International Journal of Interdisciplinary Innovative Research &Development (IJIIRD)
ISSN: 2456-236X
Vol. **06** Special Issue **01** | **2021**

Solution Combustion Synthesis of Nano-Crystalline La0.67 $(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ Powder & Its Characterizations

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ABSTRACT

Alkali earth metal doped lanthanum manganite have applications as cathode material in modern solid oxide fuel cells and as low field magneto resistance materials. A large scale production of such materials is thus needed by a low cost method which consumes minimum time and is least energy extensive. Many methods of preparation like solid state synthesis, amorphous citrate process, freeze drying, spray, sol-gel, nitrate method and self propagating high temperature synthesis etc. have been reported by various researchers.

We have synthesized $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ ***by a solution combustion method using a unique combination of oxidant and fuel by using stoichometric mixture of*** La_2O_3, $SrCO_3$, $CaCO_3$, $Mn(CH_3COO)_2.4H_2O$. ***Maintaining stoichometry in such compounds is a challenge due to the hygroscopic nature of oxides and carbonates. Therefore the oxides and carbonates must be pre-heated at proper temperature and for a definite time to get rid of the moisture.***

In the present work, we have undertaken the study of effect of preheating the initial reactants on the stoichiometry of above materials synthesized by a solution combustion method. The material has been characterized by XRD, SEM, EDX etc. which has confirmed the formation of highly homogenous nanoparticles of average size 100 nm. EDX results shows better stoichiometry of the material synthesized by preheating the oxides and carbonates.

Keywords: ***magnaite, solution combustion, SOFC, magnetoresistance***

1. INTRODUCTION

When an oxidizer & the fuel are intimately mixed in proper ratio and proper temperature is provided an exothermic reaction takes place with the evolution of heat light and ash. this is called “combustion synthesis” If the initial reactants are used in the solution form it is called Solution Combustion Synthesis

Fig-1 fire trangle

Fuel, Oxidizer and ignition temperature are the basic Components of the fire triangle while heat, light and ash are the important products that emerge from it there are several method for preparation of $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ Like Fuel Pechiney Method, Freeze-drying Method, Spray Pyrolysis, Glycine nitrate Method, Solution Combustion Synthesis. We used a solution Combustion synthesis for preparation of $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ Solution Combustion Synthesis has advantages over other methods like high productivity, low energy consumption and use of simple production facilities but it's a lengthy process [1]the combustion process has several advantages in terms of simplicity, cost effective less, energy saving, purity and homogeneity [2]the combustion-derived powders have narrow size distribution with average agglomerate particle size in the range of 0.5-5µm [3]the film particle nature of the combustion-derived powder is attributed to the low exothermic of the combustion reaction and evolution of large amount of gases (NH_3,H_2O,CO_2),which help to dissipate heat thereby preventing the oxides from sintering [4]the combustion synthesis as a preparation process to produce homogeneous, very fine crystalline, average agglomerates, multi component oxide ceramic powders without the intermediate decomposition and/or calcining steps has attracted a good deal of attention

2. Synthesis of $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$

Polycrystalline sample of $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ was synthesized from stoichometric mixture of La_2O_3, $SrCO_3$, $CaCO_3$, $Mn(CH_3COO)_24H_2O$, the powders so formed were heated in air at various temperature up to 950 ^{0}C followed by slow cooling to room temperature in Al_2O_3 crucible the sample where heated for several hours with frequent intermediate grinding there are several chemical reaction takes place during the synthesis of $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$

For lanthanum oxide & Nitric acid

La2O3+6HNO3 = 2La(NO3)3+3H2O
For Strontium carbonate & Nitric acid
SrCO3+2HNO3 =Sr(NO3)2+H2O+O2
For calcium carbonate & nitric acid
CaCO3+2HNO3 = Ca(NO3)2+H2O+CO2
Final reaction in presence of manganese acetate
$(0.67)La(NO_3)_3 + (0.33)(0.25)Sr(NO_3)_2 + (0.33)(0.75)\ Ca(NO_3)_2 + Mn(CH_3COO)_2\ 4H_2O = La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ + other product gases

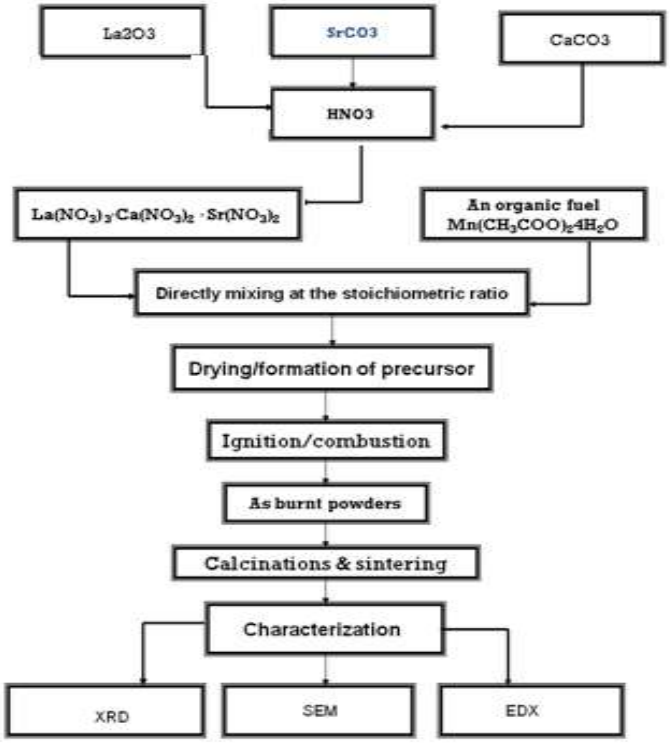

Fig-2 Flow chart for the preparation of sample

3. CHARACTERIZATION

The characterization of the $La_{0.67}(Sr_{0.25}Ca_{0.75})_{0.33}MnO_3$ powder is done by using EDX and SEM of sample

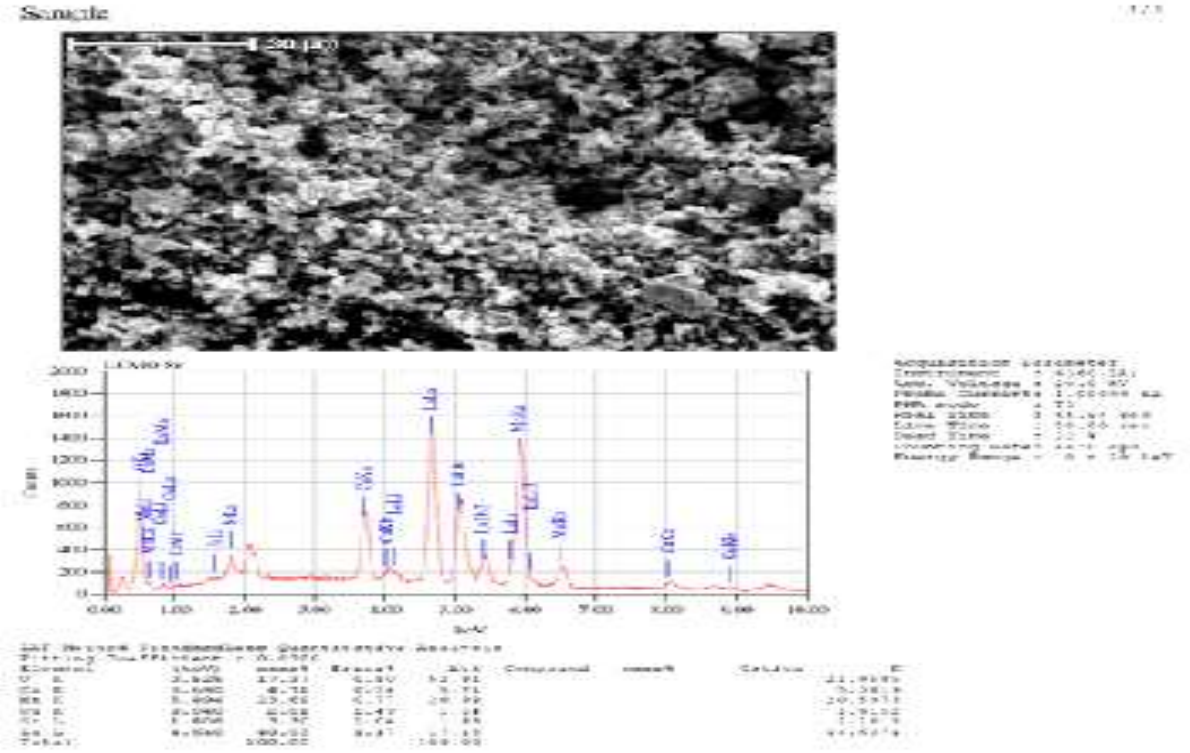

Chart-1 EDX result of material powder using heated reactants

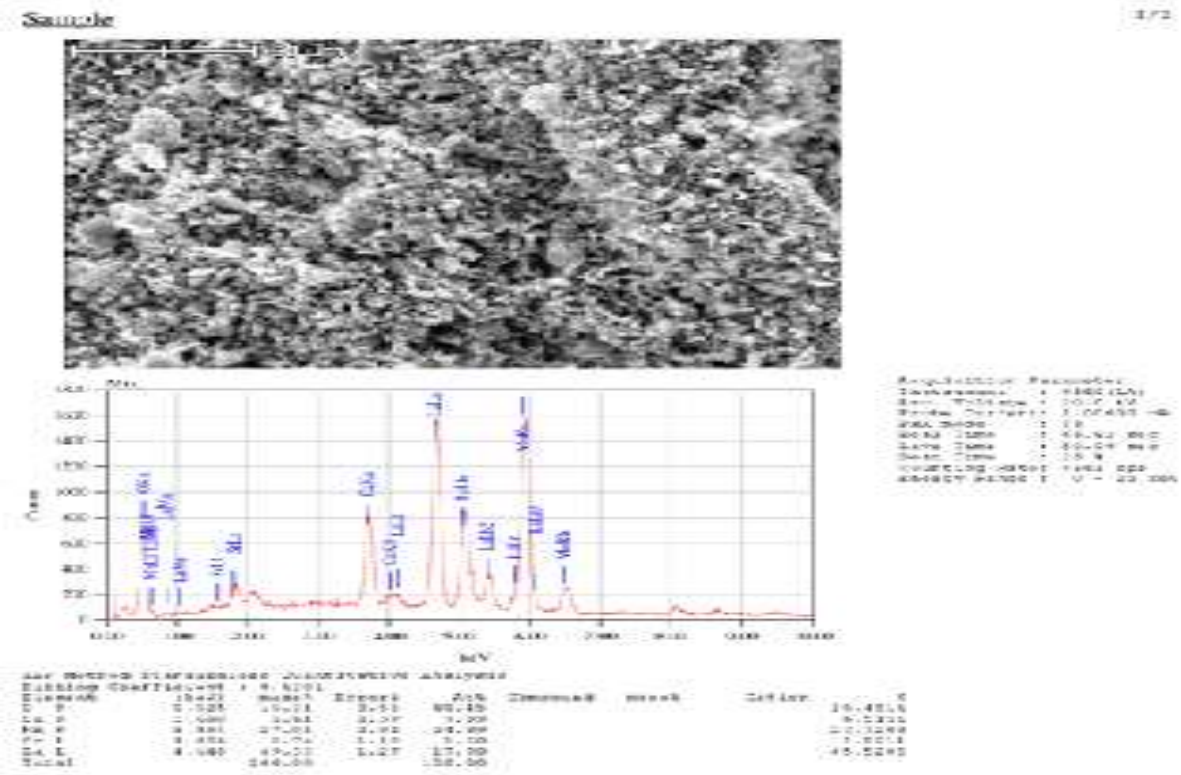

Chart-2 EDX result of material powder without heating initial reactants

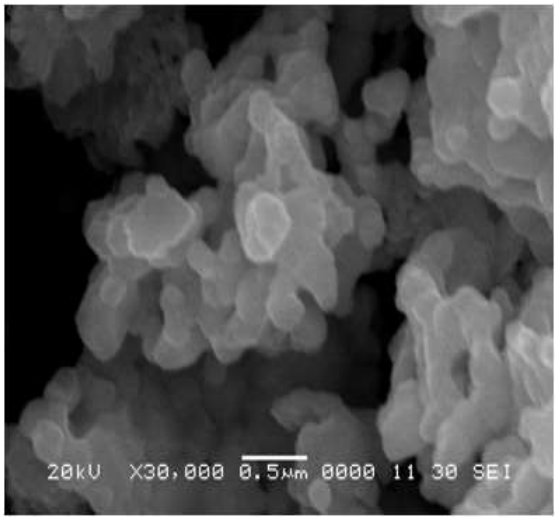

Image-1 SEM result of material powder using heated reactants

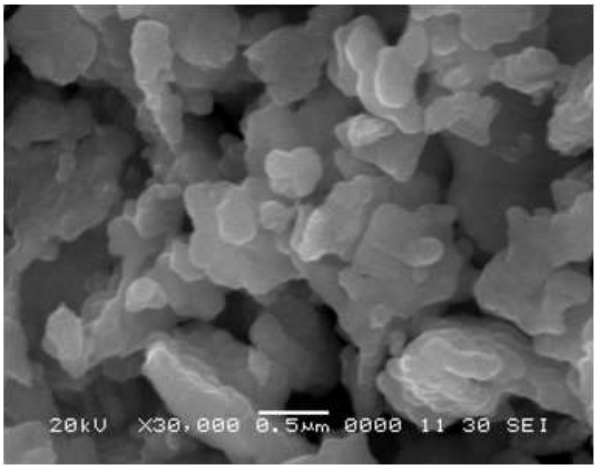

Image-2 SEM result of material powder without heated initial reactants

4. RESULT AND CONCLUSION

La_2O_3, $SrCO_3$, $CaCO_3$, $Mn(CH_3COO)_24H_2O$

1. On heating the initial reactant at various temperatures there was a considerable weight loss which suggested the presence of the moisture in the compounds.

On heating La_2O_3 at 900 ^{0}C for one hr. there was a weight loss of the 18.87%

On heating $SrCO_3$ at 200 ^{0}C for 4 hr there was a weight loss of 2.3%

On heating $CaCO_3$ at 200 ^{0}C for 4 hr there was a weight loss of 2.3%

2. stoichiometry required was Mn:La :Ca:Sr = 1:0.67:0.2725:0.08 stoichiometry of the resulting compounds obtained was Mn:La :Ca:Sr = 1:0.81:0.27:0.08 when the initial reactant were heated stoichiometry of the resulting compounds obtained was Mn:La :Ca:Sr = 1:0.72:0.28:0.06 when the initial reactant were not heated

3. The average particle size of the powder was near to 100mm

5. CONCLUSION

The desired stoichiometry of the resulting compound was not obtained accurately by both the methods used the probable reasons for this can be following

1. Manganese acetate was not heated but it is quite possible that Manganese acetate could also gain some moisture from the environment

2. furnaces used for heating the reactant powder were not very reliable when the powders where heated at 900 0C there temperature could not be maintained properly so it is quite possible that the Lanthanum oxide was over heated which might have resulted in the partial decomposition of oxide

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