Synthesis of Nano Platinum-Tungsten Supported on Gamma-Alumina Catalyst

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ABSTRACT

Nano gamma-alumina support was prepared successfully by Sol-gel method using aluminum nitrate without and with carbon nanotubes as a template at different calcination temperatures (600, 650, and 700°C) and then loaded with 0.3% of platinum and 0.3% of tungsten by the wet-impregnation method. X-ray diffraction (XRD), thermal gravimetric analysis (TGA), atomic force microscope (AFM), field emission scanning electron microscopy (FE-SEM) and (BET) surface area were used to characterize the prepared nano catalyst. The best results obtained were 389.34 m²/gm surface area, 0.468 cm³/gm pore volume, 8.07 nm pore size and 42.71 nm average particle size for the prepared nano gamma-alumina at a calcination temperature of 600°C.

Keywords: Gamma-alumina, Carbon nanotubes, Sol-gel method, Wet-impregnation method.

INTRODUCTION

The support material is often the amorphous pored refractory oxide and generally without crystalline molecular sieves, was calcined at a temperature above 800°F such as (Al₂O₃, silica-Al₂O₃, silica, γ-Al₂O₃, theta-Al₂O₃, zirconia, titania, and magnesia), but γ-Al₂O₃ is widely utilized...
because it has a high surface area [1,2]. There are many methods to prepare nano gamma aluminan such as: Sol-gel, hydrothermal, precipitation, laser ablation, sprays pyrolysis, and solution combustion [3-5]. Morphology of gamma alumina can be modified by changing some conditions of the preparation reaction like: temperature, pressure, reaction time, pyrolysis methods, type of preparation technique, and materials used in the preparation. The catalytic specifications of gamma alumina support depend on some important properties like: surface area, porosity, pore volume and particle size [6]. Gamma alumina is used in the synthesis of the catalyst as its support because of its high thermal stability and ability to form into parts small (spherical or cylindrical) mechanically stable accordingly, gamma alumina is used as catalytic support in different types of chemical reactions [7]. Active gamma alumina is usually used as a catalyst, catalyst auxiliary, supports, and adsorption. The method and techniques used in preparing alumina as well as the calcination temperature have a significant impact on its surface area and histological composition that controls the extent of activity of alumina gamma[8]. The nanoparticle alumina support is probably to give high loading of active metals, as well as high surface area, and high pore volume [9]. Modern research lacks techniques to exploit the unique properties of many nanomaterials, besides this challenge, there is a need to link these nanomaterials with microscale and macroscale platforms. The materials used in the preparation of alumina have shown special interest in carbon nanotubes (CNTs), due to the role that (CNTs) play in improving physical properties like reducing particle size and increasing surface area [10]. The main obstacle to using carbon nanotubes is their dispersion. Because carbon nanotubes lean around to conglomerate via van der Waals force, therefore a surfactant material such as (SDS) was added to it as a dispersion, this is to prevent occur agglomeration for CNTs [11-13]. One of the most important things in the performance and strength of the catalyst is the choice of a correct technique in preparing the support as well as choosing the type of support well. Alumina is used as a support on a large scale because it has many characteristics: cheap price and can be provided from several sources and different phases as well as stability and good surface area through which it can be used in many chemical reactions[8]. Nano catalysts have important properties, which are high surface area and high pore volume, compared to traditional catalysts. The effectiveness of the catalyst depends on the properties of its structure so that the particle size in the nano scale has the largest surface area and the severe possible reaction, this is the catalyst target [14]. The catalyst activity based on the surface area. If the catalyst has a high surface area leads to a higher number of sites for the chemical reaction, and the decrease in the particle size leads to an increase in the surface area while maintaining the same total mass of the material
without change [15]. Active ingredients are ingredients such as platinum (Pt), straightly assist in realizing the catalyst purposes, the percentage of these ingredients loaded on the support is responsible for the activity, selectivity, and service lifetime of the catalyst [16]. The promoter is a chemical substance added to the catalyst to improve the activity of active metals in a chemical reaction and increase the lifetime of the catalyst. Some types of promoters react with the active ingredients of the catalysts and this leads to a change in the chemical effect on the catalyst. The reaction may be caused by a change in the crystal or electronic configuration of the active part [17]. The addition of tungsten (W) to platinum (PT) is a successful promotion of a catalytic reformer unit as it has improved performance significantly and this has been shown by the low precipitation of coke on the catalyst surface, high thermal stability for catalyst and resistance to high operating temperatures. Perhaps this high catalyst stability is due to the formation of mixed surface oxides of W6+ robustly connected with Al3+ stabilization the alumina surface, this works against the rearrangement of the crystal structure at high operating temperatures, as well as the positive effect of tungsten against platinum sintering [18]. This work aims to prepare nano gamma alumina then use it in the preparation of nano Pt-W/\gamma-Al2O3 catalyst that can be used instead of the conventional catalyst used for catalytic naphtha reforming.

**EXPERIMENTAL SECTION**

**Materials**

Aluminum nitrate Al(NO3)3·9H2O (98% purity, THOMAS BAKER/India) was used as sources of aluminum, sodium dodecyl sulphate (SDS) CH3(CH2)11OSO3Na (90% purity, Central Drug House (CDH) /India) used as a surfactant, ammonia solution NH4OH(25% purity, THOMAS BAKER /India) and nitric acid HNO3 (69-72% purity, LOBAL Chemie/India) were used to adjust the pH of the mixtures, multi wall carbon nanotube (MWCNTs) (90% purity, Cheap Tubes/USA) was used to improve the surface area of alumina, chloroplatinic acid H2PtCl6. 6H2O salt (≥ 99.995 % purity, British Drug House (BDH) Company) was used as sources of platinum metal, and finally ammonium tungsten (NH4)10W12O41·5H2O salt (≥ 99.99 % purity, HOPKIN & WILLIAMS LTD. Company) was used as sources of tungsten metal.
Preparation of Nano Gamma-Alumina

Aluminum nitrate $\text{Al(NO}_3\text{)}_3\cdot9\text{H}_2\text{O}$ (73.6 gm) was dissolved in 1500 ml deionized water with stirring at temperature 80˚C for 1hr and adjusting the pH to equal 8 for the solution by addition the ammonia solution 25 wt.% . The resulting solution precipitates for 24 hr, then filtered using a vacuum system in order to produce a white gel. Preparation of a first solution consisting of multi-wall carbon nanotubes (MWCNTs) in different three ratios: 1vol%, 3vol% and 5vol% with 100 ml deionized water and 5gm of sodium dodecyl sulphate (SDS) as a dispersed, in addition to this, an ultrasonic device was used for 40 minutes to disperse multi-wall carbon nanotubes in the solution. Preparation of a second solution consisting of 1500 ml of deionized water, then add nitric acid drops to it and adjusting the pH to equal 4, then add the second solution to the previously formed white gel with stirring continuous. The resulting solution is gradually added to the first solution previously prepared with stirring and adjusting the pH to equal 4 by adding nitric acid drops at temperature 80˚C for 1hr, the final solution is filtered using a vacuum system in order to produce a gel. Finally the resultant gel was dried at 120˚C for 24 hr and then calcination at different temperatures (600, 650, and 700 °C) for 5 hr a heating rate of 10˚C/min.

Preparation of Pt-W/γ-Al$_2$O$_3$ Nan Catalyst

Pt-W/γ-Al$_2$O$_3$ catalyst were prepared by wet impregnation method. A 60 g of nano γ-Al$_2$O$_3$ powder as support previously prepared was loaded with 0.3wt% of platinum metal using $\text{H}_2\text{PtCl}_6$ 6H$_2$O salt as the metal precursor, the platinum salt was dissolved easily in an amount of deionized water depending on pore volume of support nano γ-Al$_2$O$_3$, producing a yellow to orange solution then addition the solution Pt to powder nano γ-Al$_2$O$_3$ with stirring at room temperature for 24 hr. It was observed that approximately six hours after the mixing process, the color of the solution almost disappeared indicating that the largest amount of the platinum salt compound had been impregnated, subsequently filtered by a vacuum system then dried at 110 °C for 24 hr and calcination at 450 °C for 4 hr a heating rate of 10˚C/min. The resulting powder was loaded with 0.3wt % tungsten metal using (NH$_4$)$_{10}$W$_{12}$O$_{41}.5$H$_2$O salt as the metal precursor with the same steps as platinum metal loading, also dried at 110 °C for 24 hr and calcination at 450°C for 4 hr at a heating rate of 10 °C/min [19-21].

RESULT AND DISCUSSION

Characterization of Nano Gamma-Alumina and Pt-W/γ-Al$_2$O$_3$ Nano Catalyst
XRD Analysis

According to the XRD spectrum standard $\gamma$-alumina Card (ICPDS-files no.29.0063) the comparison of the XRD spectrum with the standard XRD spectrum reveals that all peaks of prepared gamma-alumina without addition MWCNTs at calcination temperature 600°C as shown in Fig. 1 were approached the standard gamma-alumina, among them, three strong peaks (311-65b intensity-2.39 d spacing), (400-80b intensity-1.98 d spacing) and (440-100b intensity-1.4 d spacing) were accepted.

![Figure 1. XRD spectrum for nano gamma-alumina without addition calcination of (MWCNTs) at temperature 600°C.](image)

When preparing gamma-alumina with multi-walled carbon nanotubes as shown in figures 2 to 4, the comparison XRD spectra for prepared alumina samples with the (XRD) spectrum standard gamma-alumina reveals that all peaks of prepared samples of gamma-alumina with the addition of MWCNTs were similar to the standard gamma-alumina. This means that the prepared $\gamma$-alumina samples at different calcination temperatures (600, 650 and 700°C) were not affected their structure with the addition of multi-walled carbon nanotubes.

![Figure 2. XRD spectrum for gamma-alumina with addition of MWCNTs at temperature 600°C.](image)
The difference in the height of getting peaks perhaps may be due to the change in the calcination temperature used during nano gamma-alumina preparation with MWCNTs. On the other hand, the addition of MWCNTs to nano gamma-alumina may lead to reduce the intensity of all peaks in the test X-ray diffraction according to [13].

The purity of solid crystal was measured from comparing the X-ray diffraction pattern of prepared nano gamma-alumina with MWCNTs at various calcination temperatures with X-ray diffraction of standard $\gamma$-alumina shown in table 1. This evaluation demonstrated that the prepared gamma-alumina at calcination temperature 600°C is all the more closely to the standard gamma alumina.
The broadening of the peaks may be occurred due to the role of the SDS as surfactant used in the process preparation alumina which was released after calcination producing nanoparticle size for alumina [22,23].

Table 1. Comparison between standard gamma alumina and prepared gamma alumina with addition (MWCNTs) at different calcination temperatures.

| Angle (2θ) | d, spacing (Å) | Angle (2θ) | d, spacing (Å) | Angle (2θ) | d, spacing (Å) | Angle (2θ) | d, spacing (Å) |
|------------|----------------|------------|----------------|------------|----------------|------------|----------------|
| 20.462     | 4.33           | 20.860     | 4.25           | 20.636     | 4.30           | 20.012     | 4.43           |
| 31.936     | 2.80           | 31.541     | 2.83           | 31.241     | 2.86           | 31.753     | 2.81           |
| 37.603     | 2.39           | 37.557     | 2.39           | 37.111     | 2.42           | 37.385     | 2.40           |
| 39.401     | 2.28           | 39.513     | 2.27           | 39.309     | 2.29           | 39.234     | 2.29           |
| 45.964     | 1.98           | 46.030     | 1.97           | 45.767     | 1.98           | 45.830     | 1.97           |
| 60.457     | 1.53           | 60.284     | 1.53           | 60.974     | 1.51           | 60.724     | 1.52           |
| 66.761     | 1.40           | 66.572     | 1.40           | 66.793     | 1.39           | 66.772     | 1.39           |

Thermal Gravimetric Analysis

Thermal gravimetric analysis was performed for the sample MWCNTs commercial type, shown in Fig. 5, this can be explained by the gradual rise in temperature of the MWCNTs sample, accompanied by a loss of weight until 96.672% from MWCNTs is burned, it was combustion temperature up to 577 °C, therefore nano gamma alumina with (MWCNTs) was calcination at 600°C to ensure the content of (MWCNTs) was removed completely from alumina.
Surface Area and Pore Volume Analysis

The surface area of gamma-alumina powders with and without addition 3vol% of MWCNTs at different calcination temperatures are presented in Table 2 from this table it is observed that by addition carbon nanotubes the surface area of gamma-alumina powders increases and decreases, respectively, while the increase of calcination temperature has reverse effects [24]. The increase of specific surface area of gamma-alumina powders by addition MWCNTs can be attributed to the high-surface area of MWCNTs. The other possible reason is that the coarsening of alumina particles is prevented by resting the MWCNTs between particles during calcination. The increase of calcination temperature coarsens the particles and subsequently decreases the surface area of the \( \gamma \)-alumina powders [13].

Table 2. Surface area of nano gamma-alumina powders with and without MWCNTs

| Samples                  | Calcination temperature | Surface area (BET) $m^2/gm$ |
|--------------------------|-------------------------|-----------------------------|
| gamma-alumina            | 500°C                   | 285.8                       |
| gamma-alumina            | 600°C                   | 232.32                      |
| gamma-alumina with 3vol % MWCNTs | 600°C                | 389.34                      |
| gamma-alumina with 3vol % MWCNTs | 650°C                | 330.42                      |
| gamma-alumina with 3vol % MWCNTs | 700°C                | 275.6                       |
Nano Pt-W / γ-Al₂O₃ catalyst was prepared has surface area 340.1 m²/g and pore volume 0.326 cm³/g using gamma-alumina with 3vol % MWCNTs which has a surface area of 389.34 m²/g and pore volume 0.468 cm³/g. The decrease in surface area, and pore volume probably due to blockage some pores due to loading Pt, W on the prepared support gamma-alumina during wet- impregnation method as mentioned by [25,26].

**Atomic Force Microscope Analysis**

The morphology of the surface and average particle size for prepared gamma alumina was studied using (AFM), figures 6, 7 show the images of AFM on two-dimensional surface profile, irregular hexagonal structure of gamma-alumina crystal when alumina without addition MWCNTs and alumina with addition MWCNTs at calcination temperature 600°C respectively, while figures 8, 9 show AFM images for three-dimensional surface profile and indicate that low agglomerated of gamma-alumina crystal are in form of irregular hexagonal layers crystal at alumina without additional MWCNTs and alumina with addition MWCNTs at calcination temperature 600°C respectively. The prepared gamma-alumina crystal without addition MWCNTs at calcination temperature 600°C consisted of particles with diameters ranged between 25-115 nm and average diameter 72.12 nm, these are results agreeing with those obtained by the previous author [27,28].

![Figure 6. Two-dimensional surface of nano gamma- alumina with MWCNTs.](image1)

![Figure 7. Two-dimensional surface of nano gamma- alumina without MWCNTs.](image2)
Figure 8. Three-dimensional surface of nano gamma alumina without MWCNTs. Figure 9. Three-dimensional surface of gamma-alumina with MWCNTs.

The prepared nano gamma-alumina crystal with addition MWCNTs at calcination temperature 600°C consisted of particles with diameters ranged between 26-60 nm and an average diameter of 42.71nm. This change is explained by the average diameter of the particles due to the addition of multi-wall carbon tubes that reduce the particle size according to [13,29].

The AFM images of prepared nano Pt-W/γ-Al₂O₃ catalyst as revealed in figures 10, 11. The two-dimensional surface images in Fig. 10 show the irregular hexagonal structure of the prepared nano catalyst crystal. Three-dimensional surface images shown in Fig. 11 indicate that agglomerated of prepared nano catalyst crystal are in the form of irregular hexagonal layers, also spherical and rough in shape [27,28].

Figure 10. Two-dimensional surface of prepared Pt-W/γ-Al₂O₃ nano catalyst Figure 11. Three-dimensional surface of prepared Pt-W/γ-Al₂O₃ nano catalys

The prepared nano Pt-W/γ-Al₂O₃ catalyst consisted of particles with diameters ranged between 40-90 nm and the average diameter was 64.64 nm. The increase in average particle diameter of the prepared catalyst occurs due to loading Pt, W on the prepared support gamma-alumina and crystal centering during wet- impregnation method [17,30,31].

Field Emission Scanning Electron Microscopy Analysis


Field emission scanning electron microscopy (FE-SEM) images of nano gamma-alumina with 3vol % MWCNTs at calcination temperature 600 °C as shown in Fig.12. This figure shows MWCNTs bundles sitting along the nano gamma-alumina grain boundaries also inhibit the growth and congregation of alumina grains during the calcination process, resulting in the refinement of grain size. This is according to [32].

Fig. 12. a, b) FE-SEM images of the prepared nano gamma-alumina with 3vol% MWCNTS at calcination temperature 600°C.

Fig. 13 shows images of the prepared nano Pt -W/ γ-Al₂O₃ catalyst increase particle diameter and growth crystal centering of the prepared catalyst after loading 0.3 wt % of platinum and 0.3wt % of tungsten over nano gamma-alumina [30,31]. It is clear that both (Pt,W) metals were distributed almost regularly on surface gamma-alumina, maybe which is explained by the regular increase in the size of the particles after the loading process. These results are in an agreement with the results obtained from AFM analysis and test of BET surface area.
**CONCLUSION**

The nano gamma-alumina has been prepared successfully using aluminum nitrate by the Sol-gel method. The addition of MWCNTs to nano gamma-alumina as a template improves the surface area to 389.34 m²/gm and the average particle size is 42.71 nm at calcination temperature 600°C but, after loading the Pt-W/γ-Al₂O₃ by using wet-impregnation method decreased the surface area reach to 340.1m²/gm. FE-SEM results show which is highly comparable with the BET surface area and AFM analysis results.

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