



Synthesis of benzazoles via oxidative cyclization using $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ as a green nanocatalyst

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Abstract

A convenient and efficient method for the synthesis of benzimidazoles, and benzothiazoles have been developed using recyclable magnetite polydopamine-supported copper nanoparticles ($\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$) under green and mild conditions. This catalytic system enables the one-pot synthesis of benzazoles by employing available, and easy to-handle aldehydes and *o*-phenylenediamine/*o*-aminothiophenol. Utilizing sustainable and mild conditions, a variety of functionalized benzazoles has been prepared in nearly quantitative yields (79–97%) within 5–30 min at 40 °C. The method offers advantages such as high yield, short reaction time, use of air as oxidant, ease of workup, avoiding toxic organic solvents, and a simple workup protocol. The catalyst could be recycled and reused up to five times without any loss of significant catalytic activity. The catalyst's great effectiveness can be seen in the fact that the TON reaches 2633–3233 and the TOF reaches 5266–38,800 h⁻¹.

Keywords Benzimidazole · Benzothiazoles · $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ · Green chemistry · Condensation reaction · Reusable

Introduction

In recent years, there has been an increased interest toward the benzo-fused heterocyclic motifs such as benzimidazole and benzothiazole derivatives, due to their growing importance in medical and organic chemistry [1–3]. The efficacy of benzazoles including benzimidazole and benzothiazole compounds as a key pharmacophore in contemporary drug research against influenza, herpes (HSV-1), and HIV viruses has widely recognized in the scientific community [4, 5].

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They are also attractive frameworks because of their reported anti-cancer properties, anti-tumor activity, as well as anti-allergy activity properties [6–8]. To synthesize benzimidazoles and benzothiazoles, two common strategies are used. The first involves condensation of carboxylic acids or their derivatives with *o*-aryldiamines or *o*-nitroanilines or 2-aminobenzenethioles, which usually require high temperatures, acidic conditions, or utilizing of microwave irradiation microwaves [9–12].

An alternative approach entails the oxidative cyclization of *o*-phenylenediamines with aldehydes, using a diverse range of oxidants. The availability of aldehydes has led to improvements in the synthesis of benzimidazoles by condensing of *o*-aryldiamines and 2-aminobenzenethioles with aldehydes [13–19]. Some of the used oxidants and catalytic reagents are Oxone, $\text{PhI}(\text{OAc})_2$, $\text{K}_4[\text{Fe}(\text{CN})_6]$ [16, 20–25].

Although considerable development has been made, the use of nanocatalysts in heterocyclic chemistry is particularly attractive owing to their high reactivity, large surface area, potential to improve the selectivity, and recyclability [26–30].

Magnetite, also known as ferrite (Fe_3O_4), can be used as a versatile support for catalyst functionalization and usually formed effective hybrids with carbon materials, polymers, and organic molecules [31–33]. The unique properties of magnetite and polydopamine motivated us to develop a heterogeneous catalytic system for mild and efficient synthesis of benzimidazoles and benzothiazoles employing $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ nanocatalyst. This magnetic nanocatalysts exhibited remarkable stability and recyclability, thereby leading to a reduction in costs associated with the manufacturing process. These hybrids also reduce waste production, making it an environmentally friendly and greener choice for organic transformations [34–36].

Moreover, this approach benefits from the use of readily available starting materials, resource effectiveness more efficiency and less toxicity, recyclability, and environmental safety.

In the present work, the nanocatalyst was employed in an aqueous environment, which is inexpensive and more ecologically friendly than using conventional organic solvents. Using the magnetic nanocatalyst allows for easy separation of the benzazole products from reaction mixtures and simplifying the purification process.

Experimental section

$\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ nanocatalyst preparation

Fe_3O_4 nanoparticles have been prepared using the co-precipitation method [37]. $\text{Fe}_3\text{O}_4@\text{PDA}$ was prepared by dissolving 0.2 g of dopamine hydrochloride in 20 mL of DMF. A buffer solution of 10 mM Tris with a pH value of 8.5 was prepared in a flask (250 mL) by adding 0.52 g of Tris [Tris(hydroxymethyl)aminometha] in 200 mL of water/ethanol (7:3 V/V). After sonicating the obtained mixture (30 min.), the suspension of Fe_3O_4 magnetic particles was added to a solution containing 0.2 g of dopamine hydrochloride in 20 mL of DMF at room temperature, and the resulting powder— $\text{Fe}_3\text{O}_4@\text{PDA}$ —collected after 15 h, washed with ethanol/water (1:1), and dried at 60 °C. To prepare $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$, the copper-containing solution (0.05 g CuCl_2 in 20 mL of water) was added dropwise to a dispersion of $\text{Fe}_3\text{O}_4@$

PDA (0.15 g) in water (100 mL, 30 min.), and agitated for 12 h at room temperature. Finally, it was rinsed in ethanol and water, then dried for six hours in an oven at 60 °C, and collected using a magnet.

Benzimidazoles and benzothiazoles synthesis

A mixture of aromatic aldehydes (1.2 mmol) and *o*-phenylenediamine/*o*-aminothiophenol (1 mmol) was first stirred in water (5.0 mL) at 40 °C for 1 h. Fe₃O₄@PDA/CuCl₂ nanocomposite (0.03 g) was then added to the above mixture and reacted at 40 °C for the requisite period of time while monitoring the reaction progress by TLC. Upon completion of the reaction, the catalyst was separated using an external magnet for recycling and the residual reaction mixture was diluted with ethyl acetate (2 × 10 ml). The combined organic phase was dried over anhydrous.

Na₂SO₄ was filtered and evaporated under reduced pressure. The desired benzazole products were purified through crystallization with ethanol. Supporting information contains data related to the characterization of the all products and the catalyst (Figs. S1–S5).

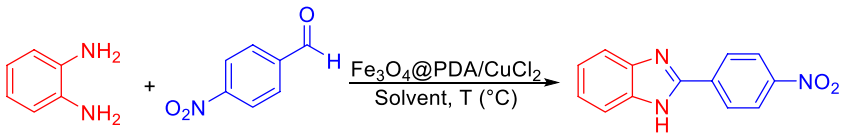
Results and discussion

In this study, Fe₃O₄@PDA/CuCl₂ nanocomposites were synthesized through a simple three-step method and characterized using different techniques such as FT-IR, XRD, SEM–EDX, VSM, and AAS (see SI). In the structure of the catalyst, polydopamine (PDA) as a new emerging benign polymer with terminal amino moieties can facilitate the immobilization of metal centers or further covalent modification [38, 39]. Furthermore, the redox properties of PDA promote the reduction of metal ions into metal nanoparticles, eliminating the need for using reducing agents. As a stabilizing agent, PDA keeps the nanoparticles from aggregation and increases their stability during catalytic processes. To sum up, the Fe₃O₄@PDA/CuCl₂ nanocatalyst integrates the catalytic activity supplied by CuCl₂, the stabilizing effects and biocompatibility of PDA coating, and the magnetic characteristics of Fe₃O₄ nanoparticles [40, 41].

After the characterization of the composite material, a thorough investigation was carried out to assess its catalytic activity in the condensation of various aldehydes with *o*-phenylenediamine, as a model reaction.

Optimization of reaction conditions

The model reaction used in this study is shown in Table 1. In the first step, 0.03 g of Fe₃O₄@PDA/CuCl₂ nanocomposite was added to 5 mL of various solvents, along with 1 mmol of *o*-phenylenediamine and 1.2 mmol of 4-nitrobenzaldehyde, and the mixture was stirred at solvent reflux temperatures. The higher yield of (**1h**) was obtained when the reaction was performed in water (Table 1, No. 1). Other solvents such as ethanol, toluene, acetonitrile, diethyl ether, dimethylformamide (DMF), and

Table 1 *o*-Phenylenediamine's model interaction with 4-nitrobenzaldehyde and the effect of solvent


No. ^a	Solvent	T (°C)	Yield (%) ^b
1	Water	Reflux	89
2	Ethanol	Reflux	80
3	Toluene	Reflux	49
4	Acetonitrile	Reflux	76
5	Diethyl ether	Reflux	62
6	DMF	Reflux	71
7	DMSO	Reflux	72
8	Water	R.T	85
9	Water	40	97
10	Water	60	91

^aReaction conditions: *o*-phenylenediamine (1 mmol) and 4-nitrobenzaldehyde (1.2 mmol), solvent (3 mL), time (5 min), amount of catalyst (0.03 g)

^bIsolated yields

dimethylsulfoxide (DMSO) gave lower yields of the product (**1h**). To find the optimal reaction temperature, the effect of different temperatures was also examined. The results showed that the highest product yield of (**1h**) was obtained at 40 °C.

Then, we studied the necessity and effect of the catalyst loading (Table 2). The reaction between *o*-phenylenediamine and 4-nitrobenzaldehyde in aqueous media without using Fe₃O₄@PDA/CuCl₂ nanocatalyst at 40 °C was found to be very slow and the corresponding product (**1h**) was obtained in low yield (Table 2, No. 1). It was observed that high yield of 2-(4-Nitrophenyl)-1*H*-benzimidazole was achieved using 0.03 g of the catalyst at 40 °C, confirming the importance of Fe₃O₄@PDA/CuCl₂ in the reaction (Table 2, No. 2–5). The model reaction was performed for each component of the nanocatalyst (under the conditions of Table 2), and CuCl₂ alone had a low efficiency (15%), while Fe₃O₄@PDA gave the desired product (1 h) in 65% isolated yield.

Having now developed and optimized conditions for synthesis of benzimidazoles, the synthesis of benzothiazole was optimized using the reaction conditions outlined in Tables 1 and 2. Optimization data showed the same effect of solvent, temperature, and amount of the catalyst, on synthesis of benzimidazole synthesis.

Synthesis of benzimidazoles and benzothiazoles under optimized conditions

A series of substituted aldehydes were employed to prove the general applicability of our present procedure (Table 3) All products were purified by recrystallization and characterized using ¹H-NMR, ¹³C-NMR, and CHN analysis. According to the results, aromatic aldehydes with either electron-deficient or electron-releasing

Table 2 The impact of catalyst amount on *o*-Phenylenediamine's model interaction with 4-nitrobenzaldehyde

No. ^a	Amount of catalyst (g)	Yield (%) ^b
1	–	15
2	0.01	50
3	0.02	69
4	0.03	88
5	0.04	88

^aReaction conditions: *o*-phenylenediamine (1 mmol) and 4-nitrobenzaldehyde (1.2 mmol), water (3 mL), time (5 min), at 40 °C were used to carry out the reaction

^bIsolated yields

groups gave high to excellent yields of the corresponding 2-substituted benzothiazole and benzimidazole derivatives.

In general, benzaldehydes containing electron-withdrawing substituents on the aromatic rings were more reactive than aldehydes with electron-donating groups resulted in a greater yield of the corresponding benzazole derivatives. In fact in these series, slow condensation reaction of electron-rich benzaldehydes can be attributed to a low electrophilicity of such substrates. When unsubstituted benzaldehydes, such as benzaldehyde and 1-naphthaldehyde, were examined as substrates, the desired benzoxazoles (**1a–b**) were obtained in high yields (Table 3, No. 1 and 2). Various benzaldehydes containing electron-deficient groups, such as halogens and -NO₂, were utilized in the reaction conditions (Table 3, No. **1c–1j**). Furthermore, this system could also be applied for higher sterically hindered aldehydes such as 2-dichlorobenzaldehyde and 2,6-dichlorobenzaldehyde (Table 3, No. **1d** and **1e**). These results showed that aldehydes bearing substituents in *meta*- or *para*-position resulted in better results than those *ortho*-substituted benzaldehydes. This variation in product yields with nature and position of substituents may be due to inductive and steric effects. The conversion of the benzaldehydes containing electron-donating substituents (-Me, -OMe, -NMe₂, and -OH) at the *ortho*, *para*, and *meta* positions was carried out successfully to produce moderate to satisfactory yields (Table 3, NO. **1k–1o**). Heterocyclic aldehydes, such as furfuryl aldehyde, exhibited excellent reactivity and the desired product was obtained with an 86% yield (Table 3, NO. **1p**). To enhance the versatility of the catalytic system, we also investigated the condensation reaction of 2-aminothiophenol with various aromatic benzaldehydes as demonstrated by the results obtained in Table 3. To our satisfaction, it was found that the reaction between *o*-aminothiophenol and benzaldehydes in the presence of 0.03 of Fe₃O₄@PDA/CuCl₂ in H₂O as solvent at 40 °C went smoothly and gave the desired products (NO. **1q–1v**).

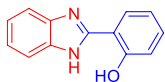
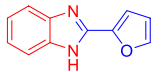
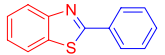
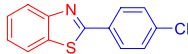
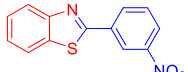
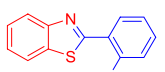
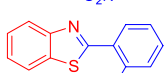
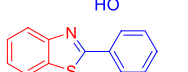
Hot-filtration test

A hot filtration experiment was performed to check the catalyst leaching and its stability (Fig. 1). In certain instances, active sites on solid catalysts may become

Table 3 Synthesis of benzimidazoles (**1a-p**) and benzothiazoles (**1q-1v**) using the Fe₃O₄@PDA/CuCl₂ nanocatalyst

No. ^a	Product	Time (min)	TOF ^b (h ⁻¹)	TON ^c	Melting point (°C) ^(ref)	Yield ^d (%)
1a		30	5467	2733	291–293 (293–294) [42]	82
1b		25	6400	2667	269–272 (270–272) [42]	80
1c		20	9400	3133	294–296 (295–297) [42]	94
1d		25	7040	2933	270–273 (272–275) [43]	88
1e		20	9000	3000	227–230 (228–230) [43]	90
1f		10	19,000	3167	290–292 (291–293) [44]	95
1g		15	12,267	3067	204–206 (206–208) [43]	92
1h		5	38,800	3233	307–309 (309–310) [43]	97
1i		5	37,200	3100	307–310 (310–311) [43]	93
1j		5	35,600	2967	211–213 (210–212) [44]	89
1k		20	8600	2867	222–225 (224–226) [42]	86
1l		25	6960	2900	262–265 (263–265) [43]	87
1m		25	6720	2800	214–216 (216–217) [45]	84
1n		15	11,600	2900	288–290 (289–291) [42]	87

Table 3 (continued)

No. ^a	Product	Time (min)	TOF ^b (h ⁻¹)	TON ^c	Melting point (°C) ^(ref)	Yield ^d (%)
1o		25	6720	2800	234–237 (233–235) [42]	84
1p		20	8600	2867	284–286 (281–284) [46]	86
1q		30	5800	2900	110–113 (112–113) [47]	87
1r		20	9200	3067	114–116 (115–116) [47]	92
1s		5	37,200	3100	186–188 (186–188) [48]	93
1t		5	35,600	2967	135–137 (137–138) [47]	89
1u		25	6720	2800	119–122 (120–121) [49]	84
1v		30	5266	2633	161–163 (162–163) [50]	79

^aReaction conditions: *o*-phenylenediamine (1 mmol) or 2-aminothiophenol (1 mmol) and benzaldehydes (1.2 mmol), water (3 mL), nanocatalyst (0.03 g) and at 40 °C

^bTurnover frequencies (TOF = (Yield/Time)/Amount of catalyst (g))

^cTurnover number (TON = TOF × Time)

^dIsolated yield

isolated during the reaction, and these eluted species can significantly impact the desired product yield. The heterogeneity of the Fe₃O₄@PDA/CuCl₂ catalyst was checked by carrying out a hot filtration test with *o*-phenylenediamine and benzaldehyde as substrates, to find out whether Cu is leaching out from the solid catalyst to the solution or whether the catalyst is truly heterogeneous in nature [51]. After continuing the reaction under optimized conditions for about 15 min., the catalyst was filtered under hot conditions from the reaction mixture with 59% formation of (**1a**). After removal of the solid catalyst, the filtrate was then subjected to the reaction conditions for an additional 15 min. and no further yield of (**1a**) was observed. This observation confirmed no metal leaching and shows that the reaction does not proceed well in the absence of the catalyst.

Catalyst recyclability

One of the most crucial characteristics of magnetic nanocatalysts is catalyst reusability. The reusability of the $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ catalyst was examined in the condensation reaction of 4-nitrobenzaldehyde and *o*-phenylenediamine under the optimized conditions. After completion of the reaction, the solid catalyst was recovered by external magnet and extensively washed with water and ethanol and dried for three hours at 70 °C. Only a marginal loss in the activity of the catalyst was observed for up to five consecutive reactions (Table 4).

In addition, the ICP-AES analysis revealed that the initial copper loaded on the surface of $\text{Fe}_3\text{O}_4@\text{PDA}$ was around 2.5 weight percent, which reaches 2.4 weight percent after 5th run. It can be concluded that the leaching of copper in the reaction mixture after the fifth cycle is extremely low.

Reaction mechanism

On the basis of the above results, and also in accordance with previous literature reports [9, 52, 53], a plausible reaction pathway is proposed as shown in Scheme 1. In the reactions, the amino group of *o*-phenylenediamine/2-aminothiophenol attacks the carbonyl group of the aromatic aldehydes, which is activated by the $\text{Fe}_3\text{O}_4@\text{PDA}/\text{CuCl}_2$ nanocatalyst, giving the intermediates **I** and **II**, respectively. This process is followed by an intramolecular ring closure to produce the N,X-acetal **III**. Finally, acetal **III** undergoes aromatization and dehydrogenation (oxidation with air) to give the final benzazole.

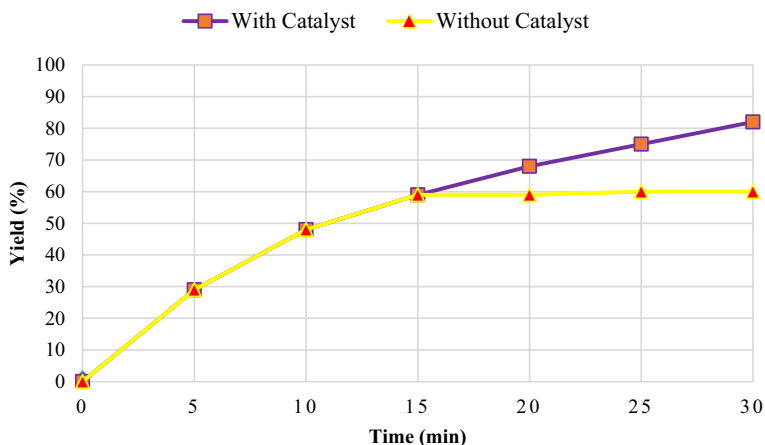


Fig. 1 Diagram illustrating the kinetic curve during the hot filtration test

Table 4 Recycling study of $\text{Fe}_3\text{O}_4@PDA/\text{CuCl}_2$

Run ^a	1	2	3	4	5
Yield (%) ^b	97	97	96	94	94

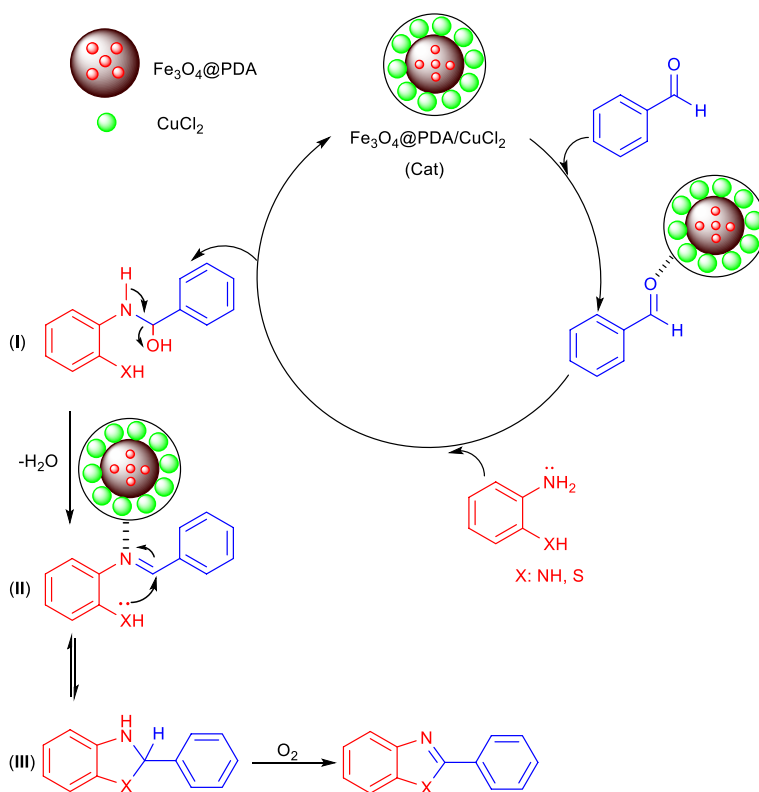
^aReaction conditions: *o*-phenylenediamine (1 mmol) and 4-nitrobenzaldehyde (1.2 mmol), water (3 mL), time (5 min), at 40 °C

^bIsolated yields

A concise evaluation of similarities and differences to prior research

The effectiveness of this nanocatalyst is compared to that of earlier published works in Table 5. The $\text{Fe}_3\text{O}_4@PDA/\text{CuCl}_2$ nanocatalyst demonstrated superior performance in synthesizing benzimidazoles and benzothiazoles.

These comparative results demonstrate the distinct advantage of utilizing heterogeneous $\text{Fe}_3\text{O}_4@PDA/\text{CuCl}_2$ nanocatalyst over the currently employed methods (based on yield, reaction time, and reaction condition). Starting materials for the synthesis of the catalysts are non-toxic, and widely available. These catalytic system



Scheme 1 Possible mechanism in the presence of $\text{Fe}_3\text{O}_4@PDA/\text{CuCl}_2$ for synthesis of benzazole

Table 5 Fe₃O₄@PDA/CuCl₂ as a catalyst in the model reaction **1h** compared to the stated techniques

No.	Catalysts	Catalysts (g)	T (°C)/time (h)	Yield (%)	TON	Solvent	Ref.
1	Fe ₃ O ₄ @PDA/CuCl ₂	0.03	40/0.08	97	38,800	H ₂ O	This work
2	[DodecIm][H ₂ SO ₄]	0.02	RT/5-24	85	850	EtOH	[54]
3	γ-Fe ₂ O ₃ @CuFAp	0.04	80/0.17	96	14,400	ACN	[55]
4	Nano-Fe ₂ O ₃	0.02	80/2-3	70	1750	H ₂ O	[56]
5	Cu(II)Schiff-base@SiO ₂	0.05	RT/2	90	900	EtOH	[57]
6	Urea hydrogen peroxide, Iodine	0.05	70/0.5-1	87	1740	DMSO	[58]
7	(BS- Cu(II))@SiO ₂	0.05	RT/6-24	89	297	EtOH	[59]

requires no time-consuming and laborious purification procedures, unlike previously described methods. The higher values of TON and TOF compared to other catalysts indicate the high efficiency and durability of the present catalyst [54–59].

Conclusions

In conclusion, magnetite polydopamine-supported copper nanoparticles were prepared to facilitate the synthesis of benzimidazoles and benzothiazoles in an aqueous medium, serving as a sustainable and environmentally friendly solvent. The methodology offers several advantages including short reaction time, high effectiveness, the potential for catalyst recycling, and ease of manipulation using an external magnet. Various benzimidazoles and benzothiazoles can be efficiently obtained under mild and green conditions in high yields. The results further illustrate the high activity and stability of the Fe₃O₄@PDA/CuCl₂ by considering its TON (up to 3233) and TOF (up to 38,800 h⁻¹).

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s11164-023-05195-0>.

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Author's contribution The authors were contributed in the development and design of the research. MB participated in investigation, data acquisition, and data analysis. Project management and conceptualization, work supervision, text revision were all done by AA. The project administration, interpretation of data, and conceptualization were conducted by DK. The final version of the manuscript has been reviewed and endorsed by all authors.

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Availability of data and materials The article's supporting information includes the statistics that back up the research's findings.

Declarations

Competing interests No conflicts of interest are declared by the authors.

Consent to participate Not applicable.

Consent for publication Not applicable.

Ethical approval Not applicable.

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