SYNTHESIS OF LUMINESCENT MATERIALS BY NUMEROUS METHODS

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A B S T R A C T

Phosphors are Luminescent materials that emit light (from infrared to ultraviolet) by excitation due to external impetus. The incident energy may be in any form of high energy beam of electrons, photons or electric field and can then be re-emitted in the form of electromagnetic radiations. For certain phosphors, these radiations fall in the visible region of electromagnetic spectrum.

This paper aims to provide the recent advances in the synthesis of such luminescent materials. Method of preparation of phosphors matters a lot for the behaviour of luminescent materials; organic and inorganic as it has made the drastic change in quality of materials which made revolution in light industry. There are various methods of preparation of such luminescent materials like; solid state reaction, wet chemical, precipitation and co-precipitation, combustion, simple chemical route, sol gel method, molten salt synthesis, etc. which are studied to get advanced and smart materials.

Keywords: Luminescent materials, Synthesis methods, Phosphor, Light industry

1. Introduction of Luminescence and Luminescent materials

1.1 Luminescence

The features of engineers and scientists who are working on different chunks of the artificial and synthetic spectrum are often rather specialized and just non overlapping. However, one of the most popular aspects that modern research in material science starts from preparation and characterization of novel materials for various luminescence applications. Luminescence is an interdisciplinary subject as it is applicable in various fields like chemistry, physics, medical science, biological science, forensic science, geology, material science, engineering technology, etc. Current research is characterized by solid interaction among other branches of solid state and between different areas of luminescence using organic and inorganic materials [Sawde, Bhake, Patil, Dhoble&Moharil, 2018]. Both experimental as well as theoretical approaches have been made for it.

Luminescence means emission of light by appropriate materials when they are relatively cool. Luminescence is defined as the emission of radiation of the light by bodies, which is in excess of that attributable to the black radiation and persist for longer duration than the period of electromagnetic radiation after the excitation stops. Traditionally, luminescence is classified as fluorescence and phosphorescence. The word "Luminescence" which includes both fluorescence and phosphorescence was first used by EilhardtWiedemann, a German Physicist; in 1888 material science, engineering technology, etc. If the emission from the material persists for a shorter duration ($<10^{-8}$ s) then it is called as fluorescence while phosphorescence persist for quite longer duration (up to several seconds). The classification of luminescence [Sawde, Bhake, Patil, Dhoble&Moharil, 2018]based on the source of excitation and their various applications are summarized in following Table 1.

Table 1 –Classification of Luminescence based on the source of excitation and their various applications.

Type of luminescence	Excitation source	Applications
Photoluminescence	Photons	Fluorescent lamps, PL-LCD, Plasma display, LASERs, LSCs, Paints, inks, Upconversion material
Thermoluminescence	Ionizing radiations	Radiation dosimetry, Archeological and geological dating, Forensic science
Cathodoluminescence	Electrons	TV set, FED, Oscilloscope, Monitors, storage tubes, Flying spot scanners, Radars
Electroluminescence	Electric field	LEDs, EL displays, Diode lasers
Radioluminescence	Ionizing radiations such as X-rays or Gamma rays.	X-ray imaging, Scintilators, dosimetry
OpticallyStimulated luminescence	Visible Photons	X-ray radiography, dosimetry
Lyoluminescence	Chemical reaction	Detectors, Analytical devices, Lyoluminescencedosimetry
Chemiluminescence	Chemical reaction	Analytical chemistry
Bioluminescence	Biochemical reaction	Analytical chemistry
Triboluminescence	Mechanical energy	Luminescent sparkles by crushing Sugar crystals
Sonoluminescence	Ultrasound	

1.2 Luminescent materials

Luminescent materials are substances called phosphors that emit light (from ultraviolet to infrared) under external energy (impetus) excitation over and above that due to black-body emission. For certain phosphors, this radiation occurs in the visible spectrum. The incident energy may be in terms of high energy photons, electrons or electric field. It can then be re-emitted in the form of photons of electromagnetic radiation. In order to categorize the radiation in the visible spectrum, the Commission Internationale de I'Eclairage (CIE) standardized the method by which color is quantified by establishing the CIE chromaticity diagram in 1931 [Blasse, &Grabmaier, 1994].

The topic of this research areas include the development of phosphors and nanophosphors for various optical applications such as making lamp phosphor, solid state lighting, CR tubes and TV screens, electroluminescent lamp and display panels, LED's, detectors for X-ray imaging, thermoluminescent dosimeters, scintillation detectors, laser crystals, dosimetry of ionizing radiations, paints, inks and whiteners, solar concentrators and in chemical and bio-chemical analysis and medical diagnosis[Miejerink, &Blasse, 1991] .This subject continues to have a major technological role for humankind in the form of applications such as organic and inorganic light emitters for flat panel and flexible displays such as plasma

displays, LCD displays, and OLED displays[Sawde, Bhake, Patil, Dhoble&Moharil, 2018]. Luminescent Materials and Applications describes a wide range of materials and applications that are of current interest including organic light emitting materials and devices, inorganic light emitting diode materials and devices, down-conversion materials, nanomaterials and powder and thin-film electroluminescent phosphor materials and devices.

2. Experimental: Role of Synthesis of phosphors

Phosphors used for various applications are generally in powder form. However, the synthesis of phosphors is not always simple and straightforward as it appears from their chemical formula. There are many constraints in phosphor synthesis while considering it for particular application. The constraints mostly are due to the physical and chemical properties of initial ingredients used, limitations and conditions of particular method of synthesis and the nature of application.

In the development of these materials, chemistry plays an important role [Blasse, 1989]. For example, there is influence of preparation on the luminescence properties of these phosphors. There is also influence of crystallinity and particle size distribution and morphology on these properties. Recently, therefore, researchers have shown more interest in preparing such materials of nano-meter dimensions. There are various methods for the synthesis of luminescent materials, looking at the chemical formula of most of the phosphors one may feel that the synthesis of the luminescent materials should be straight forward as the host materials are well known[Mimani, &Patil, 2001]. However, in practice the synthesis of the phosphors with desired characteristics can be quite tricky. The difficulties arise as one has to consider several aspects such as the incorporation of the activator at the desired sites, elimination of the unwanted impurities, specific grain size and morphology suitable for the application, cost of production, batch homogeneity and reproducibility, etc.

3.Novel methods used for preparing the phosphors

During the recent years several methods have been used for simplifying the phosphor synthesis. These can be broadly called as "Novel Synthesis". The novel syntheses not only provide simpler methods for phosphor preparation but they often provide the control over particle size and morphology as well. Principles of these novel methods and their application to preparation of a variety of class of phosphors for use in various applications are very interesting. Many novel methods have been suggested for the synthesis of these phosphors such as; solid state reaction and Solvothermal, wet chemical, precipitation and co-precipitation, combustion, simple chemical route, sol gel method, molten salt synthesis, etc. which are studied to get advanced and smart materials. Some are discussed below in brief.

3.1Solid state diffusion method and Solvothermal synthesis-As good as Twin methods as temperature reliant:

In Solid state diffusion method, the constituents are made to react through diffusion process. The temperature is just enough to have adequate diffusion to complete the reaction in laboratory time without melting the constituents. Reaction time and the temperature bear a sort of reciprocal reaction. It may not be possible every time to lower the latter sufficiently. e. g. several aluminates cannot be formed even by the solid state diffusion at temperatures below 1400 ºC.

A conventional solid-state reaction can be followed as shown in Fig.1-- Starting hosts of purity higher than 99.9% were used as raw materials for preparation of the phosphors. The reagents were weighed in stoichiometric proportions; thoroughly ground, mixed in anagate mortar, and calcined at 1000 ^{0}C for 5 hours in a covered alumina crucible. Subsequently, the calcined materials were re-ground in the mortar and re-fired at 1000 ^{0}C for 5 hours under the reductive ambience of 5% H_2/95% N_2 to reduce $^{1+}$ion to $^{2+}$ ions, respectively[Kim, Choi, & Jun, 2009].

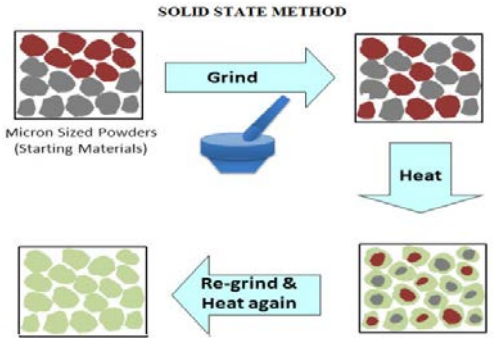

Fig. 1 - Solid State method

To prepare some materials and its precursors, solvothermal method is used which presents some advantages for several reasons. Solvothermal processes can be defined as chemical reactions or transformations in a solvent under supercritical conditions or near such a pressure-temperature domain. The specific physico-chemical properties of solvents in these conditions can, in particular, markedly improve the diffusion of chemical species.

Another open method is melting the constituents together thus obtaining the reactants in liquid phase without using a solvent. Yet again, there can be several difficulties in following this procedure. The melting points of the constituents like oxide are usually very high. The problem of obtaining a container that will withstand such high temperature without reacting with the constituents can prove unsolvable. The constituents themselves may have vastly differing melting points so that one of the constituents may vaporize well before reaching the melting point of the other**.**

Solvothermal synthesis is a method for preparing a variety of luminescent materials such as semiconductors (may be luminescent), ceramics, metals and polymers. It is comparable to the hydrothermal route (in which the fusion is conducted in a stainless steel autoclave), as shown inFig. 2.The only difference is that of the precursor solution which may not be usually aqueous (though, this is not always the case in all uses of the expression in the scientific literature). The process involves the usage of a solvent below moderate to high pressure (typically between 1 atm and 10,000 atm) and temperature (typically between 100°C and 1000°C) that facilitates the interaction of precursors during synthesis. If water is used as the solvent, the method is called "hydrothermal synthesis." The synthesis under hydrothermal conditions is usually performed below the supercritical temperature of water (374°C). The process can be used to prepare much geometry including thin films, bulk powders, single crystals, and nanocrystals. The method can be used to prepare thermodynamically stable and metastable states including novel materials that cannot be easily formed from other synthetic routes. Over the last decade, a majority (~80%) of the literature concerning solvothermal synthesis has focused on nanocrystals; therefore, this review will highlight some advances in nanocrystalline, solvothermalsynthesis **[Gersten**, 2005].

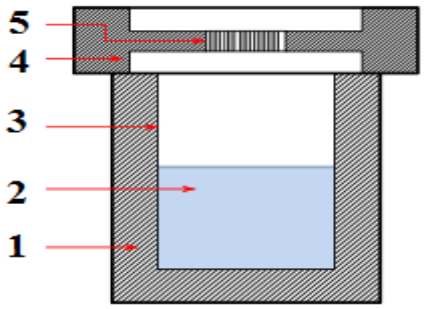

Fig. 2 - Schematic diagram of solvothermal synthesis setup: (1) stainless steel autoclave (2) precursor solution (3) Teflon liner (4) stainless steel lid (5) spring.

Using the solvothermal route one gains the benefits of both the sol-gel[Oliveira, Schnitzler, Zarbin, & Aldo 2003] and hydrothermal routes[Andersson, Österlund, Ljungström, &Palmqvist, 2002]. Thus solvothermal synthesis allows for the precise control over the size, shape distribution, and crystallinity of metal oxide nanoparticles or nanostructures. These characteristics can be altered by changing certain experimental parameters, including reaction temperature, reaction time, solvent type, surfactant type, and precursor type.Solvothermal synthesis has been used in laboratory to make nanostructured titanium dioxide[Xie, & Shang, 2007], graphene[Choucair, Thordarson, & Stride, 2008], carbon[Hu, Ma, Cheng, Liu, &Bao, 2002], chalcogenides[Li, Chen, Wang, &Proserpio, 1999] and other materials.

3.2MSS: Molten salt synthesis of compounds

Molten salts (also known as ionized salts) are a class of compounds that are solid at room temperature and pressure, and made liquid by heating. MSS is a proven alternative route to the synthesis of a wide range of compounds (such as TiO_2, ZrO_2 and Al_2O_3) [Sheikh, 2016].
MSS of compounds involve mixing reactant (s)

with an excess of salt, in a crucible, under a modified atmosphere or under air. The crucibles are made from either alumina (Al_2O_3), zirconia (ZrO_2) or SiO_2, depending upon the selection of the molten salt, to avoid any interaction with the container materials [Sheikh, 2016]. The crucible is then heated in a furnace above the melting point of the salt, where the reactants dissolve and react with in solution. During this process, the salt behaves as a solvent and/or as a reactant and upon completion, the molten salt is cooled and washed with H_2O, alcohol or mechanically separated to obtain the final product. The hydroscopic salt (such as nitrates), is pre-treated (such as drying under vacuum) to eliminate any H_2O attached to the salt [Kimura, 1991].

3.3Wet chemical method

Recently, the evaporation method is known as Wet chemical method.Wet chemical synthesis is an ideal technique used to produce fine, chemically homogeneous and pure, single-phase powders in the synthesized condition. This procedure is attractive because of its capacity to yield products at 80 - 100°C only.The molecular motions and hence the chemical reaction may proceed very swiftly in the liquid state. One can summarize the advantages of the Wet chemical synthesis are low cost synthesis technique, as very low temperature is needed (nearly 80 – 100°C), power saving method due to low temperature requirement and simplest method for the homogeneity of constituent salts as compared to solid- state diffusion method, combustion method and sol gel method etc. [Bhake, Nair, Zade, &Dhoble, 2016].

For preparing the conventional polycrystalline mixed compound phosphors, the wet chemical method is used, i.e. constituent hosts with stoichiometric ratios are dissolved in doubly distilled deionized water and then evaporated till the mixture becomes anhydrous. The compound, in its powder form obtained is evaporated at 80°C for 8 hrs. The dried samples were then slowly cooled to room temperature. The resultant polycrystalline mass was crushed to fine particles in a crucible. The powder is used in further characterization.

3.4Combustion synthesis

Combustion synthesis is also known as Self-Heat Generating Process (SHGP). In most of the novel methods, lower reaction temperatures are achieved by obtaining the reactants in fine form. An ingenious way of lowering the operating temperature is to use the heat generated in exothermic chemical reaction itself for the synthesis.

One of the earliest discovered SHGP was self-heat propagating process. In this process, the reactants are thoroughly mixed and pressed to form a bar. One end of the bar is heated to high temperature using some sort of flame. Once the exothermic reaction starts, it propagates across the length of the bar. Temperatures as high as 7000°C have been obtained in such reactions to produce nitride and carbide materials.

Exothermic reactions between metal nitrates and fuels such as urea are exploited in the combustion synthesis. Nitrate to fuel ratio can be adjusted to vary the temperature generated. Highest temperatures are attained when this ratio is such that the oxidising and reducing valencies of the oxidisers and fuel are balanced. Combustion synthesis is also known as propellant synthesis, thermally explosive synthesis, auto-ignition**,** etc. The conventional way of preparing aluminises is using the solid-state diffusion. Al_2O_3 is used as the aluminium source. It has melting point of more than 1800°C and the melting points of most of the other oxides are still higher. For this reason, even the solid-state diffusion has to be carried out at temperatures well above 1200°C and in many instances above 1700°C. Aluminates can be most conveniently prepared by the combustion synthesis involving metal nitrate and urea.

3.5Simple chemical synthesis

The general method adopted [Awad, 2012] for synthesis of especially organic chelate (compounds) is known as Simple chemical synthesis.As per the name suggested it is simple, low cost and low time consuming method. In it, desired amount of drug (such as aspirin, coumarin, etc.) is dissolved in alcohol and is de-protenated by adding NaOH solution for obtaining a basic solution. The metal salt solution is prepared by dissolving corresponding metal salt mostly nitrate or chloride in double distilled water. Both the solutions are mixed with vigorous stirring. As soon as both solutions are mixed; chelate precipitate appears in the solution. Precipitate obtained is then filtered using good quality filter paper. The filtrate is dried under the bright lamp for several hours to drive away the moisture.

The powder precipitate is then separated and sealed in glass bottle for its study. During the reaction pH is maintained at 10 in basic region. The salt to aspirin ratio is taken in 1:2 proportions. The mixed chelates may be prepared by dissolving the moieties in 99:1, 95:5, 90:10, 80:20 ratio, etc. and then carrying out the reaction with metal salts as described above.

3.6Precipitation and Co-precipitation

Some phosphors were synthesized by wet chemical method assisted by precipitation technique as in Fig. 3 which is described below. The stoichiometric amounts of hosts and dopant are taken and thoroughly mixed in double distilled water separately and solution is stirred well on magnetic stirrer to make the transparent solution. Then solution of next starting host is added drop by drop which gives a thick milky white color precipitate with water immediately. The obtained product is washed several times to separate the by-products with distilled water and allowed to dry on fine quality filter paper for 24 hours. All the pure and doped samples of phosphor can be prepared in this way. Completely dried samples were in powder form but needed a little crushing to yield fine powder for measurements[Bhake, Zade, Nair, &Dhoble, 2015].

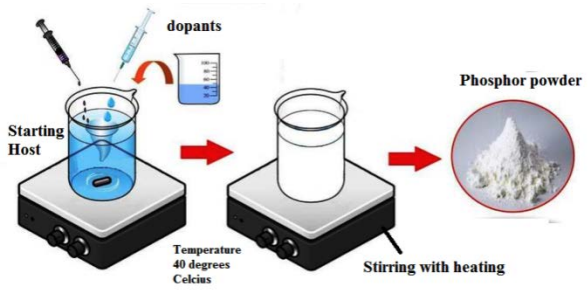

Fig. 3 - Synthesis of Phosphor by Precipitation technique

Co-precipitation is the carrying down by a precipitate of substances normally soluble under the conditions employed. Coprecipitation is one of the most appropriate techniques for the incorporation of trace RE elements into nanophosphors with a narrow size distribution, during recrystallization and solid solution formation. In comparison to other techniques, the coprecipitation technique does not require stringent reaction conditions, costly equipment or complex procedures. Sometimes, crystalline nanophosphors can be obtained directly by co-precipitation without a calcination step or post-annealing process.

3.7Sol-gel Method

The Sol-gel method is an important technique which is frequently used not only for the synthesis of nano particles but also to fabricate ceramic or glass materials in a wide variety of forms.The different forms of materials produced by this method are ultra-fine or spherical shaped powders, thin film coatings, ceramic fibres, microporus inorganic membranes, monolithic ceramics and glasses or extremely porus aerogel materials.The interest in sol–gel processing can be traced back in the mid-1800s. Sol–gel research grew to be so important that in the 1990s more than 35,000 papers were published worldwide on the process [Brinker, & Scherer 1990][Hench, & West, 1990][Klein, 1994].

Thesolis a name of a colloidal solution made of solid particles (approximately equal to 0.1to1mm) few hundred nm in diameter, suspended in a liquid phase. The gelcan be considered as a solid macromolecule immersed in a solvent. Sol-gel process consists in the chemical transformation of a liquid (the sol) into a gel state and with subsequent post-treatment and transition into solid oxide material. Following four steps [Thejokalyani, &Sawde, 2015]are used for the synthesis of nanomaterials using sol-gel method:

PREPARATION OF SOL: The starting materials used in the preparation of sol are usually inorganic metal salts or metal organic compounds such as metal alkoxides.

PREPARATION OF GEL: In a typical sol-gel process, the precursor is subjected to a series of hydrolysis and polymerization reaction to form a colloidal suspension known as gel.

DRYING AND PURIFICATION:In this step, heat treatment is given to the gel emulsion to dry it up to the level necessary for further processing.The reaction mixture is refluxed and washing is done by suitable agent several times

before separating the powder by centrifuging system or spraying it by spin coating unit to desired shape or size.

PRODUCT FORMATION:

Using different techniques, we can fabricate the particles or films of desired shape and size. For example, the centrifuge action is used to obtain the nano particles of zicronia while the thin films can be produced on a piece of substrate by spin coating or dip coating.

In this way, firstmethod allows inorganic oxides and/or their immediate precursors to be produced from simple alkoxides or chelates via low temperature hydrolysis and condensation reactions. In sol-gel synthesis one may obtain the products in reasonable time at lower reaction temperatures.

The second sol-gel technique involves ceramic synthesis by hydrolysis of metal-organic compounds, in particular metal alkoxides. For preparation of multicomponent oxides, alkoxides are mixed together in alcohol and a component unavailable as an alkoxide is introduced as a salt, for example an acetate, so that the resulting solution has the required ceramic composition.

The sol-gel method is based on the phase transformation of the sol obtained from metallic alkoxides or organo-metallic precursors. This sol, which is a solution containing particles in suspension, is polymerized at low temperature in order to form a wet gel. The solvent is removed by drying the gel and the next step is a proper heat treatment [Rajaeiyan, &Bagheri-Mohagheghi, 2013]. **[**From Wikipedia, the free encyclopedia]

4. Conclusion

There are various methods of preparation of luminescent materials like; solid state reaction and Solvothermal**,** wet chemical, precipitation and co-precipitation, combustion, simple chemical route, sol gel method, molten salt synthesis, etc. which are studied to get advanced and smart materials. Method of preparation of phosphors matters a lot for the behaviour of luminescent materials; organic and inorganic as it has made the drastic change in quality of materials which made revolution in light industry.

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