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# Microwave Assisted Synthesis of Binary Metallic Oxides for Catalysis Applications

Abdelrahman Mostafa Kamal, Sherif Bahgat, Mohamed Hammam, M. A. Radwan, M. A. Sadek,  
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**Abstract:** *Herein, versatile, and reproducible method to prepare binary metal oxides via microwave assisted synthesis. Catalysts are substances that basically speeds up chemical reactions. Ideally, bonds are formed between the catalysts and the reactants. Also, catalysts permits formation of products from the reactants. These formed products, splits off the catalyst without affecting or changing it. Catalytic kinetics studies the correlate chemical reaction rate with some properties of reactants and/or products for instance; temperature, concentration and pressure. The aim of the project is to prepare pure and bi-metal iron based catalyst by co-precipitation method and to characterize the prepared sample using X-ray diffraction. Metal oxides nanoparticles is a field of interest in catalysis, such that these oxides are used to oxidize carbon monoxide. The samples were prepared through co-precipitation method in laboratory scale. The metals used was copper, iron and cobalt. After preparing pure sample of each metal a mix of two metals were introduced in different ratios. The samples were characterized via X-ray diffraction (XRD) and then the results were compared to exist data introduced from others research, the prepared samples XRD was having a great matching with the data retrieved from internet and we found that the metal could exist in two form of oxides and even could exist as pure metal. Each peak in the XRD figure could indicate one or more phase of the metal.*

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

## I. INTRODUCTION

As the chemical manufacturing develops into more complicated and sophisticated products, a scientist called Harper thought of a new processing system and called thermal processing systems which have been invented to back the market's growing needs. Harper's thermal processing systems were always trusted for production of metal oxides and tremendously increasing their processing rates. Aluminum, silica, cobalt, tungsten, carbon and graphite, quartz, molybdenum and many more metal oxides are produced using these thermal processors.

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These furnaces designed by Harper are so innovative for these materials providing a temperature controlled environment from 300 to 3000°C. The design of Harper thermal processing system is on demand and usually specific to the desired metal oxide. [1-22]

Heterogeneous catalysis based on metal oxide supports have numerous applications including hydrocarbon refining along with other environmental applications. Cobalt as an oxide is a coloring metallic oxide which at all temperature ranges, was found to give a blue shine. A black color is expected to be produced, if it exists in an extremely high percentage. Cobalt oxide has many applications in industry as a colorant as in enamels, glass and glaze industries where black cobalt oxide is the main source of it.

Being stable at most reaction conditions, cobalt is the main coloring agent in ceramic industry. It is added with a minor amount of less than one percent in most recipes. Cobalt possess some unique characteristics. It melts readily fast upon oxidation as copper (Andy, 2015). Upon cooling cobalt would get into a crystalline structure completely if mixed with fluid frit base with an adequate percentages. As previously mentioned that cobalt is has many applications as a coloring agent and hence could be used as a stain for body and as slip stain. Drawbacks of cobalt based substances are of very high cost and hence its use in practice is extremely limited. The cobalt products stocked by the suppliers are always cobalt (71%) where the mentioned percent designated refers to cobalt metal amount. Commercially available product of cobalt, is a mixture of CoO and  $Co_2O_4$  which takes the symbol  $Co_3O_4$ . If pure cobalt oxide (CoO) is needed, the percentage of cobalt metal would reach 78.6% instead of 71% mentioned before. This extra oxygen present in the chemical structure of  $Co_3O_4$  will be released during firing. It is known that the decomposition of CoO is difficult to understand and complicated. As a matter of fact, during melting of glaze, if the kiln is not fired in reduction, powder of raw cobalt oxide would not decompose to actual CoO. Owing to the aforementioned drawback, on switching cobalt brands, color of cobalt many be adjusted and hence an adjustment of percentages is crucial. From the products of  $Co_2O_3$  is cobalt (II) oxide which undergoes decomposition at a temperature of 900°C. Moreover, cobalt II oxide, is distributed in different countries as Morocco and Canada also found in South Africa.

It is found in ores associated with other metals as sulfur, arsenic, nickel and manganese. Some of these metals may contaminate cobalt in roasting process.

Sodium carbonate, may be a potential contaminant for  $\text{Co}_3\text{O}_4$  ores. By heating  $\text{CoO}$  could be obtained by carbonate heating. If cobalt ore was roasted in a kiln, major disadvantages are observed as melting of the ore itself if was fired too much. On this very high firing, the walls of the container would melt. Notably, this process was tried by some people. From the drawbacks of commercially available cobalt oxides used in ceramic production is glaze specking unless being sieved properly according to the method of cobalt particle production. Other drawback is that commercially available products are not standardized. It is noted that amount of specking varies according to the batch and supplier.

The advantages of cobalt carbonate - which contains 63%  $\text{CoO}$  - is that it disperses easily in glaze than other forms and gives better blue coloration. It is advisable to use a cobalt blue stain for better stability and consistency of results (Hansen, 2015).

In this scientific research, we prepare and characterize Binary oxides (Cobalt, Iron, Copper based Catalyst) for different applications in catalysis as we prepare pure cobalt oxide and mixed cobalt oxide with copper oxide and we make x ray diffraction of each metal oxide to show our great work in the laboratory.

## II. EXPERIMENTAL WORK

XRD analysis were used. Iron, copper, and cobalt chlorides were reduced using 5 mL hydrazine hydrate and then microwave irradiation was applied to 5 minutes. Finally, the obtained nanoparticles washed several times with ethanol and then decanted. The nanoparticles were then dried and the regular protocols for characterization and catalyst evaluation were implemented. [23, 25]

## III. RESULTS AND DISCUSSION

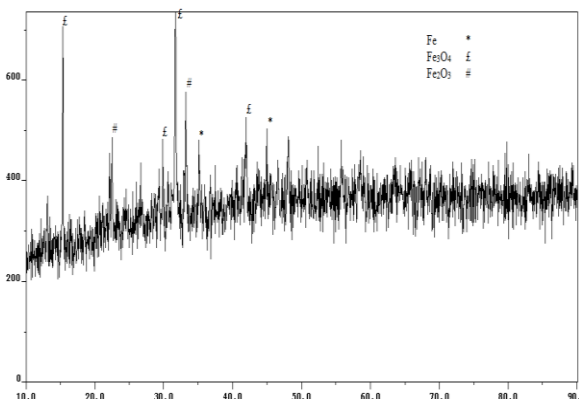


Figure 1 XRD of iron oxide nanoparticles.

Figure 1 display the XRD pattern of iron oxide nanoparticles. As shown in the figure the significant peaks are at  $2\theta = 16.2^\circ, 22.7^\circ, 28.8^\circ, 32.3^\circ, 33.7^\circ, 42.5^\circ, 46.6^\circ$ . From these peaks one can see that the sample contains three phases Fe,  $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ . But the main phase is  $\text{Fe}_3\text{O}_4$ .

Figure 2 display XRD pattern of cobalt oxide nanoparticles. The peaks at  $2\theta = 18.98^\circ, 31.68^\circ, 36.82^\circ, 44.79^\circ, 55.69^\circ, 59.37^\circ, \text{ and } 65.23^\circ$  are indicative of the presence of the

(111), (220), (311), (400), (422), (511), and (440) reflections of phases of  $\text{Co}_3\text{O}_4$ .

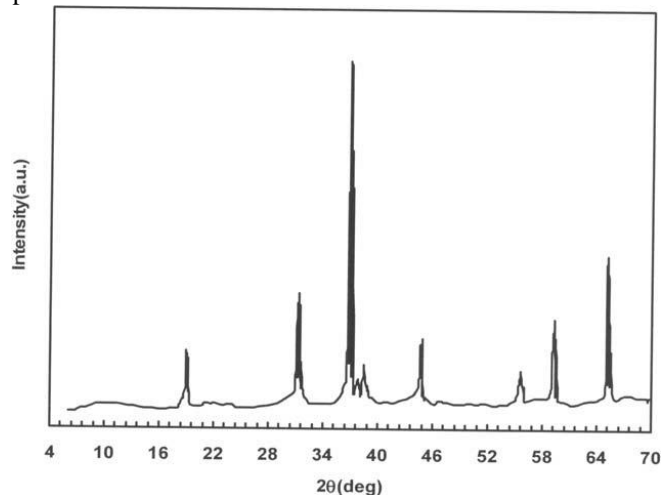


Figure 2 XRD of cobalt oxide nanoparticles.

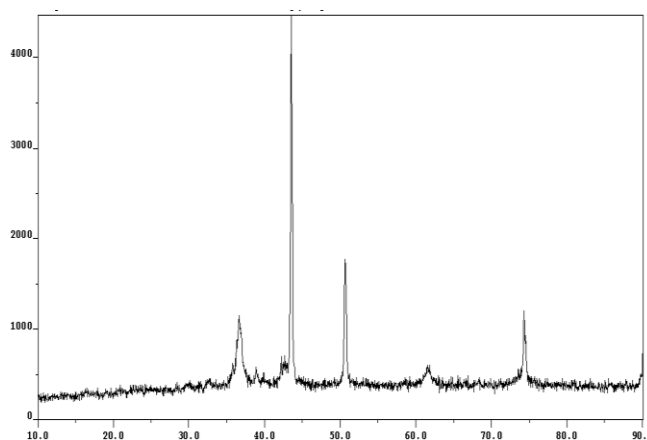


Figure 3 XRD of copper oxide nanoparticles.

The peaks at  $2\theta = 38.2^\circ, 48.3^\circ, 53.4^\circ, 58.5^\circ, 61.1^\circ, 67.5^\circ, \text{ and } 69.2^\circ$  are indicative of the presence of the (111), (202), (020), (202), (113), (022), and (113) reflections of phases of Copper oxide.

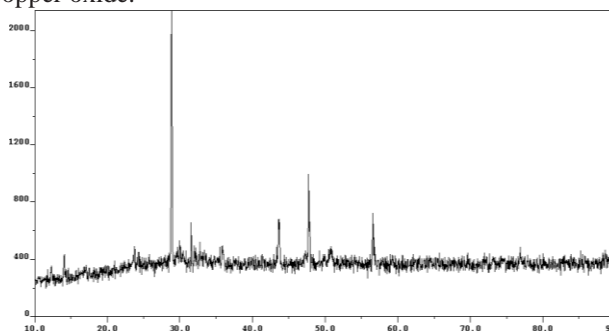


Figure 4 XRD of Iron / Copper oxide nanoparticles.

The peaks at  $2\theta = 30.5^\circ, 36.2^\circ, 37.5^\circ, 40.4^\circ, 50.5^\circ, \text{ and } 61.1^\circ$  are indicative of the presence of the (220), (-111), (311), (111), (-202), and (511) reflections of phases of mixed cobalt oxide and copper oxide. [23-39]

## IV. CONCLUSION

In conclusion, from results above as we can see these results indicates that the metal oxides are well done experimentally

By making the X-ray diffraction to each metal oxide and comparing my results to another results to guarantee my results, as the peaks of each theta (degree) in X-ray diffraction pattern of pure cobalt (II,III) oxide nanoparticles that I done are very close to another X-ray diffraction pattern of pure cobalt (II,III) oxide nanoparticles ,and the peaks of each theta degree of X-ray diffraction pattern of pure copper oxide nanoparticles are very close to another X-ray diffraction pattern of pure copper oxide nanoparticles, and the peaks of each theta degree of X-ray diffraction pattern of mixed cobalt oxide and copper oxide are very close to another X-ray diffraction pattern of mixed cobalt oxide and copper oxide, and the peaks of each theta (degree) in X-ray diffraction pattern of pure iron (II,III) oxide nanoparticles are very close to another X-ray diffraction pattern of pure Iron(II,III) oxide. Metal oxides nanoparticles is a field of interest in catalysis, such that these oxides are used to oxidize carbon monoxide. The metals used were copper, iron and cobalt. After preparing pure sample of each metal a mix of two metals were introduced in different ratios. The samples were characterized via X-ray diffraction (XRD) and then the results were compared to exist data introduced from others research, the prepared samples XRD was having a great matching with the data retrieved from internet and we found that the metal could exist in two form of oxides and even could exist as pure metal. Each peak in the XRD figure could indicate one or more phase of the metal.

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