A Versatile Flexible Polymer Actuator System for Pumps, Valves, and Injectors Enabling Fully Disposable Active Microfluidics

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To control and manipulate fluids in lab-on-a-chip (LOC) devices, active components such as pumps, valves, and injectors are necessary. However, such components are often complex and expensive to fabricate, limiting integration in disposable LOCs. A new type of flexible, all-polymer diaphragm actuator system, called Double Diaphragm Active Polymer Actuator (DDAPA), is presented as a single modular unit that can be repurposed to diverse active microfluidic components. To demonstrate the versatility of the DDAPA concept, the DDAPA devices are investigated in three different configurations: as a single operation microinjector, as a flow regulating element, and as a pump in a hybrid configuration with unibody-LOC unidirectional systems. The working principle, fabrication process, and the three examples of microfluidic components are presented. The trilayer diaphragm actuator is realized using the conductive polymer poly(3,4-ethylenedioxythiophene) polystyrene sulfonate as the actuating material and thiol-acrylate-based ionogels as solid-state electrolyte and base material. The three demonstrators show the feasibility of using the DDAPA module to inject liquids, regulate flow, and unidirectionally pump fluids up to 112 µL min\(^{-1}\) when coupled with a 3D printed unibody check valve. Hence, the presented concept with a simple mechanism and easy manufacturability, broadens the choice of disposable actuators compatible with fully disposable autonomous LOC solutions.

1. Introduction

Microfluidics, which comprises the handling and manipulation of small volumes of fluids,\(^1\) has attracted significant attention due to its capability of processing and executing complex analytical procedures while at the same time being a resource-efficient, portable technology employing low-cost plastic chips.\(^2\) It is widely used in various fields, including chemical analysis, biosensing systems, medicine development, point-of-care diagnostics, lab-on-a-chip (LOC) devices, and organ-on-a-chip devices.\(^3\) In order to control and manipulate the fluids efficiently, a microfluidic system needs some active components such as injectors, pumps, valves, and mixers.\(^4\) Various actuation mechanisms, such as pneumatic, shape-memory alloy, piezoelectric, dielectric, electromagnetic, and electrostatic, have been developed to drive such active components.\(^5\) However, there are some significant limitations of conventional actuation technology in active microfluidic devices. For example, shape-memory alloys have a relatively slow response time and they are activated using a high transition temperature which could possibly damage the fluid sample, hindering their use in biological applications.\(^6\) The use of piezoelectric and electrostatic actuators has resulted in microdevices such as micropumps and microvalves with simple structures using micromachining and photolithographic techniques.\(^7\) However, the materials used are based on rigid silicon which might not be the material of choice for single-use, disposable, and flexible LOCs. Dielectric elastomer actuators require an activation voltage of up to thousands of volts to achieve reasonable actuation, however, the high voltages involved may change the properties of the samples.\(^8\) Pneumatic valves used in polydimethylsiloxane (PDMS)-based LOC are a simple and most elegant solution to control liquid flow; however, they require additional, external equipment to control the actuation.\(^9\) In addition, most of the conventional actuators rely on hybrid integration of components which is both complex and needs some special fabrication facilities which harm cost efficiency. These characteristics limit the possibility of fully disposable, advanced microfluidic systems. Therefore, it is vital to develop easy to fabricate actuators with a simple mechanism for on-demand control of LOCs that can be potentially efficiently fabricated.

In the past decades, conducting polymers have emerged as promising materials for sensing and actuation in various applications such as cell biology, microelectromechanical systems...
These conducting polymer (CP) actuators are different from conventional actuators and they do not have the above-mentioned constraints. They are electrically driven by a very low actuation voltage (1–3 V) and can generate considerable stress and large strain.[21] This low voltage requirement is favored as it is ubiquitous to obtain in modern life such as from USB power and mobile phones. CP actuators can be miniaturized to small sizes in order to achieve kHz frequency actuation and can be integrated into other microsystems.[22] Moreover, they are soft, flexible, biocompatible, and can interface with biological materials.[13] In addition, the generated stress and strain of conducting polymer actuators can be adjusted through different actuation parameters, material selection, and geometrical design. Considering these advantages of conducting polymer actuators, they are interesting candidates for use as active components in microfluidic devices and have started to attract attention in the research community. For instance, a petal-shaped diaphragm micropump based on polypyrrole and gold-coated polynylidene difluoride (PVDF) materials has been created to reduce the edge restriction of diaphragm actuators.[14] A soft actuator with a tube-in-tube structure, constructed by coating polypyrrole on a metalized tube was developed by Wu et al. for use as peristaltic micropump.[15] Polypyrrole actuators synthesized by electropolymerization on titanium sheets were used to provide the required pressure on a PDMS tube for a new kind of valveless micropump.[16] However, these developments lack a flexible design and operation of the CP actuators for active components in microfluidics, nor do they have a simple fabrication process that could enable the low-cost fabrication needed for disposable microfluidics. The most popular forms of CP actuators are configurations as linear actuators, and bilayer or trilayer cantilever beams.[19a,b,12b,17] However, a diaphragm configuration is interesting for many potential applications in microfluidics such as pumps and valves.

In this work, a new type of all-polymer actuator based on conducting polymer tri layered diaphragm actuators was developed and various potential microfluidic applications of the new actuator are illustrated. The working principle and fabrication process are described in detail. The trilayer diaphragm actuator was realized using the conductive polymer poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) as the electromechanically active electrodes and using ionogels from thiol acrylate free radical photopolymerization as an ionically conductive layer. The actuation capability of conducting polymers such as PEDOT:PSS is based on the ion doping and the de-doping process during electrochemical oxidation and reduction.[22] When a voltage is applied to the PEDOT:PSS layers, the PEDOT:PSS layer on the cathode side is reduced while the layer on the anode side is oxidized. On the cathode, positively charged PEDOT chains are reduced toward the neutral state. Since PEDOT is doped with large immobile anions, PSS, dissociated cations, EMIM⁺, driven by a strong electric field are inserted into the PEDOT:PSS and engage with negatively charged PSS chains which cause the volume to increase. Switching the polarity of the applied potential will reverse the electrochemical reactions, that is, oxidizing the PEDOT chains resulting in the expulsion of the EMIM⁺ cations and a volume decrease.[22,23] It is suggested that during oxidation (anode) also anion insertion might occur, but since the inserted anions are smaller than the expelled cations this still results in a net volume reduction.[20,24] The solvent also plays an important role in the reversible electrochemical volume change when actuating in a liquid electrolyte.[25] In the DDAPA devices, we use an iongel as the ion source/sink to enable actuation that is independent from the surrounding electrolyte and to enable in air actuation. However, there still might be a contribution from a solvent (water) since both PEDOT:PSS[26] and ionic liquids[27] are slightly hygroscopic and since the PEDOT:PSS layers inside the DDAPA do come into contact with water from the fluidic channel. For more details on the electrochemically induced volume change, we refer to review articles that describe the different aspects in more detail.[22,28]

In the classical trilayer bending beam actuator configuration, the difference in swelling of the electrodes upon oxidation and to regulate the flow rate, and by coupling to a 3D printed one-way valve, to be configured as a pump capable of unidirectional flow rates of up to 112 µL min⁻¹. Hence for the purpose of developing active microfluidic components with simple mechanism and easy manufacturability, this work broadens the choice of materials and paves the way for the realization of active, fully disposable microfluidics for diverse LOC applications. The fabrication of the DDAPA unit is rather simple comprising only two patterning steps and lamination and might be fabricated using methods similar to those used in printed electronics, such as roll-to-roll printing and screen printing.[18]
reduction causes it to bend toward the anode and switching the polarity of the applied potential will reverse the bending. In the diaphragm configuration as presented here, where all sides are clamped, the actuator adopts a buckling motion with a convex (cathode up) and concave (anode up) geometry upon cycling the potential. The center point of the diaphragm has the maximum displacement while the edge movement is confined.

The oxidation and reduction of the PEDOT:PSS electrodes are further confirmed in Figure S1, Supporting Information, showing the cyclic voltammograms (CV) of the PEDOT:PSS electrodes with the ionogel at the different potential ranges used in this work. They show that the actuator is electrochemically driven and the redox peaks are in agreement with previous works on PEDOT:PSS in air actuators using EMIM-TFSI solid-state electrolytes based on semi-interpenetrating polymer network.[23c]

A photograph of the assembled single diaphragm actuator and the displacement measurement is shown in Figure S2, Supporting Information. The center point displacement of the single trilayer diaphragm actuator is shown in Figure 1b and Figure S3, Supporting Information for five electrochemical cycles. When a $+1$ V square wave was applied to the bottom electrode, the whole diaphragm bent rapidly upward until it gradually reached a maximum displacement of 0.16 mm at 90 s. When the polarity was reversed, the diaphragm moved downward rapidly and then reached a minimum displacement of $-0.16$ mm. The resulting absolute displacement of the diaphragm was 0.32 mm in the center point and remained constant through the following activation cycles. Then, for this technology, large displacement can be ascribed to the fact that the ionogel prepared from thiol acrylate photopolymerization has a high ionic conductivity and a low Young’s modulus. The relationship of displacement with different applied potentials is shown in Figure 1c. The displacement increased from 0.32 to 0.44 to 0.51 mm for a consecutive 0.5 V increase of the applied potential, as more ions can be driven into the electrodes at higher potentials. However, when the potential increased to 2 V, the generated peak current started to decrease progressively after each electrochemical cycle (Figure S3B, Supporting Information). The PEDOT:PSS electroactive performance is deteriorated due to over oxidation and over reduction processes at a high driving voltage, which may eventually cause irreversible damage to the PEDOT:PSS backbones.[21a,29] A potential of 1 or 1.5 V was therefore chosen in the following studies as a trade-off between actuator performance and stability.

2.2. Double Diaphragm Active Polymer Actuator Structure

A classical diaphragm pump operates through the reciprocating action of a flexible diaphragm, which changes the chamber volume and thus exerts pressure on an internally confined liquid. It has been previously reported that a micropump with only one conducting polymer diaphragm actuator generates no observable flow,[15] which is probably due to geometrical restrictions to the diaphragm deformation and the high Young’s modulus of the PVDF-based electrolyte-containing layer used. A petal-like actuated diaphragm was proposed instead, which unfortunately led to a complicated design of the micropump. In our design, which we address as a DDAPA, the proposed pump has two diaphragm actuators as the active elements as shown in Figure 2a. This design is easily achieved by bonding
two diaphragm actuators and one middle ionogel layer together using the ionogel precursor solution as the adhesive. In the middle layer, the flow channels and a circular pumping chamber are arranged. The reactive thiol groups on the surface of the soft ionogels can participate in the adhesive curing process, forming a strong bond between the layers. In a typical configuration, the device had a rectangular structure of 70 mm × 30 mm × 1.5 mm. The diameter of the circular PEDOT:PSS electrode was 21 mm, yielding an approximate chamber volume of 0.17 mL. This simple assembly method also enables patterning of multiple actuator electrodes simultaneously, for instance placing three diaphragm actuators in series to form a classical peristaltic pumping configuration as shown in Figure S4A, Supporting Information. This shows the versatility of the fabrication and bonding method in building complex fluidic structures with multiple diaphragm actuators integrated. Also, the DDAPA module is fully flexible (Figure S4B, Supporting Information), which is difficult to achieve with classical microfluidic components. To show the feasibility and demonstrate the versatility of the concept, the DDAPA was configured in three essential microfluidic components: as a single operation microinjector, as a flow regulating element, and as a pump in a hybrid configuration with unibody-LOC unidirectional systems (Figure 2), using the same DDAPA module.

Figure 2. a) Schematic illustration of the bonding process for the Double Diaphragm Active Polymer Actuator (DDAPA) The DDAPA chamber is formed by bonding the middle ionogel with two circular trilayer diaphragm actuators. Photographs of the resulting DDAPA in b) top view and c) cross-sectional view. d) Illustration of pumping mechanism. e) Schematic illustration of the DDAPA configured as microinjection tool, f) microvalve, g) and one-way micropump.
2.3. DDAPA Microinjector

First, we investigated the ability of the double diaphragm actuators to dispense a liquid, which at the same time allowed to evaluate the bonding strength of the assembled DDAPA for leakage and the capability of the diaphragm actuators to generate sufficient actuation force to propel a liquid. To illustrate the operation of the microinjector a 4.5 cm plastic tubing was connected to the DDAPA outlet. The DDAPA was operated by actuating the two trilayer diaphragm actuators by applying a square-wave voltage of 1.5V at a 180-degree phase difference. The fluid movement in the outlet tube at different times is shown in Figure 3.

In the first 90 s, the liquid moved forward in the tubing and a drop was formed and dispensed at the end of the tubing. After 90 s the polarity of voltage was reversed and the liquid flowed backward, rendering tubing empty after 180 s. This propulsion could be repeated by cycling the activation potential, resulting in the liquid moving back and forth in the tubing. This shows that the two diaphragm actuators of the DDAPA can provide significantly large deformations, generate a significant injection volume, and even retrieve volumes as a complementary suction device. Also, there is no leakage between the bonded layers, showing that a water-tight bonding is formed. The microinjector configuration of the DDAPA could be used for liquid suction and/or dispensing operations, and for dosing in combination with multiple diaphragms, which is very suitable for controlled drug microinjection.

2.4. DDAPA Flow Rate Regulator

Next, the DDAPA was configured as a flow rate regulator. A reservoir was connected to one end of the DDAPA using a tubing and placed at 5 cm above the device. The liquid in the reservoir flowed through the tubing in one direction to fill the chamber and thereafter exited through the output channel (Figure S5, Supporting Information). The number of liquid drops coming out from the outlet was counted in 10 s intervals to determine the flow rate. The output flow rate of the flow regulator for different applied voltages and frequencies is plotted in Figure 4, and Figures S6 and S7, Supporting Information. Without the actuation, flow rate was constant and varied between 1 and 2 drops every 10 s. When the diaphragm actuators were activated at 1.5 V, for the first 30 s, the flow decreased from three drops/interval until it stopped. This indicates that the volume expansion creates a negative pressure in the pump chamber, which prevents the liquid from coming out. When the polarity was reversed after 30 s, the flow rate increased. This flow cycled in sync with the applied potential cycle and this flow rate pattern was also observed when the activation voltage periods were extended to 60 and 90s (Figure S7, Supporting Information). These results demonstrate that the DDAPA structure can be used for flow rate regulation.

2.5. DDAPA One-Way Pump

Lastly, the DDAPA was configured as a one-way pump. To assess the pumping performance qualitatively and achieve a unidirectional flow, the DDAPA was coupled with two different 3D printed unibody-LOC devices: a Tesla valve and PDMS check valve, into a hybrid pump. In a unibody, the check valve, a through hole in the seat of a thin PDMS element creates an asymmetrical configuration for forward and backward flows. When pressure is applied from the top, the PDMS element will be pushed down and backward flow is reduced. When a pressure is applied from the bottom, the PDMS element is lifted, allowing the forward flow. The variation of the current and the flow rate with time for the hybrid pump at a driving voltage of 1 V is shown in Figure 5. During the first volume expansion of the diaphragms, the outward bending of the diaphragms generated a negative pressure to close the check valve and a very
small backflow at −3 µL min⁻¹ was observed at first. This backflow can be attributed to a minimum pressure front that needs to build up to close the check valve. After reversing the polarity, a positive pressure was generated by the inward bending of the diaphragms that forces the valve to open. Hence, the fluid was pumped to the outlet from the chamber immediately. The flow rate reached a peak of 21 µL min⁻¹ after 110 s and decreased thereafter. The maximum transient flow rate of the micropump in the following two pump cycles was 31 and 36 µL min⁻¹, respectively. When using the DDAPA micropump coupled to a Tesla valve, a large backflow rate was observed (Figure S8, Supporting Information). The Tesla valve was less efficient in regulating the transient flow direction, but the maximum forward flow rate (112 µL min⁻¹) was larger than that of check valve, as more pressure is needed to push open the check valves than in the Tesla valve. The average pumping flow rate for the check valve is 6.6 µL min⁻¹ and for Tesla valve, it is 14 µL min⁻¹, which are appropriate flow rates for microfluidic applications. (It is worth to mention that the flow rate in Figure 5b shows a step-wise manner due to the method used to measure the flow, that is, the flow rate values were calculated every 10 s. See Experimental Section.) The output flow rates can be further adjusted through different actuation parameters, such as applied voltage, frequency, driving signal shape, and by optimizing diaphragm thickness, electrode area, and other micropump design parameters, which will be done in our future research.

3. Conclusions and Outlook

We have developed a versatile, flexible, all-polymer soft diaphragm-based actuator driven by two PEDOT:PSS-based trilayers and illustrated possible configurations thereof for microfluidic applications. The trilayer diaphragm actuator was realized using the conductive polymer PEDOT:PSS as the actuating electrodes and ionogels from thiol acrylate free radical photopolymerization comprising EMIM TFSI ionic liquid as solid-state electrolytes and base materials. The ionogel was also utilized as an ionically conductive adhesive to bond two diaphragm actuators and one structural layer together to form the DDAPA. The bonded DDAPA showed good adhesion and no leakage. We have constructed various complex, active microfluidic components with multiple diaphragm actuators to demonstrate the versatility of the bonding methods. The fabricated DDAPA system proved their modular versatility as it can be easily repurposed, here illustrated to show the feasibility by the three different active microfluidic components presented (Figure 2): a microinjection/suction tool, a flow regulator, and as a fully disposable unidirectional hybrid pump. Contrary to previous reports using one conducting polymer diaphragm actuator, our developed DDAPA can generate liquid flow. Flow rate measurements showed the capability of the system to regulate or pump the output flow. Configured as a unidirectional hybrid pump, the DDAPA could transport fluids at flow rates up to 112 µL min⁻¹. These results demonstrate that this conducting polymer-based diaphragm actuator is a suitable option for active control in fully disposable autonomous microfluidics. Similar to the printed electronics field, where complex electronic devices, comprising several conducting polymer and passive layers, including patterning and alignment, are printed using both roll-to-roll printing and screen printing, we envision the DDAPA units to fabricated using these potentially cost-effective fabrication methods. The application illustrations presented show that the same non-optimized standard device can be used for different components. However, for a
specific application proper design of the DDAPA toward that application would be desired. For instance, the ratio between the radius of the active electrode and the deflection membrane is important to optimize.\textsuperscript{[32]} Likewise, the ratios of the PEDOT and ionogel layers would need to be optimized to get more stroke and/or force since the ratio between the active and passive layers of a CP actuator determines the achieved deflection and force.\textsuperscript{[33]} The DDAPA geometry, such as pump volume (i.e., diameter and thickness of the cavity), is of importance for the performance as well. The mechanical mounting of the DDAPA unit is also an important parameter to further investigate. Mounting the DDAPA unit between two rigid (plastic) frames, with openings for the buckling membranes will most probably enhance the performance. Future research will also involve optimizing the driving signal (e.g., applied voltage, frequency, and signal shape). Once the DDAPA unit has been tailored to the specific application specific characterization will be performed such as delivered pressure, long-term stability, and the suitability of working with diverse fluids. For instance, the long-term stability of the DDAPA depends on the driving potential,\textsuperscript{[34]} the adhesion between the active PEDOT-PSS layer and electrolyte layer,\textsuperscript{[23c,35]} and the interaction of the PEDOT:PSS layer with the passing sample media with which it is in close and continuous contact. Future work will include refining the fabrication process to achieve smaller sizes and creating complex structures such as 3D printed bellows to increase operational range, and the integration into the LOCs. Both the PEDOT: PSS electrodes and the microfluidic channels can be patterned down to several micrometers depending on the fabrication method used (photolithography, inkjet printing, screen printing).\textsuperscript{[36]} Coupled with the benefits of conducting polymer-based actuators such as low driving voltage, flexibility, and easy manufacturability, this simple fabrication process to construct all-polymer integrable components will contribute to the progress of fully disposable active microfluidics for applications such as disposable point-of-care devices. The DDAPA concept contributes a new alternative to conceive disposable LOCs with on-demand actuation capabilities.

4. Experimental Section

\textit{Materials}: Trimethylolpropane tris(3-mercapropionate) (TT), poly(ethylene glycol) diacrylate ($M_n = 700 \text{ g mol}^{-1}$) (PEGDA), EMIM TFSI and photoinitiator benzoin methyl ether were purchased from

![Figure 5. a) Schematic illustration of the setup for DDAPA one-way flow micropump. b) The flow rate and current versus time for the micropump coupled with PDMS check valve. c) A sequence of video frames showing liquid displacement in the outlet tube at different times. Flow rate with negative sign means a reverse flow. The applied voltage is 1.0 V and polarity changes every 90 s.](image-url)
Sigma-Aldrich. PEDOT:PSS (PH1000) was purchased from Heraeus. All chemicals were used without further purification.

**Preparation of Ionogel Films:** Ionogels with reactive thiol groups on surfaces were prepared as follows. An ionogel precursor solution was first prepared by mixing TT, PEGDA, ionic liquid, and photoinitiator (1 wt% of the total weight of TT and PEGDA) in an aluminum foil protected vial. The molar ratio between thiol groups and acrylate groups was kept as 1:1. The ionic liquid weight percentage was 50 wt% of the total weight of ionogel precursor solution. The ionogel precursor solution was then cast on a glass plate and covered with a UV transparent glass plate. The thickness was controlled by using a 0.5 mm thick silicon wafer spacer between the two glass plates. A portable UV lamp was used to cure the ionogel precursor solution to obtain free-standing ionogel films (LABINO AB, BigBeam DUO, output power 18W, duration of UV exposure 5 min).

**Fabrication and Characterization of Single Diaphragm Actuator:** The conducting polymer PEDOT:PSS was used in the diaphragm actuator fabrication. To increase the actuation performance of PEDOT:PSS, 5% by volume of DMSO (Sigma-Aldrich) solvent was added to the aqueous PEDOT:PSS solution. A compliant PDMS (Sylgard 184) mold was used to define the circular PEDOT:PSS electrodes and straight contact lines on the ionogel surface. Patterned PEDOT:PSS electrodes were obtained by depositing 0.5 mL PEDOT:PSS solution in the mold which was thereafter left to evaporate overnight at room temperature. The same procedure was repeated on the other side of ionogel film with the PDMS mold aligned to the first PEDOT:PSS electrode pattern to achieve a trilayer actuator structure.[21,37] A ring-shaped ionogel, used to confine the edge movements of the diaphragm actuator, was bonded around the PEDOT:PSS electrode area using the ionogel precursor solution as an adhesive and cured for another 5 min under UV exposure. A cycling step potential for 90 s at 1, 1.5 and 2 V was applied between the two PEDOT:PSS electrodes using a potentiostat (Ivium Technologies). The displacement at the center of the diaphragm was measured by a laser distance sensor (optoNCDT ILD1700-50).

**Fabrication and Characterization of Double Diaphragm Active Polymer Actuator:** The DDAPA was assembled from three layers: two single diaphragm actuators (fabricated as described above) and one middle ionogel layer. A hole with the same size as the PEDOT:PSS electrodes to form a chamber, and two channels were cut in the middle ionogel layer before assembly. The three layers were assembled one by one using the ionogel precursor solution as an adhesive and exposed to 5 min UV irradiation for each bonding step. The DDAPA was first studied as an injector. The chamber was filled with water, colored with a red food dye for visualization, and placed horizontally. A cycling step potential for 90 s at 1.5 V was applied and water movement inside the tube was recorded. The system was also tested as a flow regulator by connecting it to a water reservoir, which was placed above the device. Figure S4, Supporting Information, shows the measurement setup we used to measure the flow rate change when the double diaphragm actuator is used as a microvalve for flow rate regulation. An elevated beaker was used to provide the water pressure needed to propel the colored liquid. The beaker was connected to the DDAPA using a long, thin tubing which resulted in a slow flow of water drops. The water drops coming out from the output channel were counted in 10 s intervals as an indicator of the flow rate. A cycling step potential for 30, 60, and 90 s at potential of 1.5 V was applied to study the influence on flow regulation.

To achieve one-way flow and test its applicability as a pump for microfluidics, the device was coupled to 3D printed microfluidic unibody-LOCs equipped with flap and Tesla microvalves.[39,38] A cycling step potential for 90 s at 1 V and 90 s at −1 V was applied to drive the active pump and the resulting liquid flow in the microfluidic chips was video recorded. The flow rate was determined by analyzing the distance of the water movement every 10 s from the video frames using the software ImageJ. From the displaced water distance, the average flow rate during these 10 s intervals was calculated. Silicone tubing (ESSKA, Hamburg, Germany) of 1 mm internal diameter was used for connecting the diaphragm to the microfluidic chips.

**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.

**Keywords**

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