Dielectric Properties of Silicothermic Reduction Chromite in the Microwave Field

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ABSTRACT: The microwave absorption properties of chromite and the feasibility of microwave reduction chromite have been discussed. The results show that as the density increases, the dielectric properties of materials increase. The dielectric properties are the best (the value around 4.2) when the silica ratio is 0.5. Microwave penetration depth shows that chromite and the mixture have good wave absorption properties.

1. INTRODUCTION

Iron and chromium symbiotically form ferrochrome spinel (FeO·Cr₂O₃), whose mineralogical name is chromite.1,2 Chromite is used in metallurgy, refractory, and chemical industry and is mainly used in ferrochrome alloy and chrome metal in the metallurgical industry. In recent years, with the economy and national defense advancing, the demands of stainless steel keep growing. Chromite is an important mineral in the ferrochrome alloy industry. Because chromite resources have many problems such as small-scale mineral deposits, scattered distribution, and low grade, few of them are exploitable.3−7 Ferrochromium alloy is indispensable in the special steel industry, which can produce high-strength steel.8 Currently, there are two main processes for low-carbon ferrochromium alloy making:9 oxygen blowing and electro-silicothermic process. Both of them require agglomerating before smelting,10−13 and the long process flow, high energy consumption, and high carbon emission restrict the development of high-strength steel industry.8,14−17 Therefore, it is necessary to explore a new process for smelting low-carbon ferrochromium.

As a new type of clean and efficient heating energy, microwave energy has attracted wide attention in recent years, and microwave heating has been widely used in beneficiation and metallurgy process. As a novel technology, microwave heating has the following advantages that cannot compare: fast heating rate, integrity heating, selective heating, volume heating, environment friendliness, and high effectiveness. It can also reduce the reaction temperature, shorten the reaction time, and promote energy conservation.18−26 With these advantages, microwave heating is introduced to the silicothermic reduction chromite process. In order to determine the microwave thermal reduction process parameters, the dielectric properties of materials should be analyzed.

The dielectric properties (ε′, ε″, and tan δ) of materials are used to describe the microwave absorption characteristics in the microwave heating process.27−31 The relative permittivity is (ε) composed of a real part (ε′) and an imaginary part (ε″). The real part of the relative permittivity is known as the dielectric constant, and it indicates the material’s polarization property in electromagnetic waves. Also, the imaginary part is the dielectric loss factor, and it represents the material’s ability of converting microwave energy into internal energy.32 These parameters can be expressed as ε = ε′ − jε″. The loss tangent (tan δ) is a parameter used to describe how well a material absorbs microwave energy. It indicates the ratio of the dielectric loss factor to the dielectric constant (tan δ = ε″/ε′). The dielectric constant and loss tangent are functions of frequency, uniformity, and material temperature.33 Another important parameter in microwave heating is the penetration depth. Penetration depth (D_p) is a basic material property that determines the microwave penetration depth.34 It can be observed that the dielectric properties of materials determine...
the microwave absorption property of materials and the interaction between materials and microwave. The changes in dielectric properties with parameters such as temperature composition will also cause the change in parameters related to microwave interaction.

In this paper, the cavity perturbation method was applied to measure the dielectric properties of chromite with different apparent densities, silica ratios, and alkalinity. A simple equation is developed to quantify the penetration depth of microwave in different conditions. The particle size of materials in microwave heating can be optimized by the penetration depth and determine the microwave absorption properties of the mixture.

2. RESULTS AND DISCUSSION

2.1. Permittivity of Chromite and Ferrosilicon. The temperature rise curve of chromite powder (50 g, 200 mesh) and ferrosilicon powder (50 g, 200 mesh) is shown in Figure 1.

![Figure 1. Temperature curve of chrome and ferrosilicon in the microwave field.](image)

(microwave frequency is 2450 MHz, and microwave power is 1.5 kW). It can be concluded that the chrome powder reaches 1200 °C in 300 s, and the average heating rate is 4 °C/s. The ferrosilicon alloy powder can be heated to 1200 °C in 270 s, and the average heating rate is 4.44 °C/s. The dielectric properties of chromite and ferrosilicon are analyzed by a dielectric characteristic test system, and the results are shown in Figure 2a. Figure 2b indicates that the $\varepsilon'$, $\varepsilon''$, and $\tan \delta$ increase with temperature. The result of ferrosilicon shows that the $\varepsilon'$ increases with the temperature, but the $\varepsilon''$ and $\tan \delta$ increase with the temperature, and the $\varepsilon''$ and $\tan \delta$ stabilized after 600 °C (Figure 2a). Obviously, the dielectric analysis and the temperature rising behavior show that chrome and ferrosilicon both have good microwave absorption and that ferrosilicon is better than chrome.

The cavity of the microwave heating equipment is designed to be metal plate wall because the metal plate can completely reflect electromagnetic wave in theory. When a certain thickness of the material is laid flat on a metal plate and the electromagnetic wave is incident vertically, a part of the electromagnetic wave will be reflected back. The relationship between the reflection power and incident power can be described by the reflection loss (RL) under this condition. That is to say, the reflection loss describes the material absorption performance from the angle of energy loss in the process of electromagnetic wave reflection. Based on the transmission line theory, the reflection loss of the mixture sample at different temperatures can be calculated by taking the dielectric parameters into eq 1:

$$RL = 20 \log \left| \frac{\mu_r}{\varepsilon_r} \tan \left( \frac{2\pi f \mu_r \varepsilon_r d}{c} \right) - 1 \right|$$

where $c$ is the speed of light in free space ($c = 3 \times 10^8$ m s$^{-1}$), $f$ is the frequency of microwave ($f = 2450$ MHz), $\mu_r$ is the relative permeability and assumed to be 1 in the RL calculation, and $d$ is the thickness of the materials loaded on the metallic plate ($d = 0.01$ m).

Figure 3 shows the curve of reflection loss and temperature of chromite powder and ferrosilicon powder. It can be seen that the reflection loss decreases with the increase in temperature. Clearly, it can be divided into three parts for analysis. In the first stage (room temperature to 400 °C), the reflection loss of chromite and ferrosilicon powder tends to be stable. During the period from 400 to 800 °C, the reflection loss of both decreases. In the three stages (800–1100 °C), the decline trend of reflection loss of ferrosilicon is obviously better than that of chromite. Thus, the utilization ratio of ferrosilicon to microwave energy is higher than chromite.

2.2. Effect of Temperature on Dielectric Properties. The effect of temperature on mixture’s dielectric properties is analyzed in this part. The results indicate that there are three steps and illustrate the temperature dependence of the real part ($\varepsilon'$) and imaginary part ($\varepsilon''$) of the complex relative permittivity of chromite at 2450 MHz. In the first stage, $\varepsilon'$ and $\varepsilon''$ change slightly at 25 to 400 °C. In the second stage, at 400–800 °C, the values of $\varepsilon''$ and $\tan \delta$ increase significantly. In the last stage, the values of $\varepsilon'$, $\varepsilon''$, and $\tan \delta$ gradually stabilized after 800 °C.

The variation of $D_p$ with temperature is shown in Figure 4b. It is obvious that the $D_p$ value of the mixture is smaller at room temperature, and since the mixture of calcium oxide belongs to the semiconductor, its microwave absorption is relatively low. Meanwhile, it is seen that the mixture has a large $D_p$ (203 cm at 2450 Hz) at 300 °C, indicating a very slow microwave dissipation inside the oxide. This is due to the small permittivity of the mixture at low temperatures. As the temperature increases, the $D_p$ value decreases rapidly mainly because of a significant increase in permittivity. Since the relative permittivity is larger, the permittivity dominates the variation of microwave penetration depth. Figure 4b indicates that the $D_p$ value is less than 18 cm beyond the temperature of 600 °C. This shows that the mixture undergoes a transition from a microwave transparent material to a good microwave absorber with increasing temperature.

The variation of reflection factors with temperature is shown in Figure 5. It can be seen that as the temperature increases, the reflection loss decreases. Obviously, it can be divided into three parts for analysis. In the first stage (room temperature to 400 °C), the value of RL tends to stabilize. Next, in the second stage (400–800 °C), the value decreases, that is to say, it improved the microwave energy utilization, and in this stage, the oxide is reduced. In the third stage (800–1100 °C), the value of RL did not change further, and the oxide reduced to metals, resulting in low microwave energy utilization. There-
fore, the decrease in reflection loss value indicates that the utilization of microwave energy increases.

Figure 6 shows that with the temperature increase, the materials are gradually reduced. Combining with Figures 7 and 4, in the range of 400–500 °C, the mixture of Fe₂O₃ shows a reduction reaction (eq 2). In the range of 600–750 °C, Fe₃O₄ is converted into FeO (eq 3). Also, from 750 to 900 °C, FeO is converted into Fe (eq 4). The values of $\varepsilon'$, $\varepsilon''$, and $\tan \delta$ gradually stabilized after 800 °C. This is because the reaction enters the final reduction stage, completely converting the unconverted Fe₂O₃ into Fe₃O₄, Fe₃O₄ into FeO, and finally FeO into Fe from 1100 to 1300 °C. As the temperature continues to increase, about 1300 °C, Cr₂O₃ is converted into Cr (eq 5). The reaction can be expressed as follows:

$$\begin{align*}
6\text{Fe}_2\text{O}_3 + \text{Si} &= 4\text{Fe}_3\text{O}_4 + \text{SiO}_2 \\
2\text{Fe}_3\text{O}_4 + \text{Si} &= 6\text{FeO} + \text{SiO}_2 \\
2\text{FeO} + \text{Si} &= 2\text{Fe} + \text{SiO}_2 \\
2/3\text{Cr}_2\text{O}_3 + \text{Si} &= 4/3\text{Cr} + \text{SiO}_2
\end{align*}$$

The results show that the above reaction is exothermic, and the dielectric properties also change.

2.3. Effect of Apparent Density on Dielectric Properties.

The effects of apparent density on the dielectric constant ($\varepsilon'$), dielectric loss ($\varepsilon''$), and loss tangent ($\tan \delta$) are presented in Figure 8.

Figure 8 shows that the dielectric properties ($\varepsilon'$, $\varepsilon''$, and $\tan \delta$) of the mixture have a good linear relationship with the apparent density at room temperature. It can be seen that the dielectric properties ($\varepsilon'$, $\varepsilon''$, and $\tan \delta$) increase with apparent density, which indicates that the ability of microwave absorption increases. More microwave energy that is converted to thermal energy may contribute to the improvement of dielectric properties. The linear regression equations and correlation coefficient ($R^2$) are shown in Table 1.

The microwave penetration depth ($D_p$) was measured as the distance from the surface to the inner part of the material, where the microwave field intensity was reduced to 1/e of the original field intensity. This value can be expressed as follows: $\delta = \frac{\lambda}{4\pi}$. 

Figure 2. Temperature dependence of dielectric property of (a) chromite powder and (b) ferrosilicon powder.

Figure 3. Curve of reflection loss of chromite and ferrosilicon at different temperatures.
πε = −ε″

where $C$ is the speed of light ($c = 3 \times 10^8$ m/s$^{-1}$), $f$ is the frequency (Hz), $\lambda_0$ is the microwave wavelength in free space, $\lambda_0 = 12.24$ cm (2450 MHz), $D_p$ is the microwave penetration depth, $\varepsilon'$ is the dielectric constant, and $\varepsilon''$ is the dielectric loss factor.

\[
D_p = \frac{C}{2\pi f \sqrt{2\varepsilon' - 1 - \left(1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2\right)^{1/2}}} = \frac{\lambda_0}{2\sqrt{2\pi} \sqrt{\varepsilon' - 1 - \left(1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2\right)^{1/2}}}
\]

(6)

The microwave penetration depth of the mixture under different apparent densities is shown in Figure 9. The results show that the $D_p$ decreases as the apparent density increases. Equation 6 indicates that with the increase in the $\varepsilon'$ and $\varepsilon''$, the $D_p$ decreases. When the apparent density is constant, the $D_p$ remains basically unchanged due to the fact that the energy

Figure 4. Effect of temperature on mixture’s dielectric property: (a) dielectric constant and (b) penetration depth.

Figure 5. Curve of reflection loss of mixture at different temperatures.

Figure 6. Thermogravimetric curve of the mixture.

Figure 7. Gibbs of oxide.
density of the materials surface is greatest when the microwave enters the materials. The microwave energy is decaying when the microwave penetrates the material and transforms into thermal energy, which determines the penetration ability of microwave to materials. Based on Table 1, the material absorbed more microwave energy when the apparent density increases. This result is consistent with the conclusion presented by Peng.39 When the material penetration depth is greater than the size of the heated sample, the influence is negligible. By contrast, microwave energy penetration is limited when the sample size is larger than the penetration depth. Nonuniform heating also occurred during the microwave heating. Thus, it is important to select the appropriate apparent density under microwave heating, which is a key factor to ensure that the material heated uniformly under microwave.

2.4. Effect of Silica Ratio on Dielectric Properties. The effect of silica ratio on the dielectric constant $\varepsilon'$, loss factor $\varepsilon''$, and loss tangent $\tan \delta$ is discussed in this part (Figure 10). The results presented the variation of dielectric properties under different silica ratios and temperatures.

As can be seen from Figure 10a, the $\varepsilon'$ increases with the temperature, and there is a positive correlation between $\varepsilon'$ and the temperature. As the value of silica ratio (0.38–0.50) increases, the $\varepsilon'$ rises. However, after the silica ratio keeps increasing from 0.50 to 0.62, the $\varepsilon'$ significantly decreases. Therefore, it has the optimal numerical value, which illustrates that the materials have good absorptivity when the value of silica ratio is 0.50.

The results show that the values of $\varepsilon''$ and $\tan \delta$ stabilize with the silica ratio between room temperature to 400 °C. At 400–650 °C, data demonstrate that the $\varepsilon''$ and $\tan \delta$ increased rapidly when the silica ratio is lower than 0.50. The $\varepsilon''$ and $\tan \delta$ decrease when the silica ratio is higher than 0.50 after 650 °C. The extremum of $\tan \delta$ is reached when the value of silica ratio is 0.5. Meanwhile, the transformation efficiency of microwave energy is the highest.

In the first stage, with the increase in silica ratio, CaO in the mixed material decreases continuously, and its microwave energy conversion efficiency increases. Because there is a small part of carbonate ion (CO$_3$)$^{2-}$ in calcareous lime powder, it absorbs some energy when decomposed. In the second stage, with the increase in silica ratio, that is, the excess of SiO$_2$ is concentrated in the slag, the further reduction reaction is hindered. Therefore, with the increase in silica ratio, its dielectric performance becomes worse.

2.5. Effect of Alkalinity on Dielectric Properties. The effect of alkalinity on mixture’s dielectric property at 20–1200 °C was measured in this part (Figure 11).

The results of $\varepsilon'$ illustrate that there are slight changes with different alkalinity at 25–650 °C. Figure 8 shows the variation curve of $\tan \delta$ and $\varepsilon'$. At 25–350 °C, the values of $\varepsilon'$ and $\tan \delta$ barely changed. After that, the $\varepsilon''$ and $\tan \delta$ increase tremendously from 350 to 650 °C. After 650–1200 °C, the trend of the results illustrates that $\varepsilon''$ and $\tan \delta$ increase with alkalinity. However, a further increase in temperature results in additional reductions in $\tan \delta$.

It is an important process to control slag alkalinity in silicothemic reduction chromite. From Figure 8, the results show that during the initial stage of reaction, the influence of alkalinity in dielectric properties is not significant. During the end stage of the reaction, FeO is converted to Fe, and Cr$_2$O$_3$ is

**Table 1. Regression Equations of Silicon-Containing Chromite Ore Fine Material Dielectric Property in Different Densities ($\rho$: Apparent Density)**

| property | linear regression equation | $R^2$ |
|----------|-----------------------------|-------|
| $\varepsilon'$ | $\varepsilon' = 1.56435\rho + 0.70447$ | 0.9526 |
| $\varepsilon''$ | $\varepsilon'' = 0.02077\rho - 0.00974$ | 0.91923 |
| $\tan \delta$ | $\tan \delta = 0.00501\rho - 0.0002$ | 0.96179 |
| $D_p$ | $D_p = -531.633\rho^2 + 733.8753\rho + 67.28895$ | 0.98978 |

**Figure 8.** Effects of apparent density on dielectric constant $\varepsilon'$, loss factor $\varepsilon''$, and loss tangent $\tan \delta$ (25 °C).

**Figure 9.** Effect of apparent density on microwave penetration depth ($D_p$).
converted to Cr. Meanwhile, with the increase in alkalinity, the reaction can be expressed as follows:

$$\text{CaO} + \text{SiO}_2 = \text{CaO} \cdot \text{SiO}_2$$  \hspace{1cm} (7)$$

$$2\text{CaO} + \text{SiO}_2 = \text{Ca}_2\text{SiO}_4$$  \hspace{1cm} (8)$$

The reaction produces the stable silicate to ensure the reduction reaction.

### 3. CONCLUSIONS

This work reports on dielectric properties of the mixture measured at a microwave frequency of 2450 ± 0.5 pct MHz under an air atmosphere from 25 to above 1200 °C using the cavity perturbation technique. The following conclusions can be drawn from the obtained results:
(1) Dielectric properties (\(\varepsilon'\), \(\varepsilon''\), and \(\tan\delta\)) of chromite and ferrosilicon all have good microwave absorption. The microwave absorption capability of chromite is poor from 25 to 1000 °C compared to ferrosilicon. Also, the decrease in reflection loss value indicates that the utilization of microwave energy increases.

(2) Dielectric properties of mixture are significantly influenced by temperature especially in the mid-temperature stage. The relationship between the penetration depth and temperature further elaborates the absorption capability of the mixture, i.e., chromite and ferrosilicon exhibit strong microwave absorption capability, and both of them are excellent microwave absorbers. Reduction of the mixture suggests that the ability of microwave absorption is related to material dielectric properties.

(3) Dielectric properties of the mixture depend on the apparent density, silica ratio, and alkalinity. The dielectric properties are improved with the increase in apparent density. Furthermore, the microwave penetration depth decreases, and a linearly function is noticed at room temperature. The increase in silica ratio leads to the improvement of dielectric properties. Thus, controlling the silica ratio is beneficial to silicothermic reduction. At the initial reaction stage, the values of \(\varepsilon'\), \(\varepsilon''\), and \(\tan\delta\) remain almost constant as the alkalinity (1.6, 1.7, and 1.8) increases. However, in the latter reaction phase, the dielectric properties increase with the alkalinity. Therefore, in this work, the dielectric properties of chromite in the microwave field are evaluated and clarify the feasibility of chromite by microwave heating. Finally, the dielectric properties of the mixture and the effects of different factors on the dielectric properties of the mixture are investigated. The effect of these phenomena is helpful to study the dielectric properties and heating characteristics. This provides a theoretical basis for silicothermic smelting of low-carbon ferrochrome.

4. EXPERIMENTAL SECTION

4.1. Experimental Materials. In this experiment, the chromite powder used is supplied by a company from South Africa, and the ferrosilicon powder and industrial analytically pure calcium oxide powder are supplied by a company from Yunnan, China. The particle size of chromite is ground to 90% lower than 68.62 \(\mu\)m before the dielectric test. The crystal structures and microstructure morphology of the chromite ore are characterized by XRD (Figure 12) and SEM (Figure 13). The XRD and XRF analysis are used to characterize the composition of the chromite powder (Table 2) and ferrosilicon powder (Table 3). The XRD and XRF

Figure 12. XRD spectrum of chromite.

Figure 13. (a) SEM images of chromite. (b) SEM image of chromium. (c) Energy spectrum analysis of chromium. (d) SEM image of iron. (e) Energy spectrum analysis of iron. (f) SEM image of silicon. (g) Energy spectrum analysis of silicon.
results indicate that chromium is mainly in the form of \( \text{Cr}_2\text{O}_3 \) and iron is mainly in the form of \( \text{Fe}_3\text{O}_4 \). The SEM and energy spectrum analyses of chromite show the morphological features and that chromium and iron mainly exist in the form of a monomer (Figure 13).

**4.2. Test Principle of Dielectric Characteristics and System.**

**4.2.1. Dielectric Characteristic Test System.** The permittivity of chromite powder was measured by the cavity perturbation technique.\(^\text{38}\) The main components of the measurement system include a resistive heating furnace and a cylindrical TM\(_{0n0}\) resonant mode cavity. The system measures the differences (frequency shift and change of quality factors) in the microwave cavity response between a cavity with an empty sample holder and the same cavity with a sample holder plus the sample at each specified temperature. These differences are recorded in the vector network analyzer (E5071C Agilent) and then used to calculate the permittivity. The details of this technique and apparatus used for the measurements can be found in the published literature.\(^\text{38,40}\) The cavity mode frequency was 915 \( \pm \) 0.5 pct MHz (\( \lambda_0 = 32.28 \) cm) or 2450 \( \pm \) 0.5 pct MHz (\( \lambda_0 = 12.24 \) cm) commonly. Also, the cavity mode frequency of 2450 \( \pm \) 0.5 pct MHz (\( \lambda_0 = 12.24 \) cm) has the characteristics of improving the heating uniformity with a multimode cavity. Therefore, the cavity mode frequency used in this experiment was 2450 \( \pm \) 0.5 pct MHz, and the detailed descriptions for the measurement fundamentals and the apparatus are found in the published literature.\(^\text{41,42}\) The structure of the experimental device is shown in Figure 14.

Figure 14. Variable temperature test system structure figure of microwave dielectric property (1, insulating sleeve; 2, graphite sleeve; 3, thermocouple; 4, induction coil; 5, sample under test; 6, insulating pedestal; 7, approach switch).

In the permittivity test, the dielectric constant can be measured at room temperature to 1400 \( ^\circ \text{C} \) in an air atmosphere, and the test temperature interval is 50 \( ^\circ \text{C} \). The equipment was adjusted before measurement, and each sample side was set three times. Through the variation in frequency (\( f \)) and quality factor (\( Q \)), the absorbing capacity of the material in the quartz tube could be calculated. It should be noted that, in order to avoid too fast cooling, affecting the accuracy of measurement, it is necessary to measure the sample as soon as possible.

**4.2.2. Test Principle of Dielectric Characteristics.** The effect of a cylindrical resonator is to produce electromagnetic oscillation transfer to the load. With the increase in the output rate of internal electromagnetic performance, its quality is also improving, while it also has a loss in the cavity. If there is a dielectric in the resonant cavity, it will also lead to the loss of electromagnetic energy. Quality factor (\( Q \)), which represents the parameter of the relationship between the total energy storage and energy consumption of the cylindrical resonator, is defined as

\[
Q = 2\pi \frac{W}{W_T} = \frac{W}{P_L}
\]

(9)

where \( W \) is the total electromagnetic energy in the cavity, \( W_T \) is the amount of energy expended in a cycle, and \( P_L \) is the average power consumed by a resonant cavity in a cycle.

The total energy stored inside the resonator is zero:

\[
W = W_e + W_m = \frac{1}{2} \int \mu |H|^2 d\nu
\]

(10)
The average loss power of the resonator

\[ R_l = \frac{1}{2} \oint |J|^2 R_s ds = \frac{1}{2} R_s \oint |H_{tan}|^2 ds \]  

(11)

where \( R_s \) is the surface resistivity, \( R_s = \sqrt{\mu_0/2\delta} \); \( H_{tan} \) is the tangential magnetic field.

So, the resonator quality factor corresponding to the finite conductivity is

\[ Q_c = \frac{\omega_0 \mu}{R_s} \oint |H|^2 dv = \frac{2 \oint |\mu| |H|^2 dv}{\delta \oint |H_{tan}|^2 ds} \]  

(12)

If the average storage energy and magnetic energy in the cylindrical cavity are equal, it will result in resonance. The operating frequency (\( f_0 \)) is the resonance frequency, which reflects the oscillation law of electromagnetic energy in the resonant cavity.

In circular waveguides, the propagation constant of the \( T_{E_{mn}} \) mode is

\[ \beta_{nn} = \sqrt{k^2 - \left( \frac{\mu_{nn}}{a} \right)^2} \]  

(13)

By studying the principle of circular cavity and knowing the dispersion form of the \( T_{E_{mn}} \) mode in the resonant cavity, this means that the essence of measuring electromagnetic parameters is a comparison of predominant frequencies (\( f \)) and quality factor (\( Q \)) to obtain the dielectric parameters of the material.

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Author Contributions

T.H. performed all of the experiments, collected sample, and wrote the manuscript. L.D. and B.L. supervised the studies and edited the manuscript. Q. Guo ran the chromite and ferrosilicon samples. Q. Gui and R.G. modified the grammar of the manuscript and reviewed the manuscript. All of the authors have given approval to the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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