

***“DEVELOPMENT OF $Y_2O_3 : Er_2O_3$ SYSTEM AND ITS DISPERSION STUDY IN
DIFFERENT MEDIA”***

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

By

MUKESH KUMAR RATRE

Roll No: 107CR020



NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA
राष्ट्रीय प्रौद्योगिकी संस्थान, राउरकेला
Golden Jubilee Year 2010-2011

DEPARTMENT OF CERAMIC ENGINEERING

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Under the Guidance of

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9th May 2011

Mukesh Kumar Ratre



NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA 2011

CERTIFICATE

This is to certify that the thesis entitled, “**DEVELOPMENT OF $Y_2O_3 : Er_2O_3$ SYSTEM AND ITS DISPERSION STUDY IN DIFFERENT MEDIA**” submitted by **Mr. Mukesh Kumar Ratre** in partial fulfilment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

Prof. Sumit Kumar Pal

Dept. of Ceramic Engineering

National Institute of Technology

Date: 9.5.2011

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ABSTRACT

$Y_2O_3 : Er_2O_3$ system has been developed and its dispersion properties have been studied in the present work. Erbium Oxide has been used as a dopant in to the Y_2O_3 matrix. The amount of Er_2O_3 has been fixed to 1 mol % with respect to 1 gm of Y_2O_3 . The $Y_2O_3 : Er_2O_3$ system has been prepared by using 2 methods : Co-precipitation and Auto-combustion methods. The powders obtained have been calcined at a temperature of $900^\circ C$ to develop the desired phase. Then the phase formation has been authenticated by XRD analysis which confirmed the cubic phase of the powder obtained after calcinations. The powders were dispersed in aqueous medium using PEG-400, PEG-6000 and TritonX-100 as dispersing agent. Then the zeta potential of the dispersed system has been studied at pH values between 7-8.

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Chapter **1**

INTRODUCTION

1.1 Yttrium Oxide

1.1.1 Introduction to Yttrium Oxide

The mineral from which yttrium oxide is extracted is Xenotime (YPO_4). Y_2O_3 is used as a primary material for both inorganic compounds as well as materials science. It is basically a white coloured crystalline solid. It is the most thermodynamically stable oxide available [1].

1.1.2 Properties of Yttrium Oxide

The molar mass of Y_2O_3 is 225.81 and density is 5010 kg m^{-3} . Its melting and boiling points are 2425°C and 4300°C respectively. It is insoluble in water and soluble in alcohol. Its crystal structure is cubic in nature and follows octahedral coordination. Its properties include high thermal stability as well as good transparency to infrared radiation. Y_2O_3 has an affinity towards oxygen and sulphur [2].

1.1.3 Applications of Yttrium Oxide

Yttrium oxide is used as a common starting material for both inorganic compounds as well as materials science. It is used to make YVO_4 europium and Y_2O_3 europium phosphors that give the red color in the color TV picture tubes. It is also used to make yttrium iron garnets that are very effective microwave filters. It is used in the manufacture of high temperature superconductor $\text{YBa}_2\text{Cu}_3\text{O}_7$ [3].

The high infrared transmission added together with good erosion resistance and thermal shock resistance, makes it ideal for use as protection domes for infrared sensors. It is used in optical sensors with high sensitivity. It can also be used as X-ray detection material. It is used for the production of thermal barrier coatings. Any high

temperature component made from graphite, ceramic or metal that is in contact with reactive molten metals such as uranium, titanium, their alloys, etc. can be protected using yttrium oxide. The reactive material is in direct contact with a thin layer of Yttria but is not in direct contact with the metal or graphite or ceramic, so they can be protected. Whenever purity of the metal is one of the primary consideration during melting as well as casting operations, Yttrium Oxide can be considered as the most chemically resistant material. It can also be used as an additive for stabilizing zirconia and hence enhance its fracture toughness.

Y_2O_3 nano-particles doped with Er^{3+} shows bright upconversion emission and therefore used as upconversion bio-imaging probe [3].

1.1.4 Structure of Yttrium Oxide

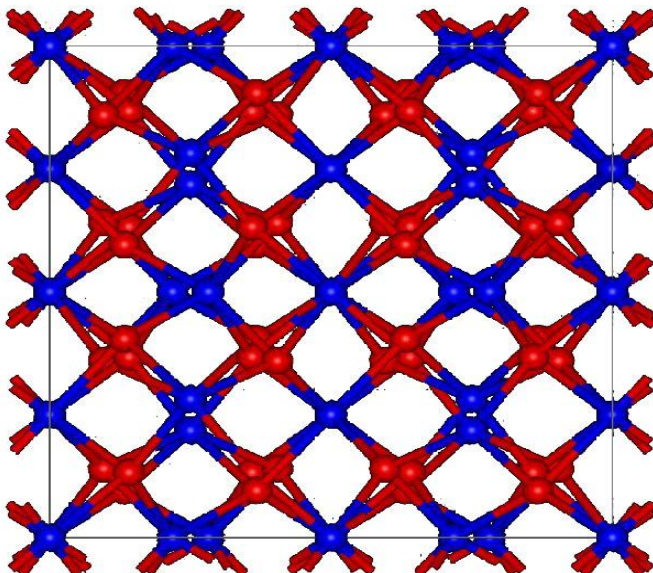


Figure 1(I) – shows the structure of yttrium oxide [21].

1.2 Erbium Oxide

1.2.1 Introduction, properties and uses

Erbium Oxide is Er_2O_3 and its IUPAC name is Erbium(III) oxide. It is a pink solid and seldom used for colouring of glass-sand and also used as a dopant for optical fibres and optical amplifiers. It was partially isolated by Carl Gustaf Mosander in 1843. It was first synthesised in its pure form by Georges Urbain and Charles James in the year 1905. Erbium oxide can also be used as a burnable neutron poison for nuclear fuel. It can react with acids to form the corresponding erbium(III) salts. Its molar mass is $382.52 \text{ g mol}^{-1}$ and density is 8.64 g cm^{-3} . Its melting and boiling points are 2344°C and 3290°C respectively [21].

Er^{3+} doped Y_2O_3 nano-particles shows bright upconversion emission and therefore used as upconversion bio-imaging probe [3].

1.2.2 Structure of Erbium Oxide

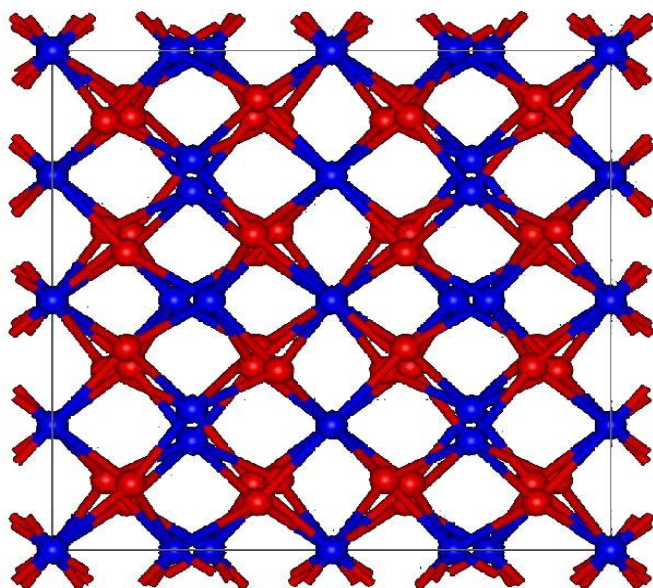


Figure 1(II) – shows the structure of erbium oxide [21].

1.3 Polyethylene Glycol (PEG)

1.3.1 Introduction, properties and uses

The molecular formula is $C_{2n+2}H_{4n+6}O_{n+2}$. It is also called as polyethylene oxide (PEO) or polyoxyethylene (POE), but it depends on its molecular weight. PEG, PEO, or POE indicates to polymer/oligomer of ethylene oxide. But, PEG refers to materials with a molecular mass below 20,000 g/mol. And, PEO refers to polymers with a molecular mass above 20,000 g/mol. POE refers to a polymer of with any molecular mass.

It is used as a dietary preventive agent against cancer in animal models [20].

1.3.1(a) PEG 400

It is harmless towards skin and easily soluble in water. It is used in the manufacture of cosmetics. It is a superior and thermally stable heating medium because of its high molecular weight, boiling point and specific heat ; and thus used in hot bath processes. It is used as a lubricant, plasticiser and synthetic leather It is also used as a flux for soldering. It is also used in toothpaste to remove dirt on the teeth [19].

1.3.1(b) PEG 6000

It is a high molecular weight polymer of ethylene oxide and is soluble in water. Because of its laminar structure, it is used as binder & dry lubricant. It is also used for pharmaceutical preparations. It can be used as a mould release agent in rubber industry. It is used as wetting agents to inhibit soap cracking and binder for facial It is used as an additional component of galvanisation bath for copper & nickel electroplating of steel and iron parts. It is a non ionic product and thus facilitate the even distribution of electrolytes [19].

1.3.2 Structure of PEG

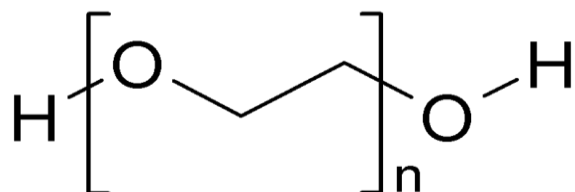


Figure 1(III) – shows the structure of PEG [21].

1.4 TritonX-100

1.4.1 Introduction, properties and uses

The molecular formula of Triton X-100 is $(C_{14}H_{22}O(C_2H_4O)_n)$. It is a non-ionic surfactant which has a hydrophilic polyethylene oxide group and a hydrocarbon lipophilic or hydrophobic group. The hydrocarbon group is a 4-(1,1,3,3-tetramethylbutyl)-phenyl group. At room temperature, it is highly viscous and thus is easier to use after heat treatment. It is used as detergent in laboratories. It can be used in DNA extraction as part of the lysis buffer. The surface tension of aqueous solutions can be reduced by its use.

1.4.2 Structure

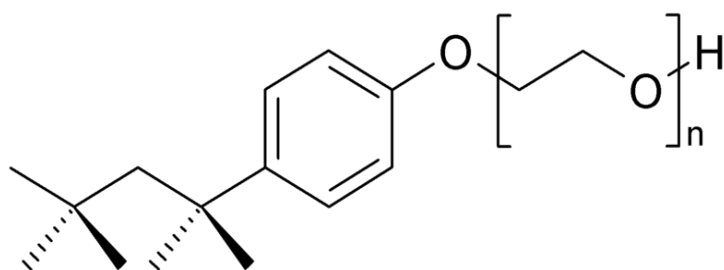


Figure 1(IV) – shows the structure of TritonX-100 [21].

1.5 Outline of the report

An introduction to the topic has been presented in this chapter. The next chapter deals with the available literature, objectives and motives of our work. Chapter 3 describes the experimental procedures and the characterization techniques used, all in detail. Chapter 4 discussed the results obtained, and seeks to provide an explanation to those results. The conclusions of the work are summarized in the final chapter.

Chapter **2**

LITERATURE REVIEW

2.1 Synthesis and microstructural properties of $\text{Y}_2\text{O}_3 : \text{Er}^{3+}$ system

Trivalent rare earth ions can be doped with upconversion luminescent materials. It can be used to make upconversion lasers and fluorescent labels and the list goes on [6-11]. But Er^{3+} ions have been widely doped, as Er^{3+} ions have energy levels which approximately coincide with that of commercially available InGaAs laser diode [12-13]. Considering the hosts which are inorganic in nature, for the use as an upconversion luminescent material, Y_2O_3 is one of the best material because of its physical and chemical stability. It also possesses relatively low phonon energy which makes it very suitable for the use as a host [14-17].

$\text{Y}_2\text{O}_3 : \text{Er}^{3+}$ powders were prepared by the homogeneous precipitation process. The raw materials were nitrates and urea. The outcome was that the monodispersed spherical powders were obtained with a particle size of 200–210 nm when the calcination of the precipitates was done at 800 °C for 2 hours duration. The morphologies of the precursor and powders were affected by the change in the solution concentration of Y^{3+} and Er^{3+} . The solution concentration should be as low as 0.015M to promote precipitation precursor for keeping uniform spherical shape. The finally prepared monodispersed $\text{Y}_2\text{O}_3 : \text{Er}^{3+}$ phosphors show upconversion luminescence properties and the upconversion luminescence intensity reached its maximum value when the concentration of Er^{3+} is at 3% [5]

Bio-imaging is a technique by which we can know the details of the biological phenomena's both in vivo and vitro. In the field of biomedical science, it is one of the most important technology [18]. In the fluorescence bio-imaging, one of the major problem that occurs is the color-fading of the fluorescence probes and it limits the time of observation. It also damages the bio-system. All of these are caused by the ultraviolet irradiation excitation. By using rare-earth doped inorganic nanophosphors and then its

upconversion emission under infrared excitation, it can be solved. Surface modification of the nanophosphors with biocompatible polymers has been done to get the required biological functionality for the use as bio-imaging probes in organic tissues. On the Er doped Y_2O_3 particles in the nano range, the surface was modified by the formation of multilayer's of polyacrylic acid (PAAc) and α -acetal-poly(ethylene glycol)-block-[poly (2-(N,N-dimethylamino) ethyl methacrylate)] for the use as upconversion nanophosphor. 100 mg of Y_2O_3 nanoparticles was dispersed in PAAc aqueous solution with varying concentrations. They were then ultra-sonicated for 3 hours. Then the prepared dispersion was diluted to get exact and required concentration for the analysis by the addition of 10mM sodium phosphate buffer and the pH was maintained at 7.4 [4].

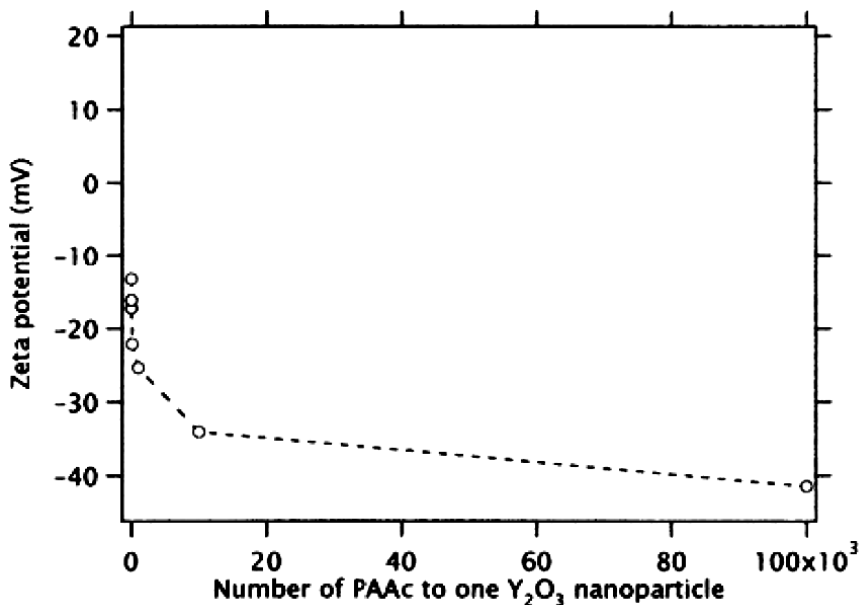


Figure 2(I) – Zeta Potential of Y_2O_3 nanoparticles [4].

$Y_2O_3 : Er^{3+}$ nanoparticle system was prepared by (i) enzymatic decomposition method (ii) homogenous precipitation method (iii) alkali co-precipitation method. The particle size varied from 20-30 nm in enzymatic decomposition to 100-200 nm in homogeneous precipitation and more than 200 nm in alkali co-precipitation methods respectively. The particles obtained can be used as a good upconversion bio-imaging probe [3].

Jingning Shan et. al [2] reported that by proper selection of ligands, precursor ratios adjustment and proper reaction conditions; highly uniform and monodisperse particles can be prepared and also the size range can be adjusted from 5 nm to 200 nm.

2.2 Summary of literature

Summary of the literature survey are :-

- (1) $Y_2O_3 : Er^{3+}$ nanoparticle system can be prepared by various methods.
- (2) Enzymatic decomposition process provided the least particle size distribution as compared to homogenous precipitation process and alkali co-precipitation process.
- (3) The particles obtained show bright upconversion emission.
- (4) The powder preparation technique plays a vital role in microstructural parameters.
- (5) By the increase in the amount of the PAAc, the zeta potential of Y_2O_3 nanoparticles decreases.

2.3 Objective of the present studies

In this paper we limited and focussed ourselves on the synthesis of the $Y_2O_3 : Er^{3+}$ powders and studied the XRD, FTIR and the zeta potential by dispersing the particles in varying concentrations of PEG-400, PEG-6000 and TritonX-100 respectively.

(1) Synthesis of the $Y_2O_3 : Er^{3+}$ powders by the precipitation and auto combustion methods respectively. For the precipitation method, HNO_3 and NH_4OH has been used and for auto combustion method, glycine has been used as fuel.

(2) Calcination of the precursors, thus formed, has been done at $900^\circ C$ and XRD has been studied for phase analysis.

(3) The transparent pellets have been formed by using KBr and the synthesized powder; and the FTIR analysis has been done to analyse the absorption and transmission spectra.

(4) The zeta potential of the prepared powders has been measured by dispersing the particles in varying concentrations of PEG-400, Peg-6000 and TritonX-100 respectively.

Chapter **3**

EXPERIMENTAL WORK

3.1 Synthesis of $Y_2O_3 : Er_2O_3$ powder

3.1.1 Precipitation Synthesis

3.1.1(a) Experimental Setup

150 ml nitric acid (HNO_3) was taken in a 500 ml beaker. Then, 1 gram of yttria was weighed and taken. Then 1 mol% with respect to 1 gram yttria, of erbium oxide was calculated and was found to be 0.0169 gram. The yttria and erbium oxide powders were added to the HNO_3 solution and then continuous stirring was done in a magnetic stirrer. The stirring was done till complete dissolution of the powders in the solution and thus a clear solution was obtained.

Then 20 ml of the above prepared solution was taken and diluted upto 200 ml with the help of distilled water. This solution was taken in a burette. Then, around 250 ml of ammonium hydroxide (NH_4OH) was taken in a 1000 ml beaker. Then, the solution from the burette was added drop wise to the NH_4OH solution with continuous stirring in a magnetic stirrer. The pH was checked at certain intervals and we had to see that it was maintained at 9. If the pH became less than 9, we added extra amount of NH_4OH to the 1000ml beaker to get the pH = 9. Then, after the precipitation process had taken place, we stopped the addition of the solution from the burette. The stirring was continued for another 1 hour. The precipitate obtained was filtered using 2 Wattman 42 filter papers by applying suction pressure. When the filtration was at its last stage, we added 200 ml of petroleum spirit to the precipitate for the removal of the residual water. The filtration process was then continued for overnight at room temperature. After the filtration process was over, we collected the filtered precipitate in a crucible and put it in a dryer for 24 hours. But the dryer had no effect on the precipitate. We calcined the precipitate at $900^\circ C$ and thus obtained the required powder. The sample name was Y_2O_3P .

3.1.2 Auto-combustion Synthesis

3.1.2(a) Experimental Setup

150 ml nitric acid (HNO_3) was taken in a 500 ml beaker. Then, 1 gram of yttria and 0.0169 gram of erbium oxide were weighed. The yttria and erbium oxide powders were added to the HNO_3 solution and then continuous stirring was done in a magnetic stirrer. The stirring was done till complete dissolution of the powders in the solution and thus a clear solution was obtained. Thus, the metal nitrates were formed in the clear solution.

In this process, glycine was used as a fuel for auto-combustion. Two compositions of glycine were used. In the first composition, metal nitrate to glycine ratio was taken as 1 : 1 and the glycine amount was calculated to be 0.6702 gram. In the second composition, the ratio was 1 : 2, and the amount of glycine was calculated to be 1.336 gram.

In the above obtained clear solution, we added the calculated glycine amount and then, the sample was heated in a magnetic stirrer with continuous stirring. After some time, gelatinous mass was formed inside the beaker. Then, we stopped the stirring and took out the magnetic needle. But the heating was continued. Then, after some more time combustion took place and powder was obtained. It was then left for 1 hour heating. After that the beaker and the powder were allowed to cool. Then, the obtained powder was taken in a crucible and calcined at 900°C . Thus, we obtained the required powder.

The sample name for 1 : 1 ratio (metal nitrate : glycine) was $\text{Y}_2\text{O}_3\text{C1}$ and for 1 : 2 (metal nitrate : glycine) was $\text{Y}_2\text{O}_3\text{C2}$ respectively.

3.2 General Characterization

3.2.1 X-Ray Diffraction(XRD)

For the study of phase analysis, the samples were calcined at 900 °C and the analysis was made with the help of X-ray diffractometer phillips PANalytical (Model: PW 1830 diffractometer, Netherland) with Cu K α radiation. The scanning was made from 15 °-80 ° for obtaining the intensity; and the scanning time period was 33min.

3.2.2 Fourier Transform InfraRed Spectroscopy(FTIR)

Fourier transform infrared spectroscopy (FTIR) is a measurement technique for collecting infrared spectra. Instead of recording the amount of energy absorbed when the frequency of the infra-red light is varied (monochromator), the IR light is guided through an interferometer. After passing through the sample, the measured signal is the interferogram. Performing a mathematical Fourier transform on this signal results in a spectrum identical to that from conventional (dispersive) infrared spectroscopy.

First, around 2 milligram of potassium bromide (KBr) and around 1-2 grain of the synthesised powder were taken in a pestle and mortar and then they were mixed properly. Then, they were pressed into pellets by applying 4 ton pressure for 120 seconds. The die and the punch were cleaned properly with acetone before pressing.

IR transmittance and absorption spectra were measured on the three transparent crystalline samples prepared by KBr pellet technique in the wavenumber range of 4400–450 cm⁻¹ on a Shimadzu FTIR (Model 8400 S) spectrophotometer.

3.2.3 Zeta Potential

Firstly, 0.1%, 0.3%, and 0.5% with respect to 0.01 gram of the synthesized powder was calculated which comes out to be 0.00001, 0.00003 and 0.00005 grams respectively. Then 100 ml of distilled water was taken in a 250 ml beaker and 0.00001 gram of PEG-400 was added to it and then stirred in a magnetic stirrer so that the PEG was dissolved in the water. After that 0.01 gram of the synthesized powder was added to the above prepared solution. Then, ultrasonication of the solution was done for 15 minutes using ultrasonic vibrator. Ultrasonication was repeated several times till we obtain a stable suspension. Then, its pH was checked using pH meter or pH paper which was around 5. Then, 0.1M Tris buffer solution was prepared. Its pH was around 12-13. Then few drops of the prepared Tris buffer solution was added to the suspension to adjust its pH to 8. Then, the zeta potential of the suspension was measured. This procedure was repeated with 0.00003 and 0.00005 gram PEG-400 respectively. All the 3 synthesized powders viz. Y_2O_3P , Y_2O_3C1 and Y_2O_3C2 were used individually for the above process described and then the suspensions were made with PEG-6000 and TritonX-100 respectively.

Chapter 4

RESULTS AND DISCUSSION

4.1 XRD analysis

$Y_2O_3:Er_2O_3$ samples prepared from the various precursors were characterized by X-ray diffractograms matching the relevant ASTM standards. They confirmed that all the synthesized powders had the cubic structure.

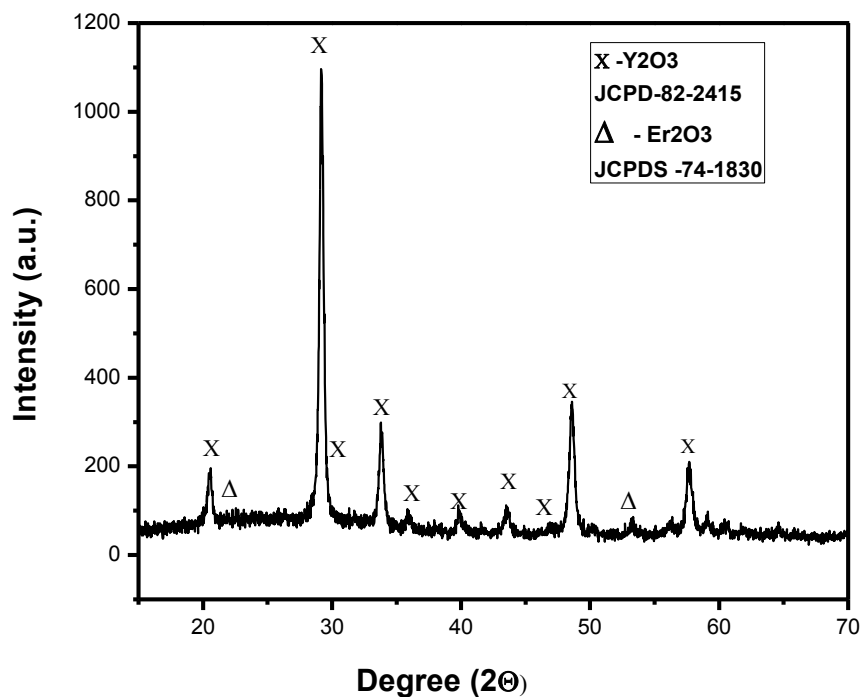


Figure 4(I) – Shows the XRD pattern of yttrium oxide doped with erbium oxide (Sample name- Y_2O_3P)

From the above figure, we can see the XRD pattern of the yttrium oxide synthesized by the precipitation technique. We can see the least existence erbium oxide phases as marked in the figure confirming that its incorporation in the yttrium oxide was successful. The erbium oxide phases have been marked as Δ and the yttrium oxide phases have been marked as X respectively. The XRD confirmed the cubic phases of both yttrium oxide and erbium oxide.

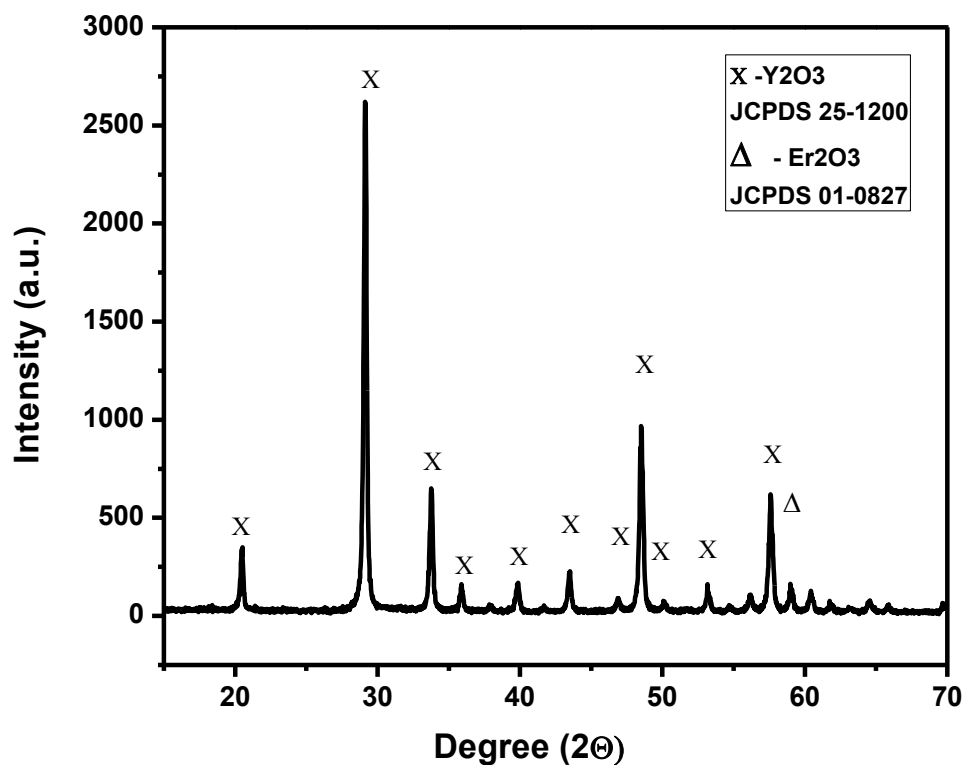


Figure 4(II) – Shows the XRD pattern of yttrium oxide doped with erbium oxide (Sample name- Y_2O_3C1)

The above figure gives XRD pattern of the yttrium oxide synthesized by the auto combustion technique. In this sample, the metal nitrate to glycine ratio was taken as 1 : 1. We can see the least erbium oxide phases in the figure confirming that its successful incorporation in the yttrium oxide. The yttrium oxide phases have been marked as X and the erbium oxide as Δ respectively. The XRD confirmed the cubic phases of both yttrium oxide and erbium oxide.

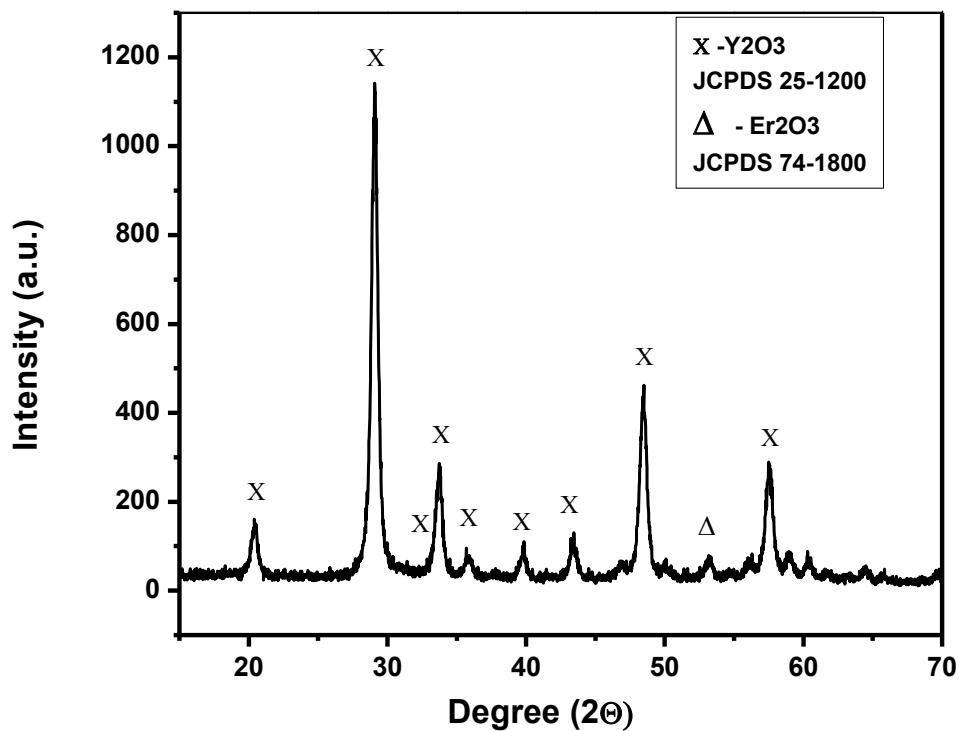


Figure 4(III) – Shows the XRD pattern of yttrium oxide doped with erbium oxide (Sample name- Y_2O_3C2)

The above figure gives the XRD pattern of the yttrium oxide synthesized by the auto combustion technique. In this sample, the metal nitrate to glycine ratio was taken as 1 : 2. We can see the least erbium oxide phases marked as Δ in the figure confirming that its incorporation in the yttrium oxide was successful. The XRD confirmed the cubic phases of both yttrium oxide and erbium oxide.

4.2 FTIR analysis

IR transmittance and absorption spectra were measured on the three crystalline samples prepared by KBr pellet technique in the wavenumber range of 4000–450 cm^{-1} .

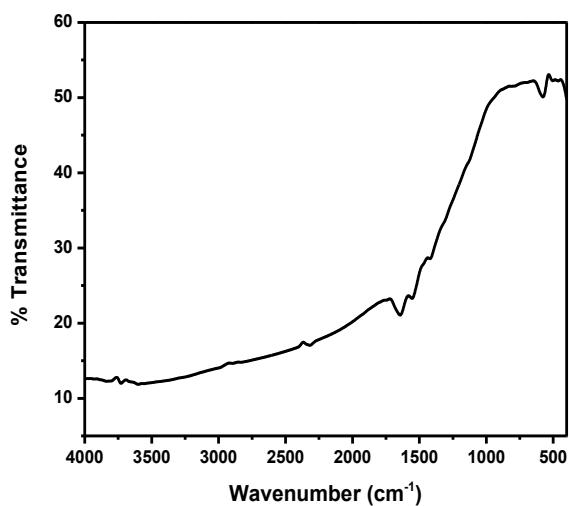


Figure 4(IV) – Transmission spectra of the Y_2O_3 synthesized through precipitation method.
(Sample name : $\text{Y}_2\text{O}_3\text{P}$)

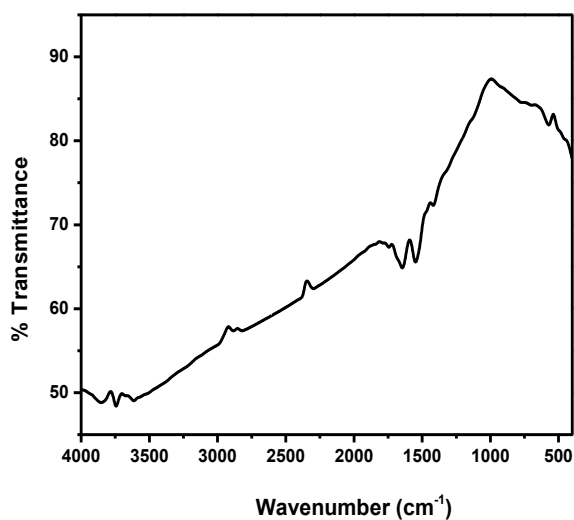


Figure 4(V) – Transmission spectra of the Y_2O_3 synthesized through auto-combustion method. (Sample name : $\text{Y}_2\text{O}_3\text{C1}$)

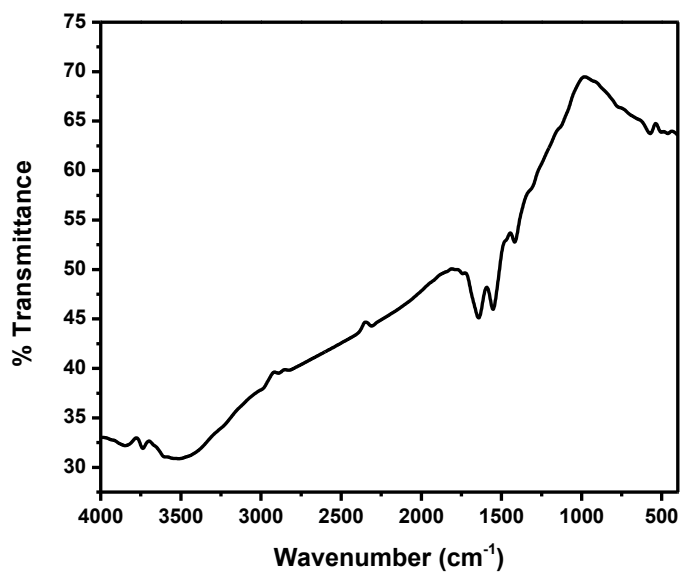


Figure 4(VI) – Transmission spectra of the Y₂O₃ synthesized through auto-combustion method. (Sample name : Y₂O₃C2)

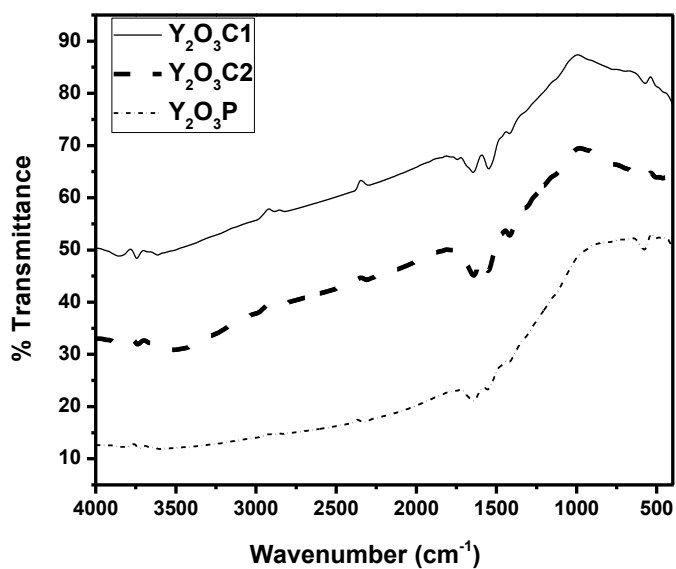


Figure 4(VII) – Transmission spectra of the all the three synthesised Y₂O₃ combined.

4.3 Zeta potential analysis

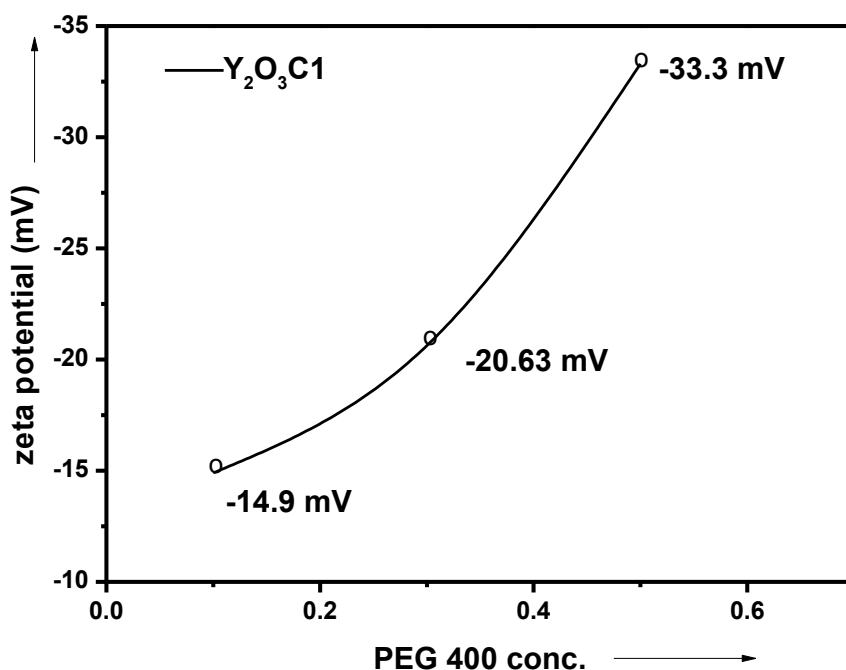


Figure 4(VIII) – Zeta potential v/s PEG400 concentration for the sample Y_2O_3C1 dispersed in water containing PEG400.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:1; in PEG-400 was analysed. The dispersed solution was stabilised by the increase in the concentration of PEG-400 in the dispersion which is indicated by the more negative value of the zeta potential as shown above. The zeta potential was highest when PEG-400 concentration was 0.5% and was -33.3 mV, which indicates that the system was most stable at that concentration. The zeta potential at 0.1% and 0.3% concentration were -14.9 mV and -20.63 mV respectively.

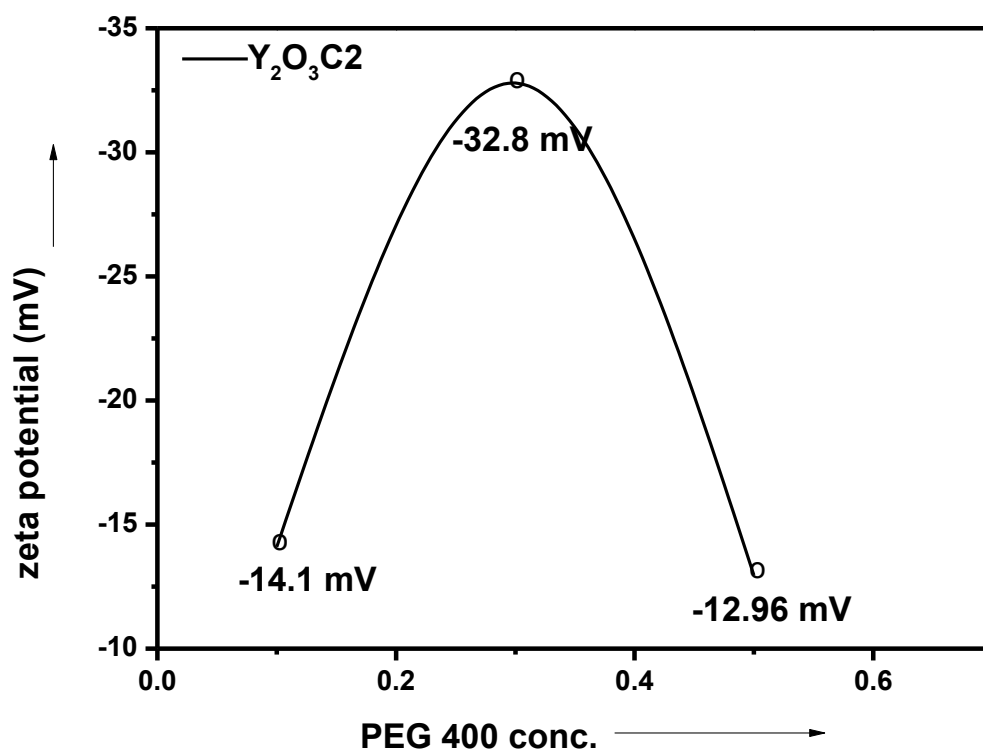


Figure 4(IX) – Zeta potential v/s PEG400 concentration for the sample $Y_2O_3C_2$ dispersed in water containing PEG400.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:2; in PEG-400 was analysed. The dispersed solution was most stable when the concentration of PEG-400 in the dispersion was 0.3% which is indicated by the more negative value of the zeta potential as shown above and the value was -32.8 mV. The zeta potential at 0.1% and 0.5% concentration of PEG-400 were -14.1 mV and -12.96 mV respectively.

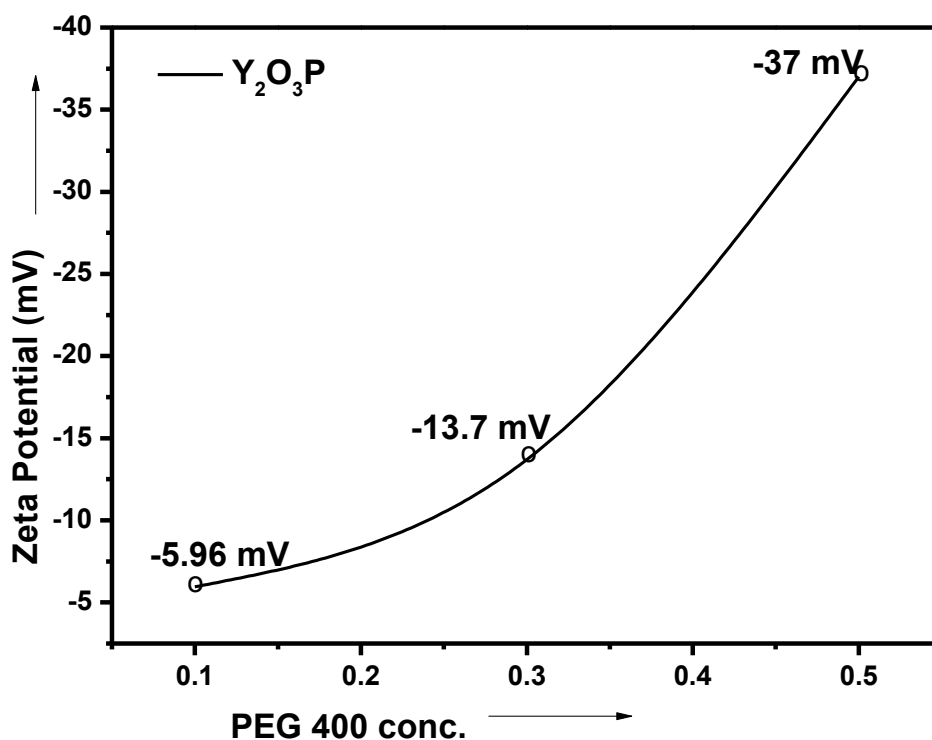


Figure 4(X) – Zeta potential v/s PEG400 concentration for the sample Y_2O_3P dispersed in water containing PEG400.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the precipitation method; in PEG-400 was analysed. The dispersed solution was stabilised by the increase in the concentration of PEG-400 in the dispersion which is indicated by the more negative value of the zeta potential as shown above. The zeta potential was highest when PEG-400 concentration was 0.5% and was -37 mV, which indicates that the system was most stable at that concentration. The zeta potential at 0.1% and 0.3% concentration were -5.96 mV and -13.7 mV respectively.

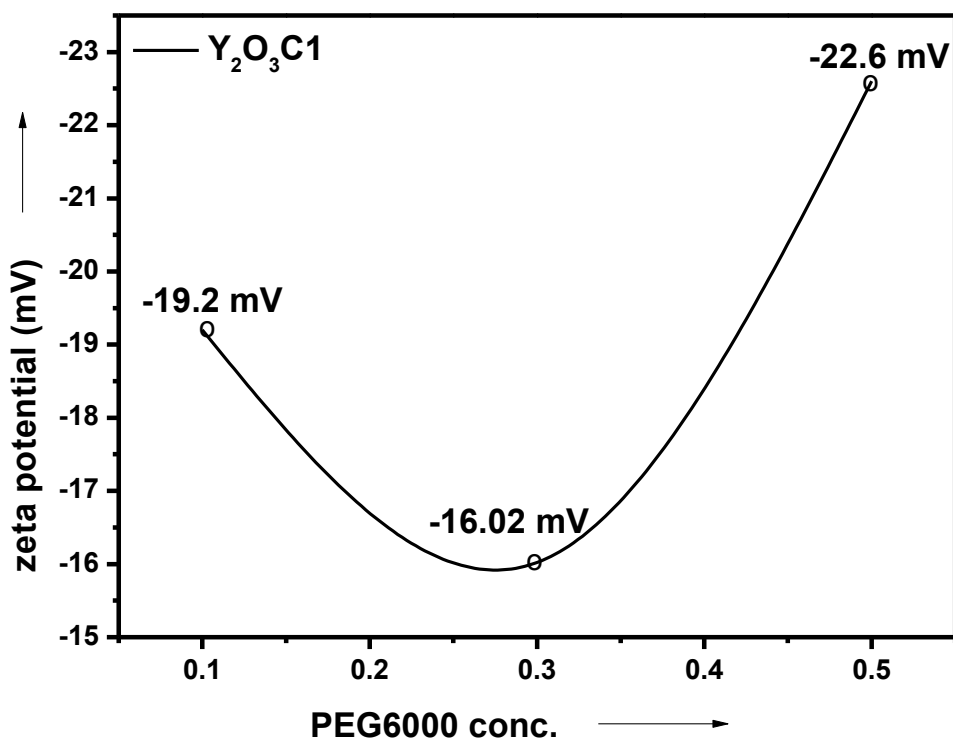


Figure 4(XI) – Zeta potential v/s PEG6000 concentration for the sample Y_2O_3C1 dispersed in water containing PEG6000.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:1; in PEG-6000 was analysed. The dispersed solution was least stable when the concentration of PEG-6000 in the dispersion was 0.3% which is indicated by the least negative value of the zeta potential as shown above and the value was -16.02 mV. The zeta potential at 0.1% and 0.5% concentration of PEG-6000 were -19.2 mV and -22.6 mV respectively.

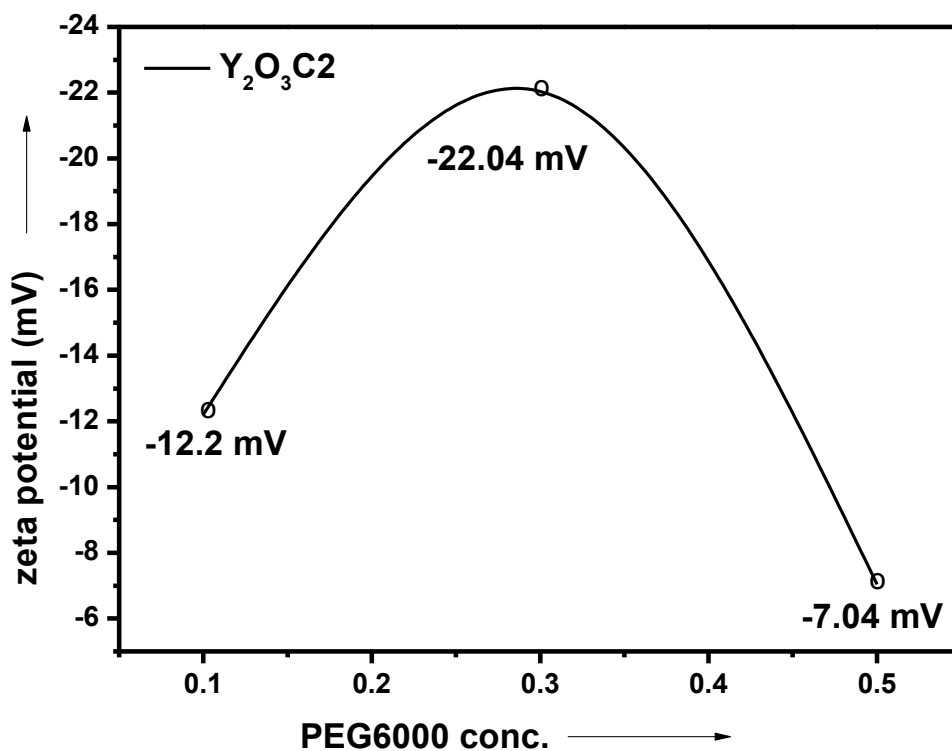


Figure 4(XII) – Zeta potential v/s PEG6000 concentration for the sample $Y_2O_3C_2$ dispersed in water containing PEG6000.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:2; in PEG-6000 was analysed. The dispersed solution was most stable when the concentration of PEG-6000 in the dispersion was 0.3% which is indicated by the more negative value of the zeta potential as shown above and the value was -22.4 mV. The zeta potential at 0.1% and 0.5% concentration of PEG-6000 were -12.2 mV and -7.04 mV respectively.

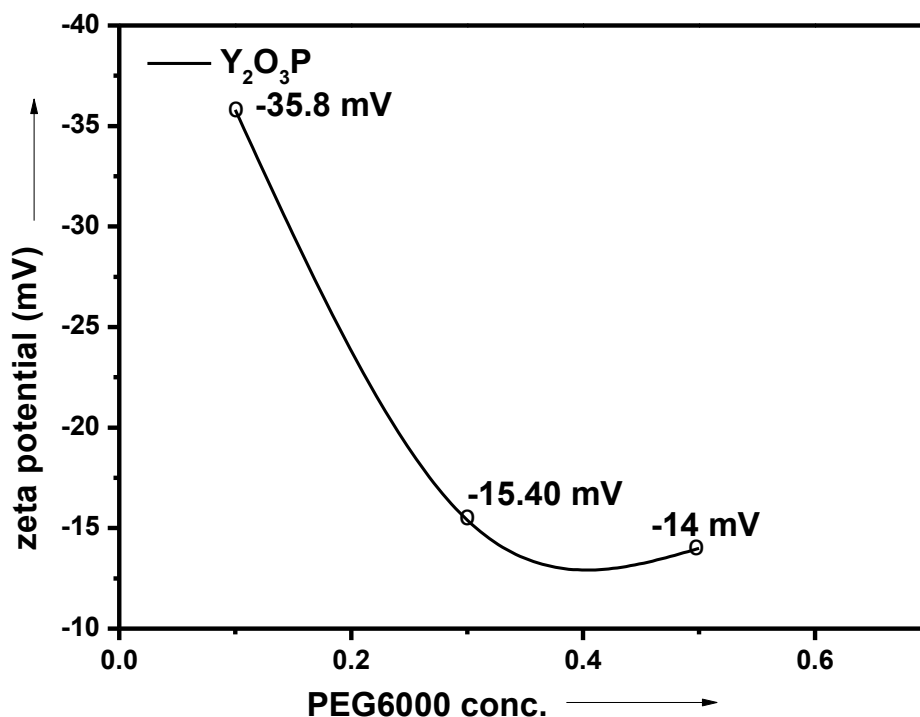


Figure 4(XIII) – Zeta potential v/s PEG6000 concentration for the sample Y_2O_3P dispersed in water containing PEG6000.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the precipitation method; in PEG-6000 was analysed. The stability of the dispersion decreased with the increase in the concentration of PEG-6000 which is indicated by the decrease in the negativity of the zeta potential. The dispersed solution was most stable when the concentration of PEG-6000 in the dispersion was 0.1% which is indicated by the more negative value of the zeta potential as shown above and the value was -35.8 mV. The zeta potential at 0.3% and 0.5% concentration of PEG-6000 were -15.4 mV and -14 mV respectively.

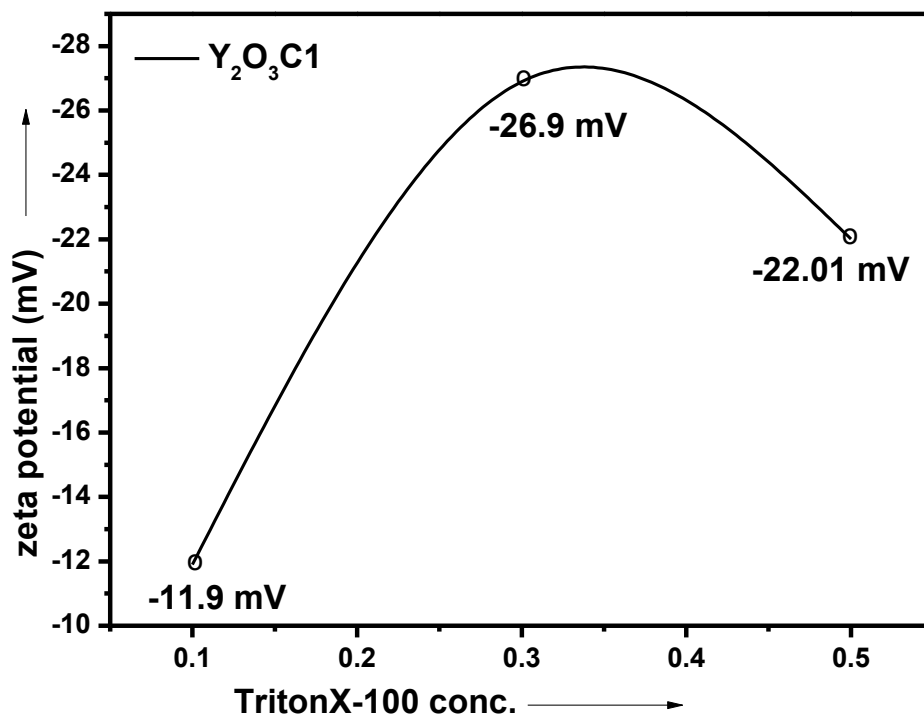


Figure 4(XIV) – Zeta potential v/s TritonX-100 concentration for the sample Y₂O₃C1 dispersed in water containing TritonX-100.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:1; in TritonX-100 was analysed. The dispersed solution was most stable when the concentration of TritonX-100 in the dispersion was 0.3% which is indicated by the more negative value of the zeta potential as shown above and the value was -26.9 mV. The zeta potential at 0.1% and 0.5% concentration of TritonX-100 were -11.9 mV and -22.01 mV respectively.

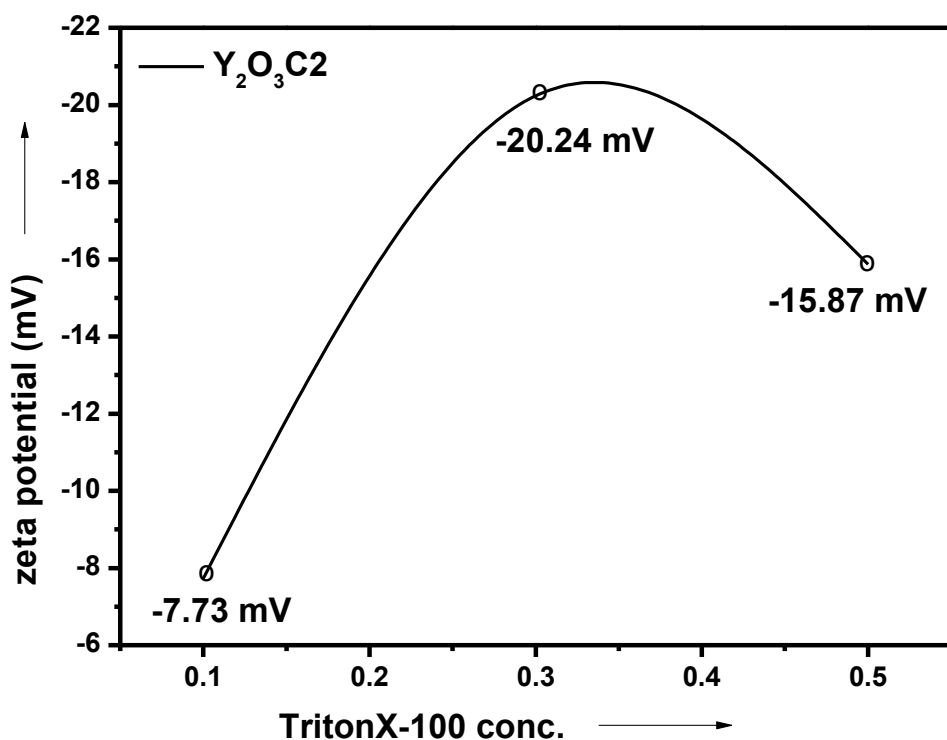


Figure 4(XV) – Zeta potential v/s TritonX-100 concentration for the sample $Y_2O_3C_2$ dispersed in water containing TritonX-100.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the auto combustion method in which the metal nitrate to glycine ratio was 1:2; in TritonX-100 was analysed. The dispersed solution was most stable when the concentration of TritonX-100 in the dispersion was 0.3% which is indicated by the more negative value of the zeta potential as shown above and the value was -20.24 mV. The zeta potential at 0.1% and 0.5% concentration of TritonX-100 were -7.73 mV and -15.87 mV respectively.

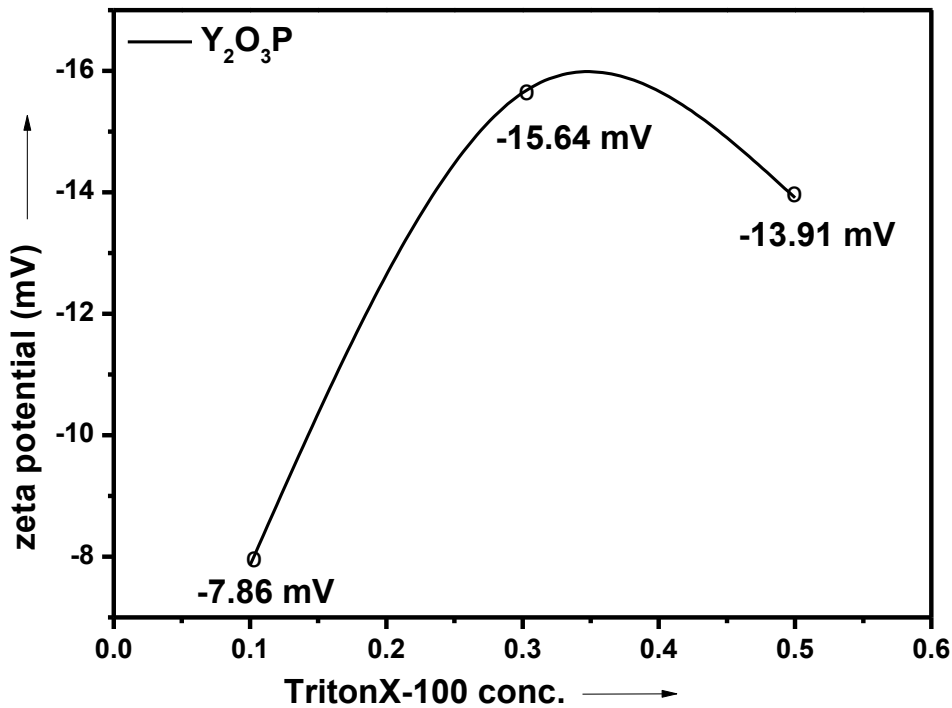


Figure 4(XVI) – Zeta potential v/s TritonX-100 concentration for the sample Y_2O_3P dispersed in water containing TritonX-100.

The pH of the dispersed solution was adjusted to 8 by the addition of 0.1M tris buffer solution. Then the zeta potential distribution of the yttrium oxide powder; prepared by the precipitation method; in TritonX-100 was analysed. The dispersed solution was most stable when the concentration of TritonX-100 in the dispersion was 0.3% which is indicated by the more negative value of the zeta potential as shown above and the value was -15.64 mV. The zeta potential at 0.1% and 0.5% concentration of TritonX-100 were -7.86 mV and -13.91 mV respectively.

Chapter 5

CONCLUSIONS

It can be concluded that Er_2O_3 doped Y_2O_3 system has been successfully prepared by co-precipitation as well as auto-combustion techniques which can be very useful for use as a bio-imaging probe. The dispersion study of the powders in aqueous media using PEG 400, PEG 6000 and triton X as dispersing agent shows different behaviour which can help in selecting proper dispersing agent during its use as bioimaging probe. For combustion synthesis the maximum zeta potential was observed for the sample $\text{Y}_2\text{O}_3\text{C1}$ where PEG 400 concentration was fixed at 0.5% of the solid loading. For co-precipitation synthesis the maximum zeta potential was observed for the sample $\text{Y}_2\text{O}_3\text{P}$ where PEG 6000 concentration was fixed at 0.1% of the solid loading. So it can be again concluded that through auto-combustion synthesis the particles are less agglomerated than the particles prepared by co-precipitation synthesis.

Chapter 6

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