



Blue-emission tuning of perovskite light-emitting diodes with a simple TPBi surface treatment

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Abstract

We demonstrate a simple approach for blue-emission tuning of quasi-2D perovskite light-emitting diodes through a surface treatment of 2,2',2''-(1,3,5-benzinetriyl)tris(1-phenyl-1*H*-benzimidazole) (TPBi). By increasing the TPBi concentration, we achieved tunable electroluminescence of the perovskite layer with wavelength shifted from the blue-green (506 nm) to blue (481 nm) regions of the visible spectrum. Fourier-transform infrared spectroscopy, scanning electron microscopy, and UV-Vis absorption spectroscopy were conducted to study the morphological and optoelectronic properties of the films. Our results suggest that the TPBi molecules accumulated on the surface and grain boundaries of the perovskite layer changed the perovskite electronic structure causing the observed blue shifts.

Introduction

In the last years, metal halide perovskites (MHPs) have become a very promising candidate for light-emitting diode (LED) applications owing to the unprecedented improvement of the device performance. The external quantum efficiencies (EQEs) of the red and green perovskite light-emitting diodes (PeLEDs) have both reached a considerable value of more than 20%,^[1,2] catching up the state-of-the-art organic LEDs.^[3] However, compared to their red and green counterparts, the performance of the blue PeLEDs still lags behind, so far exhibiting a record EQE of only ~10%.^[4] This undoubtedly impedes the further implementation of PeLEDs in white light and full-color displays as blue is one of the three needed basic colors.^[5] Further development of blue PeLEDs is, therefore, a highly active field of research.^[6,7]

In fact, achieving blue emission in LEDs has always been a challenge, owing to the difficulties of producing stable wide-bandgap emitting layers (EMLs) and designing appropriate charge transport materials for efficient charge injection.^[8] In general, there are two approaches to achieve blue emission in PeLEDs. The first way is the halogen regulation strategy.^[9] Increasing chloride content in the mixed Cl/Br perovskites can enlarge the band gap, leading to blue-shifted electroluminescence (EL). However, this method often brings about

severe spectral stability problems as the EL spectrum exhibits a red shift due to phase segregation of the mixed halide perovskites under electric field.^[10] The second way is synthesizing low-dimensional (low-D) perovskite crystals (perovskite nanocrystals, 2D/quasi-2D perovskites) with quantum effect.^[11] Reducing the size of perovskite crystals to nanoscale can widen the band gap, which has demonstrated very efficient blue-emission tuning.^[12] Nevertheless, this method requires complicated chemical synthesizing steps, and the addition of insulating organic ligands dramatically decreases the electrical conductivity of perovskite.^[8] Forming 2D/quasi-2D perovskites with large organic cations, on the other hand, avoids the use of long-chain ligands to some extent and has demonstrated extremely efficient radiative recombination.^[6,7,13] However, achieving blue emission with perovskites that use only Br is still difficult due to the presence of large-*n* phases.^[14] Moreover, as multiple phases always exist in these perovskites, precisely controlling the *n* values to achieve monochromatic blue emission is also challenging. As a result, the spectra are often a combination of several distinct peaks which are from the luminance of these co-existing low-*n* phases.^[15,16] Recently, Xing et al. addressed these problems by partially replacing the long-chain cation (phenylethylammonium, PEA⁺) with shorter ones (iso-propylammonium, IPA⁺), which slows the formation

of $n=1$ perovskite phase and improves the monodispersity of $n=2$, $n=3$, and $n=4$ phases.^[13] As a result, they achieved color-stable sky-blue emission with a single emission peak at 490 nm. However, chemical synthesizing of these organic cations and careful tuning of the precursor stoichiometry are still needed in these cases.^[17]

In this work, we demonstrate a simple and effective way of blue-emission tuning by treating the Br-based quasi-2D perovskite layer with the commonly used electron transport layer (ETL) TPBi (i.e., 2,2'',2''-(1,3,5-benzinetriyl)tris(1-phenyl-1*H*-benzimidazole)). In the active layer preparation step, different amounts of TPBi were added in the anti-solvent, and the resulting solution was dripped during the spin-coating process of perovskite. Such treatment was found to effectively passivate the defects at grain boundaries of the three-dimensional (3D) perovskite.^[18,19] Interestingly, in our quasi-2D perovskite layers, we found systematic blue shifts of the EL spectra of PeLEDs with increasing the TPBi concentration. As a result, we achieved pure sky-blue emission at ~ 480 nm with the color coordinates located at (0.11, 0.15) based on the 1 wt% TPBi treatment. This method does not require chemical synthesizing of the organic cations and, therefore, is much easier to handle. Results of the UV-Vis absorption measurements suggest that the band gap of the perovskite material was enlarged after the treatment, causing these blue shifts.

Experimental method

Materials

The materials used in this work are all commercially purchased. Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS, PVP AI 4083, FHC Solar) was purchased from the company of Heraeus. TPBi (>99.5%) was purchased from Ossila. Lead (II) bromide (PbBr_2 , 99.999%), formamidine bromide (FABr, $\geq 98\%$), phenethylammonium bromide (PEABr, $\geq 98\%$), ethyl acetate ($\geq 99.5\%$), and dimethyl sulfoxide (DMSO, anhydrous, $\geq 99.9\%$) were purchased from Sigma Aldrich. Lithium fluoride (LiF, 99.85%) and cesium bromide (CsBr, 99.9%) were purchased from Alfa Aesar.

Preparation of perovskite light-emitting diodes

For preparation of the perovskite layer, we followed the method developed by Jin et al.^[7] Briefly, 0.161 mmol PEABr, 0.09 mmol CsBr, 0.038 mmol FABr, and 0.129 mmol PbBr_2 were dissolved in the solvent of DMSO. The solution was vigorously stirred overnight before use. The NaCl-doped PEDOT:PSS (abbreviated as PEDOT-Na) precursor solution was prepared according to the method described by Huang et al.^[20] The concentration of the NaCl in the solution was optimized at 10 mg mL⁻¹. At first, the pre-patterned ITO substrates were cleaned with acetone and isopropanol in an ultrasonic bath for 10 min each. The substrates were then treated with O_2 plasma for 15 min to ensure good wetting properties for the deposition of the hole-transport layers (HTLs). The

PEDOT:PSS and NaCl-doped PEDOT:PSS HTLs were spin coated on the substrates at 4000 rpm for 30 s and annealed on a hot plate at 150°C for 30 min in air. Afterwards, the perovskite solution was spin coated on the HTLs at 4000 rpm for 2 min in a nitrogen-filled glovebox. During the spin-coating process, ~ 300 μL ethyl acetate was dripped at 21 s to facilitate crystallization and induce nanocrystal pinning of the perovskite. No further annealing step was required. Finally, the electron transport layer TPBi (45 nm) and top electrode LiF/Al (1 nm/100 nm) were deposited on the perovskite layer by thermal evaporation under a base pressure of $\sim 3 \times 10^{-7}$ torr in a vacuum chamber. The device area was defined as 24 mm².

For the TPBi treatment, different amounts of TPBi powders were added in the anti-solvent EA, and the resulting solution was dripped at the same time (21 s) during the spin-coating process of perovskite.

Characterizations and measurements

The performance of the PeLEDs was measured with a calibrated Botest LIV functionality test system inside the glovebox, with a scanning rate of 0.1 V s⁻¹ and a delay time of 1 s. Perovskite morphology was characterized by a scanning electron microscope (SEM) from Zeiss company. The Fourier-transform infrared (FTIR) spectra were measured with a Bruker Vertex 80v FTIR spectrometer in vacuum (~ 3 mbar). All spectra were measured in transmission mode and referenced to the spectrum of a bare silicon (Si) substrate with a natural oxide layer. UV-Vis absorption spectra of the TPBi-treated perovskite films were measured with a Jasco V660 spectrometer. Photoluminescence (PL) spectra were recorded with a Jasco FP-6500 spectrofluorometer using a monochromated Xenon lamp. The excitation wavelength was 370 nm.

Results and discussion

In this work, this method was utilized for the first time in the modification of the quasi-2D perovskite material. For the fabrication of the PeLEDs, we utilized a device structure developed in our previous work [Fig. 1(a)], where NaCl-doped PEDOT:PSS was used as the hole-transport layer (HTL) yielding a large increase in the device performance.^[20] Figure 1(b–e) shows the performance of the PeLEDs with different concentrations of TPBi. A detailed statistical analysis of the performance over several samples is given in Table S1. The treatment yielded a gradual blue shift of the EL of the devices with increasing TPBi concentrations [Fig. 1(b)]. The PeLED with 1 wt% TPBi showed an EL peak at 481 nm, nearly 25 nm of blue shift compared to the ones without (506 nm). The corresponding Commission internationale de l'éclairage (CIE) 1931 coordinates for each device are shown in Fig. 1(c) and Table S1. It indicates that the device emission was gradually shifted from blue green (0.057, 0.60) for the pristine sample into the blue region (0.11, 0.15) for the 1 wt% TPBi sample. This coordinate is located almost at the edge of the CIE diagram, indicating a very pure sky-blue emission. To

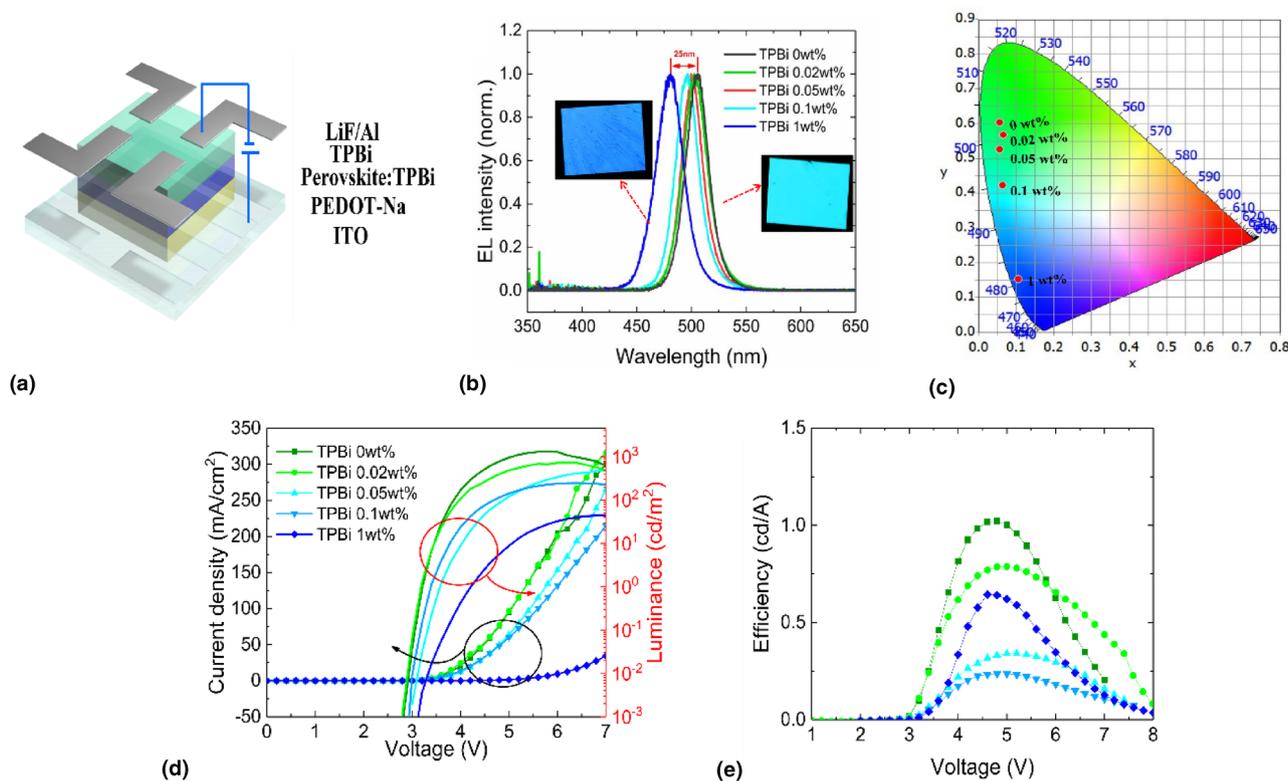


Figure 1. Characteristics of PeLEDs with different concentrations of TPBi treatment. (a) Device structure; (b) normalized EL spectra; (c) the corresponding CIE coordinates of the peaks in (b); (d) current density–luminance–voltage (LIV) characteristics; (e) current efficiency vs. voltage curves.

clarify, if there is an additional TPBi emission contribution after the perovskite was treated, we fabricated a reference device with the structure: ITO/PEDOT-Na/TPBi/LiF/Al. In this device, the TPBi served as both the EML and ETL. Figure S1 shows a broad EL peak with a maximum at ~ 380 nm and an onset of ~ 340 nm, corresponding to the typical TPBi emission.^[21] No emission in this region was observed in our TPBi-treated devices, thus, suggesting a complete energy transfer from TPBi into the quasi-2D perovskites. However, the TPBi treatments were not observed to give rise to a better device performance. The PeLEDs based on 1 wt% TPBi treatment showed only luminance of 37.9 cd m^{-2} and $\sim 40\%$ of the current efficiency (CE) of the reference devices without any treatment (Fig. 1d, e and Table S1).

We investigated the chemical interaction between the TPBi and perovskite with FTIR spectroscopy measurements shown in Fig. 2. We did not observe much difference in the IR spectra of perovskite with TPBi-treating concentration in the range of 0–0.1 wt%. However, some extra peaks appeared in the IR spectrum of the perovskite film when 1 wt% TPBi was treated. The additional peaks with wavenumbers in the range of $1250\text{--}1500 \text{ cm}^{-1}$ can be ascribed to the fingerprint region of the C=N and C–N bondings of nitrogen heterocyclic rings in the benzimidazole part of TPBi.^[22] Moreover, the broad

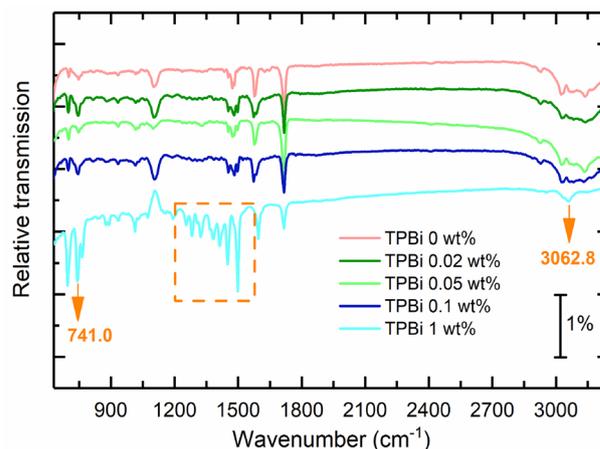


Figure 2. FTIR spectra of perovskite films with different concentrations of TPBi treatment.

peak at around 3000 cm^{-1} and the peak at 741.0 cm^{-1} can be assigned to the vibration of C–H in phenyl ring^[23,24] and the C–S bond of TPBi,^[24,25] respectively. No significant shifts were observed in these peak positions. These results confirm that TPBi remains as single molecules in the perovskite film during the treatment. Consistently with this result, other

groups demonstrated that TPBi did not chemically react with perovskite when it was used as an additive in the perovskite precursor solutions, which is due to the presence of relatively stronger Lewis bases of DMSO solvent.^[22]

Figure 3 shows the SEM images of the perovskite thin films with and without the treatments. The pristine perovskite film exhibited an extremely smooth surface with uniform and small grains [Fig. 3(a)], in good agreement with the reported quasi-2D perovskite prepared by the one-step nanocrystal-pinning method.^[7] In Fig. 3(b), however, small pinholes were observed in the perovskite films with 0.02 wt% TPBi treatment. These pinholes will create substantial shunt paths for leakage currents, leading to lower EL emission and current efficiency of the PeLEDs.^[26] In Fig. 3(c), a few white spots started to appear on top of the perovskite layer when treated with 0.05 wt% TPBi. The increasing amounts of these spots were observed when increasing the concentration to 0.1 wt% [Fig. 3(d)]. Based on the IR results shown in Fig. 2 confirming that the TPBi molecules did not chemically react with the precursors, these white spots can be ascribed to the increasing number of TPBi impurities precipitated on the surface of the perovskite films. We also note that wrinkle-like grains were formed in the perovskite films treated with 0.1 wt% TPBi [Fig. 3(d)]. An even rougher morphology with rod-like grains is observed in Fig. 3(e) when the films were treated with 1 wt% TPBi. This can be explained by the “impurity effect” in which the TPBi molecules intercalate into the grains which suppress continuous crystal growth of the perovskite.^[22,27] We think that the increasingly inhomogeneous, rough perovskite films after the TPBi treatments together with the growing number of pinholes affect the charge transport, resulting in charge imbalance

and nonradiative recombination, thus, causing the observed decrease of the overall device performance.

We furthermore ruled out an influence of the substrate on the blue shifts of the device emission. We fabricated reference PeLEDs on pristine ITO/PEDOT:PSS substrates (without NaCl doping), and the corresponding EL performance is shown in Fig S2 and Table S2. The EL spectrum of the PeLED with 0.1 wt% TPBi treatment also exhibits a 10 nm blue shift compared to the ones without [Fig. S2(b)]. These results suggest that the origin of these blue shifts is more likely due to the electronic property change of perovskite emitting layer after the treatment and not influences by the substrate properties (e.g., morphology or chemical composition). Therefore, UV-Vis measurements were performed to characterize the optical properties of the TPBi-treated perovskite films. As depicted in Fig. 4(a), the pristine perovskite film shows distinct excitonic absorption peaks at ~ 430 nm and ~ 460 nm which are corresponding to the absorption of the $n=2$ and $n=3$ layered perovskites, respectively. However, the absorption intensities of these domains declined when treated with TPBi. Also, these peaks seem to exhibit a gradual blue shift with increasing TPBi concentrations. A similar but more obvious trend was also observed in the shift of the absorption edges of these TPBi-treated perovskites. These results imply that the band gaps of the quasi-2D domains were enlarged while the low- n domains were suppressed after the treatment. However, these reduced low- n phases were not observed to change the 2D crystal structure.^[28,29] As shown in Fig. 1(b), all the PeLEDs display single EL emission peaks suggesting an efficient energy funneling from the low- n domains to the high- n domains; therefore, we think that the enlarged band gap of these high- n perovskite phases is mainly responsible

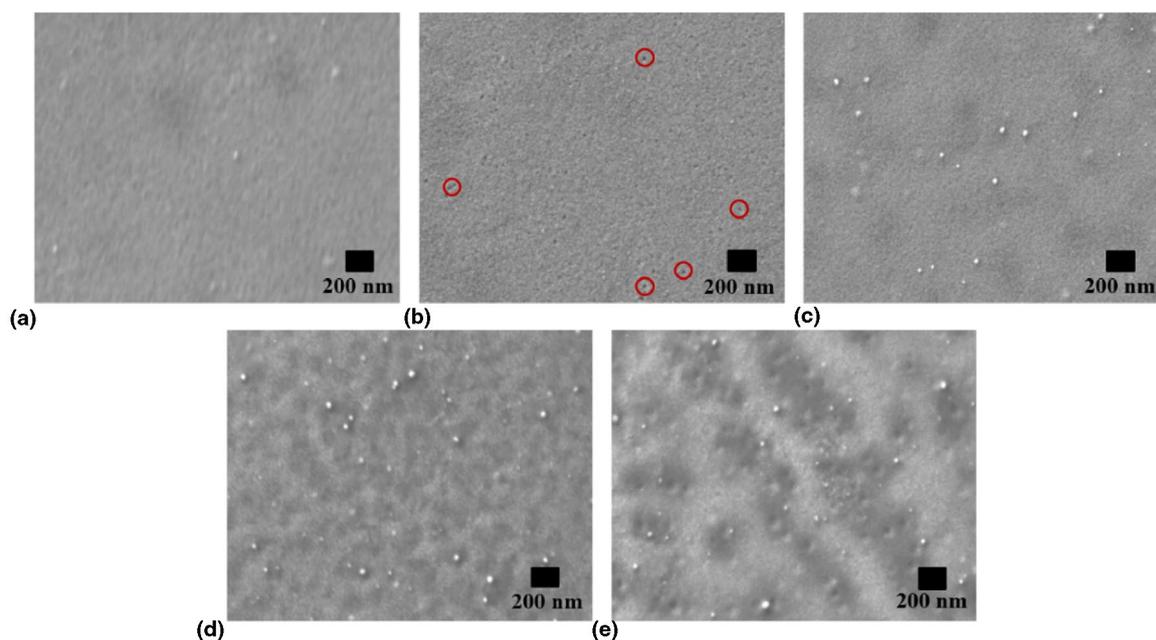


Figure 3. SEM images of quasi-2D perovskite films with the different concentrations of TPBi treatment. (a) 0 wt%, (b) 0.02 wt%, (c) 0.05 wt%, (d) 0.1 wt%, and (e) 1 wt%.

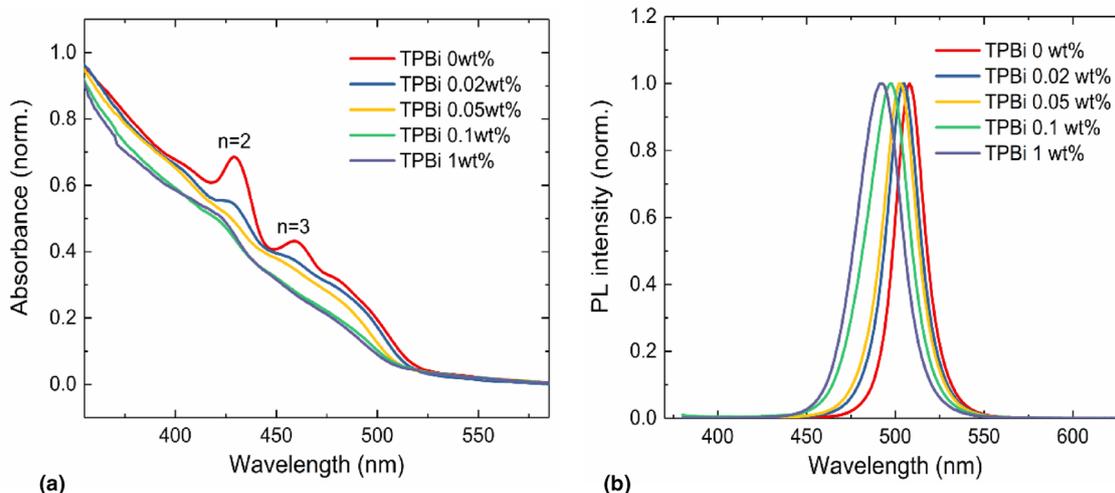


Figure 4. UV-Vis absorption spectra (a) and the photoluminescence spectra (b) of quasi-2D perovskite films with different concentrations of TPBi treatment.

for the blue-shifted characteristics, despite the reduction of the low- n phases after the treatment. The PL spectra in Fig. 4(b) show that a systematic blue shift of the PL emission peak from 508 to \sim 490 nm was achieved when the TPBi concentration was increased from 0 to 1 wt%, following the trend of the EL spectra [Fig. 1(b)]. As TPBi remains as impurities on the surface of the perovskite films (Fig. 3), we speculate that the enlargement of band gaps and the corresponding change in the electronic structure might be owing to the intercalation of TPBi to the grain boundaries of the perovskite.^[19] A similar mechanism can be found in the yttrium (III) chloride (YCl_3) doping of quasi-2D perovskite.^[30] In that work, the yttrium was found to incorporate into the perovskite grains, which increases the band gap of the grain cell and also helps confine the charges inside the grains for more efficient radiative recombination. Enlarged band gaps caused the blue shifts of the EL and PL spectra [Figs. 1(e) and 4(b)] and consequently lead to the blue emission.

Conclusion

In summary, we demonstrate a facile way of efficient blue-emission tuning of perovskite through a TPBi surface treatment. The treatment resulted in up to \sim 25 nm blue shift of the device emission with increasing the TPBi concentrations to 1 wt%, and finally, a pure sky-blue emission at \sim 480 nm was realized. UV-Vis absorption spectroscopy confirmed that the perovskite band gap was enlarged after the treatment, which would be the origin of the blue shifts. We note that the TPBi molecules remain as impurities during the treatment, which affect the growth of the perovskite crystals, causing the deterioration of the perovskite morphology and therefore, the decrease of the PeLED performance. Hence, further morphology optimization and device engineering are needed for ensuring good

performance. Also, future work should address the current stability issue of the quasi-2D PeLEDs and further explore the thermal stability of the tuning level of the TPBi treatment. However, our results open a new window for blue PeLEDs, highlighting that a simple surface treatment can also be a viable avenue for blue-emission tuning of perovskite.

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Declarations

Conflict of interest

The authors declare no competing financial interests.

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Supplementary Information

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