Electrodeposition of nickel-tungsten alloys under ultrasonic waves: Impact of ultrasound intensity on the anticorrosive properties

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ABSTRACT
Electrodeposited Ni–W alloy assisted by high-intensity ultrasound was evaluated considering the nominal power effect on the anticorrosive property. Temperature profiles demonstrated that using a nominal power of 400 W, the electrolytic bath at 30 °C reached values of 39 ± 1 °C. The maximum acoustic power corresponded to 6.7% of the nominal power value at 400 W. Increasing the nominal power from 0 to 400 W; the Ni content decreased from 85.3 to 75.2 wt%, and the W content increased from 15.1 to 25.1 wt%. The deposited coating at 200 W and 300 W had a smooth, homogeneous, and uniform surface. At 400 W, the acoustic cavitation promoted erosion, affecting the coating surface. X-ray diffraction analysis indicated that the nominal power of 200 W promoted electrodeposition of the Ni17W3 structure with the plane (111) as a preferred orientation. The crystallite size decreased for the planes (111) and (200) when increased nominal power from 100 to 200 W. The optimum condition for the improved corrosion resistance occurred with the nominal power of 200 W, providing a polarization resistance of 23.42 kΩ cm².

1. Introduction
Electrodeposited coatings can reduce corrosion in metallic materials and replace toxic metals that harm human health and the environment [1]. The electrodeposition technique consists of passing an electric current in a conductive material immersed in a solution containing the protective metal's salts to be deposited on the substrate. Various sectors, such as maritime, automotive, building, and pipelines, apply coating technology due to the continuous failure of materials in adverse conditions [2].

The modulation of mass transfer close to the cathode and conventional agitation methods improves the electrodeposited materials' properties. High-intensity ultrasound (HIUS) technology promotes the synthesis of nanostructured materials, which are not obtained by conventional methods [3]. Its application can occur through direct mechanical transmission. The vibrations from the transducer to the solid occur directly on the material surface, or indirectly, where the acoustic cavitation propagates the energy in a fluid. The sonication of an electrolyte can represent indirect transmission. Acoustic cavitation represents the interaction between ultrasound and chemical species at the molecular level. It governs the electrochemical reactions' diffusion process.

The use of ultrasonic power in electrodeposition is known as sonoelectrochemistry [4]. It can promote heterogeneous reactions by the "extreme" increase in mass transport, thermal alteration, and interfacial cleaning [5]. According to Walker and Walker, the application of ultrasound in electrodeposition has been used since 1950 [6]. The authors reported that the effects of acoustic cavitation depend on the metal and electrochemical parameters used. Although ultrasound is applied for surface cleaning processes, its use extends to modifying organic and inorganic materials [7], strongly influencing mass transport at the electrode-solution interface [8]. To Hihn et al. [9], the ultrasound during electrodeposition increases brightness, hardness, reduces internal tension, porosity, and adhesion to the substrate.

There are several studies on the benefits of ultrasound-assisted electrodeposition that include changes in surface morphology [10], grain size [11], hardness [12], crystal orientation, and improved corrosion resistance [3]. The acoustic cavitation propagated in the liquid electrolyte influences the electrochemical process. Through microstreaming, it improves mass transport [13], promoting the reduction of the diffusion layer, with consequent punctual heating.

Regarding Ni alloys, Bigos et al. [14] studied the deposition of Ni and...
Ni-Mo with different power intensities (0, 20, 30, and 40 W cm\(^{-2}\)). Ni deposition was not dependent on ultrasonic agitation; however, it affected Ni-Mo deposition, increasing the Mo content. The change in the Mo content decreased the grain size and increased the hardness of the alloy from 720 to 820 HV. The influence of ultrasonic variables in the electrodeposition process is still little discussed. The change in ultrasonic power can alter the deposition reaction and provide smooth and compact surfaces [15].

Ni coatings were deposited under five agitation conditions, involving ultrasonic power densities at 0.011, 0.124, and 0.180 W cm\(^{-2}\), using an ultrasonic bath, at a current density of 40 mA cm\(^{-2}\), pH of 3.2 at 50 °C [16]. Under static conditions and with mechanical agitation, the deposits had a smooth and uniform appearance. However, they accumulated edges in the copper substrate’s corner due to the high locally current density. The edges were also observed in the deposit produced under acoustic cavitation, however, with a less uniform surface, demonstrating erosion marks caused by the bubbles’ implosion. Nevertheless, the erosion marks produced at 0.011 or 0.180 W cm\(^{-2}\) were small concerning the substrate’s coated area.

In this way, this study aimed to evaluate the effect of ultrasound power during Ni-W alloy electrodeposition, applying nominal powers of 100, 200, 300, and 400 W. Calorimetric tests determined acoustic power, HIUS intensity, and energy density. To the authors’ knowledge, these process variables have not been determined for application in coatings, especially in Ni-W alloys. They are coatings with high microhardness and corrosion protection. The alloys were characterized regarding morphology, composition, and atomic arrangement. The polarization resistance and electrochemical impedance spectroscopy (EIS) technique evaluated the coating’s anticorrosive property.

2. Materials and methods

2.1. Chemical treatment and solution composition

The copper substrate was cut into 2 by 2 cm squares containing a stem 0.4 cm wide and 4 cm high. The cleaning was performed with sandpapers of 200, 600, and 1200 grain cm\(^{-2}\). Grease removal occurred in 1 wt% NaOH solution and surface activation in 1% (v/v) H\(_2\)SO\(_4\) solution, both lasting 1 min. The copper plates were dried in an oven at 105 °C.

Electrolytic baths were prepared to contain: Na\(_2\)WO\(_4\)-H\(_2\)O (Sigma Aldrich 99%), tungsten source salt, 0.3 M; NiSO\(_4\)-6H\(_2\)O (Synth 98%), nickel source salt, 0.3 M; Na\(_2\)B\(_4\)O\(_7\)-2H\(_2\)O (Synth 98%), as an alloying agent, 3.75 × 10\(^{-2}\) M; Na\(_2\)C\(_2\)H\(_2\)SO\(_4\) (Synth 90%), to prevent blistering in the alloy caused by slowly released H\(_2\), 1 × 10\(^{-4}\) M; (NH\(_4\))\(_2\)SO\(_4\) (Synth 99%), as a stabilizing agent, 1.3 × 10\(^{-1}\) M; and (NH\(_4\))\(_2\)C\(_6\)H\(_5\)O\(_7\) (Synth 98%), with complexing function, 0.3 M.

2.2. Ni-W electrodeposition assays and HIUS processing

Duplicated depositions were performed with and without a 19 kHz probe ultrasound, with a 13 mm probe diameter (Unique, Disruptor, 500 W, Indaiatuba, Brazil) at 100, 200, 300, and 400 W, resulting ten experiments.

The pH levels were analyzed with a pHmeter (Quimis Q400AS, Diadema, Brazil) before and after the deposition that occurred at 30 °C in a jacketed cell, lasting 15 min, using a potentiotstat/galvanostat (VersaStat 3, Princeton Applied Research) with 50 mA cm\(^{-2}\) of electric current density. The codeposition of W using the citrate complexing agent occurred according to the reactions in Eqs. (1)-(4) [17].

\[ \text{Ni}^{2+} + \text{Cit}^{3-} \rightleftharpoons [(\text{Ni})(\text{Cit})]^{-} \]  \hspace{1cm} (1)

\[ \text{WO}_4^{2-} + \text{Cit}^{3-} + \text{H}^{+} \rightleftharpoons [(\text{WO}_4)(\text{Cit})(\text{H})]^4- \]  \hspace{1cm} (2)

\[ [(\text{Ni})(\text{Cit})]^{-} + [(\text{WO}_4)(\text{Cit})(\text{H})]^4- \rightleftharpoons [(\text{Ni})(\text{WO}_4)(\text{Cit})(\text{H})]^5- + \text{Cit}^{3-} \]  \hspace{1cm} (3)

The copper plate acted as a working electrode. The counter/reference electrode was a hollow platinum mesh, as illustrated in Fig. 1. Faradaic efficiency (\(\varepsilon\)) was calculated using the coating deposit and the chemical composition detected by EDX, according to a previous study [18].

The acoustic power (Eq. (5)) or real power was calculated for each nominal power by calorimetric experiments described by Silva et al. [19]. Therefore, HIUS intensity and energy density were calculated using acoustic power, according to Eqs. (6) and (7), respectively [20].

\[ \text{Acoustic power} = mC_p \left( \frac{dT}{dt} \right) \]  \hspace{1cm} (5)

\[ \text{HIUS intensity} \left( \frac{W}{cm^2} \right) = \frac{4 \times \text{Acoustic power}}{\pi D^2} \]  \hspace{1cm} (6)

\[ \text{Energy density} \left( \frac{J}{mL} \right) = \frac{\text{Acoustic power (W)} \times \text{Processing time (s)}}{\text{Volume (mL)}} \]  \hspace{1cm} (7)

where \(m\) is the mass of electrolytic bath (g), \(C_p\) is the specific heat of electrolyte (C \(\text{g}^{-1} \cdot \text{C}^{-1}\)), \((dT/dt)\) is the change in temperature over time (°C s\(^{-1}\)), and \(D\) is the probe diameter (cm).

2.3. Chemical and physical characterization

Scanning electron microscope (SEM) with chemical analysis by X-ray dispersive energy (EDX) and X-ray diffraction (XRD) evaluated the Ni-W coating morphology and composition. The XRD diffractograms were obtained with a Philips device (XPERT model), with copper K\(_{\alpha}\) radiation at a 40 kV voltage, current of 40 mA, wavelength of 1.52 Å, step size of 0.02°, and a time per step of 1 s. Coating surfaces were evaluated using the SEM LEO brand (LEO 440i model) with the EDX detector.

2.4. Corrosion tests

Corrosion tests were performed by the Tafel extrapolation method using a three-electrode cell, with the Ag\(_{2}\)/AgCl electrode as a reference. Pt was the counter electrode. The working electrode was a coated copper plate exposing 0.0001 m\(^2\). The corrosive environment occurred at room temperature (25 °C) with NaCl (3.5 wt%). The potentiodynamic polarization curves were obtained with a scan rate of 0.167 mV s\(^{-1}\) and a potential range of 250 mV around the open circuit potential (OCP). For the EIS analysis, a frequency window from 10\(^3\) to 0.01 Hz was used, and the OCP was applied for 1 h with an amplitude of 10 mV.
3. Results and discussion

3.1. Temperature profile under HIUS conditions

Table 1 presents the acoustic power, power density, HIUS intensity, and energy density obtained from each nominal power applied in the Ni–W alloy electrodeposition bath. The calorimetric tests to obtain the acoustic power by HIUS demonstrated that the nominal power increase from 100 to 400 W linearly increased the applied energy density. Therefore, electrodeposition occurred at different energy levels, allowing the evaluation of Ni–W alloy parameters. In general, acoustic power, HIUS intensity, and energy intensity are neglected in ultrasound-assisted electrodeposition. Other process variables are not studied, such as the probe area, specific heat, and liquid medium volume [21].

Fig. 2 shows the temperature profile for each nominal power as a function of the processing time that was the electrodeposition time. The experiments started at 30 °C and reached equilibrium temperature values of 31.1 ± 0.6, 33.4 ± 0.3, 37 ± 0.1, and 39 ± 1 °C for the nominal powers of 100, 200, 300, and 400 W, respectively. Temperature profiles allow monitoring energy expenditure. Also, the increase of temperature decreases the electrolytic bath’s viscosity, reducing the acoustic cavitation effect. Temperature influences ionic strength and mobility, modifying the solubility of salts in the bath [22]. Therefore, knowing the heating level provided to the solution allows obtaining the heat rate exchanged from the deposition process.

3.2. Surface finish of the coatings

Fig. 3 shows the visual appearance of Ni–W coatings deposited under different sonication conditions. The coating appearance was quite heterogeneous without agitation, formed by three matte regions in the plate’s center. The deposition at 100 W formed matte structures located in the corner of the copper substrate. For depositions at 200 and 300 W, the coatings had a uniform and smooth appearance, with an excellent surface finish. At 400 W, small dark spots formed on the coating indicated erosion marks generated by the acoustic cavitation phenomenon. For pure Ni coatings deposited at a power density of 0.124 W cm⁻², erosion caused by cavitation was more evident than in coatings deposited at 0.011 and 0.180 W cm⁻³, demonstrating randomness in appearance [16].

3.3. Morphology, composition, and pH of the electrolytic bath

According to Table 2, the bath pH remained constant, close to 5.8, after electrodeposition, indicating that it may not have occurred H₂ evolution with H⁺ ions’ consumption. Hydrogen evolution is expected during bimetallic alloys’ electrodeposition, increasing the solution pH and forming bubbles, affecting the deposited material’s morphology [23].

Ni mass deposited had a slight decrease of 0.19 × 10⁻² g of the deposition process from 0 to 400 W. On the other hand, the W mass increased by 3.04 × 10⁻³ g with the nominal power increase from 0 to 400 W. The faradaic efficiency remained constant close to 25% considering the standard deviation, with a difference of 1% between the coating’s efficiency without ultrasound and the one with 400 W nominal power. The deposition time of 15 min was adequate for the faradaic efficiency value. For the electrodeposition lasting 1 h using real wastewater containing 0.1 M Ni, a value of 25.3% was obtained [24].

Regarding the Ni–W alloy morphology, deposition without ultrasound led to the formation of a bubble between the copper substrate and the Ni–W coating, with circular structures, as shown in Fig. 4. The deposition at 100 W nominal power promoted incomplete cracks, according to the arrows indicated in Fig. 4. The deposition process at 200 W still presented an incomplete crack, according to the arrow; however, it presented a complete aspect similar to the coating obtained at 300 W. The deposition at 400 W formed small square structures at the end of the crack outline, indicated in the circles in Fig. 4. Mohammadpour and Zare [25] observed cracks in the Ni–W alloy deposited with a current density of 38.2 mA cm⁻² and solution pH of 7.5, at 50 °C, lasting 60 min. The authors suggested that hydrogen evolution may have caused the cracks, decreasing faradaic efficiency. Moreover, it attenuates multiple islands’ lateral growth, presenting incomplete cohesion or discontinuities [26]. The crack-rich coating surface is also related to the inherent fragile nature of W [27].

The number of cracks can be related to the current density applied during electrodeposition; according to Porto et al. [28], few microcracks were observed for a current density of 10 mA cm⁻². However, the number of microcracks increased for the alloy deposited at 50 mA cm⁻². Co-W/MWCNTs nanocomposites presented a modified morphology after ultrasonic treatment [29]. Mechanical agitation gave rise to agglomerated carbon nanotubes. Increasing the nominal power to 400 W promoted the homogeneity of the microstructure. In contrast, the nominal power of 500 W resulted in material detached from the matrix, indicating a suitable nominal power for the process.

The EDX maps in Fig. 5 demonstrate the highest Ni content (84.9 wt %) without ultrasound compared to 400 W with 75.2 wt%. The W content increased from 15.1 to 25.1 wt% when the nominal power increased from 0 to 400 W. Higher W contents up to 45.5 wt% refine the grain, after which the structures become amorphous [30]. The Ni energy peaks decreased as the content decreased, and the W peak increased with the increased nominal power, according to the EDX spectra.

3.4. Phase structure analysis

In general, ultrasound technology is known to produce smooth surfaces. It distributes the particles evenly and refines the grain size [31]. The peaks indicated by the XRD pattern in Fig. 6 demonstrate the crystalline cubic structure of the Ni–W alloy, with Ni₂W₃ composition
(JCPDS card n° 65–4828) in the three peaks at 43.68, 50.88, and 74.82° with crystallographic planes (1 1 1), (2 0 0), and (2 2 0), respectively. These three peaks, although similar to the copper substrate peaks at 42.37, 49.33, and 72.33° with the respective planes (1 1 1), (0 0 2), and (0 2 2) (JCPDS card n° 01–3024), did not match the diffractograms. Besides, the coating covered the entire substrate. The peak intensity decreased from 0 to 300 W and increased in the deposition at 400 W nominal power.

The reverse pulse electrodeposition produced Ni–W nanocrystalline coatings with the same characteristic peaks, containing multilayer structures with W content of 11 and 25 wt% [32]. The widening of the XRD peaks of deposits with a higher W content suggested that the layers with 25 wt% W had a finer size, indicating a crystallite size of 26 nm.

The crystallite size calculated by the Scherer equation for all crystallographic planes indicated a decrease of 4.58 nm for the plane (1 1 1) when the nominal power increased from 100 to 200 W, as displayed in Fig. 7-a. For the plane (2 0 0), the crystal decreased 4.58 nm, comparing the process of 100 and 200 W, and increased with equal value when the

### Table 2

| Nominal power (W) | Initial/ final pH | $M_{Ni}$ ($\times 10^{-2}$ g) | $M_{W}$ ($\times 10^{-3}$ g) | Composition (wt%) | Faradaic efficiency (%) |
|-------------------|-------------------|-----------------------------|----------------------------|-------------------|-------------------------|
| 0                 | 5.9–5.8           | 2.41 ± 0.01                 | 4.26 ± 0.01                | 85.1 ± 0.3 Ni/ 15.1 ± 0.1 W | 25.68 ± 0.01             |
| 100               | 5.8–5.8           | 2.24 ± 0.07                 | 4.7 ± 0.4                 | 83 ± 2 Ni/17     | 24.6 ± 0.3              |
| 200               | 5.8–5.7           | 2.2 ± 0.1                   | 6.41 ± 0.01               | 77.8 ± 0.5 Ni/ 22.2 ± 0.5 W | 26 ± 1                   |
| 300               | 5.8–5.7           | 2.2 ± 0.1                   | 6.9 ± 0.08                | 75.9 ± 0.9 Ni/ 23.6 ± 0.3 W | 26 ± 1                   |
| 400               | 5.8–5.7           | 2.22 ± 0.01                 | 7.3 ± 0.1                 | 75.5 ± 0.3 Ni/ 24.7 ± 0.5 W | 26.6 ± 0.1               |

Fig. 3. Visual appearance of the coatings deposited under different nominal powers.

Fig. 4. SEM micrographs of the deposited Ni–W alloys under different nominal powers.
nominal power increased from 300 to 400 W. Deposited coatings of Nidoped TiN had a similar effect on crystallinity [33]. The Ni peaks’ intensity decreased as the ultrasonic intensity increased from 0 to 20 W cm$^{-2}$, indicating a decrease in Ni crystals size. However, the Ni peaks increased to some extent, using an ultrasonic intensity of 40 W cm$^{-2}$.

Although the crystallite size’s measured values may not be exact due to the Scherrer equation’s limitations [34], there was a tendency of the values corresponding to the peak of enlargement according to the EDX spectra in Fig. 5. Another factor affecting the crystallite size is the W content. It can segregate at the grain boundaries, controlling its size [35]. The crystallite size was smaller than pure Ni coatings, which are greater than 60 nm [36], and Ni-Cu/TiN composite coatings (between 14.1 and 21.8 nm) [37]. The Cu atom associated with the complexing agent sodium citrate and saccharin presented a grain refinement effect in the Ni-Cu alloy. This effect may have occurred in the Ni-W alloy. W atoms can restrict the diffusion of the nickel surface and inhibit crystallite growth.

The relative texture coefficient (RTC) was calculated according to Eq. (8) [38] to quantify the Ni-W alloy’s preferred orientation.

Fig. 5. EDX maps and their spectra for the Ni–W coatings produced under nominal power of 0 e 400 W.

Fig. 6. XRD patterns of Ni–W alloys under different nominal powers.

Fig. 7. a) Crystallite size and b) RTC according to the nominal power applied during Ni–W alloys deposition.
where \( I_{(hkl)} \) is the reflection intensity for the crystal plane \((hkl)\) of each sample and \( I_{(hkl)p} \) is the reflection intensity for the same crystal plane in a standard sample of Ni-W powder with random orientation.

\[
\text{RTC}_{(hkl)} = 100 \times \frac{I_{(hkl)}}{\sum I_{(hkl)}} \quad (8)
\]

3.5. Potentiodynamic polarization assays

Fig. 8 illustrates the Tafel plots of Ni-W alloys for different nominal powers. The Tafel extrapolation method allowed obtaining the corrosion potential \( E_{\text{corr}} \) and corrosion current \( i_{\text{corr}} \), as well as the polarization resistance \( R_p \), according to Table 3. Ultrasound-assisted electrodeposition at 200 W provided a coating with the lowest corrosion current \((0.87 \mu A \text{ cm}^{-2})\) and the highest polarization resistance \(23.42 \Omega \text{ cm}^2\). Therefore, there was a suitable energy density for the system, as approximately 100 J mL\(^{-1}\). Values higher than this energy decreased the polarization resistance, just as the deposition without ultrasound indicated 21.85 kΩ cm\(^2\). The microbubbles generated from the acoustic cavitation increased the temperature of the solution and the mass transfer. With a nominal power of 400 W, an energy density of 269 ± 2 J mL\(^{-1}\) was greater than adequate to improve corrosion resistance.

3.6. Electrochemical impedance spectroscopy assays

The semicircles obtained by the EIS technique illustrated in Fig. 9 corroborated with the results of polarization resistance. The deposited coating assisted by ultrasound with a nominal power of 200 W presented the largest capacitive arc, indicating more excellent corrosion resistance. The adjusted impedance data by the EIS spectrum analyzer software are displayed in Table 4. The Randles equivalent circuit presented the electrolyte resistance \( R_e \) in series with a constant phase element (CPE), which was in parallel with the charge transfer resistance \( R_{ct} \). CPE presents two parameters: 1) a non-ideal capacitor related to charge transfer \( Q_{ct} \); 2) a fractional power of the frequency \( n \).

The adjustments again indicated that the coating produced at 200 W had a higher \( R_{ct} \), which is equivalent to polarization resistance.

Table 3
Corrosion data of Ni-W alloys for different nominal powers.

| Nominal power (W) | \( E_{\text{corr}} \) (V vs SCE) | \( i_{\text{corr}} \) (\( \mu A \text{ cm}^{-2} \)) | \( R_p \) (kΩ cm\(^2\)) |
|------------------|--------------------------------|---------------------------------|------------------|
| 0                | 0.165                          | 1.09                            | 21.85            |
| 100              | 0.160                          | 0.98                            | 17.96            |
| 200              | 0.204                          | 0.87                            | 23.42            |
| 300              | 0.166                          | 1.43                            | 12.34            |
| 400              | 0.126                          | 1.90                            | 8.10             |

3.6. Electrochemical impedance spectroscopy assays

The adjusted impedance data by the EIS technique illustrated in Fig. 9 corroborated with the results of polarization resistance. The deposited coating assisted by ultrasound with a nominal power of 200 W presented the largest capacitive arc, indicating more excellent corrosion resistance. Therefore, there was a certain energy level supplied to the system that benefits Ni-W coating’s anticorrosion property. Similar behavior was reported by Shetty and Chitharanjan Hegde [3]. They synthesized multilayer Ni-Co alloys assisted by alternating current and ultrasound pulses using three levels of cavitational energy \((0.6, 0.9, \text{and } 1.2 \text{ W cm}^{-2})\). The authors observed an adequate processing time for the ultrasound to modulate the mass transport and decrease the corrosion rate, which occurred with up to 150 layers. With 300 layers, the corrosion rate increased to 11,270 mm y\(^{-1}\).

4. Conclusions

Ni-W alloys with anticorrosive properties were deposited on copper substrate assisted by HIUS under different nominal powers. The calorimetric tests in the bath indicated that the temperature at the end of the process reached a value of 39 °C, using the nominal power of 400 W. The physical appearance of the coatings produced at 200 and 300 W indicated a smooth and uniform aspect. Their microstructures presented refined grains. Polarization resistance tests demonstrated that ultrasound-assisted deposition at 200 W provided the highest corrosion resistance and the largest capacitive arc diameter in the Nyquist diagram, assigning an ideal acoustic cavitation level to improve the anticorrosion property of the material.

Table 4
Equivalent circuit parameters based on the Nyquist diagram data.

| Nominal power (W) | \( R_e \) (Ω cm\(^2\)) | \( R_{ct} \) (Ω cm\(^2\)) | \( Q_{ct} \times 10^{-4} \) (Ω\(^{-1}\) s\(^n\) cm\(^{-2}\)) |
|------------------|------------------|------------------|------------------|
| 0                | 12.9             | 2822             | 0.96 (n = 0.75)  |
| 100              | 20.2             | 2212             | 3.63 (n = 0.75)  |
| 200              | 15.9             | 3803             | 2.85 (n = 0.77)  |
| 300              | 16.6             | 1348             | 5.05 (n = 0.83)  |
| 400              | 19.4             | 490              | 5.05 (n = 0.95)  |
CRediT authorship contribution statement

Josiel Martins Costa: Methodology, Conceptualization, Writing - original draft. Ambrosio Florencio de Almeida Neto: Supervision, Funding acquisition, Resources, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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