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Electrochemical data on the corrosion inhibition performance of admixed *Citrus paradisi* and *Zingiber officinale* oil extracts in 0.5 M H₂SO₄ solution

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ABSTRACT

Data output on the protection performance of the combined admixture of *Citrus paradisi* and *Zingiber officinale* oil extracts (CPZO) on the corrosion inhibition of mild steel in 0.5 M H₂SO₄ solution are presented and described. CPZO effectively suppressed the electrochemical reactions on the steel surface with final inhibition values ranging between 95.16 mm/y at 1 % CPZO and 96.61 % at 3.5 % CPZO concentration. CPZO reduced the corrosion rate of the steel from the value of 184.48 % at 0 % CPZO concentration to values between 3.45 mm/y and 8.94 mm/y in the presence of CPZO extract. CPZO performance remained marginally steady with respect to its concentration. However, variation with time was significant till 240 h. Effective inhibition performance was attained at 48 h of exposure. Calculated data from ANOVA analysis shows exposure was the only statistically relevant parameter at 98.63 % compared to 0.75 % for CPZO concentration. Adsorption of CPZO molecules on the steel aligns with the Langmuir and Frumkin isotherm models with correlation coefficient values of 0.9997 and 0.7292. Thermodynamic calculations depict chemisorption adsorption mechanism with ΔG values ranging between -46.13 and -43.23 KJ/mol. Optical images of the inhibited and non-inhibited steel significantly contrast each other. © 2023 Elsevier Ltd. All rights reserved.

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1. Introduction

Application Corrosion is an irreversible and undesirable occurrence responsible to the degradation, degeneration and weakening of metallic alloys [1]. The consequences of corrosion are the deterioration of the structural and mechanical properties of alloys especially those in industrial applications such as the building, process, petrochemical, energy generation, automobile and desalination industries. The industrial operating environment where these alloys are exposed to (H₂S, CO₂, SO₄²⁻, Cl⁻ etc.) accelerates the corrosion of these alloys. Carbon steel, an Fe-C metallic alloy with the carbon content making up about 0.3 % to 2.1 % of the entire weight of the steel. It represents 90 % of the total steel volume produced worldwide. The steels are utilized as structural columns and beams, components parts of industrial machineries and subcritical to critical components where toughness and hardness properties etc. cannot be compromised. The mechanical properties of carbon steels serve as the base standards by which the properties of other metallic alloys are compared [2]. The comparative eco-

nomical cost of carbon steels immensely contributes to its high volume utilization globally. However, these steels, are vulnerable to rapid degradation in aqueous condition containing threshold concentrations of Cl⁻, SO₄²⁻, S₂O₃²⁻, NO₃⁻ etc. [3]. This leads to corrosion reaction mechanisms responsible to surface degradation and eventually decrease in the functional lifespan of the steel [4]. Corrosion controls of metals are of technical, economic, environmental, and aesthetic importance [5].

Corrosion inhibitors are chemical derivatives which alters the chemical and electrochemical conditions of environments where they are introduced. They tend to slow down the rate of corrosion of carbon steels or actively stifle the electrochemical reaction processes responsible for corrosion [6]. Inhibitors act by forming a protective film over the steel surface, selective precipitation over reactive sites on the steel, modification of the corrosive medium rendering it harmless and formation of complexes with the corrosive anions. Generally, this decreases the diffusion of corrosive anions onto the metallic surface and increases the electrical resistance of the steel surface [7,8]. In addition to industries where carbon steels are used and undergo corrosion, industrial operations also require the use of corrosion inhibitors such as acid pickling

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of steels, chemical cleaning, and processing, ore production, chemical processing plants, and oil well acidification, etc. [9]. Research into green chemical compounds is ongoing with promising results. Loto [10], Abdelaziz *et al.* [11], Gadow *et al.* [12], Bensouda *et al.* [13], Bensouda *et al.* [14], Hafez *et al.* [15] and Elhajjaji *et al.* [16] studied the corrosion inhibition of biodegradable 2-amino-4-methylpentanoic acid, *Arbutus unedo L*, *Eruca sativa*, *Artemisia Abrotanum*, *Eucalyptus* and *thyme vulgaris* examined the corrosion inhibition of carbon steel in dilute HCl solution. Results showed that the green compounds effectively inhibited the steel, increase in extract concentration results in increase in inhibition efficiency and they exhibited mixed type inhibition effect. Zheng *et al.* [17], Farahati *et al.* [18], Znini *et al.* [19], Znini *et al.* [20], Saxena *et al.* [21] and Loto *et al.* [22] studied the corrosion inhibition effect of *Eriobotrya japonica Lindl* leaves extract, water soluble penicillamine drug and cysteine, *Asteriscus Graveolens*, *Warionia Saharea*, *Saraca Ashoka* and *Camellia Sinensis* green chemical compounds on the corrosion inhibition of carbon steel in dilute H₂SO₄ solution. Performance of plant extracts as corrosion inhibitors is encouraging as possible replacement for toxic chemical derivatives. However, more research needs to be carried out to establish effective inhibition performance at lower concentrations instead of higher concentrations of the extract [23]. In contribution to the research on green chemical derivatives, the manuscript studies the inhibition effect of the combined admixture of *Citrus paradisi* and *Zingiber officinale* oil extracts on high carbon steel in 0.5 M H₂SO₄ solution.

2. Experimental methods

2.1. Materials and methods

Citrus paradisi and *Zingiber officinale* (CPZO) oil extracts purchased from NOW foods USA at 100 % purity were added individually to 0.5 M H₂SO₄ solution in volumetric concentrations of 1 %, 1.5 %, 2 %, 2.5 %, 3 % and 3.5 %. High carbon steel (HCS) rods were cut into six separate samples for weight loss measurement. The 6 samples were washed with deionized H₂O and propanone before weight loss test. The HCS samples were inserted into each acid/extract solution (with respect to inhibitor concentration) contained in 6 separate glass beakers for 240 h after initial measurement. Weight of the specimens were recorded at 24 h interval with Ohaus PA114 measuring device with resolution of 0.0001 g, maximum capacity is 110 g, repeatability is 0.1 mg and linearity is ± 0.2 mg. Weight loss was determined from the subtraction between the initial weight of HCS (maintained for 240 h) and subsequent weight measured at 24 h interval for a total of 240 h. Corrosion rate of HCS was determined from the equation below;

$$C_R = \left[\frac{87.6W}{\rho AT} \right] \quad (1)$$

W denotes weight loss (g), ρ denotes density (g/cm³), A denotes area (cm²), and T symbolizes time of exposure (h). Inhibition efficiency (η) was enumerated from the following equation;

$$\eta = \left[\frac{\omega_1 - \omega_2}{\omega_1} \right] \times 100 \quad (2)$$

ω_1 denotes weight loss of MS from the acid solution without the distillates while ω_2 denotes weight loss of HCS at precise extract concentration.

2.2. Statistical computation

Dual-factor empirical ANOVA test (F - test) was applied to compute the statistical relevance of extract concentrations and exposure time on CPZO inhibition efficiency data. The evaluation was

realized at confidence level of 95 % i.e. a significance level of $\alpha = 0.05$ in accordance with the equations below. The aggregate of squares of columns (exposure time) was enumerated as shown;

$$SS_c = \frac{\sum T_c^2}{nr} - \frac{T^2}{N} \quad (3)$$

The aggregate of squares between rows (CPZO concentration) was enumerated from equation (4)

$$SS_r = \frac{\sum T_r^2}{nc} - \frac{T^2}{N} \quad (4)$$

Total aggregation of squares

$$SS_{Total} = \sum x^2 - \frac{T^2}{N} \quad (5)$$

3. Results and discussion

3.1. Coupon analysis

Table 1 presents the corrosion rate of MS and inhibition efficiency of CPZO at specific CPZO concentrations for 240 h of exposure. General observation shows that MS corrosion rate data from 1.5 % to 3.5 % CPZO concentration significantly varies from MS corrosion rate at 0 % CPZO concentration. The high corrosion rate of MS in the absence of the extracts is due to the electrochemical action of SO₄²⁻ anion resulting in accelerated oxidation of the steel surface. While the corrosion rate of MS at 0 % CPZO was marginally stable after 24 h of exposure till 240 h, corrosion rate of MS from 1 % to 3.5 % CPZO concentration decreased significantly till 240 h. The reason for the decrease is due to the effective corrosion inhibition properties of CPZO. In the presence of CPZO, the continual decrease of MS corrosion rate shows time dependent electrochemical action even though effective inhibition performance occurred early at 72 h from observation of CPZO inhibition efficiency data. Considering the effect of variation of CPZO concentration, it is clearly visible that performance of CPZO is non-linearly proportional to its concentration i.e. it does not increase or decrease with increase in concentration as it is common with some inhibitors. This signifies the lateral interaction effect among CPZO molecules has limited influence on the performance of the extract. The inhibitor exhibits concentration non-dependent performance which increases its survivability in industrial applications. Its time dependent action also supports this assertion as effective inhibition efficiency is attained at 48 h of exposure. At 48 h, corrosion rate of MS at 1 % to 3.5 % CPZO concentration varies between 5.13 mm/y and 8.94 mm/y compared to 184.48 mm/y at 0 % CPZO concentration at 240 h. The corresponding inhibition efficiency value at this time varies between 95.16 % and 98.13 %.

3.2. Adsorption isotherm and corrosion thermodynamics

The corrosion inhibition performance of CPZO extract on MS in 0.5 M H₂SO₄ solution was further evaluated from numerical derivations and analytical plots which reveals the degree of adsorption of the molecular species of CPZO onto the steel surface at stable conditions and temperature [24]. Protonated molecular species of CPZO diffused from the aqueous electrolyte to the metal-solution boundary layer during redox reaction processes. Results from potentiodynamic polarization depict mixed-type corrosion inhibition behavior by CPZO extract indicating strong influence on the cathodic and anodic polarization behavior of the steel through surface coverage, selective precipitation, strong covalent bonding and electrostatic attraction. Adsorption of inhibitor molecules generally occurs by substitution mechanism resulting in the

Table 1
Data of MS corrosion rate and CPZO inhibition efficiency with respect to time and CPZO concentration.

Time (h)	MS Corrosion Rate (mm/y)							CPZO Inhibition Efficiency (%)					
	CPZO Conc.												
	0 % CPZO	1 % CPZO	1.5 % CPZO	2 % CPZO	2.5 % CPZO	3 % CPZO	3.5 % CPZO	1 % CPZO	1.5 % CPZO	2 % CPZO	2.5 % CPZO	3 % CPZO	3.5 % CPZO
24	78.20	33.51	31.10	28.68	31.40	31.10	26.87	57.14	60.23	63.32	59.85	60.23	65.64
48	133.45	18.87	18.87	15.10	16.15	19.32	19.63	85.86	85.86	88.69	87.90	85.52	85.29
72	161.83	15.10	13.89	10.47	11.67	13.79	14.69	90.67	91.42	93.53	92.79	91.48	90.92
96	188.86	12.30	10.87	8.00	9.74	10.72	12.30	93.49	94.24	95.76	94.84	94.32	93.49
120	188.83	10.93	9.36	6.46	8.09	9.30	10.45	94.21	95.04	96.58	95.71	95.08	94.47
144	186.04	10.06	7.90	5.43	7.20	8.00	9.31	94.59	95.75	97.08	96.13	95.70	95.00
168	183.57	10.14	6.94	4.72	6.25	7.07	8.11	94.48	96.22	97.43	96.60	96.15	95.58
192	186.21	9.89	6.19	4.15	5.77	6.57	7.25	94.69	96.68	97.77	96.90	96.47	96.11
216	188.10	8.92	5.60	3.73	5.27	5.87	6.64	95.26	97.02	98.01	97.20	96.88	96.47
240	184.48	8.94	5.13	3.45	5.53	5.34	6.25	95.16	97.22	98.13	97.00	97.11	96.61

displacement of H₂O molecules from the steel surface. Results from weight loss analysis were evaluated with familiar adsorption isotherms. Isotherms exhibiting high correlation coefficient values (Langmuir and Frumkin isotherms) were chosen and analyzed. Table 2 shows the correlation coefficient data for Langmuir and Frumkin adsorption isotherms.

The inhibition mechanism exhibited by CPZO extract on MS follows the Langmuir and Frumkin isotherm models. The Langmuir and Frumkin isotherm plots are presented in Fig. 1a and 1b. Langmuir isotherm focusses on the equilibrium between CPZO molecules and MS surface wherewith CPZO adsorption is restricted to single molecular layer. The isotherm states the adsorption of molecular species is a single layer holding only one adsorbed molecule at the surface without any interaction between molecules on different sites with equal magnitude of the heat of adsorption independent on the number of sites. The isotherm equation is as follows;

$$\theta = \frac{K_{ads}C_{CPZO}}{1 + K_{ads}C_{CPZO}} \quad (6)$$

K_{ads} indicates the equilibrium constant of adsorption. C_{CPZO} indicates molar concentration of CPZO extracts. Frumkin isotherm states the that adsorbed inhibitor molecules do exhibit lateral attraction or repulsion interaction effect with respect to the equation below;

$$\text{Log}[CCPZO * (\theta/1 - \theta)] = 2.303\text{log}K_{ads} + 2\alpha\theta \quad (7)$$

Calculated results for Gibbs free energy (ΔG) representing the energy of adsorption between the extract and MS surface in H₂SO₄ solution are shown in Table 3. The ΔG results were determined from equation (8). The equilibrium constant of adsorption (K_{ads}) for CPZO was determined from Langmuir equation due to its higher correlation coefficient value.

$$\Delta G_{ads} = -2.303RT\text{log}[55.5K_{ads}] \quad (8)$$

55.5 indicates molar concentration of H₂O in H₂SO₄, R indicates universal gas constant, T represents absolute temperature. The ΔG results obtained clearly indicates chemisorption adsorption mechanism for CPZO ionized molecules onto MS surface due to the strong electrostatic attraction and covalent bonding between the

molecular species of CPZO and charged MS surface [25]. The H₂O molecules were significantly replaced CPZO molecules which invariably limited the electrolytic transport and reaction of SO₄²⁻ anionic species. Consequentially, the redox electrochemical process responsible for corrosion are stifled, restricting the active sites from further propagating corrosion.

3.3. Optical image analysis

Optical image representations of MS (mag. x20) before corrosion test, after corrosion in H₂SO₄ solution without CPZO and after corrosion in CPZO/H₂SO₄ solution extract at mag. x20 are shown in Fig. 2(a), 2(b) and 2(c). Fig. 2(a) shows the MS morphology before corrosion test. The serrated and uneven surface is due cutting of the steel prior to weight loss analysis. The image in Fig. 2(b) is due to the electrochemical action of SO₄²⁻ anions on MS surface. Oxidation of MS in the electrolyte led to a severely degraded morphology and thinning of the steel thickness. Under this condition the steel fails completely and experiences short operational lifespan. Addition of CPZO extract suppressed the electrochemical reactions occurring on MS surface. The presence of oil residue on the steel surface after long hours of exposure shows surface coverage is principally the inhibition mode of CPZO in protecting the steel surface. The protection effect of CPZO sustained the aesthetic, physical and mechanical properties of the steel while significantly decreasing the corrosion rate of the steel as earlier observed.

3.4. Statistical analysis

ANOVA analysis employed to assess the statistical relevance of CPZO concentration and exposure time on the inhibition efficiency of CPZO on MS. ANOVA analysis data are presented in Table 4. Statistical relevance factor depicts the proportionate value of the impact of CPZO concentration and exposure time on the protection effect of CPZO after 240 h of exposure. Theoretical significance factor indicates the numerical value wherewith the mean square ratio must be greater than for the statistical relevance factor to be applicable, or less than for the statistical relevance factor to be irrelevant. The mean square ratios for CPZO concentration and time of exposure are significantly greater than the corresponding theoretical significance factor. As a result, they are statistically applicable. However, the statistical relevance value for CPZO concentration is 0.75 % indicating very minimal effect on the performance of the inhibitor. This observation coincides with the observation from weight loss analysis where increase or decrease in CPZO concentration has marginal influence. The corresponding theoretical significance factor for exposure time is 98.63 %, indicating the overwhelming time dependent characteristics behaviour of CPZO. The efficiency of the inhibitor significantly improves to effective

Table 2
Correlation coefficient data for Langmuir and Frumkin adsorption isotherms.

Adsorption Isotherms	CPZO Extract
Langmuir	0.9997
Frumkin	0.7292

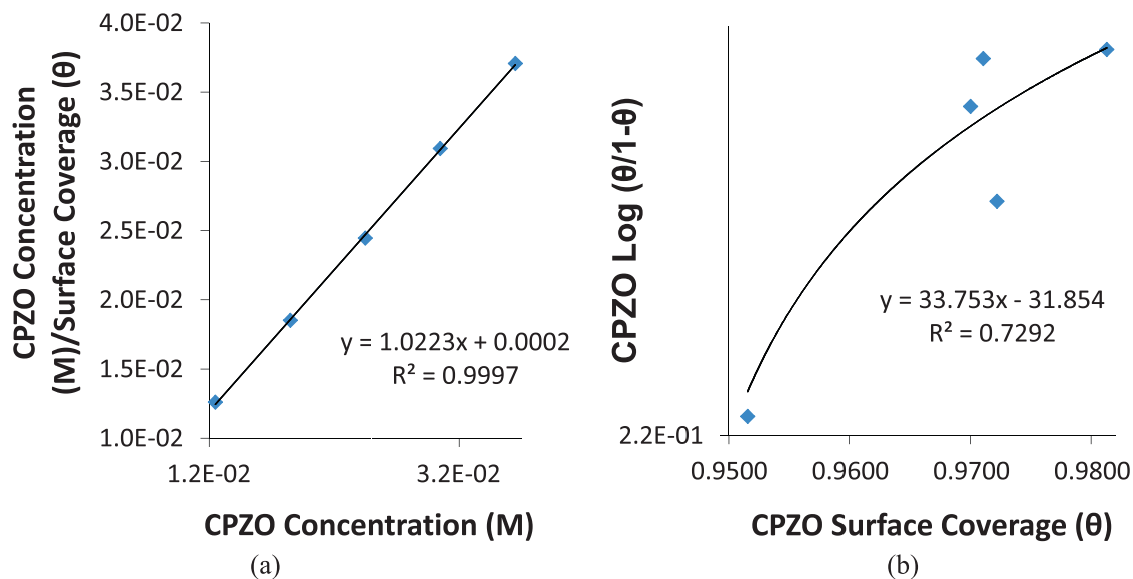


Fig. 1. Adsorption isotherm plots for CPZO extract on MS in 0.5 M H_2SO_4 solution (a) Langmuir isotherm and (b) Frumkin isotherm.

Table 3

Data for Gibbs free energy (ΔG) and equilibrium constant of adsorption (K_{ads}) in for CPZO extract adsorption on MS in H_2SO_4 solution.

LCS Samples	CPZO Concentration (M)	Surface Coverage (θ)	Equilibrium Constant of adsorption (K)	Gibbs Free Energy, ΔG (KJ/mol)
A	0	0	0	0
B	1.20E-02	0.952	1636974.9	-45.41
C	1.80E-02	0.972	1942530.4	-45.83
D	2.40E-02	0.981	2185860.2	-46.13
E	3.00E-02	0.970	1078483.5	-44.37
F	3.60E-02	0.971	932275.3	-44.01
G	4.20E-02	0.966	679036.5	-43.23

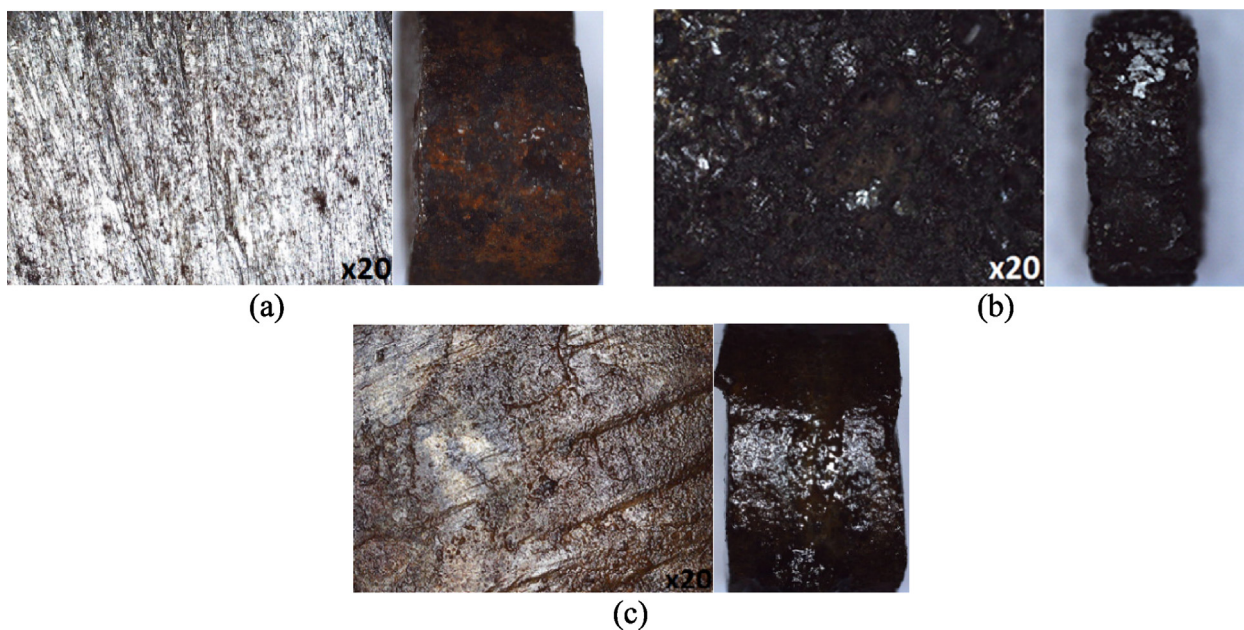


Fig. 2. Optical representations of MS (a) before corrosion test, (b) after corrosion in H_2SO_4 without CPZO and (c) after corrosion in CPZO/ H_2SO_4 solution.

values within 48 h of exposure and thereafter continues to improve till 240 h within effective inhibition data.

Calculated data for standard deviation (SD), mean and margin of error for the inhibition output of CPZO extract are presented in

Table 5. The average data depicts the mean protection effect of CPZO with respect to time of exposure. The SD values displays the degree of differentiation of CPZP inhibition output to the average CPZO inhibition value. The degree of differentiation from aver-

Table 4
ANOVA data for CPZO inhibition performance in 0.5 M H₂SO₄ solution.

Source of Variation	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance (%)
CPZO Concentration	10.83	2.45	0.75
Exposure Time	793.74	2.12	98.63

Table 5
Data for SD, mean, margin of error and proportion above 95% inhibition performance for GG, TR and GF extracts in H₂SO₄ solution.

Concentration (%)	1	1.5	2	2.5	3	3.5
SD	11.75	11.34	10.69	11.47	11.33	9.54
Mean	89.56	90.97	92.63	91.49	90.89	90.96
Margin of Error	6.99 %		Proportion of Data above 90 % Inhibition Efficiency			80 %

age value gives information on the resilience of the protection effect of the extract as time and extract concentration varies. SD values for CPZO inhibitor at all CPZO concentrations vary between 11.75 at 1 % CPZO concentration and 9.54 at 3.5 % CPZO concentration. Despite the variation being marginal but significant, the values show the lateral interaction effect among inhibitor molecule is generally stable. Hence the performance of CPZO is stable with time and concentration as shown in the minimal variation of average values with respect to concentration. The proportion of CPZO inhibition data above 90 % inhibition efficiency is 80 % at margin of error of 6.99 %.

4. Conclusion

Admixture of *Citrus paradisi* and *Zingiber officinale* extracts effectively inhibited high carbon steel corrosion in dilute H₂SO₄ solution at all concentrations studied. Effective corrosion inhibition above 80 % was attained at 48 h of exposure while at 240 h, inhibition efficiency was generally above 90 %. Inhibition effect of the extract chemisorbed on the steel from thermodynamic calculations according to Langmuir and Frumkin adsorption isotherms. Optical images of the corroded steel showed a severely degraded morphology compared to the inhibited steel whose original morphology was sustained by the protective residue of the extract. Analysis of variance (ANOVA) showed variation in extract concentration has limited influence on the corrosion behavior of the steel compared to exposure time wherewith the extract inhibition efficiency varies significantly with time.

CRedit authorship contribution statement

Roland Tolulope Loto: Supervision, Conceptualization, Writing – original draft, Visualization, Investigation, Validation, Methodology, Data curation.

Data availability

Data will be made available on request.

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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