RJC
http://www.rasayanjournal.com

RASĀYAN J. Chem.
Vol.3, No.1 (2010), 82-86
ISSN: 0974-1496
CODEN: RJCABP

SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE $LaFeO_3$ BY COMBUSTION ROUTE

S. M. Khetre[1], H. V. Jadhav[2] and S. R. Bamane[*3]
[1*]Dahiwadi College Dahiwadi,Tal.- Man, Dist-Satara, Maharashtra, India
[2]Kisanveer Mahavidhyala, Wai, Satara, India
[3]Dr. Patangrao Kadam Mahavidhyala Sangali , India.
E-Mail:sanjaykhetre@gmail.com

ABSTRACT

Orthorhombic structure perovskite $LaFeO_3$ nanocrystalline with size ~27 nm were prepared by glycine combustion method. The prepared $LaFeO_3$ nanocrystals were characterized by TG-DTA thermal analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic fluorescence microscopy (AFM) and Brunauer Emmett Teller (BET) nitrogen absorption. The $LaFeO_3$ nanocrystals are more attractive in the field of catalytic application and process can be applied to prepared more other oxide nanocrystals such as $LaCrO_3$, $LaMnO_3$ etc.
Keywords: Perovskite, $LaFeO_3$, Nanocrystals, combustion method.

INTRODUCTION

The number of catalysts used in modern chemical industries are based on mixed oxide including perovskite oxide ABO_3 where A is a rare earth element, B is 3d transition metals remain predominant[1] perovskite oxides crystals can have broad applications in advanced technologies such as catalysts, oxide fuel cell, chemical sensors, magnetic materials, electrode materials[2-4].Most important perovskite oxides, LaFeO3 nanocrystals is a very known materials[5-6] The $LaFeo_3$ has been synthesized by many methods such as wet chemical method, co-precipitation, microemulsion method, solid state reaction, mechanochemical solid reaction, thermal decompositions of heteronuclear complex, microwave and combustion synthesis.[7-12] The paper has been shown that nanocrystalline perovskite powder can be prepared by using combustion synthesis method of glycine nitrate process.[13] And this provides another possible method to produce nano $LaFeO_3$ particles in a simple way. In this paper, we examine the synthesis of $LaFeO_3$ nanocrystals via the glycine combustion method.

EXPERIMENTAL

Polycrystalline $LaFeO_3$ was synthesized by the combustion synthesis method using glycine as fuel (organic fuel). All chemical reagents were analytical grade and used without further purification. Stoichiometric quantity of solid mixture of one mole reagents i.e. La $(NO_3)_3$ nH_2O (purity 98.5%), Fe $(NO_3)_3$ $9H_2O$ (purity 98.5%) and two mole of glycine (purity 99.5%) were mixed together in a flat Pyrex disc. The solid were stirred for five minutes (clear solution was obtained). Solution formed was evaporated on hot plate in temperature range 80-90^{0}C gives thick gel. The gel was kept on a hot plate for auto combustion and heated in the temperature range of 160-180^{0}C. The nanocrystalline $LaFeO_3$ powder was formed within five minutes. This powder was grind in the mortal pestle and used for the characterization.

Instrumentation

The thermal decomposition process of the synthesized powder was investigated by simultaneous thermogravimetric and differential thermal analysis (TG-DTA) using Universal V2 .4f TA instruments to

900^0 C with heating rate 10^0/min. in static air and Al_2O_3 as a reference. X-ray diffraction XRD measurements were performed on Philips Analytic X-ray B.V. (PW-3710 Based Model) Advanced X-ray diffraction using Cu Kα 1.5406, radiation. The morphology was studied by using scanning electron microscopy (SEM) Model No. JEOL, JSM.6360. The BET surface areas were measured on a Benchman coulter SA 3100 plus instrument using adsorption at 200^0 C.

RESULTS AND DISCUSSION

When reactants were heated at 180°C the reaction proceeds by the mechanism indicated by equation number 1 and 2 give the final product LaCrO3.

$$2C_2NH_5O_2 + (9/2)\ O_2 \rightarrow N2 + 4CO_2 + 5H_2O \quad (1)$$

$$La\ (NO_3)_3 + Fe\ (NO_3)_3 + 4\ C_2H_5NO_2 \rightarrow LaFeO_3 + 8CO_2 + 10H_2O + 5\ N_2 \quad (2)$$

The characterization of $LaFeO_3$ nanocrystals:-

TG and DTA measurements were performed to study the thermal behavior of sol and the related curves are shown in fig1. At the begging synthesized powder was stable up to the temperature 400^0C. this indicate that no loss any material but beyond that temperature there was slight weight gained and this due to the adsorption of nitrogen gas supplied by the instrument. The slight Weight gained was occur up to the temperature 700^0C.After this temperature the weight loss take place due to the desorption of nitrogen gas. At 850^0C temperature the powder is stable. This weight loss and weight gained is very negligible. This weight change was in the range of 0.002 % only. These indicate that the synthesized powder was almost stable from the begging.

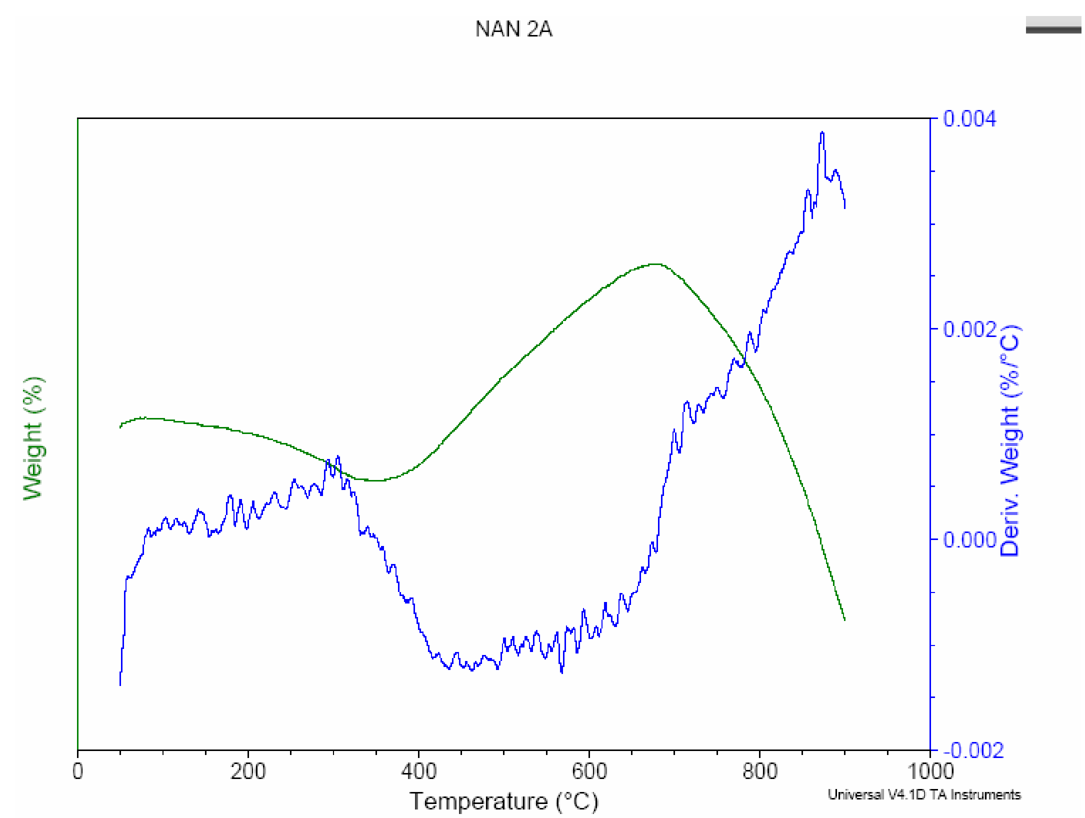

Fig.-1:TG-DTA curve of LaFeO3 as synthesized powder.

Material characterization

X-ray diffraction XRD measurements were performed on Philips Analytic X-ray B.V. (PW-3710 Based Model) Advanced X-ray diffraction using Cu Kα 1.54056, radiation. The XRD pattern shown in the fig. (2).The XRD pattern shows that the product is pure perovskite oxide $LaFeO_3$ with an orthorhombic structure. The diffraction data are good agreement with JCPD card of $LaFeO_3$ (JCPDS No.37-1493). The average crystalline size of $LaFeO_3$ spinel powder was estimated with the help of Scherrer's equation $t = 0.9\lambda/\beta cos\theta\beta$, where t is the thickness of the crystals (in angstroms), λ the X-ray wavelength and θβ the Bragg angle β is the integral breath that depends on the width of the most predominant peak at 100% intensity =1.54056 A^{o}, the wavelength of the Cu Kα source and θ is the Bragg's angle at which the peak is recorded. The average particle size 0f nanocrystalline $LaFeO_3$ was ~27 nm. $LaFeO_3$ nanocrystals are more attractive in the field of catalytic application. The surface morphology was studied by using scanning electron microscopy (SEM) Model No. JEOL, JSM.6360. The SEM images of LaFeO3 are shown in fig.3 (a), (b) and (c) as synthesized powder, at 510^{0}C and 850^{0}C temperature respectly. The SEM images reveal the product is a low density, loose and porous material that is favorable to a catalyst application. The particle size was also calculated from atomic fluroscence microscopy (AFM) recorded at 450^{0}C, 800^{0}C and 1000^{0}C temperatures.AFM images are shown in fig.4. Particle size increases as the temperature increases. The BET surface areas were measured on a Benchman coulter SA 3100 plus instrument using nitrogen adsorption at 200^{0} C. the surface area of the synthesized powder was 2.5 m^2/g.

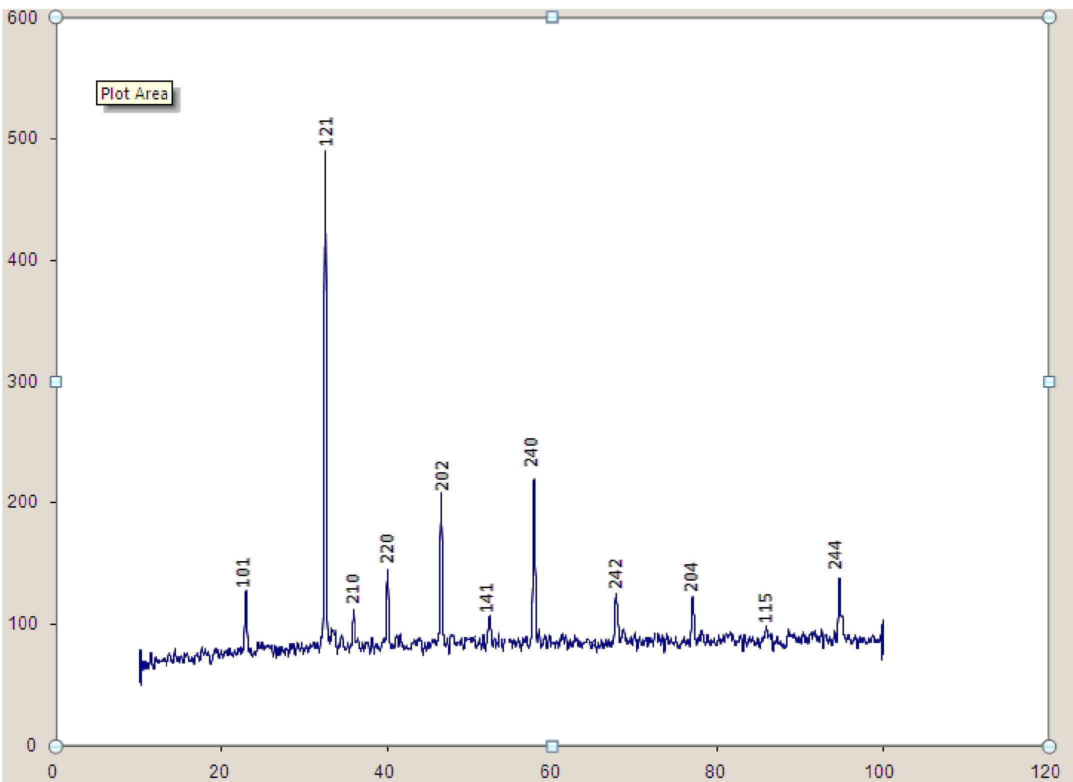

Fig.-2:XRD pattern of LaFeO3 as synthesized powder

REFERENCES

1 M.A. Pena, J.L.G. Fierro,*Chem.Rev.,***101**,1981(2001).
2 Qiwn Zhang, Fumio Saito, *J. Mater. Sci*., **36,** 2287(2001).

3 S.Nakayama, *J. Mater. Sci* , **36,**5643(2001).
4 Frank. J. Berry, Xigolin J, Ramon Gancedo, Josef. Marco, *Hyperfine interact*,**156/157** ,335(2004).
5 Wenjun Zheng, Ronghonum, Dingkum Peng. Guangyao, *Meng. Mater Lett*. , **43**,19 (2000).
6 Z. Yang, Y. Huang, B. Dong, H. L. Li, *Appl. Phys*, A,10 (2005)
7 J. J. Liang, H. S. Weng, *Ind. Eng. Chem.Res.* ,**32**,2563(1993).
8 Q. W. Zhang, E. Satio, *J. Mater. Sci.* ,**36** ,2287(2001).
9 Kuiying Li, Dejun Wang Fengqing Wu, Tengfeng Xie, Tiejing Li, *Mater. Chem.Phys.* ,**64** ,269(2000).
10 W. J. Zheng, R. H. Liu, D.K.Peng, G.Y.Meng, *Mater.Lett.,* **43** ,19(2000).
11 M.Kakihana, *J.Sol.Gel.Sci. Technol.*, **6** ,7(1996).
12 J.X.W.Qi, J.Zhou, Z.X.Yue, Z.L.Gui, T.Li , *Ceram.Ind.,* **29,**347(2003)
13 A.E.Giannakas, A.K.Ladavos, P.J.Pomonis,*Appl Catal.B.Environ.,***49,**147(2004).

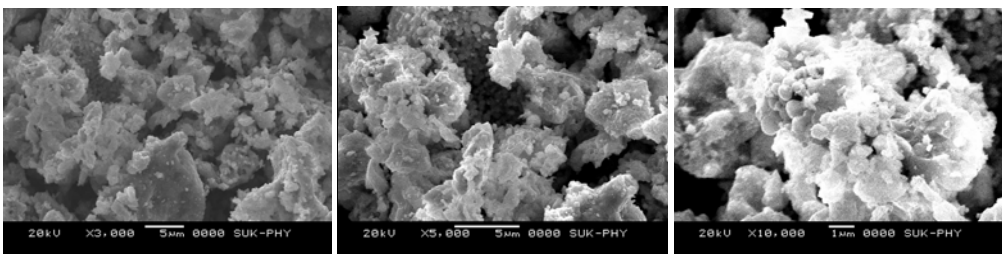

Fig.-3(a) :SEM images of $LaFeO_3$ as synthesized powder

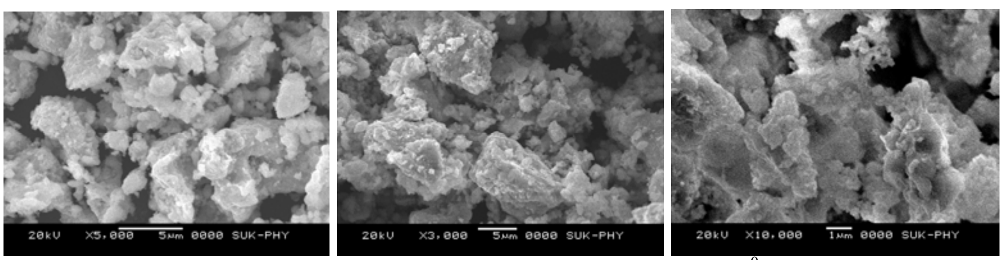

Fig.-3 (b): SEM images of $LaFeO_3$ powder at 510^0C.

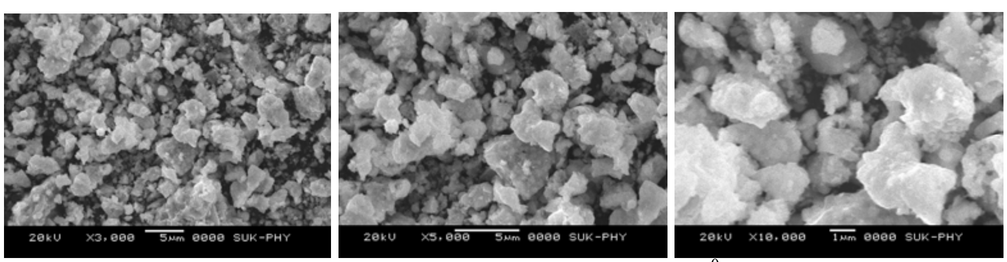

Fig.-3 (c): SEM images of LaFeO3 at 850^0C.

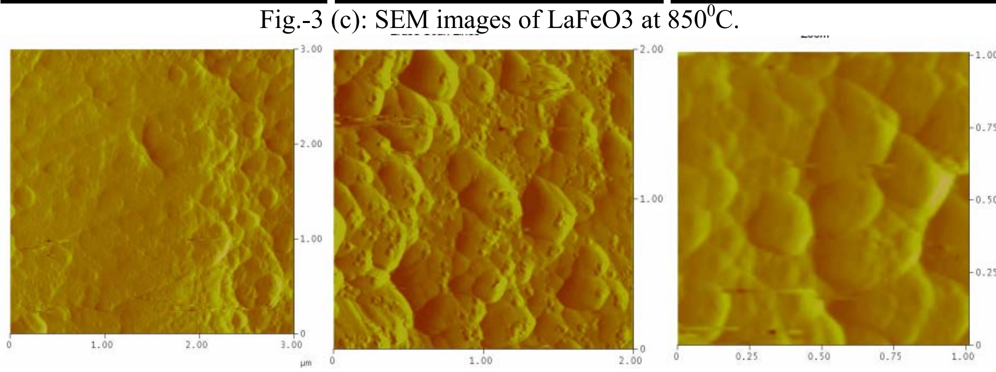

Fig.-4:AFM images of LaFeO3 heated at 450^0C, 800^0C and 1000^0C

CONCLUSION

The following conclusions can be made from the present experiment.

1. $LaFeO_3$ can be simply synthesized chemically by glycine combustion method.
2. The obtained $LaFeO_3$ crystals with fine particle size of nanometer give a relatively high value of surface area of about 2.5 m^2/g.
3. This method can be also applicable for other complex oxides such as $LaCrO_3$, $LaMnO_3$ and we foresee that the $LaFeO_3$ nanocrystals are more attractive in the field of catalytic application and process can be applied to prepared more other oxide nanocrystals.

(Received: 13 September 2009 Accepted: 25 October 2009 RJC-453)