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SYNTHESIS OF NANOPARTICLES THROUGH COMBUSTION TECHNIQUE

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ABSTRACT

The recent development and trends in the manufacturing of nanophosphors are discussed. Nanophosphors have been extensively investigated during the last decade due to their application potential for various high performance and novel displays and devices.A simple combustion method has been tried for the preparation of nanoparticles and is practiced in 65 countries. This is an effective, ecofriendly and low-cost method for production of various industrially useful nanomaterials. Number of breakthroughs in this field have been made due to the extensive research carried in last five years. The extensive research carried out in last five years emphasized the SHS capabilities for materials improvement, energy saving and environmental protection. Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to characterize the prepared product.

KEY WORDS: Nanoparticles, combustion synthesis, X-ray diffraction,

INTRODUCTION

There are a number of methods for preparing the nano-crystalline materials viz. inert gas condensation, physical vapour deposition, laser ablation, chemical vapour deposition, sputtering, molecular beam epitaxy etc. In addition, there are a number of solution-chemistry routes also. Among the available solution-chemistry routes, the combustion technique is capable of producing the nano-crystalline powders of oxide ceramics at a lower calcination temperature in a surprisingly short time. During the combustion primarily N_2, CO_2 and H_2O are evolved as the gaseous products. Therfore, carbon and hydrogen are considered as reducing electrons with the corresponding valencies 4+and 1+ whereas oxygen is considered as an oxidizing element with the valency 2- and nitrogen is assumed to have valance of zero.

Metal oxides doped with lanthanides have been received much attention for the last decades. Such materials usually show enhanced photoluminescent properties compared to the conventional sulphide phophores. In general, suitable materials for phosphorus must provide both, the high luminescence and chromatic resolution, what can be achieved by using ceramic-compounds doped with lanthanide ions. Beyond the recognized importance of these elements,due to unique features derived from the electronic configurations, the oxide host plays a fundamental role in the luminescent properties of such components.(HengleinA,1989)

Combustion technique can be described as a facile, low cost and useful route for the preparation of the powders with a high purity in nanometric scale. The theory of this technique is based on the concept of driving chemistry,where the volatile moleculesi.e. CO_2, H_2O and N_2 are released due to combustion reaction and a stable product remains. In fact,the initial materials are heated by microwave energy,prompted by burning of the fuel and nitrate groups in throughout mixture.

MATERIALS AND METHODS

The various products were prepared using the corresponding metal nitrates and a suitable fuel. Some of the examples are as follows:

Synthesis of yttrium oxide(Y_2O_3): The nano-crystalline Y_2O_3 was prepared starting yttrium nitrate $Y(NO_3)_3.6H_2O$ and glycine (NH_2CH_2COOH), which were mixed in the required molar ratios in a minimum volume of deionized water to obtain transparent aqueous solutions. Different combinations of fuel and oxidants were

used so as to tailor the powder properties. The molar concentration of the oxidants (yttrium nitrate) was kept constant at unity and the fuel concentration was varied as 1.0,1.66 and 2.0 M to give three different solutions. These solutions after thermal dehydration (at 80^0C on a hot plate to remove the excess solvent) gave highly viscous liquids. As soon as the viscous liquids were formed, the temperature of the hot plate was increased to 250^0C. At this stage, the viscous liquids swelled and auto ignited, with the rapid evolution of large volumes of gases to produce voluminous powders. Since the time for which the auto-ignition exists is rather small (<10 sec),to remove traces of un-decomposed glycine, nitrates, if any, the powders obtained after auto-ignition were calcined at 600^0C for 1 h to obtain pure and well crystalline yttria powders.

Synthesis of $NiMoO_4$ nanoparticles:
All the reagents were purchased from Merek Company and used without further purification.A stoichiometrical amounts of $Ni(NO_3)_2.6H_2O$ and $(NH_4)6Mo_7O_{24}.4H_2O$ were mixed, put into a domestic microwave oven and reacted to each other in the presence of urea as a fuel. The program of instrument was adjusted on the power of 900 W for a few minutes. The obtained product was collected and analyzed.(A. Kaddouri et al., 1998)

Synthesis of $Ca_{3-x}Al_2O_6:xEu^{3+}$ nanophosphors:
Tricalcium aluminate doped with trivalent europiumions was synthesized by the microwave-assisted combustion method using a mixture of fuels. The samples with molar composition Ca_{3-x} $EuxAl_2O_6$ ($0 \leq x \leq 0.1$) were obtained without further thermal treatment. Both fuels, urea(CON_2H_4) and glycine($C_2H_5NO_2$), as well as the metal nitrates $Al(NO_3)_3.9H_2O$ and $Ca(NO_3)_2.4H_2O$ were purchased from VETEC.

The synthesis of nanophosphors is currently a hot topic in the field of combustion synthesis(CS).The range of nanophosphors-based materials prepared by simple combustion synthesis SCS is listed in Table –A.

Table-A-Different SCS-phosphor materials, fuel used, particle size and application.

Phosphor Material	Fuel Used	Crystalline size from XRD(nm)	Application
Y_2SiO_5:Ce, Lu_2SiO_5:Ce, Gd_2SiO_5:Ce	Hexamine	20-80	Detection of ionizing radiation and dense scintillators
$SrAl_2O_4:Eu^{2+},Tb^{3+}$	Urea;urea+boric acid flux	50-80	Long lasting phosphorescence materials
Eu^{3+} activated $YAlO_3$ And $LaAlO_3$	Amonium nitrate+ urea	80	Red Phosphorous
$Gd_3PO_7:Eu^{3+}$	Glycine	40	Red phosphor
$CaWO_4:Eu^{3+}$	Citric acid Ammonium Nitrate	50-100(TEM)	Fluorescent lamps, coloured lighting for advertisement industries and other optoelectronic devices.
$MAL_2O_4:Eu^{3+}$, R^{3+} (M=Sr, Ba,Ca, R=Dy, Nd and La)	Urea	21-40	Long persistent luminescent material
Pr^{3+},Tm^{3+} doped $Gd_3Ga_5O_{12}$	Urea	30-00	Magneto optical films and materilas for solid syate lasers
$Y_2O_3:Eu^{3+}$	Sucrose	30-50	Red emmiting phosphor used in CRT screens,plasma displays, fluorescent lamps

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RESULTS AND DISCUSSION

The salient results of each system are as follows:

(a) Yttrium oxide represents a novel prototype for high temperature material for electrochemical applications.The synthesis of yttria powder with controlled powder characteristics is also of practical importance to get dense sintered product at a lower sintering temperature.(Bhargava R. N.et al.,1996).

Table1-Powder properties of nano-crystalline yttria as a function of oxidants-to-fuel ratio

Powders	O/F ratio	Observed flame temperature (^{0}C)	No.of moles of gases	Crystallite Size(mm)	Surface area(m^2/g)
1	1:1.0	No flame	13.00	8	165
2	1:1.66	1440	15.42	30	57
3	1:2.0	1200	17.50	25	147

These observations explain the finest crystalline size and the highest surface area (165m^2/g).Thus, it could be concluded that there are two competing effects that govern the final powder properties viz. the high exothermicity and flame temperature, which causes the powders to coarsen whereas the evolved gases tend to improve the powder properties.

(b) In the synthesis of $NiMoO_4$, the powder XRD measurements were performed using STOE diffractometer with monochromatised Cu Kα radiation (ʎ = 1.5418 cm^{-1}) FTIR spectra were recorded on a Shimadzu-8400S spectrometer in the range of 400-4000 cm^{-1} using KBr pellets. The UV-Vis absorption study was carried out in the wavelength range of 190-800 nm at room temperature on a UV-Vis spectrometer(Shimadzu UV-1700). FT-IR spectrum, XRD pattern and energy-dispersive X-ray analysis(EDX) energy-dispersive X-ray analysis (EDX) were used toinvestigate the structural characteristics of the synthesized $NiMoO_4$.To study the bonding nature of the resulting product was recorded the FT-IR spectrum. The strong peaks at 946 cm^{-1} with a weak shoulder at 858 and also,the bands at 586 and 414 cm^{-1} are characteristics of α- $NiMoO_4$ structure. These bands can be attributed to the vibrational modes of Mo-O-Mo and Ni-O-Mo in building block of $NiMoO_4$, respectively. XRD pattern indicated a crystalline phase of α- $NiMoO_4$ with the space group of 12/m and lattice parameters of a=9.509Å, b=8.759Å, c=6.667 Å (JCPDS Card No. 33-0948). The sharp diffraction peaks confirmed the formation of a pure crystallinity of monoclinic α- $NiMoO_4$ phase.

(c) In the synthesis of $Ca_{3-x}Al_2O_6:xEu^{3+}$, the crystal structure of the prepared samples was determined by powder x-ray diffraction analysis performed on a diffractometer SHIMADZU model XRD-6000,using Cu Kα radiation and operating at 40kV and 30 mA. The powder diffraction patterns were recorded between 10^0 to 80^0 at steps of 0.02.The morphological characteristics of the powders were analyzed by electron microscopy.The images were obtained on a Scanning Electron Microscope with Field Emission Gun(SEM-FEG) model JEOL 6700F. Photoluminescence(PL) excitation and emission spectra were collected at room temperature on a Fluorolog Horbia Jobin Yvon spectrofluorometer. The photoluminescence emission spectra of the powders exhibits at least five characteristic transitions of the ion Eu^{3+} at approximately 578,591,612,651 and 702 nm. These emission lines can be ascribed to the $^5D_0 \rightarrow {}^7F^0$, $^{7F}{}_1$,$^7F_2$,$^7F_3$ and $^7F_4$ transitions respectively. It is well established that the emission spectrum of trivalent europium ions is strongly influenced by its site symmetry.(Chen L. M. et al., 2004)

Future scope/conclusions:

In recent years, combustion synthesis has not only opened new vistas for the preparation of various novel nanosize oxides and composites, but also succeeded in continuous synthesis method of nanopowders and development of various supported catalysts and coating. As a result,conditions are mature for breakthrough in these areas over the next several years. Promoters of nanotechnology,an approach defined on the basis of a length scale, have done a

greatjob of selling the idea that 'smaller is better'. Similarly, now it is time to commercialize combustion synthesis method by selling the reality of 'simple is smarter and better'.

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