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**Effect of combustibles on the structural properties of LaFeO₃
synthesized by auto-combustion**

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Abstract

Lanthanum ferrite, LaFeO₃, is a perovskite type material with an orthorhombic structure that was synthesized by auto-combustion of different fuels from where we have obtained the structure (in the presence of amine group no further calcination was needed). This perovskite structure was confirmed by : XRD, Infra-red spectroscopy (IR), UV-visible spectroscopy and laser particle size.

Key Words : LaFeO₃, perovskite, auto-combustion, XRD, IR, UV-visible and laser particle size.

1. INTRODUCTION

Perovskite oxides consist of large families among the structures of oxide compounds, and several perovskite-related structures are currently recognized. Typical structures consist of large-sized 12-coordinated cations at the A site and small-sized 6-coordinated cations at the B site [1-2].

Perovskites induce a great interest because of a variety of properties presented in these materials according to the choice of the elements A and B [3] : conducting (insulating, superconducting, ionic conduction, electronic conducting) piezoelectricity, ferromagnetism and magnetoresistivity, catalysis at high temperature [4].

One of the most common perovskite oxides [5-6], LaFeO₃ nanoparticles exhibited good photocatalytic properties such as water decomposition and dye degradation under visible light irradiation [7-8].

The perovskite oxides are usually synthesized by a variety of methods including the reaction in the solid state [9], co-precipitation technique [10-11], polymerized complex method [12], combustion synthesis [13], spray drying, the cryochemical method (freeze-drying), and the sol-gel (using in particular the amorphous citrate complexes) [9].

2. MATERIALS AND METHODS

The method of preparation used for this study is an auto-combustion process in aqueous method proposed for the first time in 1990 by Chick in the synthesis of chromites and manganites powders, which is called Glycine Nitrate Process (GNP) [14]. The precursors are generally used to synthesize the compound LaFeO_3 : Lanthanum nitrates $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and Iron nitrates $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ as oxidizers, citric acid and glycine as fuels, which are dissolved in distilled water and also using ammonia with citric acid to adjust the pH to 7 for preparing three samples :

Sample 1 : LaFeO_3 prepared by citric acid as a fuel.

Sample 2 : LaFeO_3 prepared by citric acid as a fuel by adding ammonia to adjust the pH to 7.

Sample 3 : LaFeO_3 prepared by glycine as a fuel.

The solutions are then mixed at a room temperature in the stoichiometric proportions. The mixture is heated between 50 to 60 minutes until obtaining combustion and cinders. The resulting products are submitted to grinding until we had a fine powder and it requires the calcination only with sample 1 prepared by citric acid.

3. RESULTS AND DISCUSSIONS

The figures (1, 2, 3 and 4) shows the 3 samples after combustion and grinding :



Fig. 1- Sample 1 : Cinders obtained.

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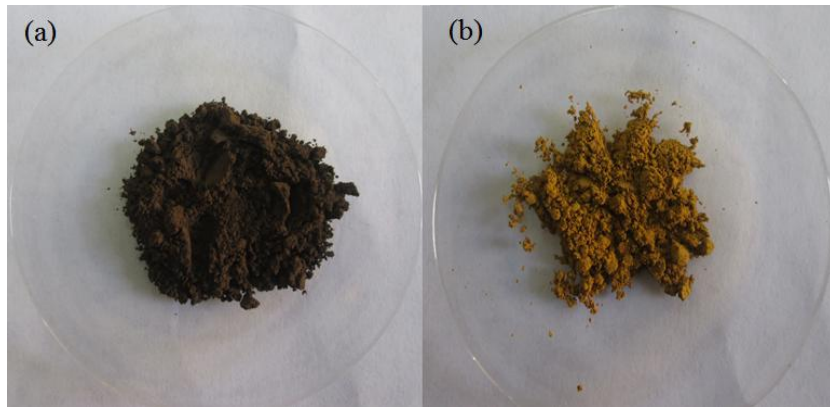


Fig. 2- Sample 1 : (a) : brown powder before calcination, (b) : yellow powder after calcination.

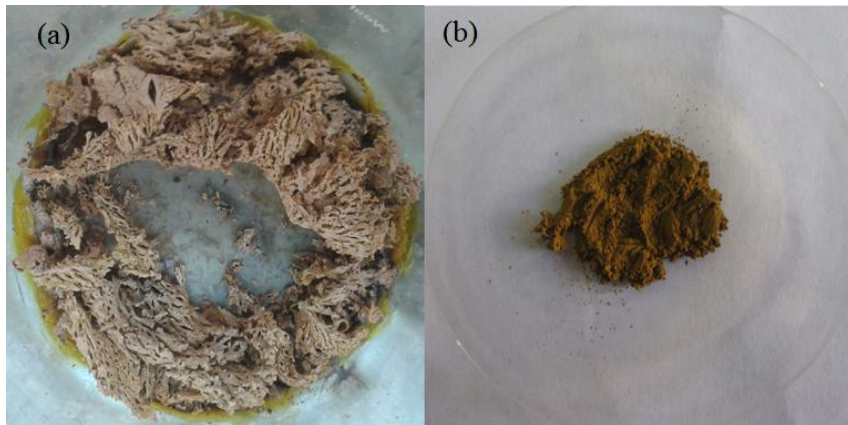


Fig. 3- Sample 2 : (a) : cinders obtained, (b) : yellow powder obtained without calcination.

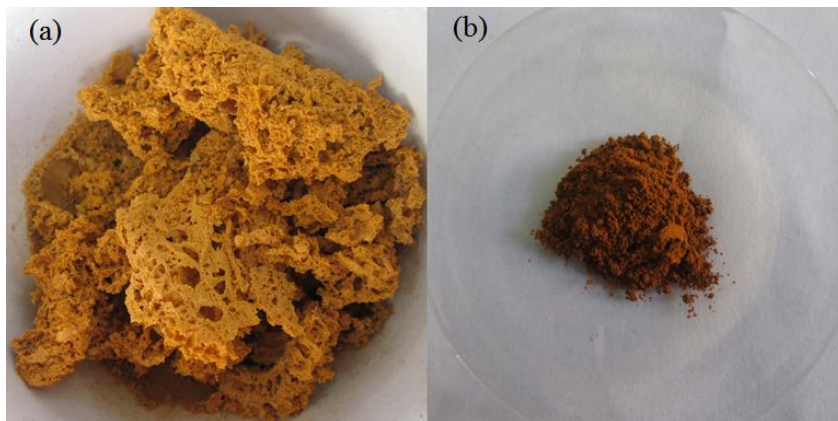


Fig. 4- Sample 3 : (a) : Cinders obtained, (b) : orange powder obtained without calcination.

3.1. X-ray diffraction

The XRD measurements (Fig. 5) shows that the perovskite oxide LaFeO_3 is a product with an orthorhombic structure and Pnma space group with parameters of : $a = 5.566 \text{ \AA}$, $b = 7.854 \text{ \AA}$ and $c = 5.553 \text{ \AA}$ ($\alpha = \beta = \gamma = 90^\circ$). The diffraction data are in a good agreement with ASTM card of LaFeO_3 from software X'pert highscore (ASTM no: 00-037-1493).

3.2. Infra-red spectroscopy

It is seen for the three samples (Fig. 6,7) an intense band at 560 cm^{-1} attributed to vibrations of the Fe-O band in the perovskite LaFeO_3 . The presence of this band confirmed the formation of the perovskite phase in those three samples.

3.3. UV-visible spectroscopy

In the above spectra of the three samples (Fig. 8,9) we can see that the maximum wavelength is : $\lambda_{\text{max}} = 326 \text{ nm}$.

This wavelength ($\lambda_{\text{max}} = 326 \text{ nm}$) indicates that the LaFeO_3 compound prepared by the auto-combustion method is a photocatalytic material type according to the work published in [15]. The difference in the values of the wavelengths refers to the experimental conditions.

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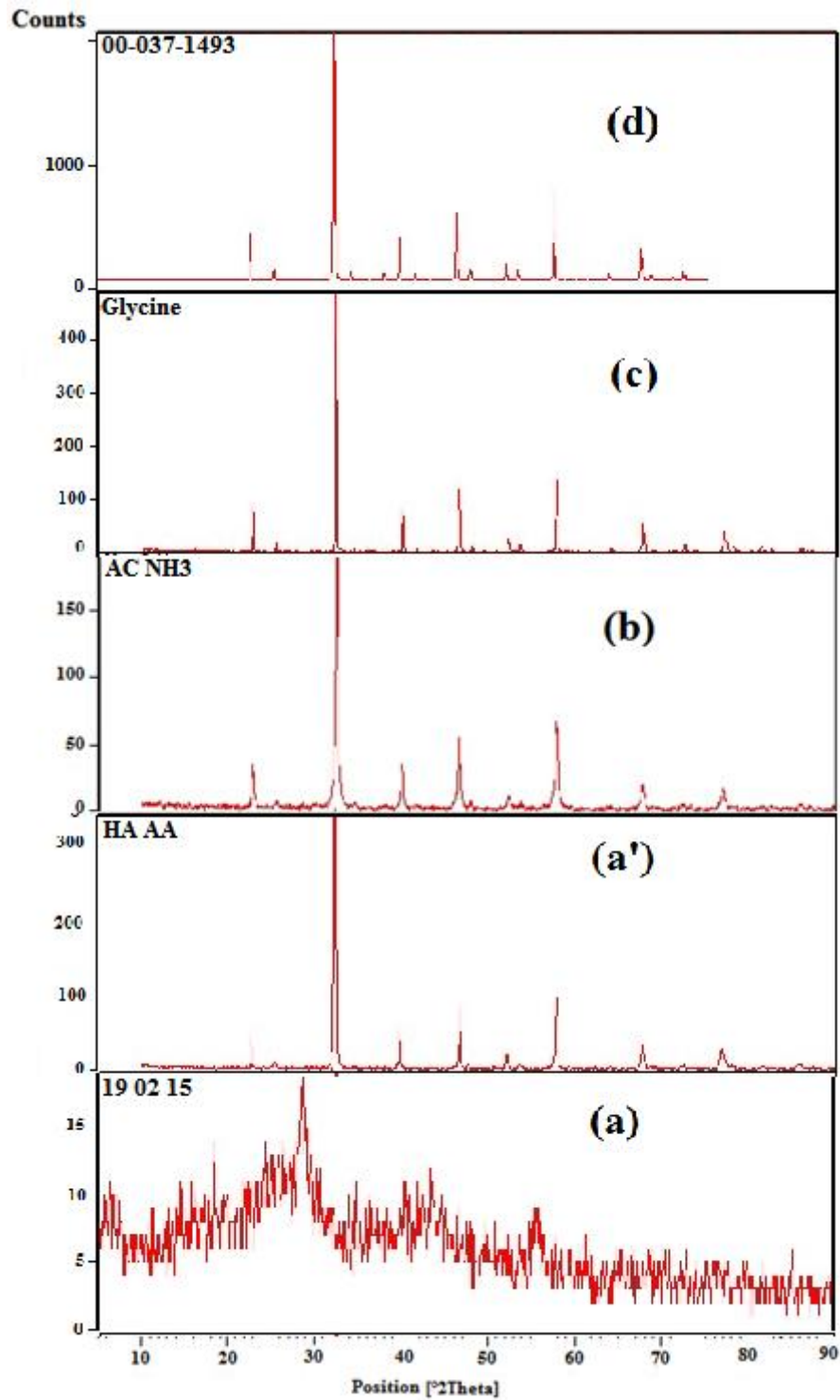


Fig. 5- diffractograms : 1st sample : (a) before calcination, (a') after calcination, (b) : 2nd sample, (c) : 3rd sample and (d) : ASTM card.

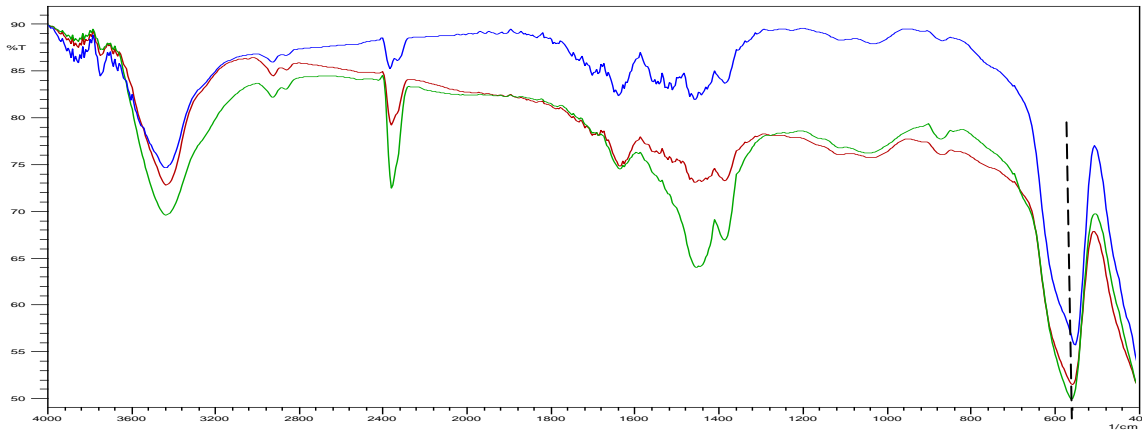


Fig. 6- Infrared spectra of the three samples.

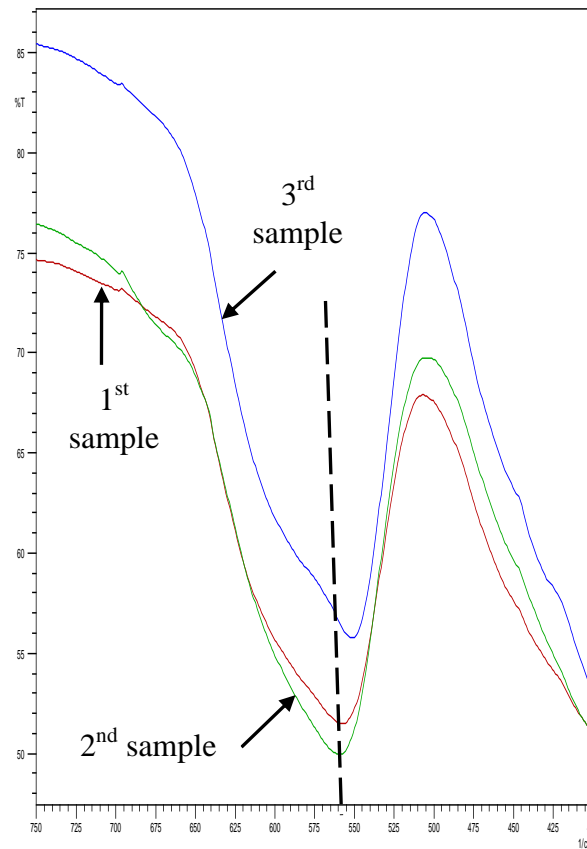


Fig. 7- The Fe -O band of 3 samples .

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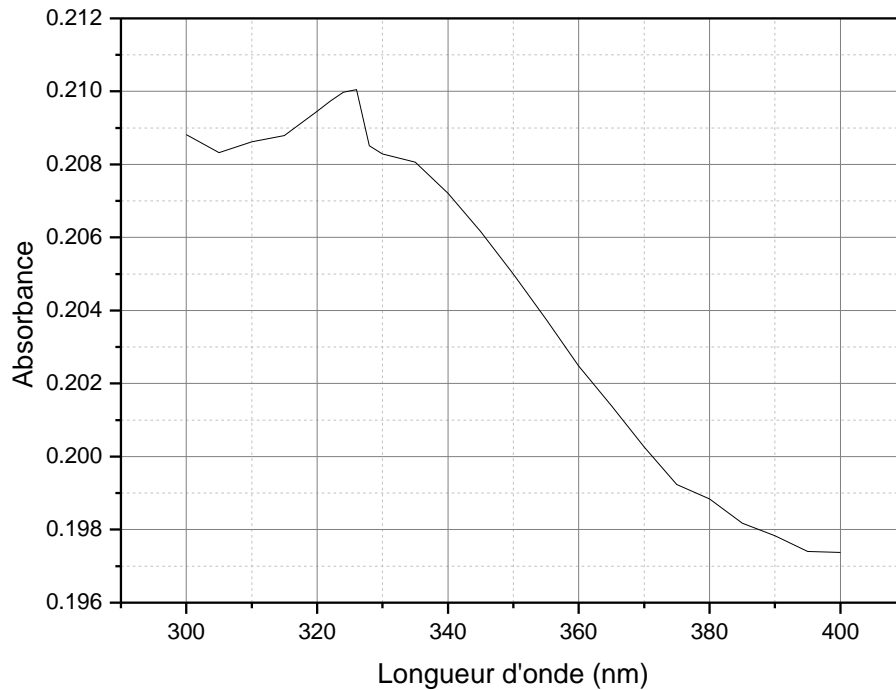


Fig. 8- UV-visible spectrum of the 1st sample before calcination.

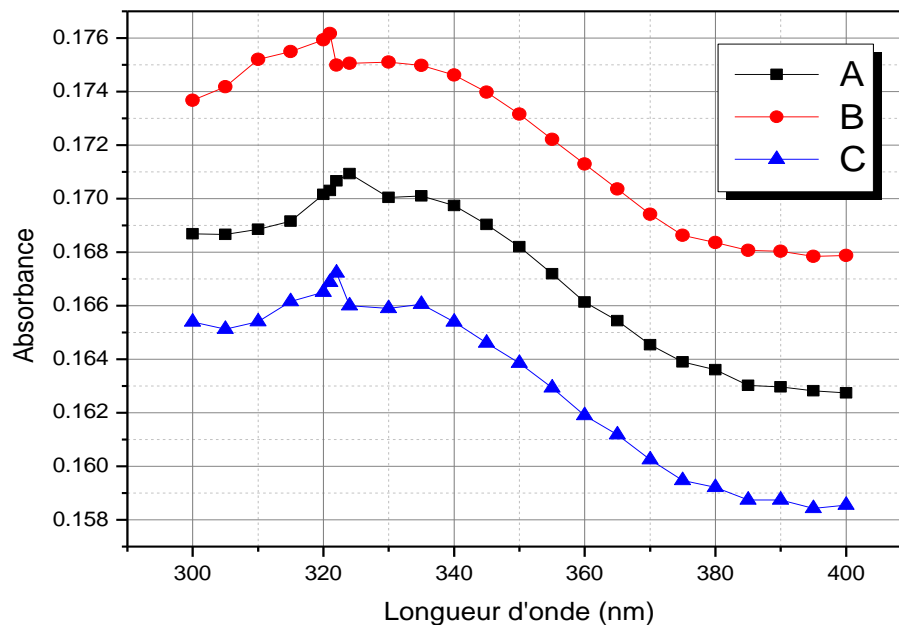


Fig. 9- UV-visible spectrum of : A : 1st sample after calcination, B : 2nd sample, C : 3rd sample.

3.4. Laser particle size

The figure and table below show the particle size distribution for the three samples :

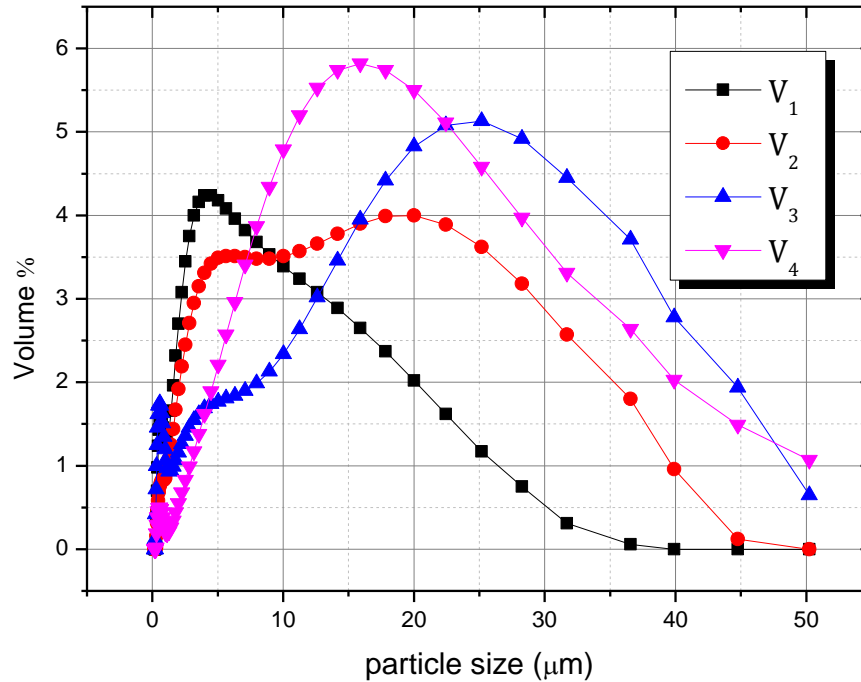


Fig. 10- Particle size distribution of : V₁ : 1st sample after calcination, V₂ : 1st sample before calcination, V₃ : 2nd sample, V₄ : 3rd sample.

Table 1 : Particle size distribution value of the three samples.

Samples	Volume distribution (%)	Particle sizes (μm)
1 st sample before calcination	3.51	7.096
	4	22.44
1 st sample after calcination	1.58	0.632
	4.24	4.74
		5.024
2 nd sample	1.75	0.564
	5.13	28.25
3 rd sample	0.50	0.564
	5.82	17.825

4. CONCLUSION

The objective of this work was studying the synthesis of the mixed oxide LaFeO_3 perovskite by auto-combustion method to reach a structural powder by different fuels without calcination.

The synthesis of the material prepared in this work by auto-combustion method is considered as an exothermic reaction of redox where nitrates are oxidants and the carboxyl group is the reducing agent , but the amine group is the complexing agent of the transition metal so it is the responsible of LaFeO_3 oxide formation without calcination in a short period of time.

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