INVESTIGATIONS AND COMPARISON OF THERMOGRAVIMETRIC AND DIFFERENTIAL THERMAL ANALYSIS OF MOLYBDENUM TRIOXIDE AND ITS NANOCOMPOSITE POWDERS

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ABSTRACT

The objective of the present work was to investigate and compare thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) of MoO₃, MoO₃-CuO, MoO₃-V₂O₅ and MoO₃-TiO₂ composite nano powders using ball synthesis techniques at different intervals of time. The synthesized composite nanopowders were characterized by spectroscopic characteristics such as Powder X Ray diffraction (PXRD), Scanning Electron Microscopy (SEM), Electron Dispersive X Ray Spectroscopy (EDS), Thermo gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA). The orthorhombic phase of V₂O₅, MoO₃, TiO₂ and monoclinic phase of CuO were revealed by X-Ray peak profile demonstrates. MoO₃, MoO₃-CuO and MoO₃-TiO₂ SEM photographs Indicate the unusual particle forms and MoO₃-V₂O₅, show abnormal circular structures of vanadium flakes deposits. The EDS spectrum confirms the presence of the elements of the prepared samples. From the thermal analysis it has observed that thermo gravimetric and differential thermal analysis plots for MoO₃ - CuO nanocomposite powders shows high thermal stability up to 7100C as there is a negligible weight loss which gives the better results. The as-synthesized nanocomposites can also be used at a high temperature as stable lubricants.

KEYWORDS: MoO₃, Ball Milling, Nanocomposite, TGA and DTA

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INTRODUCTION

Because of their interesting applications as catalysts, transitional metal oxides as new class of research materials, and sensors as well as photchromatic appliances. Molybdenum oxide (MoO₃) is desirable among such products due to its structural and optical properties [1-2]. Nanotechnology also offers a wide range of tribological research. In general, molybdenum oxide exhibit allotropy (exist as 3 distinct phase structures) as stable orthorhombic (α -MoO₃), Monoclinic (β - MoO₃) and Hexagonal (h-MoO₃) [3]. It has a significant area of applications such as smart material (photochromatic material), lubricants and catalysts. MoO₃ is a solid lubricant because its coefficient of friction at high temperatures of approximately 645°C is excellent. MoO₃ has been analyzed for friction and wear at high temperatures with aluminum bronze. Significant results were achieved. MoO₃-bases nano for their superior tribological properties, the composites MoO₃-PbO and MoO₃-ZnO are studied at high temperatures. MoO₃ has many applications due to its special thermal, mechanical, magnetic or electrical properties in addition to its frictional properties [4-6].

Due to their significant friction reduction properties, copper oxide nano powders can also be used as solid (stable) lubricants. Most lamellar / layered solid lubricants fail in open air and at elevated temperatures of above

495 °C for lubrication applications as they become oxides [7]. A mixed oxide can be used for improved friction outputs at various high temperatures such as MoO_3/CuO alloy or composite [8-9]. Titanium dioxide (TiO_2) is widely used as an opacifier in lubricants and lubricant additives and other applications, and for its ultraviolet - resistant properties where the powder disperses light, unlike organic ultraviolet absorbers, and reduces ultraviolet damage, mainly due to the higher particulate matter [10-11]. Vanadium Pentoxide is a semi-conductive material which, in addition to solid state chemistry and material science, has a lot of interest in tribology [12]. Vanadium catalysts – based on the availability of multiple Oxidation States and therefore the potentially interesting vanado catalysts are known to be effective in oxidation chemistry [13-15]. The method of ball milling, chemical and physical vapor deposition, sol - gel method, etc., are different methods of synthesis to obtain nanocomposites. The aim of the present work was to compare thermo gravimetric analysis and differential thermal analysis of various molybdenum trioxide nanocomposites and its allied mixtures synthesized by the process of planetary ball milling [16].

MATERIALS AND METHODOLOGY

Molybdenum tri oxide, Vanadium Pentoxide, Copper Oxide and Titanium dioxide were procured from Merk chemicals, Mumbai. The chemical reagents are analytical and used without further purification. The reagents (chemical) used in the synthesis are with purity above 99.5 %. Oxides of Molybdenum, Titanium, Copper and Vanadium powders of mean size 10 microns were selected. Weight ratios of oxides of Molybdenum and copper as 2.3:1 & 3.3:1, 1:1 ratio for oxides of Molybdenum, Copper and Vanadium were homogeneously mixed separately [17-18]. As shown in Fig.1, mechanical synthesis was done with a 1:10 powder / ball ratio. The milling was performed for two hours with intervals of five minutes for every twenty minutes and a rotational velocity of 250 rpm respectively. A synthesis process is made from the ball mill of 100 ml bottle (vial) and Zirconium balls [19]. The mixtures obtained were taken in a crucible and calcinated at 250 °C in the air for 2 hours.

Figure 1: Planetary Ball Milling Equipment.

RESULT AND DISCUSSION

Charaterisation

Powder X-Ray Diffraction Patterns

Powder X-Ray diffraction (XRD) patterns of MoO_3 , $\text{MoO}_3 - \text{CuO}$, $\text{MoO}_3 - \text{TiO}_2$ and $\text{MoO}_3 - \text{V}_2\text{O}_5$ nanocomposite powders were carried on a XPert Pro-diffractometer with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.56\text{\AA}$). The measurements were taken at room temperature from 10° to 80° on position (2θ) with a step size of 0.1° in 50 KeV, 10mV current[20-21]. Various XRD patterns are as shown in Fig.2 to Fig.5.

Figure 2: X-Ray Diffraction Peaks of Ball Milled MoO_3 Nano Powders.

Figure 3: X-Ray Diffraction Peaks of Ball Milled $\text{MoO}_3 - \text{V}_2\text{O}_5$ Composite Nano Powders.

Figure 4: X-Ray Diffraction Peaks of Ball Milled $\text{MoO}_3 - \text{CuO}$ Composite Nano Powders.

Figure 5: X-Ray Diffraction Peaks of Ball Milled $\text{MoO}_3\text{-TiO}_2$ Composite Nano Powders.

Synthesised nano powder diffraction peaks are well-suited with regular JCPDS data diffraction files [22]. With respect to the milling time, no variation in phase structures are observed.

Scanning Electron Microscopy with EDS Patterns

SEM micrographs were taken from a CarlZeiss SEM and EDAX pictures were taken from ZESS EVO20.

Figure 6: Schematic Representation of the Basic SEM Components.

SEM equipment morphology studies as shown on Fig.6 are observed in SEM pictures [23]. Scanning electron microscopy is a significant method for the topography of sample characterization and provides information on the mechanism of growth, shape and dimensions of particles. Various SEM images of MoO_3 , $\text{MoO}_3\text{-CuO}$, $\text{MoO}_3\text{-TiO}_2$ and $\text{MoO}_3\text{-V}_2\text{O}_5$ nanocomposite powders are as shown below from Fig.7 to Fig.11[24-25].

Figure 7: Scanning Electron Microscope Images (a, b, c) and EDS Pattern(d and e) of MoO₃ Composite Powders.

Figure 8: SEM Images and EDS Pattern of MoO₃-V₂O₅ Composite Nano Particles.

Figure 9: (a, b, c, d) SEM Images of 2.3:1 and 3.3:1 MoO₃-CuO Composite Nano Particles.

Figure 10: Electron Dispersive Patterns of a) 2.3:1 and b) 3.3:1 MoO₃-CuO Composite Nano Particles.

Figure 11: SEM Images and EDS Patterns of MoO₃-TiO₂ Composite Nano-Powders.

Morphology obtained from MoO₃, MoO₃ - CuO, MoO₃ - TiO₂ SEM pictures confirm irregular forming particle size and display irregular spherical shaped structures and in MoO₃ - V₂O₅ deposited with vanadium flakes[26, 30,31]. The EDS spectrum confirms the fact that the samples prepared are constituent elements.

Thermogravimetric and Differential Thermal Analysis

Thermogravimetric analysis (TGA) and Differential Thermal analysis (DTA) play a critical role in understanding the thermal stability and relative loss of weight of the material [27-28]. The analysis are performed under nitrogen atmosphere of 25 cm³ min⁻¹ at a ramp rate of 5°C min⁻¹ using TGA/DTA instruments SDT Q500. The sample weighs around 10 milligrams, the thermogravimetric and differential thermal analysis[29-30] plots of MoO₃, MoO₃ - CuO, MoO₃ - TiO₂ and MoO₃ - V₂O₅ are as shown in the Fig.12 to Fig.15(a,b).

Figure 12: Thermogravimetric and Differential Thermal Analysis Plots of MoO₃ Nano Particles.

MoO₃ nano powders demonstrate excellent thermal stability up to 638⁰C with a marginal 0.75 percent weight loss

Figure 13: Thermogravimetric and Differential Thermal Plots of MoO₃-V₂O₅ Nano Particles.

MoO₃ –V₂O₅ shows a negligible weight loss of 2.3% with an excellent thermal stability at 672⁰c.

Figure 14: Thermogravimetric and Differential Thermal Plots of MoO₃-TiO₂ Composite Nano Particles.

MoO₃ – TiO₂ composite nano particles has a good thermal stability with a weight loss of 2.2 % at 695⁰c

Figure 15: a) TGA Curve and b) TDA Curve of MoO₃-CuO (3.3:1 and 2.3:1) Composite Nano Powders.

MoO₃ and CuO (with weight ratios 3.3:1 and 2.3:1) nano composite powders are stable upto 710⁰ C as there is a insignificant weight loss.

CONCLUSIONS

To sum up, the current experiment has been conducted in a cost - effective manner to produce MoO₃, MoO₃ — CuO, MoO₃ — TiO₂ and MoO₃ — V₂O₅ composite nano powders for industrial commercial use. We successfully synthesized composite nanopowders using the simple and cost - effective mechanical activation method. At different times, powders were collected and the mechanical milling effect on the nano composite was performed to PXRD and no structural changes were observed with respect to the grinding/milling level. For topography study of the samples SEM analysis is important for characterizing technique and provides information about the mechanism of growth, shape and the size of particles. The TGA and DTA curves of MoO₃ and CuO ball milled synthesized composite nano powders at 4 hours show great thermal stability to 710⁰ C, as weight loss is negligible. The synthesized thermally stable MoO₃ - CuO nano composites are therefore suitable for use at elevated temperatures in metal insulating systems, semiconductors and solid lubricants.

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