**Effect of gamma rays irradiation in the structure, optical, and electrical properties of samarium doped bismuth titanate ceramics**

Abstract: Ceramics have enormous potential in several emerging technologies, including nuclear reactors. Materials with chemical inertness, high-temperature operation, and physical properties stability under applied radiation with high energy are all desired in this field of technology. Given these broad specifications, bismuth titanate ceramics may prove to be a valuable material. Regarding this task, the effect of gamma rays on the structural, optical, and ferroelectric properties of samarium-modified bismuth titanate ceramics was investigated. The Bi$_{3.15}$Sm$_{0.85}$Ti$_3$O$_{12}$ (BSmT) compound was irradiated for 0, 50, 100, and 200 kGy using a $^{60}$Co gamma source at a dose rate of 10 kGy h$^{-1}$. The phase structure confirmed the orthorhombic, single-phase nature even after gamma irradiation. The results show that the unit cell volume decreases from 966.39 to 962.38 Å$^3$ with an increase in gamma dose from 0 to 200 kGy. The X-ray photoelectron spectroscopy study shows an irradiation-induced defect in the host matrix. The results show that the bandgap energy, dielectric constant, Curie temperatures, and remnant polarization slightly decreased with an increase in gamma irradiation. According to the findings of this study, the BSmT exhibits adequate stability against gamma irradiation, which offers tremendous promise in their utilization in nuclear reactor technology.

**1 Introduction**

As the aerospace and nuclear sectors expand, there is a growing need for innovative memory devices and actuator materials that can withstand harsh conditions like high temperatures and radiation. The perovskite materials based on lead titanate (PZT) are utilized for these objectives due to their high remnant polarization, Curie temperature ($T_C$), piezoelectric constant, and ability to tune their physical properties by doping. Unfortunately, some drawbacks can happen to these materials, such as their environmental toxicity, limiting their use in this objective [1–6]. Another piezoelectric can be used in place of PZT when operating at
extremely high temperatures or after a large, accumulated radiation dose, despite being more expensive and generally less efficient (lower $d_{33}$) than PZT [6]. Aurivillius family materials with bismuth layer-structured ferroelectric ceramics have been considered a promising candidate for the potential piezoelectric ceramic for high-temperature applications due to their remarkable characteristics, including a high Curie temperature ($T_c$), high resistivity, low aging rate, good temperature stability, and the absence of the toxic element lead [7–14].

The BiT compound is one of the Aurivillius family materials that can be described by the general formula of $(\text{Bi}_2\text{O}_3)^{2+} (\text{A}_{m-1}\text{B}_m\text{O}_{3m+1})^{x-}$, which consists of fluorite-like $(\text{Bi}_2\text{O}_3)^{2+}$ layers and perovskite-like $(\text{A}_{m-1}\text{B}_m\text{O}_{3m+1})^{x-}$ blocks stacked alternately along the $c$-axis [15–17]. Perovskite-like $(\text{A}_{m-1}\text{B}_m\text{O}_{3m+1})^{x-}$ block has excellent possibilities in component flexibility, which allows monovalent, divalent, trivalent ions or their mixtures at A-site with dodecahedral coordination, such as Na$^+$, Ca$^{2+}$, Sm$^{3+}$, La$^{3+}$, Nd$^{3+}$, etc., and transition metal cations at B-site with octahedral coordination, such as Fe$^{3+}$, Co$^{3+}$, Ti$^{4+}$, Nb$^{5+}$, W$^{6+}$, etc. The $m$ varies from 1 to 6, representing the number of $[\text{BO}_6]$ octahedral layers in the perovskite-like blocks [15–17]. Exposure of bismuth layer-structured ferroelectrics to a radiation environment is predicted to cause defects such as ionizing and displacement effects. Trapped charges alter the electrical characteristics of the bismuth layer-structured ferroelectric by producing a local electric field, and irradiation may create multiple electron–hole pairs on the host lattice due to ionizing effects.

When a lot of energy is used to expose something, the atoms in the lattice structure shift about from where they normally would be, displaced atoms may leave vacancies and contribute to interstitial defects by upsetting the positions of surrounding atoms, therefore facilitating the propagation of the impurity [18–21], quick, heavy, and rapid ion irradiation has been established as a valuable strategy for changing the microstructures of ceramics [22–24]. When a material is subjected to energetic ions, it produces a broad range of defect states. They affect the material's structural, optical, and electrical characteristics [25–27]. These variations are highly influenced by the incoming ion's mass, the energy of the irradiation, and the radiation fluence [28]. The characteristics of structurally dependent materials may be changed in the presence of gamma radiation, affecting their performance. These modifications are strongly influenced by radiation factors such as linear energy transfer, dosage, and energy, as well as material characteristics and structure [29]. Since bismuth is one of the more volatile elements during the sintering process, rare-earth elements such as Sm$^{3+}$ ions were chosen to combine with Bi$^{3+}$ ions to reduce the metal's volatility and increase sinterability [30]. There have been relatively few investigations on the impact of gamma radiation on modified bismuth titanate ceramics [31], and a comprehensive literature search revealed that no studies on gamma-ray irradiated Bi$_{3.15}$Sm$_{0.85}$Ti$_3$O$_{12}$ ($\text{BSmT}$) ceramics had been published. Systematic research is required to comprehend the influence of irradiation on the structural, optical, dielectric, and ferroelectric properties of BSmT ceramics. Various gamma dosages barely influenced the properties of BSMT ceramics and showed good stability against gamma irradiation, which holds great potential for their use in nuclear reactor technology.

### 2 Experimental details

The detailed preparation methods of Bi$_{3.15}$Sm$_{0.85}$Ti$_3$O$_{12}$ abbreviated as ($\text{BSmT}$) ceramics, are as follows. The polycrystalline material was synthesized using the traditional solid-state reaction technique, using Bi$_2$O$_3$ (Sigma Aldrich 99.9%), Sm$_2$O$_3$ (Sigma Aldrich 99.9%), and TiO$_2$ (Sigma Aldrich 99.9%) powders as the starting ingredients. After determining the appropriate quantities of starting components based on their respective chemical formula, a wet milling process was conducted for 24 h using acetone and zirconia balls. The slurry that underwent milling was subjected to drying in an electric oven operating at 100°C. After drying, the slurry was further subjected to calcination at 850°C for 6 h. The powders that had undergone calcination were subjected to an additional 24 h re-ball milling process using high-energy ball milling. The milled powders were combined with 1 wt% polyvinyl alcohol aqueous solution, serving as a binder for the pressing process. Subsequently, the powders that had undergone the sieving process were subjected to uniaxial pressing to fabricate disc-shaped pellets measuring 10 mm in diameter. The pellets were subjected to a sintering process at a temperature of 1,150°C for 3 h. The sintered ceramic samples underwent irradiation with gamma rays emitted by a $^{60}$Co source. The irradiation was conducted at a dosage rate of 10 kGy/h, and the absorbed doses applied to the materials were 50, 100, and 200 kGy. The irradiation process was carried out using the Nordion GC-220 model. The sample’s phase structure was investigated using X-ray diffraction (XRD) analysis using an X-ray diffractometer (PAN analytical X’pert Pro) before and after irradiation. The surface images were performed using field emission scanning electron microscopy (JSM-7600F). A UV-2600 (Shimadzu UV-spectroscopy) was used to test the optical properties in the 200–900 nm wavelength range. Defects were confirmed using X-ray photoelectron spectroscopy (XPS; JPS-9200). An Agilent E4980A precision LCR meter was used to evaluate the painted samples’ dielectric characteristics as a temperature
function. Radiant Precision Premier II Analyzer was used for ferroelectric measurements at a 10 Hz frequency at room temperature.

3 Results and discussion

3.1 XRD analysis

The XRD patterns of BSmT ceramics recorded at room temperature before and after gamma irradiation are shown in Figure 1. The measured patterns demonstrated that all samples, before and after irradiation, exhibited a single orthorhombic phase that matched well with the standard crystal data corresponding to BIT Card no. 73-2181 in orthorhombic crystal symmetry and space group Fmmm [32,33]. There was no indication of secondary phases in examining the acquired data for irradiated ceramics, showing that neither gamma irradiation nor defects generated by significant structural disordering resulted in secondary phases. The XRD patterns indicated that the primary change was connected with peak broadening, lattice strain, and atomic positions due to an external effect such as lattice distortion induced by gamma irradiation. At the same time, the most diverse diffraction patterns were seen for materials irradiated with doses greater than 100 kGy. They displayed decreased intensity and moved to the upper 2θ sides, indicating a reduction in unit cell volume, as illustrated in Figure 1d. According to the literature, the interpretation of crystal lattice deformation may induce variations in structural characteristics and unit cell volume of the crystal lattice. Tensile stress causes deformation to increase or decrease during irradiation.

Consequently, the crystal lattice's properties and volume may shrink [34]. The shrinkage of the unit cell following irradiation may be the cause of this reduction in lattice parameter. The X-ray data were refined to Rietveld refinement analysis using the Fullprof program to confirm the integrity of the absence of impact of the crystal phase of the BSmT sample after radiation, as shown in Figure 2a. The correctness of the studied structure was determined by looking at the values of the R-factors $R_p$%, $R_{wp}$%, $R_{exp}$%, and $\chi^2$. A plot discrepancy between the observed and computed patterns was also utilized to assess the Rietveld refinement's quality. Table 1 summarizes the structural parameters estimated from this refinement. Peak widening is expected to rise with increasing gamma dosage, leading to a reduction in crystallite size. Figure 2b shows the obtained parameters. According to the acquired results, a change in crystallite size and lattice strain was seen in the case of the sample with radiation of more than 100 kGy, indicating a rise in crystal lattice distortion. These defects might cause the unit cell to shrink compared to the ideal BSmT crystal before irradiation.

3.2 Morphological analysis

Figure 3 shows the typical pre- and post-gamma irradiation morphologies of polished and thermally etched BSmT samples. Grain size and density were shown to decrease with increasing gamma exposure. There was some grain size and orientation diversity among all samples, but overall, they had a plate-like shape. Grain length and thickness were both visibly reduced with increasing gamma dose. Gamma irradiation may significantly decrease the anisotropies of surface and grain boundary energies and mobilities. There were random-aligned grains visible in the samples. Grain size and grain orientation decreased and became more disordered in samples with a high gamma dose, such as 100 and 200 kGy. This finding agreed with the XRD result shown in Figure 2d, where the preferred orientation of (117)-type planes was found in BSmT ceramics. While looking at how gamma-ray irradiation affects grain size, the average length of grains developed along the c-axis was observed. These findings are consistent with previous research that has compared the morphology of ferroelectric thin films before and after being exposed to gamma rays [35,36].
Figure 2: (a) XRD refinements performed on BSmT ceramic, with the observed data shown in black circles, the refinement profile in red, the discrepancy between the measured and estimated diffraction patterns in blue, and Bragg reflections indicated in pink. (b) Variation of lattice constant and unit cell volume with gamma dosage. (c) Variation of peak broadening and (117) peaks shifting with gamma dosage is represented. (d) Represents the variation of crystallite size ($D_p$) and lattice strain with gamma dosage performed on BSmT ceramic.

Table 1: Rietveld refinement results of XRD data of BSmT ceramics irradiated with different gamma doses and measured at room temperature

<table>
<thead>
<tr>
<th>Parameter</th>
<th>@ 0 kGy</th>
<th>@ 50 kGy</th>
<th>@ 100 kGy</th>
<th>@ 200 kGy</th>
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<tr>
<td>Crystal system</td>
<td>Orthorhombic</td>
<td>Orthorhombic</td>
<td>Orthorhombic</td>
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<tr>
<td>Space group</td>
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<td>SG no.</td>
<td>69</td>
<td>69</td>
<td>69</td>
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</tr>
<tr>
<td>$a$ (Å)</td>
<td>5.44638 ± 0.01</td>
<td>5.44082 ± 0.01</td>
<td>5.44330 ± 0.01</td>
<td>5.40420 ± 0.01</td>
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<tr>
<td>$b$ (Å)</td>
<td>5.40754 ± 0.01</td>
<td>5.40827 ± 0.01</td>
<td>5.40280 ± 0.01</td>
<td>5.404 ± 0.01</td>
</tr>
<tr>
<td>$c$ (Å)</td>
<td>32.813 ± 0.01</td>
<td>32.813 ± 0.01</td>
<td>32.823 ± 0.01</td>
<td>32.772 ± 0.01</td>
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<tr>
<td>$a/b$</td>
<td>1.00718</td>
<td>1.00601</td>
<td>1.0075</td>
<td>1.00004</td>
</tr>
<tr>
<td>$V$ (Å$^3$)</td>
<td>966.39</td>
<td>965.52</td>
<td>965.29</td>
<td>962.38</td>
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<tr>
<td>Density (g/cm$^3$)</td>
<td>7.771</td>
<td>7.79</td>
<td>7.864</td>
<td>8.005</td>
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<tr>
<td>Measured density (g/cm$^3$)</td>
<td>7.541</td>
<td>7.543</td>
<td>7.524</td>
<td>7.336</td>
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<tr>
<td>Relative density</td>
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<td>96.82927</td>
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<td>FWHM of (117) peak</td>
<td>0.069228</td>
<td>0.089187</td>
<td>0.094155</td>
<td>0.134634</td>
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<tr>
<td>2θ of (117) peak</td>
<td>30.071</td>
<td>30.079</td>
<td>30.080</td>
<td>30.093</td>
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<td>The crystallite size (nm)</td>
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<td>$R_{ex}$ (%)</td>
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<td>$\chi^2$</td>
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3.3 Optical properties analysis

Figure 4a displays the UV–visible spectra of successively irradiated base sodium borosilicate glasses. The optical spectra of unirradiated BSmT ceramics exhibit a central band at 400 nm, indicating a high charge transfer ultraviolet absorption. Visible-region absorption spectra show a more significant improvement with increasing gamma

![Figure 3: SEM images of surface morphology of BSmT ceramics before and after gamma-ray irradiation.](image)

![Figure 4: (a) UV–vis absorption spectra, and (b) plots of $(\alpha E)^2$ versus $E$ of the BSmT samples before and after gamma irradiation.](image)
irradiation dosages. Many factors caused during the irradiation process [19,37–39] may account for the observed increase in UV absorption intensity, as suggested by earlier publications. Ionizing radiation may cause many electron-hole pairs to form on BSMT, and the trapped charges influence the material’s electrical characteristics by creating a local electric field.

Additionally, atoms in the lattice structure vary from their original location due to displacement effects when a sufficiently significant energy is applied. Atoms that move from their original sites because of the displacement effect may create vacancies there while also contributing to interstitial defects by disrupting the positions of neighboring atoms, causing the impurity to spread [19,37–39]. In addition, gamma irradiation may prevent defects from spreading by trapping them on the sample’s exterior [40]. Changes in the induced band as a function of gamma-ray dosage [41] provide strong evidence for an increasing number of the aforementioned caused defects in the lattice. Hence, the improved absorption spectra in the irradiated samples may result from combining the abovementioned causes. The study of optical absorption is an intriguing approach to learn about the band structure and energy bandgap of the irradiated samples by examining optically induced transitions. The optical bandgap of the materials under investigation was calculated using Tauc’s formula [42]:

\[(ahv) = C(hv - E_g)^n,\]

where \(C\) is a constant, \(h\nu\) is the energy of a photon, \(n\) is an exponent reflecting the band transition that relies on the type of optical transition (\(n = 1/2\) yields the direct energy bandgap, and \(a\) is the absorption coefficient. Extrapolating the slope line of the \((ahv)^2\) versus \((h\nu)\) as shown in Figure 4b. The values obtained were 3.25 eV for samples exposed to no radiation, 3.15 eV for those exposed to 200 kGy, 3.04 eV for those exposed to 100 kGy, and 2.95 eV for those exposed to 600 kGy. The optical absorption edge thus shifts into the visible spectrum. Changes in the induced bandgap energy as a function of gamma-ray dosage [41] provide strong evidence for an increasing number of the aforementioned caused defects in the lattice.

To prove the presence of defects caused by gamma irradiation, post-irradiated and irradiated samples of 200 kGy by gamma rays were chosen. Figure 5(a)–(d), depicts high-resolution XPS spectra with associated fitting findings of Ti 2p and O 1s. The peak spectra of Ti4+ 2p3/2 and Ti4+ 2p1/2 show that the binding energy is indicated at 456.92, 457.62 eV and 462.42, 463.62 eV for the BSMT sample before and after gamma irradiation, respectively, and these banding energies are similar to those of TiO2. Additionally, the spin–orbital splitting energies between the Ti4+ 2P3/2 and Ti4+ 2P1/2 peaks are determined to be 5.5 and 6 eV for the sample before and after irradiation, confirming the oxidation state of Ti4+.

Figure 5: High-resolution XPS spectra of Ti 2p and O 1s in the BSMT sample: (a) and (b) before and (c) and (d) after gamma irradiation with 200 kGy dosage.
Figure 5a shows no signal similar to Ti$^{3+}$ found for the non-irradiated sample, which may be attributable to its low Ti$^{3+}$ content. Further two signals are located in the irradiation sample with binding energies of 455.58 and 462.23 eV referred to as Ti$^{3+}$ 2p$^{3/2}$ and Ti$^{3+}$ 2p$^{1/2}$, respectively [43]. The concentrations of Ti$^{3+}$ ions in Ti for the irradiation sample were evaluated using the equation described in ref. [44], which was revealed to be 22%.

The primary peak at 529.54 and 529.49 eV in Figure 4(b) and (d) for the samples before and after gamma irradiation, which attributed to an oxygen lattice in the bulk Ti–O bond [45]. The oxygen vacancies are responsible for the other peak recorded at 530.53 and 530.93 eV. The oxygen vacancies content in the BSmT sample was determined to be 5.13 and 9.54% before and after 200 kGy gamma ray irradiation. As a result, it is inferred that the irradiated sample contains Ti$^{3+}$ surface defects and oxygen vacancies, which may play an essential role in improving the absorption spectra and decreasing the bandgap energy, confirming the preceding discussion.

### 3.4 Dielectric permittivity analysis

Polarization and loss processes in materials are heavily influenced by the material's composition, frequency, and temperature range. The three types are electronic or ionic conduction, dipolar relaxation, and atomic or electronic polarization. To investigate the effect of gamma rays on the dielectric properties of BSmT ceramics, post-irradiated and irradiated samples of 200 kGy by gamma rays were chosen, as depicted in Figure 6(a) and (b). The dielectric constant values before and after irradiation rose with increasing temperature, with the highest value recorded on the temperature dependences for the samples investigated, corresponding to the ferroelectric to the paraelectric phase transition. Furthermore, a diffused phase transition is occurring, which may be due to the fact that the difference in ionic radii between Sm and Bi may cause a local structural distortion, preventing long-range dipole alignment and driving polar nanoregions. As a result, the observed diffused phase change is caused by the substitution of Bi$^{3+}$. This inhomogeneous distribution causes compositional variability, resulting in microscopic heterogeneity with variable Curie points and observable diffuseness [46]. The Curie temperature value ($T_c$) was shifted from 568 to 496°C after irradiation with a gamma dose of 200 kGy; also, from Figure 6, we can see that the dielectric constant decreased from 648 to 632 after gamma irradiation. The reason behind reducing the phase transition after gamma irradiation can be explained by resorting to structural features. Table 1 shows that the lattice deformation ($a/b$) decreased with increasing gamma dose, which may explain why the Curie temperature shifted to the lower temperature location. On the other hand, SEM images reveal a decrease in grain size with increasing gamma dose, which leads to a reduction in $T_c$. The decreasing grain size may result in the formation of internal stress, which reduces the phase transition temperature [47]. Other researchers observed the same behavior in other ceramic materials [48–50].

### 3.5 Ferroelectric permittivity analysis

The ferroelectric properties at room temperature, both pre- and post-gamma irradiation, are shown in Figure 7(a).
The gamma irradiation was conducted at a specific dosage, while the measurements were performed at a frequency of 10 Hz and a maximum applied field of 73 kV/cm. The exposure to gamma rays resulted in a slight decrease in saturated and remnant polarization levels. However, the ferroelectric properties remained intact after exposure to a total gamma-ray dose of 200 kGy. Figure 7(b) illustrates the changes in the \(P_r\) and \(E_c\) parameters due to augmented gamma-ray irradiation, reaching a maximum of 200 kGy. After irradiation, the rate of change of the \(2P_r\) value exhibited a slight reduction compared to the BSmT without irradiation. The decrease in ferroelectric polarization may be primarily due to mobile defects, such as oxygen vacancies and gamma-ray stored energy [51,52]. Mobile defects, such as oxygen vacancies, exhibit an interaction with the polarity of the domain, leading to their migration in a manner that minimizes the overall energy. Mobile defects have the potential to aggregate in the domain boundaries, creating a depolarization field inside the grain. Consequently, the interaction between these mobile defects and domain polarity might lead to degradation in the ferroelectric properties of the irradiated materials. Although both samples were fabricated using the same conventional solid-reaction technique, it is evident that they exhibit differences in microstructure, indicating a little but conceivable effect. The XPS investigation in Figure 5 confirms the specific mechanism by which irradiation generates defects in ferroelectrics. A comprehensive examination was conducted on many sources to ascertain potential defects from gamma irradiation. Based on the results of our research, it has been shown that gamma rays have the potential to induce atomic displacement inside the matrix when interacting with matter [53]. When the energy reaches a sufficient level, it may cause the removal of atoms from their original positions, forming point defects [54,55]. In Figure 4, it is shown that energy levels inside the bandgap may be introduced as recombination sites, which are characterized by a reduction in bandgap energy. Modifying material properties, such as conductivity, mobility, and electron density, may occur via interaction with radiation, with the extent and kind of radiation determining the specific effects [56].

4 Conclusions

In summary, this study demonstrates a novel approach for modifying the structural, optical, and ferroelectric characteristics of samarium-modified bismuth titanate ceramics. This study used the solid-state reaction approach to create BSmT ceramics for high-energy mechanical ball milling. A comprehensive investigation was carried out into the effects of gamma irradiation on structural, optical, dielectric, piezoelectric, and ferroelectric characteristics. A \(^{60}\)Co gamma source irradiated the BSmT compound for 0, 50, 100, and 200 kGy. Even after a high dosage of gamma irradiation, the phase structure revealed the orthorhombic single-phase nature. Increasing gamma irradiation dosage reduced the band gap from 3.25 to 2.95 eV, decreasing the dielectric constant. The rationale for this reduction in bandgap was explored using defect creation caused by irradiation, which was corroborated by the XPS analysis. The temperature-dependent dielectric studies demonstrate that BSmT ceramics have a low dielectric loss and

Figure 7: (a) Measurement results of \(P-E\) hysteresis loops before and after gamma-ray irradiation on BSmT ceramics. (b) Measurement results of the variation of the \(P_r\) and \(E_c\) values of BSmT ceramics with gamma-ray irradiation.
temperature-dependent dielectric behavior, showing that they have outstanding dielectric property stabilities, which is crucial for high-temperature device applications. The ferroelectric investigation was conducted at room temperature, and the findings show that a higher gamma dosage causes a modest reduction in residual polarization. According to the research, lead-free compounds derived from irradiation BSmT are promising for photovoltaic applications.

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**Conflict of interest:** The authors declare that they have no conflicts of interest.

**Ethical approval:** The conducted research is not related to either human or animal use.

**Data availability statement:** All data that has been used is included in the manuscript.

**References**


