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Study of Nanosized Al_2O_3 Powder Synthesized via Aldo-Keto Gel Method

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Abstract

Inorganic Al_2O_3 nanopowder was synthesized by aldo-keto gel method. The prepared sample was characterized by X-ray powder diffraction (XRD) and it is found that the average particle size was 31 nm. The Debye-Scherrer's formula was used to calculated standard particle size. The ultraviolet-visible (UV-VIS) spectra show strong absorption in UV region of 224 nm with weak absorption bands in the region of 300–350 nm. The photoluminescence (PL) properties were studied by fluorescence spectrophotometer (F-7000) indicating the emission of radiation of 399 nm when nanopowder excited by 224 nm radiation. The characteristic band gap of Al_2O_3 nanopowder was found to be 4.7 eV by aldo-keto gel method.

Keywords: Nano-powder, Ultraviolet-Visible spectra, Band Gap, X-ray powder diffraction, Photoluminescence.

Introduction

Aluminum oxide (Al_2O_3, alumina) is one of the most functional oxide ceramics. These attractive properties of Al_2O_3 create it as a most studied oxide material. The Al_2O_3 is widely applicable in firebricks, abrasives and integrated circuit (IC) packaging[1,2]. It has been used in numerous fields of engineering such as heat-resistant materials, coatings, abrasive grains, advanced ceramics and cutting materials. This is for the reason that Al_2O_3 is hard, highly resistance towards acids and bases, permit very high temperature applications [3-5].

Nano-sized powders have been paying attention due to their wide applications in the variety of field. There are numerous methods for synthesis of nano-alumina, and these are categorized into chemical and physical methods[3, 6-9]. The flame sprays pyrolysis method to synthesized agglomerate-free nano-sized Al_2O_3 particles with a size range of 5–30 nm reported by Tok *et al.*[3]. Shizhong *et al.* reported nano-sized Al_2O_3 powder synthesized by thermal metal organic chemical vapor deposition (MOCVD) combined with plasma[10]. Rodica *et al.* discussed synthesis and characterization of alumina nano-powder obtained by sol-gel method[11]. $Al_2O_3:Cr^{3+}$ was also used as red emitting phosphor in ruby laser. Among the several methods, in our work we use aldo-keto gel method for synthesis of nano-sized Al_2O_3 powder.

Methodology

The precursor Al $(NO_3)_3.9H_2O$ (2M) was taken in china clay basin and add some DD water in it. Acetone (1M) and Benzaldehyde (1M) were added to this mixture. Slowly heated and stirred the sample continuously to 120°C on hot plate, the pale yellow mixture was obtained. The color of the mixture changes to dark reddish brown. The process to forming gel started at close to 130 °C with the evolution of dark yellowish brown fumes. The mixture is then permitted to cool. Red gel is formed after cooling. The gel is further heated slowly to 300 °C. Dark red foam was formed with evolution of yellowish brown fumes. On further slow heating, pyrolysis of foam was started at 250 °C and completes up to 300 °C and shining black material was formed at 300 °C and burns into flame at 375 °C started. It was finally sintered at 1000 °C for 2 h. A snow-white powder of Al_2O_3 was obtained. The process concerned in the reaction was represented as a flow chart in Figure-1.

Results and Discussion

XRD investigation: Figure-2 showed the XRD pattern of Al_2O_3 by aldo-keto gel method. The formation of the nano crystalline phases was confirmed by X-ray diffraction. The XRD pattern for sample agree well with ICCD card No. 01-075-0783 for aldo-keto gel method.

The Al_2O_3 lattice possesses Trigonal structure with a space group R-3C (167) with lattice parameters a = 4.7517 A°, b = 4.7517 A° and c = 12.9650 A° and interfacial angles $\alpha = \beta = 90^0$ and $\gamma = 120^0$ for aldo-keto gel method. The average crystalline size of Al_2O_3 sample was calculated from the Debye Scherrer equation and the crystalline size was found to be 31 nm[12].

$$D_{(hkl)} = \frac{k\lambda}{\beta\cos\theta}$$

Where β the full width at half maximum (FWHM) of the (h k l) peak, k is the shape factor, λ the wavelength of X-ray and θ is the angle of diffraction.

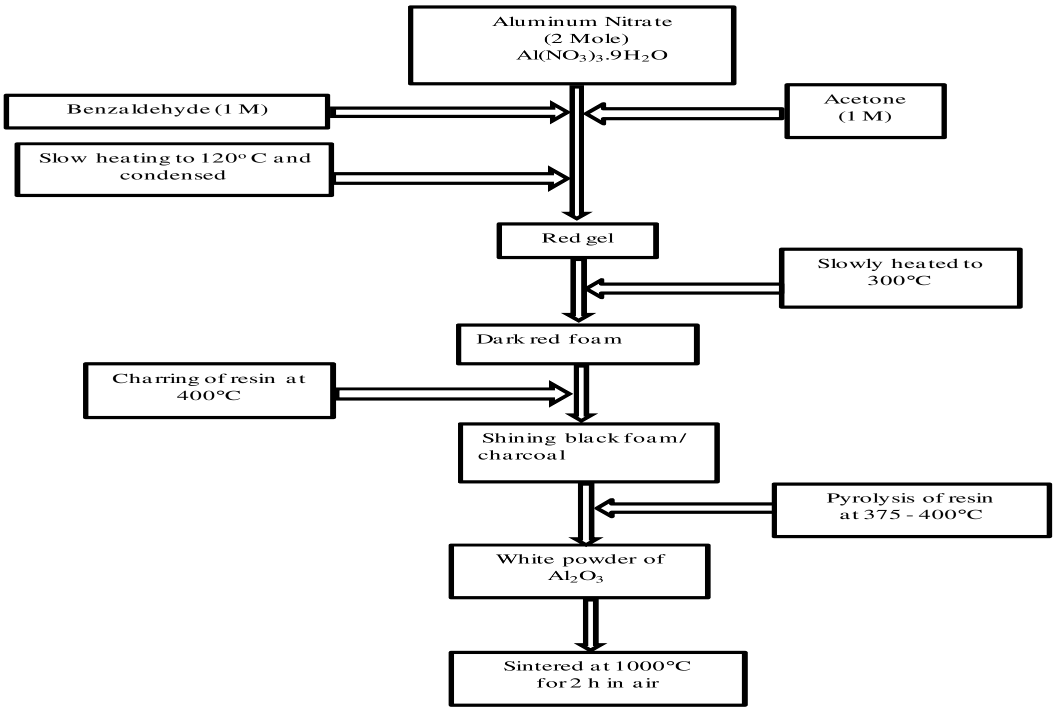

Figure-1
Flow chart of Al_2O_3 synthesized by aldo-keto gel method

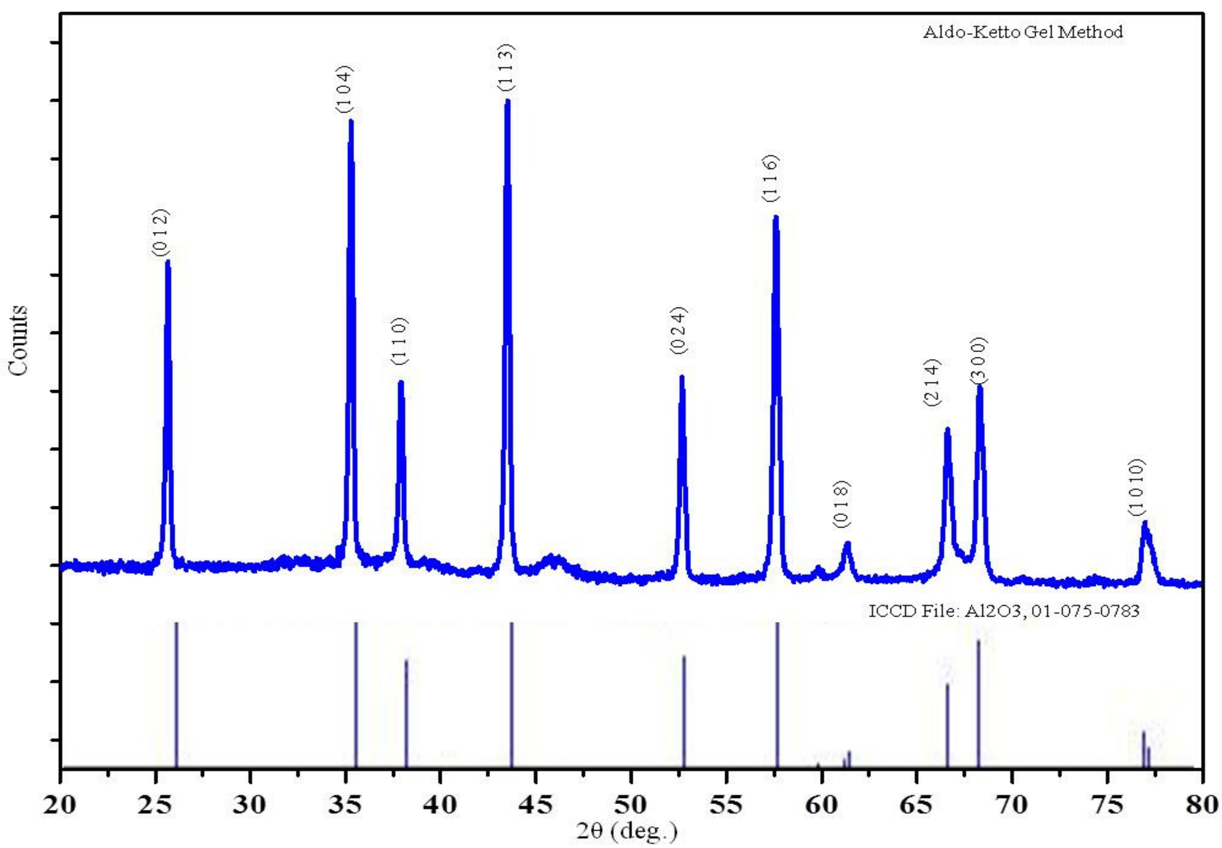

Figure-2
XRD patterns of the nano-sized Al_2O_3 powder synthesized by aldo-keto gel method

UV-VIS analysis: Figure-3 represented the UV–VIS absorption spectra of Al_2O_3 by aldo-keto gel method. The spectra show evidently a strong band-to-band absorption in the UV region. The UV–VIS. spectra represent a whole accord with that of nano-sized Al_2O_3 material in the literature[13]. It showed an absorption peak at 224 nm with feeble band in the region of 300–350 nm corresponding to the charge transfer bands from Al–O. The value of band gap was determined by extrapolation of the linear part of (αhν) plotted against the photon energy (eV). From the graph the band gap was found to be E_g = 4.7 eV via aldo-keto gel method. The formation of nano-sized Al_2O_3 powder is confirms from the blue shift in band gap[13]. This can be explained on the basis of defect chemistry[1, 14]. The optical band gap is depends upon the size of a particles, it is increase with a decrease in the particle size[14].

Fluorescence analysis: The concepts of self-fluorescent nano-crystals are said only when material in quantum state[1]. PL emission spectra showed intense blue luminescence range from 315 to 550 nm under the excitation of wavelength 224 nm at room temperature. The higher crest value of the emission was observed at 399 nm. The fluorescence wavelength varies by varying the particle size. It is well identified in quantum dots[13].

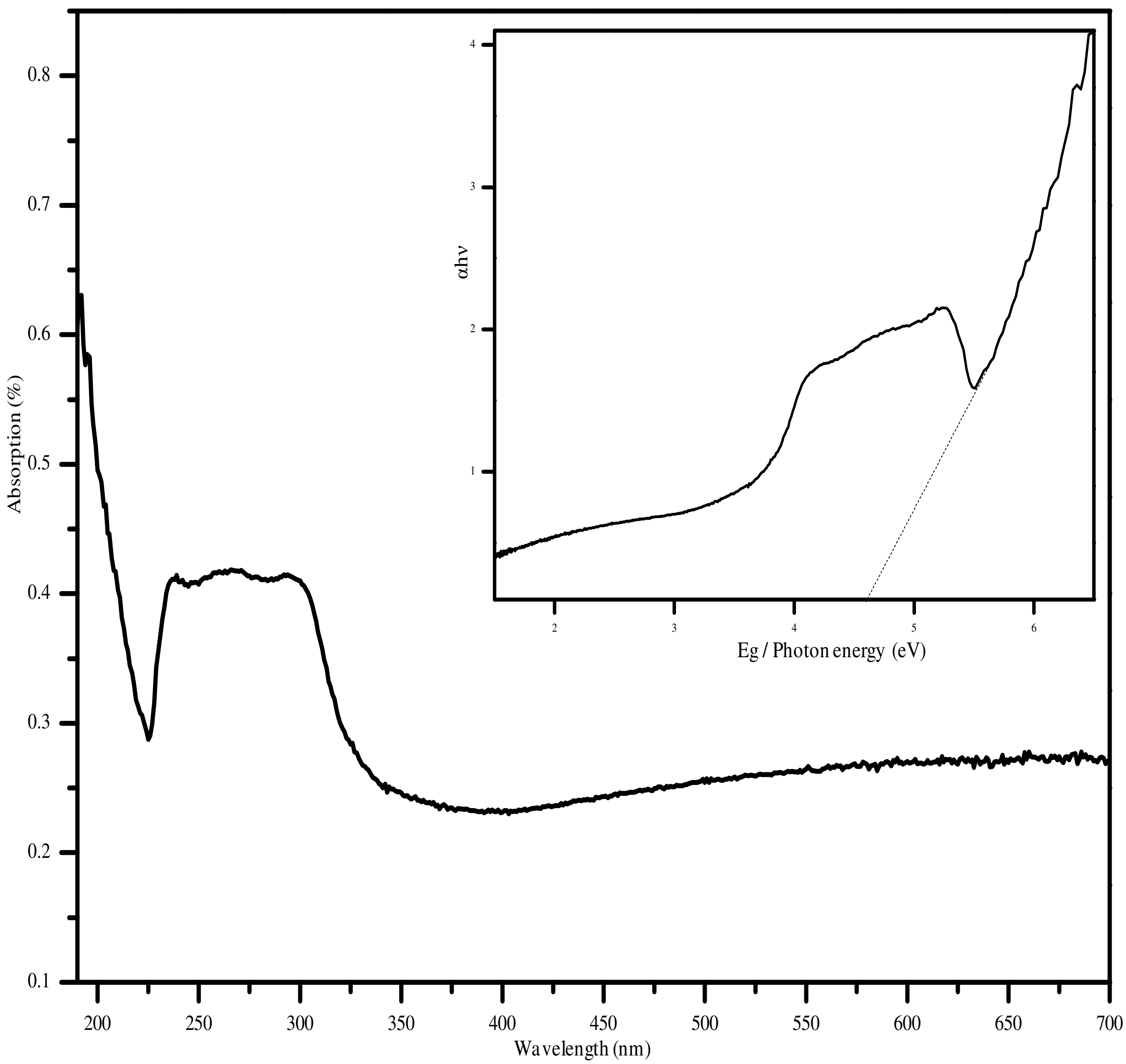

Figure-3
UV–Visible spectra of nano-sized Al_2O_3 powder synthesized aldo-keto gel method

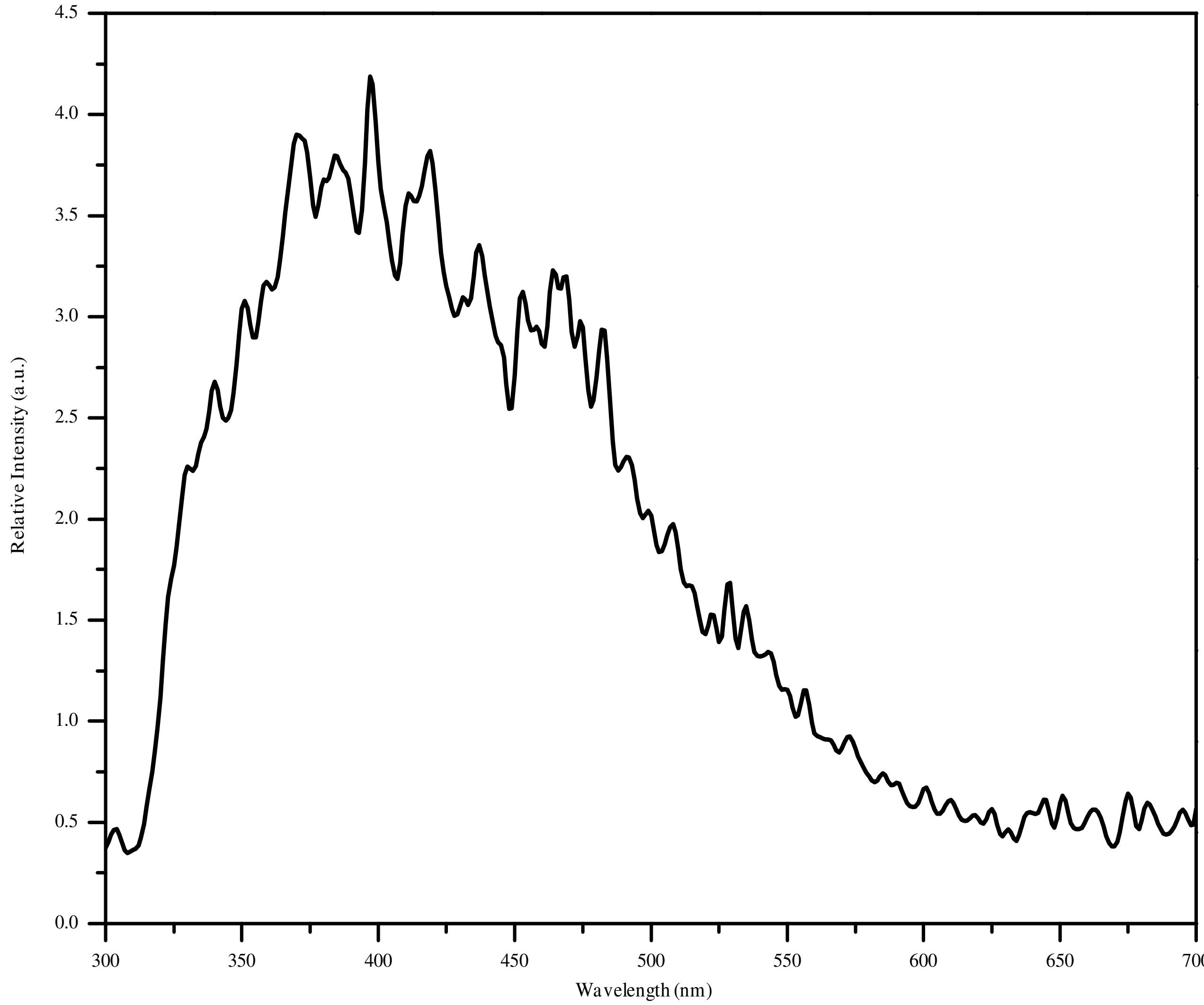

Figure-4
PL spectrum of nano-sized Al_2O_3 powder synthesized by aldo-keto gel method

Conclusion

Inorganic Al_2O_3 nano-powder has been successfully synthesized by using a novel aldo-keto gel method. The XRD model well agrees with ICDD file and ropes a whole crystalline nature of the synthesized materials. The band gap was found to be E_g = 4.7 eV by aldo-keto gel method. The material shows a superior UV absorption at 224 nm and a blue emission peaks at 399 nm by aldo-keto gel method. The prepared material might perhaps find applications as biocompatible materials, and ceramic sensing.

Figure-1

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References

1. Bajaj N.S. and Omanwar S.K. (2015). Low-temperature stearic acid sol–gel synthesis of α-Al_2O_3 quantum dots and its optical properties. *J. Sol-Gel Sci. Technol.,* 75, 1–5.
2. Krell A., Blank P., Ma H., Hutzler T. and Nebelung M. (2003). Processing of high-density submicrometer Al_2O_3 for new applications. *J. Am. Ceram. Soc.* 86**,** 546–553.
3. Tok A.I.Y., Boey F.Y.C. and Zhao X.L. (2006). Novel synthesis of Al_2O_3 nano-particles by flame spray pyrolysis. *J. Mater. Process Technol.*, 178, 270–273.
4. Perry R.H. (1984). Chemical Engineers Handbook, 6th Ed., McGraw-Hill, New York, 23-26.
5. Tikkanen J., Gross K.A., Berndt C.C., Pitkanen V., Keskinen J., Raghu S., Rajala M. and Karthikeyan J.

(1997). Characteristics of the liquid flame spray process. *Surf. Coat. Technol.*, 90, 210–216.

6. Wu J.M. (2001). Nano-sized amorphous alumina particles obtained by ball milling ZnO and Al powder mixture. *Mater. Lett.*, 48, 324– 330.

7. Sharma P.K., Varadan V.K. and Varadan V.V. (2003). A critical role of pH in the colloidal synthesis and phase transformation of nano size α-Al_2O_3 with high surface area. *J. Eur. Ceram. Soc.*, 23, 659– 666.

8. Wen H., Chen Y., Yen F. and Huang C. (1999). Size characterization of alpha alumina crystallites during phase transformation. *Nanostruct. Mater.*, 11, 89-101.

9. Tani T., Madler L. and Pratsinis S.E. (2002). Synthesis of zinc oxide/silica composite nanoparticles by flame spray pyrolysis. *J. Mater. Sci.* 37, 4627–4632.

10. Han S., Chen J., Zheng P. and Qing P. (2011). Characterization of Nanosized Al_2O_3 Powder Synthesized by Thermal-Assisted MOCVD and Plasma-Assisted MOCVD. *J. Chem. Eng.*, 30, 83-88.

11. Rogojan R., Andronescu E., Ghiţulica C. and Ştefan B. (2011). Synthesis and characterization of alumina nano-powder obtained by sol-gel method. *U.P.B. Sci. Bull.*, 73, 1454 -2331.

12. Nemade K.R. and Waghuley S.A. (2014). Low temperature synthesis of semiconducting α-Al_2O_3 quantum dots. *Ceram. Int*, 40, 6109–6113.

13. Irimpan L., Nampoori V.P.N., Radhakrishnan P., Deepthy A. and Krishnan B. (2007). Size dependent fluorescence spectroscopy of nanocolloids of ZnO. *J. Appl. Phys.*, 102, 063524.

14. Koparkar K.A., Bajaj N.S. and Omanwar S.K., (2015). Combustion synthesis and photoluminescence properties of Eu^{3+} activated $Y_2Zr_2O_7$ nano phosphor. *Indian J. Phys*, 89, 295–298.