

SYNTHESIS, STRUCTURAL CHARACTERIZATIONS AND BIOLOGICAL PROPERTIES OF Cd(II) AND Zr(IV) PEROXO COMPLEXES CONTAINING SCHIFF BASE DERIVED FROM CINNAMALDEHYDE AND *o*-AMINO BENZOIC ACID

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Abstract. Schiff base (SB) containing two peroxo complexes of Cd(II) and Zr(IV) ion were synthesized and characterized by thin layer chromatography (TLC), elemental analyses, conductivity measurements, magnetic moment measurements, UV-Vis. , IR and ESI-MS spectral studies. The metal ions were participated in direct complexation with the Schiff base ligand derived from cinnamaldehyde and *o*-aminobenzoic acid in a single pot. IR spectra showed that the Schiff base ligand coordinated to the metal ion through nitrogen of azomethine group and oxygen of carboxyl group (COO⁻) & peroxo group (O-O). ESI-MS spectra confirm the molecular mass of the complexes. The molar conductance values indicated the non-electrolytic nature of both the complexes. Antibacterial activity of the complexes was tested against four pathogenic bacteria, two Gram-positive *Staphylococcus aureus* & *Bacillus cereus* and two Gram-negative *Escherichia coli* & *Shigella dysenteriae* with standard *Kanamycin-30*. Also, the antifungal activities of both the Cd(II) and Zr(IV) complexes were tested against four pathogenic fungi *Aspergillus flavus*, *Penicillium species*, *Candida species* and *Aspergillus niger* with standard *Fluconazole-50*. The antimicrobial activity results showed that Cd(II) complex possesses more potential antibacterial activity than the Zr(IV) complex against all the tested organisms and both the complexes showed moderate to strong antifungal activity against all the tested pathogenic fungi.

Keywords: Schiff base, Peroxo complexes, Cinnamaldehyde, *o*-Aminobenzoic acid, Antimicrobial activity.

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1. Introduction

Schiff bases are important type of ligands possessing versatile applications (Bitu *et al.*, 2019). They are well known for their biological applications as antibacterial, antifungal, antiviral, anticancer and antitumor agents (Hossain *et al.*, 2017; Rahman *et al.*, 2015). Again, metal-amino acid (Alcock *et al.*, 1985) as well as peroxo-metal system has increasing interest due to their application in synthetic and coordination chemistry (Chaudhuri, 1988; Karlin, 1985; Mimoun, 1983). This helps us to understand biologically important molecules and promoted to establish rational synthetic routes to peroxo complexes containing an amino acid (Mimoun, 1987). Many peroxo complexes of transition metals having fantastic catalytic activity are excellent sources of active

oxygen atoms (Herrmann *et al.*, 1996; Salles *et al.*, 1997). Importantly, peroxy complexes undergo oxygen transfer reactions. Many of these complexes possess to have potential antimicrobial activity such as antibacterial activity, antifungal activity and cytotoxic properties (Kudrat-E-Zahan *et al.*, 2004; Alam *et al.*, 2004).

Keeping these facts in view, the present research deals with the studies on the synthesis, spectral and potential biological properties of Cd(II) and Zr(IV) peroxy complexes containing Schiff base derived from cinnamaldehyde and *o*-aminobenzoic acid during the single pot reaction.

2. Materials and Methods

2.1. Reagents and chemicals

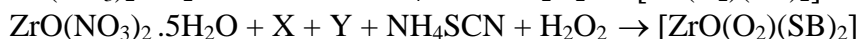
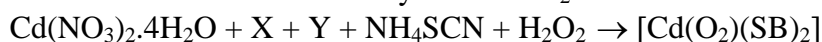
All the reagents and chemicals were analytical grade and used as supplied. Zirconium oxynitrate and cadmium nitrate salts were purchased from Loba Chemie pvt. Ltd. (India). Cinnamaldehyde and *o*-aminobenzoic acid were purchased from BDH Chemicals Ltd. (England). Silica gel used as the stationary phase of thin layer chromatography (TLC) was purchased from Thomas Baker (India).

2.2. Physical Measurements

A Mettler PM-200 electronic balance was used to carry out all the weighing operations. An electro thermal melting point apparatus (model AZ 6512) was employed to obtain the melting point of the complexes. Molar conductance of the complexes in 1.0×10^{-3} mol/dm³ DMSO solutions were measured at room temperature using a type CG 857 No. 71798 SCHOTT-GERATE Gumbo, Germany, digital conductivity meter and a dip-cell with platinized electrodes. The magnetic susceptibility was measured by a SHERWOOD SCIENTIFIC magnetic susceptibility balance. Infrared spectra were recorded as KBr disc with a NICOLET 310, FTIR spectrophotometer, Belgium, from 4000-400 cm⁻¹. UV-visible spectra were recorded with a SHIMADZU DOUBLE BEAM spectrophotometer (model UV-1650pc).ESI- Mass spectral data were obtained on ESI-esquires 3000 Bruker Daltonics spectrometer. C, H, N analyses were performed by PerkinElmer 2400 organic elemental analyzer-II at Okayama University, Japan.

2.3 General method for the preparation of the Cd(II) and Zr(IV) complexes:

For Cd(II) complex (0.6169 g, 2 mmol) of Cd(NO₃)₂.4H₂O and for Zr(IV) complex (0.6426 g, 2 mmol) of ZrO(NO₃)₂.5H₂O was dissolved in 5 mL ethanol in a 50 mL beaker. A solution of (0.5304 g, 4 mmol) of cinnamaldehyde was made in 5 mL ethanol in a 50 mL beaker. A solution of (0.5485 g, 4 mmol) of *o*-aminobenzoic acid was also made in 5 mL ethanol in another 50 mL beaker. Then a solution of ammonium thiocyanide (0.3044 g, 4 mmol) was prepared in 5 mL ethanol in another 50 mL beaker. The four solutions were mixed in a round bottom flask and stirred for half an hour at room temperature and then allowed to stand for several minutes. After that about (8 mmol) of 30 % H₂O₂ was poured into the beaker. The mixture was stirred for 22 hours at room temperature. The completion of the reactions was monitored by thin layer chromatography (TLC). After completion of reaction a solid precipitate was observed. The resulting precipitate was filtered, washed with cold ethanol and diethyl ether and dried in vacuum desiccator over anhydrous CaCl₂.



Where,

X = Cinnamaldehyde

Y = *o*-Aminobenzoic acid

SB = Schiff base derived from X and Y during reaction.

2.3.1. Cd(II) Complex, [Cd(O₂)(SB)₂]:

Color: Green, Melting point: 285⁰C, Yield: 75 %, Molar conductance (Ohm⁻¹cm²mol⁻¹): 9.5, Magnetic moment (μ_{eff} /B.M.): 0.47, FT-IR (cm⁻¹): 1619s, $\nu(\text{C}=\text{N})$; 1533w, $\nu_{\text{asy}}(\text{COO}^-)$; 1384, $\nu_{\text{sy}}(\text{COO}^-)$; 884s, $\nu_1(\text{O}-\text{O})$; 601w, $\nu_2(\text{M}_\text{o}^\text{o})$; 726s, $\nu_3(\text{M}_\text{o}^\text{o})$, 474s, $\nu(\text{M}-\text{O})$; 437m, $\nu(\text{M}-\text{N})$; UV-Vis. (λ_{max} /nm): 400. Elemental analysis for [CdC₃₂H₂₄N₂O₆], (M.W.= 644.95): Calculated: C, 59.59; H, 3.75; N, 4.34 %. Found: C, 59.07; H, 3.87; N, 4.39 %.

2.3.2. Zr(IV) Complex, [ZrO(O₂)(SB)₂]:

Color: Deep yellow, Melting point: >300⁰C, Yield: 70%, Molar conductance (Ohm⁻¹cm²mol⁻¹): 7.0, Magnetic moment (μ_{eff} /B.M.): 0.53, FT-IR (cm⁻¹): 1620s, $\nu(\text{C}=\text{N})$; 956m, $\nu(\text{M}=\text{O})$; 1534w, $\nu_{\text{asy}}(\text{COO}^-)$; 1404, $\nu_{\text{sy}}(\text{COO}^-)$; 884w, $\nu_1(\text{O}-\text{O})$; 602w, $\nu_2(\text{M}_\text{o}^\text{o})$; 727s, $\nu_3(\text{M}_\text{o}^\text{o})$, 475m, $\nu(\text{M}-\text{O})$; 437m, $\nu(\text{M}-\text{N})$; UV-Vis. (λ_{max} /nm): 405. Elemental analysis for [ZrC₃₂H₂₄N₂O₇], (M.W.= 639.77): Calculated: C, 60.08; H, 3.78; N, 4.38 %. Found: C, 59.87; H, 3.84; N, 4.43 %.

Here, the related band intensities for FT-IR are denoted by s, m, and w representing strong, medium, and weak band respectively.

3. Results and Discussion

3.1 Elemental Analysis and Conductivity Measurement

The molar conductance of both the complexes in 10⁻³ M dimethyl sulphoxide (DMSO) solution were measured at 30°C. The molar conductance value (7.0 & 9.5 $\Omega^{-1}\text{cm}^2\text{mol}^{-1}$) indicate that both the complexes are non-electrolytic in nature (Elachi *et al.* 2019). The elemental analysis data are in good agreement with the proposed structure (Fig. 1) of the complexes.

3.2 Magnetic Moment and Electronic Spectra

The observed values of effective magnetic moment ($\mu_{\text{eff}} = 0.47$ to 0.53 B.M.) at room temperature suggested that both the complexes possess to have no unpaired electron i.e. they are diamagnetic in nature indicating no changes in the oxidation state of the metal ions upon complexation. The UV-Vis. spectral data of the complexes exhibited absorption bands between 400 to 405 nm region due to the charge transfer band only (Tsumaki, 1938; Agarwal *et al.*, 1995).

3.3 IR spectral studies

IR spectral data of the complexes showed $\nu(\text{C}=\text{N})$ stretching frequency in between 1619-1620 cm⁻¹ indicate the presence of azomethine linkage in the complexes. The appearance of absorption band in the range (1533-1534 cm⁻¹) and (1384-1404 cm⁻¹) indicates the asymmetric $\nu_{\text{asy}}(\text{COO}^-)$ and symmetric $\nu_{\text{sy}}(\text{COO}^-)$ stretching frequencies respectively. Further, the presences of $\nu(\text{M}-\text{O})$ stretching frequency in the range 474-475 cm⁻¹ and $\nu(\text{M}-\text{N})$ stretching frequency at 437 cm⁻¹ in the spectra of the complexes indicating the coordination through O and N atom (Djordjevic *et al.*, 1993; Shinohara *et al.*, 1991; Ray *et al.*, 2015; Fujisawa *et al.*, 1991; Kurosawa *et al.*, 1991; Schmidt *et al.*, 2001; White *et al.*, 1972; Porta *et al.*, 1990).

Three IR active vibrational modes due to the presence of metal peroxo group in the complexes are observed in the IR spectra. These are predominantly O-O stretching ν_1 , the symmetric ν_2 stretch and the antisymmetric ν_3 stretch. The characteristics ν_1 (O-O) modes of the complexes appear at 884 cm^{-1} whereas the ν_2 and ν_3 modes appear in between $601\text{--}602\text{ cm}^{-1}$ and $726\text{--}727\text{ cm}^{-1}$ respectively. In case of Zr(IV) complex appearance of band at 956 cm^{-1} indicates the presence of $\nu(\text{M}=\text{O})$ stretching.

3.4 ESI-MS spectral studies

The ESI mass spectra of the peroxo complexes are shown in Fig. 1. The observed peaks in the spectra confirm the elemental composition of the ions observed and supports the proposed structure of the synthesized complexes (Fig. 2). The most abundant fragment ions are given in Table 1.

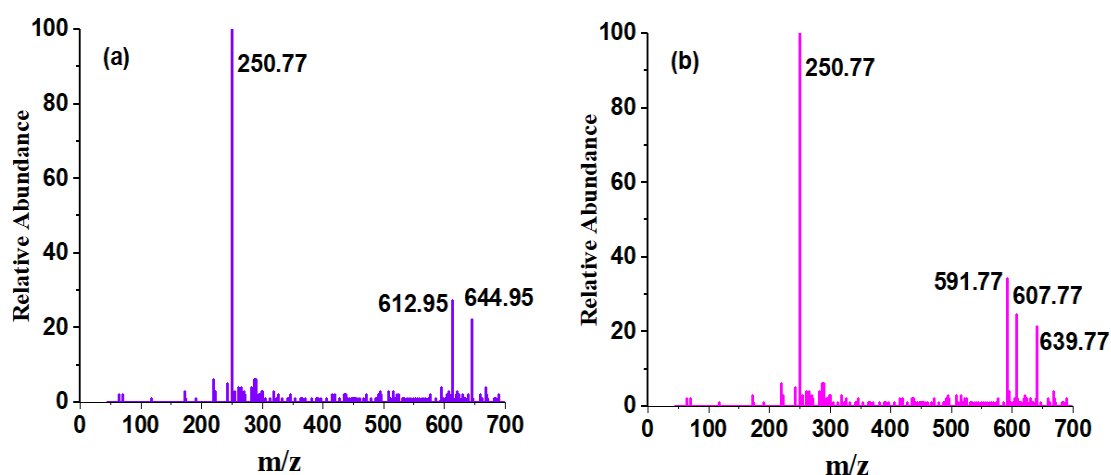


Figure 1. ESI-MS spectra of the complex (a) $[\text{Cd}(\text{O}_2)(\text{SB})_2]$ and (b) $[\text{ZrO}(\text{O}_2)(\text{SB})_2]$

Table 1. ESI-MS spectral data of the peroxo complexes

$[\text{Cd}(\text{O}_2)(\text{SB})_2]$	m/z (%)	$[\text{ZrO}(\text{O}_2)(\text{SB})_2]$	m/z (%)
Observed Ions		Observed Ions	
$[\text{Cd}(\text{O}_2)(\text{SB})_2]^+$ (644.95 g mol^{-1})	644.95 (22.34)	$[\text{ZrO}(\text{O}_2)(\text{SB})_2]^+$ (639.77 g mol^{-1})	639.77 (21.45)
$[\text{Cd}(\text{SB})_2]^+$	612.95 (27.42)	$[\text{ZrO}(\text{SB})_2]^+$	607.77 (24.65)
$(\text{SB})^+$	250.27 (100.0)	$[\text{Zr}(\text{SB})_2]^+$	591.77 (34.24)
		$(\text{SB})^+$	250.27 (100.0)

Based on the above analyses the proposed structure (Fig. 2) of the synthesized peroxy complexes of Cd(II) and Zr(IV) are shown below.

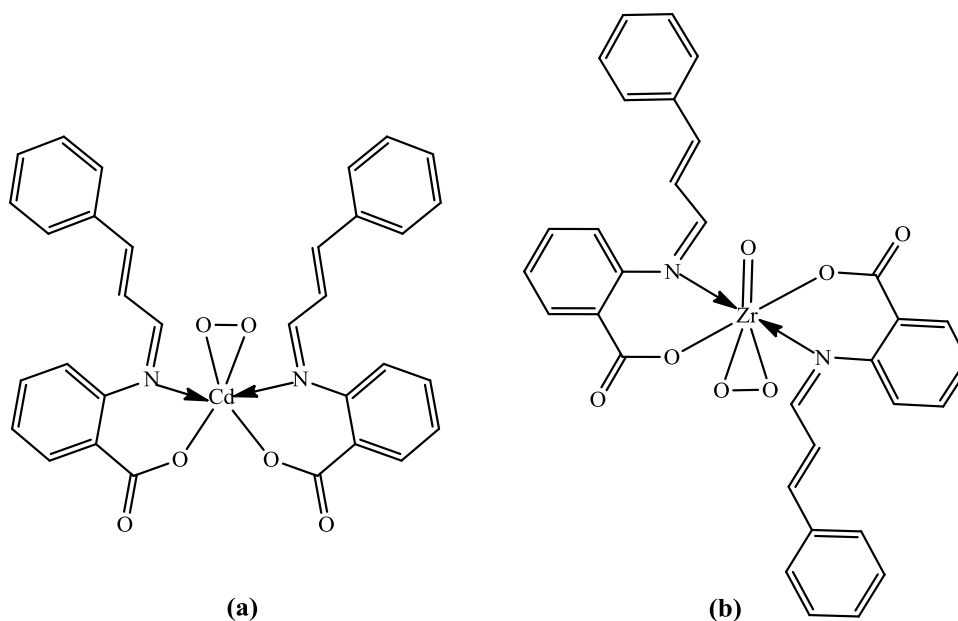


Figure 2. Proposed structure of the (a) Cd(II) complex and (b) Zr(IV) complex

4. Biological Activities of the Metal Complexes

4.1. Antibacterial Activity

There exist different methods to determine the susceptibility of the pathogenic microorganism to the synthesized compounds. Among these methods the disc diffusion method is largely acceptable for performing the antibacterial screening of the complexes (Kudrat-E-Zahan *et al.*, 2015; Latif *et al.*, 2019; Kudrat-E-Zahan *et al.*, 2019). Antibacterial activity of the complexes expressed by measuring the diameter of zone of inhibition are tabulated in Table 2. The complexes (50 µg/0.01 mL in DMSO solution) were screen for their antibacterial activity against four pathogenic bacteria namely *Escherichia coli*, *Shigella dysenteriae*, *Staphylococcus aureus* & *Bacillus cereus*, with *Kanamycin (K-30)* standard. The result showed that Cd(II) complex possess to have more potential antibacterial activity than the Zr(IV) complex against all the tested bacteria. This can be explained due to enhanced lipophilic property of the central metal ions as a result of chelation with ligand moieties (Rios *et al.*, 1988). The results are graphically represented in Fig.3.

Table 2. Antibacterial activity of the Cd(II) and Zr(IV) complexes with standard *Kanamycin*

Compounds	Diameter of zone of inhibition (mm) for 50 µg/disc of the samples			
	Gram Negative		Gram Positive	
	<i>Escherichia coli</i>	<i>Shigella dysenteriae</i>	<i>Staphylococcus aureus</i>	<i>Bacillus cereus</i>
[Cd(O ₂)(SB) ₂]	18	13	23	15
[ZrO(O ₂)(SB) ₂]	09	06	11	06
Kanamycin -30	25	22	28	21

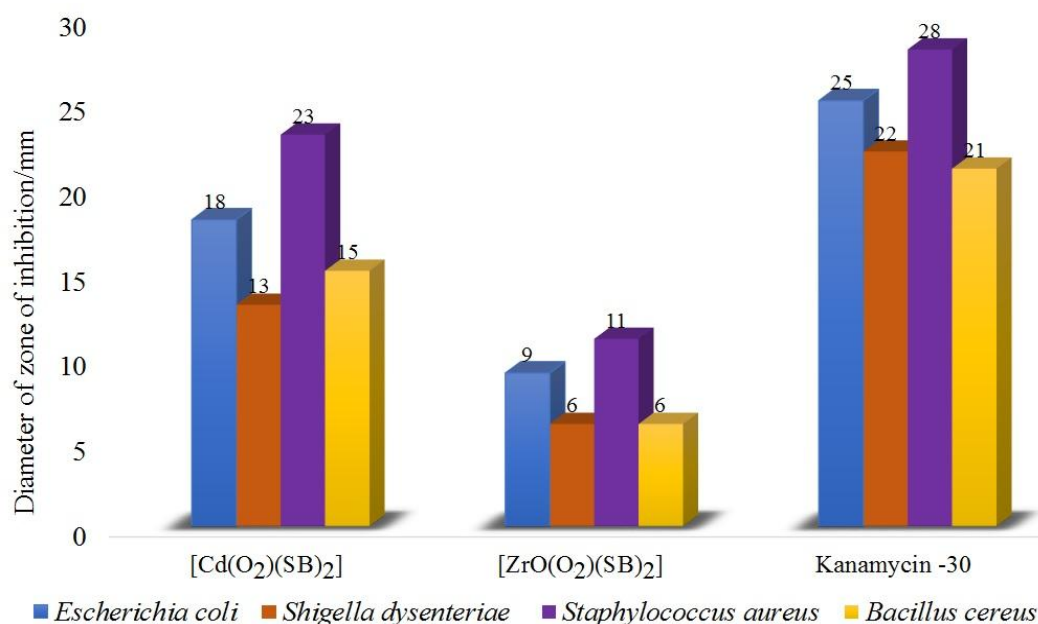


Figure 3. Graphical representation of antibacterial activity of the complexes against *E. coli*, *S. dysenteriae*, *S. aureus* and *B. cereus*.

4.2. Antifungal Activity

The antifungal activities of both the Cd(II) and Zr(IV) complexes (100 µg/disc) were tested against four pathogenic fungi *Aspergillus flavus*, *Penicillium species*, *Candida species* and *Aspergillus niger* with standard *Fluconazole* (F-50 µg/disc) (Nasrin, 2015). The measured diameter of zone of inhibition for the antifungal activity of the complexes are tabulated in Table 3. From the result, it is observed that both the complexes showed moderate to strong activity against all the tested pathogenic fungi. The metal complexes showed greater antifungal activity as compared with free ligand. This can be explained on the basis of chelation theory (Mohapatra *et al.*, 2019). The results are graphically represented in Fig.4.

Table 3. Antifungal activity of Cd(II) and Zr(IV) complexes with standard *Fluconazole-50*.

Compounds	Diameter of zone of inhibition (mm) for 100 µg/disc of the samples			
	<i>Aspergillus flavus</i>	<i>Penicillium species</i>	<i>Candida species</i>	<i>Aspergillus niger</i>
[Cd(O ₂)(SB) ₂]	09	08	11	07
[ZrO(O ₂)(SB) ₂]	08	10	12	08
Fluconazole-50	23	21	24	22

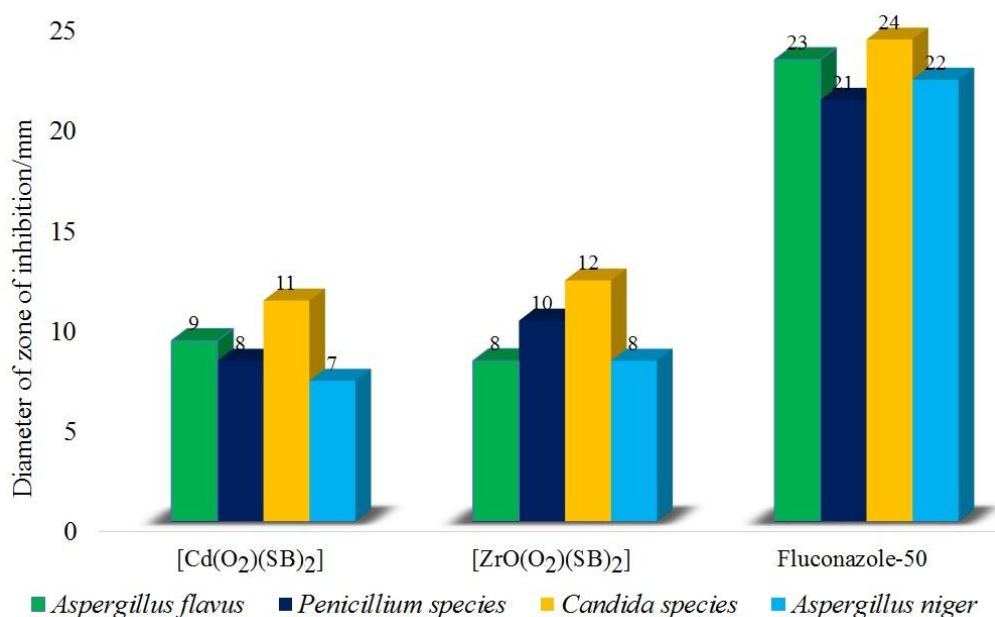


Figure 4. Graphical representation of antifungal activity of the complexes against *Aspergillus flavus*, *Penicillium species*, *Candida species* and *Aspergillus niger*

5. Conclusion

In this paper we have explored the synthesis, characterization and biological investigation of the peroxo complexes of Cd(II) and Zr(IV) ion containing Schiff base (SB) derived from cinnamaldehyde and *o*-aminobenzoic acid. The completion of all reactions were monitored by using thin layer chromatography (TLC). IR spectral data indicated that the ligand coordinated to the central metal ion through N and O atoms. Elemental analyses, conductivity measurements, magnetic moment measurements, UV-Vis, FT-IR and ESI-MS spectral data confirmed the proposed structure of metal complexes. The result of antimicrobial activity showed that Cd(II) complex possesses more potential antibacterial activity than the Zr(IV) complex against all the tested organisms and both the complexes showed moderate to strong antifungal activity against all the tested pathogenic fungi.

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Conflict of Interests

Authors have declared that there exist no conflict of interests.

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