Integration of Graphene, Nano Sulfur, and Conducting Polymer into Compact, Flexible Lithium–Sulfur Battery Cathodes with Ultrahigh Volumetric Capacity and Superior Cycling Stability for Foldable Devices

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Lithium–sulfur batteries, as one of the most promising next-generation batteries, attract tremendous attentions due to their high energy density and low cost. However, their practical application is hindered by their short cycling life and low volumetric capacity. Herein, compact, flexible, and free-standing films with a sandwich structure are designed simply by vacuum filtration, in which nanosulfur is homogenously coated by graphene and poly(3,4-ethylene-dioxythiophene):poly(styrenesulfonate) (PEDOT:PSS). This unique hierarchical structure not only provides a highly conductive network and intimate contacts between nanosulfur and graphene/PEDOT:PSS for effective charge transportation, but also offers synergistic physical restriction and chemical confinement of dissoluble intermediate lithium polysulfides during electrochemical processes. Therefore, these conductive compact films, used directly as cathodes, show the highest reversible volumetric capacity of 1432 Ah L\(^{-1}\) at 0.1 C and 1038 Ah L\(^{-1}\) at 1 C, and excellent cycling stability with a minimal decay rate of 0.04% per cycle over 500 cycles at 1 C. Meanwhile, remarkable rate performance with a high capacity of 701 mAh g\(^{-1}\) at 4 C is also achieved. Soft-packaged batteries based on this flexible cathode are further fabricated and demonstrate excellent mechanical and electrochemical properties with little capacity decay under folded state, highlighting the practical application of our deliberately designed electrode in a flexible power system.

To meet the increasing demand for efficient and economical electrical energy storage systems promoted by the rapid development of transportation and grid applications, it is necessary to develop low-cost advanced batteries with high energy density and long-term cycling stability. Lithium–sulfur (Li–S) batteries, as one of the most promising substitutes for commercial lithium-ion batteries, have attracted extensive attentions because of their high theoretical energy density (2600 Wh kg\(^{-1}\) or 2800 Wh L\(^{-1}\)), and low cost and environment benignity of element sulfur. However, the large-scale production and commercial application of Li–S batteries have been hindered by the following issues: 1) the intrinsic low electronic conductivity of sulfur (5 × 10\(^{-30}\) S cm\(^{-1}\)) and their discharging products (Li\(_2\)S or Li\(_2\)S\(_2\)); 2) formation of dissoluble intermediate lithium polysulfides (LiPSs) and the "shuttle effect" of LiPSs, resulting in low Coulombic efficiency, rapid capacity fading, and short cycling life; 3) large volume change (up to 80%) during the discharge–charge process, leading to the instability of electrode structure; and 4) relatively low areal or volumetric capacity, compared with state-of-the-art lithium-ion batteries.

To address the issues aforementioned, extensive studies have been conducted to design rational electrodes with elaborate composition and structure. The most common strategy is to combine sulfur with nanostructured carbonaceous materials with high conductivity and enhanced physical confinement of LiPSs, such as microporous or mesoporous carbon, carbon nanotubes, carbon fibers, and their hybrids. However, the nonpolar interaction between nanostructured carbonaceous materials and LiPSs is not sufficient to restrain the shuttle effect of LiPSs. Recently, various polar hosts are deliberately engineered for high-performance Li–S batteries because of strong chemical interactions between polar hosts and LiPSs. Metal oxides or sulfides due to their strong binding energy with LiPSs, are widely used as ideal candidates to trap LiPSs. For example, Nazar’s group synthesized core–shell sulfur–MnO\(_2\) composites as cathode for Li–S batteries, and, due to excellent efficiency of MnO\(_2\) in trapping LiPSs, the capacity fading rate per cycle is only 0.039% over 1700 cycles. Nevertheless, the intrinsic poor electrical conductivity of many metal oxides or sulfides and low volumetric energy density of cathodes with nanostructured metal oxides or sulfides restrict their practical application. Meanwhile, other polar hosts, such as heteroatom (N, S, B, P, Cl, and O)-doped carbonaceous materials or polymers, with functional groups, have also been evaluated.
group systematically studied the effect of different conductive polymers on Li-S batteries and found that PEDOT was superior to the other polymers both in rate performance and cycling stability.\[40\] Nevertheless, Li-S batteries with long-term cycling stability remain a significant challenge. Meanwhile, in order to accommodate the volume change during the electrochemical process, cathode materials with different pore sizes have been studied.\[10–12,14,15\] However, these void spaces increase the whole volume of cathodes and instead decrease the volumetric capacity, which is critical to the practical application of Li-S batteries.\[11\] Therefore, it is necessary to design appropriate cathodes with high volumetric capacity and long-term cycling stability.

Herein, we prepare flexible and binder-free sulfur cathodes by integrating nanosulfur, graphene, and poly(3,4-ethylene-dioxathiophene):poly(styrenesulfonate) (PEDOT:PSS) (SGP), for the first time, to achieve significant improvement in cycling stability and desired volumetric capacity. First, graphene oxide (GO) nanosheets with large surface areas provide sufficient contact sites for sulfur growth, and the highly conductive and compact PEDOT:PSS-graphene network facilitates charge transportation, which results in better sulfur utilization and high-rate performance. Second, functional groups in PEDOT:PSS and residual oxygen-containing groups in graphene provide strong LiPS confinement through chemical interaction. Third, flexible graphene and PEDOT:PSS can accommodate volume change during the charge–discharge process. Finally, as a whole, the compact flexible film results in high volumetric capacity. Benefiting from these advantages, the hybrid electrodes achieve the highest volumetric capacity (1432 Ah L\(^{-1}\)) at 0.1 C, outstanding cycle stability (80% capacity retention after 500 cycles at 1 C), and excellent rate performance. We further investigate the electrochemical performance of SGP as cathodes for soft-packaged batteries and find that the capacity decreases little even at a folding angle of 180°, and remains 91.5% of the initial capacity after 80 cycles under the folded state.

Preparation of flexible SGP films is schematically shown in Figure 1. Typically, sodium thiosulfate (Na\(_2\)S\(_2\)O\(_3\)) was first dissolved into aqueous GO dispersion, followed by ultrasonic and magnetic stirring, respectively. Hydrochloric acid (0.1 m) was added dropwise into the above well-mixed solution under strong magnetic stirring to obtain GO-S solution (step I), and the color of the solution changed from yellow brown to dark brown. Before reduction of GO, PEDOT:PSS aqueous dispersion was added and stirred for 30 min for thorough adsorption of PEDOT:PSS on the surface of GO induced by mutual strong interactions (step II).\[42,43\] During step II, the color of solutions finally became black after reduction. Meanwhile, because of the synergistic effects of \(\pi-\pi\) interaction between rigid PEDOT molecules and 2D graphene and electrostatic interactions between negative charged PSS molecules,\[42\] even after reduction at 95 °C for 1.5 h, the reduced graphene oxide (rGO) was well dispersed in solution without obvious aggregation. In contrast, a large amount of S-rGO (SG) aggregates was formed due to the hydrophobicity of rGO. By vacuum filtration, flexible and free-standing SGP films were obtained (step III), as shown in Figure 1. Due to the conductive networks of rGO and PEDOT:PSS within the SGP composite, the SGP films show a very high electrical conductivity of 36.5 S cm\(^{-1}\). Even after being bent for 100 times under a bending angle of 180°, the SGP films showed almost unchanged electrical conductivity of 35.9 S cm\(^{-1}\), indicating that SGP possessed excellent mechanical flexibility.

During the process of in situ growth of nano-S on GO sheets, because of their large surface area and functional groups, GO sheets not only provide sufficient nucleation sites for sulfur growth, but also anchor sulfur particles and limit their further growth.\[8,44\] Thus, as revealed by transmission electron microscopy (TEM) in Figure 2a, sulfur nanoparticles are homogenously distributed on GO sheets. To further accurately analyze size distribution, sulfur particles in Figure 2a were carefully measured and the obtained size distribution is shown in Figure S1 (Supporting Information). The size distribution is relatively narrow with an average diameter of about 15 nm. Because of the \(\pi-\pi\) interaction between PEDOT and graphene, PEDOT:PSS molecules are inclined to coat tightly on the surface of S-rGO to form sandwich structure, as shown in Figure 2b. Furthermore, the relatively homogenous distribution of elements C, O, and S (Figure 2c–e) also supports the above observation. The uniform nano-S particles can enhance the utilization of sulfur, while the conductive and compact

![Figure 1](https://www.advmat.de)  
**Figure 1.** Schematic illustration of the SGP cathodes: I) in situ growth of nano-S on GO; II) addition of PEDOT:PSS into S-GO solution and reduction of mixture solution to yield a stable dispersion of PEDOT:PSS-functionalized S-rGO; III) vacuum filtration of SGP solution to produce compact flexible SGP films.
sandwich network facilitates ion or electron transportation and restricts the shutting of LiPSs, which have key effects on improvement of electrochemical performance of Li–S batteries. The morphology of the SGP films was further investigated by scanning electron microscope (SEM). As shown in Figure 3a, there are no obvious particles but a large number of wrinkles on the surface, illustrating the homogenous distribution of nano-S particles. The mapping images of elements C, O, and S from top view and side view, as shown in Figure 3b,f,g,d,h, respectively, further demonstrate the homogenous incorporation of nano-S into the SGP films, which is in accordance with the results of TEM. The side-view SEM image clearly reveals a layered structure with some pores between layers for SGP film (Figure 3e). Owing to the excellent effect of PEDOT:PSS on preventing aggregation of rGO nanosheets, the layered SGP film, with a thickness of about 19.5 µm (Figure S2, Supporting Information), is compact but still porous to some extent. This unique condensed structure can not only entrap dissoluble polysulfide and accommodate volume change during the charge–discharge process but also facilitate ion/electron transportation and provide high volumetric loading of active materials.[41,45]

The detailed structure and composition of SGP films were investigated by X-ray diffraction (XRD), Fourier transform infrared spectra (FTIR), Raman spectroscopy, and thermo gravimetric analysis (TGA). As shown in Figure 4a, in both nano-S-graphene composite (SG) and SGP, there are three dominant peaks at 23.4°, 26.0°, and 27.7°, corresponding to the (222), (026), and (040) planes,[15] which are in good agreement with the characteristic reflection peaks of pure sulfur. In addition, there exists a broad peak at 23.6°, indicating the interlayer stacking of rGO. FTIR spectroscopy was used to further investigate the chemical bond structure. As shown in Figure 4b, the peaks at 1061, 1627, and 1732 cm⁻¹, which are assigned to C=O, O=C–O, and C=O bonds, respectively, are relatively weak, demonstrating that GO is partially reduced.[44] Meanwhile, the successful incorporation of PEDOT:PSS in SGP films is proven by the appearance of

![Figure 2](image1.png)

**Figure 2.** a) TEM of nano-S grown on GO, b) images of SGP, and corresponding mapping of element c) C, d) O, and e) S.

![Figure 3](image2.png)

**Figure 3.** SEM images of a) top view and e) side view of SGP films, and corresponding mapping of elements b,f) C, c,g) O, and d,h) S.
680, 832, 976 (attributed to the C=S bond in the thiophene ring), 1074 (the S=O stretching vibration of SO$_3^-$ groups), 1187 (vibration mode of ethylenedioxy groups), and 1311 and 1511 cm$^{-1}$ (C=C and C=C stretching of the quinoid structure of the thiophene in PEDOT). Which, albeit weak, are in good agreement with these characteristic bands of pure PEDOT:PSS. The residual oxygen-containing groups on rGO and successful incorporation of PEDOT:PSS are very helpful in entrapping soluble LiPSs. Meanwhile, the strong polar–polar interactions between functional groups in PEDOT:PSS/rGO and LiPSs because of the strong polar–polar interactions between fundamental groups in PEDOT:PSS/rGO and LiPSs. Raman spectroscopy, as shown in Figure S3 (Supporting Information), also demonstrates that the reduction of GO both in SGP and SG inferred from the decrease of intensity ratio of G (ordered) and D (disordered) band (located at 1589 and 1350 cm$^{-1}$, respectively), and incorporation of PEDOT:PSS proved by the appearance of band at 1440 cm$^{-1}$. TGA test was performed to determine the sulfur contents in SG and SGP, as shown in Figure S4 (Supporting Information). The sulfur contents in SG and SGP are 72.2% and 56.4%, respectively. SG cathodes were fabricated by mixed nano-S-rGO composites with super P and polyvinylidene fluoride (PVDF) at weight ratio of 8:1 in order to make sulfur content in SG and SGP cathodes comparable.

The highly conductive flexible SGP films with a volumetric sulfur loading of ≈1 g cm$^{-3}$ were directly used as cathodes without binder or collectors. Electrochemical performance of SGP cathodes was first investigated by charge–discharge tests at current rates of 0.1, 0.2, 0.5, 1, and 2 C, respectively. In contrast, capacities of SG cathodes at the same condition are 1085, 919, 780, 629, and 481 mAh g$^{-1}$. More importantly, SGP cathodes can still deliver discharge capacity of 701 mAh g$^{-1}$ at 4 C, while that of SG is only 75 mAh g$^{-1}$. After 100 cycles, the specific capacity of SGP cathodes still retains 1210 mAh g$^{-1}$ with Coulomb efficiency above 99%, which is much better than that of SG cathodes (Figure S7a, Supporting Information). To demonstrate the superior rate performance of SGP cathodes, discharge–charge tests were carried out at current densities from 0.1 to 4 C, as shown in Figure 5c. The SGP cathodes deliver reversible capacities of 1391, 1225, 1147, 1013, and 883 mAh g$^{-1}$ at 0.1, 0.2, 0.5, 1, and 2 C, respectively. In contrast, capacities of SG cathodes at the same condition are 1085, 919, 780, 629, and 481 mAh g$^{-1}$. More importantly, SGP cathodes can still deliver discharge capacity of 701 mAh g$^{-1}$ at 4 C, while that of SG is only 75 mAh g$^{-1}$, demonstrating superb rate performance of SGP cathodes. Moreover, discharge capacity of SGP cathodes recovers to 1180 mAh g$^{-1}$ when the current density is back to 0.2 C. The unique compact structure with coating of conductive PEDOT:PSS and graphene is very helpful in prompting charge transportation and restricting shuttling of LiPSs within the electrodes during discharge–charge processes, and thus leads to excellent rate performance. Meanwhile, benefiting from compact sandwich structure of SGP cathodes with a high volumetric sulfur loading of ≈1 g cm$^{-3}$, high volumetric capacity is also expected, which is very critical in consideration of commercialization. Reversible volumetric capacities of SGP cathodes are 1432, 1261, 1181, 1043, 909, and 722 Ah L$^{-1}$. The initial discharge specific capacity of SGP cathodes is smaller than that of SG cathodes, indicating the improved electrochemical kinetics of SGP cathodes with the incorporation of PEDOT:PSS. Even at 4 C, SGP cathodes can deliver discharge specific capacity of 701 mAh g$^{-1}$, exhibiting superior rate performance. Cyclic voltammetry (CV) profile of SGP cathodes was performed at 0.1 mV s$^{-1}$ in the range of 1.6–2.8 V, as shown in Figure S6 (Supporting Information). At the first cycle, there are two pairs of cathodic peaks at 2.33 and 2.05 V, which are related to the formation of long-chain LiPSs from sulfur and generation of Li$_2$S or Li$_2$S$_2$, respectively, while the anodic peak at 2.4 V is attributed to the formation of S$_8$ from Li$_2$S or Li$_2$S$_2$, which are in good agreement with discharge–charge profiles. Meanwhile, the second and third CV curves show similar shape with slight shift with the first one, indicating that SGP cathodes possess good cycling stability.

The cycling performance was first tested at a relative small current density of 0.2 C. As shown in Figure 5b, the initial discharge specific capacity of SGP cathodes is 1357 mAh g$^{-1}$, while that of SG cathodes is only 1238 mAh g$^{-1}$. After 100 cycles, the specific capacity of SGP cathodes still retains 1210 mAh g$^{-1}$ with Coulomb efficiency above 99%, which is much better than that of SG cathodes (Figure S7a, Supporting Information). To demonstrate the superior rate performance of SGP cathodes, discharge–charge tests were carried out at current densities from 0.1 to 4 C, as shown in Figure 5c. The SGP cathodes deliver reversible capacities of 1391, 1225, 1147, 1013, and 883 mAh g$^{-1}$ at 0.1, 0.2, 0.5, 1, and 2 C, respectively. In contrast, capacities of SG cathodes at the same condition are 1085, 919, 780, 629, and 481 mAh g$^{-1}$. More importantly, SGP cathodes can still deliver discharge capacity of 701 mAh g$^{-1}$ at 4 C, while that of SG is only 75 mAh g$^{-1}$, demonstrating superb rate performance of SGP cathodes. Moreover, discharge capacity of SGP cathodes recovers to 1180 mAh g$^{-1}$ when the current density is back to 0.2 C. The unique compact structure with coating of conductive PEDOT:PSS and graphene is very helpful in prompting charge transportation and restricting shuttling of LiPSs within the electrodes during discharge–charge processes, and thus leads to excellent rate performance. Meanwhile, benefiting from compact sandwich structure of SGP cathodes with a high volumetric sulfur loading of ≈1 g cm$^{-3}$, high volumetric capacity is also expected, which is very critical in consideration of commercialization. Reversible volumetric capacities of SGP cathodes are 1432, 1261, 1181, 1043, 909, and 722 Ah L$^{-1}$.
at 0.1, 0.2, 0.5, 1, 2, and 4 C, respectively (Figure S8, Supporting Information), which are the best volumetric capacities for Li–S battery cathodes that have been reported so far (Figure 5e and Table S1, Supporting Information).[20,23,50–57]

The cycling stability of SGP cathodes at high current density was further investigated, as shown in Figure 5e. The long-term cycling performance was tested at 1 C for 500 cycles after three cycles of activation at 0.2 C. The SGP cathode delivers an initial specific capacity of 1008 mAh g\(^{-1}\), and retains 80% (806 mAh g\(^{-1}\)) of the initial capacity after 500 cycles at 1 C, which is much better than that of SG cathode (only 539.1 mAh g\(^{-1}\) was retained after 200 cycles, as shown in Figure S7d, Supporting Information). The decay rate is only 0.04% per cycle, demonstrating excellent long-term cycling stability at high current density, which is also superior to most of the metal-based or heteroatom-doped hosts for cathodes of Li–S batteries (Table S2, Supporting Information). What’s more, the electrochemical performance of our SGP is also superior to other polymer-based lithium–sulfur batteries (Table S3, Supporting Information).

In order to verify the role of SGP films in anchoring LiPSs, adsorption experiments were carried out. As shown in Figure 6a, after addition of SG and SGP, respectively, into

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**Figure 5.** Electrochemical performance of SGP and SG cathodes. a) Charge–discharge curves of SGP cathodes, b) cycling performance of SGP and SG cathodes at 0.2 C, c) rate performance of SGP and SG cathodes, d) comparison of volumetric capacity between SGP and representative electrodes (see details in Table S1 of the Supporting Information), and e) cycling performance of SGP cathodes at 1 C for 500 cycles.

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**Figure 6.** a) Digit pictures of neat Li\(_2\)S\(_6\), Li\(_2\)S\(_6\) solution absorbed by SG and SGP, b) ultraviolet–visible absorption spectra of Li\(_2\)S\(_6\) solution without or with addition of SG or SGP composites, and c) EIS plots of SG and SGP cathodes before and after 100 cycles at 0.2 C.
Li$_2$S$_6$ solution for 5 h, the color of Li$_2$S$_6$ solution with SG films becomes lighter, while Li$_2$S$_6$ solution with SGP films is colorless, demonstrating strong adsorption ability of SGP for Li$_2$S$_6$. Ultraviolet–visible absorption tests were performed to further quantitatively investigate concentration change of Li$_2$S$_6$ after addition of SG and SGP. As shown in Figure 6b, it is clearly seen that, compared with that of pure Li$_2$S$_6$ solution, intensities of absorption peaks for both solutions with SG and SGP decrease, while the absorbance intensity of Li$_2$S$_6$ solution with SGP is much smaller than that of Li$_2$S$_6$ solution with SG. The absorption experiments firmly uphold our speculation that PEDOT:PSS and graphene in SGP cathodes can synergistically anchor LiPSs and therefore strongly suppress shuttle effect during the charge–discharge process, resulting in less capacity loss and thus better long-term cycling stability.

In addition, electrochemical impedance spectroscopy (EIS) experiments (Figure 6c) of SG and SGP cathodes were conducted before and after 100 cycles at 0.2 C, respectively. In the Nyquist plots, the diameter of the depressed semicircle in the high-to-medium-frequency region corresponds to charge transfer resistance, while the sloping line in the low-frequency region is assigned to Warburg impedance.[58] Before cycle, according to the results of equivalent circuit mode (Figure S9, Supporting Information), $R_{ct}$ of SGP cathodes is about 50.3 Ω, which is much smaller than that of SG cathodes (75.1 Ω), implying the superior electrochemical kinetics of SGP cathodes because of the intimate contacts between ultrasmall S particles and robust conductive graphene/PEDOT:PSS network. Meanwhile, after 100 cycles at 0.2 C, $R_{ct}$ of SGP slightly increases to 52.2 Ω, while that of SG rises up to 88.2 Ω, implying less deposition of nonconductive Li$_2$S or Li$_2$S$_2$ and better structure stability in the SGP electrode.[58] SEM images of SGP cathodes and the element mapping pictures, as shown in Figure S10 (Supporting Information), also demonstrate the structure stability of SGP cathodes after 100 cycles.

According to the results we discussed above, the well-designed SGP cathodes have the following advantages. First, intimate contacts of nano-S with robust conductive graphene/PEDOT:PSS not only facilitate charge transportation, leading to high utilization of sulfur and excellent rate performance, but also physically trap LiPSs and accommodate volume variation during discharge–charge processes. Second, the strong chemical interaction between functional groups in PEDOT:PSS or rGO and LiPSs can largely suppress the shuttle effect of LiPSs, resulting in superior long-term cycling stability. Third, the compact sandwich structure accommodates high volumetric sulfur loading, which is beneficial to high volumetric capacity. Therefore, our SGP films can serve as superior lithium–sulfur battery cathode with excellent rate performance, long-term cycling stability, and ultrahigh volumetric capacity.

To further verify the practical application for flexible batteries, thicker SGP films (about 30.3 µm) with a higher areal sulfur loading of $\approx$3 mg cm$^{-2}$ were directly used as cathodes for soft-packaged Li–S batteries. As shown in Figure 7a, light-emitting diode (LED) was lightened up by soft-packaged Li–S batteries in various folding angles (0°, 90°, 180°, and back to 0°) with no obvious change in brightness. Figure 7b shows the cycling stability in various folding angles. Before bending, the initial areal capacity of soft-packaged Li–S batteries is about 3.3 mAh cm$^{-2}$. The capacity decreases slightly with the increase of bending angles, and retains 3.15 mAh cm$^{-2}$ after refattling, indicating excellent cycling stability of SGP films as flexible cathodes. The long-term cycling test shows that areal capacity decreases slightly from 3.28 to 3.08 mAh cm$^{-2}$ after 80 cycles.
under the 180° folding state (Figure 7c). These results demonstrate the excellent mechanical and electrochemical properties of SGP sandwiches as flexible cathodes for soft-packaged foldable Li–S batteries, showing great potential in wearable electronics application.

In conclusion, flexible and free-standing films with nano-S sandwiched between graphene and PEDOT:PSS were fabricated via vacuum filtration. Benefiting from the ultrasmall sulfur particles, excellent mechanical and high conductive graphene and PEDOT:PSS, and strong interaction between LiPSs and the functional groups in PEDOT:PSS or graphene, the compact layered SGP cathodes display high initial capacity of 1584 mAh g\(^{-1}\) at 0.1 C, excellent rate performance with capacity of 701 mAh g\(^{-1}\) at 4C, and superior long-term cycling stability with 80% retention of the initial capacity after 500 cycles at 1 C. Meanwhile, with a high volumetric sulfur loading of ≈1 g cm\(^{-3}\), the SGP cathodes show the highest volumetric capacity of 1432 Ah L\(^{-1}\) among all reported Li–S battery cathodes. What’s more, soft-packaged batteries based on the SGP cathodes are further fabricated and show excellent mechanical and electrochemical properties with little capacity decay under folded state. We believe this study provides a promising route to develop high-performance Li–S battery cathodes by elaborate integration of functional components and hierarchical structures, which may pave the way for next-generation flexible/wearable power sources.

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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