

Influence of La³⁺ substitution on the structural, magnetic and electrical characteristics of Sr₂FeTiO₆ double perovskites

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ABSTRACT

In this study, the La³⁺ ions are substituted at the Sr²⁺ site in Sr₂FeTiO₆ double perovskites using the solid state reaction method. The impact of La³⁺ion substitution on the Sr₂FeTiO₆ physical characteristics have been studied. The SrTiO₃ structural parameters were utilised as starting model for performing the Rietveld refinement of powder X-ray diffraction patterns. Cubic symmetry with *Pm3m* space group was determined for the prepared materials. Due to the larger ionic radii of La³⁺ ion, the La substituted samples showed slight increase in the lattice cell parameters leading to volume expansion. The La-substituted samples don't exhibit any significant morphological differences when compared to parent compound. The diffused reflectance measurements also show no significant changes in the band gap values of La-substituted samples in comparison with the pure compound. The magnetization studies exhibit the enhanced ferromagnetic nature of La-substituted sample. Thermally activated charge carriers are also evident from the analysis of temperature dependent dielectric measurements.

1 Introduction

Exploring the double perovskite materials for their magnetic and electrical properties is mostly carried out by alkaline earth or lanthanide element substitution at the A-site and transition metals at the B site. Due of the diverse variety of properties they exhibit, perovskite and double perovskite oxides with the general formulas ABO_3 and $A_2BB'O_6$ are of great interest. Double perovskites have attracted a lot of attention over the years due to their unique electrical and magnetic properties [1]. They are basically

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described by the equation A₂BB'O₆, where the A site is occupied by alkaline earth metals like Ba, Ca, Sr, and other similar ions, as well as rare earth ions with larger ionic radii [2]. The transition metal cations, alternatively, occupy the B octahedral positions, while lanthanides with smaller ionic radii do so for rare earth ions [3–6]. Double perovskites have an unusual combination of magnetic, dielectric, and conductivity properties depending on the degree of octahedral tilting [7, 8]. Interesting properties like magneto capacitance, multiferroicity [9, 10], magnetodielectric [11], ferroelectricity [12], spintronics for magnetic storage devices [13], thermopower [14] and thermoelectricity [15] towards technological applications have been shown by these perovskite and double perovskite materials.

Half metallic Sr₂FeMoO₆, which displayed extremely high magneto-resistance at room temperature, was used to conduct the initial research on the magneto-resistance of strontium-based double perovskites [16-18]. The compound Sr₂FeMoO₆ was used to study the magneto-caloric effect because it exhibited ferromagnetic ordering and may be used for ferromagnetic refrigeration [19]. It was this substance that led to the discovery of the random anisotropy magnetic effect [20-22]. Low field colossal magneto resistance at room temperature is a feature of the compound Sr_2FeMoO_6 [23]. The compound Sr₂FeCoO₆, which derives from the substitution of the Co-ion for Mo-ion, induces a spin glass state with mild ferromagnetic ordering [24]. A ferromagnetic to paramagnetic transition that happens when Mo-ions are swapped out for Mn-ions is investigated using dielectric measurements [25]. There has been very few research on strontium-based double perovskites with Ti ions at the B/B' site. Sr₂TiMnO₆ exhibited canted antiferromagnetic behaviour [26], Sr₂TiMoO₆ was utilised to research thermoelectric applications [27], and Sr₂FeTiO₆ compound showed spin glass behaviour [28]. Since transition metals are substituted for rare earth elements in the B site and alkaline earth elements in the A site, the current work examines how these modifications affect the magnetic and electrical properties.

The Sr_2FeTiO_6 [29] exhibited relaxor ferroelectric and magnetic spin glass properties that are interesting to observe [28]. This may be possible because the structure is a solid state mixture of $SrTiO_3$ and $SrFeO_3$. The Ti ion is known for its structural stability with its choice of crystallographic position. Similarly, the Fe ions produce disorder in the structure. In the current work, Mo ion has been replaced with Ti ion while La ion has been substituted in the A site, producing the double perovskite $Sr_{2-x}La_xFeTiO_6$. The examination of the literature reveals that there have been no previous reports on the synthesis or characterisation of rare earth substituted Sr_2FeTiO_6 double perovskite compounds. In order to investigate $Sr_{2-x}La_xFeTiO_6$ compounds, the synthesis has been carried out using a conventional solid state reaction technique. The following discussion covers the experimental details and results.

2 Experimental

High purity powders of SrCo₃ (99% purity, Spectrum Veagents and Chemicals), La2O3 (99% purity, Alfa Aesar), TiO₂ (99% purity, Alfa Aesar), and Fe₂O₃ (99% purity, Alfa Aesar) are combined in stoichiometric ratios in atomic weight% and ground for nearly two hours in an agate mortar. The $Sr_{2-x}La_{x-1}$ FeTiO₆ compounds (x = 0, 0.2, 0.4, 0.6, and 0.8) have been synthesised using the solid state reaction method. In the beginning, the powdered powders were calcined at room temperature for 8 h at 1000 °C. The calcined materials were then reground for two hours and sintered at 1200 °C for eight hours. For a homogeneous mixture of substances, a similar approach has been used and the samples were sintered to 1200 °C for eight hours for another two times with intermediate grindings. Using a D8 Advance Bruker diffractometer, a powder x-ray diffraction analysis of the produced compounds was carried out at room temperature. To estimate the structural parameters, Rietveld refinement was performed using Full Prof software. Physical electronics (PH15000 Versa Probe III Scanning XPS Microprobe) device was used to conduct the x-ray photoelectron spectroscopy investigation in order to determine the oxidation states. Gaussian Lorentzian fitting with Shirley background is used to fit the spectrum obtained for La, Sr, Fe, Ti, and O using the CASA XPS software. For the examination of morphology, the Quanta 200 FEG FESEM apparatus was used to produce the field emission scanning electron micrographs. Utilizing a Perkin Elmer LAMBD instrument, the diffused reflectance spectrum was obtained from a UV-Vis-NIR spectrophotometer. The Lakeshore VSM 7410 S equipment was used to conduct the



Fig. 1 a–c PS survey spectrum of the $Sr_{2-x}La_xTiFeO_6$ (x = 0, 0.2, 0.8) compounds. d–f Sr3d, Fe2p and Ti2p elemental spectrum of Sr_2TiFeO_6 compounds



Fig. 2 a & b C1s and O1s spectrum of Sr_2TiFeO_6 compound. c & d C1s and O1s spectrum of $Sr_{1.8}La_{0.2}TiFeO_6$ compound. e & f C1s and O1s spectrum of $Sr_{1.2}La_{0.8}TiFeO_6$ compound.

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Table 1Binding Energyvalues of C1s and O1sSpectrum

C 1s Fit	STFO	LSTF01	LSTFO4	O 1s Fit	STFO	LSTFO1	LSTFO4
Fe–C	283.4	283.3	284.5	М–О	528.1	526.8	529.3
С–С	284.7	285	285.4	М-О	531.1	528.3	531.6
(284.8 eV)							
C-O-C ($\sim 286 \text{ eV}$)	_	_	_	М-О	_	529.4	
O–C=O (288.5 eV)	_	_	_	C=O	_	531.2	_
. ,				С–О	_	_	_

 $STFO = Sr_2TiFeO_6, LSTFO1 = La_{0.2}Sr_{1.8}TiFeO_6, LSTFO4 = La_{0.4}Sr_{1.6}TiFeO_6$

Table 2Binding Energyvalues of elements obtainedfrom XPS spectrum

	Sr ₂ TiFeO ₆ (STFO) # (eV)	La _{0.2} Sr _{1.8} TiFeO ₆ (LSTFO1) # (eV)	La _{0.8} Sr _{1.2} TiFeO ₆ (LSTFO4) # (eV)
C 1s	284.7	285	284.5
O 1s	531.1	531.2	531.6
La 4d	_	836.7	838.1
Ti 2p	457	456.6	457.8
Fe 2p	710.4	710	710.5
Sr 2d	133.2	133.5	133.9

The values of Binding Energy as obtained from curve fitting of experimental data



Fig. 3 a XRD patterns of the prepared samples. b–f X-ray Diffraction Rietveld refinement patterns of the Sri_{2 – x}La_xTiFeO₆ (x = 0, 0.2, 0.4, 0.6, 0.8) compounds

magnetic measurements at room temperature. Dielectric readings at various frequencies and temperatures were analysed using the Wagner Kerr LCR 4275 m device.

Table 3 The data obtainedfrom Rietveld Refinement of		R _p	R _{wp}	R _{exp}	χ^2	a (Å)	b (Å)	c (Å)	Volume (Å ³)
XRD data is listed	Sr ₂ TiFeO ₆	4.42	5.76	4.83	1.44	3.8996	3.8996	3.8996	59.3029
	Sr _{1.8} La _{0.2} TiFeO ₆	5.82	7.68	4.94	2.42	3.9009	3.9009	3.9009	59.3600
	Sr _{1.6} La _{0.4} TiFeO ₆	5.60	7.24	5.09	2.03	3.9028	3.9028	3.9028	59.4457
	Sr _{1.4} La _{0.6} TiFeO ₆	4.78	6.57	5.36	1.51	3.9077	3.9077	3.9077	59.6710
	Sr _{1.2} La _{0.8} TiFeO ₆	5.00	6.71	5.41	1.54	3.9109	3.9109	3.9109	59.8169

Table 4 The structural parameters of Sr₂TiFeO₆

Atom	<i>x</i> Sr ₂ TiFeO ₆	Ŷ	Z	Occupancy
Sr1	0.5	0.5	0.5	1
Ti1	0	0	0	0.5
Fe1	0	0	0	0.5
0	0.5	0	0	3.0

Table 5Important Bondangles and Bond lengths ofSr2TiFeO6

Bond	Bond angles								
		Sr ₂ TiFeO	6 Sr _{1.8} La _{0.2} TiFeO	6 Sr _{1.6} La _{0.4} TiFeO ₆	5 Sr _{1.4} La _{0.6} TiFeO ₆	Sr _{1.2} La _{0.8} TiFeO ₆			
Sr-O-	–Sr	90°	90°	90°	90°	90°			
Fe–O	–Fe	180°	180°	180°	180°	180°			
Ti-O-	-Ti	180°	180°	180°	180°	180°			
Fe–O	-Ti	180°	180°	180°	180°	180°			
Fe–O	–Sr	90°	90°	90°	90°	90°			
Ti-O-	–Sr	90°	90°	90°	90°	90°			
Bond	Leng Sr ₂ ' (Å)	gths TiFeO ₆	Sr _{1.8} La _{0.2} TiFeO ₆ (Å)	Sr _{1.6} La _{0.4} TiFeO ₆ (Å)	Sr _{1.4} La _{0.6} TiFeO ₆ (Å)	Sr _{1.2} La _{0.8} TiFeO ₆ (Å)			
Sr– O	2.7	574	2.7584	2.7597	2.7532	2.7654			
Ti– O	1.94	498	1.9505	1.9514	1.9539	1.9555			
Fe– O	1.94	498	1.9505	1.9514	1.9539	1.9555			
	2.7	574	2.7584	2.7597	2.7632	2.7654			

3 Results and discussion

3.1 X-Ray Photoelectron Spectroscopy Analysis (XPS)

The oxidation states of the ions in the Sr_2FeTiO_6 (SFTO), $Sr_{1.6}La_{0.2}FeTiO_6$ (SLFTO1), and $Sr_{1.2}La_{0.8}$ -FeTiO₆ (SLFTO4) compounds were determined using XPS. The survey spectrum for the SFTO, SLFTO1, and SLFTO4 samples is shown in Fig. 1. All of the elements from the compound's stoichiometric combination are present, according to the obtained survey spectrum of the samples. In the SFTO compound, the Fe ion is present in the + 3 oxidation state. This is evident by the fact that the binding energy value of the Fe- $2p_{3/2}$ peak position determined by fitting at 710.7 eV closely resembles the binding energy value reported for the Fe₂O₃ molecule [30]. Additionally, it is observed that the Ti and Sr ions are present in + 4 and + 2 oxidation states, respectively, with reference to the binding energy values of TiO₂ and SrCO₃. The Fig. 2 displays the carbon and oxygen peak fits for each prepared compound. The binding energy values determined using the fit obtained for elemental scans are displayed in the Tables 1 and 2. The binding energy values thus obtained for the SLFTO1 and SLFTO4 samples also demonstrate the existence of Sr, Fe, and Ti in the + 2, +3, and + 4 oxidation states, respectively as well as La ion in the + 3 oxidation state. The XPS study therefore validates the incorporation of La ions into the double perovskite structure of Sr₂FeTiO₆.

Fig. 4 a SEM micrographs of $Sr_{2-x}La_{x}TiFeO_{6}$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds. b EDX images of $Sr_{2-x}La_{x}TiFeO_{6}$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds









(c)

(**d**)



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Fig. 4 continued

(a)





	1621 193	2					
Element At. No.		Mass	Mass Norm.	Atom	abs. error [%]	rel. error [%]	
		/	[%]	[%]	[%]	(1 sigma)	(1 sigma)
	Sr	38	46.37	43.26	19.01	2.07	4.46
	0	8	26.95	25.14	60.52	6.20	23.01
	Ti	22	15.78	14.72	11.84	0.57	3.59
	Fe	26	10.24	9.55	6.59	0.42	4.14
	La	57	7.84	7.32	2.03	0.36	4.57
			107.19	100.00	100.00		





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Element	At. No.	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Sr	38	29.73	28.93	12.67	1.33	4.49
0	8	27.28	26.54	63.66	5.23	19.16
La	57	20.67	20.11	5.56	0.69	3.32
Fe	26	13.18	12.83	8.81	0.47	3.60
Ti	22	11.93	11.60	9.30	0.43	3.57
		102.79	100.00	100.00		

(**d**)



Fig. 4 continued

3.2 Powder X-ray diffraction (XRD) analysis

Figure 3a shows the $Sr_{2-x}La_xFeTiO_6$ (x = 0, 0.2, 0.4, 0.6, 0.8) samples powder x-ray diffraction patterns at room temperature. Using the FULLPROF Suite programme, the diffraction pattern was refined using the Rietveld method. The matching six-coefficient polynomial function is used to fit the background, while the pseudo-Voigt function is utilised to fit the peaks. As a starting point for the refinement, the SrTiO₃ structural parameters with cubic symmetry and pm3m space group has been used. According to the results of Rietveld refinement, all of the La-substituted Sr₂FeTiO₆ compounds crystallize in single phase cubic symmetry system without any secondary phases. The dependability factors demonstrate a reasonable fit between the experimental and calculated patterns. The optimized XRD patterns are presented in the Fig. 3b-f, and the Tables 3 and 4 contains the calculated lattice parameters, reliability factors, bond angle, and bond distances. Significant bond lengths and bond angles are listed in Table 5.

3.3 SEM and EDS Analysis

Figure 4a shows the double perovskite SLTFO's surface microstructure. Because different-sized grains were present and evenly dispersed across the whole surface, the sample was likely prepared without voids. All samples exhibit the same morphologies and appear to be homogeneous and highly uniform. Due to the greater processing temperature, each sample has significant particle sizes and aggregates. The distribution, size, and form of the grains in the microstructure have revealed that the examined double perovskite samples are polycrystalline in nature. The micrographs also show the existence of small particles produced during preliminary grinding. Figure 4b shows the EDS patterns for the double perovskite SLTFO. The prepared sample's EDS spectra contained the elements Sr, Ti, Fe, La, and O, which ruled out the inclusion of any extraneous metals or elements. The element ratios utilised to prepared the samples are roughly matched by the atomic percentage values of each sample, as listed in Table 6.

3.4 Diffused Reflectance Spectroscopy (DRS) analysis

Figure 5a shows the diffuse reflectance UV-Vis spectra of $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6, and0.8) that were taken in the range of 200 to 800 nm. Using the Kubelka-Munk function, the graph is plotted between $hv(1-R)^2/2R$ vs. hv(eV) as given in Fig. 5b. The intercepts are measured by extrapolating the sharp edges and by drawing a line along a sharp edge in order to determine the energy band gap value. Peak intensity rises with the La concentration in the compounds. The charge transfer transition between the doped La^{3+} and O^{2-} (valence and conduction band) in the lattice structure may have been brought on by a significant absorption. As the concentration of La³⁺ rises, the molecule's reflectance value rises as well, which suggests that its absorbance properties are weakening. The absorption coefficient is derived using the Kubelka-Munk (KM) function using diffuse reflectance spectra, as illustrated in equation:

$$f(R) = \frac{\alpha}{s} = \frac{\left(1 - R\right)^2}{2R}$$

where f(R) represents the KM function, α is the absorption coefficient, s is the scattering coefficient, and R is the reflection coefficient. The band gap values for the samples have been determined using the straight line fit in the graph shown in Fig. 5b. Sr₂ _xLa_xTiFeO₆ double perovskites have energy band gaps ranging from 2.0 to 2.6 eV for x = 0.2, 0.4, 0.6,and 0.8. The band gap values in the samples gradually increase as the La³⁺ doping concentration increases [31, 32].

3.5 Magnetization study

The vibrating sample magnetometer was used to analyse magnetization curves in order to evaluate the samples' magnetic properties. Figure 6 displays the pure and La-substituted Sr₂TiFeO₆ samples' fielddependent magnetization (M-H) curves. The Figure illustrates the narrowing of the hysteresis loops

Table 6 Values of atomicweight % obtained from EDS	Details of the compound	Weight %					Band gap value (eV)
analysis of Sr _{2-x} La _x TiFeO ₆ compounds and their Band		La	Sr	Ti	Fe	0	
Gap Values	Sr ₂ TiFeO ₆	0	46.13	13.59	13.15	22.80	3.4
	Sr _{1.8} La _{0.2} TiFeO ₆	7.32	43.26	14.72	9.55	25.14	2.0
	Sr _{1.6} La _{0.4} TiFeO ₆	13.96	35.22	12.09	9.55	29.17	2.3
	Sr _{1.4} La _{0.6} TiFeO ₆	20.11	28.93	11.60	12.83	26.54	2.6
	Sr _{1.2} La _{0.8} TiFeO ₆	19.35	34.07	14.37	9.79	22.42	2.4



Fig. 5 a Diffused reflectance graph and b KM plot for the determination of energy band gap of $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds



Fig. 6 Magnetization curves obtained for $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6 and 0.8) compounds using VSM



Fig. 8 Temperature dependent resistivity plots of $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds

Table 7	Magnetization	properties of	Sr_2xLa_x	TiFeO ₆ (x =	= 0.0, 0.2,	0.4, 0.6,	0.8) compounds
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Compound details	Magnetic saturation (M_S) (emu/g)	Coercivity (H _c) Gauss	Magnetic retentivity (M_R) emu/g	Squareness ratio (M _R /M _S)
	04.540	2057.0	11.020	0.4400
$Sr_2 I1FeO_6$	24.542	2057.0	11.020	0.4490
Sr _{1.8} La _{0.2} TiFeO ₆	6.0613	7.9661	4.9895	0.8231
Sr _{1.6} La _{0.4} TiFeO ₆	5.2049	25.124	6.6069	1.2693
Sr _{1.4} La _{0.6} TiFeO ₆	6.3589	1575.1	1.3397	0.2106
Sr _{1.2} La _{0.8} TiFeO ₆	55.701	2198.6	30.548	0.5484



Fig. 7 a Temperature and b Frequency dependent dielectric constant plots of $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds

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Fig. 9 Arrhenius plots for the determination of activation energy of $Sr_{2-x}La_xTiFeO_6$ (x = 0.0, 0.2, 0.4, 0.6, 0.8) compounds

with the La concentration. Saturation was attained at a maximum applied magnetic field of 15 KOe. There is a noticeable increase in the magnetization (saturation of magnetization, Ms) in this field with an increase in lanthanum. According to the magnetization value obtained at the maximum applied field, the Sr₂TiFeO₆ compound has ferromagnetic (FM) ordering. Due to the enhanced magnetization observed for the applied magnetic field, the highest magnetization values of the La³⁺ substituted samples show a substantial increase. Due to its high crystallinity and homogeneous morphologies as observed in SEM micrographs, the saturation magnetization (Ms) value for the Sr₂TiFeO₆ sample is relatively low when compared to La³⁺ ion replacement materials. This is a result of the formation of the magnetic perovskite structure as well, which happens when the most magnetic ions, as opposed to nonmagnetic lanthanum ions, are substituted.

The coercivity (H_C) of the samples increases from 2057 G for pure to 2198 G for the sample with maximum La^{3+} replacement, as shown in the Table 7. Remanent magnetization (M_R) values (between 11.020 emu/g and 30.548 emu/g) rise parallel to the increase in La^{3+} ion concentration. The intrinsic and extrinsic features of the material affect coercivity and remanent magnetization.

The likelihood of substituent-induced structural disorder in the synthesised compounds is demonstrated by the gradual increase in coercive field and remanent magnetization, which also points to the integration of La^{3+} ions into the crystal lattice of the Sr_2TiFeO_6 combination. La^{3+} ions are thought to be present in the Sr_2TiFeO_6 structure, which is supported by the observed magnetization behaviour in the La^{3+} -substitution samples.

3.6 Dielectric analysis

Figure 7a illustrates the temperature dependent dielectric constant plot for various frequencies. The measured values of dielectric constant do not change, upto to about 415 K. However, after this temperature is reached, the observed values of dielectric constant start to rise. The frequency-dependent dielectric constant graph is shown in the Fig. 7b at various temperatures. For all substances, it is discovered that the dielectric constant value decreases with frequency. The variation of resistivity with temperature is shown in Fig. 8. The Arrhenius plot used to determine the activation energy (E_a) is shown in Fig. 9. Utilizing a straight-line equation and linear fitting, the activation energy is determined, and the results are displayed graphically. It is concluded from analysing the resistivity curves that the resistivity of the materials decreases with increase in temperature. This is due to the fact that thermally activated charge carriers are evident from the activation energy values of the samples (Fig. 9). This may happen due to the release of electrons and creation of more defects in the sample with increase in temperature which is generally observed in double perovskite materials.

4 Conclusion

Polycrystalline double perovskite $Sr_{2-x}La_xFeTiO_6$ (x = 0, 0.2, 0.4, 0.6, 0.8) samples were successfully synthesized having single phase nature using solid state reaction method. X-Ray photoelectron spectroscopy study proves that Sr, La, Fe, and Ti ions exist in + 2, +3, and + 4 oxidation states in the double perovskite structure. The cubic *Pm3m* structure for the system with randomly arranged Fe and Ti cations at the octahedral sites were confirmed through the Rietveld refinement of X-ray diffraction data. With the substitution of lanthanide at the A-site of the double perovskite compound, the magnetic and conductivity properties were modified. The sample with highest La substitution showed a better ferromagnetic curve when compared to parent compound. The thermally activated charge carriers added to the conductivity of the prepared samples at high temperatures as revealed in temperature dependent dielectric studies.

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Author contributions

JSP: Conceptualization of work, experimental work and discussion of data, creation of initial draft of the article, AN: Supervision of the work carried out for the article, discussion of obtained experimental data, VA: Discussion of obtained experimental data, MD: Discussion of obtained experimental data, MM: Discussion of obtained experimental data, KSK: Supervision of the work carried out for the article, discussion of obtained experimental data, finalization of article.

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Data availability

All data generated or analysed during this study are included in this published article.

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