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Review: Perovskite Structure, Characterization, & Synthetic Methods

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Abstract

The perovskite compounds are interesting molecular material for various applications. The structure may have ions of different size and charge reflecting variety of composition according to structural requirement of target application fields like,electric, magnetic, optic catalytic, sensor and solar cell etc. The reviewfocuses on the synthetic aspects of mesoporous perovskite oxides compounds and also on metal halide perovskite nano crystals (MHP NCs). The reactions conditions, starting materials and process of manufacture have been discussed.The various factors related toeffects on synthesicstrategy and their effecton properties of perovskites have also been taken under consideration. The structure of perovskite material and the different characterization techniqueshavealso been discussed, e.g. powder XRD, single crystal XRD, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and thermal study likethermo gravimetric analysis (TGA), differential scanning calorimetry (DSC),differential thermal analysis (DTA)and FTIR etc. have taken under considerations. The hard and soft template synthesis colloidal method, electrostatic spinning, hydrothermal method for perovskite oxide synthesis have been discussed. Hot injection method and ligand assisted reprecipitation method (LARP) for pervoskite halides have also been discussed.

Keywords : Perovskite, synthesis, characterization, colloid, template, hot-injection.

1. Introduction

Nowadays the perovskite materials are the area of interest among the scientists. Although the first perovskite material has been synthesized in 19th century but the actual research on these compounds in various application purposes e.g. electronic systems [1,2], light emitting devices, florescent materials, magnetoresistant uses, catalysis [3], sensors[4] and bio sensors[5], fuel cells[6], solar cells[7], photo chromic, electro chromic applications[9] are now in use. Keeping in mind the structural aspects, skills, methods, techniques of characterization as well as various synthetic routes of different types of perovskite oxide and perovskite halide nano materials have been overviewed. The fundamental procedures with the merits and demerits of these routes have also been taken under consideration.

2. Metal-oxide perovskite

During the synthesis of perovskite the oxygen stoichiometry change in the structure is reflected in the tuning of properties, at different electronic and functional level. Several methods for synthesis have been reported.

2.1 Structure of metal oxide perovskites

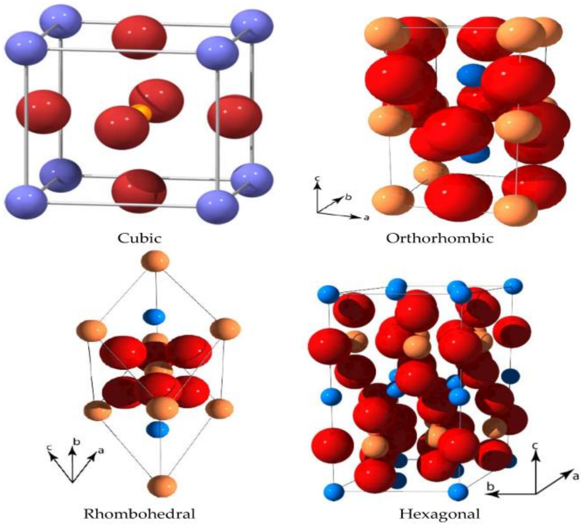

Fig-1 Structure of perovskite material

Before proceeding to synthesis, it is essential to understand the general structure (Fig-1) of these oxides. The structure of the metal oxide pervoskite family have the general formula ABO_3 in which A stands for alkaline or rare earth metal and B stands for transition metal. The twelve fold octahedral cage structure of oxygen around A type cation and six fold coordinated octahedral oxygen atom surrounding around B type cation generally observed with flexibility due to structural distortions variations in the size, nature and valency of both the cations may be reflected in oxygen deficiency or oxygen excess and also in redox sensitive sites in the perovskite. The great deal of the physical and chemical properties changes observed due to following reasons:

(a) Change in structural parameters of crystal lattice.

(b) Variation at compositional level by change in metal cations A and B.

(c) Morphological level changes like layer structure or double perovskite. The mesoporous nano structured materials have attracted the researchers very much because of their large surface area and more number of active sites within them.

2.2 Materials and methods

The synthetic procedures and the various parameters related to synthesis are discussed in this part.

2.2.1. Method based on the soft template

The synthesis process when mediated with the assistance of some soft template systems, the procedure became more facile. The different types of soft templates are:

(i) Non-ionic surfactants (e.g. co-polymer pluronic P 123)[10]

(ii) Cationic surfactants (e.g. alkyl tri methyl ammonium compound)

(iii) Anionic surfactants (e.g. long chain sulphonic derivative)

(iv) Organic soft template

2.2.2. EISA (Evaporation induced self assembly)

This process involves following step wise [11] procedure.

(i) Homogenous sol preparation of soft template and precursor compound.

(ii) Solvent evaporation and coating for increase the concentration above the CMC (critical micelle concentration) with self assembly of inorganic processor material in micelle.

(iii) An equilibrium maintenance between the surrounding environment and the meso structured film.

(iv) Thermally assisted pre-treatment before the removal of template detection and final network formation[12,13].

Limitations of this method:

Temperature susceptibility for stability and crystallinity of meso porous structure are the limitation of this method. In the modified EISA method, by the use of chelating agent [14-16]like urea, citric acid, acetic acid the homogenous solution can be prepared more easily. In these methods the perovskite oxides $La_{1.7}Ca_{0.3}N_{1-x}Cux$, $SrTiO_3$, $BaTiO_3$ etc. have been synthesized.

Merits of modified EISA method

(i) Highly ordered structure.

(ii) Wide surface area.

(iii) Enchance volume

(iv) Reduced pore size distribution.

2.2.3 Method based on hard template/nano costing/repeated template technique

As the name implies, in this method the template [17-19]which the been removed after the synthesis can be used in next repeated synthesis. The perovskite $LaFe_xCo_{1-x}O_3$ with KIT-6 template and $LaFeO_3$ or $LaNiO_3$ with SBA-15 template has been prepared by hard template method. There are nonporous in nature. General method involves the following steps:

(i) The hard templates (SBA-15, KIT-16 and MCM-48 or mesoporous carbons (MK-1, MK-3)[20-22] and homogenous solution of metal salt precursor are mixed with suitable chelating agents (like citric acid) in proper stoichiometric ratio.

(ii) The next step is calcination.

(iii) Removal of silica using solution of sodium hydroxide or hydrogen fluoride in water.

Demerits of the method

Filling of silica requires longer time due to different types of interactions such as H-bonding, Van der waals interactions between silica and metal ion.

The functionalization of silica with some functional group like $CH_2=CH_2$ are in use. Double solvent with hydrophobic solvent method can provide better results [23].

2.2.4 Colloidal template method

The colloidal crystal templates (e.g. organic polymer spheres like polystyrene PS, poly methyl meth acrylate (PMMA) are used for preparation of nano porous perovskite. Three common procedures are generally used[24].

(i) The colloidal crystal which is already prepared previously is used and infiltrated with the precursor.

(ii) The template sphere of uniform size and shape are used for co-precipitation.

(iii) Colloidal spheres coated with coating layer forming composite spheres are used.

$La_{0.6}Sr_{0.4}FeO_{3-8}$, E_4FeO_3, $Eu_{0.6}Sr_{0.4}FeO_3$, $LaMnO_3$, La_2CuO_4, $La_{0.6}Sr_{0.4}MnO_3$[25-30] are prepared by this method using organic surfactants and chelating agents e.g. glycine, citric and, F-127, L-lysine and polyethylene glycol etc.

Draw-back of the method :

(i) Sometimes due to very thin wall as compare to pore size the three dimensional porous structure may collapse during the removal of template.

(ii) This methods requires enough time for the synthesis.

2.2.5 Electrostatic spinning method

Several km long with 100nm diameter nano fibers can be prepared using this method. High voltage electro spinning of the precursor and polymer/polymer alloy/polymer loaded with nano particles are used and after the drying process precursor nanofiber associated with polymer and metal cations of definite stoichiometry can be synthesized. Calcination is used to removes the polymer and pure material of perovskite oxides synthesized using this method are : $LaCoO_3$, $La_{0.75}$ $Sr_{0.25}$ MnO_3, $LaFeO_3$, $La_{0.5}$ $Sr_{0.5}CoO_3$-x, $BiFeO_3$, La0.5 $Sr_{0.5}$ $Co_{0.8}$, $Fe_{0.2}$ O_3, La0.6, Sr0.4, C01-x, Fe_xO_{3-5}, and $PrFeO_3$[31-38].

Use of organic compounds like citric acid etc. is favourable to maintain good dispersion during the synthesis of provskite oxides.

2.2.6 Hydrothermal Technique :-

This method generally used for pervoskite based titanates [39-43]e.g. $PbTiO_3$, $BaTiO_3$, $SrTiO_3$, $LaFeO_3$, and $LaNiO_3$. This technique involves TiO_2 or titanates in layered protonated template form and spheres of TiO2 can be prepared with different strucureal parameters like pore size, porosity etc. Ostwald ripening or in situ crystal formation involved during the synthesis Kirkendall effect has involved the polymer template e.g. poly vinylchloride, poly vinyl alcohol and additives Na_2SiO_3. $9H_2O$, have also been reported to involve in synthetic routes by this method.

The advantage of this method is good yield, low energy consumption with low air pollution effect.

3 Metal halide perovskites

3.1 Structure of metal halide perovskite

In the general formula of perovskite ABX_3, X is halide in this group of compounds Atom A is generally larger than B. In cubic structure cation B has 6 fold coordination and surrounded octahedrally B anions. The cation A has cuboctahedrally coordinated with 12 fold coordination.

3.2 Metal halide perovskite nanocrystal synthesis

The photovoltaic and opto-electronic features of perovskite metal halide nano crystal (MHPNCs) attracted researcher very much. The synthetic strategies used for their synthesis are as follows :

3.2.1 Colloidal method

The properties of MHPNCs can be controlled by using specific synthetic strategies. There are :-

(i) “Top down” (ii) Bottom up approach.

In top down technique macroscopic solid reactants are used and fragmented either by means of mechanical way like ‘ball-milling’ or the “chemical method” while the “bottom up” method requires gas liquid phase reactions [44].

PSCI: Polar solvent controlled ionization

3.2.2 Hot injection (H.I.) method

In H.I. method [45] rapid injection of a precursor material is added to the solution of ligand, remaining precursor materials and solvent of high boiling points.

The NCs with narrow range of size range are formed due to difference in the nucleation and growth stages. Several factors like reaction time temperature of injection of precursors and precursor surfactants ratio are important factors for control over size and shape of nano crystal (Table-1).

S.No.	MHPNCs	Specification
1.	$CsPbX_3$	Cs-oleate injection PbX_2 salt solution temperature 40-200 ^0C Solvent : Octa- decane, carboxylic acide and amines.
2.	$MAPbX_3$ (X=Br, I and Cl)	$PbCl_2/PbBr_2$ or PbI_2, methyl amine, solution temperature 140-200^{0}C oleyl amine and oleic acid capping ligands.
3.	$CsPbBr_{1-x}I_x$	Anti solvent : iso-propanol, n-butanol or acetone
4.	$CsPbX_3$ (X=Cl, Br or I)	Three precursor method PbO_2, NH_4X and are mthyl ammonium salt are used instead of PbX_2.
5.	$FAPbX_3$	Synthesis with alkyl ammonium halide precursor
6.	Cs_2AgBrX_6	Silyl halides are used. Method is useful in the synthesis of Pb free metal halide perovskite

Table : 1 Details of synthesis for different metal halide perovskites

The control [46] of shape and size of metal halide, perosvskite nano crystals can be done as follow :-

(i) Varying the chain length of carboxylic acid.

(ii) Temperature variation during the process.

3.3 LARP or ligand assisted re precipitation :-

In this approach the perovskite nano crystals is prepared[by dissolving the precursor salts in a solvent and after getting equilibrium concentration the equilibrium state of the reaction is disturbed either by changing the temperature or by addition of any other polar solvent or co-solvent so that the precipitation occur continuously to get the equilibrium state again.

$$\begin{matrix} Reactantsalts \\ in \\ (goodsolvent) \end{matrix} + organicliqud \xrightarrow{BadSolvent} Reprecipitation\&perovskitenanocrystals$$

4 Characterization :-

Perovskites are characterized by power XRD or single crystal XRD. The TGA, DTA and DSC techniques are used for study of thermal stability of the material. Scanning election microscopy (SEM), transmission electron microscopy (TEM) are utilized for the study surface characterization, the FTIR (Fourier Photo Electron Microscopy) are used for complete identification of perovskite materials (Table-2).

S.No.	Technique [49,50]	Information
1.	XRD (X-ray diffraction) (a) Powder XRD (b) Single Crystal XRD	Porticle size, lative parameters and lattice structure
2.	SEM (Scanning electron microscopy)	Morphology and surface structure analysis.
3.	TEM (transmission electron microscopy)	Helps for selection of preparation method, reaction condition etc.
4.	HRTEM (High resolution transmission electron microscopy)	Morphology and crystal structure characteristics.

5.	BET (Brunauer-Emmett-Teller)nitrogen adsorption	Surface structure analysis helps in selection of synthetic routes, reaction condition for synthesis to get desired shape size and properties.
6.	Thermal study (i) Thermal gravimetric (ii) Differential scanning calometry (DSC) (iii) Differential thermal analysis (DTA)	Thermal stability of the compound analyzed.
7.	FTIR (Fourier transform infra-red)	The bonding behavior and structural information can be obtained from FTIR.
8.	XPS (S-ray photoelectron spectroscopy	Structural composition can be obtained from XPS analysis

Table-2 various characterization of perovskites.

5. Result and discussion

Metal halide perovskite nano crystals especially in case of lead halide perovskite the 3D crystal structure have been reported but due to toxkicity of Pb several attempts made to replacement of Pb with other group IV metals are reported with the composition Cs_2SnI_6oD and 2D structure, Cs_4PbX_6 are and $CsPbX_5$ "perovskite related" structural composition and designated as zero dimensional and two dimensional structures respectively.

In the synthesis of MPH NCs the size and shape of NCs and properties exhibited by them are interrelated and the control on the structural parameters can be governed by : (i) temperature regulation during the reaction (ii) use of ligands of different nature, varying the chain length of amine or carboxylic acid. (iii) Use of ligand of different nature.

The synthesis of metal oxide pervoskite can be done by any of the methods using soft, hard and colloidal template electron spinning or hydrothermal method. All these methods have their own advantages and disadvantages. Soft template method is best for large scale synthesis but pure size can't be controlled and have low surface and requires expensive soft template. On the same time, the hard template method provides uniform pore size pervoskite with high surface area but in low yield and restricted to limited oxides. Colloidal method is although useful in the synthesis of perovskite oxides of large pore size but some times collapse of pores may occur and it requires long process time with low yield. Similarly, electros pining technique provide pore sizecan not be controlled and surface area is also low hydrothermal method is bet in many respects but can be used for limited perovskite oxide only.

6. Conclusion :-

The perovskite oxides and perovskite metal halide nano crystals both have numerous applications in multifarious field like catalysis, sensors, fuel industrial application, opto electronics, gyro magnetic uses and photovoltaic applications.

Therefore, it is very important to understand the synthesis and characterization of these compounds in a systematic manner. In this review an attempt has made together information about the synthetic aspects of both the nano porous metal oxides and metal halide perovskite nano crystals. In reaction condition and their effect on the overall product synthesis has been taken under deep consideration the use of organic ligands pore formation surfactants and precursors are essential for the synthesis of nano porous metal oxide perovskites, the metal halide pervoskite nano crystals can be synthesized by using proper temperature regulation, good organic acid and amine with metal halide precursor in suitable solvent or solvent mixture recently, the stability, surface structure and product yield are under consideration of researchers for getting better results with perovskite materials.

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Competing Interests

I am working on the chromophoric metal complex for DSSC. I have introduced with perovslite materials. I am working on a project on soler cell funded by 'Research and Development Program', U.P., India and also appreciated by 'RAFM-2022', organized by Department of Physics, ASRD College, Delhi, India. I am thankful to both of these authorities.

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