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Synthesis of Chalcogenide Material (CMnTS) By Hydrothermal Method, Characterization and Its Application

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Abstract

The present research paper concentrate on the synthesis, characterization and applications of the quaternary chalcogenide material Cu2MnSnS4 (CMnTS) using the hydrothermal method. CMnTS has attracted considerable attention in recent years because of its suitable band gap, excellent optical absorption properties, earth-abundant constituent elements, low cost, and non-toxic nature. These characteristics make it an environmentally friendly and sustainable semiconductor material for renewable energy and electrochemical applications. In this study, CMnTS was successfully synthesized using metal chloride precursors and thiourea as the sulfur source in a Teflon-lined stainless-steel autoclave under controlled temperature and pressure conditions. The hydrothermal technique was chosen due to its simplicity, cost-effectiveness, and ability to produce highly crystalline materials with controlled morphology. The structural properties of the synthesized material were examined using X-ray diffraction (XRD), confirming the formation of the desired crystalline phase. The optical properties were investigated using UV–Visible spectroscopy, which revealed a suitable band gap for solar energy harvesting applications. Furthermore, cyclic voltammetry analysis was carried out to evaluate the electrochemical behavior and charge storage capability of CMnTS. The results indicate that CMnTS exhibits strong potential for application in solar cells, photocatalytic removal of heavy metals from wastewater, and as an anode material in high-energy-density batteries. Overall, this study demonstrates that the hydrothermal method is an efficient and reliable approach for synthesizing high-quality CMnTS suitable for advanced energy-related applications.

Keywords: *chalcogenide, cmts, hydrothermal synthesis, characterization techniques, cyclic voltammetry, renewable energy.*

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Introduction

Nanotechnology in recent era has been revolutionary and high impact field in scientific sectors. It is a discipline that deals with material sized between 1-100 nanometers, creation of nanoparticles by manipulating at nanoscales. The nanoparticles show unique mechanical, electrical, chemical, physical properties then their parent material. They are used in medical equipments, coatings, electronics, energy materials etc. We are witnessing the rapid increase of the global energy demand especially after the industrial revolution. In the last decades world oil prices shows extreme volatility and hazardous impacts of the conventional energy sources on the environment draw our attention towards the need of a sustainable clean energy supply. Prominent renewable energy source such as solar energy is a good alternative to conventional energy sources. Chalcogenide material can be used as absorber layer in solar cell which can give us more efficiency in solar energy production. Recently, much attention has been focused on the development of more safe, high-energy density, long life and low-cost batteries to meet our energy demand as our daily life includes electric vehicles, portable electronics etc. However, lithium-ion batteries which we were using from decades have low theoretical specific capacity and much lesser energy density of electrode material. To obtain high theoretical storage capacity, the metal chalcogenides can be most promising anode material for batteries. There are two general approaches to the synthesis of nanomaterials and fabrication of nanostructures, (A) Top-down approach (B) Bottom-up approach. CMnTS is a chalcogenide material which is basically a special family of compounds that include one of the chalogen elements from the group 16 of the periodic table. Characterization of chalcogenide material is done with the help of various characterization techniques to study structural, optical, electrical properties.

The following objectives are determined in the research paper

1. To synthesize CMnTS materials by bottom up approach with the help of hydrothermal synthesis.
2. To determine the crystallite size of CMnTS using XRD analysis.
3. To confirm the crystal geometry based on JCPDS data and lattice parameters.
4. To investigate the optical band gap through UV-Vis spectroscopy.
5. To analyze the electrochemical behavior using cyclic voltammetry, identifying anodic and catholic peak currents and their corresponding potentials.

Literature Review:

Some of the literatures that have been used for the proceeding of this research paper are as follows:

Nanotechnology principles and practices by Sulbha K Kulkarni (2007)[1] discuss the basic of nanotechnology including introduction of nanomaterials, synthesis method, characterization techniques, properties of nanomaterials etc. Walus.E,Manecki.M,Cios.G,Tokarski.T in their research paper published in Materials (2021)2 investigated & studied how the choice of sulfur precursor influence the hydrothermal synthesis of CMnTS. Sulfur precursor like sodium sulphide (Na_2S) & thiourea directly impacts the final nanomaterial structure & phase purity. K.Byrappa & M. Yoshimura in their handbook of Hydrothermal technology (2001)3 had a overview of hydrothermal processes particularly behavior of materials under high pressure & high temperature. This book discuss the design and operation of hydrothermal reactors such as autoclaves, supercritical water reactors etc. Le Donne A, Trifiletti V. and Binetti S. (2019)4 have stated in their research on new earth abundant thin film solar cells based on chalcogenides, developing sustainable and cost effective photovoltaic technologies are crucial because global energy demand is continuously increasing and there is need to reduce carbon emission. PV market is dominated by crystalline silicon but its high energy requirements and material constraints limits its long term use. On the other hand, quaternary chalcogenides like CMnTS is cost effective, earth abundant, have high absorption coefficients, high carrier concentrations can be fit for thin film photovoltaic applications. Noemie Elgrishi, Kelley J. Rountree, Braina D. McCarthy, Eric S. Rountree, Thomas T. Eisenhart, and Jillian L. Dempsey (2017)5 have explained their research in practical beginners guide to cyclic voltammetry. Authors emphasize on cyclic voltammetry (CV), one of the widely use electrochemical techniques,this research paper gives practical introduction on how to obtain and interpret CV data adequately. All the fundamental components including electrolytes, electrodes, solvent system of CV experiment were describe in the research. Practical guide on cyclic voltammetry explains how electrode preparation, scan rate affects the voltammogram. Moses O. Alfred and others (2025)6 in Chalcogenides: recent advanced in their environmental applications highlighted that chalcogenides have photocatalytic applications like useful in water treatment, heavy metal reduction & ions removal.

Methodology:

There are two approaches to perform nanomaterial synthesis i)top-down approach ii)bottom-up approach, broadly categorized into physical, chemical and biological methods. Among the various methods such as solgel method, chemical vapour deposition method, physical vapour deposition method, hydrothermal and combustion method, here we are particularly interested in hydrothermal synthesis.

Hydrothermal Synthesis:

Hydrothermal synthesis is a widely used technique for preparing nanomaterials with controlled size, shape, and crystalline. It involves chemical reactions in an aqueous solution at elevated temperatures (typically 100–250°C) and pressures in a sealed autoclave. Under these conditions, the solubility of reactants increases, promoting the formation and growth of crystalline nanoparticles. The process mimics natural mineral formation and allows precise control over the reaction environment, leading to high-purity and uniform nanostructures.

Fig.1 Autoclave & Teflon

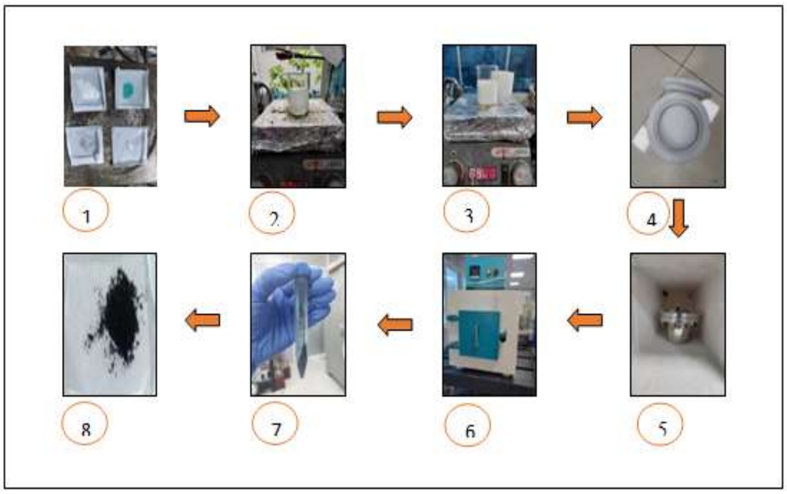
Fig. 2 Muffle Furnace

Process of Synthesis:

CMnTS synthesized by hydrothermal method which is basically a solution reaction-based approach. Chalcogenide material (CMnTS) were synthesized using sulfur precursor (thiourea CH_4N_2S). Source of Tin, Copper and Manganese is $SnCl_2.2H_2o$ (Tin(II) Chloride Dihydrate), $CuCl_2.2H_2O$ (Copper(II) Chloride Dihydrate),$MnCl_2.4H_2$ O(Manganese(II) Chloride Tetra hydrate) respectively. Molar ratio of Cu, Mn, Sn, S taken as 2:1:1:4. The weight for respective element taken by molecular concentration formula i.e.,

W = $\boldsymbol{M*N*Q/1000}$

Where,
M = Molecular weight
N = Molarity
Q = Quantity of Solution
Fig. 3 (1) Precursor used in synthesis (2) precursor solution (3) Homogeneous solution (milky white) (4) solution poured in Teflon (5) Autoclave (6) Muffle furnace (7) Centrifuge (8) dried powder



So, for the Synthesis of CMnTS $CuCl_2.2H_2O$ (0.376gm), $SnCl_2.2H_2O$ (0.315gm), $MnCl_2.4h_2O$(0.277gm), Thiourea
(0.852gm) were used. We took those precursors in one beaker and poured the 80 ml distilled water in it. For purpose of stirring, we place that beaker on heater cum stirrer along with magnetic needle inside the beaker. Solution was stirred until it become homogeneous. Next we took 10µl acetic acid and drop wise it was added in precursor solution with the help of pipette, and stirred it for few minutes. Resulting solution was poured in autoclave and it was placed in muffle furnace. Temperature of muffle furnace was kept at 140˚c and the process of heating carried out for nearly 18 hours. After cooling down the temperature we centrifuge the synthesized sample solution and placed it under IR lamp thereafter we obtained the dry powdered sample.

3. Reaction involved in process:

Metal chlorides, thiourea were dissolved in solvent in the first step of synthesis mechanism of CMnTS.

$CuCl_2 \rightarrow Cu^{2+} + 2Cl^-$

$MnCl_2 \rightarrow Mn^{2+} + 2Cl^-$

$SnCl_2 \rightarrow Sn^{2+} + 2Cl^-$

CMnTS synthesis mechanism has chemical reaction given as follows:

$$\mathbf{2CuCl_2 + MnCl_2 + SnCl_2 + 4CH_4N_2S + 8H_2O \rightarrow Cu_2MnSnS_4 + 8NH_4Cl + 4CO_2}$$

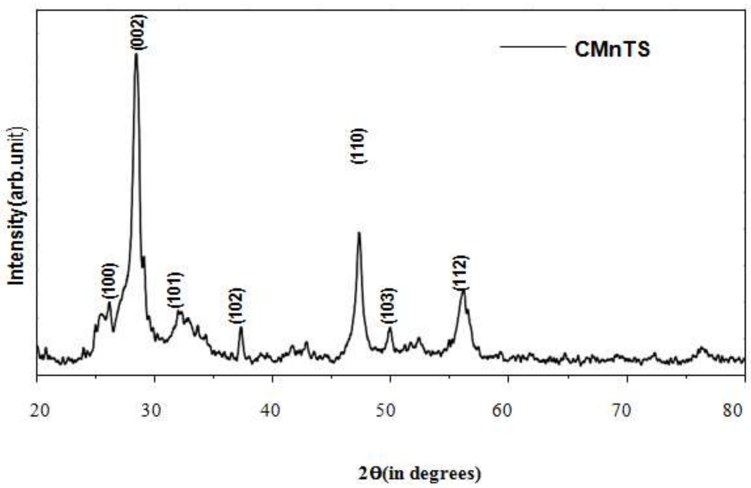
Characterization technique:

The hydrothermally synthesized nanomaterials can be characterized using the range of analytical techniques, like XRD, UV Visible spectroscopy, SEM, FTRI, etc. In this research paper, we have performed and analyzed- XRD and UV visible spectroscopy.

Results and Discussion:

1. XRD Characterization:

The X Ray Diffraction spectrum of synthesized CMnTS nanoparticles shown in figure confirms the hexagonal wurtzite structure according to JCPDF data. Wurtzite structure of CMnTS has unit cell parameters a = b = Å and c = 5.209 Å. The average size of the nanoparticles has been calculated using the Debye- Scherrer formula, where, λ is the x-ray wavelength (1.504nm), θ is the Bragg diffraction angle, and β is the full width at half maxima.



Graph 1: XRD pattern of CMnTS

From the diffracted peaks obtained in the XRD spectrum, the average crystallite size of the synthesized CMnTS is determined with the help of the Scherer formula given below:

$\boldsymbol{D=0.9\lambda\ /\beta cos\theta}$

Where,
λ= Wavelength of X- rays = 1.54178 Å
β = Full Width at Half Maximum (FWHM) (in radian),
θ = Angle of diffraction.
The Average Crystallite size of CMnTS material is = 8.45nm
The formula used to determine inter-planer distance is,

$(\boldsymbol{2dsin\theta=n\lambda})$

Where,
n = Order of diffraction = 1,2,3......;
λ= Wavelength of X- rays = 1.54 Å;
θ =Angle of Diffraction and
d =Inter-Planar Distance,
d = $\boldsymbol{\lambda}\ /(\boldsymbol{n}=1)$
Calculation of miller indices and crystalline size for prominent peaks of XRD is shown by following observation table.

Table 1: XRD findings

2θ	Crystalline size	Crystalline strain percent	Miller indices
28.111	5.011	2.97	(002)
47.182	10.261	0.88	(110)
55.961	10.077	0.76	(112)
	AVG=8.45nm		

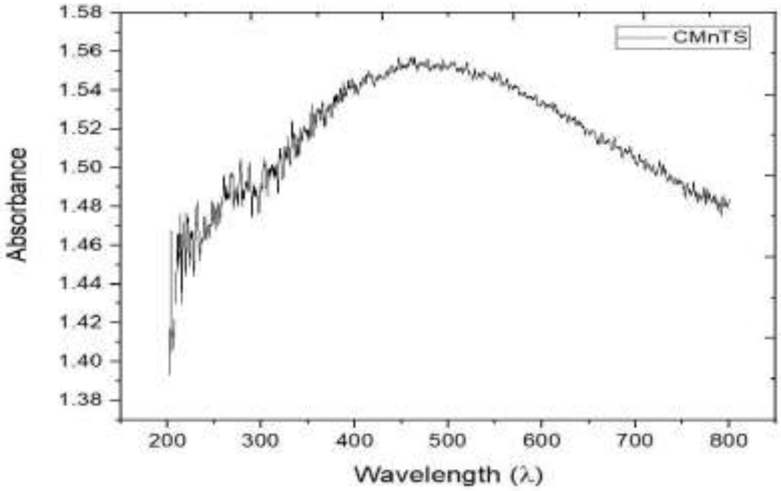
For hexagonal unit cell the inter-planar spacing d can be given as,

$$\frac{1}{d^2}=\frac{4}{3}\frac{h^2+hk+k^2}{a^2}+\frac{l^2}{c^2}$$

Here, h, k, 1 are miller indices. a, c are lattice parameters.

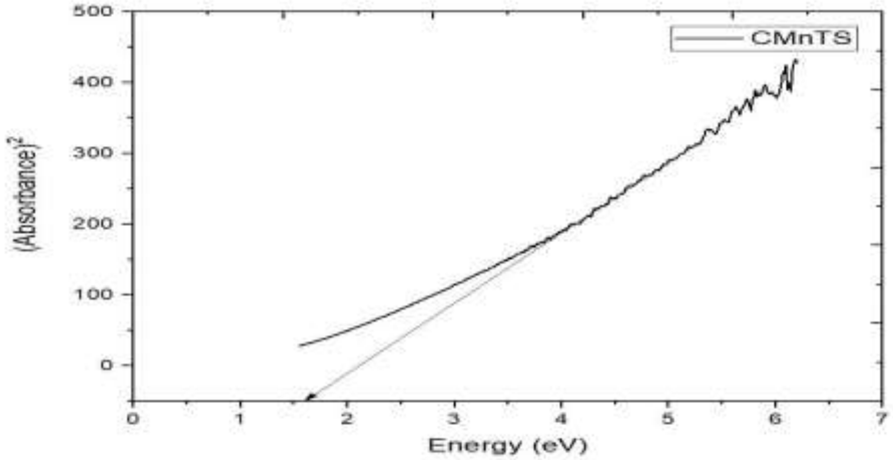
We have found that,
a= 3.24 Å (a = 3.25Å Standard value) c= 5.21Å (c = 5.2Å standard value) From the JCPDS data of Cent's and calculated lattice parameters it is concluded that cant's has hexagonal geometry.

2. Optical Studies:
The optical properties of CMnTS such as energy band gap were studied by using UV -Visible spectroscopy.
The below figure shows the spectra recorded in the wavelength range of 210-795 nm.



Graph 2: UV-vis Spectroscopy spectrum of CMnTS material.

However, no significant features were observed beyond 795 nm wavelength. Therefore, the optical spectra below are plotted for 210nm to 795nm wavelength range.

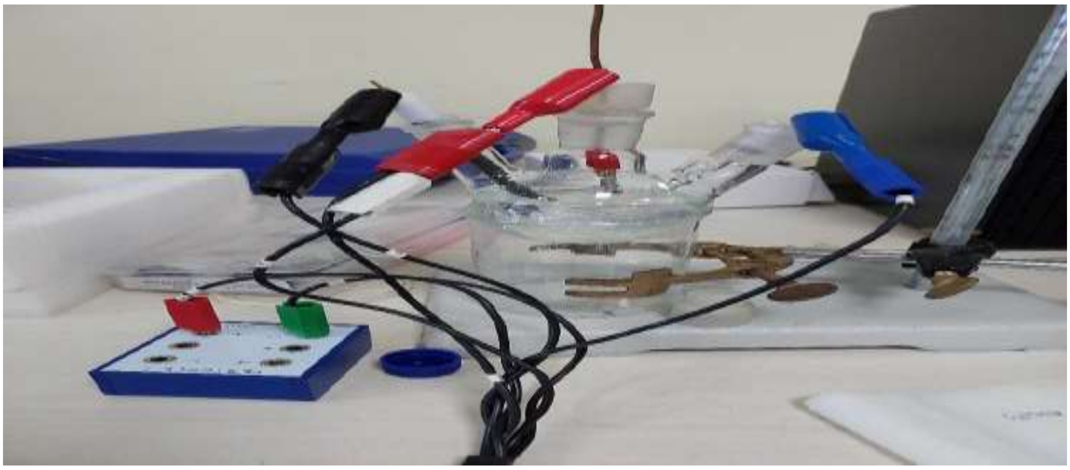


Graph 3: Tauc plot from UV data.

The curve drawn between Energy (hv) and (α)2 is used to determine the band gap of CMnTS nanoparticles, here v is the frequency and α is the optical absorption coefficient. The band gap energy obtained by extrapolating curve is found to be approximately 1.59 eV for CMnTS.

3. Cyclic Voltammetry:

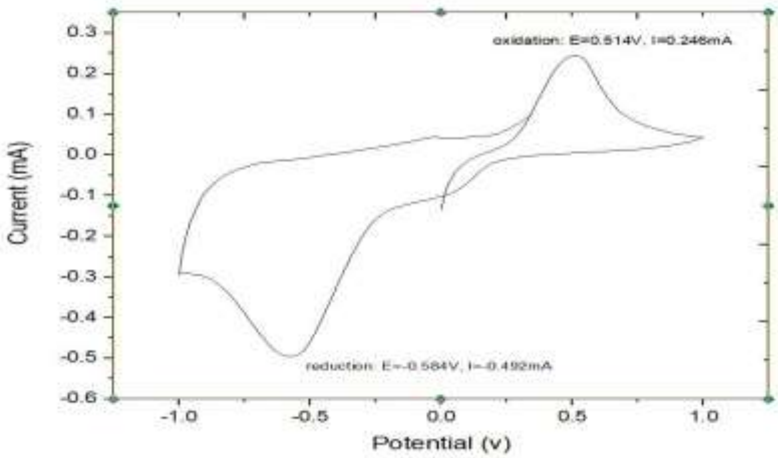
Cyclic Voltammetry is a powerful and popular electrochemical technique that gives information about analyze. CV is also invaluable to study electron transfer initiated chemical reaction and it is performed by cycling the potential of working electrode and the measuring the resulting current. The potentiostat is used to control the working electrode potential. CV setup consists of three electrodes called as working electrode (Silver electrode), Counter electrode (Graphite electrode), Reference electrode (Silver chloride). To perform cyclic voltammetry, the electrolyte solution is first added to an electrochemical cell along with a reference solution and the three electrodes.



Three Electrode Setup

In the CV, current is recorded by sweeping the potential back and forth (from positive to Negative and negative to positive) between the chosen limit. This process is repeated multiple times during a scan and the changing current between the working and counter probes is measured by the device in real time. The result is a characteristic duck-shaped plot known as a cyclic voltammogram. The electrochemical behavior of the material can be studied with the help of information obtained from CV.

The x-axis in the below cyclic voltammogram represent the parameter that impose on the system, here the applied potential (E), while the y-axis is the response, here the resulting current (i) passed. The anodic and cathodic peaks in the voltammogram give us the potential at which the material is oxidized and reduced. For the forward scan positive signal is provided while the voltage is switched after the first half-cycle followed by negative signal which inverts the nature of the voltammogram for the second half-cycle. In cyclic voltammogram shown above, the scan starts at -1.0 V sweep forward to more positive oxidative potentials. Initially the potential is not sufficient to oxidize the analyte. Current reaches to maximum at point called as anodic peak current for oxidation at the respective anodic peak potential. The Process for reduction is exactly opposite to that of oxidation having cathodic peak current at the respective cathodic peak potential. Anodic and cathodic peak currents should be an equal in magnitude but with opposite sign.



Graph 4: Cyclic Voltammogram

4. Summarize Result:

The Average Crystallite size of CMnTS material is 8.45nm. From the JCPDS data of CMnTS and calculated lattice parameters it is concluded that CMnTS has hexagonal geometry.

From the optical studies we got the band gap of CMnTs material i.e., 1.59eV.

We got the anodic peak current at (0.246mA) and cathodic peak current at (-0.492mA) for corresponding anodic voltage (E= 0.514V) and cathodic voltage (E= -0.584V) from Cyclic Voltammogram.

Application of CMnTS Material:

1. **For photo catalytic heavy metal reduction and ions removal:**

Heavy metal pollutants in water are regarded as a serious threat for water security. Therefore, several methods such as adsorption, precipitation, photo catalysis, electrochemical and membrane filtration were assigned for heavy metal removal from water. Among them, photo catalysis is considered to be one of the best methods for the removal of dissolved metal ions in wastewater. High efficiency, low cost and direct use of natural solar energy are preferential benefits for photo catalytic technique. Chalcogenides based nanomaterials are widely used as photo catalysts due to their narrower band gaps that correspond to the visible light absorption. Three types of mechanism can be considered for the photo catalytic removal of metal ions i.e. direct reduction by photo generated electrons, indirect reduction and oxidative removal by holes or radicals.

2. **Photovoltaic (Thin film solar absorbers):**

Recently, much attention has been focused on the developed of more safe, high energy density long life, and low-cost batteries to satisfy our energy demand as our daily life includes the electric vehicles, portable electronics etc. However, Lithium-ion batteries prepared and available in commercial market since the 1990, even though their theoretical specific capacity, energy density of electrode material is relatively low. Various metal sulphides, metal oxides, have been developed with preferable capacities, but their output voltages are not sufficient. Hence, chalcogen materials are used as anode due to their excellent theoretical capacities, low cost and no toxicity. Despite silicon solar cells currently use the PV (photo-voltaic market), the extremely more versatile thin film-based devices made up of chalcogenide material have almost matched them in performance and have room for improvement. The thin films based on earth abundant elements were strongly investigated as PV absorbers; in particular CZTS, CMnTS, CFTS and related sulfur selenium alloy are used. The CMnTS have direct band gap around 1.2 to 1.6 eV and have high absorption coefficient which is suitable to be used for absorber layer in solar cell.

Conclusion:

CMnTS were synthesized by simple hydrothermal method. Further the synthesize CMnTS were characterized by XRD and UV absorption spectroscopy in order to reveal Crystalline size, Crystalline structure, Crystalline strain and band gap respectively. The preferential crystallographic orientation of the CMnTS material synthesized was found to be along (0 0 2), (1 1 0), and (1 1 2) planes. The crystalline parameter found by XRD confirms that the synthesized CMnTS is of hexagonal structure. Optical characterization performed using UV-visible spectroscopy revealed direct band gap of 1.59ev,placing the CMnTS within the optimal energy range for photovoltaic and photocatalytic application CMnTS which is semi conductive nature can be used as anode material for Batteries as it is having High theoretical storage capacity.

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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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