



REDUCTION OF p-NITROPHENOL TO p-AMINOPHENOL BY USING NiO CATALYSTS: A COMPARATIVE STUDY

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Different nickel oxide catalysts have been prepared by simple precipitation method using different nickel precursors and using surfactant sodium dodecyl sulfate (SDS) or without surfactant. The prepared catalysts have been characterized by using XRD and FT-IR. The particle sizes have been calculated by using the Scherrer equation. The nickel oxide catalyst prepared by using nickel acetate as precursor and surfactant SDS has shown less particle size as compared to other catalysts prepared by using other precursors. Also, nickel oxide catalyst prepared using nickel acetate precursor and surfactant SDS has shown higher catalytic activity for reduction of p-nitrophenol (PNP) to p-aminophenol (PAP) using sodium borohydride (NaBH₄). Also, for the said reaction effect of concentration of p-nitrophenol on catalytic efficiency has been studied.

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pharmaceutically significant value-added products. The wide commercial use of arylamines has led to the improvement of new and efficient protocols for the reduction of nitroarenes. The reduction of nitroarenes with sodium borohydride is one of the most simple, cleanest and most accepted methods, but the very slow hydrolysis of sodium borohydride makes this method unusable^{20,21} until some catalyst is used.

Introduction

Countless chemical protocols have been depended upon the catalysis, and the catalysts play a crucial role in the production and manufacture of chemicals and materials because catalytic reactions generally occur under milder conditions compared to the noncatalytic reactions.^{1,2,3} The use of heterogeneous catalysts is one of the best attractive alternatives for synthetic strategies and adopted by organic chemists for increasing the efficiency of a wide range of organic reactions.⁴

Recently, transition metal oxides as catalysts are to tailor and design according to their sizes, structure in nanodimensions and therefore their surface chemistry and catalytic properties.⁵⁻⁹ An increasing number of illustrations are accessible in the literature where nickel-based nanoparticles have been used as catalysts during organic transformations.^{10,11}

Nickel oxide (NiO) is a p-type semiconductor material with magnetic power and wide band-gap energy (3.6-4.0 eV). It has been exploited in many different areas such as gas sensing, biomedical, electrochemical, supercapacitors, photovoltaic devices, memory storage, fuel cells, conducting materials, and electrode materials.¹² Also, NiO nanoparticles have been used as heterogeneous catalysts for various organic transformations.¹³⁻¹⁶ The nanoparticulate NiO catalysts have shown an edge over the bulk NiO catalysts since they have a higher surface to volume ratios.¹⁷ NiO nanoparticles have been synthesized by various routes such as chemical precipitation method, microemulsion, electron spray synthesis, laser ablation, and hydrothermal method.^{18,19}

Arylamines are useful raw materials for several industries such as for the synthesizing rubbers, paints, plastics and

In this work, we have been prepared NiO nanomaterials by simple precipitation method (with and without surfactant) using two different precursors (nickel acetate and nickel nitrate). Prepared catalysts were characterized by XRD, FT-IR and applied for catalytic reduction of p-nitrophenol to p-aminophenol.^{ES}

Experimental

The main starting materials were nickel(II) acetate, nickel(II) nitrate hexahydrate, sodium hydroxide (NaOH), sodium dodecyl sulfate (SDS) and ethanol, all of them were analytical grade and purchased from Kemphasol and Sigma-Aldrich. These chemicals were used as received without further purification.

Synthesis of NiO nanoparticles

In this preparation, we have prepared two separate solutions; a solution of 16.65 g of nickel acetate in 83.25 mL of deionized water, and a solution of 10 g sodium hydroxide in 250 ml of deionized water. Amount of 6.34 g of surfactant sodium dodecyl sulfate has been added to above-mentioned nickel acetate solution with continuous stirring. Next, the prepared sodium hydroxide solution was added dropwise with constant stirring to the solution of nickel acetate and surfactant. The mixed solution was stirred by a magnetic stirrer at room temperature for 1 h. The resultant light-green solution was kept at room temperature for settle down. The formed precipitate was then filtered, washed with deionized water several times and dried at 50°C for 24 hours. Finally, the obtained powder has been calcined at 500°C for 4 hrs. The