Ensembles of Photonic Beads: Optical Properties and Enhanced Light—Matter Interactions

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Light management is of paramount importance to improve the performance of optoelectronic devices including photodetectors, optical sensors, solar cells, and light-emitting diodes. Photonic crystals are shown as an effective metamaterial for trapping light among their various photon management functions. Herewith, it is demonstrated that spherical photonic crystals, or in other words, photonic beads, possess a stronger light-trapping effect compared to the planar counterpart. The photonic beads are fabricated by colloidal self-assembly under microdroplet confinement employing microfluidic devices. The light—matter interactions are illustrated by the emission intensity and lifetime of the embedded emitters, namely carbon dots and upconversion nanoparticles (UCNPs). The bandgaps of the photonic beads are selected according to the emission and excitation peaks of the light emitters, whereby the emission or excitation peak overlaps the blue edge or red edge of the photonic bands, respectively. Significantly stronger emission and extended luminescence lifetime are observed in photonic beads ensemble in comparison to the planar photonic crystals, demonstrating enhanced light trapping owing to the spherical geometry, which introduces additional microcavity effect. Photonic beads represent a perfect hierarchical light manipulation system. Combining both photonic and microcavity resonator effects, photonic beads potentially find applications in light harvesting, sensing, lighting devices, and light-triggered manipulations.

1. Introduction

Enhanced light—matter interactions can be categorized into enhanced light absorption and enhanced light emission, which is fundamental to efficient energy conversion in the biological and artificial photosynthesis systems, and to the design of light-emitting devices, respectively.[1] Light—matter interactions are intrinsically multiscale; therefore, hierarchically structured materials integrating features of nano- to microlength scales exhibit unique and often advantageous optical properties.[2]

Photonic beads (PhBs), or spherical photonic crystals (PhCs), represent one of these hierarchical nanomaterials exhibiting intricate light—matter interactions.[3] While the periodic dielectrics contrast presented in photonic crystals induces a forbidden region for electromagnetic waves, namely the photonic bandgap (PBG), additional light—matter interactions observed on photonic beads arise from the spherical geometry. These interactions are 1) isotropic photonic bandgap properties versus the angle-dependent photonic bandgap in 2D and 3D photonic crystals, hence, representing materials with full bandgap,[4] 2) grating diffraction on curved surfaces,[5b] and 3) heterogeneous light—matter interactions from crystalline outer shell to internal random dense packing.[5b]

In addition to photonic bandgap per se, the band edges have also been found to induce intricate light—matter interactions. Since the photons at the bandgap edges possess reduced group velocity, resulting in a light-trapping effect called the “slow photon,”[5] the bandgap edge photons would have stronger
interactions with the dielectric materials, specifically for the blue and red edge coupling with the phases of low and high dielectric constant, respectively. The bandgap edge modulation provides powerful means to control light and has found broad applications in miniature lasers, light-emitting diodes,[6] solar cells,[7] and photocatalysis,[8] sensing, and actuations.[9] The slow photon effect can be understood as the mean free path of light being significantly extended without increasing the material thickness. Through theoretical investigations, Deparis et al.[10] proposed the concept of loose confinement in subwavelength size regions to explain how the blue and red edges play their roles in enhancing light absorption.

An immediate following question is how to introduce secondary nanostructures to further extend the light trapping of slow photons? Li and co-workers[11] have shown that the slow photon can be further slowed down by introducing higher order structures such as the B—R—B sandwich-structured trilayer TiO$_2$ inverse opal (B refers to blue edge in resonance with TiO$_2$ electronic absorption edge; R refers to red edge in resonance), which resulted in an additional fivefold photocurrent density in comparison to monolithic photonic crystals. By introducing metal, the plasmonic effect has also shown intensification of light—matter interactions in addition to the photonic effect.[12]

However, there appears to be a lack of in-depth understanding on the effect of additional spherical geometry on light trapping in PhBs.[16]

A very intriguing aspect of slow photon effect in photonic crystals is its influence on the photoluminescence (PL) of emitters embedded in photonic crystals. Previous works[9b,13] demonstrated that when the photonic bandgap edges overlap the emitters’ emission wavelength, the emission intensity would be considerably enhanced owing to the coherent scattering effect, and that when the photonic bandgap edges overlap emitters’ excitation wavelength, the emission enhancement is even stronger due to stimulated excitation. Such effects have given rise to the concept of photonic crystal phosphors,[14] yet, the photonic bandgap-modulated light—matter interactions in PhB ensembles have not been clearly characterized, particularly in comparison to their planar counterparts.

Herein, we show that the spherical geometry of the PhBs provides an additional leverage to slow down the selected band of photons, hence further enhancing the light—matter interactions. In this work, the PL enhancement in the photonic structures was employed as the indicator for demonstrating the enhancement strength of micro- and nanostructures on light—matter interactions. We fabricated PhBs through water-in-oil emulsion confinement using microfluidic technology. A single layer of photonic beads was then prepared for studying the ensemble optical properties, and the photoemission dynamics in PhBs were characterized to illustrate the light-trapping effect. The fluorescence matched with photonic bandgap edges was significantly enhanced in comparison with planar photonic crystal counterpart. We found that the bandgap-modulated PhB represents a superior hierarchical structure for confining the light.

### 2. Results

#### 2.1. PhBs and PhB Ensemble

Two different sizes of polystyrene spheres with respective diameters of 205 ± 5 and 377 ± 5 nm were synthesized in-house and employed for the fabrication of PhBs and PhCs. The polystyrene (PS) sphere size selection serves for the following study on photoluminescence enhancement. The size and the shape of the polystyrene spheres were characterized using a scanning electron microscope (SEM; Figure S1, Supporting Information). Table 1 provides a summary of the samples that are used in this study.

PhBs prepared by microfluidic technique with 205 nm polystyrene spheres as the constituent particles were observed under an optical microscope showing a bright blue color, as depicted in the Figure 1a. The SEM image in Figure 1b shows the surface of bead at high magnification with quasiorder packing, which may reduce the iridescence and produces saturated color.[15] The SEM image of the bead shown in Figure S2 (Supporting Information) exhibits a perfectly spherical shape with a diameter of 78 μm in size. The cross-sectional image of the bead in Figure 1c shows uniform packing at the surface for about 2 μm depth and a disordered packing toward the core.

The uniform bright blue color of the bead could be due to its spherical nature and closely packed PS spheres.[16] Figure 1d shows the SEM image of the PhBs’ ensemble, which illustrates a monolayer deposition of the PhBs in a random close packing. The average film thickness was measured as ≈73 ± 30 μm.

Unlike typical characterization of photonic beads where individual photonic beads would be measured,[3b] we evaluated the reflectance spectra for “n” number of beads (Figure 2a), using diffused reflectance spectroscopy. The photonic bandgap or stopband was found to be centered at 488 nm (Figure 2a), and the inset displays the optical image of the PhB ensemble on the glass film, showing the uniform, angle-independent blue color. The following Bragg–Snell equation was used to estimate the photonic bandgap[17]

$$\lambda = 2dn_{\text{eff}} = \left(\frac{8}{3}\right)^{1/2} D \left(\sum n_i^2 V_i\right)^{1/2}$$

where $\lambda$ is the wavelength of reflected light, $n_i$ is the refractive index of the two phases 1.59 for polystyrene and 1 for air, $V_i$ is the volume fraction of the two phases 0.74, $d$ is the lattice constant, and $D$ is the constituent particle diameter, which is 205 nm in this case. The calculated bandgap wavelength was found to be 488.7 nm, matching perfectly well with the experimental measurement.
Figure S3a (Supporting Information) shows that at different viewing angles, the PhB-488 nm displays a constant reflection peak in contrast to planar PhCs, whose reflection peak wavelength changes on changing the angle (Figure S3b, Supporting Information). The uniform structural colors induced by spherical photonic beads have been employed in sensors and displays.\[18\]

The PhB-902 nm beads and film show whitish color without observable grating diffraction effect as shown by the optical image of the film on a glass substrate in the inset of Figure 2b. The PhB-902 nm bead shown in Figure S4 (Supporting Information) exhibits a spherical shape with a diameter of 80 ± 10 µm. The recorded reflection spectrum of PhB-902 nm is presented in Figure 2b, exhibiting a distinctive reflective peak centered at 902 nm. The red side of the reflection peak intriguingly shows a higher reflectivity compared to the blue side of the peak. On observing the reflection–transmission data of PhB-902 nm film in Figure 2b, light transmission increases above the critical threshold wavelength. Moreover, Figure 2a indicates that the light with energies greater than the observed reflection peak does not appear to transmit through the PhB film. However, the photons with energies lower than the threshold can be transmitted through. This observation on PhB films is consistent with the optical characterizations performed on individual PhBs,\[16\] suggesting the macroscopic optical properties of the PhB ensemble reflect the microscopic optical properties of individual PhBs with high fidelity.

2.2. Light Trapping in Photonic Beads as Illustrated by Embedded Emitters

Fluorophore emission in photonic structures could be significantly altered owing to the intricate and convoluted light–matter interactions.\[9b\] For probing the light-trapping effect of the photonic beads, fluorescent nanomaterials, carbon dots (CDs),\[19\] and upconversion nanoparticles (UCNPs)\[20\] were employed as the emitters.
2.2.1. Case 1: Carbon Dots’ Emission Peak Overlaps with PhB Bandgap

In the first instance, we studied the enhancement effect when the fluorophore emission peak falls on the edge of the photonic bandgap of the PhB. The size selection of the polystyrene sphere was based on the emission of carbon dots. Figure S5 (Supporting Information) shows that the as-synthesized carbon dots show an absorption peak centered at 420 nm in the UV–vis spectrum and emission centered at 476 nm in the fluorescence (PL) spectra. We have positioned the carbon dots’ emission peak at the photonic bandgap with a slight offset toward its blue edge using polystyrene spheres of 205 nm in diameter (PhB-488 nm) (Figure 3a). Another photonic beads’ film (PhB-902 nm) comprised of photonic beads with a bandgap at 902 nm far away from the carbon dots’ excitation and emission peaks was also employed for comparison purposes (see Figure S6 in the Supporting Information). To compare the effect between the spherical and planar PhCs, the same amount of carbon dots was infiltrated in the corresponding planar PhC films of the same surface area. Figure 3c,d (i,iv) shows the PhB-488 nm and PhB-902 nm beads without carbon dots under excitation and emission peaks and Figure 3c,d (ii,v) shows the PhB-488 nm and PhB-902 nm beads with carbon dots under white light. We notice that the intensity of PhB-488 nm beads (Figure 3c (iii)) under 365 nm excitation light has a higher intensity in comparison to that in amorphous PhB-902 nm beads (Figure 3d (vi)).

The confocal microscopic image in Figure S7a (Supporting Information) shows the unvarying emission throughout the PhBs, suggesting that the carbon dots are uniformly distributed on the surface. It is worth mentioning that the glass substrate with the same amount of carbon dots coated on the surface did not show fluorescence of similar intensity, indicating that PhB structure can intensify the fluorescence signal. Figure S7b (Supporting Information) depicts the optical images taken at different angles of a single PhB infiltrated with carbon dots, where no change in the PL intensity could be observed.

Figure 3b shows the PL spectra of carbon dots in matching PhB (PhB-488 nm), matching planar photonic crystal (PhC-488 nm), non-matching PhB (PhB-902 nm), and the control PS thin-film sample (referred to as “amorphous”). By embedding in PhB-488 nm the carbon dots showed a striking 27-fold enhancement in comparison to that in amorphous PhB film. This performance is significantly higher than its planar counterpart PhC-488 nm, which induced a tenfold enhancement. Since the reflection peaks of the PhB-488 nm and PhC-488 nm at 488 nm almost coincide with the carbon dots’ emission peak at 476 nm, a coherent scattering effect is likely induced, resulting in enhanced fluorescence. However, the spherical photonic crystal structure has generated almost threefold higher fluorescence intensity compared to its planar counterpart. This enhancement can only be attributed to the secondary architecture, namely the spherical geometry.

When carbon dots were embedded in PhB-902 nm, whose photonic bandgap has no influence on either the absorption or emission of the carbon dots, a sevenfold PL intensity increase was noted in comparison to the control sample. This fluorescence intensity increase can be attributed to the enhanced light scattering and better dispersion matrix for carbon dots. The fourfold PL intensity difference between PhB-488 nm and PhB-902 nm can be solely attributed to bandgap modulation, by which the photonic bandgap and the carbon dots’ emission are in resonance.
2.2.2. Fluorescence Lifetime Analysis

Fluorescence lifetime provides rich information on fluorophore behavior in the time domain. We performed the fluorescence lifetime study on the carbon dot–infiltrated PhB ensemble using a 366 nm pulsed laser, and the fluorescence emission intensity at a fixed emission wavelength was collected in a period of time to obtain the fluorescence intensity decay profile. The fluorescence decay kinetics of the carbon dots could be deconvoluted with a biexponential decay function, indicating that the CDs may have two types of PL origins, consistent with the most reported CDs.\[9b,c\] Figure 4 displays the PL decay curve and the average lifetime of carbon dots on its own as a thin film, carbon dots in PhC-488 nm, in PhB-902 nm, and in PhB-488 nm are obtained as 5.4, 7.1, 8.0, and 9.1 ns, respectively. Figure 3a shows that the carbon dots’ emission peak is closely aligned with the bandgap of PhB-488 nm and PhC-488 nm (perpendicular direction); therefore, the propagation of the emitted photons is hindered. Such a trapping effect due to the photonic crystal structure is shown by the extension of the fluorescence lifetime in PhC-488 nm, namely 7.1 ns versus 5.4 nm of bare carbon dots, e.g., 31% extension. Surprisingly, the fluorescence lifetime of carbon dots in PhB-902 nm has been extended at a higher rate of 48%. Since the bandgap of PhB-902 nm does not affect either the excitation or the emission, such a light-trapping effect should be entirely due to the spherical geometry of the photonic beads. The average fluorescence lifetime measured from carbon dots in PhB-488 nm as expected shows the strongest extension, 69%, with both photonic and cavity effects at work.

2.2.3. Case II: UCNPs’ Excitation Peak Overlaps PhB Bandgap

We employ $\beta$-NaGdF$_4$:Yb,Er$@$NaYF$_4$ UCNPs to study the enhancement effect of PhBs when its bandgap is modulated against the excitation maximum of the emitters. In an upconversion luminescence process, multiple photons of lower energy are absorbed sequentially and converted into one emitted photon of higher energy, of which the conversion efficiency is low, typically around 1%. Therefore, it is highly desirable to design optically favorable nanostructures to enhance the quantum yield of UCNPs. In $\beta$-NaGdF$_4$:Yb,Er$@$NaYF$_4$ UCNPs, $\beta$-NaGdF$_4$ works as host and Yb$^{3+}$ ions function as sensitizers that absorb 980 nm photon wavelength, which stimulates (Er$^{3+}$ ions) activators by transferring energy. After receiving a second photon or having undergone a transition of energy, the excited Er$^{3+}$ electrons are driven to a higher rate of energy, leading to a radiative decay through several available transitions of distinct energy levels.\[22\] Figures 5a and Figure S8a (Supporting Information) show that the $\beta$-NaGdF$_4$:Yb,Er$@$NaYF$_4$ UCNPs absorption centered at 977 nm in the vis–NIR absorption spectrum and its emission has three peaks at 521, 540, and 653 nm (Figure S8b, Supporting Information), respectively.

We have positioned the UCNPs’ excitation maximum at the red edge of the PBG of PhB-902 nm, which is fabricated with PS spheres of 377 nm in diameter (Figure 5a). The PhB-488 nm described above provides a perfect nonmatching photonic beads’ structure for comparison since its photonic bandgap does not overlap with either the absorption or any of the emission peaks of the UCNPs (Figure S9, Supporting Information). The luminescence spectra (Figure 5b) show that the PhB-488 nm provides a negligible emission enhancement, which could be explained that the size of the constituent particles, namely 205 nm, is too small in comparison to the excitation and emission wavelengths; hence, the scattering effect is small.

The microscope images of individual PhB-902 nm and PhB-488 nm beads without UCNP under white light are presented in Figure 5c.d (i,iv) with Figure 5c.d (ii,v) showing them with infiltrated UCNPs under white light. The observable collected emission light from UCNPs in iii) PhB-902 nm bead is greater than that in vi) PhB-488 nm bead, under 980 nm excitation light (EL).

Figure 5b displays the fluorescence spectra of $\beta$-NaGdF$_4$:Yb,Er$@$NaYF$_4$ UCNPs in PhB-902 nm, PhC-902 nm, PhB-488 nm, and the control amorphous film. The emission of $\beta$-NaGdF$_4$:Yb,Er$@$NaYF$_4$ UCNPs is complex with multiple peaks. Comparing the highest peak at 540 nm, the PL intensity of the UCNPs in PhB-902 nm shows 6.5-fold and almost 2-fold enhancement in comparison to the control film and the PhC-902 nm, respectively. As already explained by earlier works,\[8b,4\] when the fluorophore excitation maximum falls on the photonic bandgap edge, the slow photon effect gives rise to stimulated excitation. In other words, the excitation light is trapped in the material; hence, it could excite more emitters. Therefore, both PhB-902 nm and PhC-902 nm enhance the PL.

Figure 4. PL decay of carbon dots’ film, CDs incorporated in PhC-488 nm, PhB-488 nm, and PhB-902 nm.
of UCNPs. However, the luminescence intensity of UCNPs in PhB-902 nm is nearly twofold as high as that in PhC-902 nm, which can be attributed to the spherical geometry of the PhB.

Interestingly, we observe that PhB-902 nm is the only matrix that dramatically improved the 653 nm emission peak; a 25-fold increase can be observed in comparison to the control sample. Since the 653 nm emission enhancement is only pronounced in PhB-902 nm, but not in PhC-902 nm, it is reasonable to consider that the PL enhancement of 653 nm is primarily due to the spherical geometry of photonic crystal, likely caused by the microcavity resonance. Overall, the strong and selective emission tuning effect of the bandgap-modulated PhBs on UCNPs is significant and offers application opportunities.

3. Discussion

Figure 3b indicates that while PhB-488 nm and PhC-488 nm reflection wavelengths coincide with carbon dots’ emission peak, the carbon dots’ emission intensity in the former is almost three times as high as that in the PhC-488 nm. This stark difference can be solely attributed to the spherical geometry of the photonic beads. Since the design of the bead is circularly structured, the light waves may be guided on the surface of the bead as in an optical resonator, where the light wave is contained. Since the emitters are infiltrated in the spherical photonic crystal that has a photonic crystal shell and largely amorphous core, the resonant structure may be alike the whispering gallery mode resonator.[23] The measured extended lifetime of carbon dots in PhB-488 nm, 9.1 ns, versus those in PhC-488 nm, 7.1 ns, also supports the conclusion that the emitted light of carbon dots is well trapped on the PhB-488 nm due to the photonic bandgap modulation and the microresonator structure.

The further slowing down of the “slow photons” is more evident in the case of UCNPs in PhB-902 nm, where the UCNPs’ absorption is enhanced by the spherical photonic crystal. While the emission enhancement induced by the slow photons due to photonic structure is reflected by comparing the emission spectra of PhC-902 nm and the control sample, the difference between the emissions in PhB-902 nm and PhC-902 nm is purely caused by the spherical geometry of the photonic structure. The excitation light of 977 nm is trapped by the photonic structure and circulates in the surface layer of the microbeads, thereby giving rise to the much increased light—matter interactions, increasing the external quantum efficiency by tenfold. Moreover, the different levels of enhancement exerted on the multiple emission peaks of the UCNPs suggest that PhCs and PhBs may be used for manipulating the color output of UCNPs in addition to the emission intensity augmentation as shown in Table S10 (Supporting Information).
We also hypothesize that the microcavity resonance effect is induced not only by the spherical shape of the photonic crystal, but also by the heterogeneity of the constituent sphere arrangement in the PhBs. Figure 1c shows that the PhB has a crust of ≈2 µm thick of highly ordered crystal layer, while underneath the polystyrene spheres appear to be randomly packed. Such a clear demarcation of the two different phases in the PhBs may be an important reason for generating the microcavity resonator effect, since there is a strong scattering effect between the two phases when light passes.[24] Figure 6 illustrates the effect of slow photons confined by the microcavity resonator in a PhB.

4. Conclusion

We demonstrated the slow photons trapped in the photonic structures can be further “slowed down” by secondary architectures, such as the spherical geometry of the photonic crystal. By studying the luminescence intensity and the lifetime of the emitters in PhBs and planar PhCs, we demonstrated significantly improved luminescence intensity simply by confining the light using the hierarchical structures of the PhBs. The PhBs represent a perfect hierarchical light manipulation system integrating both photonic and microcavity resonator effects, which has potential applications in light harvesting, sensing, lighting devices, lasers, and light-triggered manipulations.

5. Experimental Section

Materials: Hexadecane, hexane, and styrene 99% stabilized with 10–15 ppm 4-tertbutylcatechol were purchased from Alfa Aesar; acrylic acid (AA) from Aldrich; potassium persulfate (KPS) from Chemsupply; and hypermer 2296 was gifted by Croda lubricants. Glass slides of premium pathology grade were used as the substrates for making a thin film.

Synthesis of Polystyrene Spheres: A volume of 1.5 mL styrene was diluted in a round bottom flask with 23 mL of distilled water and heated to 60 °C with continuous stirring and with the solution maintained in an inert atmosphere. About 0.20 g of KPS, 0.25 mL of AA, and 40 mL of distilled water were added in another flask and heated to 60 °C. The beaker containing KPS, AA, and distilled water was poured into a flask containing styrene, and the solution kept under moderate stirring for 10 h. The solution was washed repeatedly with distilled water and the supernatant removed by centrifuging at 9000 rpm.

Synthesis and Fabrication of PhBs and PhB Film: To generate PhBs, a polytetrafluoroethylene-based microfluidic device (T-junction) was used. Polystyrene spheres in aqueous solution of (10% V) were used as the dispersed phase, and hexadecane was used as the continuous phase with 2% hypermer 2296 as the surfactant (supplied from Croda lubricants). Scheme 1 shows the schematic image of the fabrication method. The size of droplets generated at the T-junction device was controlled by adjusting the flow rates of the continuous phase (100 µL min⁻¹) and dispersed phase (90 µL min⁻¹) using syringe pumps. The droplets were collected onto a glass Petri dish (hydrophobic) filled with mineral oil and 2.5% hypermer 2296 as the surfactant and kept at 60 °C. The selection of appropriate medium for the collection phase was vital for the collection.

Preparation of Carbon Dots: Solvothermal synthesis of organosilane-functionalized CDs was performed at low temperature (150 °C) using N-(β-aminoethyl)-γ-aminopropyl methyldimethoxy silane (AEAPMS) as the solvent and citric acid as carbon source, which was described in the previous research.[23]

Preparation of Lanthanide Nanoparticles: β-NaGdF₄:Yb,Er@NaYF₄ core–shell upconversion nanoparticles were synthesized by successive layer-by-layer (SLBL) technique, which has been detailed in ref. [22].

Scheme 1. The fabrication process of photonic bead ensembles.
of beads, and the mineral oil/hypermer mixture was chosen based on the interface property of the continuous and dispersed phases. The collected droplets provide a confined geometry for the solidification of polystyrene beads during the evaporation-induced self-assembly process. The PhBs were then washed with hexane to remove the oil phase and were made into film on a glass substrate by doctor blade technique.[12] Preparation of Fluorescent PhB and PhC Films: In the subsequent fluorescent thin-film preparation, a volume of 40 µL of carbon dots’ aqueous solution with a weight concentration of 21.2 g L⁻¹ was drop cast onto the PhB and PhC films and was allowed to dry. An amorphous PS thin film was prepared as the control sample by annealing the PS sphere packing at 150 °C for 60 min for destroying the nanostructures. For studying the effect of PhB bandgap on fluorescence excitation modulation, β-NaGdF₄:Yb,Er@NaYF₄ upconversion nanoparticles were employed as the fluorophore. A volume of 40 µL lanthanide nanoparticles in hexane solvent with a weight concentration of 51 g L⁻¹ was dropped on to the film and was allowed to dry.

Characterization: The reflection property of the PhBs was characterized by using diffused reflectance spectroscopy from Ocean Optics. The surface morphology of photonic spheres and beads was examined by SEM JEOL 7001 operated at 15 kV. Fluorescence measurements were carried out using a Thermo Scientific Lumina fluorescence spectrometer and a laser scanning microscope (Olympus FV1000). Lifetime measurement was carried out using a confocal laser scanning microscope (Olympus FV1000). Lifetime measurement was carried out using Horiba Delta flex time-correlated single photon counting (TCSPC) system at an excitation wavelength of 366 nm.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

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