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Influence of Sr Substitution on the Phase Formation and Electrochemical Properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ Perovskites

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$\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ ($x = 0, 0.15, 0.30$ and 0.50) perovskite powders were synthesized by a modified Pechini sol-gel method. Differential thermal analysis revealed that heat treatment at approximately 800°C is required for complete formation of the perovskite phase. X-ray diffraction analysis showed that the as-synthesized powders possess an orthorhombic perovskite-related structure (Pnma), while heat treatment at 800°C results in the formation of a single-phase rhombohedral perovskite structure (R-3c). Scanning electron microscopy demonstrated that the powders consist of agglomerated nanoparticles with grain sizes ranging from 50 to 200 nm. Electrochemical investigations revealed that Sr substitution significantly influences the capacitive properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$. The highest specific capacitance of 118 F g^{-1} was obtained for $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_{3-\delta}$, which is attributed to an optimal balance between electronic conductivity and the concentration of electrochemically active $\text{Mn}^{3+}/\text{Mn}^{4+}$ redox centers. The results demonstrate that controlled Sr substitution is an effective approach for tailoring the structural and electrochemical properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$, making these materials promising candidates for supercapacitor electrode applications.

Keywords: perovskite, phase formation, XRD, morphology, $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$, Sr substitution, modified Pechini method, electrochemical properties, supercapacitors.

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Introduction

Lanthanum-based perovskite oxides have attracted considerable attention because of their structural flexibility, mixed-valence manganese ions, high chemical stability and tunable electrical properties. Lanthanum-based perovskite materials exhibit notable optical [1, 2], magnetic [3, 4], and catalytic properties [5]. Owing to their high power conversion efficiency, and capacity for isomorphic metal substitution, they have been widely

investigated for diverse applications, including catalysts [6-8], chemical sensors [9], solid oxide fuel cell electrodes [6], hydrogen storage [10], optoelectronic devices [11], solar cells [12], and magnetic materials [13-15]. Among these, Ag-decorated $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_{3-\delta}$ perovskite nanoparticles have been proposed as promising electrode materials for supercapacitor applications [16].

By substituting lanthanum with strontium in the LaMnO_3 crystal lattice, the valence state of manganese can be tuned, which in turn strongly influences electrical

conductivity, magnetic behavior, and charge transport. Owing to these properties, $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ is highly promising for diverse applications, ranging from cathode materials for solid oxide fuel cells [17] to electrode materials for supercapacitors [18–22] and other energy-conversion and storage devices.

Recent studies have demonstrated that Sr substitution significantly improves the electrochemical performance of LaMnO_3 -based electrodes [23–26]. In particular, enhanced capacitance has been attributed to improved electronic conductivity, optimized $\text{Mn}^{3+}/\text{Mn}^{4+}$ ratios, and favorable microstructural characteristics. However, the reported electrochemical performance strongly depends on the synthesis route, thermal treatment and Sr concentration, while the relationship between crystal structure, morphology and electrochemical behavior remains insufficiently understood. Therefore, further investigation of the influence of Sr substitution on the structural and electrochemical characteristics of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ remains an important task for optimizing electrode materials for supercapacitors.

The aim of this work is to investigate the effect of Sr substitution on the phase formation, morphology and electrochemical properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ synthesized by the modified Pechini method and to determine the optimum Sr concentration for supercapacitor electrode applications.

I. Materials and Methods

Perovskite materials with the composition $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ ($x = 0; 0.15; 0.3; 0.5$) were synthesized using a modified the Pechini method. The starting reagents included lanthanum nitrate, strontium nitrate (in the required compositions), manganese nitrate, citric acid as a complexing agent, and ethylene glycol as a polymerization agent.

The phase composition of the obtained samples was analyzed by X-ray diffraction using Shimadzu XRD 7000 instruments with $\text{Cu(K}\alpha\text{)}$ radiation ($\lambda = 1.541 \text{ \AA}$). The experimental diffractograms were then analyzed using the Rietveld method in FullProf software [27–29].

Thermal characteristics of the samples were investigated using the STA Jupiter 449 F3 system. Data

were recorded over a temperature range of 25–1200 °C at a controlled heating rate of 20 °C/min under an argon flow of 20 ml/min.

The morphological features of the samples were examined using a JSM 6700F field-emission scanning electron microscope. Imaging was performed in secondary electron mode at an accelerating voltage of 10 kV and a beam current of 0.75 nA.

To investigate the electrochemical properties of the $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ /electrolyte systems, a standard three-electrode electrochemical cell was employed. The active layer of the working electrode was prepared by mixing the investigated material with acetylene soot, serving as a conductive additive, and polyvinylidene fluoride. The components were combined in a mass ratio of 85:10:5. All measurements were carried out in an aqueous 3 M KOH solution, which served as the electrolyte. The preparation of the working electrodes and the electrochemical testing procedure were similar to those reported previously for oxide-based supercapacitor materials [30, 31]. Electrochemical testing was carried out using an Autolab PGSTAT/FRA 12. The specific capacitance of the materials was determined from the integral area of the cyclic voltammetry curves [32].

II. Results and discussion

X-ray diffraction patterns of the as-synthesized $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ powders obtained after the autocombustion stage are shown in Fig. 1a. The diffraction peaks of these as-synthesized powders correspond predominantly to an orthorhombic perovskite-related structure (space group Pnma) together with a small amount of secondary phases, indicating that the crystallization process is incomplete after autocombustion. Increasing the Sr content slightly broadens the diffraction peaks of the as-synthesized powders, which may be associated with the formation of nanosized crystallites and increased structural disorder. Therefore, an additional heat treatment was required to obtain a single-phase perovskite material.

To determine the appropriate temperature for the final heat treatment, differential thermal analysis (DTA/TG) was performed on the as-synthesized powders obtained

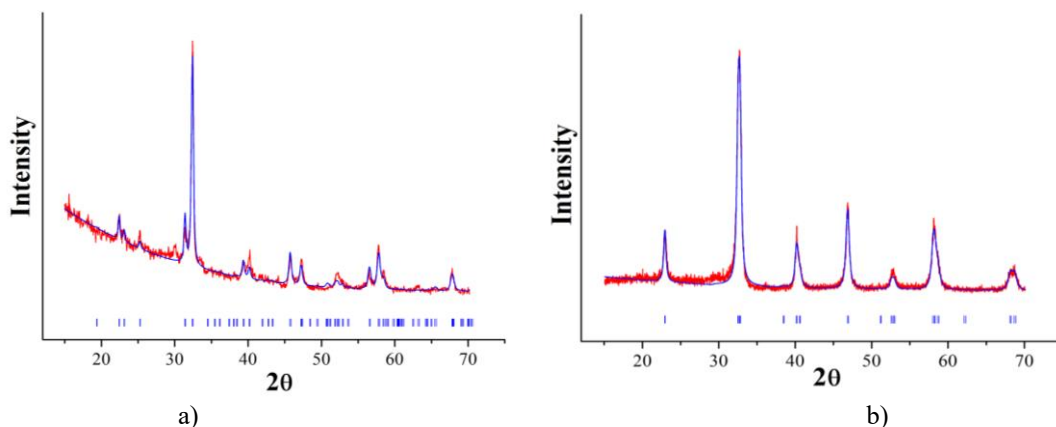


Fig. 1. XRD patterns $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ powders ($x = 0$): (a) as-synthesized samples after autocombustion and (b) samples after heat treatment at 800°C.

after autocombustion. The thermal behavior of the samples depends on the Sr content, which affects the $\text{Mn}^{3+}/\text{Mn}^{4+}$ ratio and consequently influences the crystallization process of the perovskite phase.

The DTA/TG curves (Fig. 2) show that the main thermal transformations occur below 800 °C. The exothermic effects observed within the 400–800 °C range are associated with decomposition of precursor compounds and crystallization of the perovskite phase. With increasing Sr content, these thermal effects become more pronounced, indicating enhanced precursor reactivity and faster phase formation.

The temperature interval of 280–320 °C corresponds to the combustion of the organic components formed during the modified Pechini process, while the exothermic peak at 650–700 °C is associated with crystallization of the rhombohedral perovskite phase. Above approximately 800 °C, no significant mass changes are observed, indicating completion of the phase formation. Therefore, 800 °C was selected as the optimum heat treatment temperature to obtain a single-phase perovskite while preserving the nanostructure.

After annealing at 800 °C, all $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ samples exhibit well-defined diffraction peaks characteristic of a highly crystalline rhombohedral perovskite structure (space group R-3c) (Fig. 1b). Rietveld refinement confirmed that the annealed samples are single-phase

within the detection limit of X-ray diffraction. A gradual shift of diffraction peaks with increasing Sr concentration indicates successful incorporation of Sr into the perovskite lattice. X-ray phase analysis indicates that the average size of coherent scattering domains, calculated according to the Debye-Scherrer and Williamson-Hall approaches [33, 34], is approximately 20 nm, reflecting the nanoscale character of the crystallites.

SEM images of the surfaces of samples after heat treatment at 800 °C are presented in Fig. 3. For the undoped sample (Fig. 3a), a uniform surface with an almost continuous granular layer is observed. Particle sizes range from 50 to 80 nm, with minimal porosity. At $x = 0.5$ (Fig. 3b), the microstructure displays the most pronounced fragmentation. The surface exhibits macroporosity, complex topography, and larger agglomerates composed of smaller particles, confirming the significant influence of Sr substitution on the microstructure developed during heat treatment.

Figure 4a presents CV curves of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ materials with compositions $x = 0$; 0.15; 0.3 and 0.5, recorded in the potential window from -0.2 to 0.6 V at a scan rate of 1 mV/s in KOH electrolyte. The curves exhibit an almost rectangular profile with slight distortions, characteristic of pseudocapacitive behavior accompanied by contributions from redox processes. The CV curve of the $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ sample with $x = 0$ exhibits a

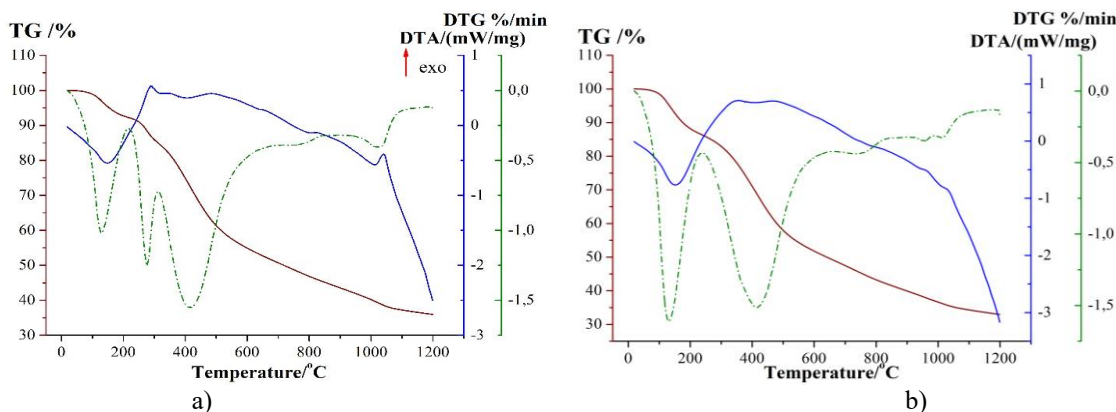


Fig. 2. DTA, TG, and DTG Curves of (a) $\text{La}_{0.85}\text{Sr}_{0.15}\text{MnO}_{3-\delta}$ and (b) $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_{3-\delta}$ powders.

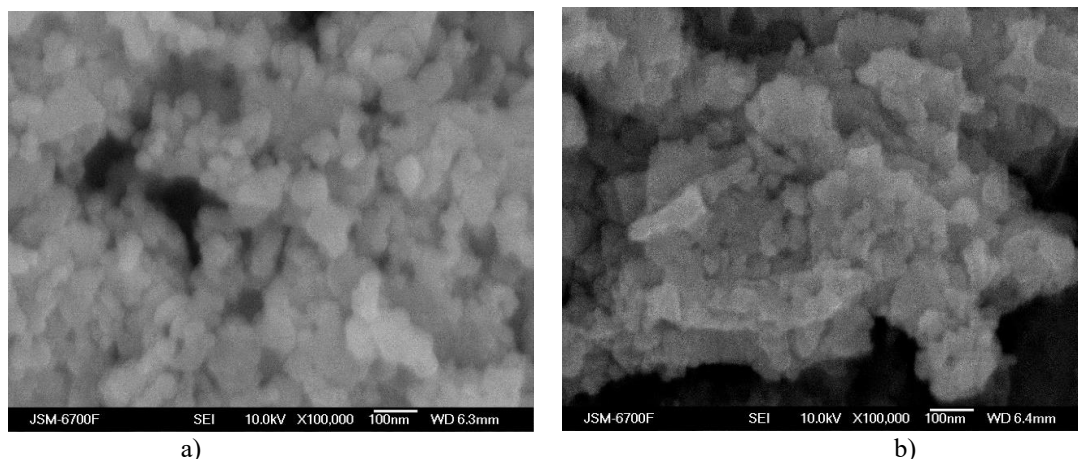


Fig. 3. SEM of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ surfaces after heat treatment at 800 °C for $x = 0$ (a) and $x = 0.5$ (b).

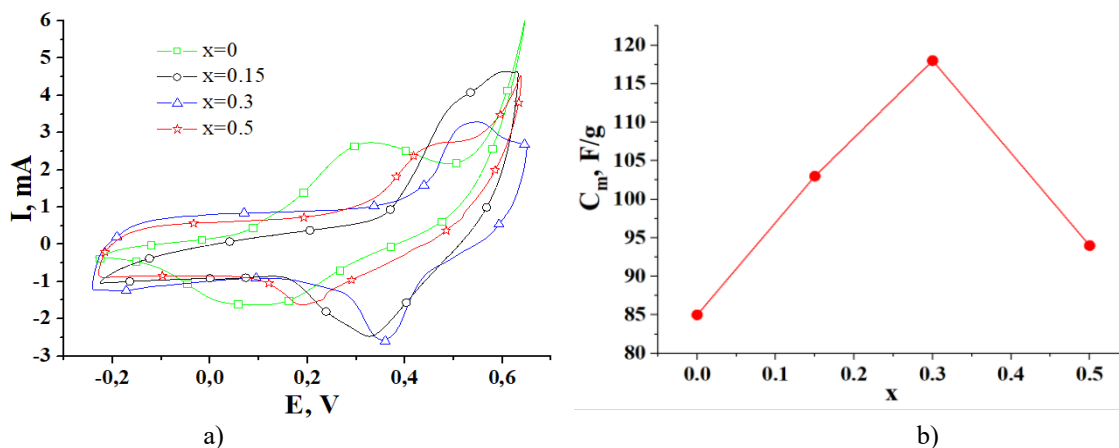


Fig. 4. CV curves of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ samples (a) and specific capacitance values (b) as a function of Sr substitution.

pseudocapacitive profile with moderately pronounced redox peaks. In contrast, the curves for $x = 0.15$ and $x = 0.3$ display higher currents and more distinct redox peaks, reflecting the highest electrochemical activity among the compositions studied. At $x = 0.5$, the curve shape changes slightly: the redox peaks become less defined, and the maximum current decreases.

The substitution of Sr for La in the LaMnO_3 perovskite lattice has a pronounced effect on the electrochemical properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$. The specific capacitance increases with Sr content up to $x = 0.3$, reaching a maximum value of 118 F g^{-1} for $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_{3-\delta}$ (Fig. 4b). This behavior can be attributed to an optimal $\text{Mn}^{3+}/\text{Mn}^{4+}$ ratio, which improves electronic conductivity and provides favorable conditions for reversible redox processes. However, further increase in Sr content to $x = 0.5$ leads to a decrease in capacitance, most likely due to excessive substitution, structural disorder, and a reduced number of electrochemically active redox centers. Therefore, $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_{3-\delta}$ can be considered the most promising composition among the studied samples for further supercapacitor research.

Sr substitution significantly affects the electrochemical properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$. Cyclic voltammetry measurements confirmed the capacitive behavior of all investigated compositions. The highest specific capacitance of 118 F g^{-1} was achieved for $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_{3-\delta}$, indicating that this composition provides the most favorable balance between structural stability, electrical conductivity and the concentration of electrochemically active $\text{Mn}^{3+}/\text{Mn}^{4+}$ redox centers.

The obtained results demonstrate that controlled Sr substitution is an effective approach for tuning the structural and electrochemical properties of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ perovskites, making these materials promising candidates for supercapacitor electrode applications.

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Conclusions

$\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ perovskite powders were successfully synthesized by the modified Pechini method and heat-treated at 800°C . X-ray diffraction analysis revealed that the as-synthesized powders possess an orthorhombic perovskite-related structure (Pnma), whereas heat treatment at 800°C leads to the formation of a single-phase rhombohedral perovskite structure (R-3c) for all investigated compositions.

Based on the data of DTA/TG/DTG analysis, the following characteristic temperature intervals were identified: $\sim 280\text{--}320^\circ\text{C}$ – organic burnout; $\sim 650\text{--}700^\circ\text{C}$ – exothermic peak associated with crystallization of the perovskite phase; $\sim 800^\circ\text{C}$ – mass stabilisation and completion of perovskite phase formation.

Scanning electron microscopy demonstrated that the obtained powders consist of agglomerated nanoparticles with grain sizes ranging from 50 to 200 nm.

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Вплив заміщення Sr на фазоутворення та електрохімічні властивості перовскітів $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$

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Перовскітні порошки $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$ ($x = 0; 0,15; 0,30; 0,50$) синтезовано модифікованим золь-гель методом Печіні. Диференціальний термічний аналіз показав, що для повного формування перовскітної фази необхідна термічна обробка за температури близько 800 °С. За даними X-променевої дифрактометрії встановлено, що синтезовані порошки до термообробки мають орторомбічну перовскітоподібну структуру з просторовою групою $R\bar{3}m$, тоді як термічна обробка при 800 °С приводить до формування однофазної ромбоєдричної перовскітної структури з просторовою групою $R\bar{3}c$. Растрова електронна мікроскопія продемонструвала, що порошки складаються з агломерованих наночастинок із розмірами зерен від 50 до 200 нм. Електрохімічні дослідження засвідчили, що заміщення Sr істотно впливає на ємнісні властивості $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$. Найвище значення питомої ємності – 118 Ф/г – отримано для $\text{La}_{0,7}\text{Sr}_{0,3}\text{MnO}_{3-\delta}$, що пов'язано з оптимальним співвідношенням між електронною провідністю та концентрацією електрохімічно активних окисно-відновних центрів $\text{Mn}^{3+}/\text{Mn}^{4+}$. Отримані результати показують, що контрольоване заміщення Sr є ефективним підходом до цілеспрямованого регулювання структурних та електрохімічних властивостей $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$, що робить ці матеріали перспективними для застосування як електроди суперконденсаторів.

Ключові слова: перовскіт, фазоутворення, X-променевий структурний аналіз, морфологія, $\text{La}_{1-x}\text{Sr}_x\text{MnO}_{3-\delta}$, заміщення стронцієм, модифікований метод Печіні, електрохімічні властивості, суперконденсатори.