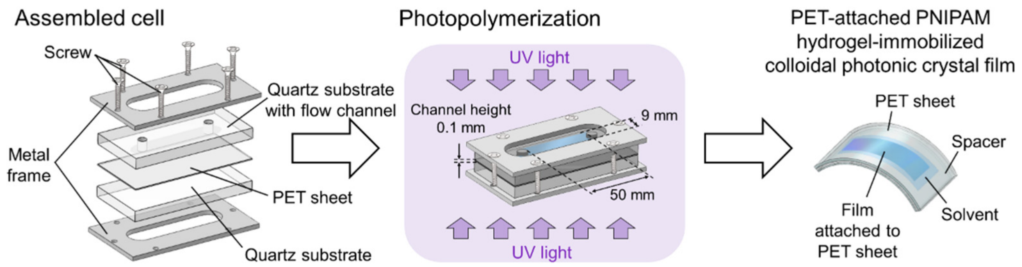


Figure 1. Schematic of the preparation process of the polyethylene terephthalate-attached poly(*N*-isopropylacrylamide) hydrogel-immobilized colloidal photonic crystal film.

2.3. Characterization

The reflection spectra of the colloidal photonic crystals at normal incidence were measured before and after UV light irradiation using a fiber spectrometer (Fastevert S-2630, Soma Optics, Ltd., Tokyo, Japan). The samples were placed on a thermal stage (KM-1, Kitazato Corporation, Tokyo, Japan) and the temperature was increased. The photograph and reflection spectrum at normal incidence were recorded at various temperatures using a charge-coupled device camera (XCD-V60CR, Sony, Tokyo, Japan) and fiber spectrometer (Fastevert S-2630 or S-2431 model II, Soma Optics, Ltd., Tokyo, Japan), respectively. The temperature distribution on the film surface was measured using an infrared thermal imaging camera (R300SR, Nippon Avionics Co., Ltd., Yokohama, Japan).

3. Results and Discussion

Figure 2a shows the reflection spectrum (at normal incidence) of the colloidal photonic crystals on the PET sheet in the cell before and after UV light irradiation. Before irradiation, a strong reflection peak is observed at 867 nm. Considering the Bragg condition, this peak originates from the (111) lattice planes of a face-centered cubic (FCC) structure aligned parallel to the cell surface [27]. The Bragg equation is as follows:

$$\lambda_{111} = 2n_c d_{111}, \tag{1}$$

where λ_{111} and d_{111} are the Bragg wavelength and lattice spacing of FCC (111) planes, respectively, and n_c is the refractive index of the colloidal photonic crystals. The value of n_c can be approximated as the volume-weighted average of the refractive indexes of the components [28]:

$$n_c = n_p\phi_p + n_{pol}\phi_{pol} + n_w\phi_w, \tag{2}$$

where n_p, n_{pol}, and n_w are the refractive indexes of polystyrene particles (n_p = 1.59), polymer (n_{pol} = 1.45), and water (n_w = 1.33), respectively. The volume fractions of the polystyrene particles (ϕ_p), polymer (ϕ_{pol}), and water (ϕ_w) exhibit the relationships $\phi_w = 1 - \phi_p - \phi_{pol}$ and $\phi_{pol} = 1.03\ \phi_p$ [29]. Furthermore, ϕ_p was determined from the geometrical properties of the FCC structure using d_{111} and particle diameter d as:

$$\phi_p = \frac{2\pi}{9\sqrt{3}}\left(\frac{d}{d_{111}}\right)^3. \tag{3}$$

By substituting $\phi_p = 0.1$ (determined from the particle concentration of the preparation) in Equations (2) and (3), λ_{111} was calculated as 871 nm using Equation (1), which is in good agreement with the observed Bragg wavelength.

Under UV light irradiation, the NIPAM monomer and BIS crosslinker were photopolymerized to immobilize the non-close-packed colloidal photonic crystals almost intact in the PNIPAM hydrogel film and were simultaneously attached to the PET sheet by graft polymerization [24]. A slight reduction in the intensity and blue shift of the Bragg reflection peak are observed after UV irradiation, probably because of the evaporation of water during photopolymerization. When the cell was disassembled, a PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film was obtained. The film was easy to handle and exhibited good flexibility because of the support of the PET sheet, as shown in Figure 2b.

Figure 3a,b show the photographs and reflection spectra at normal incidence, respectively, of the PET-unattached and PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal films at various temperatures. As the temperature increases, the PET-unattached film shrinks and exhibits a red color at 31.5 °C. With further increase in the temperature, the film shrinks more and warps above 32.4 °C. The film ceases to shrink above 34.0 °C. In the reflection spectrum, the Bragg peak markedly blueshifts with increasing temperature and then saturates to approximately 600 nm above 34.0 °C. These behaviors are typical of PNIPAM hydrogel-immobilized colloidal photonic crystals [18,19]. In contrast, the PET-attached film exhibits better color changes without any change in the film area or warping. The full color spectrum from red to violet was displayed in a temperature range of approximately 3 °C (31.0–34.2 °C). Figure 3c shows the Bragg wavelengths of the PET-unattached and PET-attached films as a function of temperature. The Bragg wavelength of the PET-attached film is consistently lower than that of the PET-unattached film. While the Bragg wavelength of the PET-unattached film saturates to approximately 600 nm above 34.0 °C, that of the PET-attached film decreases further and reaches 332 nm at 35.1 °C. The thermosensitivities, $\Delta\lambda/\Delta T$, of the PET-unattached and PET-attached films were calculated from the Bragg wavelengths at different temperatures (Figure 3d). The thermosensitivity was enhanced by attaching the PNIPAM hydrogel-immobilized colloidal photonic crystal film to the PET sheet. While the maximum thermosensitivity of the PET-unattached film is −76 nm/°C at 31.5 °C, that of the PET-attached film is enhanced to −171 nm/°C at 32.8 °C.

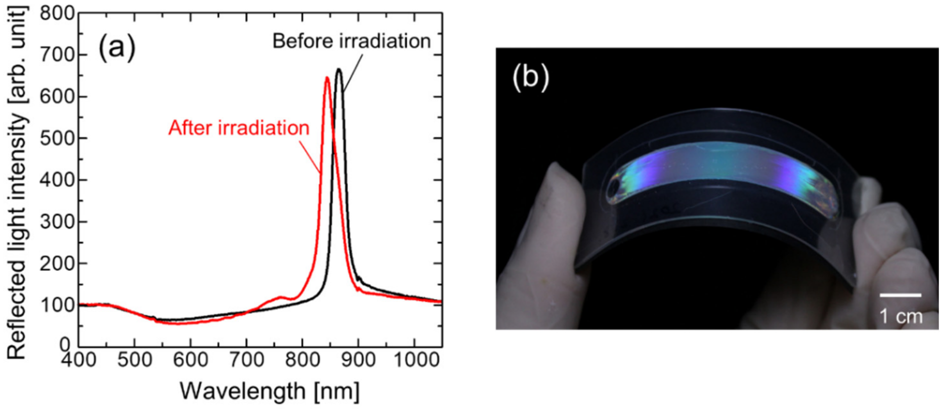


Figure 2. (**a**) Reflection spectrum (at normal incidence) of the non-close-packed colloidal photonic crystals on the polyethylene terephthalate (PET) sheet in the cell before and after ultraviolet light irradiation. (**b**) Photograph of the PET-attached poly(*N*-isopropylacrylamide) hydrogel-immobilized colloidal photonic crystal film covered with the PET sheet.

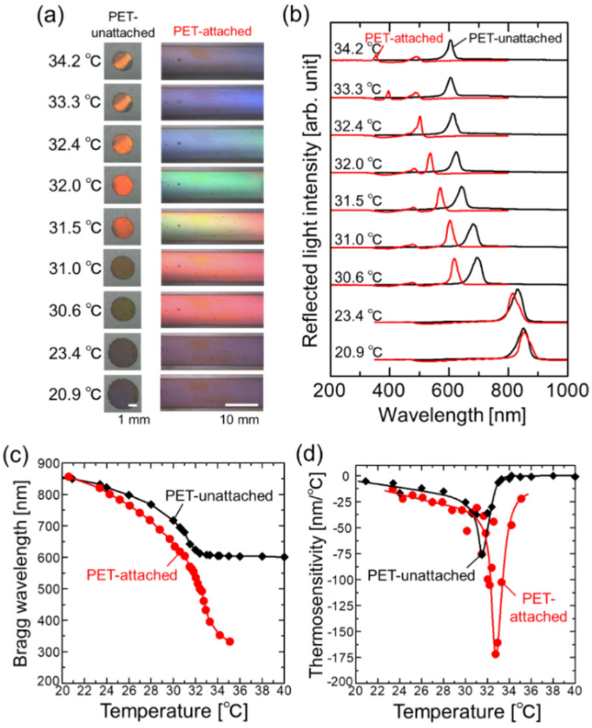


Figure 3. (**a**) Photographs and (**b**) reflection spectra of the polyethylene terephthalate (PET)-unattached and PET-attached poly(*N*-isopropylacrylamide) (PNIPAM) hydrogel-immobilized colloidal photonic crystal films at various temperatures. (**c**) Bragg wavelengths and (**d**) thermosensitivities of the PET-unattached and PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal films as a function of temperature.

The enhanced thermosensitivity of the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film arises from the anisotropic contraction of the PNIPAM hydrogel [23,29]. The PET-unattached PNIPAM hydrogel-immobilized colloidal photonic crystal film maintained the FCC structure during contraction with increasing temperature. However, for the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film, the contraction in the thickness direction increased because the in-plane shrinkage was suppressed. The observed color or Bragg wavelength is derived from the lattice spacing parallel to the thickness direction; hence, the Bragg wavelength exhibited a greater blue shift. Furthermore, the colloids in the lattice planes perpendicular to the thickness direction remained apart and did not interfere with each other during contraction. This resulted in a smaller lattice spacing parallel to the thickness direction in the maximum shrinkage state. Thus, the ultimate Bragg wavelength of the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film reached a much smaller value of 332 nm.

The temperature range in which the full color spectrum from red to violet is displayed can be easily tuned by adding a poor solvent such as ethylene glycol to PNIPAM. The lattice spacing of the colloidal crystals embedded in the PNIPAM hydrogel reduces with an increase in the C_{EG}. Therefore, the temperature range exhibiting the full color and Bragg wavelength of the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film shifts to lower values with increase in the C_{EG}, as shown in Figure 4a,b. A film with C_{EG} = 20 wt.% exhibited the full color spectrum between 24.3 and 27.9 °C. Although the maximum thermosensitivity decreased to −122 nm/°C at C_{EG} = 20 wt.% and 27.4 °C, the value is considerably high (Figure 4c).

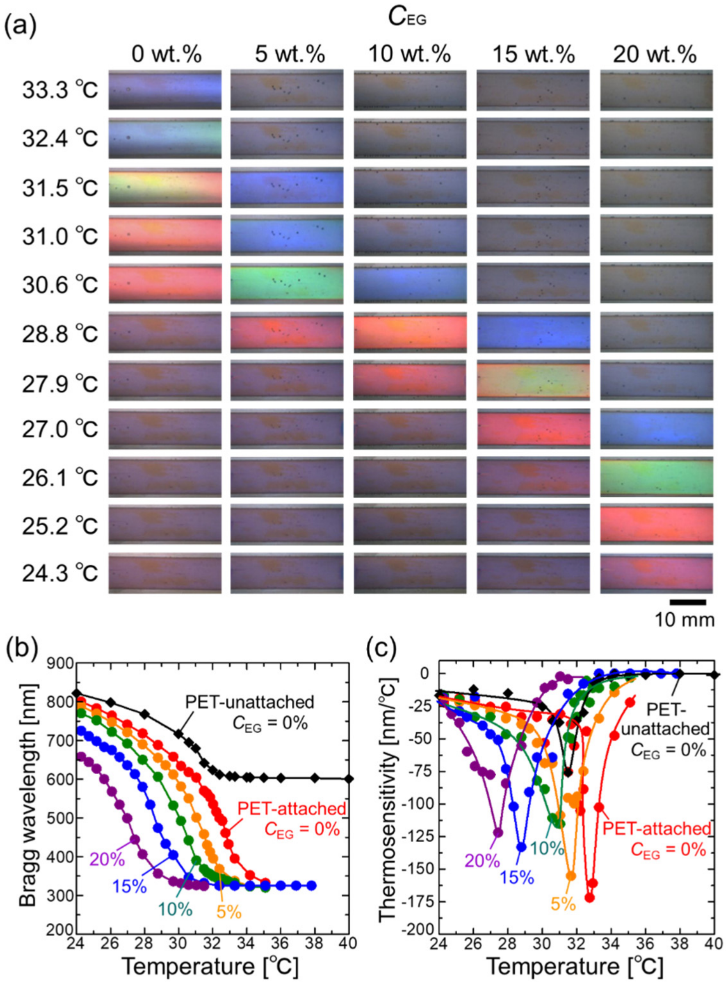


Figure 4. (**a**) Photographs of the polyethylene terephthalate (PET)-attached poly(*N*-isopropylacrylamide) (PNIPAM) hydrogel-immobilized colloidal photonic crystal films with different ethylene glycol concentrations (C_{EG}) at various temperatures. (**b**) Bragg wavelengths, and (**c**) thermosensitivities of the PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal films with different C_{EG} as a function of temperature.

The PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film was used to visualize the temperature distribution. When a copper wire heated to 40 °C was brought closer to the film, the film displayed the full color spectrum from violet to red spreading radially outward close to the copper wire (Figure 5). The temperatures marked at different positions in Figure 5 were determined using thermography. The correspondence between the color and temperature at each position is in reasonable agreement with that shown in Figure 4a.

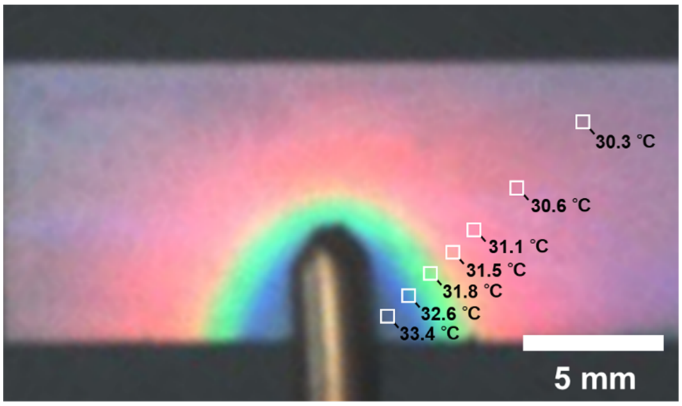


Figure 5. Photograph showing the color change of the polyethylene terephthalate-attached poly(*N*-isopropylacrylamide) hydrogel-immobilized colloidal photonic crystal film (prepared with ethylene glycol concentration of 0 wt.%) when a copper wire heated to 40 °C was brought closer to the film.

The PET-attached PNIPAM hydrogel-immobilized colloidal photonic crystal film changes color in response to changes in the surface temperature of the human body without the application of external heat. Figure 6 shows the color changes occurring in the film attached to a human arm while the arm was swinging. After resting for 30 min in a room maintained at a temperature of 23.0 °C, the surface temperature of the film (as measured by thermography) stabilized to 31.1 °C. In the initial state, the film color was red. When the arm was swung from side to side using the elbow as a fulcrum, the film temperature increased to 31.5 °C after 6 s, and the film color began changing to yellow. After 5 and 6 min, the film temperature increased to 31.9 and 32.6 °C, and the film color changed to light green and blue, respectively. When the arm stopped swinging, the film color returned to light green, yellow, and red, in that order, over approximately 77 min, corresponding to a decrease in the surface temperature of the arm.

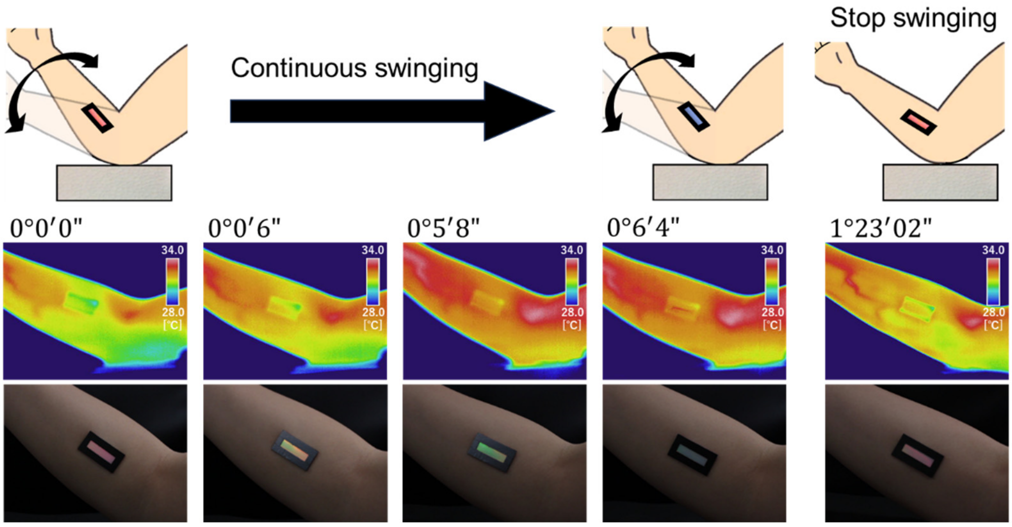


Figure 6. Photographs and thermography images of the polyethylene terephthalate-attached poly(*N*-isopropylacrylamide) hydrogel-immobilized colloidal photonic crystal film (prepared with ethylene glycol concentration of 0 wt.%) attached to the human arm when the arm was continuously swung from side to side and then stopped.

4. Conclusions

We demonstrated chameleon-inspired color-changeable films that respond to small changes in the surface temperature of the human body. The films were successfully prepared by immobilization of non-close-packed colloidal photonic crystals in a thermosensitive PNIPAM hydrogel film and simultaneous attachment to a flexible PET sheet through photopolymerization. Attaching the film to the PET sheet ensured high thermosensitivity and film quality; the full color spectrum from red to violet was displayed without any change in the film area and film distortion in a small temperature range of approximately 3 °C, which encompasses the surface temperature of the human body. Furthermore, the temperature range of the full color spectrum was easily tuned by adding a poor solvent such as ethylene glycol to PNIPAM. The color-changeable film supported by the PET sheet was easy to handle and exhibited excellent flexibility. When attached to a human arm, the film exhibited color changes in response to the changes in the body temperature without the application of external heat. Although the PET-attached film is sandwiched between PET sheets, tight sealing is necessary for long-term use to prevent evaporation of the swelling solvent. This study is expected to advance fundamental research and applications of artificial chameleon skins.

Author Contributions: T.K.: conceptualization, data curation, funding acquisition, supervision, writing—original draft, writing—review & editing. M.S.: data curation, investigation, visualization. Y.H.: data curation, investigation, validation, visualization. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by JSPS KAKENHI Grant Number 18H01721.

Data Availability Statement: The data that support the findings of this study are available from the corresponding authors upon reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

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