Smart Textiles with Janus Wetting and Wicking Properties Fabricated by Graphene Oxide Coatings

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The interaction between water and fibers is critical in the physiological comfort of garments, especially inner wears. Antigravity directional water transport and ultrafast evaporation are the two key indicators to be expected of a high-performance moisture management textile. However, it is practically still challenging to make the textiles with continuous directional liquid moisture transport and outstanding prevention of water penetration in the reverse direction. In this work, a Janus functional textile achieved by graphene oxide (GO) coating is developed, with the GO coating side on the textile working as the outer side for its good moisture absorbing and spreading features and the reduced GO coating side serving as the inner layer because of its hydrophobicity. Performance of the as-prepared textile is characterized by moisture management tester, exhibiting remarkable accumulative one-way transport index R (1145%) and a desired overall moisture management capacity (0.77) within 120 s, the negative R value (~690.4%) indicates an ultrahigh directional liquid moisture transport capacity. The Janus textile can provide a source of inspiration for the development of more adaptive textiles and garments to maximize personal comfort in demanding situations under hot and humid environments.

1. Introduction

Comfort is one of the most critical factors for textiles. It is mainly implicated by the moisture management of the textiles. This issue has received increasing attentions especially in the fields of sportswear, activewear, and workwear. Moisture management textiles refer to the garments with one-way transport property, allowing the moisture to transport away from the body of a wearer. People tend to sweat or emit perspiration heavily in many conditions, for example, in a humid and hot environment, or in a state of intensive exercise. In these cases where perspiration adheres to human body, inefficient moisture transport can not only affect the thermal-physiological comfort but also result in discomfort and possible skin conditions. Therefore, materials with excellent directional moisture transporting capability are necessary to maintain the comfort the textiles and performances of the wearers.

In this regard, moisture wicking technology has been applied as one of the promising approach. The efficiency of moisture wicking depends on several parameters, which are structural design, surface properties of substrate, micro-structure of pores, and capillary force (FCF). Various technologies are being employed, including single layered textiles composed with surface-modified hydrophobic microfiber. This kind of textiles are commonly developed from polyester and polypropylene, exhibiting with high moisture releasing and low moisture carrying. Mild finishing is required for this single layered microfiber-based textiles to enhance their moisture transport capability. Another applicable approach for wicking technology is the utilization of tailored microfibers. Coolmax fiber has been designed to improve the moisture transport performances of the resultant textiles. It shows considerable moisture transport capabilities, however, this kind of single layered textiles are unable to reserve liquid and prevent it from crossing the textiles in the reverse direction, that is, this is a bidirectional liquid moisture transport textile.

Janus textiles refer to those with asymmetric properties on each side. They have attracted ever increasing attentions owing to their potential benefits in moisture management. This kind of textiles possesses more effective liquid moisture transport performances owing to the independent tailoring and design of each layer. In the context of our work, such Janus materials with directional moisture transport capability can be fabricated in two main strategies: 1) combining the features of hydrophilic and hydrophobic layers by coating them on each side of a cloth, and 2) forming a hydrophobicity – hydrophilicity features and the reduced GO coating side serving as the inner layer because of its hydrophobicity. Performance of the as-prepared textile is characterized by moisture management tester, exhibiting remarkable accumulative one-way transport index R (1145%) and a desired overall moisture management capacity (0.77) within 120 s, the negative R value (~690.4%) indicates an ultrahigh directional liquid moisture transport capacity. The Janus textile can provide a source of inspiration for the development of more adaptive textiles and garments to maximize personal comfort in demanding situations under hot and humid environments.
Textiles are designed to have their inside hydrophobic and outside hydrophilic to generate the pressure gradient and enlarge the FCF from inside to outside. Such an amphiphilic gradient will thus push the moisture out, facilitating the transportation and evaporation of the moisture to the surrounding atmosphere. Babar and Zhao et al. employed a one-step electrospinning process to prepare an inter-connected hierarchical fibrous membranes, the resultant CNW/PA-Ag nanofiber/net composite membranes shows excellent moisture management behaviors as well as considerable antibacterial activity. Baber and Miao et al. developed a breathable and dyeable nanofibrous membrane with outstanding moisture-management properties. Wang et al. developed a temperature-adaptive reversible moisture transportation diode by introducing two kinds of polymer onto each side of a cotton fabric to achieve a pressure gradient. They realized the reversed directional moisture transport under different temperatures. Yan et al. reported a method to fabricate a multi-scaled interconnected fibre porous janus membranes to improve directional moisture transport via electrospinning, a PU/HPPAN janus membrane was developed and a mechanism of inter- and intra-porosity was proposed.

Wet chemical process is a convenient, feasible, and economical fabric functionalization method. It can easily change the surface hydrophilicity and hydrophobicity of the fabric, which is also the main process of current commercial moisture management textiles.

The wet chemistry process suffers from several limitations, such as the residues of monomeric compounds and unexpected thickness of the resin, resulting in potential threat to the health of the wearer and negative influences to the hand feeling of the subsequent fabrics.

Graphene oxide (GO) and reduced graphene oxide (rGO) as single layer materials possess superior properties, such as good mechanical flexibility and large surface to volume ratio, ultrafast diffusion rate of water molecules between GO nanosheets, and difference surface properties of GO and rGO. This enables GO and rGO to bond with substrate via Van der Waals force rather than other chemical bonding, emerging as potential candidates in moisture management finishes. Several novel materials and finishes used to achieve moisture management behaviors exhibiting drawbacks as complicated and non-environmentally friendly fabrication process, unexpected odor of residual finish agent and still texture to the textiles, polluting the environment, and affecting the comfort properties as well.

If GO and rGO are deposited onto each side, the textile can possess the two different surface properties. The modified textile with an asymmetric gradient across the fabric thickness could be tested for its one-way water transport ability.

Herein, we report a simple and facile approaches to develop a smart Janus double side coated textile with excellent one-directional liquid moisture transport performance through spray coating. Superhydrophobic rGO coating layer, deposited by spray coating followed by thermal reduction, were served as inner side next-to-skin, while the hydrophilic GO nanosheets, selected as the coating layer on outer side, were directly spray coating onto the opposite side of textiles. It is hoped that the coated cotton fabric possesses a wettability gradient (FWG) structure that can drive liquid moisture transport from the hydrophobic rGO side (inner layer) to the hydrophilic GO side (outer layer) rapidly. The micro-sized roughness on cotton fibers and porosities between them work as capillary channels to pull the perspiration out. Numerous oxygenated groups on GO surfaces interact strongly with water molecules and the cavities with hydrophilic surfaces create ultrafast diffuse rates between GO sheets, promoting perspiration, diffusion, and evaporation. This process prevents the perspiration from being trapped inside the cotton fiber grooves, thereby achieving the dry and comfort feeling.

2. Results and Discussion

A smart Janus double surface coated textile aimed for the one-way water transport was prepared with GO and its derivative rGO through an easy to operate and low-cost strategy as schematically depicted in Figure 1. GO sheets can form func-

Figure 1. Schematic diagram to depict the production procedures for fabricating the smart Janus double side coated textile.
tional coating through intermolecular bonding with cotton fiber. To prevent the penetration of GO sheets to the opposite side, one side of the cotton fabric was first treated with a viscous starch solution by screen printing. The treated cotton fabric then underwent a single-side spray coating process with GO dispersion, followed with air drying and washing off the starch treatment. The GO deposited on the single-side coated cotton fabric was then under reduction to prepare rGO coating. GO has various functional groups, for example, carboxyl, carbonyl, hydroxyl, and epoxy groups. When GO dispersion was sprayed onto the fabric, it became quickly deposited onto the fabric surface. The reduction process converted many of the accessible ones, making the outer surface hydrophobic. By repeating the GO spray coating procedure on the opposite side, a double side coated cotton fabric was fabricated. Commercially available cotton fabric was used as the substrate. It possesses good breathability and softness, ideal for cheap mass production.

Surface morphological changes at each step were observed by scanning electron microscopy (SEM), with representative images shown in Figure 2. As shown in Figure 2a, the cotton fibers in the fabric all have flat ribbon shape with a natural twist. Figure 2b is the SEM image after GO coating on the cotton fabric, displaying a distinct morphological change after the treatment of GO. It is obvious to find the wrinkled structure on the coated surface, indicating the firm coating of the GO sheets via assembly with the cotton fibers. The wrinkled structure can also be seen clearly on the surface of the cotton fabric after the thermal reduction process (Figure 2c) to convert GO into rGO on the cotton fibers. These observations reveal the wrinkling of roughening upon GO coating, but the reduction process appears to create further wrinkle creases on the fiber surfaces.

Fourier Transform Infrared Spectroscopy (FTIR) was further conducted to investigate the composition and structure of the modified cotton fabric (Figure 3a). FTIR spectrum of the pristine cotton fabric displayed peaks at around 3340, 2900, 1650,
of 10 and 22° values the modified cotton fabric samples, which are at the 2θ value of 15°(101), 16.6°(101), 22.9°(002), and 34.7°(040), corresponding to monoclinic cellulose-Iβ (C6H10O5)n.[38] Comparison of the three curves shown in Figure 3d shows no characteristic peak of GO and rGO from the diffraction measurements. It is thus difficult to find the characteristic peak of GO and rGO in the pattern of the modified cotton fabric samples, which are at the 2θ values of 10 and 22°, respectively.[39] This is because the X-ray beam penetrates the fabric sample and characterizes the inside structures of the bulk cotton fabric. In contrast, the GO and rGO coatings were deposited onto the fiber surfaces, as evident in Figure 3a,b from FTIR and Raman spectra. The results could imply that GO and rGO coating nanosheets are thin enough to help achieve the functional roles of water absorption and repellence without affecting hand property and comfort feeling.

Static water contact angle (WCA) was measured to detect the changes in hydrophilicity or hydrophobicity of both sides. The amphiphilic changes indicate surface wettability. Figure 4a illustrates the shape retaining of the water droplet on the rGO coating side for more than 45 s, with no significant change in the spherical shape (Movie S1, Supporting Information). This result indicates that the inner layer with the rGO coating has high contact angle and the surface is hydrophobic, providing a good water repellence. On the contrary, for the GO coating side (Figure 4b), the spherical shape of the water droplet is not well maintained on the fabric surface. Instead, the water droplet quickly collapsed and was absorbed by the cotton fabric as soon as it came into contact with the fabric surface, showing the power of the hydrophilic fibers to spread and disperse liquid moisture within 1 s (Movie S2, Supporting Information).

The wettability change is attributed not only to the GO and rGO coating layers onto the surfaces of fabric, but also to the structure of the cotton fibers and the morphology of the fabric. They work together as an effective entity for moisture transport and diffusion. First, cotton is widely referred to a porous fiber containing a lot of hydrophilic groups on cellular macromolecules, with good absorbability of moisture.[40] As can be seen from Figure 2a, cotton fibers are rather hollow inside, leaving a sufficient storage space for water molecules. The large surface area helps improve water absorption performance. Second, the structural arrangement between the macromolecules results in a large fraction of micro- and nano-pores between them, equipping cotton fibers with vast accessible areas for amphiphilic modifications.[41] Upon GO coating to form the hydrophilic side, the water molecules can directly form hydrogen bonds with the polar groups on the GO surface and the hydrophilic groups on fiber macromolecules. Surface roughness and fiber networks provide the FCF to improve fiber wicking property within the gradual changes of WCA from > 90° on the inner surface to 0° on the outer surface. On the inner hydrophobic side with the rGO coating, the hierarchical roughness and convex nano-asperities offered by the coated cotton fiber network can block the liquid–air interface for small pores or cavities due to the high contact angles, thereby preventing the micro and nano-pores or grooves on the inner fiber surface being filled by water molecules.[42] Thirdly, the woven structure within the cotton fabric offers many crossed points that interconnect the capillary pores inside, thus bridging the FWG between the two sides of the fabric. The structural network together with the two distinct surface amphiphilicity forges an internal driving force to orient and drive the directional moisture transport. Upon contact of the moisture with the hydrophobic side of the fabric, it intrudes the spacing of the threads and then become expelled to the opposite hydrophilic side.

Figure 4c offers schematic depiction to illustrate how moisture is driven from the rGO coated inner side to the GO coated outer side (atmospheric side). The human body produces heat energy under various activities, sensible, and insensible perspiration can be adsorbed by the fabric in a relatively short time in a humid and hot environment, and can be transported from the hydrophobic inner side rapidly and continuously to the atmosphere (outer hydrophobic side), thus maintaining the skin surface dry and making the wearer feel comfortable. If the moisture remains in the inside of garment and cannot transport to the outer surface of clothing to be evaporated, the temperature of the body remains in a high level. If cooling is not happening, more perspiration will emit after a period of time. If the moisture is transported outside and evaporated, heat energy is released during this process and human body cools down even if the temperature does not get decreased.
Figure 4d presents the probable liquid moisture transport mechanism from the hydrophobic (positive) and hydrophilic (negative) side of the smart Janus double side coated textiles. From the hydrophobic rGO coating side, two opposite forces, upward hydrophobic force (FHF) and downward hydrostatic pressure (FHP), act when the textile is exposed to liquid moisture. FHP is determined by the size of water droplet, while the FHF depends on the surface properties of the top layer. The gravitational force of the perspiration generates sufficient FHP to counteract the FHF in the opposite direction, facilitating the
penetration of the moisture. Moreover, a wettability difference exists in the two layers drive the liquid moisture from the top layer to bottom layer, the force developed by this difference is regarded as the FWG. Once the water droplet come in contact with the surface of bottom layer, a high FCF come in action on the water and push it off the top layer, owning to this high FCF, liquid moisture is absorbed and diffused rapidly in the horizontal direction. During the water transport process, fiber volume expands after moisture absorption, resulting in the size changes of the capillary channels on the different sides of the fabric. Fibers on the hydrophilic side absorb water and swell, with the channel sizes at the GO side becoming narrow (Figure 4d).[42] FCF generated in pores or channels serves to drive the moisture from the rGO inner side to GO outer side. Due to the surface amphiphilic nature and size range of the pores and cavities, it is hoped that the diffusion process can make fast expelling of perspiration from the inside of garment to air environment, with the exact diffusion rates being controlled by the GO nanosheets. Figure 4e illustrates the mechanism of liquid moisture transport from the negative direction (i.e., hydrophilic GO coating side on the top), when the water droplet is dripped onto the surface of GO coated side, under the effective combination of FCF and hydrophilic properties of top layer, the liquid moisture is rapidly dissipated throughout this layer. Additionally, for the smooth transport of liquid moisture, FHP also facilitates the liquid moisture dissipation in GO coating layer. As the liquid is dispersed in large area of GO coating layer, the mass per unit area of liquid decreases, resulting in a signification reduction of the FHP per unit area.

Furthermore, the effective combination of reduced FHP, FWG, FCF, and FHF prevent the liquid moisture from moving to the bottom layer. The liquid moisture is therefore reserved on the GO coating layer and unable to reach the external surface of the rGO coating layer when the liquid is added from a negative direction.

Moisture management tester (MMT) results were further studied to support the mechanistic processes as suggested on the basis of the WCA results. Figure 5 listed the moisture management profiles of modified cotton, flax, and polyester fabric samples, where the black and red lines correspond to the water content on bottom side (GO coating, outer layer) and on top side (rGO coating, inner layer) with respect to time, illustrating the quantitative analysis of liquid moisture transport behavior within 120 s. It was observed that water content in both layers had an increase when water was continuously dripped into the surface of the top layer (i.e., rGO coating side). Once the water feeding was stopped (after 20 s), there is a significant difference in the liquid transport rate. For modified flax fabric, top layer reserved a large amount of water during the test. On the contrary, in terms of modified cotton fabric, a lot of water was pulled off and throughout the rGO coating layer. This superior liquid moisture transport is attributed to the micro-roughness structure of cotton fiber, which enhance the capillary action on the liquid moisture to drive them away from the top layer. The outstanding liquid moisture transport capability was ascribed to the surface modification by GO nanosheets, enhancing the affinity to water as well as providing more liquid moisture transport channels. The high water content on the top layer

Figure 5. Moisture management profiles from surface modified materials plotted against time: a) cotton fabric, b) flax fabric, c) polyester fabric, d) cotton fabric in negative direction. Inner and outer refer to rGO coating and GO coating sides, respectively. Schematics showing the relative water content after the initial periods on top and bottom surfaces of the modified materials denote e) cotton fabric, f) flax fabric, g) polyester fabric, h) cotton fabric in negative direction, with blue symbolizing high water level and black low water level.
represents water retention, which is attributed to its natural water absorption and storage capacity. The rGO coating does not achieve superior hydrophobic effect as in the case of flax fabric. This behavior is also supported by the WCA outcome as shown from Figure S3, Supporting Information, indicating the relatively poor wetting gradient and capillary actions. The water content on the top layer for the modified polyester fabric increases after a delay of around 40 s (Figure 5c), the long wetting time results to the accumulation of perspiration and contributes to the discomfort of wearer.

Figure 5e–g shows water content and spreading in the top and bottom layers of the double side coated cotton, flax, and polyester fabric, respectively. It can be seen that for all modified textiles, majority of water fall on the top layer transported to the bottom layer. Figure 5e demonstrates water content in the top and bottom sides of modified cotton fabric after 120 s of the MMT testing, water also dispersed on complete sample area of the bottom layers for modified cotton fabric. Where blue shade represents the high level of water and black refers to the low level of water. The results show that the water content in the top and bottom layers shows vast difference even during the short testing period, with the water content in the top layer is significantly lower than that in the bottom layer, confirming the hypothesized mechanism that the moisture was transported form the inner hydrophobic layer to outer hydrophilic layer.

Furthermore, when the liquid is added in reversed direction (from hydrophilic GO coating side), a large amount of liquid moisture spreads in horizontal direction under the action of FCF rather than moving in a vertical direction as explained in Figure 4e. The obtained values of R and overall moisture management capability (OMMC) are ~690.4% and 0.31, respectively, indicating that the liquid (sweat) cannot diffuse easily from the next-to-skin surface to the opposite side and will accumulate on the top surface of the fabric. This negative R value confirm that the double side coated cotton fabric possesses excellent one-way liquid moisture transport capability.

To further characterize the modified textiles, Figure 6 compares the key parameters relating to the moisture management of the modified fabrics with different substrates. For all specimens, the top surface wetting time is longer than the bottom (Figure 6a), and this is because the top (inner) layer is with the rGO coating showing hydrophobic property. The wetting time WTt (top surface) and WTb (bottom surface) are defined as the time periods in which the top and bottom surfaces of the fabric just start to be wetted, respectively, after the test commences, defined as the time in second(s) when the slopes of total water contents on the top and bottom surfaces become greater than tan(15°). Therefore, it takes longer time to be wetted than the bottom hydrophilic layer, with the bottom layer absorbing moisture quickly. As cotton and flax are natural fibers and they originally have good moisture absorbability, while the polyester fiber is only capable of containing moisture in small quantity, the wetting time in the top layer is much longer than that of the modified nature fibers. Quicker wetting is one of the significant factors to provide better moisture management.

The maximum wetted radius (MWR) and spreading speed (SS) are the indicators of horizontal wicking properties of textiles. Double side coated cotton fabric possesses relatively large MWR and high SS, and perspiration is therefore spread out
to a large wetting area and dries out quicker, promoting wearing comfort. The accumulative one-way transport capability (R) is the area difference between the liquid moisture content curves of the top and bottom surfaces of a textile with the respect to time. The OMMC is an index of the overall capability of a fabric to transport liquid moisture as calculated by combining three tested parameters: MWR for top and bottom, R and the maximum liquid moisture SS on the top and bottom surfaces. OMMC values can be calculated from Equation (1) based on AATCC test Method 195-2011:

$$\text{OMMC} = 0.25\text{MAR} + 0.5R + 0.25\text{SS}$$  

Figure 6e,f displays the R and OMMC values of the corresponding modified fabrics, with all the fabrics being developed by the two-step strategy exhibiting the outstanding directional moisture transport capability with R values greater than 400. This indicates that perspiration of wearer can be better transported from the skin side to the outer side. This process results are not only the dry feeling of skin but also the easily evaporation of sweat. In order to have good moisture management properties, a fabric should have good horizontal wicking properties (MWR and SS) and outstanding vertical wicking (R) simultaneously. The modified cotton fabric fulfills these criteria and shows outstanding one-directional liquid moisture performance, thus giving the best moisture management properties among all the modified fabric.

3. Conclusion
A smart textile with Janus wetting and wicking properties was fabricated through a novel method of surface modifications to a cotton fabric. With GO and rGO coatings deposited on each side of the cotton, fabric is capable of moisture management via the combination of inner hydrophobic and outer hydrophilic surface coatings. The fabric is capable of absorbing and transporting liquid moisture rapidly from the inside to outside surface and being evaporated in the surrounding atmosphere. The fabrication process is easy to operate and environment friendly. The results as obtained from various characterizations support the structural features underlining mechanistic processes of directional moisture management driven by the capillary actions across the modified cotton fabric, showing great potential for garment comfort applications in various modern working or living situations.

4. Experimental Section
Materials: Cotton fabric (plain woven, 7.5 cm × 7.5 cm, 190 g m⁻² thickness: 0.5 mm), flax fabric (plain woven, 7.5 cm × 7.5 cm, 168 g m⁻² thickness: 0.3 mm), and polyester fabric (plain woven, 7.5 cm × 7.5 cm, 171 g m⁻² thickness: 0.4 mm) were used after cleaning with NaOH solution under sonication. The graphene oxide dispersion (9 mg mL⁻¹) was purchased from ICCCAS (ShanXi, China). Sodium hydroxide (NaOH) and Sodium perborate tetrahydrate (NaBO₃·4H₂O) were purchased from Sigma-Aldrich. ECE Phosphate Reference Detergent (B) was purchased from SDL Atlas. Deionized (DI) water was used as solvents throughout the experiments. A screen mesh was used to screen printing the starch viscous liquid with mesh count of 200.

Fabrication of the Janus Fabric: Fabrics were first treated with NaOH solution to remove the impurities such as oil, wax, and pollution. A viscous starch liquid was used as a mask to adhere on one side of cotton fabric. The graphene oxide dispersion was diluted to 0.5 mg mL⁻¹ with DI water to prepare the spray-coating solution for another side. The spray-coating procedure was carried out by using a spray gun. After that, the fabric was air-dried at 20 °C and followed by DI water rinsing to remove the starch treatment. Single-side GO coated fabric was put into the oven for 1 h under the temperature of 200 °C to reduce the coated GO into rGO. Then repeat the single-side spray-coating process on the side that was starch treated before. After air drying at 20 °C, the double-side coated cotton fabric was fabricated. Modified flax and polyester fabric were prepared by the same strategy.

Fastness to Washing Test: Such as-prepared double side coated fabric underwent a washing fastness test according to ISO 105-C06: color fastness to domestic and commercial laundering. 4 g of ECE Phosphate Reference Detergent (B) and 1 g of sodium perborate tetrahydrate (NaBO₃·4H₂O) were dissolved into 1 l water to make test solution. The test sample underwent a washing test at 60 °C for 30 min, with 50 mL test solution and 25 steel balls inside the port. After the washing test, the detergent attached on the sample was rinsed and air-dried.

Characterization and Performance Investigation: Scanning electronic microscopy (SEM) measurements were conducted by a ZEISS Ultra 55 scanning microscope with prior gold sputtering to observe surface morphology. The composition and structure were characterized by FTIR on Nicolet FTIR 5700. Elemental analysis and crystal structure of samples were investigated by XRD. XRD analysis was performed on the PANalytical X’Pert X’Celerator. Raman spectroscopy was carried out via a Renishaw System 1000 Raman Spectrometer including Modu-laser 514 nm Argon-ion laser to characterize the crystal structure, disorder, and defects in graphene-based materials. Surface tension test was conducted with a Kruss 100 to measure and record the WCA changes on the fabric. Moisture management properties were characterized by using the Sdlatlas MMT in accordance to the ASTM D1776-2008 standard; bottom and upper sensors were concentrically placed in the machine, keeping the testing area as 8 cm × 8 cm, top sensor will drop out a drop of water (which is normal saline to simulate perspiration of human body) on the top surface of the sample. Samples are all washed and ironed to remove wrinkles to avoid inconsistent testing performances. Water will wet and continue to transfer to the back side and close to the bottom sensor. This measuring process lasted for two minutes recording the following indexes: wetting time top/bottom, absorption rate top/bottom, MWR, top/bottom, SS top/bottom, figure of water content versus time as well as figure of water location versus time. The liquid water transport properties, also known as the moisture management capacity, were determined by the absorption rate bottom, SS bottom, and one-way transport capacity; this value ranges from 0 to 1. The closer to the value of 1, the better water absorption and transmission.

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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