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Synthesis, Characterization and ionic studies of Mo^{6+} ions doped Nanocrystals

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ABSTRACT

The researches on small particles generally known as nanostructures materials have become more familiar during the last two decades in various fields of chemistry and physics. These materials are of major significant, and the technology of their production and use is rapidly into a powerful industry and in applied research. Therefore systematic work has been done to study the effect of dopants of Mo^{6+} ions into the cerium sulphide nanocrystals. In view of the importance of ionic conductivity, Mo^{6+} ion doped and undoped nanocrystals are synthesized by sol - gel method and subjected to UV-visible; Fourier transforms infrared spectroscopy, X-ray diffraction, scanning electron microscopy and transmission electron microscopy study. These nanocrystals are found to be essentially ionic conductors. The conductivity (σ) of undoped 1% and 5% Mo^{6+} ion doped nanocrystals were measured at various temperatures 33^0C - 3300C and at frequency of 100 kHz. The results suggested that the conductivity of undoped nanocrystals at 33^0C is found to be 1.12×10^{-7} Scm^{1-} while at 330^0C is 1.45×10^{-4} Scm^{1-}. The conductivity of 1% of Mo^{6+}ion doped nanocrystals at 33^0C is 6.53×10^{-7} Scm^{1-} and at 330^0C is 4.91×10^{-4} Scm^{1-}. The conductivity of 5% Mo^{6+}ion doped nanocrystals at 33^0C is 8.06×10^{-6} Scm^{1-} and at 330^0C is 7.40×10^{-4} Scm^{1-}. The conductivity of doped nanocrystals increases largely with increasing concentration of Mo^{6+} ion dopants. The results also suggested that conductivity values of Mo^{6+} ion doped nanocrystals are greater than those of undoped nanocrystals at all temperature respectively

Keywords: *Nanocrystals, X-ray diffraction, scanning electron microscopy, Transmission electron microscopy, Conductivity*

I. INTRODUCTION

Nanotechnology offers an extremely wide range of potential applications from electronics, optical communications and biological systems to new materials [1-3]. It is interesting to note that the applications of nanotechnology in different fields have distinct different demands, and thus face very different challenges, which need different approaches. For examples, for application in medicine or in nanomedicine, new instruments to analyze tissues literally down to the molecular level, sensors smaller than a cell allowing to look at ongoing functions and small machines that literally circulate within a human body pursuing pathogens and neutralizing chemical toxins. Due to these reasons there is great interest of the scientific community toward nanotechnology in recent years. Among them, metal sulphide nanocrystals are structurally and technologically interesting materials which have been greater importance than any other nanomaterials [4-5]. The physico-chemical properties of metal sulphides [4] and doped with metals ions [6-7], exhibit an acute dependence on size. These properties are of special importance because these are related to the industrial uses such as sensors, catalysts, absorbents displays, lighting, and lasers [3-4]. A number of dopants of transition metal ions such as Co, Mn, Cu and Fe and rare earth ions such as Tb^{3+}, Eu^{3+}, and Ce^{3+} ions are used in metal oxide and metal sulphide nanocrystals which can lead to an increase of conductivity of nanocrystals [8-11]. It belongs to a class of materials that combines high electrical conductivity with optical transparency and thus constitutes an important component for optoelectronic applications, solid state gas sensor, oxidation catalyst and transparent conductor [12-14]. A survey of literature reveals that no systematic work has been done to study the effect of dopants of Mo^{6+}ions into the cerium sulphide nanocrystals. Therefore, it was considered worthwhile to study the conducting behavior of Mo^{6+}ions doped nanocrystals. In view of the importance of ionic conductivity, open circuit voltage (OCV) studies and structural characterization of some newly synthesized undoped and Mo^{6+}ions doped nanocrystals with various concentrations of Mo^{6+}ions were carried out. The conductivity (σ) value of undoped and Mo^{6+} ions doped nanocrystals were obtained at different temperature while OCV value of undoped and Mo^{6+}ions doped nanocrystals were obtained at 27^0C temperature. In the present investigation the conductivity and OCV of undoped nanocrystals were also measured in order to see the effect of addition of Mo^{6+} ions. The undoped and Mo^{6+}ion doped nanocrystals have been characterized by SEM (scanning electron microscope) and TEM (transmission electron microscope) indicating crystalline size 35 – 80nm. An attempt has also been made to explain the structure of conducting nanocrystals on the basis of IR and X-ray diffraction pattern. Thus, in this paper for the first time we are reporting systematic characterization, electrical conductivity and OCV measurement of undoped and Mo^{6+} ion doped nanocrystals and also explain the effect of change of dopants concentration and their structure respectively. The undoped and Mo^{6+} ion doped nanocrystals are conducting in nature has large application in industries and solid state batteries [15].

2. EXPERIMENTAL

2.1 Materials

Chemicals used for investigation of nanocrystals were all of analytical grade. Ceric ammonium sulphate $(NH_4)_4Ce(SO_4)_4$ $2H_2O$ (Qualigens), Sodium sulphide ($Na_2S.7H_2O$) (Qualigens), ammoniummolybdate, $(NH_4)_2MoO_4.2H_2O$ and Ethylene glycol(CH_2OHCH_2OH) were taken from Ranbaxy for preparing the nanocrystals.

2.2 Preparation of undoped and Mo^{6+} ion doped nanocrystals

Method used for preparing Cr^{3+} ion doped sulphide nanocrystals have been described in a previous publication [1]. Similar techniques were employed for preparation of both undoped and Mo^{6+} ion doped nanocrystals. The undoped cerium sulphide nanocrystals was prepared by mixing 1:1 molar ratio of ceric ammonium sulphate and sodium sulphide in 100ml of distilled water and mixed well with magnetic stirrer for 4 hours and filtered and then refluxed with ethylene glycol at 80^0C to give cerium sulphide nanocrystals. For preparing Mo^{6+} ion doped nanocrystals different amount of 1% and 5% of ammoniummolybdate were taken. In all these cases of preparation three round bottle flask were taken. In one round bottle flask appropriate amount of ceric ammonium sulphate and sodium sulphide in 1:1 molar ratio was taken to prepare control (undoped) cerium sulphide nanocrystals while in other two round bottle flask appropriate amount of ceric ammonium sulphate and sodium sulphide in 1:1 molar ratio was taken and the dopants of 1% and 5% of ammoniummolybdate salt was added in each test tube to prepare the Mo^{6+} ion doped nanocrystals. The reaction mixture in each case was dissolved in 100ml of distilled water and mixed well with help of magnetic stirrer for 4 hours. The pH value of undoped and doped solution was measured with help of pH-5-25cw microprocessor pH/mv meter. The reaction mixture in each case was decanted and refluxed in ethylene glycol at 80^0C to obtain undoped and Mo^{6+} ion doped nanocrystals. The nanocrystals thus obtained were dried in dark and placed in sample tube and stored in desiccator. The undoped cerium sulphide nanocrystals were brown while Mo^{6+} ion doped nanocrystals were yellow. The purpose of the investigation was to study the changes brought about by the introduction of a metal Mo^{6+} ion along with control undoped system which were synthesized simultaneously.

2.3 Characterizations

In order to characterize the undoped and Mo^{6+} ion doped nanocrystals the UV – visible reflectance spectra were recorded on a Shimadzu 160A spectrophotometer. The FT-IR spectra of nanocrystals were run in the range of 4500 - 50 cm^{-1} using KBr discs on a Perkin Elmer FT-IR spectrometer. The XRD patterns of the compound were taken on a Phillips Analytical X-ray B.V. diffractometer using Cu α radiation with 2θ = 5-80^0 from BARC Kalpakkam. Scanning electron microscope (SEM) and Transmission electron microscopy (TEM) images were taken on Phillips Model – CM200 operating voltages 20 - 200 kv with resolution 2.4A^o from IIT Bombay.

2.4 Electrical conductivity measurements

The electrical conductivities of undoped, 1% and 5% Mo^{6+} ion doped nanocrystals were measured by four probe a.c. impedance method [16] by using HIOKY 3550 LCR Tester. In this method pellets of the samples under investigation were prepared by giving pressure of 5t by hydraulic pressure. After measuring the thickness of the pellet with the help of a screw gauge, silver epoxy was coated on the both side. The pellet was allowed to dry completely before measuring the electrical impedance with the help of the LCR Tester. The variations of the conductivity with the temperature of undoped and doped nanocrystals were studied in the range of 33- 330^{0}C at 100 kHz frequency by using equation (1).

$$\sigma = G \quad l/A \qquad (1)$$

where G is the impedance, l is the thickness of the pellet and A is cross section area of the pellet.

2.5 Open circuit voltage measurement (OCV)

Open circuit voltage of undoped and Mo^{6+} ion doped nanocrystals were measured after fabrication of solid state batteries by using high impedance multimeter (Phillips model PM 2718). All chemicals used in the fabrication of solid state batteries were of analytical grade. Iodine (Glaxo), Graphite (Qualigens), Silver (Qualigens) and undoped and Mo^{6+} ion doped nanocrystals were used as electrolyte without further purification. Three batteries were prepared in the present investigation in which silver was used as anode, synthesized nanocrystals as the electrolyte and a mixture of graphite and iodine in the ratio 9:1 as the cathode. These three materials were powdered separately and successively pressed in a pelletizing die by applied pressure of 5t cm^{-2} to give the desired battery [17]. The OCV value of following solid state batteries of undoped and Mo^{6+} ion doped nanocrystals were measured at temperature (27^{0}C) with humidity level of 45%.

Ag | $[(H_2O)(OH)Ce(S)_2Ce(OH)(H_2O)]$ | **C+I$_2$** (Undoped)

Ag | $[(H_2O)(SH)(OH)Mo\text{-}(O)_3\text{-}Mo(OH)(SH)(H_2O)]^{2+}$ | **C+I$_2$** (1%, and 5% Mo^{6+} ion doped)

3. RESULTS AND DISCUSSION

Elemental analysis results confirmed the presence of Mo^{6+} and S^{2-} ions in doped nanocrystals and Ce^{3+}, S^{2-} ions in undoped nanocrystals respectively. The pH value of undoped solution is found to be 7.21 while for Mo^{6+} ion doped solution pH values are 6.82 - 5.25. These pH values decreases from 7.21 to 5.25 clearly indicated that after doping Mo^{6+} ion solution becomes acidic which is light yellow color. The UV–vis. spectrum of undoped $[(H_2O)(OH)\ Ce\ S_2\ Ce(OH)(H_2O)]$ show strong and sharp absorption peak at 302 nm and Mo^{6+} ion doped $[(H_2O)(SH)(OH)Mo\text{-}(O)_3\text{-}Mo(OH)(SH)(H_2O)]^{2+}$nanocrystals indicates broad absorption bands at 320nm, 430nm and 480 nm. The results obtained from IR spectra of Mo^{6+} ion doped nanocrystals show absorption bands at 582.64 cm^{-1} and 648.73 cm^{-1} which suggested the υMo – S and υMo – O stretching. The band at 1432.12 cm^{-1} and 3347.83 cm^{-1} may be due to coordination of H_2O and OH to Mo metal ion and the coordination occurs through the – O ion which is clearly stipulated in IR-spectra at 2365 cm^{-1} respectively. The absorption peak appeared at 800 cm^{-1} in IR spectra confirmed that undoped compounds are in tetrahedral symmetry and Mo^{6+} ion doped nanocrystals. – at 1100 cm^{-1} show hexagonal symmetry[18,19].

The XRD studies of undoped crystalline nanoparticles show the four sharp and well defined diffraction lines at 2θ = 28.43^0, 47.29^0, 56.13^0 and 76.37^0 respectively which can be assigned to the (1 0 1),(1 1 1), (0 0 4) and (1 0 5) reflection planes of the hexagonal lattice while 1%, and 5% Mo^{6+} ion doped nanocrystals shows broad diffraction patterns which indicates synthesized nanocrystals are metallic cluster. The broadening of peaks in X – ray diffraction patterns after addition of different amount of Mo^{6+}ions reveals that Mo^{6+} ion goes into the crystal lattice and occupied the place of Ce^{3+} ion to obtained hexagonal symmetry.

The crystallite sizes of undoped and Mo^{6+} ion doped nanocrystals were calculated by using Debye Scherer equation [20].

$$L = 0.9\,\lambda / \beta \cos\theta \qquad (2)$$

Where L is the average crystallite domain size perpendicular to the reflecting planes, λ is the X-ray wavelength (1.5418A^0), β is the full width at half maximum and θ is the diffraction angle. The crystallite size of undoped and Mo^{6+} ion doped nanocrystals were calculated by using equation (2). It is clearly represented that undoped nanocrystals are uniform and range in size from 35 to 47nm while 1% of Mo^{6+}ion doped nanocrystals are range in size from 60 to 72nm, and 5% of Mo^{6+} ion doped nanocrystals are range in size from 76 to 88nm respectively. The crystallite size of Mo^{6+}ion doped nanoparticles are found to be larger than those of undoped nanocrystals. XRD results further suggested that when concentration of dopants increases size of nanocrystals also increase. It is clearly indicated that more and more Mo^{6+} ion goes in to crystal lattice and expansion of crystals lattice takes place due to larger coordination and clustering tendency of Mo^{6+} ion.

Fig.1 show the scanning electron microscope (SEM) of 5% Mo^{6+} ion doped nanocrystals which indicated that size of Mo^{6+}ion doped nanocrystals is 2µm.

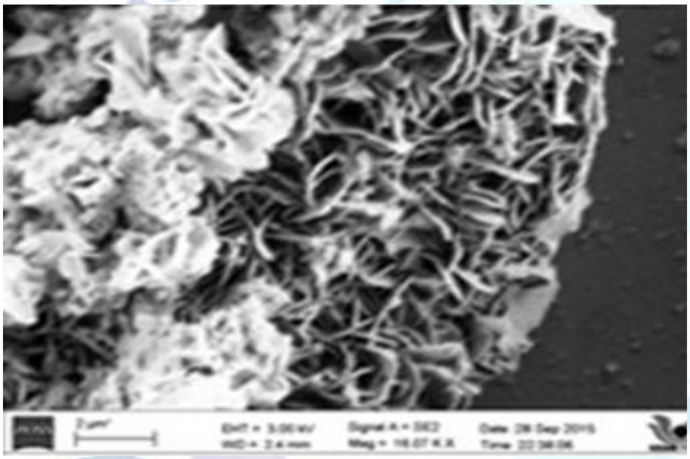

Fig.1. SEM of 5% Mo^{6+}ion doped nanocrystals

Fig.2 represent the typical TEM image of 5% Mo^{6+} ion doped nanocrystals which is in size 80nm.The diffraction rings have been indexed in the figure are clearly due to a hexagonal lattice structure prior to the X– ray diffraction measurements.

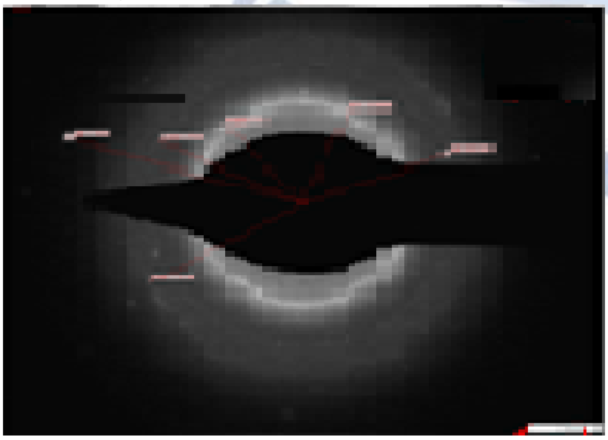

Fig.2. TEM image of 5% $Mo6^{+}$ ion doped nanocrystals.

The conductivity (σ) of undoped, 1% and 5% Mo^{6+} ion doped nanocrystals were measured at various temperatures (i.e.33-330⁰C) at frequency of 100 kHz given in Table 1. The results given in table 1 suggested that the conductivity of undoped nanocrystals at 33^0C is found to be 1.12 x 10^{-7} Scm^{1-} while at 330^0C is 1.45 x 10^{-4} Scm^{1-}. The conductivity of 1% of Mo^{6+}ion doped nanocrystals at 33^0C is 6.53 x 10^{-7} Scm^{1-} and at 330^0C is 4.91 x 10^{-4} Scm^{1-} which is greater than those of undoped nanocrystals. The conductivity of 5% Mo^{6+}ion doped nanocrystals at 33^0C is 8.06 x 10^{-6} Scm^{1-} and at 330^0C is 7.40 x 10^{-4} Scm^{1-} which is greater than those of 1% Mo^{6+} ion doped nanocrystals. The results presented in Table 1 also suggested that conductivity values of 1% and 5% of Mo^{6+} ion doped nanocrystals are greater than those of undoped nanocrystals at all temperature respectively.

The three different batteries were also fabricated by using undoped, 1% and 5% Mo^{6+}ion doped nanocrystals as electrolytes with corresponding OCV values are listed in Table 2. The OCV values for 1% and 5% of Mo^{6+} ion doped cells II and III are 0.521 and 0.634V respectively. When undoped cerium sulphide is used as electrolyte (cell I) the OCV value is low as 0.325V. This clearly suggests that the addition of percentage of Mo^{6+} ions increases markedly the OCV value in the order; OCV (5% Mo^{6+}) > OCV (1% Mo^{6+}) > OCV (undoped). On comparing the results obtained from Cr^{3+} ion doped nanocrystals published in paper [1] shows that conductivity and OCV values of the Mo^{6+} ion doped nanocrystals are higher than those of Cr^{3+} ion doped nanocrystals. This is because of the fact that upon addition of Mo^{6+} ion dopants, enhance expansion of crystal lattice than Cr^{3+} ion doped nanocrystals therefore more and more ions trape in to the lattices and these ions easily migrate towards the electrodes and consequently causes larger conductivity and OCV values. The conductivity of undoped nanocrystals at 33^0C is ~10^{-7} Scm^{-1} and OCV is 0.325V which is much less than Cr^{3+} and Mo^{6+} ions doped nanocrystals. This is probably undoped nanocrystal is not able to provide sufficient mobile of H^+, OH^- and SH^- ions. If these nanocrystals doped with Mo^{6+} ions the conductivity and OCV values increases continuously with increasing concentration of dopants of Mo^{6+} ions and temperature respectively. The increase in conductivity and OCV values with increase in concentration of dopants of Mo^{6+}ion may be simply due to the expansions of crystal lattice resulting in a more opened – up structure suitable for conduction [16]. However, this increase of conductivity and OCV values may also be explained by the two possible reasons.

The first possibility is that when dopants of Mo^{6+}ion added to undoped nanocrystals which are reduced to Ce^{3+} ion and consequently causes expansion of crystal lattice through which movement of electrons and ions occurs readily. Thus increase in conductivity and OCV values appears due to increase in mobility of electrons and ions across the crystal lattice.

The second possible reason for increase in conductivity and OCV values is that during the nanocrystals formation undoped nanocrystals react with dopants of Mo^{6+} ion to form $[(H_2O)(SH)(OH)Mo\text{-}(O)_3\text{-}Mo(OH)(SH)(H_2O)]^{2+}$ nanocrystals in which Mo^{6+} ion has strong coordination power than Ce^{3+} ion. Therefore, Ce^{3+} ion is replaced by Mo^{6+} ion and has more conducting in nature. The results of conductivity and OCV measurements indicated that the prepared undoped and Mo^{6+} ion doped nanocrystals are used as solid state batteries [15]. On the basis of IR and X-ray diffraction studies it has been shown that undoped nanocrystals contain Ce – S, Ce – O and Ce – OH_2 bonds while the dopants of Mo^{6+} ions are used to it, Ce^{3+} ions get replaced by Mo^{6+}ions resulting in formation of Mo – $(O)_3$ – Mo bonds and Ce^{3+} ions gets free in the crystal lattice which are responsible for ionic conduction. When more and more Mo^{6+} ions added to undoped nanocrystals more and more Ce^{3+} ions replaced due to larger coordination ability of Mo^{6+} ions and consequently increases conductivity and OCV values. On the basis of above discussion a proposed possible structure and model for undoped and Mo^{6+} ion doped nanocrystals are given in Fig.3.

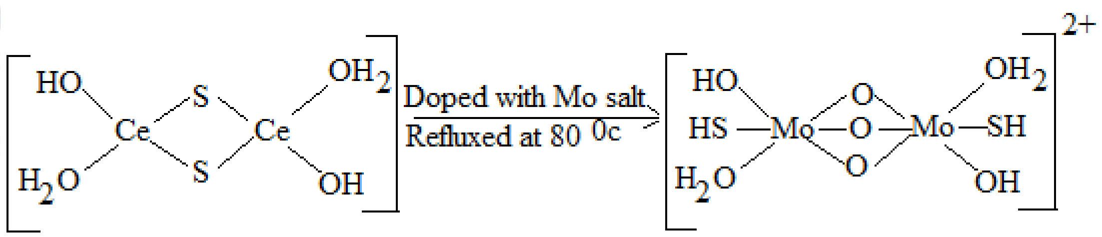

Fig.3. Proposed structure for undoped and Mo^{6+} ion doped nanocrystals.

These results shows that lattice sites are occupied by Mo^{6+} ions, therefore, broadening of peaks in X-ray diffraction pattern occur, which clearly indicated that when concentration of dopants of Mo^{6+} ions increases broadening of X- ray diffraction patterns also increase consequently size of nanocrystals increases. This increase in size with increase of dopants concentration also ascertained by scanning electron microscope (SEM), transmission electron microscope (TEM). The electrical conductivity and OCV studies provide support to the size of nanocrystals respectively. The expansions of crystal lattice after addition of dopants of Mo^{6+} ions are also explained by UV- visible spectrum through the splitting peaks. Thus the synthesized Mo^{6+} ions doped nanocrystals are much more importance in solid state batteries.

4. CONCLUSIONS

We have prepared a more useful undoped and Mo^{6+}ion doped nanocrystals. On the basis of electrical conductivity and OCV studies reported above, it can be concluded that electrical conductivity and OCV values of Mo^{6+}ion doped nanocrystals are higher than undoped nanocrystals and corresponding values increases with increasing concentration Mo^{6+}ions. The Mo^{6+} ion doped nanocrystals are potential electrolytes for the fabrication of solid state batteries. Among the nanocrystals investigated the lattices of nanocrystals were hexagonal with well defined crystalline nature.

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Table1. Conductivity (σ) values for undoped and Mo^{6+} ion doped nanocrystals at different Temperature.

Nanocrystals	Temperature °C	1/T x 10^3 K^{-1}	Conductivities(σ) Scm^{-1}
Undoped	33	3.26	1.12 x 10^{-7}
	330	1.65	1.45 x 10^{-4}
1% Mo^{6+} ion doped	33	3.26	6.53 x 10^{-7}
	330	1.65	4.91 x 10^{-4}
5% Mo^{6+} ion doped	33	3.26	8.06 x 10^{-6}
	330	1.65	7.40 x 10^{-4}

Table2. Open circuit voltage (OCV) values of solid state batteries using Mo^{6+} ion doped and undoped Nanocrystals as electrolyte

Cell	Anode	Electrolyte	Cathode	OCV (V)
I	**Ag**	$[(H_2O)(OH)\mathbf{Ce}(S)_2\mathbf{Ce}(OH)(H_2O)]^{2-}$ (Undoped)	**C + I_2**	0.325
II	**Ag**	$[(H_2O)(SH)(OH)Mo\text{-}(O)_3\text{-}Mo(OH)(SH)(H_2O)]^{2+}$ (1% Mo^{6+} ion doped)	**C + I_2**	0.521
III	**Ag**	$[(H_2O)(SH)(OH)Mo\text{-}(O)_3\text{-}Mo(OH)(SH)(H_2O)]^{2+}$ (5% Mo^{6+} ion doped)	**C + I_2**	0.634

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