Journal of Physics: Condensed
Matter

**PAPER** • **OPEN ACCESS**

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To cite this article: Sangchul Roh *et al* 2024 *J. Phys.: Condens. Matter* **36** 425101

OPEN ACCESS
IOP Publishing
Journal of Physics: Condensed Matter
J. Phys.: Condens. Matter **36** (2024) 425101 (7pp)
https://doi.org/10.1088/1361-648X/ad6110

# Transparency-changing elastomers by controlling of the refractive index of liquid inclusions

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Received 30 April 2024, revised 11 June 2024
Accepted for publication 9 July 2024
Published 19 July 2024

## Abstract

Complex materials that change their optical properties in response to changes in environmental conditions can find applications in displays, smart windows, and optical sensors. Here a class of biphasic composites with stimuli-adaptive optical transmittance is introduced. The biphasic composites comprise aqueous droplets (a mixture of water, glycerol, and surfactant) embedded in an elastomeric matrix. The biphasic composites are tuned to be optically transparent through a careful match of the refractive indices between the aqueous droplets and the elastomeric matrix. We demonstrate that stimuli (e.g., salinity and temperature change) can trigger variations in the optical transmittance of the biphasic composite. The introduction of such transparency-changing soft matter with liquid inclusions offers a novel approach to designing advanced optical devices, optical sensors, and metamaterials.

Supplementary material for this article is available online

Keywords: optical metamaterials, liquid inclusions, stimuli-responsive soft matter

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## 1. Introduction

Many widely used products consisting of multiphasic complex materials (e.g., hand creams, paints, salad dressings) appear opaque because they contain particles or droplets dispersed throughout another fluid [1–6]. The refractive indices of the dispersed phase inclusions (particles or droplets) are usually different than the surrounding media, causing incident light to scatter as it passes through the multiphasic materials. The dispersed particles or droplets refract the propagating light, resulting in the white appearance of the multiphasic complex materials. In addition to many other factors, such as particle size and volume fraction, the tuning of the refractive indices between dispersed phase and continuous phase endows biphasic materials with a wide range of opacity (or light transmittance) [7]. However, biphasic materials can look optically clear when the refractive indices between dispersed phases and continuous phases are carefully matched [8].

Adaptive materials changing optical transmittance in response to external stimuli can find applications such as light shutters, smart windows or sensors [9–17]. For example, polymer-dispersed liquid crystals (PDLCs) exhibit switching between a transparent state and an opaque state by turning the external electric field on and off [15, 18, 19]. PDLCs are biphasic materials comprised of liquid crystal droplets and polymer matrix. The electric field can control the refractive index (RI) of liquid crystalline droplets, leading to the RI match or mismatch between liquid crystalline droplets and the polymer matrix. Thus, biphasic materials with on-demand control of the refractive indices of the dispersed phases could provide an engineering platform for designing advanced optical systems, such as ones including active and adaptive optical metamaterials [20, 21].

We report here the design of another class of simple and efficient transparency-changing composites, which is based on elastomeric composites (polydimethylsiloxane, PDMS) with embedded aqueous droplets. We explore the optical property (light transmittance) of these biphasic composites by varying the RI of the internalized aqueous droplets. We further demonstrate the tunability of the light transmittance of the composites in response to external stimuli including osmotic pressure difference and temperature.

## 2. Materials and methods

To prepare elastomeric biphasic composites (figure 1(a)), mixtures of water and glycerol were emulsified in liquid-phase PDMS precursor (Sylgard$^{\mathrm{TM}}$ 184 Silicone Elastomer Kit, Dow). The PDMS liquid precursor consisted of a silicone base and curing agent, that was mixed by a 10:1 (w:w) ratio. The homogeneously blended PDMS precursor was degassed in a vacuum chamber. Water–glycerol (WG) mixtures (Millipore water and glycerol from Sigma-Aldrich), containing 0.1 wt.% of Tween$^{®}$20 as an interface stabilizer, were added to sheared PDMS precursor using a syringe pump (0.1 ml min$^{-1}$, New Era) in a dropwise manner. The PDMS precursor was sheared by a ServoDyne mixer (Cole-Parmer, Model # 50003) equipped with a three-bladed impeller (1.5 "Lab Hydrofoil with 3/8" Bore Mixer-Direct) at 150 rpm. After adding the desired amount of WG mixture, the PDMS was agitated for 15 min for homogeneous droplet distribution in the PDMS matrix. The dropwise addition of the WG mixture into the sheared PDMS precursors allows the WG droplets to be uniformly distributed in the PDMS medium up to ≈50 vol.% of the droplet phase. The WG/PDMS blends were then poured into an acrylic mold. The WG/PDMS-containing mold was sealed to prevent water evaporation. After crosslinking the WG/PDMS blends at an elevated temperature (85 °C), elastomer biphasic composites (EBCs) were prepared.

The light transmittance of EBCs was measured with a UV–Vis spectrophotometer (Jasco, Japan). For the investigation of light transmittance change of EBCs in aqueous solutions, the EBCs were immersed in aqueous solutions contained in cuvettes of 10 mm optical path length. Millipore water in the same type of cuvette was used as an optical reference. The morphologies of EBCs were characterized by an optical microscope (Olympus BX-61) and a scanning electron microscope (Verios).

## 3. Results

In our initial experiment, we prepared EBCs containing 10 vol.% of WG mixture in which the glycerol concentration varied from 40 to 80 wt.%. Most EBCs had a translucent to opaque appearance. However, EBC containing droplets of glycerol concentration of 60 wt.% appeared visually clear and optically transparent (figures 1(b) and (c)) with ≈100% of optical transmittance at all visible light wavelengths (400–900 nm) (figures S1(a) and (b)). However, a small deviation of water concentration in the droplet phase gave rise to a dramatic decrease in the transmittance ($T_{40\ wt.\%}$–$T_{41\ wt.\%} \approx 55\%$) (figure 1(b)). The variation in the transmittance can be more pronounced as the vol.% of droplet phase in the PDMS medium increased, indicating that most of the incident light was scattered in all directions in the composites and barely reached to the detector of the UV–Vis spectrophotometer. Further, the WG mixture allowed us to include various dyes (Brilliant green, Fluorescein sodium salt, Bromophenol blue, and Allura red) that dissolve molecularly in the droplet phases. As these dyes were completely dissolved in the WG mixtures without scattering from particulates, the EBC composites attained their color of florescence emission spectra without compromising the material clarity (figure 1(d)). Although relatively simple, this capability may be valuable for making clear colored or fluorescent PDMS, whose color is derived from common hydrophilic dyes, which are otherwise molecularly immiscible with the silicone matrix.

The optical transmittance ($T$) of biphasic materials can be approximated by the Beer-Lambert law, $T = I/I_0 = \exp(-NL\pi RQ)$, where $N$ is the number density of droplets, $R$ is the radius of the droplets $I_0$ and $I$ are the intensity of incident light and the light attenuated after optical pathlength $L$ by an extinction factor, $Q$. According to the literature, when the refractive indices between dispersed

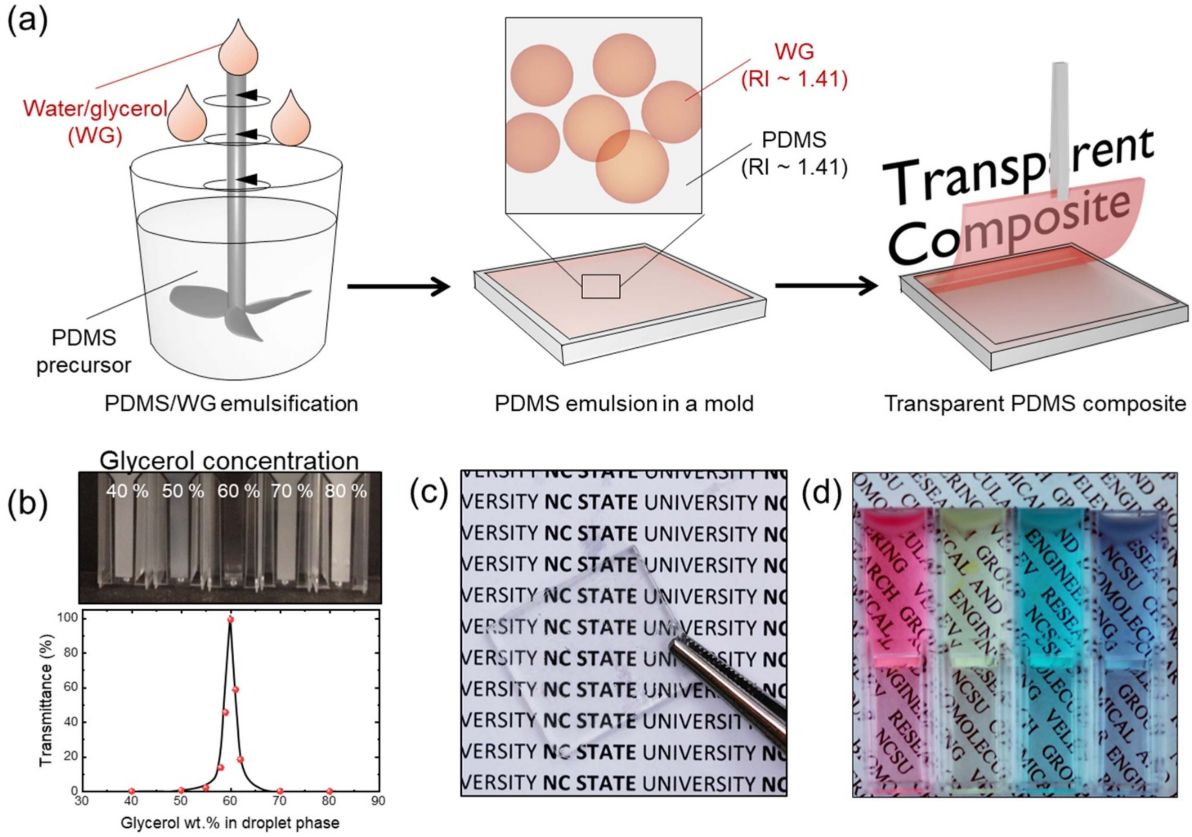

**Figure 1.** (a) Schematics of the silicone composite fabrication. Water–Glycerol (WG) mixture containing 0.1 wt.% of Tween 20 is added in a dropwise manner to liquid PDMS precursor under shear. The WG in PDMS emulsion is then molded and thermally crosslinked at 85 °C. (b) A Transmittance of the PDMS composites (10 mm optical pathlength, fixed light wavelength of 650 nm) containing 10 vol.% of liquid phase in which glycerol concentration varies from 40 wt.% to 80 wt.%. The line was added to guide the eye. (c) A block of a 1 mm thick elastomeric biphasic composite (EBC) with 30 vol.% of liquid phase containing 60 wt.% glycerol in water. The composite is optically transparent, and the letters behind the composite are clearly visible. (d) Colored composites with dyes dissolved in the WG droplet phase. The dyes are Allura red (0.0001 wt.%), Fluorescent sodium salt (0.0001 wt.%), Brilliant green (0.00007 wt.%), and Bromophenol blue (0.0001 wt.%) from left to right.

phase and continuous phase are matched, $Q$ converges to 0, resulting in ~100% transmittance [8, 22, 23]. Based on the relation, we interpreted that the RI of the WG droplet phase (pure water $n \approx 1.33$ and glycerol $n \approx 1.47$) are matched to that of the PDMS phase ($n \approx 1.41$) at a glycerol concentration of 60 wt.%; thus the EBCs are optically clear (figure 1(b)). Indeed, the transmittance of the composites is very sensitive to the water and glycerol mixing ratio (figure 1(b)).

Our experimental results and the analysis in figure 1 suggest that the optical transmittance of EBC can be varied in response to stimuli that trigger the change in the RI of the droplet phase. Among various stimuli, we observed EBCs to change their transparency when they are immersed in aqueous solutions of glycerol as an immersion medium for EBCs. When a 1.0 mm thick EBC containing RI-matched WG droplets (60 wt.% of glycerol in water) was immersed in a vial filled correspondingly with 60 wt.% glycerol solution, the EBC is completely transparent and hidden in the WG solution (figure 2(a)). On the other hand, when the EBC was immersed in a vial filled with water in the absence of glycerol (figure 2(b)), the EBC became hazy over time. Microscopically, while the RI-matched WG droplets were hidden in the PDMS matrix and barely seen (figure 2(a)), the WG droplets of opaque EBCs that were incubated in pure water were directly visible with an optical microscope (figure 2(b)). Thus, we hypothesized that contact of the EBC with aqueous environments triggers water infusion into the droplets and RI mismatch between the droplets and the PDMS matrix, leading to the optical emergence of the droplets and transmittance change in EBCs (figure 2).

Prior studies have reported that water can diffuse across a PDMS medium with a diffusivity of $\sim 10^{-9}$ m$^2$ s$^{-2}$ [24–26]. Consequently, PDMS is permeable to water despite its inherent hydrophobicity. Thus, we expect that water from the immersion medium diffuses into the WG droplet, leading to the dilution of WG droplets. The directional diffusion of water molecules from the immersion medium towards the WG droplets can take place due to the difference in the osmotic pressure between WG droplets and water in the immersion medium ($\Delta\Pi \approx 36$ MPa, see also SI) [27]. If the WG mixture in the EBC droplets is diluted by water inflow, the RI of the droplet phase decreases over time, which may lead to RI mismatch and light scattering from the droplet/PDMS surfaces (figure 2(b)).

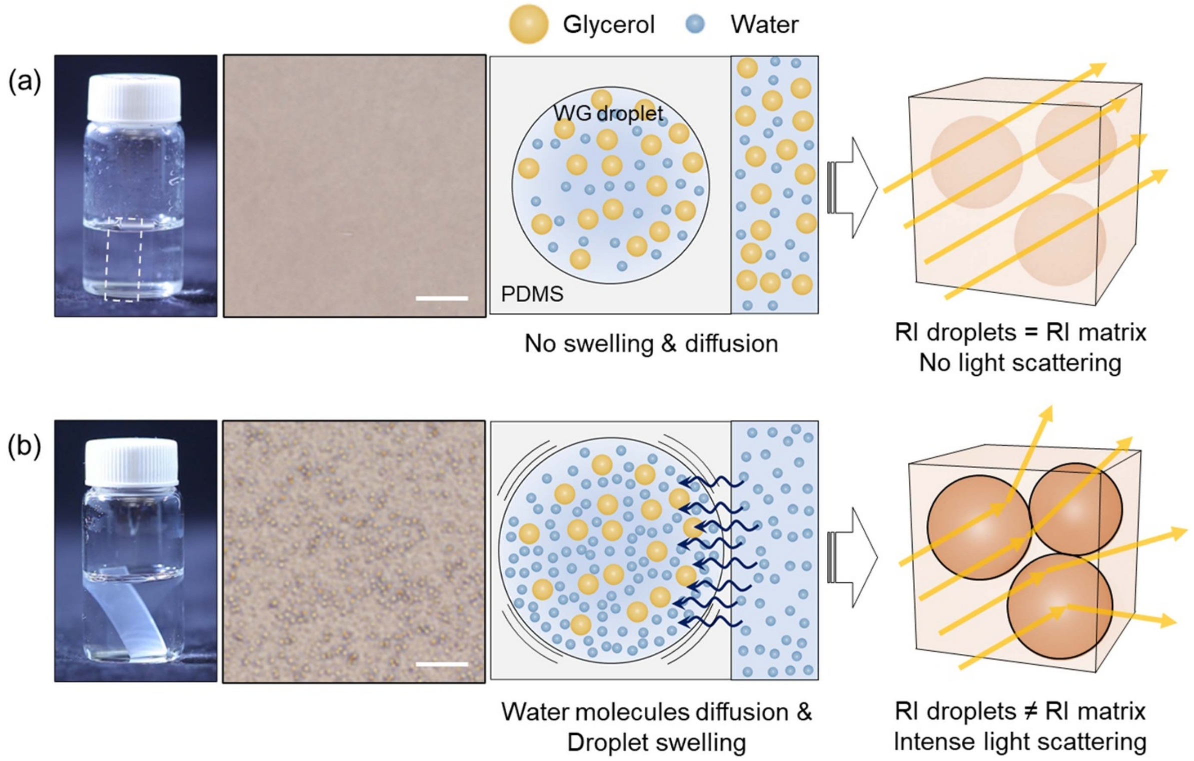

**Figure 2.** Optical response of EBCs to aqueous immersion media. The EBCs include 30 wt.% of RI-matched WG droplets. (a) An EBC immersed in 60 wt.% glycerol in water (isosmotic condition). The white dotted rectangle indicates the location of EBC in the vial on the left. The WG droplets in the PDMS composite remain invisible and the EBC remains transparent. (b) An initially transparent EBC immersed in pure water for 30 s. The EBC swells with water as the water molecules diffuse from the immersion medium into the droplets. Due to the infusion with additional water, the RI of the droplet phase decreases and the WG droplets begin to scatter incident light. As a result, the EBC becomes opaque. Scale bars: 20 $\mu$m.

To explore our proposed mechanism (figure 2) of the optical property change in EBCs, we measured the swelling ratio and transmittance change as a function of time with RI-matched WG droplets by immersing sheets of the EBC material in water and glycerol mixtures of varying composition. In these WG media, the osmotic pressure varies from 0 to 36 MPa at 0–60 wt.% of glycerol (see also figure S2) [27]. The swelling ratio of EBCs increased over time except for EBCs that were incubated in an isosmotic medium (60 wt.% of glycerol in water). As the wt.% of glycerol in the immersion medium increased, the swelling was more rapid (figure 3(a)). Regardless of the glycerol content (or osmotic pressure), however, the swelling rate decreased over time, as an indication of the approaching of new medium-EBC equilibrium. These experiments led us to conclude that the osmotic pressure difference between WG droplets and the immersion medium dictates the amount of water uptake.

To understand further the relation between optical appearance and swelling ratio, we measured the optical transmittance of EBCs in water and glycerol mixtures (glycerol contents: 0–60 wt.%) as a function of time (figure 3(b)). Except for the EBC immersed in the isosmotic condition, transmittances of all EBCs decreased over time. The change in the transmittance was more rapid as the amount of glycerol in the immersion medium decreased, leading to distinct variations in the opacity of EBCs immersed for the same time period (inset of figure 3(b)).

According to figures 3(a) and (b), as the swelling ratio increases, the optical transmittance exponentially decreases, which is consistent with the deviation of the RI ratio, $m$, of the droplets and medium from 1. Therefore, we conclude that the transmittance of the composite is directly correlated to the amount of infused water, which in turn is regulated by the osmotic pressure difference. The loss of transmittance is likely completely balanced by the internal and external scattering of the light, as none of the materials used adsorbs at visible wavelengths. The transmittance increases as the osmotic pressure of the water medium increases (figure 3(c)), thus the osmotic pressure difference between the WG droplets and the immersion medium triggers changes in the transmittance of EBCs. In other words, the EBCs can optically report the osmotic pressure of immersion media.

Having enabled the capability of changing the transparency of EBCs via the osmotic pressure of the media, we evaluated the optical properties of EBCs immersed in NaCl aqueous solutions (10–24 wt.%). Similar to glycerol and water mixture media, the transmittance of EBCs in the NaCl aqueous solutions decreased over time (figure S3) and was shown to exhibit distinct variations in the transparency correlated to the NaCl concentrations (figure 4). Especially, at the saturation point of

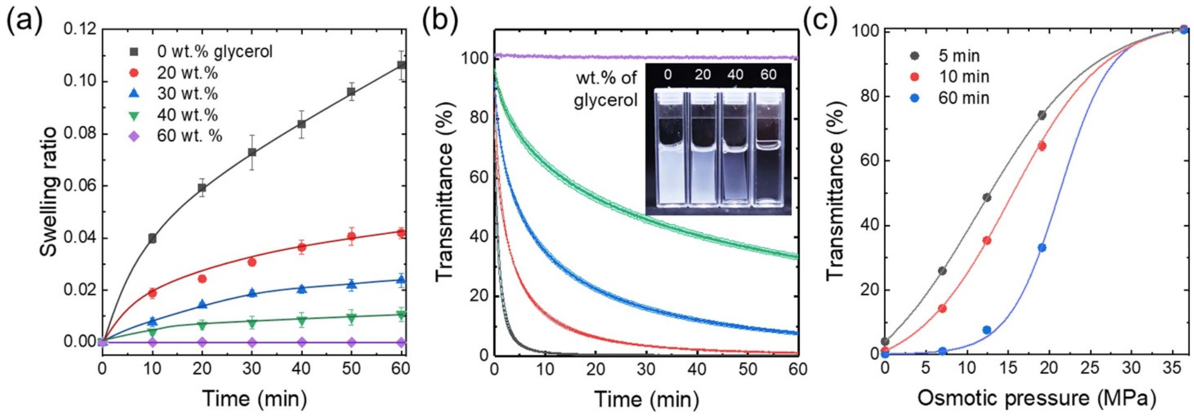

**Figure 3.** Changes in swelling and transmittance of EBCs immersed in WG media of varying compositions. (a) Swelling ratio of EBCs in media of varied wt.% of glycerol in water. The line was added to guide the eye. (b) Transmittance change of the EBCs over time with the variation of water/glycerol ratio at 650 nm. The inset picture shows EBCs immersed in the WG media for 5 min. The color of the curves corresponds to the legend in (a). (c) The dependency between osmotic pressure and transmittance. The transmittance was read from the EBCs immersed in WG media for 5 min, 10 min and 1 h of (b). The EBCs contain 30 wt.% of RI-matched WG.

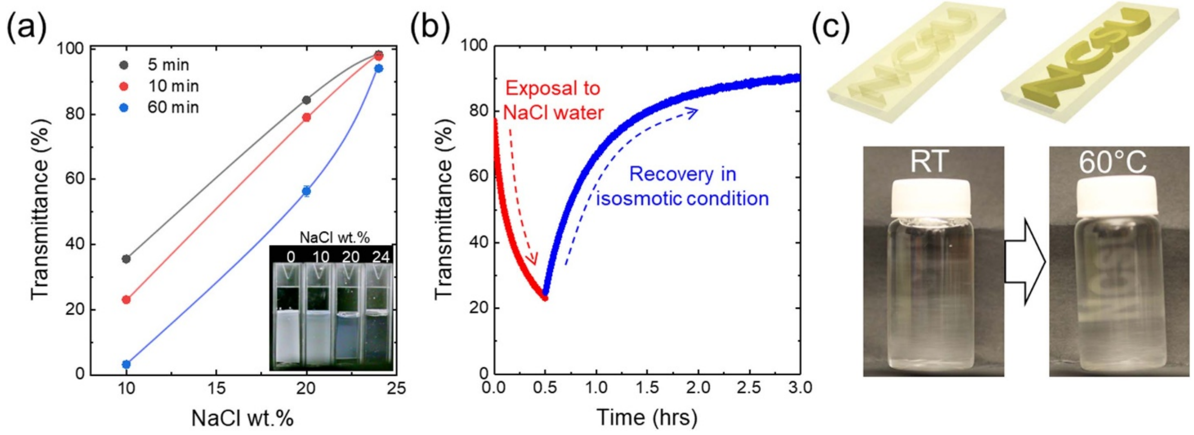

**Figure 4.** (a) Transmittance of EBCs immersed in NaCl aqueous solutions at varied NaCl concentrations (10−24 wt.%). The inset is a photograph of EBCs incubated for 5 mins in NaCl aqueous solutions. The line was added to guide the eye. (b) The loss and recovery of the transmittance of an EBC, immersed in 15 wt.% NaCl/water medium for 30 min followed by immersion in isosmotic solution. (c) Encryption and revealing of the letter 'NCSU' encoded inside an EBC. A PDMS block embedded with letters consisting of EBCs is immersed in vials with WG solution and heated. The EBCs contain 30 wt.% of RI-matched WG.

NaCl in water (24 wt.%) at room temperature, the EBCs exhibited <10% decrease in transmittance, indicating that the water barely diffused into the WG droplets due to similar osmotic pressure of the WG droplet phase (∼36 MPa) and the NaCl solution (∼31 MPa) [28].

When the EBCs incubated in NaCl solutions were immersed back in an isosmotic solution, the ≈100% transmittance of the EBCs was recovered. For example, an EBC was shown to exhibit a transmittance drop to 24% in 15 wt.% of NaCl solution in 30 min, but once immersed back into an isosmotic liquid, ≈90% of the transmittance was recovered in 3 h and the EBCs became fully transparent in <12 h (figure 4(b)). The EBC fully regenerated its transparency even after > 20 cycles of repeated exposure to the NaCl solution followed by an isosmotic solution. Further canvassing experiments revealed that the osmotic pressure-dependent transmittance changing capability is not limited to NaCl solutions but also occurs in solutions of $CaCl_2$ or water-soluble polymers (e.g., polyethylene oxide), indicating that EBCs can be used as an alternative method to visually estimate osmotic pressures, second virial coefficient, and molecular weight of polymers in a various aqueous medium.

While we focus on osmotic pressure-driven transparency-tuning, the EBCs can respond to numerous other stimuli. For example, the EBCs also respond to temperature changes. Although the RI between the PDMS and droplet phases in the EBCs is matched at room temperature, the RI of each phase has a different dependency on temperature RI($T$). Therefore, the RI between the two phases became mismatched as the surrounding temperature increased. The EBC became opaque at elevated temperatures, which indicates that the RI ratio diverges from 1. The capability of changing transmittance as a function of temperature can be useful for designing smart windows because EBCs may be able to sustain the temperature of buildings by regulating the sunlight radiation entering through the windows. Similar trends in changes in the optical transparency (transparent → opaque) was also observed when the EBCs were simply dried in the air. We believe that the change in the transparency takes place as a result of loss of water from the droplet phases upon drying. In contrast to water-uptake induced decrease in RI of droplets, which was the case for EBCs incubated in aqueous phase, the RI of the droplets may increase when they become enriched with glycerol upon water evaporation. It is presumed that the transmittance change behavior is dictated by the difference in the chemical potential, $\Delta\mu = {\mu_{\text{water}}}^{\text{air}} - {\mu_{\text{water}}}^{\text{droplet}}$, thus, EBCs could also in the future be used to design a new functional soft materials that optically report humidity of air [29, 30].

Finally, we demonstrated how the heating response can be used in EBC samples with embedded internal patterns of 'invisible' WG droplets. We prepared a pure PDMS mold carved with the letters, 'NCSU' (figure 4(c)). The space was further filled with a WG/PDMS biphasic mixture and crosslinked. When heated in the isosmotic W/G solution, the initially hidden letters that were embedded in the pure PDMS matrix were subject of temperature-induced RI mismatch, revealing the hidden text. When the composite was exposed to room temperature again, the letters became hidden again, indicating that the switching between translucent and transparent state is reversible by changing the surrounding temperature with a switching time of ∼10 min. Note that this change also can be triggered by osmotic pressure. The on-demand spatioselective response may allow the EBCs to be utilized as information encryption and anti-counterfeiting devices [31].

## 4. Conclusions

We demonstrate a new class of optically transparent, soft, biphasic composites that are formed via inclusions of RI-matched droplets in an elastomeric matrix. Based on Beer–Lambert law, the optical transmittance of the biphasic materials changes dramatically as a function of the RI ratio between the dispersed phase and the matrix. We demonstrated that as the droplet RI is a function of the environmental conditions such as osmotic pressure or temperature, the EBCs change their light transmittance in response to external stimuli. While we focus on osmotic pressure and temperature as stimuli in this report, the use of the biphasic system to change light transmittance by other stimuli that control the RI of droplets is highly applicable. Overall, our approach toward transparency-changing systems based on biphasic composites has the potential to enable the fabrication of soft materials for applications in semiquantitative measurements of various environmental stimuli. It is also expected that the switching time of the composite can be further engineered by varying the number density of droplets, droplet size, and PDMS crosslinking density. Thus, other possible applications for new class of transparency changing materials include coatings for smart windows, color-changing materials, and other soft optical metamaterials with on-demand RI-controlling capabilities [32].

## Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

## Acknowledgments

This work is supported by the National Science Foundation Grants CBET-1935248 and partially CBET 2133983. This project has received support from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie Grant Agreement No 956248.

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