

Room Temperature Curable Copper Nanowire-Based Transparent Heater

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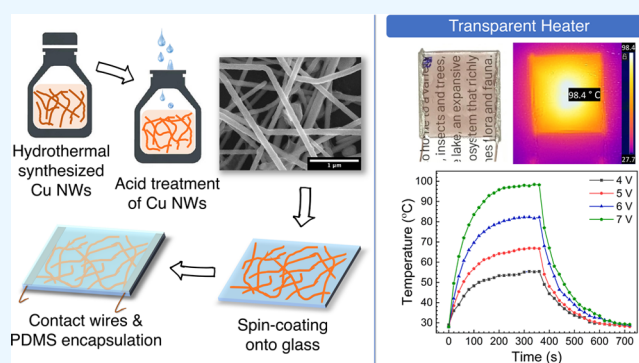
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ABSTRACT: Copper nanowires (Cu NWs) are a promising alternative to silver NWs to develop transparent conducting films (TCFs) due to their comparable electrical conductivity and relative abundance. Postsynthetic modifications of the ink and high-temperature postannealing processes for obtaining conducting films are significant challenges that need to be addressed before commercial deployment of these materials. In this work, we have developed an annealing-free (room temperature curable) TCF with Cu NW ink that requires minimal postsynthetic modifications. Organic acid pretreated Cu NW ink is used for spin-coating to obtain a TCF with a sheet resistance of 9.4 Ω /sq. and optical transparency of 67.4% at 550 nm. For oxidation protection, the Cu NW TCF is encapsulated with polydimethylsiloxane (PDMS). The encapsulated film is tested as a transparent heater at various voltages and shows good repeatability. These results demonstrate the potential of Cu NW-based TCFs as a replacement for Ag-NW based TCFs for a variety of optoelectronic applications, such as transparent heaters, touch screens, and photovoltaics.



1. INTRODUCTION

Metal nanowires (NWs) are identified as suitable alternatives for indium tin oxide (ITO) to fabricate low-cost, flexible, and transparent conductive films (TCFs).¹ Highly abundant copper (Cu) can offer conductivity on par with those of other metallic NWs, such as silver (Ag) and gold (Au), at low cost.^{2–6} However, the fabrication of TCFs with Cu NWs and their commercialization posts challenges due to the lack of robust methods that give pure Cu NWs with high oxidation stability.⁷ Similarly, the use of capping agents/reagents for ink formulation also needs to be investigated. The development of annealing free methods to fabricate Cu NW films is also a limitation. Among these limitations, issues associated with formulation and stability of the ink have been addressed to a certain extent.^{8,9} However, development of annealing free conducting Cu NW-based films is still challenging and needs further research.

Pristine Cu NW films are nonconductive due to the formation of an oxide layer on the Cu surface. One approach to reduce the oxide film is by annealing in a reducing atmosphere such as forming gas (10% H₂ and 90% Ar) at 200–300 °C.⁹ The above method results in conductive Cu NW films but is also restricted to select substrates due to the annealing conditions. A few nonannealing methods have also been reported in the literature. For instance, Zhong et al. used

laser light irradiation to make the Cu NW films conductive, and this process also enables roll-to-roll production of flexible conducting substrates.¹⁰ Tran et al. developed pulsed laser-irradiated Cu NW films prepared by polyvinylpyrrolidone (PVP)-based Cu NW ink.¹¹ Kim et al. developed Cu NW inks with silicone and polyacrylate-based additives using which Cu micropatterns, with reverse offset printing and laser irradiation, were obtained.¹² Although the pulsed laser irradiation technique offers an annealing free method and a promise for large-scale fabrication, it needs sophisticated facilities that may compromise the goal of developing low-cost conducting electrodes.

Apart from the above physical approaches, the conductivity of the Cu NW-based film can be enabled by chemical routes such as dissolving the surface oxide layer using organic acids. Mayousse et al. used glacial acetic acid treatment to fabricate a sintering free conductive Cu NW electrode.¹³ Duong et al. also developed PVP-based NW inks for the fabrication of

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conductive films using acetic acid treatment.¹⁴ In addition to acetic acid, formic¹⁵ and propionic acids⁹ have also been used for the dissolution of the oxide film on the Cu surface. However, in the above procedures, conductive films are prepared by vacuum filtration of the Cu NW ink on cellulose-based membranes followed by transfer of the film onto the required substrate and dipping in organic acids.¹³ In this approach, poor substrate adhesion was noticed and the film floated away from the substrate during or after the acid treatment.^{11,14} This can be avoided with further modification in the procedure, such as preheating the substrate and solvent dipping, which increases complexity and makes the process unsuitable for large area coating.^{11,16} In an alternative method, PVP stabilized Cu NWs were subjected to preorganic acid treatment and then applied onto heated substrates through spray coating. However, this process necessitates a substantial amount of ink and results in significant wastage.¹⁷

In this study, we present a facile method for the fabrication of copper nanowire-based transparent conductive films (TCFs). Copper nanowires were synthesized using a hydrothermal method and the formation of face-centered cubic (FCC) Cu NWs was confirmed through X-ray diffraction (XRD) analysis. The resulting NWs had a diameter of 115 ± 14 nm and were subjected to a simple propionic acid treatment. Subsequently, TCFs were fabricated through a spin coating procedure. The efficacy of the resulting TCFs was evaluated in terms of their transparency and electrical conductivity. To prevent oxidation, the resulting film was encapsulated with polydimethylsiloxane (PDMS), and these encapsulated films remained stable for several weeks under ambient conditions. These were evaluated for a potential transparent heater application and showed good stability and repeatability.

2. EXPERIMENTAL SECTION

2.1. Materials. Copper chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), D-glucose, octadecylamine (ODA), and propionic acid (AR) were obtained from SRL Chemicals, India. Isopropyl alcohol (IPA) was obtained from Merck. Poly(dimethylsiloxane) (PDMS) was prepared from Dow Corning Sylgard 184 silicone elastomer. All the reagents were used as received. For the experiments, Milli Q water ($18.2 \text{ M}\Omega \cdot \text{cm}$) was used.

2.2. Synthesis of Cu NWs. Cu NWs were synthesized using a hydrothermal method.¹⁸ In this route, 0.76 g of ODA, 0.1077 g of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$, and 0.0975 g of glucose were taken in a 100 mL borosilicate vessel, and 50 mL of Milli Q water was added to the vessel. The contents were subjected to stirring at 1800 rpm for 8 h. After continuous and vigorous stirring, the above solution turns to a stable thick blue emulsion, which was then transferred to a Teflon lined autoclave, which was heated at 120°C for 24 h followed by cooling at room temperature. The obtained reddish brown solution was transferred to a 50 mL centrifuge tube, and Cu NWs were allowed to settle down at the bottom.

2.3. Purification of Cu NWs. The Cu NW precipitate settled at the bottom of the centrifuge tube, and the supernatant was carefully decanted. Milli Q water was added to the above precipitate, which was then divided into two centrifuge tubes in equal portions and spun at 3000 rpm for 5 min. After decanting the supernatant, the precipitate was washed with IPA twice at 3000 rpm for 5 min. The obtained precipitate was washed with 5 vol % propionic acid in IPA at 3000 rpm for 5 min. The final precipitate obtained after the

acid wash was directly dispersed in 25 mL of IPA. A stable NW dispersion in IPA was obtained by sequential vortex mixing and sonication of the wires for 15 min.

2.4. Fabrication of the Transparent Conductive Film (TCF) and PDMS Encapsulation. Uniform Cu NW-based conducting films were prepared by spin coating method. The dispersion was treated with propionic acid (the final concentration of the propionic acid in the Cu NW dispersion in IPA was 5 vol %) and vortex mixed for 15 min. The obtained ink was spin-coated for 30 cycles on a pre-cleaned soda lime glass substrate ($2.5 \times 2.5 \text{ cm}^2$) at 750 rpm for 30 s using $100 \mu\text{L}$ of ink per cycle. Spin coating was carried out at room temperature, and no annealing was carried out post-spin coating. Electrical contacts on the coated film were made using silver epoxy and copper wires. To avoid oxidation, the coated film was encapsulated in PDMS prepared by mixing the base and curing agent in a 10:1 ratio. A uniform encapsulation layer was obtained by drop-casting PDMS over the spin-coated pattern and curing at 120°C for 1 h.

2.5. Characterization. XRD of the Cu NW films was recorded on a RIGAKU Miniflex 600 Benchtop sixth Generation X-ray diffractometer, with Cu $K\alpha$ radiation of wavelength 0.154 nm. High resolution scanning electron microscopy (HRSEM) images were recorded on an Apreo S SEM. The sheet resistance of the films was measured using a JANDEL RM 3000 four probe measurement system. A Keysight U3606B Multimeter was used to measure the device resistance. The optical transmittance of the film was obtained from JASCO (V-570) UV–visible spectrophotometer. For the transparent heater application, DC voltage was supplied to the fabricated device using Keysight U3606B through the Cu wire contacts. Temperature distribution on the film was obtained by recording infrared (IR) images using a thermal imaging camera, an FLIR ONE PRO with a spatial resolution of 160×120 pixels, where a pixel size is $12 \mu\text{m}$, with a temperature sensitivity of 70 mK.

3. RESULTS AND DISCUSSION

3.1. Structural and Morphological Analysis. In the alkylamine mediated approach for the synthesis of Cu NWs using glucose, an environmentally benign reducing agent, good colloidal dispersion and stability can be seen. Undisturbed sample vials of the obtained dispersion show settlement of the Cu NWs at the bottom of the vessel (Figure 1a). The supernatant, with unreacted impurities, can be easily separated

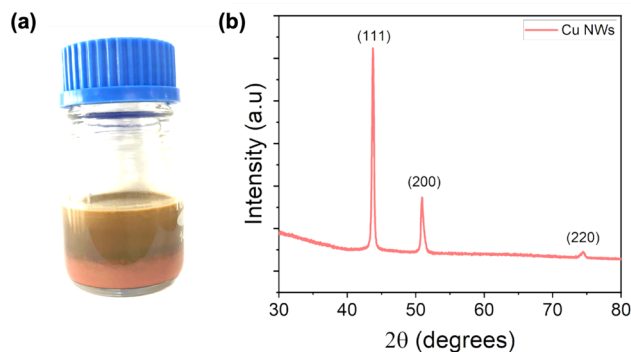


Figure 1. (a) Synthesized Cu NWs settled at the bottom of the vial, from where they can be easily separated. (b) XRD of purified Cu NWs. The peaks can be matched to pure Cu (JCPDS-04-0836).

and the NWs purified, as described in the Section 2.3. The purified Cu NWs exhibited good dispersion in IPA with metallic luster. XRD of the Cu NWs is shown in Figure 1b, and the peaks can be directly matched to the cubic phase of Cu (JCPDS-04-0836). In contrast, in the ethylenediamine (EDA) based approach to synthesize Cu NWs using toxic hydrazine as a reducing agent, the NWs form an agglomerate at the surface of the reaction solution, and their separation and purification is not trivial.^{19,20}

SEM images of the Cu NW film are shown in Figure 2. The lengths of the wires are in the micrometer range while the

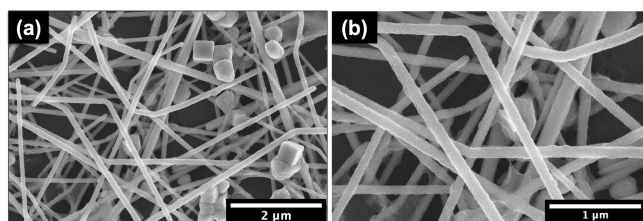


Figure 2. (a) SEM image showing micrometer-sized Cu NWs with kinks and bends and (b) magnified SEM image showing the pristine nanowires with a smooth surface.

average diameter is 115 ± 14 nm of the NWs. Kinks and bends, generally induced by the twinned defects present in the seeds, can also be seen on some of the wires, as reported earlier.^{18,21,22} Upon closer examination of the nanowire surface, as depicted in Figure 2b, it is evident that the surface is uniform and smooth, with no observable crest effects or roughness caused by oxidation, which is consistent with previous reports.^{8,23,24}

3.2. Optical and Electrical Analysis of Cu NW TCFs.

The Cu NWs dispersed in IPA were treated with 5 vol % propionic acid to remove the oxide layer formed on the NWs before spin coating. The number of coating layers in the TCF was optimized by measuring sheet resistance, and this also depends on the type of application for which these films are designed. For heater applications, TCFs with sheet resistance less than $10 \Omega/\text{sq.}$ have been shown to be suitable, with a good response time at low voltages.^{25–27} Figure 3 reveals how the sheet resistance of the Cu NW film decreases with increasing number of spin coating cycles on glass and PET substrates. This is because with an increase in the number of cycles the amount of Cu on the substrate also increases. On glass, the spin-coated Cu NW film showed good conductivity with an average sheet resistance of $48 \pm 8 \Omega/\text{sq.}$ after 20 layers. The

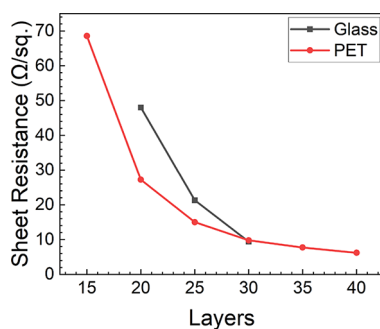


Figure 3. Decrease in sheet resistance of the Cu NWs film with increasing number of spin-coated layers on both glass and the PET substrate.

sheet resistance further decreased to $21.3 \pm 1.5 \Omega/\text{sq.}$ and $9.4 \pm 0.4 \Omega/\text{sq.}$ after 25 and 30 layers, respectively. Hence, a 30 layer film was used for transparent heater studies. Table 1

Table 1. Comparison of the TCF Properties of Cu NWs in This Work with Those in the Literature

S. No.	Material	Fabrication	Transmittance (%)	Sheet resistance ($\Omega/\text{sq.}$)
1 ²⁹	Cu NW/ alumina/ polyimide	Thermal evaporation/ ALD/spin coating	90	8
2 ³⁰	Cu NWs	Spray coating	86	25
3 ³¹	Cu NWs/ PMMA	Spray coating/spin coating	80	6.1
4 ³¹	Cu–Ni core–shell NWs	Spray coating	76	300
5 ²⁶	Cu NWs	Spray coating	77	12.6
6 ³²	Cu NWs	Spray coating	83.4	10.3
This work	Cu NWs	Spin coating	67.4	9.4

compares the fabrication process, optical, and electrical properties of Cu NW-based TCFs. The fabrication methods in the literature either have used a nonsolution based approach or require postannealing treatment, which is not required in this work. Figure 4a shows the optical transmittance spectrum

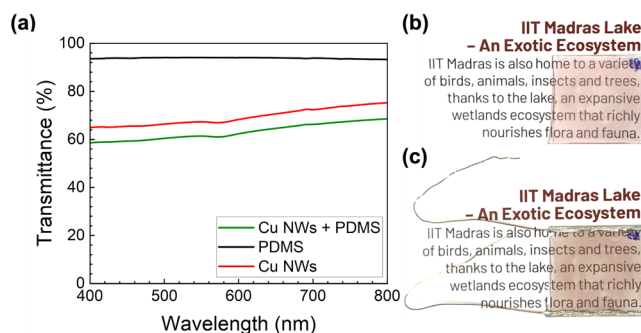


Figure 4. (a) Transmittance of a 30 layer spin-coated film, film with PDMS encapsulation, and bare PDMS. (b) Digital image of a 30 layer film on bare glass and (c) after PDMS encapsulation. There is minimal change in the overall transmittance post encapsulation.

of the device, bare PDMS, and Cu NWs. The transparent heater device showed 61.4% transparency at 550 nm. A small valley at 575 nm is observed in the transmittance spectrum, which corresponds to the surface plasmon resonance in Cu NWs. This plasmon resonance enhances the absorption of the incident wavelength, extending the application of Cu NWs as TCF in solar cells and photodetectors.²⁸ A digital photograph of the as-such spin-coated film is shown in Figure 4b, which is then converted into the heater with silver epoxy/copper wire contacts and PDMS encapsulation as seen in Figure 4c.

3.3. Transparent Heater Performance. Typically, bare Cu NW TCFs, when exposed to ambient conditions, suffer oxidation. The process is accelerated if the films are used for heater applications and exposed at higher temperatures. To improve the temporal and thermal stability of the Cu NW TCFs, they were coated with PDMS, which served as an oxidation protection layer. The device resistance (measured in a two-probe configuration between the external Cu con-

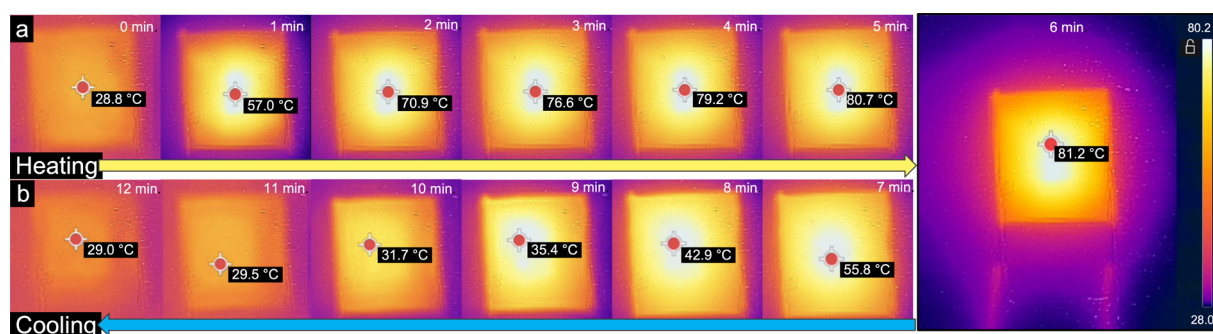


Figure 5. IR images (a) at equal intervals of 1 min indicating the attained temperature of the Cu NW film under an applied voltage of 6 V and (b) showing the same in the cooling stage when the voltage is turned off. An enlarged image at the maximum temperature (6 min) is shown along with the temperature scale bar.

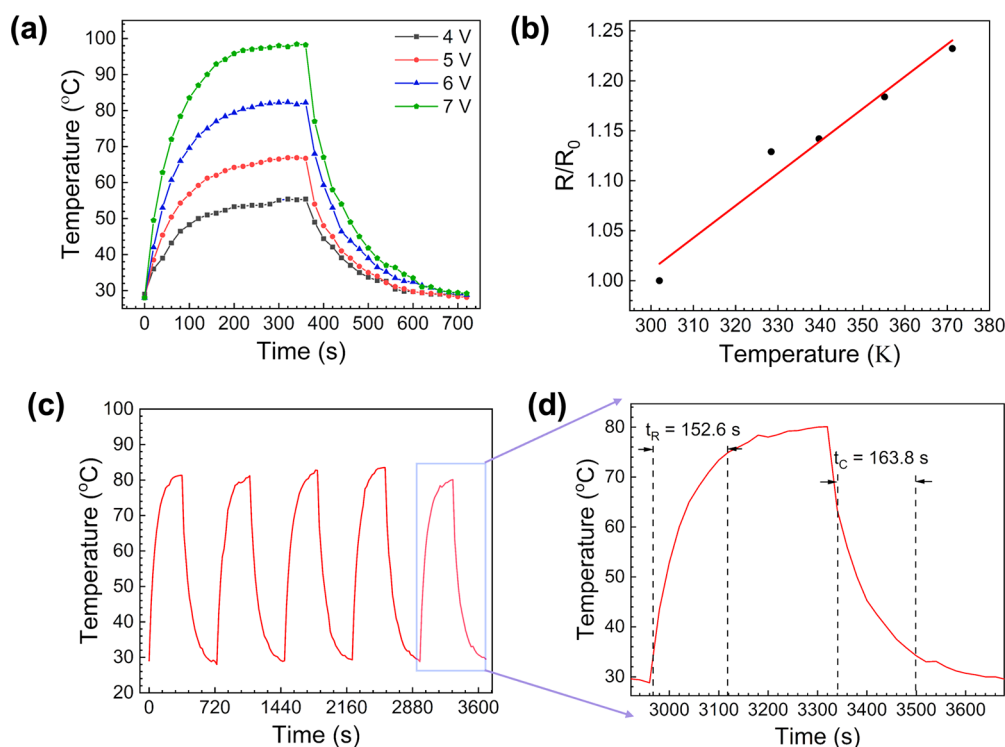


Figure 6. (a) Transient temperature response at different applied voltages. (b) Normalized resistance vs temperature plot showing positive temperature coefficient of resistance. (c) Temperature response curves at 6 V for 5 cycles, and (d) expanded curve to evaluate response and cooling time of the device.

nections) was found to be 31Ω post encapsulation. The performance of the device was assessed by joule heating. Figure 5 illustrates the IR images taken at 1 min intervals of the $2.5 \times 2.5 \text{ cm}^2$ film under an applied voltage of 6 V during heating and cooling. It shows that the film reached a temperature of 57°C in 1 min and gradually rose to 81°C in 6 min, after which the temperature was stabilized. During cooling, it took approximately 6 min for the film to return to its initial temperature, with most of the drop occurring in the first 2 min. Furthermore, the study found that there is a uniform distribution of heat across the films with the maximum temperature at the center and temperature decreasing toward the edges.

Figure 6a depicts the transient temperature response of the heater at different applied voltages, varying from 4 to 7 V. At each voltage the heater was held for a long enough time to achieve steady-state. By analyzing the resistance change during

heating, the temperature coefficient of resistance (TCR), α , was obtained. For this, the resistance at steady-state temperature, R_T , was normalized with the resistance at room temperature, R_0 , and plotted against the temperature. Figure 6b shows the linear response of the heater, in accordance with the equation given below,

$$R_T = R_0(1 + \alpha(T - T_0))$$

The TCR (α) of the Cu NWs film was calculated as $3.23 \pm 0.3 \text{ mK}^{-1}$, which is less than the TCR values reported for bulk Cu, i.e., $4.27\text{--}4.44 \text{ mK}^{-1}$.³³ This difference in the TCR values for the Cu NW film can be attributed to the junction resistance between the wires which results in higher resistive films as compared to the bulk.^{34,35} A similar behavior was seen in the case of Ag NW-based films.²⁵ A temperature increase of $\sim 54^\circ\text{C}$ was attained at 6 V, which is sufficient for applications such as defogging, defrosting, and thermochromic displays.^{36–38} A

series of successive heating and cooling of the encapsulated film was carried out to examine its cyclic thermal stability with time. The transient temperature response at 6 V for 5 cycles of 720 s per cycle is plotted in Figure 6c, where the applied voltage was turned on and off every 360 s. The heating (t_R) and cooling times (t_C) for the device at 6 V were evaluated to be 152.6 and 163.8 s, respectively, which are comparable to values reported earlier for Cu NW-based transparent heaters^{26,31,39} (Figure 6d).

The performance of the heater in terms of response time, TCR, and temperature sensitivity can be further enhanced by reducing the sheet resistance of the Cu NWs film. This can be done by increasing the number of coating layers, which will also reduce the transmittance of the device.⁴⁰ Hence, the sheet resistance and the transmittance of the device can be optimized to achieve the acquire transparent heater performance based on the specific application.

4. CONCLUSION

Highly crystalline Cu NWs were synthesized using an environmentally friendly alkylamine-mediated approach. The obtained NWs were pretreated with propionic acid to remove the surface oxide layer and uniformly dispersed in IPA. The Cu NW dispersion was spin-coated on a glass substrate for 30 cycles, forming a TCF with a sheet resistance of $9.4 \pm 0.4 \Omega/\text{sq.}$ and transparency of 67.4% measured at 550 nm. High-temperature postannealing was avoided using the acid-wash treatment. To demonstrate the applicability of the Cu NW-based TCF, a transparent heater device was fabricated with an encapsulant layer of PDMS with a TCR value of $3.23 \pm 0.3 \text{ mK}^{-1}$. The device was exposed to voltage cycles of 6 V, giving an $\sim 54^\circ\text{C}$ temperature difference with high thermal stability. The deposition of these Cu NWs can be further extended to heat sensitive flexible substrates, due to annealing free fabrication of films, widening their application to flexible optoelectronic devices. A water-based ink formulation for direct ink writing presents an eco-friendly option that enables direct patterning of transparent electronic devices. This approach is further supported by the abundance of copper compared to silver, which makes Cu NWs a more sustainable and environmentally friendly option for printed electronics.

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Notes

The authors declare no competing financial interest.

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