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Synthesis and structural investigation of polyhedron Co_3O_4 nanoparticles: Catalytic application and as fuel additive

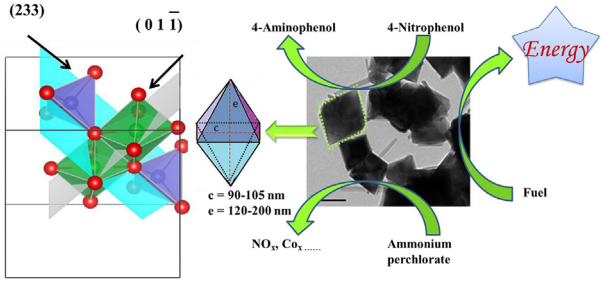
Saba Jamil[a,*], Muhammad Ramzan Saeed Ashraf Janjua[b,**], Shanza Rauf Khan[a]

[a] Laboratory of Superlight Materials and Nano Chemistry, Department of Chemistry, University of Agriculture, Faisalabad 38000, Pakistan
[b] Department of Chemistry, King Fahd University of Petroleum & Minerals (KFUPM), Dhahran 31261, Saudi Arabia

HIGHLIGHTS

- Cobalt oxide polyhedrons are prepared by microwave assisted wet chemical synthesis.
- Synthesized product is a good catalyst with an excellent reducing capability.
- Surface properties of product significantly increase the calorific value of fuel.

GRAPHICAL ABSTRACT



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ABSTRACT

Cobalt oxide (Co_3O_4) polyhedron are synthesized by using microwave assisted wet chemical synthesis approach. The structural model of Co_3O_4 has been predicted on the basis of X-ray diffraction analysis. It is observed that positions of all cobalt ions are not same. The structural parameters of unit cell are calculated. The calculated values of various structural parameters have compared with that of standard values here. The morphology of product has been analyzed with help of scanning and transmission electron microscopies. The lengths of all dimensions of polyhedron are measured from TEM images. The catalytic activity of Co_3O_4 nanoparticles are tested against dry and aqueous media reactions. Moreover the role of nanoparticles as an additive in kerosene as fuel is also studied. The properties of fuel in the absence of nanoparticles are found to be different from that of presence of nanoparticles.

1. Introduction

Nowadays metal oxide nanoparticles are being extensively used in field of imaging, sensing, biotechnology and catalysis due to their unique quantum size properties [1–9]. The surface area-to-volume ratio of nanoparticles is very high and activity of catalyst depends upon it [3,10–14]. So synthesis of crystalline and porous nanomaterials [15–17] is a milestone achieved by material chemists in this era. Nanoparticles are mostly synthesized by wet-chemical methods like co-precipitation [18–22], sol–gel [23–27], hydrothermal [28–31] and micro-emulsion [32–35], because morphology and chemical composition of prepared product can be effectively controlled by these methods [36–39]. Therefore wet-chemical methods [40–43] are most favorable route of researchers to prepare nanomaterials of well-defined morphologies and a narrow size distribution [44–46]. The properties of coinage metals (copper, cobalt etc) based nanoparticles are comparable to that of noble metals (palladium, platinum etc) [47–50]. But coinage metals are cheaper than noble metals. So usage of coinage metals based

* Corresponding author.
** Corresponding author.
E-mail addresses: saba_hrb@yahoo.com (S. Jamil), Janjua@kfupm.edu.sa, Dr_Janjua2010@yahoo.com (M.R.S.A. Janjua).

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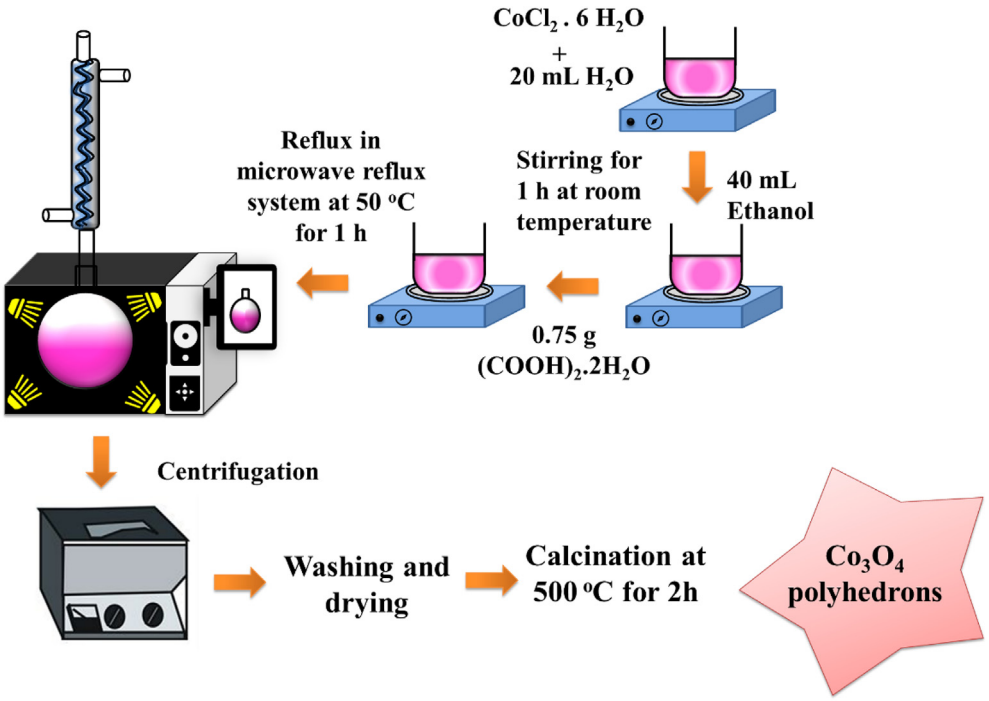

Fig. 1. Schematic representation of synthesis of Co_3O_4 polyhderons using microwave assisted wet chemical approach.

nanoparticles is cost effective. Therefore we diverted our attention towards catalytic application of cobalt oxide (Co_3O_4) nanoparticles [51–53] rather palladium nanoparticles. We employed Co_3O_4 nanoparticles as catalyst for complete combustion of methane to make this process more efficient and cost effective. These type of catalysts are very significant in the quest of innovative energy sources to meet global energy crises [54]. The metals combine with oxygen in different proportions to form their oxides. These metal oxides show some specific properties that increase their applications in various fields of life [45,55]. The positive oxidation states allow transition elements to form many different ionic and partially ionic compounds [56,57]. The formation of complexes causes the d orbitals to split into two energy sublevels, which enables many of the complexes to absorb specific frequencies of light [58]. Thus, the complexes form characteristic colored solutions and compounds. Complexation reactions sometimes enhance the relatively low solubility of some compounds. Fabrication of nano and micro forms of crystalline and porous Co_3O_4, is a milestone achieved by material chemists owing to its idiomatic and momentous attributes. This domain has acquired various manoeuvres such as the thermal decomposition of cobalt precursors under oxidizing condition (210–815 °C), chemical spray pyrolysis (350–400 °C), chemical vapour deposition (550 °C), and the traditional sol–gel method (above 260 °C) to synthesize cobalt oxides and cobalt carbonate [59]. Yet, all of the aforementioned procedures require surpassingly high reaction temperature and are subsequentlysecluded for the bulk production of nano crystalline Co_3O_4. Wet-chemical syntheses, such as co-precipitation, sol–gel, hydrothermal and micro-emulsion techniques can effectively control the morphology and chemical composition of prepared product, therefore it is the most favorable route to prepare cobalt compounds at nano and micro scale [60]. According to best of our knowledge, polyhedron Co_3O_4 is first time presented as catalyst for dry and aqueous media processes at same time in this work. No one has previously reported one catalyst for dry and aqueous media processes at same time.

In this study, unique polyhedron Co_3O_4 nanoparticles are prepared by using microwave assisted technique. The composition of product is analyzed by X-ray diffraction technique (XRD). Values of various structural parameters are also calculated. The morphology of Co_3O_4 nanoparticles lattice is analyzed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The catalytic property of prepared Co_3O_4 nanoparticles is tested for the thermal decomposition of ammonium perchlorate (AP), reduction of 4-nitrophenol (4-NP) and oxidation of carbon monoxide (CO).

2. Experimental

2.1. Materials

Cobalt chloride hexahydrate ($CoCl_2.6H_2O$), ammonium perchlorate (AP) and 4-nitrophenol (4-NP) are purchased from Sigma-Aldrich, USA. Sodium borohydride ($NaBH_4$) and oxalic acid dihydrate ($C_2O_4H_2.2H_2O$) are obtained from Scharlau Company, Germany. All the chemicals are used as such without any further purification. Deionized water is used during all solution preparation and reaction studies.

2.2. Synthesis of cobalt oxide nanoparticles

Cobalt chloride (1.5 g) is added into 20 mL deionized water followed by the addition of 40 mL ethanol. Then reaction mixture is stirred at room temperature for 1 h. Later 0.75 g of oxalic acid dihydrate is added into reaction mixture. Then reaction mixture is transferred into a flask of 100 mL capacity and refluxed in microwave reflux oven (APEX) of EU Chemical Shanghai Qiyao, Limited for 60 min at 50 °C. After microwave treatment, light pink precipitates are collected by centrifugation and washed several times with deionized water at room temperature. Then the obtained product is dried in vacuum at 60 °C for 6 h. Then dried product is grinded and calcined at 500 °C for 2 h, hence final product is obtained. Complete scheme of product preparation is given as Fig. 1. All the steps are clearly shown in this figure.

2.3. Applications of cobalt oxide nanoparticles

Co_3O_4 polyhedrons is used as catalyst for thermal degradation of AP and reduction of 4-NP. 9.9 g AP and 0.1 g Co_3O_4 polyhedrons are mixed and percentage mass loss at different temperatures is measured on thermogravimetric analyzer.

4-NP (1.8 mL of 0.14 mM), $NaBH_4$ (0.5 mL of 50 mM) and Co_3O_4