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Hydrothermal Preparation of Palladium Supported on Magnetite for Catalysis Applications

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Abstract: ***Herein, versatile, and reproducible method to prepare Pd/Fe_3O_4 via hydrothermal synthesis. The vital role of this catalyst is in its potential use in CO oxidation catalysis. The Pd/Fe_3O_4 shows a distinctive activity. TEM images confirmed that Pd nanoparticles of 8-12 nm have a well dispersion on the surface of magnetite (Fe_3O_4). Moreover, the prepared catalyst is recycled with remarkable catalytic activity. This outstanding activity is mainly a direct result of the strong metal-support interaction. The defect sites in the reduced iron oxide act as nucleation centers that enable anchoring of Pd nanoparticles leading to prevention of agglomeration.***

Index Terms: ***Hydrothermal, Palladium, Fe_3O_4, Nanotechnology.***

I. INTRODUCTION

The synthesis of magnetic nanoparticles has attracted researchers as one of the hot topics in the field of nanostructured materials due to its superior physical and chemical properties; especially when combined with other metal nanoparticles.[1-22]

Heterogeneous catalysis based on metal oxide supports have numerous applications including hydrocarbon refining along with other environmental applications. Carbon monoxide existence is a serious critical issue in pollution control as it can cause severe side effects and even death. [23-30] Hence; there are endless efforts to design new catalysts that could be effectively used in low temperature CO oxidation in order to decrease environmental pollution.[31-36]

The magnetic nanoparticles is considered as a hot topic research area as it is well known that one of the drawbacks in catalysis field is the issues associated with catalyst separation like recovery and recycling from the reaction mixture. Hence, those newly synthesized magnetically recoverable nanomaterials are of vital importance toward development of green chemical synthetic routes as it could be easily recovered using a magnet.[30-34]

There are several challenges in the field of chemical industry like pollution, environmental issues, process safety, and scale-up issues that could be minimized via using micro-reactor technology. [32-35] Palladium-catalyzed reactions are important in the field of organic synthesis although there are some disadvantages of using palladium in such reactions including its high price and also the high toxicity of the metal residue which is considered as a critical issue, especially in the field of pharmaceutical industry. [36-39] Those disadvantages could be minimized by using very small amounts of palladium catalysts, however, there will be still some issues associated with homogeneous catalysis including recyclability issues. [40-42] The key for solving this technical problem could be simply by using heterogenized homogeneous catalyst or heterogeneous catalyst as it is widely known. Heterogeneous catalysis has economic and environmental advantages if compared to the stoichiometric reactions due to the catalyst increasing ability of being easily separated and recycled.[35-44]

Nanotechnology is widely used in several catalysis applications. Tremendous achievements in the research field of air quality and environmental protection using catalysis. [35-40] Fe_3O_4 nanoparticles are considered among the ideal catalyst supports that used for CO oxidation due to its good chemical stability, low toxicity, and magnetic properties. Recently, there are some reported disadvantages of the various adopted strategies of synthesis like using hazard chemicals, multi-steps synthesis, and complicated and harsh reaction conditions.[33, 36-40]

In this scientific research, we used a facile one-step hydrothermal synthesis approach to prepare Pd/Fe_3O_4 nanocomposite with an excellent catalytic activity towards carbon monoxide oxidation catalysis.

The hydrothermal synthesis is considered as one of the unique synthetic tools especially in the field of nanomaterials synthesis. The crystal growth is obtained via using a steel pressure vessel. The German geologist Karl Emil was the first to introduce the hydrothermal growth of crystals, followed by Robert Bunsen. The hydrothermal synthesis is considered as an essential branch of inorganic chemistry as it can be used to prepare a wide group of materials including quartz, metal oxides, nanomaterials,…etc.

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II. EXPERIMENTAL WORK

TEM and XPS analysis were implemented. Ferric acetyl acetonate along with benzyl ether were added, then, palladium acetyl acetonate was also added while stirring.

Olylamine and oleic acid mixture were also added while stirring for 2 hours. Finally, the obtained nanoparticles washed several times with ethanol and then decanted.

The nanoparticles were then dried overnight under vacuum at 303 K, then the regular protocols for characterization and catalyst evaluation were implemented. [23, 25]

III. RESULTS AND DISCUSSION

Figure 1 display a TEM image of 10 wt% Pd/Fe_3O_4 nanoparticles prepared by via hydrothermal synthesis technique. The nanoparticles reveal a remarkable degree of well dispersion as the particles have small sizes ranging from 8 to 12 nm.

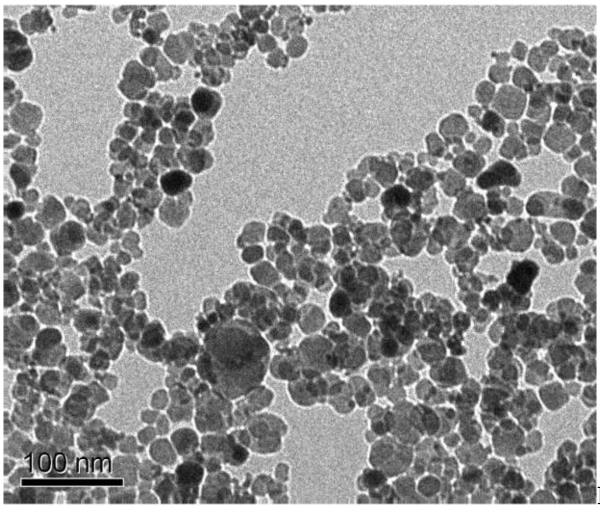

Figure 1 TEM of Pd nanoparticles supported on iron oxide

Figure 2 display the XRD patterns of Pd/Fe_3O_4 nanoparticles prepared by the hydrothermal synthesis. The pure nanoparticles of palladium show crystalline particles (JCPDS-46-1043).

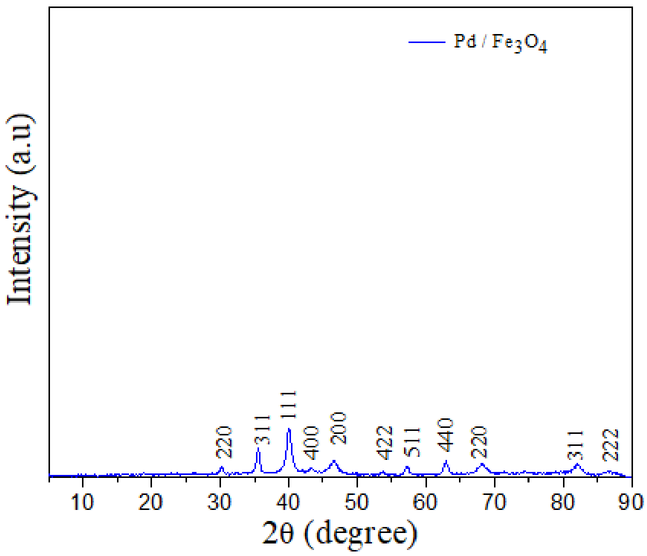

Figure 2 (a) XRD of Pd/Fe_3O_4 nanoparticles.

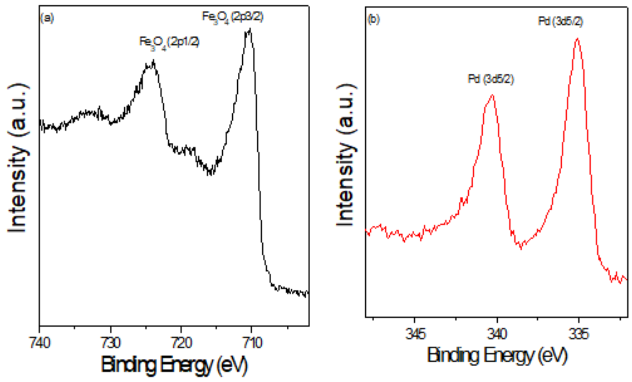

Figure 3 (a) XPS (Fe) (b) XPS (Pd) for Pd/ Fe_3O_4.

Fe (III) was confirmed by the characteristic peaks at 724.2 eV and 710.5 eV ascribed to $2p^{1/2}$ and $2p^{3/2}$ respectively. The Fe (III) $2p^{3/2}$ peak at 710.5 eV includes Fe (II) $2p^{3/2}$ around 708 eV. For Pd, binding energies were observed at 334.8 eV and 340.1 eV, ascribed to Pd^0 and Pd^{2+}, respectively.[24, 26-33]

Generally, the XRD pattern clearly indicates that the produced catalyst is enriched with Fe_3O_4 and metal Pd (0).

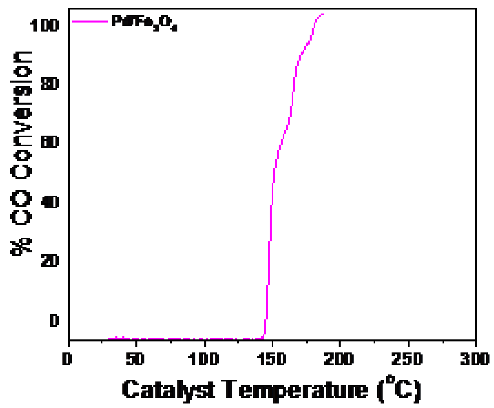

Figure 4 CO oxidation catalysis of 10 wt % Pd/Fe_3O_4 nanoparticles.

Figure 4 reveals the catalytic oxidation of carbon monoxide over Pd / Fe_3O_4. The catalytic performance of pure Pd nanoparticles and magnetite were measured and it is obvious that they both have limited catalytic activity with 100% conversion at 209 °C and 185 °C, respectively.

However, 10-wt% Pd/Fe_3O_4 catalyst has 100% conversion temperatures 136.5 °C indicating a higher activity relative to pure Pd nanoparticle catalyst due to supported catalytic systems in order to avoid aggregation and sintering. [23-39]

IV. CONCLUSION

In conclusion, Pd/Fe_3O_4 nanoparticles were prepared via hydrothermal synthesis. This synthetic technique enables us to avoid reproducibility issues compared other methods. The prepared Pd/Fe_3O_4 catalyst is highly active while being used for CO oxidation.

Optimization of the reaction conditions will alter the decisive parameters that dominated the catalytic activity of the designed catalyst including particle size, chemical composition, and shape.

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