

SYNTHESIS AND THE OPTICAL BEHAVIOUR OF ERBIUM-DOPED BORO-TELLURITE ZINC NIOBIUM BARIUM GLASS

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Received: June 11, 2024; Revised: August 07, 2024; Accepted: February 26, 2025

Abstract

This study investigates the physical, structural, optical, and luminescence properties of erbium-doped boro-tellurite zinc niobium barium (ErBT) glass. The glass was characterized by exploring the impact of erbium doping on its physical, structural, optical, and luminescence behavior. The density, molar volume, and refractive index describe the physical properties, ranging from 3.73 to 4.11 g/cm³, 33.82 to 36.47 cm³/mol, and 1.72 to 1.79, respectively. Structural analysis, utilizing techniques such as X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR), reveals that the glass network is amorphous, and the vibrations of the molecules determine the internal structure of the glass. Optical properties were examined through absorption spectroscopy, providing insights into the energy levels of 4f from the ⁴I_{15/2} ground state to various excited states, such as ⁴F_{5/2}, ⁴F_{7/2}, ²H_{11/2}, ⁴S_{3/2}, ⁴F_{9/2}, ⁴I_{9/2}, ⁴I_{11/2} and ⁴I_{13/2}. These transitions are positioned at wavelengths of 450, 487, 532, 541, 655, 799, 980, and 1535 nm, respectively. Moreover, NIR luminescence measurements were performed to evaluate the luminescence behavior of the erbium-doped glass, which shows state-level energy can be assigned to the ⁴I_{13/2} → ⁴I_{15/2} transition at 1535 nm, emission properties, and potential applications. The results demonstrated the modification of the ErBT glass's optical behavior and the enhancement of its luminescence characteristics through the doping of erbium. These results contribute to the fundamental understanding of erbium-doped glasses and provide guidelines for applications in optoelectronics, lasers, and optical amplifiers. Further exploration in this field has the potential to novel applications and optimize the performance of erbium-doped ErBT glass in various photonic devices.

Keywords: Erbium; Fourier Transform Infrared; Luminescence; X-Ray Diffraction

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DOI: <https://doi.org/10.55766/sujst5266>

Suranaree J. Sci. Technol. 33(2):010418(1-7)

Introduction

In advanced materials science and photonics, the investigation of innovative optical materials with incomparable capabilities will lead to breakthrough knowledge. Among the many materials that academics are interested in and compete with, rare-earth-doped glasses have become an attractive option, presenting an entrance to a new area of optical exploration. The creation and thorough examination of distinctive glass compositions are key to harnessing the latent potential of light in various technological applications.

This study examines the properties of erbium-doped boro-tellurite zinc niobium barium glass as a potential optical material. This composite combines the individual advantages of each element. Boro-tellurite, distinguished by its unique combination of boron and tellurium, introduces optical properties into the structure of the glass. Boro-tellurite is well known for its low phonon energy and high thermal stability, positioning it as a well-suited candidate for applications with elevated optical transparency and efficiency levels (Rammah, 2019). As a fundamental component within the glass matrix, zinc confers structural stability and durability upon the material (Mondal *et al.*, 2020). Furthermore, incorporating niobium, chosen due to its vacant d-orbital, is known for strengthening nonlinear optical sensitivity (Iordanova *et al.*, 2020). Lastly, the pivotal role played by barium is its high resistance to extreme radioactive radiation, increased density, and enhanced optical transparency (Kalenda *et al.*, 2020). The interest in rare-earth ions, especially erbium (Er), stems from their exceptional ability to make light stronger and controllable. Glasses containing erbium have left lasting impacts in the scientific and technological world because erbium produces specific kinds of light in the near-infrared range. These special glasses have transformed how data is sent, improved medical tools, and sparked new ideas for enhancing laser performance.

The methodologies employed in the synthesis of the glass will be elucidated. Subsequently, a detailed exploration of the related to the doping of Erbium ions will ensue. Following this, a comprehensive analysis of the optical behavior exhibited by the absorption spectra. This research aims to leverage the advantageous attributes of boro-tellurite, zinc, niobium, and barium. Its main goal is not solely to enhance the scientific comprehension of rare-earth-doped glass systems but also to stimulate a new era of innovations in the field of optical materials and technologies domain.

In summary, this study explores a comprehensive exploration of a highly promising

optical material, the erbium-doped boro-tellurite zinc niobium barium glass. By utilizing the unique properties of its constituent elements, this research aims to unlock the latent potential of rare-earth-doped glasses, particularly erbium, for the future of optical materials and technology. This investigation not only seeks to enhance our scientific understanding of rare-earth-doped glass systems but also endeavors to catalyze a new area of innovation in various applications, particularly in laser technology.

Experiment

The glass samples utilized in this study were prepared through the melt quenching technique, with various compositions represented by the formula: $(30-x)\text{TeO}_2-30\text{B}_2\text{O}_3-10\text{ZnO}-20\text{BaO}-10\text{Nb}_2\text{O}_5-x\text{Er}_2\text{O}_3$, where x ranges from 0.00 to 2.50 mol%. The raw materials, including TeO_2 , B_2O_3 , ZnO , BaO , Nb_2O_5 , and Er_2O_3 , were carefully weighed and mixed in six separate batches of 10 g each using a porcelain crucible. The substances were then stirred in the crucible to achieve a perfect mixture. Subsequently, the resulting powder was placed in crucibles and subjected to melting at 1150°C for 1.5 hours using an electric furnace. Afterward, the molten samples were then poured into preheated graphite molds and annealed for 3 hours at 350°C . Each sample was precisely cut into dimensions of $1.0 \times 1.5 \times 0.3 \text{ cm}^3$. Various properties of these glass samples were investigated. The density (ρ) was determined by weighing the samples using a densitometer (model AND HR-200). The refractive index (n) was measured with a Presidium Refractive Index Meter II. The ground glass samples were subjected to X-ray diffraction (XRD) analysis using a Rigaku SmartLab X-ray Diffractometer. The UV-3600 Shimadzu UV-VIS-NIR spectrophotometer was employed to record the UV-Vis-NIR absorption spectra of the samples. Additionally, measurements of excitation, and emission spectra, were obtained by using laser luminescence with an InGaAs source as the excitation source.

Results and Discussion

1. Physical properties

Density is a measure of how compact a material or substance is. In this study, density is measured with an accuracy of $\pm 0.0001 \text{ g/cm}^3$.

Figure 1 presents the values of density, molar volume, and refractive index for various concentrations of erbium oxide. As the erbium oxide content increases, both the density and refractive index of ErBT glasses increase. This increase is attributed to a higher number of bridging oxygens which is a result of doping of erbium instead of tellurite with lower molecular mass and atomic radius (Muhammad Noorazlan *et al.*, 2013). On the other hand, the molar volume does not exhibit a clear trend, as the rate of change in molar mass (M) exceeds that of density (ρ) (Muhammad Noorazlan *et al.*, 2013).

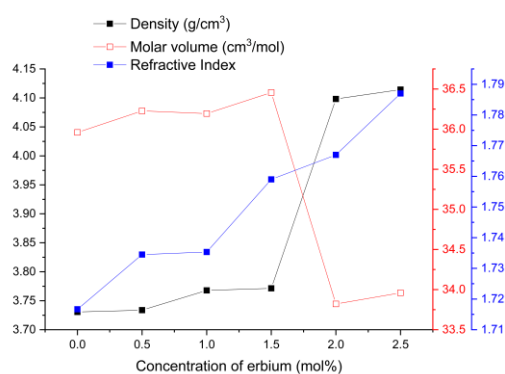


Figure 1. The physical properties of ErBT glasses with various concentrations of Er_2O_3

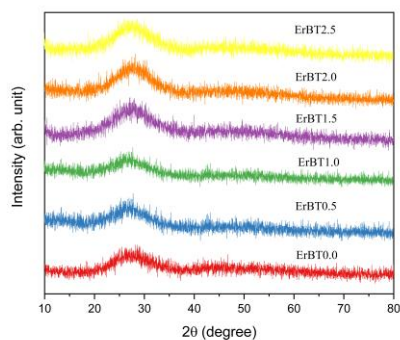


Figure 2. XRD pattern of ErBT glass

2. Structural properties

2.1 X-ray Diffraction (XRD) Pattern: To ascertain the structural characteristics of the materials, X-ray diffraction (XRD) measurements were carried out utilizing Cu $K\alpha$ radiation (with parameters set at 40 kV and 15 mA) along with a graphite monochromator. The scanning range covered 2θ (θ representing the Bragg angle) from 10° to 80° . The XRD pattern of the glass samples prepared is shown in Figure 2. The XRD data confirmed that these glasses exhibited a non-crystalline or amorphous nature, as the spectra displayed broad humps without any distinct sharp peaks. (Hou *et al.*, 2021).

2.2 Fourier transform Infrared (FTIR): The FTIR spectra of the glass samples in this study, as shown in Figure 3 and detailed in Table 1, display prominent transmission peaks at specific wavenumbers, notably at 669, 833, and 1333, and in the range of 2000-2500 cm^{-1} . These observed transmission peaks are summarized in Table 1. The transmission peak at 669 cm^{-1} is a distinctive characteristic associated with pure TeO_2 glass (Madhu & Srinatha, 2020). The structural pattern of tellurium-based glasses is determined by the trigonal pyramid TeO_3 and bipyramid TeO_4 configurations. The transmission in the 600-700 cm^{-1} range in such glasses is due to the stretching vibrations of the Te-O bonds within the TeO_3 and

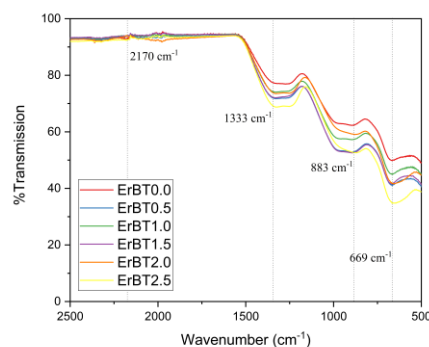


Figure 3. FTIR transmittance spectra of ErBT

Table 1. Assignments of FTIR

Band position	Assignments	Reference
669 cm^{-1}	The stretching vibration mode of TeO_4 units is interconnected by bridging oxygen.	(Madhu & Srinatha, 2020)
833 cm^{-1}	Stretching vibrations in BO_4 units.	
1333 cm^{-1}	Relaxation of the asymmetric stretching of the B-O bond within BO_3 groups.	
2000-2500 cm^{-1}	OH group.	

TeO₄ groups. Notably, the TeO₃ group exhibits a higher frequency position compared to the TeO₄ group, and typically, the TeO₃ group's absorption band falls within the 650-700 cm⁻¹ frequency range. The shifts in these bands are influenced by variations in the glass network composition. In pure B₂O₃ glass, the 883 cm⁻¹ frequency signifies the presence of the boroxol ring (Madhu & Srinatha, 2020). However, in the glass system under investigation, the absence of the 883 cm⁻¹ peak suggests the absence of boroxol rings in the glass structure. Instead, it mainly consists of BO₃ and BO₄ groups, which may be randomly connected in the network. This observation is consistent with the gradual replacement of the boroxol ring with BO₃ and BO₄ groups within the glass structure. The peaks located at 1333 cm⁻¹ in the spectra are attributed to [BO₄] groups (Madhu & Srinatha, 2020). The bands in the 2000-2500 cm⁻¹ range correspond to the stretching vibrations of hydrogen groups involved in hydrogen bonds. This may be caused by the inclusion of H₃BO₃ (used as a substitute for B₂O₃) in the glass composition.

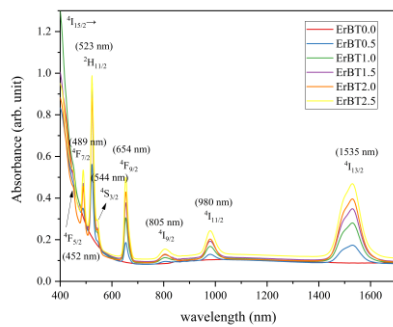


Figure 4. The absorbance spectra of ErBT glasses with various concentrations of Er₂O₃

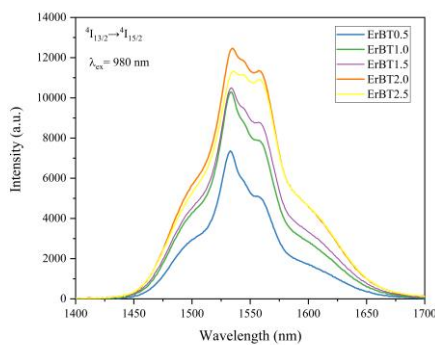


Figure 5. The laser NIR emission spectra of ErBT glasses with various concentrations of Er₂O₃

3. Optical properties

3.1 Absorbance spectra: UV-visible and near-infrared absorbance spectra were obtained for both the undoped and doped with Er³⁺ ions. By analyzing the absorbance data, determine the transition energies of Er³⁺ ions in the glasses. The absorption spectrum covers the entire internal absorption transition of the Er³⁺ ion, which spans from 400 to 1700 nm. The assignment of these absorbance bands is presented in Figure 4, showcasing representative spectra across a range of erbium concentrations, from 0.00 to 2.50 mol%. All the observed absorbance bands can be attributed to the 4f-4f transitions of Er³⁺ ions originating from the ⁴I_{15/2} ground state and leading to various excited states. Specifically, eight distinct bands are corresponding to transitions from the ground state of Er³⁺ (⁴I_{15/2}) to different excited states, namely ⁴I_{13/2}, ⁴I_{11/2}, ⁴I_{9/2}, ⁴F_{9/2}, ⁴S_{3/2}, ²H_{11/2}, ⁴F_{7/2}, and ⁴F_{5/2} transitions. These transitions are associated with specific wavelengths 452, 489, 523, 544, 654, 805, 980, and 1535 nm, respectively (Ding *et al.*, 2023). Notably, it is evident that as the concentration of erbium increases, each absorbance peak exhibits a proportional increase, consistent with the principles of the Beer-Lambert law. Additionally, the absorbance spectra were analyzed to calculate the Judd Ofelt parameter (JO), which is a crucial factor in determining the suitability of the material for laser applications.

4. Luminescence properties

4.1 NIR Emission spectra at 1.53 nm:

Figure 5, displays the near-infrared (NIR) emission spectra of Er³⁺-doped ErBT glasses. The measured NIR emission spectra shows a broad emission peak centered at 1535 nm, corresponding to the ⁴I_{13/2} → ⁴I_{15/2} transition, with 980 nm serving as the excitation source (Agawane *et al.*, 2019). The NIR emission spectrum clearly shows that the emission intensity gradually rises to a critical concentration of 2.00 mol%, but it begins to decrease beyond this concentration. This decrease indicates the occurrence of the concentration quenching effect. This effect can be attributed to the increased interactions between Er³⁺ ions at higher concentrations, resulting in a reduced distance between them. These close interactions lead to non-radiative processes, the formation of ion pairs, and the clustering of Er³⁺ ions. Therefore, the gradual increase in Er³⁺ ion concentration leads to a weakening of the emission intensity. Therefore, 2.00 mol% is identified as the optimal concentration for Er³⁺ ions in these glasses. This observation provides evidence that concentration quenching and the subsequent reduction in emission intensity are associated with non-radiative energy transfer processes.

4.2 Lifetime: Figure 6, presents the decay curve profiles of the $^4I_{13/2}$ level in Er^{3+} -doped boro-tellurite zinc barium niobium glasses as a function of the Er^{3+} ions concentration. These decay curves were fitted by a single exponential function, as described in the literature (Serqueira *et al.*, 2013), across all Er^{3+} ion concentrations. The lifetime of the $^4I_{13/2}$ level was determined to be 0.644, 0.575, 0.562, 0.553, and 0.499 milliseconds for the ErBT0.50, ErBT1.00, ErBT1.50, ErBT2.00, and ErBT2.50 glasses, respectively. The observed decrease in the lifetimes of the $^4I_{13/2}$ level with an increase in Er^{3+} ions concentration can be attributed to the heightened occurrence of non-radiative processes, such as energy transfer among Er^{3+} ions. This phenomenon ultimately results in luminescence quenching (Kummara *et al.*, 2020). When specifically examining the lifetime of erbium at a concentration of 2.00 mol%, which serves as the quenching point, it is noteworthy that the experimentally determined lifetime closely aligns with the value calculated from the Judd Ofelt parameters, as presented in Table 3.

4.3 Judd Ofelt (JO) parameter: The optical absorption spectra of glasses doped with rare earth ions play a crucial role in unraveling the radiative characteristics of emission transitions. The distinct absorption lines stemming from the 4f-4f electronic transitions can exhibit either electric or magnetic dipole characteristics. Qualitative assessments of the intensities of these transitions have been independently developed by Judd and Ofelt. Over time, the Judd-Ofelt theory has proven to be a valuable tool for analyzing the intensity of trivalent lanthanide ions. Early pioneering work in this field, addressing the intensities of f-f transitions within the lanthanide series and systematic intensity parametrization, was conducted by Carnall and colleagues (Carnall *et al.*, 1968). In Table 2, we present the experimental (f_{exp}) and calculated (f_{cal}) oscillator strengths. These oscillator strength data offer insights into the symmetry and bonding nature of rare earth ions incorporated within the glass matrix. The presence of the calculated root-mean-square (rms) value demonstrates a strong fit and accurate correspondence between the measured and calculated oscillator strengths.

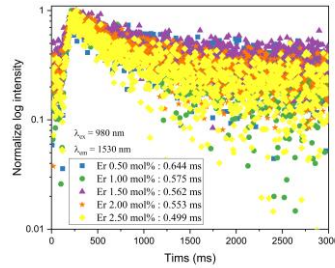


Figure 6. Luminescence decay curves of the ErBT glasses

Table 2. The experimental (f_{exp}) and calculated (f_{cal}) oscillator strength of the ErBT2.0 glass

Transition $^4I_{15/2} \rightarrow$	$f_{exp} (\times 10^{-6})$	$f_{cal} (\times 10^{-6})$
$^4I_{13/2}$	13.5900	13.4064
$^4I_{11/2}$	5.2370	6.0896
$^4I_{9/2}$	2.6610	2.3731
$^4F_{9/2}$	17.5600	17.6946
$^4S_{3/2}$	5.5990	5.3283
$^4H_{11/2}$	48.2600	48.2115
$^4F_{7/2}$	19.3800	19.8850
$^4F_{5/2}$	7.3280	6.4736
rms = 0.490		

Table 3. J-O parameters (Ω_i) and quantum efficiency for ErBT2.0 glass and compared with value reported in other works

Glass code	JO parameter			Trends
	$\Omega_2 (\times 10^{-20})$	$\Omega_4 (\times 10^{-20})$	$\Omega_6 (\times 10^{-20})$	
ErBT2.0 (present)	24.88	9.21	11.76	$\Omega_2 > \Omega_6 > \Omega_4$
TLEA4 (Sazali <i>et al.</i> , 2014)	4.97	1.69	2.27	$\Omega_2 > \Omega_6 > \Omega_4$
SN0.3WE (Yusof <i>et al.</i> , 2017)	6.16	0.88	1.42	$\Omega_2 > \Omega_6 > \Omega_4$
Bi (Babu <i>et al.</i> , 2015)	11.21	1.63	3.12	$\Omega_2 > \Omega_6 > \Omega_4$
Na (Babu <i>et al.</i> , 2015)	20.45	3.15	4.27	$\Omega_2 > \Omega_6 > \Omega_4$

Table 3 provides a list of the JO intensity parameters (Ω) for the ErBT glass sample, along with data from previous studies for compare. The Ω_2 value, associated with the concentration of erbium, is measured at 24.88×10^{-20} , while Ω_4 stands at 9.21×10^{-20} , and Ω_6 is calculated as 11.76×10^{-20} . Notably, in this glass system, the trend for the Ω parameters follows the sequence $\Omega_2 > \Omega_4 > \Omega_6$. The Ω_2 parameter pertains to the asymmetry around the site of the rare earth (RE) ions, while Ω_4 and Ω_6 are indicative of bulk properties such as the rigidity and viscosity of the host matrices (Moustafa *et al.*, 2017). Furthermore, Ω_2 's distinct characteristic is its sensitivity to the centrosymmetric nature of the coordinate structure, the type of chemical bonding (covalent or ionic), the polarizability of ligand ions, and its susceptibility to changes in the local environment of the RE ions. On the other hand, Ω_6 is primarily associated with the rigidity of the glass host. The higher Ω_2 value compared to the previous one suggests increased covalency (less ionic character) and greater asymmetry around the Er^{3+} ions. In a broader context, the variation in JO intensity parameters (Ω) in the present glasses reflects site-to-site variations leading to changes in symmetry around the Er^{3+} ions.

Table 4. shows the radiative parameter including emission wavelength (λ_p) (nm), effective bandwidths ($\Delta\lambda_p$) (nm), transition probabilities (A_R) (s^{-1}), stimulated emission cross-sections (σ) (multiplied by 10^{-20}) (cm^2), radiative lifetimes (τ_R) (ms), branching ratio (β_R), and quantum efficiency (η). These parameters were calculated following this literature (Yusof *et al.*, 2017). All the results demonstrate strong potential for laser applications as each parameter value compares favorably with those from other studies. The parameter $\Delta\lambda_p$ indicates the emissivity peak width, where a larger value signifies a broader peak. However, Table 4 shows that it has the lowest value, which is advantageous for lasers because narrow peaks

indicate high energy-density light. Conversely, other parameters such as A_R , representing the probability per unit time that an atom or molecule in an excited state will spontaneously decay to a lower energy state while emitting a photon, require high values. High transition probabilities indicate a greater likelihood of spontaneous emission, which is crucial for determining the radiative properties of the material. The stimulated emission cross-section (σ) quantifies the probability of stimulated emission occurring when an excited atom or molecule interacts with an incident photon. A larger stimulated emission cross-section indicates a higher probability of a stimulated emission, which is important for the efficiency and gain of laser materials. Quantum efficiency (η) is the ratio of the number of photons emitted to the number of photons absorbed by a material. High quantum efficiency means that a large proportion of absorbed photons result in emitted photons, enhancing the material's efficiency for laser applications. In most cases, all the above values are considered superior when compared to other works. Therefore, it can be concluded that this type of ErBT2.0 glass has a strong potential to be an excellent laser medium.

Conclusions

The ErBT glasses with various concentrations of Er^{3+} ions have been synthesized, and their physical, structural, optical, and near-infrared (NIR) luminescence properties were assessed for potential laser applications. Judd-Ofelt (JO) intensity parameters and radiative properties were analyzed, revealing that the Ω_2 parameter has a relatively high value. This suggests a greater covalence and/or higher asymmetry in the ErBT2.0 glass compared to other compositions. Additional parameters, such as effective bandwidths, transition probabilities, stimulated emission cross-sections, and quantum

Table 4. The characteristics of emission, including emission wavelength (λ_P) (nm), effective bandwidths ($\Delta\lambda_P$) (nm), transition probabilities (A_R) (s^{-1}), stimulated emission cross-sections (σ) (multiplied by 10^{-20}) (cm^2), radiative lifetimes (τ_R) (ms), branching ratio (β_R), and quantum efficiency (η) have been systematically investigated for the emission transitions within ErBT2.0 glass

Glass code	λ_p	$\Delta\lambda_p$	A_R	σ	τ_R	β_R		η
						cal	exp	
ErBT2.0 (present)	1530	99	1453	3.42	0.688	1	1	85%
Er/Yb(0.5/0) (Zhang <i>et al.</i> , 2018)	1560	106	152	0.51	4.62	1	1	-
LiBEr5 (Rajagukguk <i>et al.</i> , 2019)	1532	100	133	3.96	7.10	1	1	-
2Er BiPbTB (Arunkumar & Marimuthu, 2015)	1532	102	682	0.84	1.472	1	1	65
10BiBNCE (Mariyappan <i>et al.</i> , 2020)	1532	112	237	0.53	4.30	1	1	-

efficiency, also indicate the superior performance of ErBT2.0 glass. Emission at 1535 nm was observed, attributed to the $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition of Er^{3+} ions under 980 nm diode laser excitation. Analysis of decay curves revealed that the lifetime of the $^4I_{13/2}$ level of Er^{3+} ions decreases with increasing Er^{3+} ion concentration. The findings not only expand the understanding of rare-earth-doped glasses but also establish a benchmark in the development of high-performance optical materials. In comparison to prior studies, the ErBT2.0 glass exhibits superior radiative properties and optimal performance characteristics, validating its potential for groundbreaking applications in photonics.

Acknowledgment

The authors express their sincere gratitude to the Center of Excellence in Glass Technology and Materials Science (CEGM) for its invaluable contribution of expertise and state-of-the-art equipment, which played a pivotal role in facilitating and advancing the research conducted in this study. N. Sangwanate would like to thank Suan Sunandha Rajabhat University for supporting this research.

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