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RESEARCH PAPER

Preparation of 2,3-Disubstituted Quinazoline-4(3H)-One Derivatives in the Presence of CuO/Graphene Oxide as an Effective Catalyst

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ABSTRACT

In this research, a series of 2,3-disubstituted quinazoline-4(3*H*)-one derivatives has been synthesized in high selectivity by one-pot multicomponent reaction through the reflux method. The performance of various techniques was studied comparatively on the attributes of the product and catalyst by applying different characterized tests. The copper oxide/graphene oxide composites (CuO/GO nanocomposite) were well prepared using a co-precipitation method and characterized to confirm their structure and composition. According to the obtained data, the reflux method provides a mild process, and as a result, time and energy are saved. The reaction was efficiently promoted by 20 mg of CuO/GO composite as a robust and heterogeneous nano-sized catalyst. As expected, the proposed heterogeneous nano-sized catalyst that was designed and prepared, performed well in promoting the studied products (up to 93%). High to excellent yield, saving energy and time, chemical/thermal stability, eco-friendliness, and reusability of nanocatalyst (5 runs) are several of the outstanding advantages of this research.

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INTRODUCTION

Graphene Oxide (GO) has been substantially applied in energy storage [1], adsorption [2], catalyst [3], photocatalyst [4], and solar cells [5]. GO is easily soluble in water owing to many hydroxyl groups and hydrophilic reactive oxygen groups on its surface. The presence of –OH and reactive O on the GO or rGO offers various avenues, for instance, great solubility, and the formation of a complex with metal ions: aluminum oxide, iron oxide, and copper oxide [6]. Recently, the rGO surface has been modified with a range of metals oxide, metals, semiconducting materials, and various nanostructures that include Fe_3O_4 [7], TiO_2 [8], Au [9], Pd [10], and many more. This novel hybrid remarkably improves the ability of these structures to perform special tasks in several applications. These hybrid nanostructures often demonstrate improved properties and enhanced functionalities, due to synergic effects between nanoparticles and rGo nanosheets.

The preparation and application of morphology and size-controlled Cu-based nanostructures have always received great attention in materials science and chemistry fields [11]. Especially, Cu-based nanostructures offer distinguished advantages, for instance, facile synthesis, inexpensive cost,

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and high stability which makes them a great candidate for practical applications [12-13]. Although remarkable progress was achieved in the development of CuO/rGO nanostructures, their application in organic reactions as a heterogeneous catalyst needs more exploration [14-17]. Therefore, CuO nanostructures with well-defined shapes on rGO nanosheets are required to study its catalytic performance and establish highly efficient heterogeneous catalytic systems in organic synthesis.

Quinazolines scaffolds are found in numerous alkaloids [18-19]. In medicinal chemistry and organic products preparation, substituted quinazoline scaffolds play chief roles owing to their biological and pharmacological effects (*e.g.* anti-bacterial [20], anti-fungal [21], anticonvulsant [22], anti-hypertensive [23], and anti-cancer [24]). The preparation of substituted quinazolinones scaffolds is hitherto implemented in the presence of diverse catalysts, for instance, *p*-toluenesulfonic acid [25], silica sulfuric acid [26], Fe_3O_4 nanoparticles [27], and Gallium (III) triflate [28].

This paper aims to design and prepare CuO/rGO composite as a robust and reusable nanocatalyst reported for fabricating the 2,3-disubstituted quinazoline-4(3H)-one derivative. A co-precipitation method was used to fabricate the CuO/rGO composite. The experimental data have revealed that the designed nano-sized catalyst has a higher catalytic performance than the reported catalyst. In the following, 2,3-disubstituted quinazoline-4(3H)-one derivative was prepared by a three-component reaction of isotonic anhydride, various aryl aldehyde, and amine components using CuO/rGO composite.

MATERIALS AND METHODS

Synthesis route of GO

GO sheets were prepared via the oxidation of natural powders (Hummers' procedure) [29]. In an ice bath (0 °C), graphite powder (3 g) and potassium permanganate (18 g) were mixed and slowly added to the mixture of H_3PO_4:H_2SO_4 (1:9 ratio), exactly 40 and 360 mL, respectively. After that, the mixture was kept at 50 °C and stirred for 12 h. Next, the resultant solution was first cooled to room temperature and then, H_2O_2 (3 mL, 30%) was added to the ice bath. The resulting mixture was filtered. The obtained solid was washed with distilled water, diluted hydrochloric acid, and ethanol. Finally, the obtained solid was dried in a vacuum oven (30 °C).

Synthesis route of nano-sized CuO/rGO composites

The nano-sized CuO/rGO composite was synthesized through a co-precipitation procedure in an alkaline medium [30]. Added rGO powder (0.2 g) to DI water (100 mL) and the mixture was sonicated at 40 °C for 60 min. The nitrate salts of

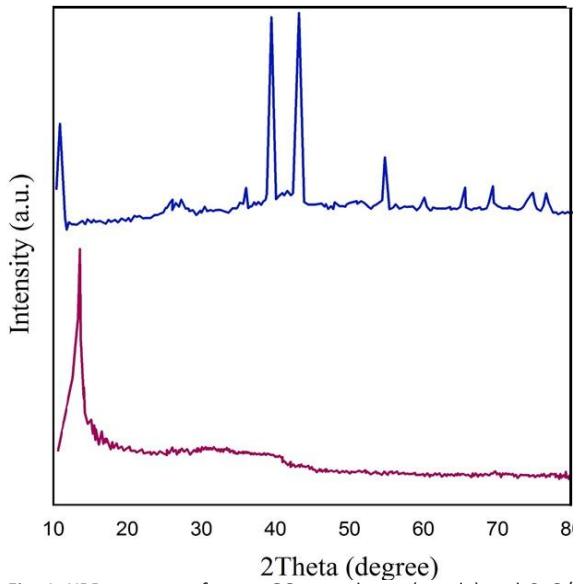

Fig. 1. XRD patterns of pure rGO nanosheets (purple) and CuO/rGO nanocomposites (blue).

the copper $^{(II)}$ (1.5 g) and nickel $^{(II)}$ (1.5 g) were added to the above-dispersed solution, respectively. The resulting mixture was stirred for 2 h at room temperature. For pH 10-11, ammonia solution (0.5 *M*) was droply added and then, stirred for 2 h at room temperature again. The solid product was washed with DI water, dried, and calcined at 400 °C for 2 h, yielding the nano-sized CuO/rGO composite.

Typical method for the synthesis of quinazolinone derivatives

Typically, the isatoic anhydride (1 mmol), various aryl aldehyde (1 mmol), amine components (aryl amines, aliphatic amines, and ammonium acetate), CuO/rGO (0.25 g, catalyst), and ethanol as green solvent (7 mL), were mixed at reflux conditions for an appropriate duration of time. Thin layer chromatography (TLC) technique was used to investigate the completion of the reaction. The desired compounds were characterized by melting point, FT-IR, ^{1}H & ^{13}C NMR, and CNHS.

RESULTS AND DISCUSSION

Characterization of CuO/rGO nanocomposites

The XRD patterns of pure rGO nanosheets and CuO/rGO nanocomposites are shown in Fig. 1. Based on Fig. 1a, a district diffraction peak at *2 theta:* 11.75° is related to rGO nanosheets [31]. The CuO/rGO nanocomposites show typical diffraction peaks at *2 theta*: 32.7, 3.8, 38.5, 49.1, 53.6, 58.2, 61.5, 65.9, 66.6, 68.2, 72.4, and 75.6 degrees (JCPDS Card No. 80-1916) (Fig. 1b) [32]. These peaks confirmed the presence of the CuO nanostructures. Also, Miller's index (001) was seen in the final XRD pattern.

The investigation of functional groups of pure rGO was done by FT-IR analysis. The FT-IR spectrum of rGO is shown in Fig. 2a. The absorption band at 3450 cm^{-1} and also a weak peak at 1620 cm^{-1} come from the -OH stretching and bending peaks, respectively. Absorption peaks at 1170, 1575, 1691, and 2923 cm^{-1} were related to C-O, C=C $_{aromatic}$, C=O, and C-H_{sp2} groups, respectively (Fig. 2a) [33]. From the final spectrum, the new band at 537 cm^{-1} is related to the Cu-O band. According to this, the CuO/rGO nanocomposite was formed (Fig. 2b).

The composition was tested by EDX analysis. The elemental test results of the CuO and CuO/rGO nanocomposite are tabulated in Fig. 3. As seen, the chemical composition of the pure CuO and CuO/rGO nanocomposite contain C, Cu, and O elements without any purities.

The surface morphology of pure CuO and CuO/rGO nanocomposite was studied via the FE-SEM technique. The FE-SEM results are shown in Fig. 4. The morphology of pure CuO nanoparticles formed a spherical structure (Fig. 4a). From Fig. 4b, the surface of CuO nanoparticles was fully

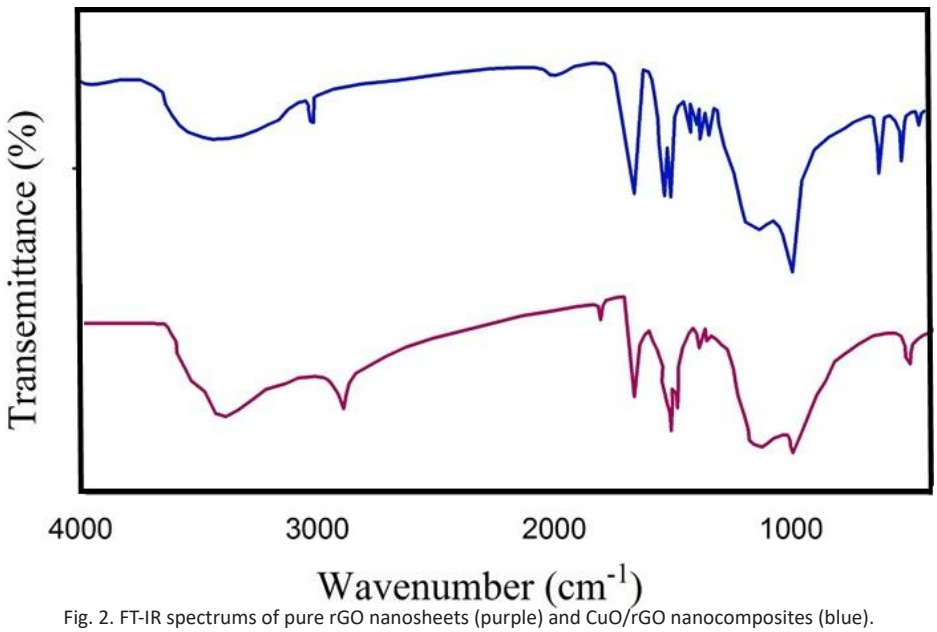

Fig. 2. FT-IR spectrums of pure rGO nanosheets (purple) and CuO/rGO nanocomposites (blue).

covered by rGO nanosheets.

Catalytic performance of CuO/rGO nanocomposite in preparation of 2,3-disubstituted quinazoline-4(3H)-one scaffold

The preparation of 2,3-disubstituted quinazoline-4(3*H*)-ones can be found in various published reports [34-37]. It is interesting to note that every one of these methods can prepare these compounds. Thus, a comprehensive comparative investigation was conducted to highlight the performance of the catalyst and optimization of the methodology (Table 1). The reported outcomes (Table 1) revealed that the as-prepared final product in the presence of the CuO/rGO catalyst has some advantages including the highest yield of synthetic product and reasonable reaction time in mild conditions.

The one-pot tandem reaction: isatoic anhydrate (1 mmol), benzaldehyde (1 mmol), and diethylamine (1 mmol) was primarily chosen as a sample reaction to study of catalytic performance of the as-prepared CuO/rGO nanocomposites. The reaction was optimized by scrutinizing various conditions (solvents and catalyst dosage). The empirical data are summarized in Table 2. From empirical data, the protic solvents revealed the best results. The protic solvents have a greater

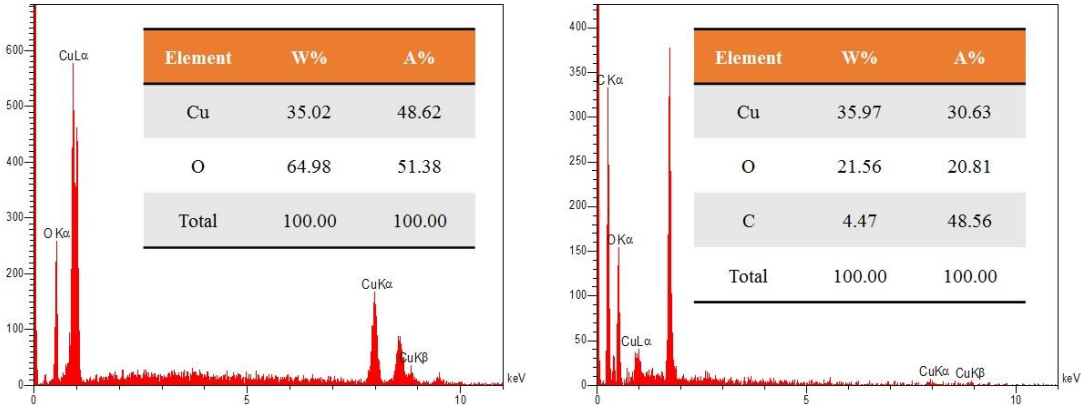

Fig. 3. EDX analysis of pure CuO nanoparticles (a) and CuO/rGO nanocomposites (b).

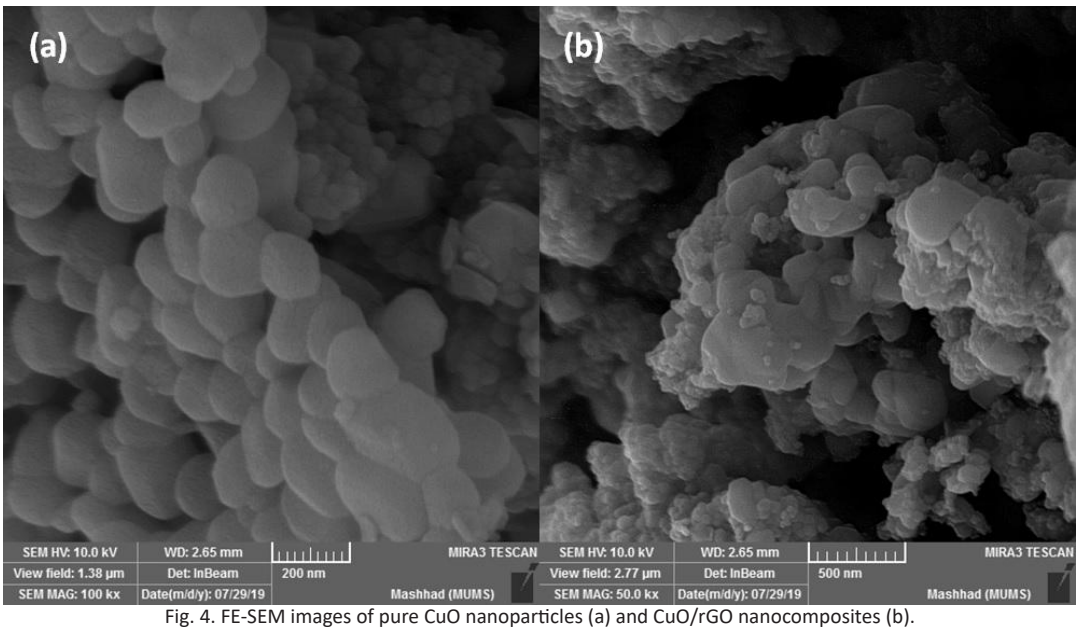

Fig. 4. FE-SEM images of pure CuO nanoparticles (a) and CuO/rGO nanocomposites (b).

ability to solve anion intermediates. The activation energy was therefore decreased and the compounds were formed with a significant yield in a short period [38]. Ethanol was also chosen as the most suitable solvent. After that, to study various catalysts, pure CuO, pure rGO, and CuO/rGO composite were tested. We observed that convincing data were provided when the CuO/rGO composite was present in the reaction. The catalyst dosage was also checked. The reaction yield was improved from 85% to 93% when the catalyst dosage was adjusted to 10, 15, and 20 mg. The catalyst dosage was increased (up to 25 mg), but the reaction yield did not change. To expand the scope of our protocol, different electron-withdrawing/donating aryl aldehydes and amine components were employed for the preparation of 2,3-disubstituted quinazoline-4(3H)-ones under optimization conditions. The finding demonstrated that electron-withdrawing aryl aldehydes gave the obtained product excellent yields (Table 3).

A reaction mechanism in preparation of 2,3-disubstituted quinazoline-4(3H)-ones

An acceptable reaction mechanism is proposed in Scheme 1. In short, the designed nanocatalyst has played a vital Lewis acid role. Therefore, the acidic sites of CuO/rGO composite bind with electron pair O atom of carbonyl and π electron of C=N group. The primary amine attack on

Table 1. Comparison of various conditions for the preparation of 3-ethyl-2-phenyl quinazoline-4(3*H*)-ones.

No.	Catalyst (dosage)	Solvent	Conditions	Time	Yield	Ref.
1	*p*-Toluenesulfonic acid (0.6 mmol)	Ethanol	Reflux	5.5 h	70%	[25]
2	Silica sulfuric acid (0.11 g)	Ethanol	Reflux	3 h	90%	[26]
3	Gallium (II) triflate (1 mol%)	Ethanol	Reflux	40 min	91%	[28]
4	CuO/rGO nanocomposite (20 mg)	Ethanol	Reflux	35 min	93%	*This job*

Table 2. Optimization of conditions for the fabrication of the 2,3-disubstituted quinazoline-4(3*H*)-ones[a].

No.	Solvents	Catalyst dosage	Conditions	Time (min)	Yield (%)[b]
1	Dichloromethane	20 mg	Reflux	65	< 13
2	Acetonitrile	20 mg	Reflux	40	54
3	Water	20 mg	Reflux	52	68
4	Ethanol	Nano-CuO 15 mg	Reflux	40	70
5	Ethanol	Nano-rGO 28 mg	Reflux	50	61
6	Ethanol	10 mg	Reflux	50	85
7	Ethanol	15 mg	Reflux	45	90
11	Ethanol	20 mg	Reflux	35	93
12	Ethanol	25 mg	Reflux	35	93

[a] Reaction conditions: Isatoic anhydrate (1 mmol), benzaldehyde (1 mmol), and diethylamine (1 mmol) in the presence of CuO/rGO composites as a catalyst.
[b] Isolated yield

Table 3. Fabrication of 2,3-disubstituted quinazoline-4(3H)-ones using the CuO/rGO composites under reflux conditions. [a]

No.	amine	R'	Time (min)	Yield (%)	m.p. (˚C) *Observed*	m.p. (˚C) *Reported*	TOF	Ref.
1	NH_3	H	35	90	234-235	234-235	1.54	[32]
3	$EtNH_2$	4-NO_2	35	93	191-192	190-192	2.05	[33]
3	$EtNH_2$	4-OMe	43	89	125-127	125-128	1.94	[33]
4	$EtNH_2$	4-Cl	35	92	111-112	108-112	1.97	[33]
5	$EtNH_2$	4-Br	38	92	109-110	110-112	2.28	[34]
6	$EtNH_2$	4-CN	37	90	192-193	190-192	1.91	[34]
7	(*p*-Tolyl)ethylamine	4-Me	48	88	145-146	144-146	2.36	[34]
8	Benzylamine	H	50	89	152-153	152-153	2.17	[35]
9	Propylamine	H	35	91	124-125	--	2.18	*Our Job*
10	Butylamine	H	50	88	101-103	--	2.28	*Our Job*
11	Butylamine	4-NO_2	36	92	242-243	242-244	2.24	[33]
12	Butylamine	4-Cl	39	90	204-205	203-205	2.17	[34]

[a] Reaction conditions: isatoic anhydride (1 mmol), various aryl aldehyde (1 mmol), amine components (1 mmol), and ethanol (7 mL) under ultrasound probe-treated in the presence of nano-scale TiO_2@SiO_2 composite
[b] Isolated Yield
TOF: (mmol of reagent×Yiled) ÷ mmol of catalyst

activated C=O (isatoic acid). Next, decarboxylation was observed and intermediate (I) was formed. An aryl aldehyde carbonyl group is the target of nucleophilic attack in the next step (intermediate II). The cyclization and intramolecular nucleophilic attack of intermediate (II) on imine was done to form the intermediate (III). In the final step of the mechanism, the obtained product was formed via oxidation.

Reusability of CuO/rGO Catalyst

The investigation of reusability is one of the critical characteristic features of catalysts. Hence, the model reaction was conducted again (under optimized conditions). After the accomplishment of the reaction process, the as-prepared catalyst was recovered. It was then washed with cold acetone three times (3×12 mL), dried at 40 °C for 5 h, and reused for further cycles with a fresh

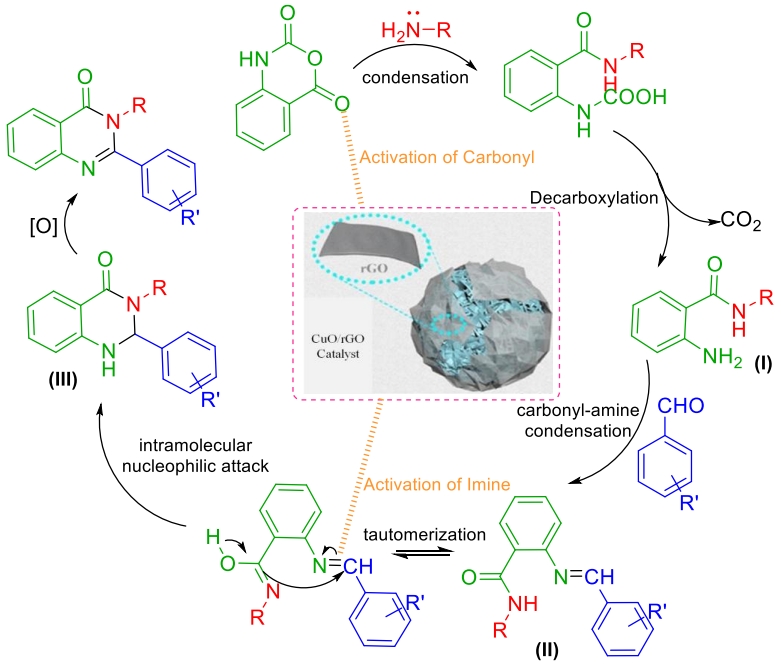

Fig. 5. Proposed reaction mechanism.

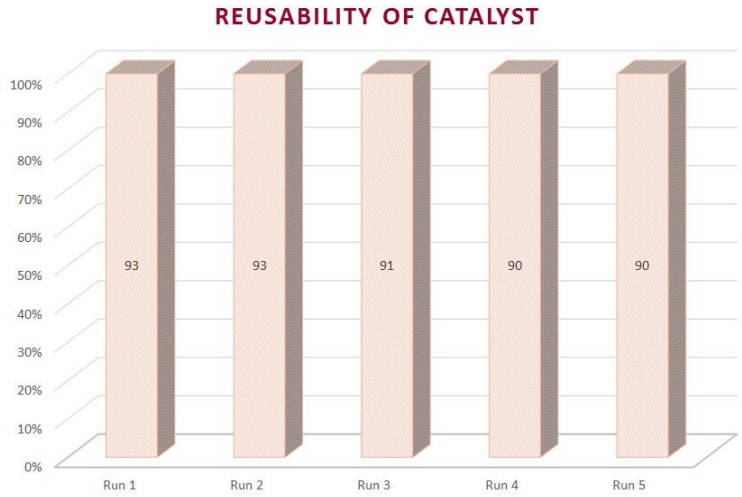

Fig. 6. Reusability of CuO/rGO composites.

surface. Findings revealed that the CuO/rGO heterogeneous catalyst could well be used for seven cycles with no dramatic loss in its efficiency (Fig. 5).

CONCLUSION

As a result, we have used the co-precipitation method as a facile strategy to design the robust CuO/rGO composite as a catalyst. Moreover, 2,3-disubstituted quinazoline-4(3*H*)-one derivatives was well synthesized via reflux conditions. Also, high to excellent yields within short reaction time, reduced catalyst dosage, green solvent, and reusability of catalyst (5 runs) are other advantages of this research. For future studies, the prepared products have the potential to become an oral antibacterial drug and also, the role of catalyst morphology in the reaction process, the use of a catalyst prepared via plant extraction, re-checked drug design methods, and computational chemistry in aliphatic aldehydes can help improve research in this field.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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