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ORIGINAL ARTICLE **OPEN ACCESS**

Studies On Synthesis, Characterization, and Gas Sensing Properties of Dysprosium Doped Aluminum Trioxide

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ABSTRACT

Nanoparticles of Dysprosium doped Aluminium trioxide gas sensors having the chemical formula DyAlo3 (x=0.5) have been synthesized with sol-gel combustion approach and sintered at 600 ℃ and 750 ℃ . The morphology and composition of the obtained samples are examined and described using TGA, DTA, FESEM, XRD, EDS techniques. XRD results confirmed the formation of nanoparticles. The EDS findings show that there are no additional elemental impurities in the Dyalo3 NPs that have been prepared. Gas sensing properties were tested for NH3, LPG, acetone. For NH3, t h e highest sensing response for 100ppm was obtained at 350 ℃ . For LPG highest sensing response was found for 1000 ppm at 300 ℃ . For acetone highest sensing response was found for 1000ppm at 400 ℃ . In this case; t he operating temperature and sensitivity of the gas sensor are low. The response of t h e DyAlo3 pellet was observed to be fast(~3s) to 100ppm for NH3, while the recovery was quick (~30s).

Keywords: *Nanoparticles, Sol-gel auto combustion method, TGA, XRD, FESEM, EDS gassensor, LPG, NH3, acetone.*

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INTRODUCTION

Acetone, NH3, LPG (Liquefied Petroleum Gas) are hazardous air pollutants. LPG is inflammablegas. It is used as an explosive fuel in both industrial and home settings. It is called to be cooking gas. Cooking gas contains mainly butane [1], odorless, colorless; nontoxic gas. LPG is a good solvent for rubber and petroleum compounds, while steel and copper alloys are typically non-corrosive. It is commonly combined with sulfur (methyl mercaptan ethyl mercaptan) compounds that have a foul smell. Sothat any leakage may be immediately detected. This gas is possibly dangerous since it has the potential to produce explosions if it leaks out by accident. It was noted that at concentrations up toa certain point, there is significant leakage. It's much greater than the gas's lower explosive limit (LEL) in air. As a result, there is a significant need for monitoring it, as well as new problems [2] in the domestic and industrial fields for control and safety applications. Several well recognized materials for NH3 & LPG gas sensingare ZnO [3-7] SnO2 [8,9,10], Ru-SnO2 [11] SnO2-Cu [12,13] TiO2 [14]. Ammonia is produced by the decomposition of proteins in the bodies of plants and animals. Ammonia has a characteristic of pungent smell like public urinals, cattle yards. Ammonia gas is neither combustible nor a supporter of combustion. The physical properties of Ammonia are colorless gas having a characteristic pungent smell, lighter than air, highly soluble in water, high toxicity, easily liquefied cooling by compressing. It is used in the manufacture of fertilizers like ammonium sulfate and ammonium nitrate, medical diagnosis, chemical technology, food process, firepower plants, environmental protection, as a reducing reagent, laboratory reagent, and good solvent.

Gas sensors detect the composition and concentration of gas by converting it into an electrical signal via chemical and physical effects. Gas sensors are often used in the detection of explosives, flammables, harmful & toxic gases, and environmental management [15]. Lately, the majority of gas sensors were surface-controlled resistance sensors, whereas semiconductor resistance gas sensors mostly utilized metal oxide semiconductors as their sensitive material[16]. Because of the enormous number of free electrons within the conduction band as well as oxygen vacancies in the surface, the metal semiconductor's material surface has high adsorption capabilities and high reactivity, and it is affected by surface gas action. Therefore, measurements based on electrical properties are possible [17]. Metaloxide materials have good chemical and physicalcharacteristics, are inexpensive to create and

have easy production procedures [18]. Thus, they were becoming more popular in gas sensing. Metal oxide (SnO_2, WO_3, NiO, etc.) real-
time gas sensors have become more popular in recent years owing to their high sensitivity, short reaction times, and ability to choose many gases (LPG, CO, H_2, etc.) Rare earth metal doping is a highly efficient way for adjusting and controlling AlO_3 gas sensing capabilities, as shown in past studies.
The purpose of the current work is to study the electrical and structural properties of $DyalO_3$ nanoparticles. To study characterization, TGA, XRD, FESEM, EDAX. To Prepare
$DyAlO_3$ pellets by the hydraulic press and to investigate their sensing properties for LPG, NH_3, and acetone gas.

MATERIAL AND METHODS

Synthesis of $DyalO_3$ NP's: $DyalO_3$ NPswere synthesized by the auto combustion method. The Dy (0.5) Al (0.5) O_3 powders were prepared using Aluminum Nitrate Al $(No_3)3.9H_2O$, [Molychem], "Dysprosium (III) Nitrate pentahydrate" Dy $(NO_3)3.5H_2O$ [Alfa Aesar], Citric Acid $C_6H_8O_7.H_2O$ [Molychem] and double-distilled water. Citric acid has been employed as a chelating agent because it aids in the development of a homogenous mixture(Metal cations).
The process of reaction was conducted at room temperature in an air environment. The prepared solution was stirred on a magnetic stirrer to form a gel at 80° temperature. The prepared powders were annealed at 600° and 750°.
The crystalline structures of the nanostructures prepared from $DyalO_3$have been investigated using X-ray powder diffraction (Philips EXPERT MPD) to make X-ray diffractometer with CU-Kα radiation (λ=0.154nm). The morphologies and microstructures of produced samples were described employing FESEM: "Field Emission Scanning Electron Microscope" (FESEM make Model of FEI: Apreo Lovac Retractable STEM 3+Detector, Aztec standard EDS, Detector of DBS, System–resolution 127ev on Mn-Kα; Leica ultra-Microtome EM UC7 (sputtercoater) with EDS ("Energy Dispersive X-ray Spectroscopy").

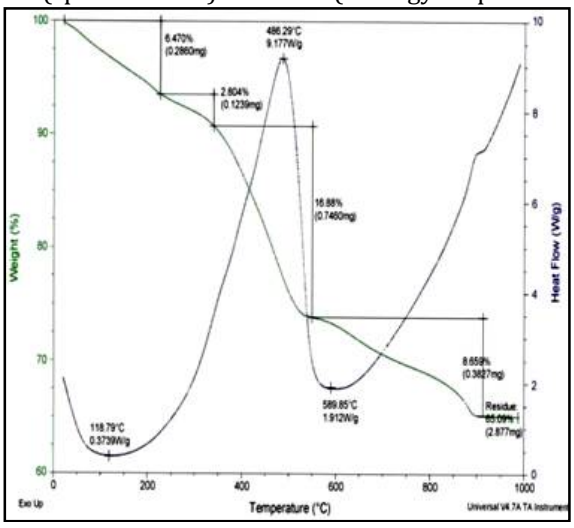

Figure: 1 - DSC - TGA of $DyalO_3$

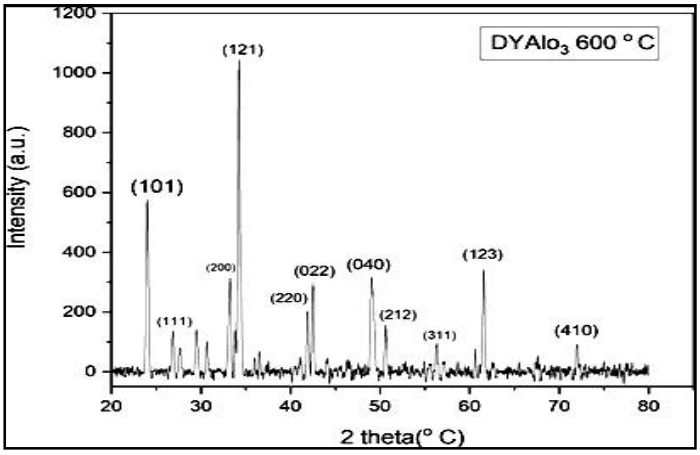

Figure: 2a. XRD of $DyalO_3$ at 600 ^{0}C

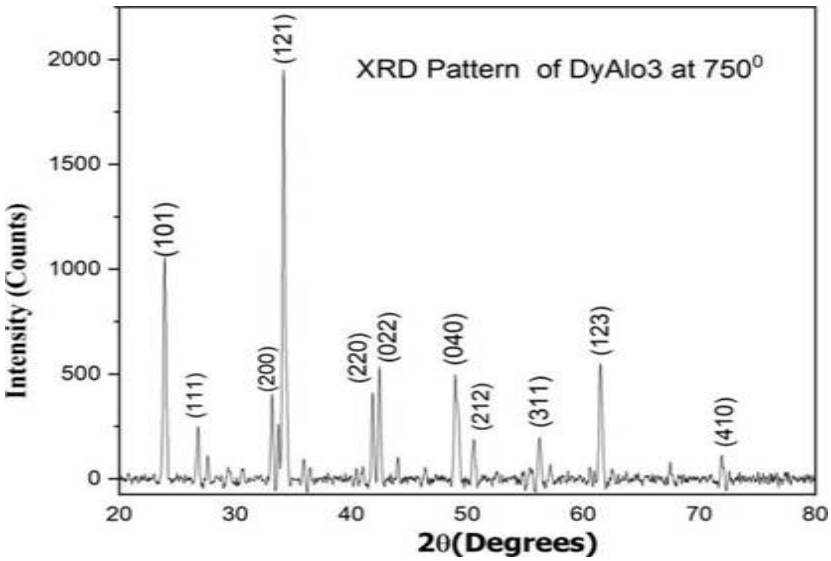

Figure: 2b. XRD of Dyalo3 at 750 ^{0}C

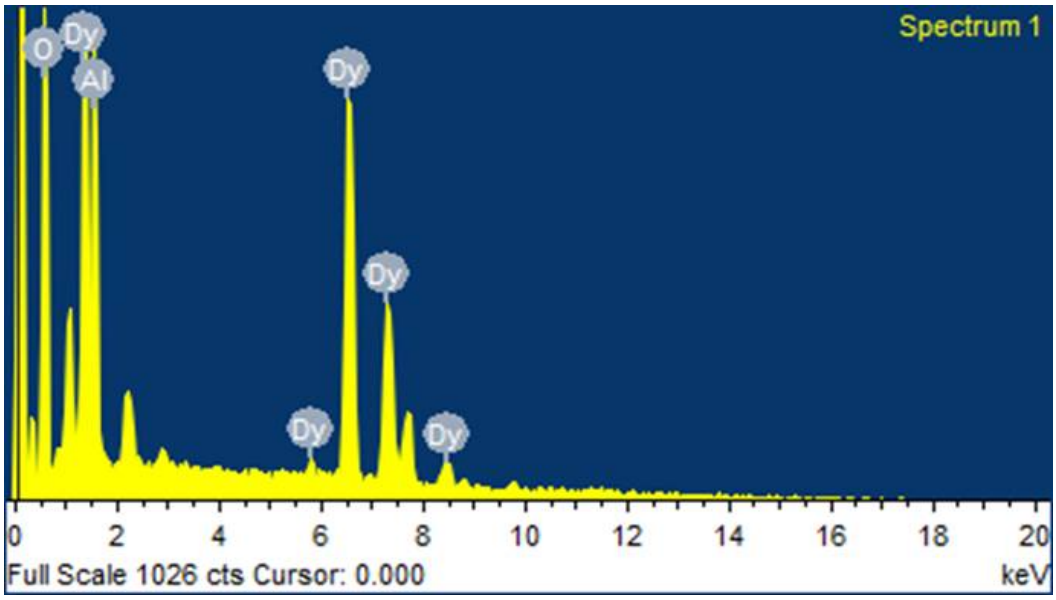

Figure 3: EDS spectrum of $DyAlO_3$ nanostructure

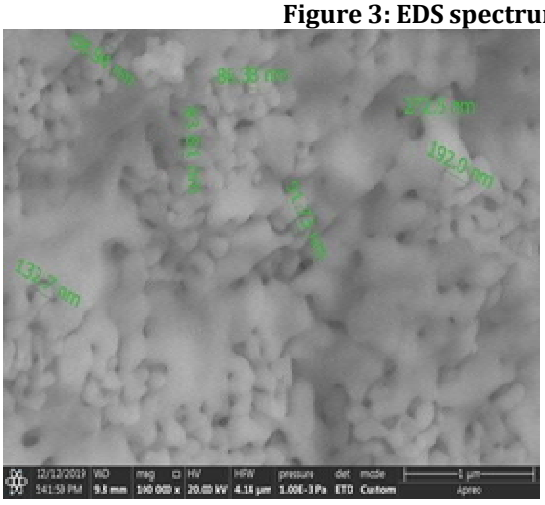

Figure 4.a

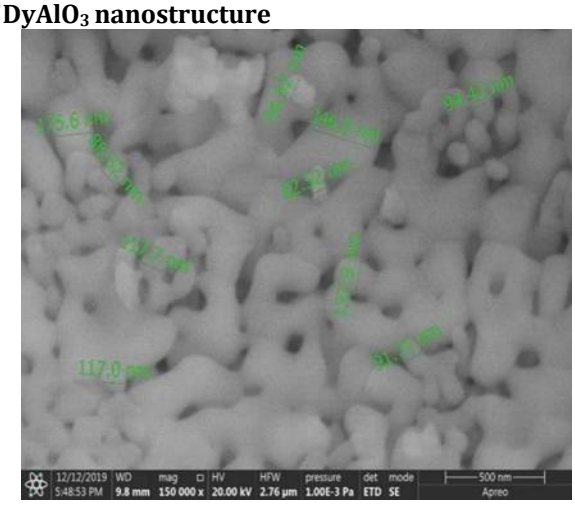

Figure 4.b

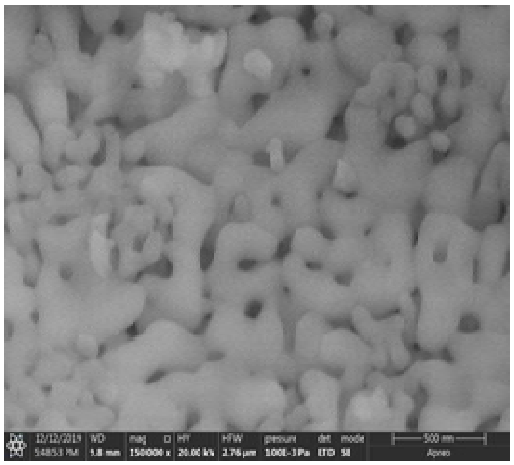

Figure 4.c

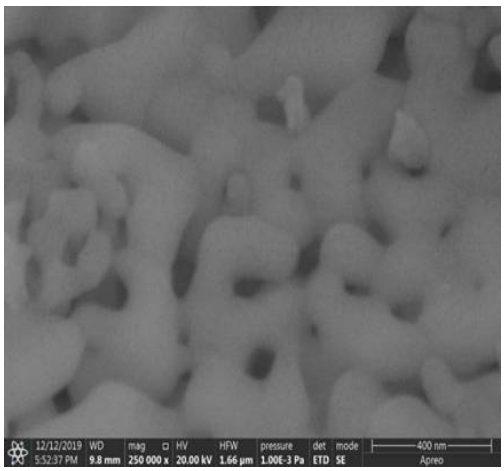

Figure 4.d

Fig 4: FESEM images of $DyAlO_3$

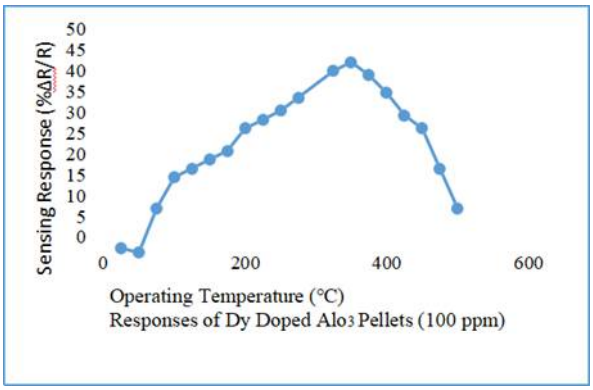

Figure: 5a (NH_3)

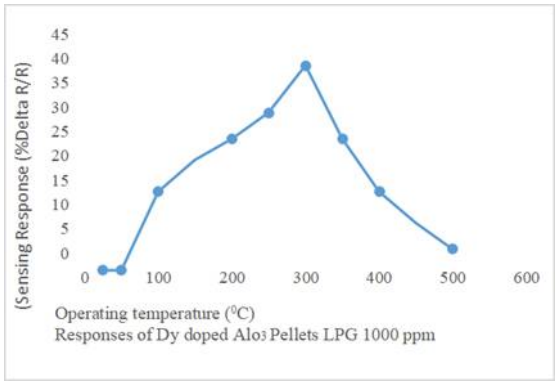

Figure: 5b (LPG)

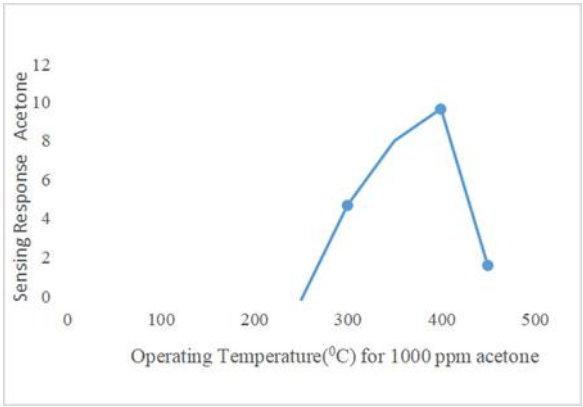

Figure: 5c (Acetone)

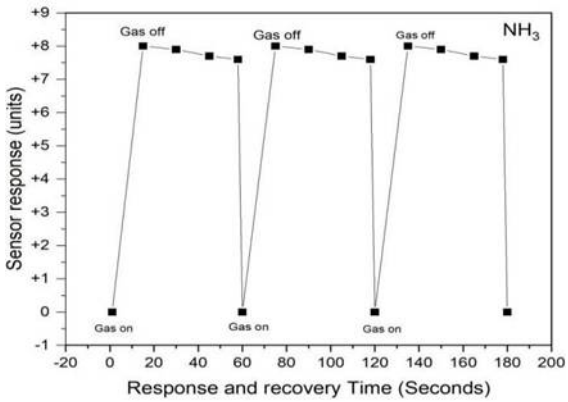

Figure 6: 100 ppm (NH_3)

RESULTS AND DISCUSSION

Thermo-Gravimetric analysis of DyAlO3 particles figure 1 indicates the weight loss rate of the particles as a temperature function. The particles indicate a weight loss (6.470%) between 0°C & 200°C. Another weight loss (2.804%) from 200 to 350 °C. The greater weight loss rate (16.88 %) was found between 350°C and 550°C, which might be due to the physically adsorbed water evaporation on the surface and in particles pores. Decomposition of Dy-Al Hydroxide may cause further weight loss (8.659 percent) between 550°C and 900°C. Above 900°C, the TGA curve does not show any weight loss. The X-ray diffraction study conducted on the prepared $DyAlo_3$ showed that the acquired $DyAlo_3$ powder is having a crystal system orthorhombic structure, space group Pnma with an excellent crystalline character as per standard data (JCPDS, card file no. reference code 00- 039- 1437). Figure 2a and figure 2b show the XRD pattern of $DyAlo_3$ NPS. No distinctive peaks of any impurities were identified, indicating that good quality of $DyAlo_3$ NPs was produced. The crystallite size was assessed from the Pattern of XRD using

Scherrer's eq d=kλ/βcosθ [8].
Here k=0.9 denotes the shape factor, λ indicates the CUKα radiation's (1.5406A°) X- ray wavelength, β signifies the FWHM of the respective diffraction peak & θ denotes the Braggs diffraction angle. The average size of Crystallite that corresponded to the XRD peaks was determined to be 29.30 nm. The Nanocrystalline structure of DyAlo3 NPs was indicated by the existence of strong structural peaks in XRD patterns and crystallite sizes smaller than 100 nm. Figures 4(a), (b), (c) & (d)indicate the typical FESEM images of DyAlo3 NPs respectively. The minimum diameter of Dyalo3 NPs came out to be 68.94 nm.
EDX analysis was used to validate the production of DyAlo3 Nanocomposite. During EDX measurement various zones were focused as well as corresponding peaks are revealed in figure 3. The Spectra shows both Dy and Alo3 in the produced composite nanostructure. In spectrum, the quantity of Dy, Al, and O was 15.38, 19.01, and 65.62, respectively, measuredin atomic %. Sustaining the XRD findings the EDS Spectra of $DyAlo_3$ NP's is given in Figure 3. The EDS findings reveal that there are no additional elemental impurities in the prepared $Dyalo_3$ NPs. The sensing response againsttemperature for NH3 is revealed in figure 5a, the highest sensing response for 100 ppm was obtained at 350 ^{0}C. For LPG maximum sensing response was obtained for 1000 ppm at 300^{0}C as shown in figure 5b. For acetone maximum sensing response was obtained for 1000ppm at 400^{0}C as shown in figure 5c. The response of the DyAlo3 pellet was found to be quick (~3s) to 100ppm of NH3, but the recovery was quic ~30s) figure 6. The rapid oxidation of the gas may be responsible for the quick reaction.

CONCLUSION

In this work dysprosium doped Aluminium trioxide nanoparticles can be successfully synthesized by auto combustion approach and carefully described by TGA, XRD, EDS, FESEM, SEM respectively. Theaverage crystallite size determined to correlate to the peaks seen in XRD is 29.30nm. The sensing response for NH3 was maximum at 350°C operating temperatures for 100 ppm. Thesensing response for LPG was maximum at 300°C operating temperature for 1000 ppm. The sensing response for acetone was maximum at 400°C operating temperature for 1000 ppm. All results indicate that the present DyAlo3 Pellet showed maximum response for high temperature.

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