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Temperature and saturation dependence in the vapor sensing of butterfly wing scales

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A B S T R A C T

The sensing of gasses/vapors in the ambient air is the focus of attention due to the need to monitor our everyday environment. Photonic crystals are sensing materials of the future because of their strong light-manipulating properties. Natural photonic structures are well-suited materials for testing detection principles because they are significantly cheaper than artificial photonic structures and are available in larger sizes. Additionally, natural photonic structures may provide new ideas for developing novel artificial photonic nanoarchitectures with improved properties. In the present paper, we discuss the effects arising from the sensor temperature and the vapor concentration in air during measurements with a photonic crystal-type optical gas sensor. Our results shed light on the sources of discrepancy between simulated and experimental sensing behaviors of photonic crystal-type structures. Through capillary condensation, the vapors will condensate to a liquid state inside the nanocavities. Due to the temperature and radius of curvature dependence of capillary condensation, the measured signals are affected by the sensor temperature as well as by the presence of a nanocavity size distribution. The sensing materials used are natural photonic nanoarchitectures present in the wing scales of blue butterflies.

1. Introduction

Photonic crystals are periodic dielectric nanocomposites capable of interacting with electromagnetic (EM) radiation in a spectrally selective manner [1]. These materials are constructed from two non-absorbing media that possess different refractive indices, and the sizes of the building elements are comparable with the wavelength of the EM radiation for which propagation is forbidden in the nanoarchitecture. Certain frequency ranges cannot propagate inside the structure; they are fully reflected from the surface. This wavelength range is called the photonic band gap (PBG), and the reflected color is called structural color because it is based on the properties of the nanocomposite.

A particularly rich variety of such structural colors can be found in the insect world; many butterflies and beetles show exciting colorations originating from photonic nanoarchitectures [2–4]. In the case of butterflies, the photonic nanoarchitecture responsible for the color is usually located in the wing scales. In the scales, the nanocomposite is constituted mainly from chitin and air, and the nanocomposite may contain a vast variety of different nanoarchitectures. These nanoarchitectures result in a rich variety of colorations that can be used for various communication purposes. Butterflies may use their structural color for sexual communication [5], for cryptic behavior [6] and for warning potential predators [7]. Because the color influences the survival and reproduction chances, it is governed by strong evolutionary pressures developed over millions of years. One can attempt to use this "natural" wisdom that has accumulated over the course of many millennia for potential human applications.

In common gas/vapor sensors, nanostructured materials are often used to increase the specific surface area, but the sensing mechanism is usually based on electric resistance measurements on oxide layers [8]. However, there is a special group among chemical sensors based on artificial photonic crystal structures in which changes in the dielectric constant are detected by the PBG shift effect [9]. As discussed above, natural and bioinspired nanostructures could act as efficient light-tailoring devices. Beyond altering the propagation of light, nanostructures may also change the surface wetting properties [10]. The color change of the butterfly wing as a valuable source of photonic crystal-based sensors was shown for the first time in *Morpho*-type structures [11]. Later, we showed that in the case of Polyommatini butterfly-based sensors, different spectral signals appear for different vapors; therefore, they can be used as a chemical-selective sensor material [12]. Recently, we investigated the relation between the color and the PBG-type structures of nine closely related lycaenid butterfly species, all of which display a structural blue color in their dorsal wing

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surfaces [13]. The Hungarian Polyommatini fauna contains more than 30 species; therefore, we have a ready-made good-quality sensor material, which is a considerable advantage over artificial structures in which the reproducible construction of appropriate macroscopic samples is a slow and expensive process. Moreover, the sample preparation is simple because it only involves the cutting of a wing piece to the desired size. Additionally, these nanoarchitectures are produced in an environmentally friendly manner; if the butterflies are bred under laboratory conditions, the use of their wings does not harm the natural richness of the environment.

In our earlier work [14], we compared the optical sensing properties of the scale-covered dorsal wing surfaces of Polyommatini butterfly species using optical spectrophotometry, and the species with the highest relative spectral change were selected. Furthermore, we investigated the temperature dependence of the relative spectral change and showed that lower temperatures increase the shift of the spectral position, which enhances the spectral response signal [15]. The explanation of the spectral change mechanism when butterfly wings colored by PBG-type materials are exposed to air with vapors of different volatiles concerns the capillary condensation of the different vapors inside the chitin-air nanocomposite [15]. The distribution of pores with small curvatures in the nanostructure (Fig. 1.a) promotes the formation of the ink-bottle effect [16], which results in hysteresis in the condensation of the vapors, whereas lower temperatures enhance the magnitude of the spectral changes. The condensation inside the pepper-pot structure is similar to the condensation in a colloid silica-sphere multilayer [17]. In this paper, a red shift of the reflectance peak similar to that of butterfly wings was reported.

In real sensors operating in an ambient air environment, the changes in temperature (the diurnal or yearly cycle) cannot be completely separated from the changes in vapor concentration. Therefore, it is worthwhile to investigate the possible cross effects that may arise from the temperature and concentration changes, such as:

- The constant concentration gas flow with changing sample temperature
- Testing the effect of the continuously changing vapor concentration on the reflectance measured at a constant temperature

2. Experimental

The Polyommatini and Morpho specimens were provided by the curated collection of the Hungarian Natural History Museum.

We worked with butterflies collected from 1900 to the present. The wing is composed of a dry material made of chitin with a complex structure. The photonic crystal-type structure located in the wing scale defines the wing color, and this is a constant species-characteristic value: identical reflectance spectra can be measured from a wing that is 100 years old and from a modern wing. When used as a gas detector, a bottleneck can be the contact with chemicals that damage the chitinous nanostructure. However, all the specimens we have currently used fully recovered after exposure to vapors of various volatiles.

To obtain insight into the nanostructures, detailed scanning (SEM) and cross-section transmission (TEM) electron microscopy images were taken. To avoid charge buildup during the SEM observations, 15 nm of sputtered gold was deposited onto butterfly wings mounted on conducting carbon tape. For TEM sample preparation, wing pieces measuring a few millimeters were embedded in a resin, and 70 nm thick sections were cut using an ultramicrotome. These slices were placed on a copper TEM grid. The vapor-sensing experiments were conducted using computer-controlled gas/vapor-mixing equipment and an airproof gas cell. The mixing equipment consists of two digital mass flow controllers (Aalborg) that provide a constant gas flow output of 1000 ml/min. The vapor concentration was set by switching the flow controllers to allow synthetic air (Messer: 80% N_2, 20% O_2, others <20 ppm) and saturated volatile vapor pass in the required ratio. The prepared vapor mixture was placed into the aluminum, airproof gas cell with the butterfly wing. The cell has a quartz glass-slide cover to allow UV transmission.

Optical spectroscopy was performed using an Avantes HS 1024*122TEC fiber optic spectrophotometer. A UV/Vis/NIR light source was used to illuminate the sample. The incident light was perpendicular to the sample, and the reflected light was collected by an off-normal optical fiber that was oriented to an angle yielding a maximum signal reflected from the butterfly wing. To analyze the temporal-spectral dataset, a MATLAB code was implemented that allowed us to create 3D surfaces and colored maps. In addition, using the MATLAB code, we were able to create 2D line-cuts from the 3D datasets that describe the temporal evolution of the reflectance signal at a given wavelength.

To change the sample temperature, a miniature Peltier element was placed under the wing inside the gas cell. The current of the Peltier element was controlled by a NI DAC card and a Labview code.

Based on our earlier investigations [14], the *Polyommatus icarus* butterfly species was selected to examine the selectivity, thermal dependence and sensitivity as a gas-sensor material. We also examined *Morpho aega* wings because this species has a narrow and intense reflectance maximum and because their photonic crystal-type nanostructure is well described [18].

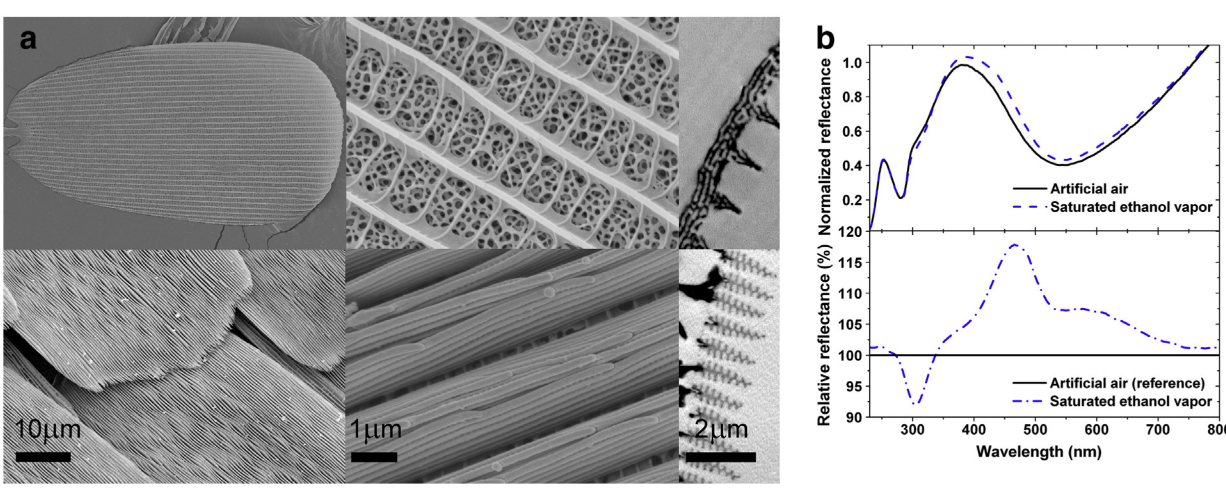

Fig. 1. a) Scanning and transmission electron micrographs of *P. icarus* (top) and *M. aega* (bottom) wing scales. b) Transforming the shift by recording the relative reflectance using the wing in artificial air as a reference (in (b) the lower panel).

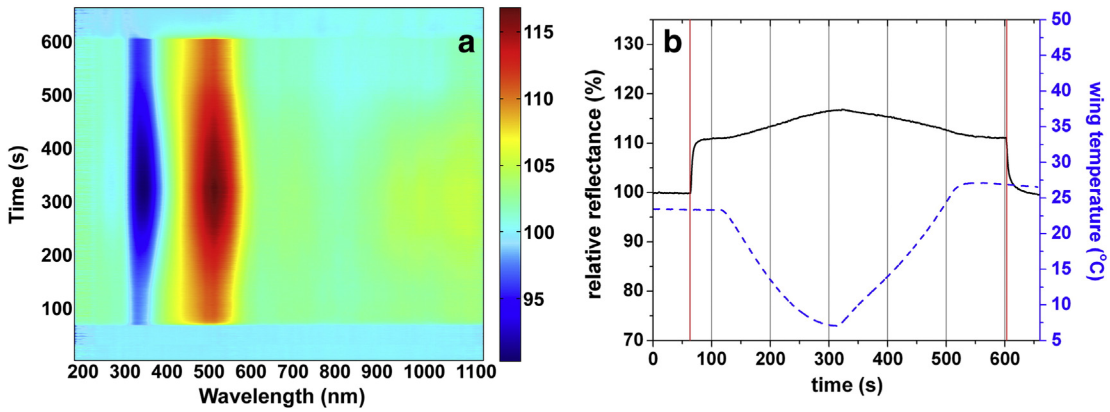

Fig. 2. *P. icarus* spectral response for 20% ethanol. a) Time-dependent, color-coded 2D spectral map of the signal. b) Line-cut from the map at 505 nm and the corresponding temperature profile (vertical red lines help to guide the eye at the on/off switching of vapor). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

To show the changes caused by different vapor concentrations, the optical reflectance spectra of the samples were first recorded while the gas-sensing cell was purged with synthetic air. In this case, the Avantes white diffuse reference reflector was used as a reference, and the characteristic blue peak in the reflectance spectrum can be observed. Using the vapor mixture, the variations from the original reflectance spectrum can be observed (Fig. 1.b). These differences are small compared to the original signal; therefore, in the subsequent vapor-sensing experiments, we used the original reflectance spectrum (measured in synthetic air) as a reference rather than the white diffuse standard. This means that during the gas-sensing experiments, the variations from the original color were measured and the observed shapes of the variations are dependent on the vapor concentration and type [12]. Because the temporal evolution describes the gas-sensing process that is time- and temperature-dependent [15], it is adequate to use the presentation of reflectance in 3D (or a color-coded map in which the relative reflectance variations are color coded (Fig. 2.a)) complemented with the wing temperature data.

In the first experiment, we tested the constant concentration of the gas flow with the changing sample temperature. Initially, synthetic air was flowed through the sample-holder cell; we then switched to 20% ethanol in air (Fig. 2). After a rapid (few seconds) appearance of the reflectance change, the signal tended to be saturated. At that moment, we began to continuously cool the wing, followed by heating back to the initial room temperature. When the temperature and the signal stabilized, we finished the experiment by switching back to flowing pure synthetic air through the measurement cell.

When we tested the effect of the continuously changing vapor concentration on the reflectance measured at a constant temperature, a 0%–100%–0% path was navigated (through 40 equal steps) at 23° (Fig. 6) and at 16° (Fig. 7).

3. Results and discussion

Shining, colorful butterfly wings are the result of various scale structures [4]. Wings with open nanostructures are efficient for gas-sensing applications. The sponge-like, pepper-pot, quasi-ordered [19] photonic crystal structure of the *P. icarus* and the *M. aega* scales with multilayer parallel lamellar ridges is sufficiently open. (Fig. 1.a). As in the case of the abovementioned two species (living in very different geographical regions), different types of nanoarchitectures may generate colors that appear more or less similar in hue. However, within one structural family, such as the pepper-pot type, the structural variations are associated with clear differences in color, as the colors are used by butterflies living in the same habitat for sexual communication [13].

In the temperature profile and in a line-cut from the color map (Fig. 2.a) measured for *P. icarus* at 505 nm (the highest signal peak), one can see (Fig. 2.b) that the signal increase and decrease correlates with the temperature change. This result is in good agreement with our previous work [15], in which an increase in sensitivity with a decrease in temperature was shown. The higher spectral response is a consequence of more extended vapor condensation into the scale nanoarchitecture pores at lower temperatures. Taken together, these findings clearly show that the sensing mechanism of butterfly wing vapor sensors is based on the phenomenon of

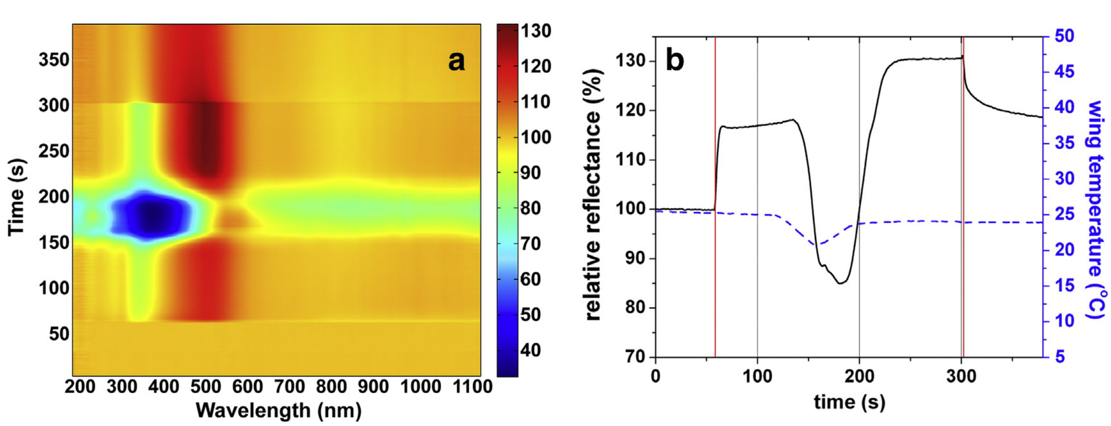

Fig. 3. *P. icarus* spectral response for 100% ethanol. a) Time-dependent, color-coded 2D spectral map of the signal. b) Line-cut from the map at 505 nm and the corresponding temperature profile (vertical red lines help to guide the eye at the on/off switching of vapor). Note in (a) the offset in the time of the minimum (blue) and maximum (red). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

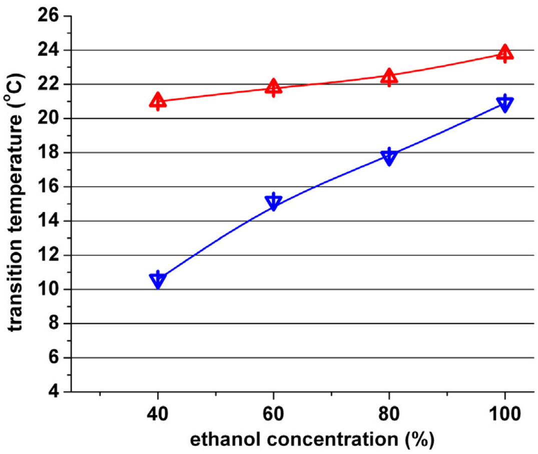

Fig. 4. Transition temperatures of spectral breakdown (blue down arrows) and recovery (red up arrows) temperatures observed at different ethanol vapor concentrations for *P. icarus* wings. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

capillary condensation and explains why the modeling work using vapors to fill the cavities of the photonic nanoarchitecture was unsuccessful in reproducing the experimentally observed sensitivities [20]. The refractive index differences of the various volatiles in the liquid state are significantly larger than the corresponding differences in the vapor state. As the phenomenon of capillary condensation is dependent on temperature and the radius of curvature [16], at a given temperature, the available nanopores will be filled with condensate up to a certain level. The increase in the number of filled nanopores will produce the increase in the detected signal.

The experiment described above was conducted with similar results at different vapor concentrations. Because the superior limit is an interesting case, the measurement with 100% ethanol is shown in Fig. 3. The first difference is in the signal amplitude and slope when switching from air to air + ethanol. Then, with a temperature decrease of a few degrees, a rapid and abrupt signal breakdown is observed. At that moment, we did not continue the cooling. A time delay was observed for the spectral signal to recover after the temperature returned to the initial value. This result indicates the presence of a strong hysteresis. With respect to these breakdown and recovery temperatures, we observed a correlation with the applied concentrations. As shown in Fig. 4, for 20%, there is no breakdown, and for 40 to 100%, we observed a linear dependence. Despite the very different scale nanostructures, the blue reflector of the *M. aega* wing with an identical experimental protocol provides very similar results. For 20% ethanol (Fig. 5.a), the nearly symmetrical shape of the maximum is similar to the one measurement for *P. icarus* (Fig. 2.a). At higher concentrations, the breakdown appears, and at 100% (Fig. 5.b), the initial reflectance returns only after the purging of the cell with air (red arrow). The breakdown phenomenon and the time delay in the recovery of the signal are the consequence of the complete filling of all the nanopores with liquid. In this state, the photonic band gap may be strongly altered to completely vanish. The recovery is delayed because of the so-called ink-bottle effect [16], meaning that the larger filled pores connected to the open space by narrower necks can be emptied only slowly.

A concentration-dependent difference worth mentioning when comparing the 100% experiment with the 20% case (Fig. 2) for *P. icarus* is that while for the 20% vapor mixture, the negative peak (blue color on the map) develops in parallel with the positive peak (red color on the map), in the case with 100% ethanol, the blue and red color maxima are offset in time. One can also observe the same offset for *M. aega*, but in this case, the time sequence of the offset is reversed and the red maximum develops first followed by the blue minimum. This difference may be attributed to the structural differences of the two nanoarchitectures (see Fig. 1); a more detailed investigation is beyond the scope of the present paper.

Concerning the cross effects arising from variations in concentration as a function of time, at room temperature (23 °C), one can observe a signal increase with the concentration (Fig. 6.a). Note the asymmetry in the curvature of increase and decrease (line-cut at Fig. 6.b), which indicates the saturating effect of high vapor concentrations. The small pit at the top shows the emergence and almost immediate termination of a signal breakdown. When the sample temperature was maintained at 16 °C (Fig. 7) at a certain concentration, strong breakdown and recovery were observed, similar to that observed in the variable temperature experiments with a 100% ethanol concentration (Fig. 5.b). However, the recovered state in this case is not identical to the initial one. These characteristic and outstanding changes in the reflectance occur when the vapors condensate into the nanoarchitecture, completely filling it; following this, the liquid completely covers the whole wing piece. We previously described a similar phenomenon when the humidity from the normal ambient air condensates in moth and butterfly wings, changing their color [21]. On the one hand, this color change is a result of a complete filling of the air holes in the photonic structure, and in this way, the refractive index of air is replaced by the refractive index of the liquid. This alone produces a complete hue change in the reflected color (from blue for air filling to green for water or ethanol filling). On the other hand, it is known from SEM studies and is also observable with a higher magnification light microscope that the wing scales are connected to the wing membrane at an angle of approximately 10° as measured from the plane of the wing membrane.

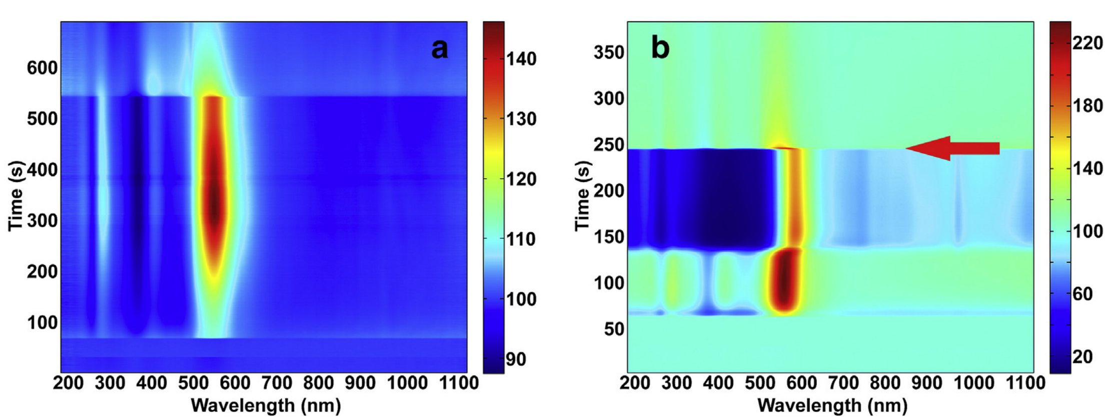

Fig. 5. *M. aega* time-dependent, color-coded 2D spectral response maps for a) 20% ethanol b) 100% ethanol. Note in (a) the parallel development of minimum (blue) and maximum (red) and their offset in (b). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

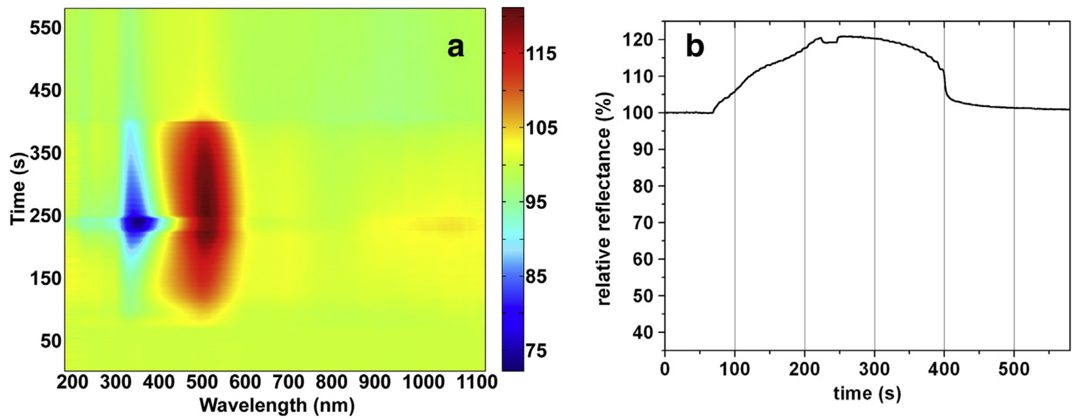

Fig. 6. *P. icarus* time-dependent, color-coded 2D spectral response map at 23° for ethanol. The concentration is increasing from 0 to 100% followed by decreasing to 0. a) Color coded map of signal; b) Line-cut from the map at 505 nm.

When completely submerged in a liquid, the surface tension makes the scales adopt a position parallel with the membrane. In this way, the well-defined light illumination and collection fiber angle for the maximum signal in the reflectance setup are now invalid. The observable transition from the normal state to the breakdown state and back is associated with the progression of the liquid/atmosphere interface from one scale to another. In the case of *M. aega,* which was observed under an optical microscope (Fig. 8), one can clearly observe blue (synthetic air filling), blue and green with some scales half-filled (just the transition), and dark green (all scales filled and covered by liquid). As the green state is darker, a lower reflectance will appear in the spectral measurements. A similar decrease in spectral intensity and a maximum red shift due to liquid infiltration were previously observed in beetles [22,23].

The presented color-changing phenomena also occur if a drop of ethanol is placed on the wing surface, and the liquid directly penetrates into the photonic crystal structure [18] and covers the whole nanoarchitecture, but no such penetration can occur with water as the scale surface is superhydrophobic. The top surface of the scales facing in a direction opposite to the wing plane with the intricately structured nanoarchitecture (with protrusions of a few 10 or 100 nm distance) acts as a superhydrophobic surface, preventing water droplets from wetting the wing. In our experiments, when we used liquid water droplets—even when we sprayed water in very fine droplets—the spherical droplets rolled off the wing. However the water vapors (or other liquids) could easily enter the 3D structure and condensate inside the nanocavities of the PBG material. After condensation, the liquid can practically fill the entire scale, including the space between the scales and wing membrane.

4. Conclusions

We demonstrated gas-sensing measurements for *P. icarus* and *M. aega* butterfly wing scales with a proportional signal intensity dependence on concentration, but this proportionality is corrupted at a certain concentration-dependent threshold sample temperature at which the entire volume of the nanoarchitecture is completely filled with the liquid phase of the vapors to be measured. The observed color breakdown is attributed to the temperature dependence of the capillary condensation process [15]. After the nanopores of the PBG material are completely filled with condensate, the further condensation process on the already present liquid surface will result in the complete submersion of the scales in liquid (Fig. 8), leading to the complete loss of color signal in the spectral range used to evaluate the vapor concentration. A further factor contributing to signal loss is the change due to the surface tension effect between the scales and wing plane if the scale layer is completely covered by liquid. Despite the fact that the abovementioned filling is a reversible process, the complete liquid filling that results in the color breakdown hinders the correct interpretation of the spectral response, and further valid data could be obtained only after the structure recovered from complete filling. From the perspective of sensor applications, this clearly raises the question of sensor temperature control or at least the precise measurement of the sensor temperature to avoid erroneous data.

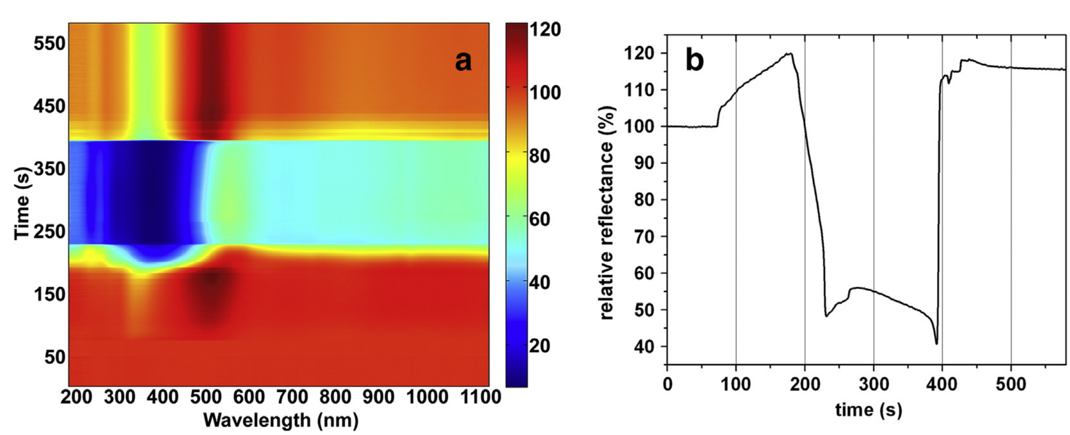

Fig. 7. *P. icarus* time-dependent 2D spectral response map at 16° for ethanol. The concentration is increasing from 0 to 100% followed by decreasing to 0. a) Map of signal; b) Line-cut from the map at 505 nm.

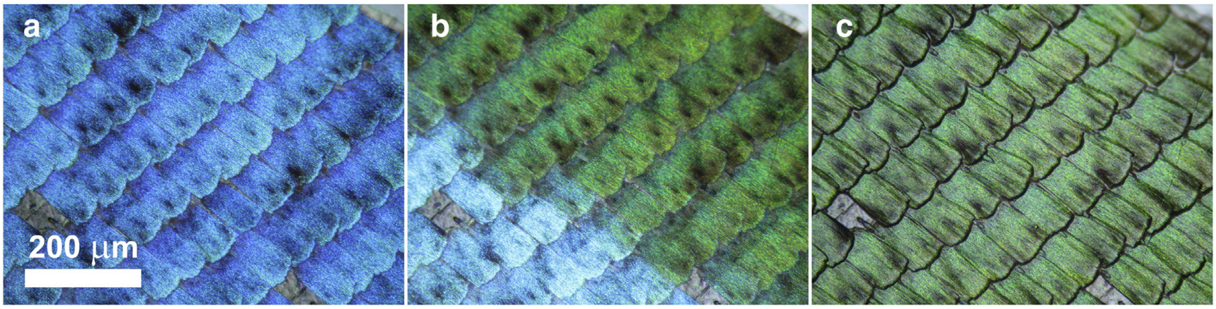

Fig. 8. *Morpho aega* wing scales; optical microscope image of the effect of ethanol vapors. Left: in pure synthetic air; middle: just at the transition of the reflectance spectra (Lower-left corner: air + ethanol vapors, right-upper corner: liquid ethanol); right: the completely filled (covered by ethanol) state.

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