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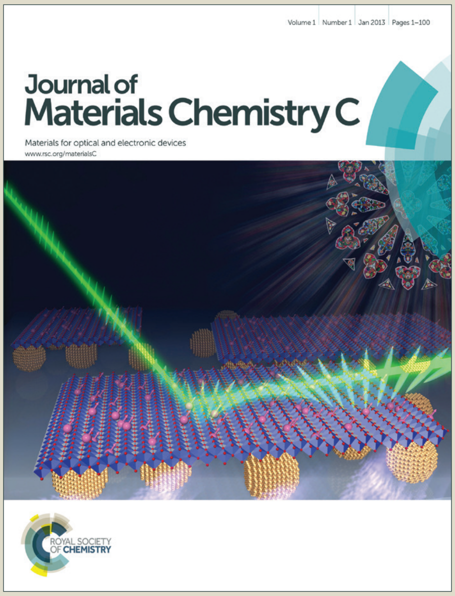

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Improved transparency switching in paraffin-PDMS composites

H. N. Apostoleris,[a] M. Chiesa[a]† and M. Stefancich[a,b]

Composites of paraffin wax and polydimethylsioxane (PDMS) have previously been demonstrated to exhibit thermally-induced switching between a transparent 'on' state and opaque 'off' state; however, the transmittance of the on state in previously investigated composites was found to decrease with increasing wax content, limiting the achievable off-on contrast. We present a new paraffin-PDMS composite formulation that exhibits essentially no decrease in on-state transparency up to a 10% wax concentration. We furthermore identify the underlying cause of the switching as a microstructural transformation from a homogeneous, transparent state to an inhomogeneous, light-scattering state as a result of the phase transition of the paraffin. We then present a model based on the theory of turbid media that allows for the estimation of the scattering power of a composite of known composition based on easily observed parameters, and suggests ways in which the contrast between the on and off states may be further enhanced.

Introduction

Materials with dynamically controllable optical properties are sought after for applications in multiple domains. Controllable transparency has applications in lighting control in buildings with smart and privacy windows [1, 2, 3, 4], or in low-consumption displays [5, 6]. Controllability of the refractive index in materials exhibiting large thermo-optic coefficients has been exploited in components for optical communication. [7, 8] Another recently emerging field of application is the realization of reactive non-mechanical sun tracking for solar energy systems, which may exploit either a change in refractive index or in transparency. [9, 10] Regardless of the application it is of great general interest to develop optical switching materials that are easily fabricated from common, cheap and environmentally benign substances. [11] Recently, composites of paraffin wax and polydimethylsiloxane (PDMS) have been shown to exhibit strong switching between a transparent "on" state, at high temperature, and a light-scattering "off" state at low temperature. [12] Since any light-scattering material acts a reflector for sufficient thickness, an optically thick paraffin-PDMS layer can therefore act not only as a transparent-opaque switch but also as a transparent-reflective switch. One drawback of the composites previously studied has been that they do not become fully transparent but rather exhibit some scattering of light even in the on state, an effect that was shown to increase with increasing wax content. In the present work, we demonstrate experimentally that this need not be the case if the appropriate paraffin is selected. However, we also show by means of new measurements that the reflectance of the off state is, in general, significantly lower than that which could be inferred from previously reported data. [12] We therefore seek a deeper theoretical understanding of the switching mechanism which will guide future research aimed at creating composites with enhanced off-state scattering power, thereby achieve the maximum possible contrast between the transparent and reflecting states.

The fact that paraffin-containing composites undergo transparency switching seems initially unsurprising, since it is well known, probably since long before modern science, that paraffin wax itself undergoes an opaque-to-transparent shift when it melts. Our investigation, crucially, demonstrates that switching in paraffin-PDMS composites, while it occurs based on the same *principle* as the switching in paraffin wax, in fact does not depend on the switching of the paraffin itself and can occur even if a paraffin is used that does not exhibit strong transparency switching, a possibility that will be discussed in the main body of the paper. In this paper we present a theoretical model explaining the switching in the composite material as the result of a first-order phase transition that transforms the microstructure from a homogeneous 'on' state that transmits light to a two-phase 'off' state characterized by small dispersed crystals that scatter light. The optical behavior of the off state can be understood in terms of the well-established theory of light scattering by turbid media, which, combined with the Lorenz-Mie theory of scattering by single small particles, allows relation of the macroscopic optical characteristics of the material to microscopic parameters that can, in principle, be controlled during fabrication.

Experimental

Samples are prepared by blending melted paraffin wax (Sigma Aldrich product 76228) with a manufacturer-provided melting temperature of 44-46°C, with a PDMS elastomer (Sylgard 184) and its standard curing agent in the desired ratios, ranging from 1.25 to 20 wt % paraffin, and cured by heating in an oven at 70°C for 2-3 hrs. Samples used for transmission vs. thickness measurement were made by blending a 1:9 solution of Sigma Aldrich Paraffin 76228 in decane with Sylgard 184 silicone elastomer from Dow Corning and standard Sylgard curing agent in a ratio of 10:10:1. The material was poured into circular molds (3D printed in PLA to the desired shape) on a polycarbonate substrate and baked in an oven at 80°C for 4 hours to evaporate the decane and induce crosslinking. Samples used to measure composition dependence (Figure 1e) were fabricated by an alternate method in which PDMS and melted paraffin are mixed directly, without including a solvent, and cured in 1-cm thick polystyrene cuvettes. The optical properties are quantified by means of transmittance measurements on samples of known thickness and composition, using an Oriel Sol3A solar simulator as the light source. Optical data are collected using an Ocean Optics USB4000 spectrometer connected to an integrating sphere to collect all forward-scattered light, thereby measuring total diffuse transmittance. Transmitted spectra are measured by placing the material over the aperture. Reference spectra are taken with the receiving aperture of the integrating sphere uncovered in the case of the mold-cured samples and with an empty cuvette covering the

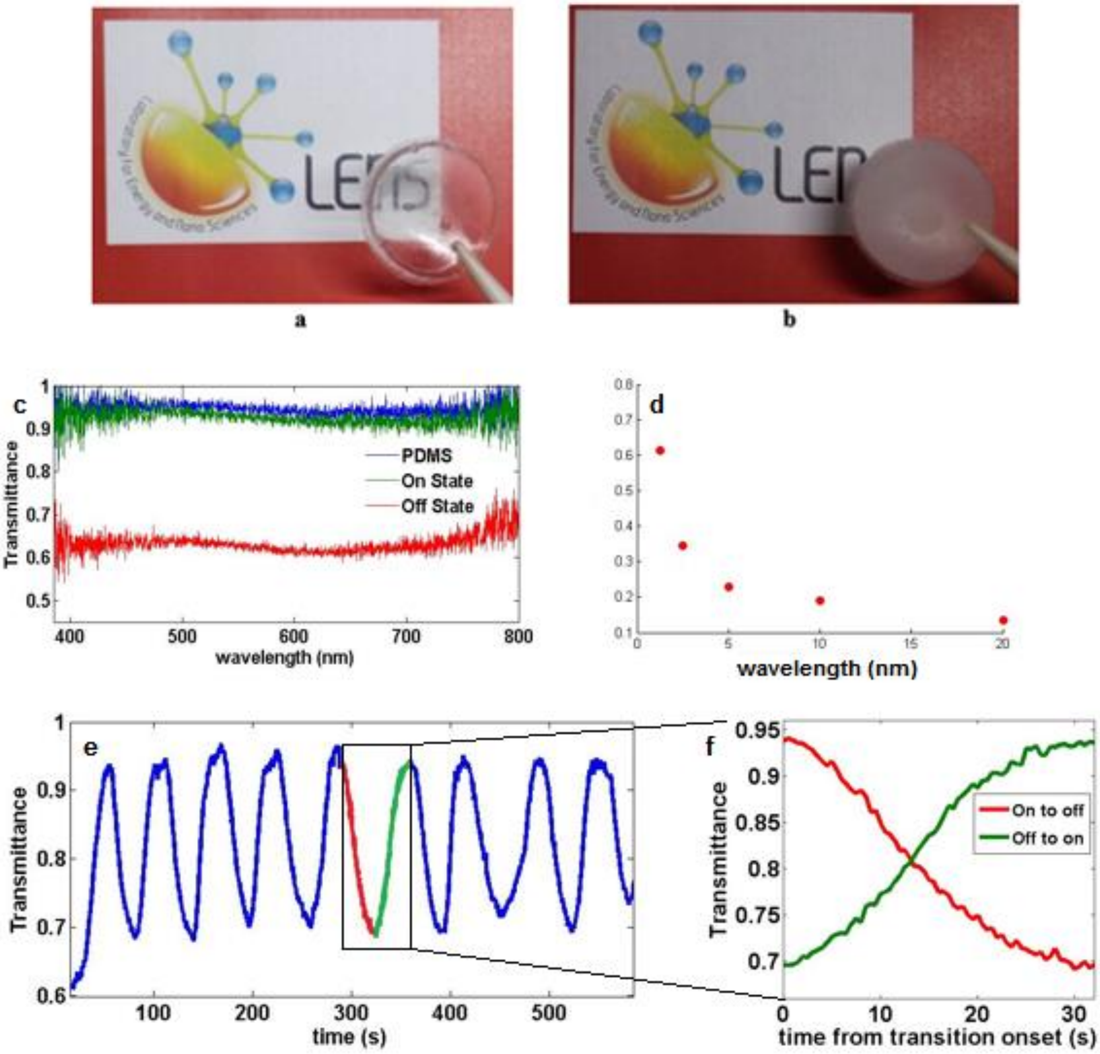

FIG. 1. A sample with 10% wax concentration in the a) ON (hot) and b) OFF (cold). c) Transmittance spectra for the on and off state of a single layer 1.3 ± 0.1 mm thick compared to an equal thickness of PDMS; e) Transmittance vs. wax concentration shows transmittance decreases with increasing wax content but saturates for high wax fractions. e) transmittance of a sample under repeated cycling and cooling demonstrates reversibility of switching; f) switching time for on and off transitions.

aperture in the case of cuvette-cured samples. Overall transmission spectra are determined by the relative change in the transmitted spectrum with respect to the reference spectrum. Data are obtained for transmittance vs. thickness, measured by stacking multiple samples of equal thickness, and for transmittance vs. composition. For comparison to the results of Zhang et al., cuvette-cure 10% wax samples were subjected to an alternate using a UV-Vis spectrometer as described in [12]. Microscope imaging is performed on samples ~200 µm in thickness, viewed in transmission on an optical microscope at 10x and 40x magnification, to investigate the microstructure.

The paraffin refractive index is measured using a prism refractometer constructed in-house to measure the (average) refractive index of melted paraffin. The solid refractive index is determined by means of the measured density change between the solid and liquid state according to the theory described by Guseva and Liefman [13] relating refractive index to density. The measured liquid and solid indices are found to be approximately 1.48 and 1.52, respectively.

Results and Discussion – Experiment

When removed from the oven, the cured samples are transparent, appearing identical to pure PDMS ('On' state, Figure 1a). When cooled below the melting temperature of the wax, they become white and opaque ('off' state, Figure 1b). Total transmittance measurements of a 10% wax sample with thickness 1.3 mm in both states show the transmittance T of the hot and cold states (Figure 1c) as a function of wavelength. It can be seen that the transmittance of the on state is essentially the same as that of pure PDMS; this is in contrast to the result of Zhang et al. which observed a significant decrease in on-state transmittance for wax concentrations as low as 4%. Measurements of samples of varied wax concentration cured in 1 cm thick cuvettes show the dependence of off-state transmittance on composition (Figure 1d). Consistent with previous observation[12], off state transmittance decreases with increasing wax content, but appears to saturate at about $T = 0.1$ at high wax concentrations. Additionally, samples with very high (20%) wax concentration do not become fully transparent but rather translucent when heated. We believe this may be attributed to the saturation of the PDMS matrix with wax. For this reason we have selected 10% as the appropriate wax concentration, as it approaches the maximum off-state reflectance without producing any loss of on-state transmittance. Figure 1c, obtained by cyclically heating and cooling the same sample placed over the aperture of the integrating sphere, shows that the switching between the opaque and transparent state is fully reversible. The transition occurs on a timescale of about 30 s (Figure 1f).

While the samples measured in Fig. 1e have the same thickness as those measured by Zhang et al., the measured transmittance is much higher ($T = 0.2$ for a 5% wax sample in our measurement compared to $T < 0.02$). We attribute this difference to our use of an integrating sphere, which captures all transmitted light in a 180° angular range, as opposed to a UV-vis spectrometer which captures light only from a narrow angular range. Insofar as we are interested in the reflectance R of the material, it is the total diffuse transmittance which is of interest as it obeys the relation $T + R = 1$. This result further suggests that there is significant room to enhance the reflectance of the off state, a task which must be guided by a strong understanding of the underlying mechanisms determining the optical properties.

The result of the UV-Vis transmittance measurements are shown in Figure 2, for the on (Figure 2a) and off (Figure 2b) state. While this technique does not accurately quantify the total transmittance of a scattering sample, which as described previously is the main quantity of interest for our research, it allows a comparison with the work of Zhang et al. [12], who used this technique, to support our claim that the material presented here exceeds the originally reported performance as a transparency switch. Comparison to the corresponding results in reference 12 reveals a large reduction in the off-state transmittance relative the results of [12], without a corresponding reduction in the on-state transmittance. We believe that this difference is due to the selection of a different wax which does not contain any suspended particulates in the melted state, therefore the higher wax concentration does not result in increased scattering in the on state when the wax is melted.

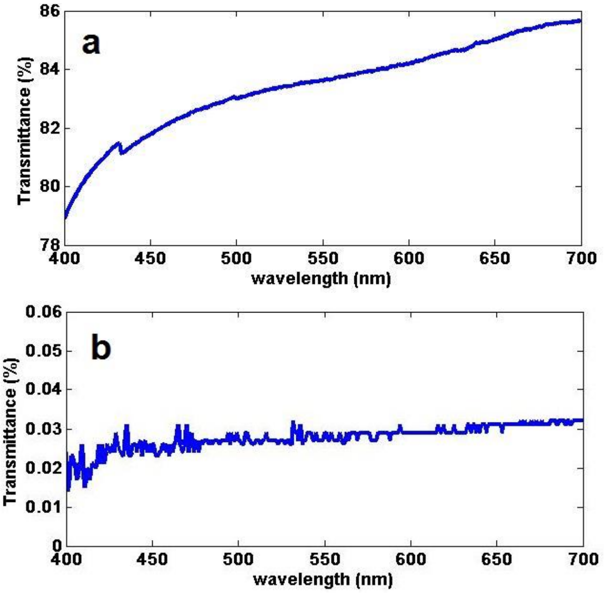

FIG. 2 UV-vis transmittance of a 10% paraffin composite in the on (a) and off (b) states. The sample can be seen to exhibit increased scattering in the off state with respect to the results of ref. 12 with essentially no reduction in transmittance of the on state.

With regard to the physical mechanism of the switching, the microscope imaging presented here identifies precisely the microstructural change accompanying the transition (Figure 3). Time lapse images at 40x magnification of a sample undergoing a hot-to-cold transition (Figure 3a) show the nucleation and growth of small paraffin crystallites distributed throughout the sample as it cools, resulting in a sharp transition between a homogeneous hot state and a heterogeneous cold state. The change in microstructure can then be explained simply as the result of a first-order phase transition in which some portion of the wax crystallizes to form small domains whose size is determined by the crystallization kinetics of the system. The refractive index of each domain is the solid refractive index of the paraffin of which it is composed, which in our material has an index contrast of about +7% with the PDMS, according to our measurements and optical data for PDMS elastomers reported in the literature. [14] The change in optical properties is directly attributed to the change in microstructure: the formation of crystals transforms the initially homogeneous sample into a turbid material, in which light it scattered by multiple interactions with small dispersed particles whose refractive index differs from that of the medium. The size of the crystals can vary somewhat from sample to sample, as seen in figure 3b (10% wax concentration, crystal diameter ~5µm) and figure 3c (2%, ~2 µm). Whether the crystal size varies systematically with the composition or whether it is primarily determined by other factors remains an open question. The behavior is somewhat different in the sample with 20% wax concentration, in which we observe bubble-like structures, which we believe to be 'pockets' of wax, that are present (albeit melted) even when the sample is heated above the melting point of the wax (2d). The presence of permanent wax pockets, which have a significant refractive index contrast with PDMS in both their solid and liquid state, means that full transparency is never achieved, since there is scattering from these structures in both states. Similarly, if a wax is used that has some suspended particulates in the melted state, this would result in the loss of transparency of the on state which was observed in previous work [12], and successfully eliminated here.

Our results demonstrate the importance of distinguishing between the opaque-to-transparent shift that occurs in melting wax and the shift that occurs in the paraffin-PDMS composite. Transparency switching in wax depends on the fact that wax itself is a composite of different hydrocarbons (alkanes) with slightly different refractive indices. [13] When a wax solidifies, the different components separate into small domains of pure alkanes. Due to the refractive index contrast *between domains*, light is scattered by multiple refractions at the interfaces between the domains. In a paraffin-elastomer composite, that scattering depends not on the index contrast between the components of the paraffin but rather on the contrast between the paraffin crystals and the elastomer (in this case, $n_{PDMS} \approx 1.43$ [14]). This raises the possibility – which we have, in fact, realized in our samples -- of an appropriately engineered composite having a scattering power substantially greater than that of the employed paraffin itself.

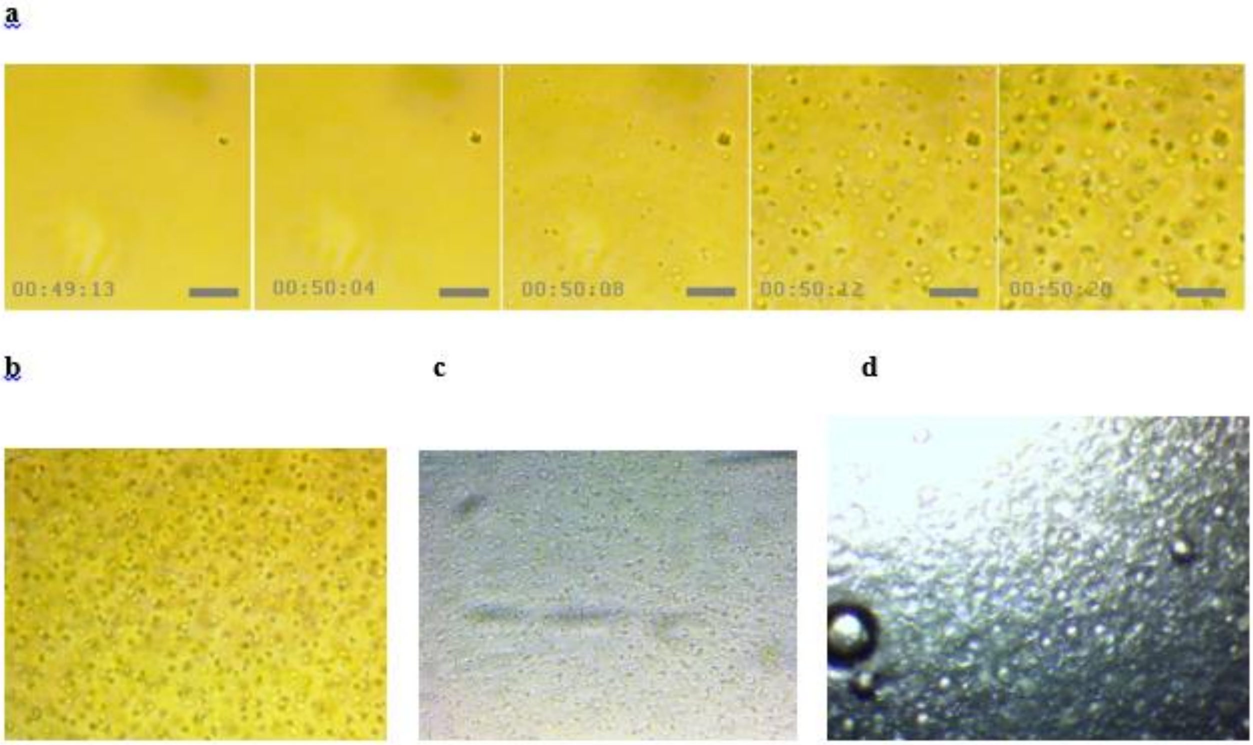

FIG. 3. a) Time lapse imaging of a sample with 10% wax concentration undergoing hot-to-cold transition reveals the formation of dispersed crystals that scatter light. Scale bar = 20 µm. b) 10% sample in off state. c) 2% wax. Horizontal edge = 250 µm. Images taken at 40x magnification with objective NA=0.65 d) 20% sample in the on state. Unlike the samples of lower wax content, the structure of this sample is not homogeneous at high temperature, with the results that this sample does not become fully transparent when heated. Horizontal edge = 1 mm. Image taken at 10x magnification with objective NA = 0.25.

Theory and Simulation

The understanding of the off state as a turbid medium composed of roughly spherical scattering centers of similar size allows us to construct a theoretical framework that quantitatively relates the optical properties to microscopic parameters. In our theoretical development we address two main issues. The first is to verify the explanation of the transparency switching offered in the previous section by confirming that the macroscopic optical properties can in fact be predicted by a model based on the established theory of light scattering by small particles. The second issue is to determine how modifications of the microstructure affect the overall optical properties. This question can be investigated theoretically with a few straightforward calculations and numerical modeling based on light scattering theory, and the results again compared to experimental data.

The process of light scattering by turbid media has been studied rigorously for nearly a century [15, 16, 17, 18] and many theoretical frameworks exist for the description of this phenomenon. The samples used for transmittance measurements, whose results are compared to the predictions of the model, are optically thick. Attempts to conduct experimental measurement on thin samples, whose thickness is on the order of the single-scattering length, were unsuccessful due to difficulties in fabricating sufficiently homogeneous thin samples. Our theoretical model must therefore represent the case in which a single photon undergoes many scattering interactions as it passes through a sample (the multiple-scattering case). This problem is notoriously difficult to solve in general, [15] and we therefore resort to numerical methods, using a Monte Carlo ray-tracing method often employed to model light scattering in biological tissue [16]. The simulation depends upon the bulk scattering coefficient *s*, and the scattering phase function or an approximation thereof. These scattering parameters, in turn, are determined by applying the superposition principle [17] and the scattering model of Henyey and Greenstein [18] to the single-particle scattering parameters calculated from the Lorenz-Mie theory. [19] The inputs for these calculations are the particle size, concentration and refractive index that can be determined from our experimental measurements.

A. **Calculating Lorenz-Mie parameters.** Many tools exist for the determination of Mie scattering functions; in our work we use the Mie Scattering Calculator developed by Scott Prahl (retrieved from http://omlc.org/calc/mie_calc.html, 29-10-2014). The required parameters are the diameter of the scattering particle, the wavelength of the incident light and the refractive index of the particle and the medium. From these we calculate the scattering cross section C_s and the phase function $\varphi(\theta)$, giving the strength of the scattering interaction and the angular profile of the scattered light, respectively. The phase function is transferred to the Henyey-Greenstein model in an aggregated form by the *anisotropy factor g*, defined as the expected value of cos θ on $\varphi(\theta)$. For our calculations, the average crystal size is estimated by inspection of the microscope images. In figure 2b (10% wax concentration), the typical crystal diameter is about 5 µm; in figure 2c, (2%), the average diameter is 2 µm. The particle refractive index is independently measured at 1.52, (see experimental section), and the PDMS matrix index at 1.43, the refractive index of PDMS at 546 nm [14]. The calculations for the sample shown in Figure 2c (10% wax content) give a C_s = 57 µm^2 and g = 0.9864.

B. **Calculation of the scattering coefficient.** The scattering coefficient *s*, representing the expected number of scattering events per unit path length, is determined from the Mie cross sections via the superposition principle as described by van de Hulste, by which

$$s = \sum_{i=1}^{N} C_{S_i} \tag{1}$$

where *N* is the number of scattering centers per unit volume. Since the crystals appear to be comparable in size throughout a given sample, we treat them as having essentially the same cross section C_S, in which case the above formula reduces to

$$s = NC_S \tag{2}$$

where *N* is the number density of crystals in a given sample. We can estimate this by counting the number of crystals visible in an image and taking this as the number contained within a focal volume defined as the image area multiplied by the focal depth. In the case of the M=40x, NA=0.65 objective used to obtain these images, the focal depth d_{focus} will be about 2 µm. Since this is small compared to the crystal size, we must consider that the observed features only *intersect* the focal volume rather than being completely contained within it. Therefore we are actually exploring a depth of a bit less than $d_{focus} + 2d_{\text{particle}}$. We take this depth to be 10 µm. Counting 507 crystals in a volume of 250 x 190 x 10 µm, we obtain $N = 1.0 \times 10^6$ mm^{-3}, corresponding to an estimated scattering coefficient of 57 mm^{-3} according to Equation 2.

C. **Monte Carlo simulations.** The Monte Carlo ray tracing simulations are performed using a built-in algorithm in a commercial ray tracing package (TracePro). A thick layer of a material is assigned bulk scattering properties according to the discussed Henyey-Greenstein model with the previously obtained host refractive index, scattering coefficient and anisotropy factor. The scattering algorithm propagates a ray by a random distance determined by the scattering coefficient before it undergoes a scattering event. The deviation angle after scattering is randomly assigned from the phase function and the process is repeated until the ray has exited the medium. Since the medium has an assigned refractive index, all geometrical optics phenomena, including Fresnel reflection and TIR at the interfaces, are included.

Ultimately, the model provides the thickness-dependent total transmission that can be directly compared with our experimental results. For comparison to the experimentally obtained transmittance vs thickness data, simulation data are obtained by creating a 'stack' of bulk scattering samples, defined according to the above discussed model, in Tracepro. A small gap in the simulation is included between each 1.3 mm thick disk to account for the interfaces between experimental samples which is assumed to contain an air gap. Figure 4 displays both the simulation results and experimental data.

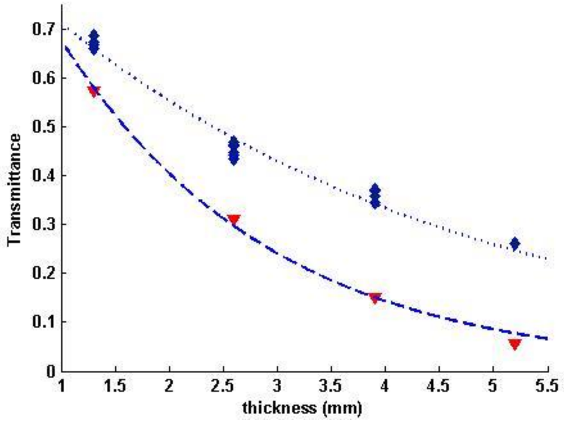

FIG. 4. Experimental data (♦) for transmission vs thickness measurements of stacked samples (▼) with Monte Carlo modelling results for the estimated scattering parameters *g=0.9867, s* = 57 mm^{-1}

Results and Discussion -- Simulation

Considering the transmittance of a single layer (1.3 mm), we find that the simulation agrees with the data within 20%. The experimental and simulated curves can be seen to follow a similar trend that can be adequately modelled as an exponential, although the physical interpretation of the exponents is at present unclear.

The model makes several important predictions regarding the relationship between microstructure and optical properties. First, it predicts that the scattering power should vary inversely with crystal diameter *d*, provided that the total crystal volume fraction remains the same. This can be demonstrated by noting that the scattering cross section $C_S = Q_S C_g$ where C_g is the geometric cross section $\pi(d/2)^2$ and Q the scattering efficiency C_S/C_g. Then we have for the scattering coefficient [21]

$$s = Q(d)C_gN = \frac{Q(d)C_gf}{v} = \frac{3Q(d)f}{4d}$$

where v is the particle volume $4\,\pi(d/2)^2/3$. We note that Q is a function of d, but this effect can be ignore either in the case where we investigate a small range of d, in which case Q is slowly varying over the observed range, or a large range, in which case Q oscillates about the geometric limit $Q = 2$. Our observations show that crystal size can vary significantly from sample to sample (Figure 2b-c), so controlling this parameter will be critical to enhancing the contrast between the two states of the material.

Furthermore, with the guidance of the model, different materials can be selected to enhance the scattering behavior in the off state. The simplest approach would be to simply use different paraffin with a higher average index, or use fractional distillation to select the desired pure alkanes.

Conclusions

We have herein described a new formulation of the transparency-switching paraffin-PDMS composite which exhibits a fully-transparent on state for wax concentrations as high as 10%. This is an enhancement over the variant presented by Zhang et al, in which on-state transmittance was strongly dependent on wax content. Our composite also exhibits enhanced off-state scattering power relative to previously reported formulations. Additional optical measurements reveal that the material is still far from ideal as a transparent-reflective switch, however, we have established that considerable further improvements can be made to enhance the reflectance of the off state. To guide or work in this area we have proposed a model based purely on scattering physics that explains qualitatively, and to a reasonable extent quantitatively, the scattering of light by paraffin-PDMS composites. We find that the optical behavior can be fully explained by the formation of wax crystals in the PDMS matrix which can be melted and recrystallized reversibly under thermal stimulus, creating a turbid medium that scatters light according to well-established theory. The dependence of the switching on a simple first-order phase transition between this turbid state and the homogeneous state in which the wax is melted means explains the reversibility of the switching between the two states.

Further investigation of alternative recipes and fabrication techniques to achieve these modifications is likely to be a fruitful topic for future research. Due to its simplicity of fabrication and operation, the low cost and environment benignity of the components, we expect this material, with further research, to become a convenient and versatile candidate for a variety of applications where controllable transparent-reflective switching behavior is required.

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Notes and references

[a] Department of Materials Science and Engineering, Masdar Institute, Abu Dhabi, 54224, UAE

[b] National Research Council, IMEM, Parco Area delle Scienze 37/A - 43124 Parma, Italy

†Author to whom correspondence should be addressed: mchiesa@masdar.ac.ae

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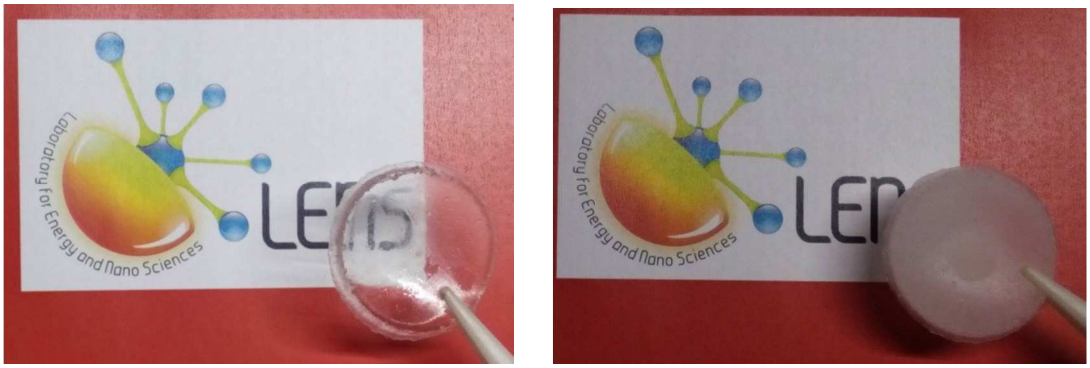

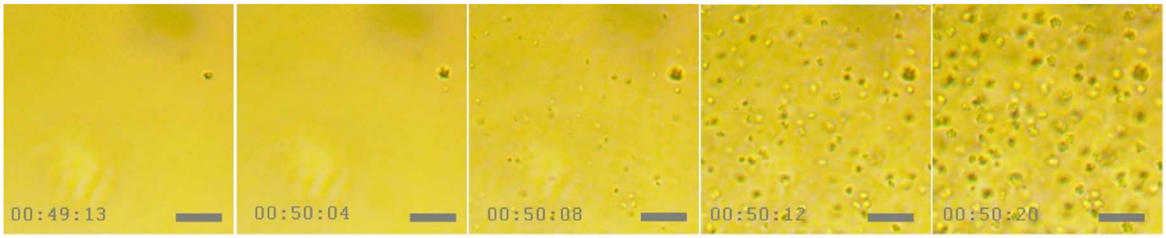

We present a composite of paraffin wax and polydimethylsiloxane (PDMS) with improved thermally-activates transparent-opaque switching. We discuss the potential use of the material as a transparent-reflective switch and present a model based on the theory of light scattering by turbid media that explains the switching behavior in terms of a microstructural change resulting from the phase transition of the wax, and propose ways of further enhancing the switching behavior.