

Original Article

Correlation between Structural and Optical features of NiO–ZnO Hybrid Oxide for sensing applications

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**Abstract**

Recent study deals with preparation of Hybrid oxides NiO–ZnO thick film sensors by adding ZnO different weight odd percent proportions mainly 1 wt. %, 3 wt.%, 5 wt.%, 7 wt.% and 9 wt.% in Ni–O which acts as functional material by considering glass substrate using screen printing technique and were characterized by several standard techniques. Structural was done by X-ray diffractometer (XRD), morphological and elemental composition was studied by Field Emission Scanning Electron Microscopy (FESEM) and EDAX technique. The XRD plots matched perfectly with JCPDS card No. 01-075-0197 for NiO and JCPDS card No. 00-036-1451 for ZnO. ZnO has Hexagonal Wurtzite structure while NiO face centred cubic (FCC) crystalline structure were confirmed from XRD analysis. Using morphological properties of NiO–ZnO thick films; concluded that the surface of NiO–ZnO nanoparticles is smooth and uniform. Energy dispersive X-ray analysis (EDAX) spectra show presence of excess oxygen as well as non-stoichiometric nature of Ni, Zn and O. FTIR and UV spectroscopy used to study optical properties of prepared hybrid oxides NiO–ZnO thick films and it shows presence different vibrations and functional groups in the prepared mixed binary oxides also to get the band gap energy of prepared thick films respectively.

Keywords: Hybrid oxides, thick film sensors, additives, spectroscopy

Introduction

Now a days nanomaterials has more popular due to the evolution of new techniques and processes. Using different synthesis techniques nanostructured metal oxides with different geometrical shapes have been produced [1, 2]. Because of their unique structure and various properties of nanomaterial have emerged as a advanced class of material science [3]. Nanocomposites are composites made up of at least two phases, one or both of which are nanoscale. Metal oxide nanoparticles and nanocomposites used in a variety of applications mainly to enhance the electrical, optical, and gas sensing capacity [4].

Nanotechnology is the framework for advancing our understanding of nanoscale materials and phenomena. Sensors are gaining a lot of interest since they can gather chemical information from the environment in real time, and as a result, they will have a growing impact on daily living [5]. Because nanoparticles increased optical emission and absorption due to electron movement from one state to another, nanodevice manufacture has considerably aided nanomaterials and nanotechnology research and development. Optoelectronic nanodevices benefit from this phenomenon [6, 7].

Nickel oxide (NiO) films have a wide forbidden band gap [8]. NiO is a semiconductor from group of transparent conductive oxides (TCO) [9, 10]. NiO is a reversible magnetic compound [11] with a melting point of around 2000 °C, implying that it is durable and chemically stable. Solar cells, gas sensors, anti-reflection layers, optoelectronics, and other applications of NiO are the most popular [10–13].

Zinc oxide (ZnO) is a TCO that belongs to the family of semiconductor chemical compounds [14]. At ambient temperature, has wide band gap and an excitonic bonding energy of 60 MeV, making it ideal for characterising and optoelectronic device applications, gas sensors, optical devices, and other applications [14–16].

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To improve physico-chemical characteristics heterogeneous bandgap semiconductor metal oxide namely zinc oxide (ZnO) is intrinsically n-type and nickel oxide (NiO) is a p-type semiconductor with a large direct bandgap of 3.37 eV and 3.6 eV respectively nano-materials are combined together to create nanocomposite, [17]. Hence it shows NiO-ZnO nanocomposite has got a lot of attention. [18-20].

In the present research work, the authors have emphasized the preparation of Hybrid oxides NiO-ZnO thick film sensors on glass substrate by using screen printing technique. This present work focus on the study of structural, and optical properties of prepared NiO-ZnO thick film sensors.

Materials and Methods

In current research work, commercially available AR grade (99.99 % purity) NiO and ZnO powder was used. For the preparation of mixed oxide binary thick films, NiO used as a

1. X-Ray Diffraction Technique

On a Rigaku diffractometer (DMAX-5000), an X-ray diffractometer with Cu-K α radiation and wavelength (λ)

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where, D= Crystallite size, K= Scherrer constant and its value 0.94, β = Full width of half maxima (FWHM), and λ = Wavelength of X-ray source.

2. FESEM and EDAX

The surface morphology of prepared thick films was characterized using a Field Emission Scanning Electron Microscope (FESEM) [Model JOEL 6300 LA GERMANY. To analysis the elemental composition; Energy dispersive X-ray spectrometer (JOEL-2300, Germany) was used.

2.2 Optical properties

The optical properties of prepared hybrid oxides NiO-ZnO thick films were studied by using Shimadzu IR Affinity-1 $\alpha = A(Eg-h\nu)^n / h\nu$

Where, A is a constant, Eg is the semiconductor band gap and 'n' is a number that decide type of a gap. the value n

Results and Discussion

Structural properties

1. X-Ray Diffraction

Figure 3 shows the XRD pattern of NiO-ZnO thick films. With reference to crystalline structure ZnO has hexagonal Wurtzite structure while NiO has Bunsenite face centred cubic (FCC). As per NiO patterns observation, peaks were found at the diffraction angles: $2\theta = 37.28^\circ$, 43.38° , and 62.94° corresponding to the Miller indices (111), (200), and (220), respectively. It is seen that this sample possess preferred orientation along (200) plane. These lines are in a very good matching with those of JCPDS File no. 01-075-0197. ZnO patterns appeared at $2\theta = 34.48^\circ$, 36.28° , and 62.90° corresponding, respectively, (002), (101), and (103) orientations of hexagonal wurtzite structure with a preferential orientation along (002) peak according to the

functional material and ZnO as an additive. The ZnO was added in different weight percent proportions viz. 1 wt. %, 3 wt.%, 5 wt.%, 7 wt.% and 9 wt.% in the functional material NiO. Films were prepared using a clean glass substrate. To clean glass substrate; distilled water and acetone were used. Inorganic (NiO-ZnO) to organic material ratio 70:30 was maintained and also EC as local binder and butyl carbitol acetate (BCA)) composition was used for film preparation. Using BCA thixotropic paste was prepared in preparation of thick films. Screen printing set up is used for preparation of thick films of mixed binary oxides NiO-ZnO. To remove impurity residue; thick films were retained under IR lamp for 30 minutes and then ready for further work.

1. Structural Features

By using XRD, FESEM, and EDAX to study the crystal structure, morphology, and elemental composition was studied respectively. Weight difference method was used to determine thickness of the prepared films.

1.54 Å.U., XRD patterns of prepared thick films were recorded. Debye Scherrer's formula, Equation 4, was used for calculation of the crystallite size [21].

(4)

and double beam UV-Visible spectrophotometer (Model-JASCO,V-730) for FTIR and UV spectroscopy respectively. Value of energy gap (Eg) is a significant parameter of semiconductors that decides optoelectronic applications; for this Tauc plot method was used. Equation 5 is used to find the optical band gap (Eg) of prepared thick films based on optical absorption spectra.

equal to 1/2 for direct gap while n=2 for indirect gap compound.

standard patterns (JCPDS File no. 00-036-1451) card [28]. As per structural analysis the crystallite size was determined using Debye-Scherrer formulas explained. The crystallite size (D) of NiO-ZnO thick films was found to be 117.1 Å.U., 232.0 Å.U., 93.5 Å.U., 141.8 Å.U., and 139.7 Å.U., for 1 wt. %, 3 wt.%, 5 wt.%, 7 wt.% and 9 wt.% respectively. The XRD results suggest that the hybrid oxides NiO-ZnO nanocomposite is composed of crystalline NiO and ZnO nanostructures. Diverse structural parameters such as crystallite size, FWHM, dislocation density, and micro strain of NiO-ZnO thick films has been calculated. Table 2 illustrated the outcomes of structural Parameters from XRD. From table 2, this is confirmed that the compatibility between the changes in micro strain and the dislocation density according to changes in the additive in wt%.

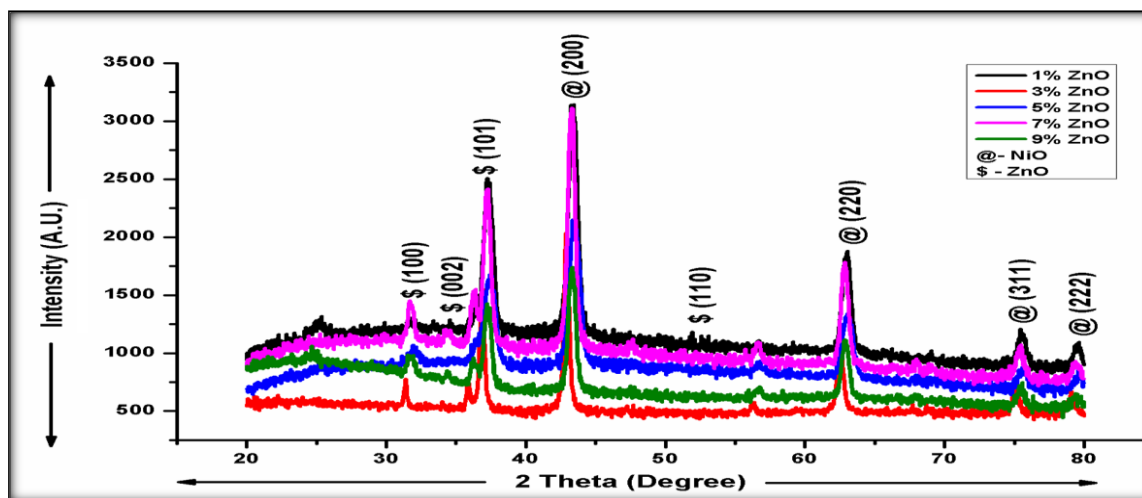


Fig.3 XRD pattern of NiO-ZnO thick films

Table 2 Structural Parameters from XRD of NiO-ZnO thick films

Additive wt%. ZnO	2θ(Degree)	FWHM	Crystallite size (nm)	Dislocation density (nm ⁻²)	Micro strain ε (%)
1%	43.37	0.76278	11.71	7.292×10^{15}	0.0031
3%	42.91	0.38439	23.20	1.857×10^{15}	0.00156
5%	43.45	0.95628	9.35	11.43×10^{15}	0.00388
7%	43.27	0.62963	14.18	4.973×10^{15}	0.00256
9%	43.31	0.63928	13.97	5.123×10^{15}	0.00259

2. Field Emission Scanning Electron Microscopy (FESEM)

The FESEM tool was used to examine the morphology of prepared NiO-ZnO thick films. Figure 4 depicts the surface morphology of films. Figure 4(a-c) depicted NiO-ZnO nanoparticles with a near round form. The round form nanoparticles are scattered casually and incorporated with ZnO, as shown in Figure 5(d, e). The penetration of ZnO into NiO nanoparticle network similar

NiO-ZnO nano heterojunctions applied for UV-photo detectors [29, 30]. The surface of NiO-ZnO nanoparticles is smooth and homogeneous, with a small size distribution, as seen in FESEM images of NiO-ZnO thick films. FESEM micrographs also revealed an extremely porous structure. The growth of a very porous structure may occur as a result of additive effects. Some particles appear to be spherical in shape, and they are strongly agglomerated.

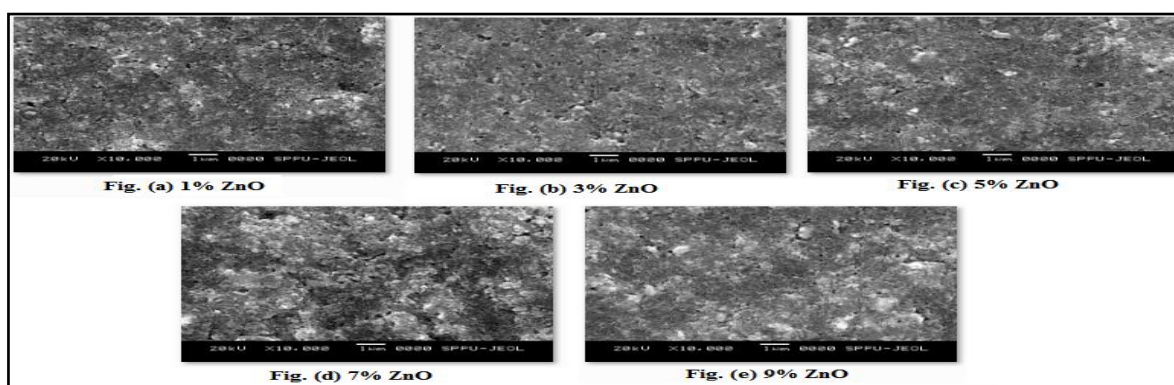


Fig.4 (a-e) FESEM micrographs of NiO-ZnO thick films

3. Energy dispersive analysis

Figure 5 shows the EDAX spectra of NiO-ZnO thick films. From Figure 4, it is clear that the film only contained Zn, Cu, and O according to the EDAX outcomes. No other impurities were found in the films. Energy dispersive analysis (EDAX) spectra shows the film has oxygen excess and also shows that the presence of Ni, Zn and O are non-stoichiometry. It has been observed that the

weight percentage of Zn is increased with increase additive in the NiO. The elemental composition of prepared thick film was described from EDAX; shows presence of nickel, zinc and oxygen with diverse atomic percentages with concentration of additive. From, EDAX plots, the atomic ratio of Ni/Zn in the resultant NiO-ZnO nanoparticles is well consistent with the composition was confirmed.

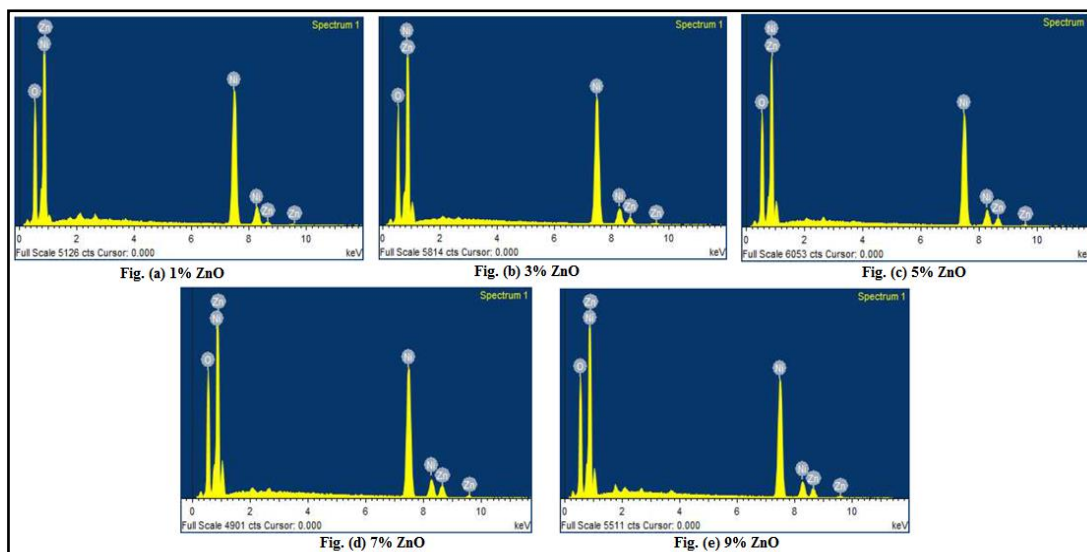


Fig. 5 (a-e) EDAX spectra of NiO-ZnO thick films

The mass percent and atomic weight percent of Zn, Ni, and O are nearly identical. From the EDAX spectra, it is found that atomic weight percentages of Ni, Zn, and O

are nonstoichiometric. The atomic and weight percentages of analysis of film elements are tabulated in Table 3.

Table 3 Elemental composition of NiO-ZnO thick films

Additive wt%.	Element	Weight %	Atomic %
1% ZnO	Ni	58.25	28.42
	Zn	2.34	1.03
	O	39.41	70.56
3% ZnO	Ni	55.51	27.19
	Zn	5.28	2.32
	O	39.21	70.49
5% ZnO	Ni	52.91	25.59
	Zn	6.82	2.96
	O	40.27	71.45
7% ZnO	Ni	52.60	25.89
	Zn	8.43	3.73
	O	38.97	70.38
9% ZnO	Ni	52.43	25.27
	Zn	7.05	3.05
	O	40.52	71.67

Optical properties

1. **Fourier Transform Infrared Spectrometry** The FTIR spectrum of NiO-ZnO nanocomposite was recorded to categorize chemical bonds present in the compound. Regarding metal oxides, the IR absorption goes to under 1000 cm^{-1} .

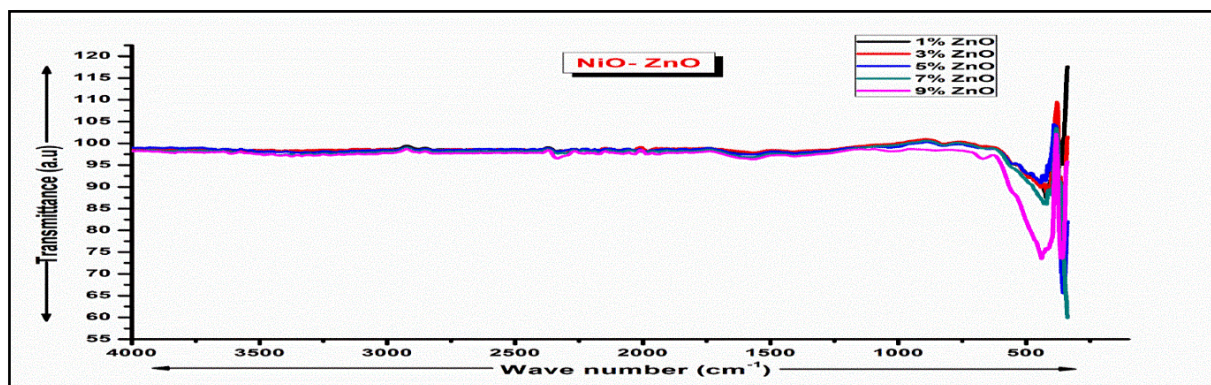


Fig. 6 Transmittance versus wavenumber of NiO-ZnO thick films

Understanding of dominant functional group recorded in the range of $4000\text{--}400\text{ cm}^{-1}$ at room temperature from FTIR. Figure 6 depicts the FTIR spectra of NiO-ZnO thick films. From FTIR spectra; IR absorption bands of the Ni-O bond observed in the range of $470\text{--}426\text{ cm}^{-1}$ are accredited to the Ni-O stretching vibration mode. The IR absorption bands at 498 cm^{-1} , 434 cm^{-1} and 412 cm^{-1} corresponded to ZnO, while the IR absorption bands at 471 cm^{-1} and 434 cm^{-1} resembled NiO-ZnO binary nanocomposite. Thus, their distinct peaks are integrated. These findings up closer the XRD data, gave confirmation presence of ZnO and NiO in the binary composite [29, 31].

2. UV Spectroscopy

The optical properties plays an important role in determining the band gap of the material. UV-Vis absorption analysis explores the optical properties of semiconductor materials. Optical absorption spectrum of heterogeneous NiO-ZnO thick film samples have been recorded using UV-Vis-NIR spectrometer in the wavelength range of $200\text{--}800\text{ nm}$. In the literature, it was reported that ZnO has an absorption band at 3760 A.U. [32], while NiO has sharp absorption peaks at 2080 and 3050 A.U. [33]. Also the discrete absorption peaks of NiO-ZnO nanocomposite at 3030 A.U. and 3710 A.U. were stated [29]. Similar results were shown in Figure 7. Figure 7 shows absorbance versus wavelength of NiO-ZnO thick films.

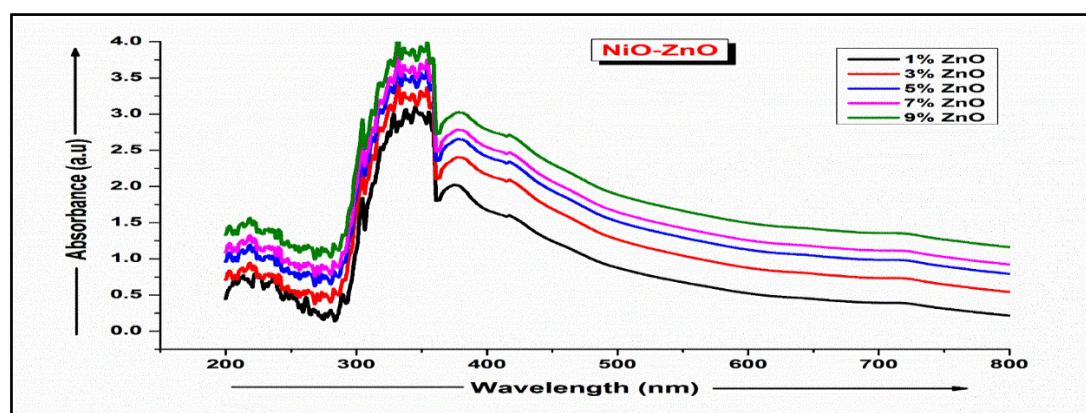


Fig. 7 Absorbance versus wavelength of NiO-ZnO thick films

The investigation of a photocatalytic active material's bandgap is an important tool for estimating its photocatalytic response. Tauc plot decides the bandgap energy of the NiO-ZnO nanocomposite [34]. By adding ZnO to n type semiconductor make p-n heterojunction and a p-n heterojunction minimizes the band gap results by review, which is consistent with the current literature review [29, 34, 35]. The energy band gap (E_g) of NiO-

ZnO thick films at 1 wt. %, 3 wt.%, 5 wt.%, 7 wt.% and 9 wt.% was found to be 4.86, 4.65, 4.24, 3.84, and 3.32 eV respectively. Finally concludes that the energy band gap of heterogeneous oxides NiO-ZnO thick films decreases as ZnO additive increases in wt%. The NiO-ZnO composite has a lower bandgap than the oxides, based on these findings. This result shows the prepared NiO-ZnO films can be used as an preparation of solar cell or LED. [29].

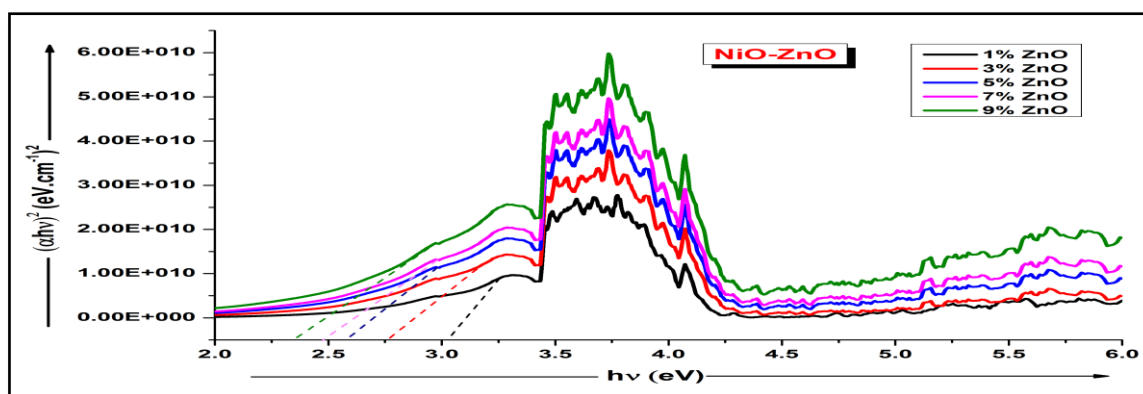


Fig. 8 Tauc plot of NiO-ZnO thick films

Conclusion

Hybrid oxides NiO-ZnO thick films were prepared on glass substrate using standard screen-printing technique. The structural properties of NiO-ZnO thick films was carried out by XRD, FESEM as well as EDAX. The optical

properties of prepared Hybrid oxides NiO-ZnO thick films were studied by FTIR and UV-visible spectroscopy. XRD results confirmed the formation of NiO-ZnO nanocomposites. The XRD plots coincides with JCPDS card No. 01-075-0197 and JCPDS card No. 00-036-1451 for NiO

and ZnO respectively. XRD confirms the ZnO has hexagonal Wurtzite structure and NiO has Bunsenite face centred cubic (FCC) crystalline structure. FESEM micrographs of NiO-ZnO thick films clearly shows presence of NiO-ZnO nanoparticles on surface. Energy dispersive analysis (EDAX) spectra shows the film has excess oxygen and also shows that Ni, Zn and O are present in non-stoichiometry ratio. From FTIR, different vibrations and functional groups in the prepared mixed binary oxides were determined. Using Tauc plot formula, it was found that the band gap decreases as additive wt % increases and hence prepared NiO-ZnO thick film can be used as photovoltaic applications.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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