Characterization Method for the Swelling Effect of an Insulating Washing Agent on Silicone Rubber in Power Systems

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ABSTRACT: Insulating washing is a valid method to recover the electrical performance of silicone rubber composite insulators under accumulation contamination; however, its swelling effect may deteriorate the electrical and mechanical properties after live washing. The swelling effect of the insulating washing agent on the electrical and mechanical properties of silicone rubber and its mechanism were investigated by macroscopic and microscopic characterization methods. The results showed that the swelling degree of silicone rubber increased as the immersing time increased and the increasing trend gradually slowed down. The degradation in mechanical properties is much greater than that in electrical properties, and the tensile strength at break dropped greatly by 16.3% at 20 min of immersing. Physical defects such as holes and cracks were observed by scanning electron microscopy. The main and side chains were partially broken, and the inorganic flame retardant Al(OH)$_3$ was decreased by Fourier transform infrared spectroscopy. The damage to the microstructure caused by the swelling effect under the action of the washing agent is the main reason for the decrease in the mechanical properties of silicone rubber.

1. INTRODUCTION

In recent years, silicone rubber has been flourishing in the modern industrial domain, especially in electric power systems, due to its excellent insulation and mechanical properties, among which composite insulators made of silicone rubber have been widely used in AC and DC power equipment at different voltage levels. However, under the long-term effects of electrostatic force, the aerated solids in the air inevitably accumulate on the surface of the composite insulators, leading to the pollution flashover or abnormal flashover of the composite insulators, which poses a great threat to the safe operation of the power grid. With access to large-scale distributed power supply, the rotating inertia reserve capacity of the power grid is getting lower and lower, the voltage and frequency stability of the power grid under low carbon energy sources are becoming more and more prominent, the cost of power outage maintenance and the safety risk caused to the system are getting higher and higher, and therefore, live line washing technology has become the main means of cleaning insulators.

At present, live line washing technology has been applied to the cleaning of electrical equipment, communication equipment, and the railway system. It is divided into physical washing and chemical washing, in which, physically, live line washing mainly includes hot washing, mechanical cleaning, hot water vapor washing, and so forth, but it is gradually replaced by chemical cleaning because of its poor cleaning performance, waste, and other shortcomings. Live chemical cleaning is a kind of non-power-off cleaning method. The insulating washing agent is sprayed onto the surface of the equipment through a spray gun, and its excellent permeability and solubility cause the pollution layer to fully infiltrate, decompose, and fall off. Insulating washing agents are usually organic solvents, among which hydrocarbon cleaning agents have been widely used because of their good cleaning effect, safety, and no biological toxicity. However, it has been found that under the action of an insulating washing agent, composite insulators will produce a swelling effect, resulting in the degradation of electrical and mechanical properties of insulators, which may lead to flashover or string fracture accidents, posing a great threat to the safe operation of the power system. Recent studies have focused on the swelling behavior of rubber materials and the cleaning properties of insulating washing agents. Seeley determined the solvent–rubber interaction parameters and effective crosslink density and evaluated three methods for measuring the swelling effect.

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of silicone rubber networks using the solvent swelling method. Fukumori\textsuperscript{13} investigated the effect of tensile stress on vulcanized rubber swelling behavior using a real-time pulsed nuclear magnetic resonance method and volumetric measurements. Neff\textsuperscript{14} proposed a continuous mechanical model based on Helmholtz free energy density and performed finite element simulation analysis on the swelling behavior of natural rubber under different boundary conditions. Choe\textsuperscript{15} determined the effect of insulating oil viscosity on the mechanical and electrical properties of silicone rubber after swelling and chemically analyzed the swollen silicone rubber by thermogravimetric analysis. Ca\textsuperscript{16} used the gas chromatography–mass spectrometry (GC–MS) technique to compare the main components and volatility of four washing agents, as well as the physical and chemical properties of each component of the washing agent. Zhao\textsuperscript{17} compared the effect of different cleaning methods on the cleaning effect of cleaning and repairing agents on the outer surface of silicone rubber insulators by the hardness test, EDS test, and aging test. Although the swelling effect of rubber materials has been studied for 60 years, little attention has been paid to the swelling effect of insulating washing agents on composite insulators and their adverse effects.

In this paper, the swelling test, hydrophobicity test, and tensile test are carried out on silicone rubber to evaluate the effect of the insulating washing agent on its electrical and mechanical properties under the swelling effect. Microscopic characterization methods such as scanning electron microscopy (SEM)\textsuperscript{19} and Fourier transform infrared (FTIR) spectroscopy\textsuperscript{20} are combined to further analyze its microscopic influence mechanism, providing an experimental and theoretical basis for the safe implementation and improvement of live washing of electrical equipment made of silicone rubber materials.

2. EXPERIMENTAL SECTION

2.1. Sample. A silicone rubber material with exactly the same formulation as the shed of the composite insulator was selected as the research object. It is a three-dimensional network structure with linear polydimethylsiloxane as the raw rubber, supplemented with the flame retardant Al(OH)\textsubscript{3} and other additives cross-linked by high-temperature vulcanization. The molecular structure of the vinyl polydimethylsiloxane is shown in Figure 1. In the swelling test, hydrophobicity test, and EDS test, and FTIR spectroscopy test, it was cut into disc-shaped samples with a 130 ± 0.5 mm diameter and a 2 ± 0.2 mm thickness. In the fracture tensile strength test, according to the standard GB/T 528-2009,\textsuperscript{20} it was cut into dumbbell-shaped samples with 25.0 ± 1.0 mm length, 4.0 ± 0.1 mm width, and 2.0 ± 0.2 mm thickness of the narrow part. The disc-shaped and dumbbell-shaped samples are shown in Figure 2.

The insulating washing agent EL99-110 used in the test is a common alkane washing agent, its main components are n-heptane, cyclohexane, undecane, N-methylpyrrolidone, and antistatic agent. Its core performances are shown in Table 1, with the advantages of high voltage resistance, fast volatility, and high flash point, in line with the standard GB/T 25098-2010.\textsuperscript{19}

\begin{table}[h]
\centering
\caption{Core Performances of Insulating Washing Agent EL99-110}
\begin{tabular}{|c|c|}
\hline
Performance index & Measured results \\
\hline
Appearance & Transparent and uniform, without stratification and precipitation \\
Volatilization rate & 108.01 g/(h·m\textsuperscript{2}) \\
Residual amount after 24 h of volatilization & 1.32 × 10\textsuperscript{-4} g/cm\textsuperscript{2} \\
Power frequency breakdown voltage & 35.91 kV \\
PH value & 7.4 \\
Insulation resistance & 1.8 × 10\textsuperscript{11} Ω \\
Density & 0.76 g/cm\textsuperscript{3} \\
\hline
\end{tabular}
\end{table}

2.2. Experimental Methods. 2.2.1. Swelling Test. Silicone rubber molecules are three-dimensional network structures. The dynamic process of volume expansion of silicone rubber caused by the infiltration of small molecules in the washing agent solution into the gap of its molecules is swelling. The swelling degree can be expressed by the swelling index \( S_1 \)\textsuperscript{21} and diameter change.

\[ S_1 = \frac{(M_2 - M_0)}{M_1} \] (1)

where \( M_0 \) is the mass of the weighing bottle, \( M_1 \) is the mass of the sample before swelling, and \( M_2 \) is the sum of the mass of the sample and the weighing bottle after swelling.

The swelling test was carried out according to the standard HG/T 3870-2008.\textsuperscript{21} The specific steps of the test were as follows. First, the clean silicon rubber disc-shaped samples were put into an oven at 50 ± 2 °C for complete drying and then taken out, and the mass and diameter of the samples were measured using an electronic balance and a vernier caliper, respectively. The samples were then completely immersed in the washing agent for swelling treatment, and the test temperature was controlled at 30 ± 1 °C. After swelling for different times, the samples were taken out, and the washing agent on the surface was sucked dry with clean and dry filter paper. The mass and diameter of the sample after swelling were measured again. Finally, the swelling index \( S_1 \) and diameter changes of the samples at different swelling times were measured. The swelling degree was calculated to comprehensively evaluate the swelling characteristics of silicone rubber in the washing agent. Considering that the live washing time generally does not exceed 20 min, and it
2.2.2. Hydrophobicity and Hydrophobicity Transfer Test. Hydrophobicity is an important index of the antipollution flashover performance of insulating materials. The hydrophobicity test was carried out according to the standard GB/T 24622-2009, and the static contact angle method was adopted. The static contact angle is the angle between the tangent line of the edge of the water droplet and the surface of the material. The larger the static contact angle, the smaller the contact surface between the water droplets and the material, and the better the hydrophobicity of the material. The specific steps of the test were as follows. First, the samples were divided into two groups, one of which was only for swelling treatment and the other group for artificial contamination treatment after swelling; the swelling time was consistent with the swelling test. In the contamination group, the samples were coated with 0.1 mg/cm² NaCl and 0.5 mg/cm² kaolin and fully transferred for 96 h. Then, 30 μL of deionized water was slowly and continuously added to the surface of the two groups of samples and horizontally filmed using a high-definition camera. Finally, the static contact angles of five water droplets on the surface of the sample at different swelling times were recorded, and the average value was taken for each measurement. The artificial contamination effect and static contact angle θ measurement are shown in Figure 3.

![Figure 3. Artificial contamination effect and static contact angle θ measurement.](image)

2.2.3. Tensile Test. The tensile test was carried out on the standard GB/T 528-2009. A microcontrolled electrohydraulic servo universal testing machine produced by a company in Jinan was used for the test. The samples dried and treated at different swelling times were placed on the universal testing machine and stretched to break at a rate of 500 ± 50 mm/min. The accuracy of the measurement of the maximum tensile force was ensured by uniform stretching, and the maximum tensile force of the samples was recorded in the breaking process. The temperature and swelling time of the test are consistent with that of the swelling test. Tensile strength at break $T_{SW}$, an evaluation index of mechanical properties, was selected to evaluate the influence of the washing agent on the mechanical properties of silicone rubber samples at different swelling times.

$$T_{SW} = \frac{F_b}{WH}$$  \hspace{1cm} (2)

where $F_b$ is the maximum tensile force of the sample from tensile to break and $W$ and $H$ are the width and thickness of the narrow part of the sample, respectively.

2.2.4. SEM Test. SEM can be used to microscopically image the surface morphology of materials on the basis of the interaction between electrons and materials. To further analyze the effect of swelling on the surface microscopic morphology of silicone rubber, the dried samples and samples treated at different swelling times were fixed on the sample clip of the electron microscope by conductive silver adhesive for vacuum treatment, and then the surface morphology was scanned. According to the swelling characteristics of silicone rubber, the swelling time was selected as 1, 4, and 10 h, respectively, and the test temperature was $30 \pm 1 ^\circ C$. The Jsm-7500f scanning electron microscope was used for the test, which has a resolution of up to 1.0 nm and can achieve electron optical magnification of the sample surface from 25 to 1,000,000 times at an accelerating voltage of 15 kV.

2.2.5. FTIR Spectroscopy Test. FTIR spectroscopy is a method based on chemistry and metrology that achieves a qualitative and quantitative analysis of the structure and chemical bonds of the measured object according to the position and shape of the absorption peaks of characteristic functional groups in the FTIR spectra. The magnitude of the absorption peak corresponding to the characteristic functional group is positively correlated with the content of that group. The higher the absorbance of the absorption peak in a certain band, the stronger the ability to absorb spectra in the band, and the higher the content of the corresponding characteristic functional group in the band. To determine the changes in the microstructure of silicone rubber before and after swelling with the washing agent, the FTIR spectroscopy test was carried out on samples dried and treated at different swelling times, respectively, and the test temperature and swelling time were consistent with the SEM test. The Fourier infrared spectrometer was manufactured by the American PE company, with a spectral range of 350–8300 cm⁻¹ and a resolution better than 0.4 cm⁻¹.

3. RESULTS AND DISCUSSION

3.1. Effect of the Washing Agent on Swelling Characteristics of Silicone Rubber. The swelling index $S_1$ and diameter change in silicone rubber reflect the influence of the washing agent on its swelling degree. The curves of the swelling index $S_1$ and diameter of the silicone rubber sample with swelling time under the action of the washing agent are shown in Figures 4 and 5, respectively. Figures 4 and 5 show that the swelling index and diameter of the sample increased with increasing swelling time, and the growth trend gradually slowed. After 20 min of swelling with the washing agent, the swelling index and diameter growth rate of silicone rubber were 6.8 and 4.7%, respectively. While after 4 h of swelling, the swelling index and diameter growth rate of silicone rubber were only 32 and 15%. The reason for this phenomenon is that at the early stage of swelling of silicone rubber, due to the similar solubility, the alkanes in the washing agent were influenced by the polysiloxane group, methyl group, and other oleophilic groups at a faster speed into the silicone rubber,
produced molecular force with the oleophilic group inside, and existed in the form of a bound state in the silicone rubber. As the swelling proceeded, the spacings between its molecular chains and chain segments were expanded, and the small molecules of the washing agent penetrated the internal gaps of silicone rubber in the form of a free state. As the gaps were gradually filled, the infiltration and precipitation of small molecules of the washing agent would be toward the direction of dynamic balance, so the swelling rate gradually slowed down.

In practical engineering, the feasibility of the insulating washing agent can be first judged by the changes in the mass and diameter of silicone rubber. Also, it is necessary to take the swelling characteristics as a group of evaluation indicators in the development of the operating rules for the live washing of composite insulators.

3.2. Effect of Swelling on Hydrophobicity and Hydrophobicity Transfer. In order to explore the effect of swelling on the hydrophobicity of silicone rubber under the action of a washing agent, the static contact angle method was used. Figure 6 shows the curve of the static contact angle $\theta$ of clean and artificially contaminated silicone rubber samples with swelling time. It illustrated that the static contact angle $\theta$ of silicone rubber had no obvious relationship with swelling time. Before and after swelling, the $\theta$ of clean silicone rubber fluctuated in the range of 92–98°, and the $\theta$ of artificial contaminated silicone rubber fluctuated in the range of 119–128°, indicating that the hydrophobicity of silicone rubber increased after contamination, but the swelling had no obvious effect on its hydrophobicity and hydrophobicity transfer.

Its excellent hydrophobicity is mainly due to the migration of hydrophobic groups methyl in the side chain and hydrophobic siloxane molecules in the main chain of the molecular chain. After swelling of clean silicone rubber, although the spacings between the molecular chains and chain segments were extended, the hydrophobic groups were not damaged, so the swelling had no obvious effect on its hydrophobicity. After contamination, small siloxane molecules inside the silicone rubber diffused into the contamination layer, enhancing the surface hydrophobicity, but the structure of the hydrophobic groups did not change, so the swelling time had no significant effect on its hydrophobicity transfer.

3.3. Effect of Swelling on Tensile Strength at Break. In the tensile test, the tensile strength at break $T_{sb}$ was used to evaluate the effect of swelling on the mechanical properties of silicone rubber. Figure 7 shows the curve of the tensile strength at break $T_{sb}$ with a swelling time of the silicone rubber sample. Under the swelling action of the washing agent, the mechanical properties of silicone rubber were significantly reduced. The tensile strength at break decreased with the increasing swelling time, and the decreasing trend gradually became slower. When the swelling time was 20 min, 40 min, 1 h, 2 h, and 4 h, it decreased by 16.3, 26.4, 30.1, 34.8, and 37.3% compared to 3.28 MPa before swelling.

This is because after the silicone rubber absorbed the washing agent and swelled, the degree of its expansion increased after contamination, but the swelling had no obvious effect on its hydrophobicity and hydrophobicity transfer.
increased, and the spacings between molecular chains increased, leading to the breakage of the molecular chains, thus reducing the tensile strength at break. At the early stage of swelling, the molecular gap of silicone rubber could contain washing agent molecules, and more washing agent molecules entered into its interior, resulting in obvious breakage of its molecular chains. However, in the subsequent swelling stage, the silicone rubber-absorbed washing agent gradually reached saturation, and the molecular chains basically no longer broke; therefore, its tensile strength at break at 4 h of swelling only decreased by 14.7% compared to that at 40 min.

3.4. Effect of Swelling on the Surface Morphology of Silicone Rubber. In order to analyze the damage of the surface morphology of silicone rubber caused by swelling under the action of the washing agent, the scanning electron microscopy (SEM) test was carried out. The SEM images of the surface morphology of dry silicone rubber samples magnified by 1000—10,000 times are shown in Figure 8. As can be seen in Figure 8, the surface of the dry sample was flat, had a complete structure, and had no microcracks or holes. The inhomogeneous fine particles on the surface of the sample were inorganic flame retardant Al(OH)$_3$ in silicone rubber. The SEM images of the sample surface after swelling for 1, 4, and 10 h in the washing agent are shown in Figures 9—11. It can be seen in Figures 9—11 that after swelling in the washing agent for 1 h, the surface of the sample became rough, and small holes appeared locally, accompanied by a large number of small precipitates. The structure between precipitates was loose and disorderly, and there were a lot of gaps. After swelling for 4 h, the number of small holes and the volume of precipitates increased significantly. After swelling for 10 h, cracks of different sizes appeared on the surface of the sample, and the surface morphology damage was the most severe.

Table 2. Bands Corresponding to the Main Characteristic Functional Groups of Silicone Rubber in the FTIR Spectra

| band (cm$^{-1}$) | characteristic functional group | band (cm$^{-1}$) | characteristic functional group |
|-----------------|--------------------------------|-----------------|--------------------------------|
| 700             | Si(CH$_3$)$_3$                  | 1240—1280       | Si—CH$_3$                      |
| 760—840         | Si(CH$_3$)$_2$                  | 2960            | C—H in —CH$_3$                 |
| 1000—1100       | Si—O—Si                        | 3200—3700       | —OH in Al(OH)$_3$              |
seen in Table 2, the band of 1000–1100 cm\(^{-1}\) can reflect the content of Si–O–Si in silicone rubber, the bands of 700, 760–840, and 1240–1280 cm\(^{-1}\) can reflect the content of Si(CH\(_3\))\(_2\), Si(CH\(_3\))\(_3\), and Si–CH\(_3\), respectively, in silicone rubber, and the band of 3200–3700 cm\(^{-1}\) can reflect the decomposition of flame retardant Al(OH)\(_3\).

The FTIR spectra of the silicon rubber sample in the dry state are shown in Figure 12, and the peak values of absorption peaks of each characteristic functional group remain unchanged, indicating that no new chemical bonds were formed on the surface of the silicone rubber under the action of the washing agent; that is, no chemical reaction occurs. After swelling, the peak values of absorption peaks of some characteristic functional groups in the samples showed a significant decrease, and the decreasing amplitude increased with the increasing swelling time, while the peak value of absorption peak of C–H in −CH\(_3\) increased slightly.

After swelling with the washing agent for 10 h, the peak value of absorption peak of C–H in −CH\(_3\) increased from 0.154 to 0.248, indicating that the presence of alkanes, the main component of the washing agent, was detected inside the silicone rubber, proving the swelling of the silicone rubber in the washing agent. There are free small-molecule siloxanes (SiO(CH\(_3\))\(_2\))(\(n\) = 3–21) in silicone rubber for composite insulators, which are mainly residual uniformly distributed uncross-linked siloxane oligomers during the manufacturing process, siloxane oligomers generated by chain breakage caused by aging, and siloxane oligomers artificially added to enhance hydrophobicity. The peak values of absorption peaks of Si–O–Si, Si(CH\(_3\))\(_3\), Si(CH\(_3\))\(_2\), and Si–CH\(_3\) decreased from 0.902, 0.583, 0.910, and 0.575 in a dry state to 0.693, 0.347, 0.716, and 0.443, respectively, indicating that the main- and side-chain contents of the silicone rubber decreased after swelling. The reason is that part of the free small-molecule siloxanes in the silicone rubber migrated into the washing agent. With the increase of small molecules of washing agent penetrating the interior of silicone rubber, the migration of small-molecule siloxanes was hindered, so the decrease in the rate of the content of Si–O–Si, Si(CH\(_3\))\(_3\), Si(CH\(_3\))\(_2\), and Si–CH\(_3\) characteristic functional groups gradually slowed down with the increasing swelling time. The peak value of absorption peak of −OH decreased from 0.091 in a dry state to 0.035, indicating that the flame retardant Al(OH)\(_3\), which is one of the main fillers of silicone rubber, was decomposed and precipitated during the swelling process. Combined with the SEM image, it can be seen that the small precipitates generated after the swelling of silicone rubber were small-molecule siloxanes migrated out of silicone rubber and particles produced by the decomposition of the flame retardant Al(OH)\(_3\). Therefore, the smoothness of the silicone rubber surface was reduced, and even various defects were formed, which ultimately led to the decline in its mechanical properties.

As can be seen in Figure 13, the characteristic functional groups of the sample were the same as those of the dry sample after swelling with the washing agent for different times, and no new groups were generated, and the bands of each characteristic functional group remained unchanged, indicating that no new chemical bonds were formed on the surface of the silicone rubber under the action of the washing agent; that is, no chemical reaction occurs. After swelling, the peak values of absorption peaks of some characteristic functional groups in the samples showed a significant decrease, and the decreasing amplitude increased with the increasing swelling time, while the peak value of absorption peak of C–H in −CH\(_3\) increased slightly.

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The literature pointed out that the smaller the area of the absorption peaks of Si–CH\(_3\) and Si–O–Si, the higher the aging degree of silicone rubber. The areas of the absorption peaks of Si–O–Si and Si–CH\(_3\) before and after swelling were calculated, respectively, according to the FTIR spectra of the silicone rubber surface before and after swelling with the washing agent, as shown in Figure 14. The relative content of Si–O–Si and Si–CH\(_3\) decreased by 26 and 33% after swelling for 10 h. The results showed that the aging process of silicone

![Figure 12. FTIR spectra of dry silicone rubber.](http://pubs.acs.org/journal/acsodf)

![Figure 13. FTIR spectra of silicone rubber after swelling with the insulating washing agent for different times.](http://pubs.acs.org/journal/acsodf)

| Characteristic Functional Group | Absorbance at 700 | Absorbance at 760–840 | Absorbance at 1000–1100 | Absorbance at 1240–1280 | Absorbance at 2960 | Absorbance at 3200–3700 |
|--------------------------------|------------------|---------------------|----------------------|---------------------|-----------------|----------------------|
| dry                           | 0.583            | 0.910               | 0.902                | 0.575               | 0.154           | 0.091                |
| swelling for 1 h              | 0.456            | 0.824               | 0.810                | 0.517               | 0.220           | 0.055                |
| swelling for 4 h              | 0.386            | 0.758               | 0.737                | 0.468               | 0.235           | 0.045                |
| swelling for 10 h             | 0.347            | 0.716               | 0.693                | 0.443               | 0.247           | 0.035                |
rubber was accelerated under the swelling action of the washing agent, and the longer the swelling time, the more serious the aging of silicone rubber.

4. CONCLUSIONS

In this paper, the macroscopic influence of insulating washing agent on silicone rubber was obtained through the swelling test, hydrophobicity test, and tensile test, and its microstructure change was further discussed with microscopic characterization method, which can provide a reference for the study of live washing of silicone rubber. The following conclusions were obtained from the tests:

(1) When the composite insulator was cleaned with the insulating washing agent, the swelling effect of silicone rubber occurred. The swelling of silicone rubber was mainly reflected in a significant increase in volume and mass. The growth of the swelling rate increased with swelling time, but the growth rate gradually slowed down.

(2) Its hydrophobicity and hydrophobicity transfer had no obvious change after swelling, but its hydrophobicity increased after contamination.

(3) Its mechanical properties were greatly reduced after swelling. Swelling in a short time leads to a great decline in mechanical properties, and the decline in mechanical properties gradually became slower as the swelling time increased.

(4) After swelling with the washing agent, the surface morphology changed. Its surface became rough, covered by a large number of small precipitates, and physical defects such as holes and cracks appeared. With increasing swelling time, the damage to the silicone rubber surface became more severe.

(5) The FTIR spectroscopy test showed that with increasing swelling time, more uncross-linked free small-molecule siloxanes in silicone rubber migrated into the washing agent, the silicone content decreased, and the inorganic flame retardant Al(OH)$_3$ decomposed and precipitated, and its aging degree was aggravated. This is also the reason for the reduction of the smoothness of the silicone rubber surface and the formation of various defects, which ultimately led to the decline in its mechanical properties.

**ASSOCIATED CONTENT**

* Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.2c05367.

Swelling analysis equations; SEM image of silicone rubber before and after swelling; and FTIR spectra of silicone rubber before and after swelling (PDF)

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**Notes**

The authors declare no competing financial interest.

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