



Improved catalytic performance of cobalt oxide for selective oxidation of benzyl alcohol

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Abstract

In this article, Co_3O_4 nanoparticles were synthesized by the uncomplicated microwave treatment. The Co_3O_4 nanoparticles have characterized by transmission electron microscopy, Brunauer–Emmett–Teller specific surface area dimension and the X-ray diffraction. The preparation of cobalt oxide nanoparticles afford a fresh avenue for the application of catalytic properties and the represents a promising way for the synthesis of novel inorganic nanoparticles.

Keywords Co_3O_4 nanoparticles · TEM · XRD · Synthesis of novel inorganic nanoparticles

1 Introduction

Freshly, amid that the several types of metal matrix nanomaterials arrived through the appearance that the nanometric features for nanocrystalline metallic arrangements suitable to the multi-functional properties. Freshly, enthusiasm lengthwise with the enhancement of nano materials separation know-how, nano structured perovskite ABO_3 -category possessions with the augmented surface area must be acknowledged rehabilitated notification by way of the impending manufacturing substance for draw off production accomplish [1]. The huge demand for the operation of substance is nanoscience and nanotechnology different from their micro measure complements in their magneto-optical, electro-optical, chemical and mechanical and the surface area to volume proportion which signifies them as an effective tool for biomedical uses. Nowadays, Co_3O_4 possesses interesting properties and thus has attracted various researchers for studying their probable Co_3O_4 catalyst shows catalytic activity

[2]. Co_3O_4 catalysis for organic responses by the metals motionless characterizes unique for the greatest influential device in organic synthesis [1], with a unique reference for unequal transition-metal catalysis authorizing highly enantio selective responses to be implemented. Outstandingly, that the amalgamation methods for the procurement for the mixed metal oxides must remain explained in the nonfiction earlier, for example that the solid-state response or ceramic process [3], co-precipitation [4], hydrothermal process [5]. Presently, synthesis underneath the hydrothermal circumstances can deliver a uninterrupted, small-step path to make into nano-sized oxide. Though, novelty technique frequently involves protracted reply period after the little infections remain functional (temperature) [6].

Nowadays, a hot topic is oxidation of benzyl alcohol cutting-edge present organic combination for carbonyl products. Hot investigation of the selective oxidation around wonderful experiment towards preclude over-oxidation of the harvests, in which were habitually supplementary

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sensitive to be tarnished than the reactants to products. Nowadays, that the benzaldehyde was a central underdone quantifiable in the separation for the other organic compounds and the ranging from pharmaceuticals toward plastic additives.

Awake to our information, that the chief meaning for our analysis remains toward demonstrate that the catalytic movement into cobalt oxide in liquid phase reaction. The catalysts were characterized by TEM, BET surface area performance and the powder XRD.

2 Experimental procedure

2.1 Resources for chemicals

Chemical used for the cobalt nitrate and the dissimilar fuel glycine besides *L*-arginine were hand-me-down as the preliminary quantifiable (Merck Chemicals, India) and they were rummage-sale as acknowledged without additional decontamination.

2.2 Synthesis of Co_3O_4

The altered fuels *glycine* and *L*-*arginine* and the cobalt nitrate were used as forerunners intended for this response. The imitation routine is established on a redox reaction. Presently, stoichiometries of metal nitrate salts and the (supporting materials) fuels were chosen seeing the total reducing and the oxidizing valences of the raw materials and they were enumerated in equivalence ratio equal with ratio. Presently, that the stoichiometric quantities of cobalt nitrate and *glycine* and *L*-*arginine* were dissolved unconnectedly in 10 ml of de-ionized water and the poured into a silica crucible and the stirred for 15 min to obtain a clear solution. For the preparation of pure cobalt oxides by the using the microwave combustion performance, that the precursor mixture in *glycine* and *L*-*arginine* was placed into a domestic microwave oven and the exposed to the microwave energy in a 2.45 GHz multimode cavity at 850 W for 10 min. After the achievement of the retort, that the solid precipitous was obtained and then it was wash away with ethanol and the desiccated at 90 °C for 1 h and was considered by means of cobalt oxides samples *glycine* and *L*-*arginine* for samples (A and B).

2.3 Characterization of nano cobalt oxide

The structural lessons of the cobalt oxides samples was strong-minded by X-ray diffraction (XRD, RIGAKU, Dmax22000) by using a diffractometer with Cu K α radiation at $\lambda = 0.154$ nm. A cobalt oxide was long-established by the gold coated compacts of powder sample using

scanning electron microscope EVO 40. Transmission Electron Microscopy (TEM) was approved out on JEOL 2100 F and FEI Philips Morgagni 268D. The nitrogen adsorption–desorption isotherms of the samples were slow by the using an automatic adsorption instrument (Quantachrome Quadrawin gas sorption analyzer) for the purpose of the surface area and the total pore volume.

2.4 Catalytic examination

Catalytic oxidation for benzyl alcohol stands approved available in a consignment apparatus the functioned underneath impressive conditions. 5 mmol of oxidant (H_2O_2) is additional along with 0.5 g of the cobalt oxide and the fillings were heated at 80 °C in acetonitrile medium for 8 h in a one necked rotund bottom flask armed with a reflux condenser and thermometer.

The yield of the carbonyl compounds formed was calculated by the following formula.

$$\text{Conversion (\%)} = \frac{\text{moles of benzyl alcohol reacted}}{\text{total moles of benzyl alcohol}} \times 100$$

The selectivity of the products was calculated by the following formula.

$$\text{Selectivity (\%)} = \frac{\text{moles of product formed}}{\text{moles of benzyl alcohol reacted}} \times 100$$

The yield of the products was calculated by the following formula.

$$\text{Yield (\%)} = \frac{\text{mole amount of product}}{\text{initial mole amount of benzyl alcohol}} \times 100$$

3 Results and discussion

3.1 XRD analysis

The XRD decorations for cobalt oxide precipitates organized by the conventional and microwave methods presented in a displayed Fig. 1. XRD, spreading results of the samples authorization the peaks experiential at $2\theta = 31.56, 36.78, 44.90, 55.13, 59.57$ and 65.45 container be situated indexed that the (220), (311), (400), (422), (511) and (440) mineral (hkl) flat surface for the cubic segment of Co_3O_4 , respectively and remain in good agreement with the JCPDS Card No. 76-1802 standard card of cubic structure of Co_3O_4 . Not at all peaks of other contaminations container are observed in the XRD design, illustrative that the high purity of all the prepared Co_3O_4 . The methodical examination accomplished in this work has permissible the documentation, for the

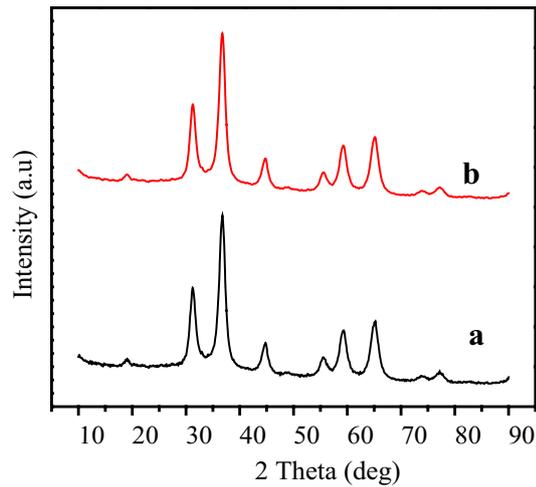


Fig. 1 XRD pattern of Co_3O_4 nanoparticles samples A and B prepared by microwave method

three energies examined, of a general reply to the dissimilarity of the countless dispensation fuels; though in some cases a dissimilar properties has been presented by the cobalt oxides formed.

The average crystallite size was obtained from XRD peaks using Scherrer equation [12].

$$L = \frac{0.89\lambda}{\beta \cos \theta}$$

where, L = average crystallite size (\AA), λ = wavelength of the incident X-ray beam (1.54 \AA), β = the FWHM (rad), and θ = is the Bragg's angle. The average crystallite size for the samples A and B was found to be 26.9 and 23.3 nm, respectively.

3.2 TEM analysis

Figure 2 shows that the TEM images for the Co_3O_4 nanoparticles synthesized samples A and B. On low resolution, it can be noticeably experimental by the TEM (Fig. 2a, b) that the spherical cobalt oxide frame work is an aggregate for the individual rod-like particles, self-possessed of primary nanoparticles weighted lengthways that the length direction. Present study, propensity of altered [from temperature and the ratio (1:1) fuels also] accumulation/accretion intensities connecting that the development for the small agglomerates of a insufficient particles was piercing out likewise for certain perovskite for metal oxides. The value of the regular particle size is nearby to the unkind crystallite size (26.1 nm to 32.6 nm) strong-minded from the control founded on the XRD data. The insignificant modifications (XRD and TEM) are inherent to the scheming or evaluating inaccuracies.

3.3 Surface area investigations

The nitrogen adsorption/desorption isotherm standards by the 77 K of the all samples are particular in presented Table 1. Cobalt oxides specific surface area and the micropore volume container remain increased, while maintaining a comparatively continuous pore diameter by means of the optimum amount (cobalt oxides) for filler loading in the cobalt oxides. The specific surface area was gotten by the using BET equation to amount the adsorbed nitrogen amount to the cobalt oxides. The average micropore diameter remained gotten by the H-K method [7], and then the total pore volume besides micropore volume stayed attained by the t -plot. After the

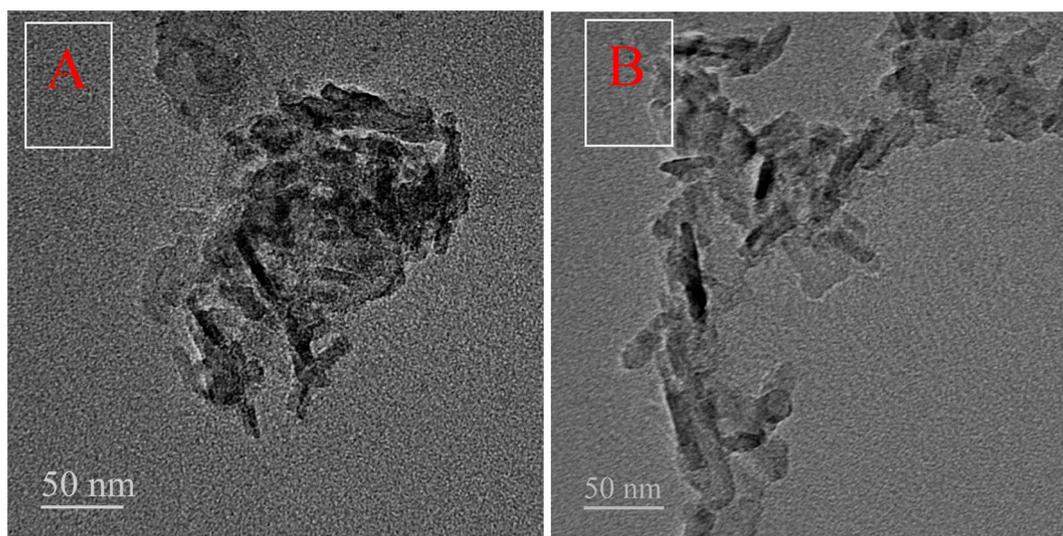


Fig. 2 HR-TEM images of Co_3O_4 nanoparticles samples A and B prepared by microwave method

Table 1 BET surface area, average pore diameter, pore volume and their crystallite size (nm) of cobalt oxides prepared by conventional and microwave methods

Catalyst	S_{BET} (m ² /g)	R_p (Å)	V_p (cm ³ /g)	Crystallite size (nm) from XRD
Sample A	62.59	26.12	0.1123	23.89
Sample B	88.90	19.34	0.1307	19.78

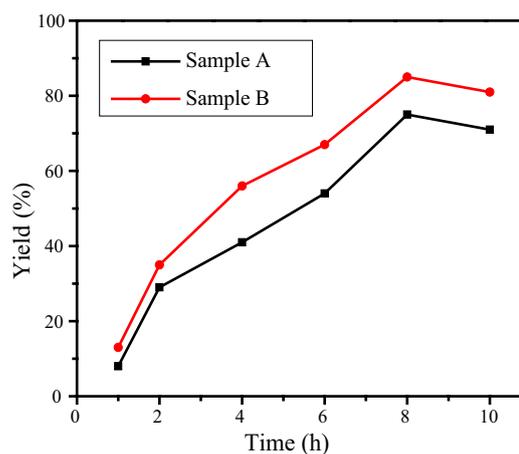
opinion of understanding of the impending presentations of cobalt oxides constructed resources for the catalysis arenas; specific surface area is a significant limitation to contemplate. The specific surface area (S_{BET}) composed with the pore radius (R_p) and the pore volume (V_p) and crystallite size (nm) are presented in Table 1. By associating the consequences of BET surface area through the catalytic happenings, it is originate that there is a straight association among the catalytic movement and the surface area [8–10].

3.4 Catalytic test

Near inspect that the catalytic oxidation belongings cobalt oxides substances towards the catalytic oxidation of benzyl alcohol, the reaction circumstances remained altered by the studying the consequence of the oxidant (NaOCl, H₂O₂ and TBHP), solvents (acetonitrile, *n*-hexane, DMSO and DMF) and reaction temperature (20–100 °C), at 1–10 h of reaction time using 0–0.5 g of samples (A and B) catalysts. Established on the discovery of this instruction, it can be said that the factory-made materials are settled for the choosy oxidation of aromatic alcohols and the can be taken up as a catalyst to study the oxidation reactions of numerous additional organic compounds. Consequently, this catalytic system is appropriate for the discriminating oxidation of alcohols.

3.4.1 Outcome of time

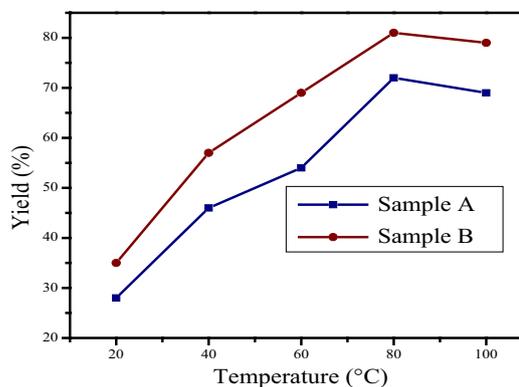
Oxidation for benzyl alcohol is consequence of time on the reaction demonstrated in Fig. 3. A suitable reaction period for foremost assurance for a picture-perfect response for oxidation rejoinder. In the contemporary revision, that reaction mixture was examined at various time intermissions in instruction to scholarship the consequence of rejoinder interval for oxidation of benzyl alcohol. Then the increase in the conversion is observed as the time profits. Nevertheless the selectivity toward benzaldehyde decreased unremittingly. This is outstanding to the uninterrupted oxidation of the product benzaldehyde, in which is preferred with cumulative time. For additional educations of the present response a reaction interval of 6 h was designated in order to accomplish an

**Fig. 3** Effective of catalytic reaction time (h) Co₃O₄ nanoparticles samples A and B

considerable adaptation of benzyl alcohol and the selectivity of benzaldehyde.

3.4.2 Upshot of temperature

The corrosion of through the preservative supplementary reaction circumstances and the changing that the response infection, catalytic oxidation responses of benzyl alcohol were achieved, and the Fig. 4, bounces the difference curvatures of benzaldehyde yield (namely the conversion curvature of benzyl alcohol in debts to benzaldehyde as the solitary product) through time on dissimilar infections. It can be found that the reaction rate increases with increasing temperature, subsequent in the augmentation of benzaldehyde yield in the similar old-fashioned of period. Though, after the infection growths to 80 °C, the discrepancy of the reaction rate has develop very small. In accumulation, in discussion of that the glass transition temperature of cross linked benzyl

**Fig. 4** Effective of catalytic reaction temperature (°C) Co₃O₄ nanoparticles samples A and B

alcohol (yields is decrease) is about 100 °C, successively, for this reaction scheme, the appropriate response temperature should be designated as 80 °C.

3.4.3 Influence of catalyst amount

Inspiration quantity of the reagent, ounce size of catalyst, quantities for the reactants, measure of solvent, oxidant and the reaction combination are imperative variables in this study. Figure 5 demonstrates the vicissitudes in the development of benzaldehyde by the changing the amount of catalyst from 0.1 to 0.5 g at 80 °C for 8 h. The consequences designate that the benzaldehyde greater than before with an growing amount of substance pending the thoroughgoing conversion of attained when 0.5 g of catalyst was used.

It consumes remained institute that the lower yield of benzaldehyde with the lower the amount of catalyst (0.1–0.3 g) was outstanding to the attendance of scarcer catalytic sites. While, increasing the amount for catalyst outside 0.4–0.5 g, consumes improved the produce of benzaldehyde. This may seemingly owing to the obtainability of additional active sites of the catalyst. Cumulative that the catalyst quantity to 0.5 g better-quality the yield by 94% within 8 h of time. Conversely, that the transformation and the harvest continue nearly the matching upon further accumulative that the catalyst quantity from 0.6 to 0.7 g, in which proposes that the great amount of catalyst is not needed to improve the reaction invention [11]. Consequently, in this response, the finest quantity of catalyst is 0.5 g.

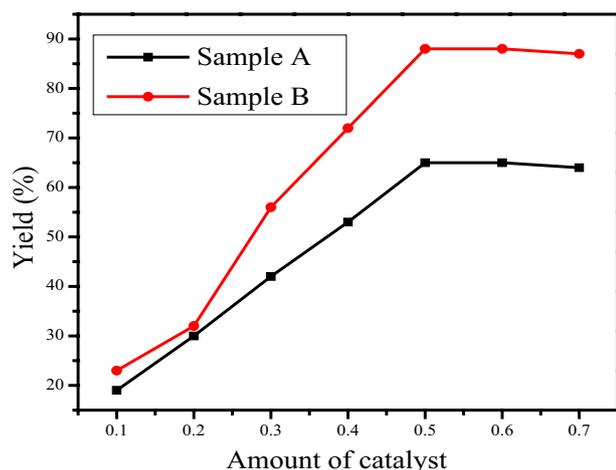


Fig. 5 Effective of amount of catalyst (0.1–0.7 g) Co_3O_4 nanoparticles samples A and B

3.4.4 Effect of the solvent

Likewise, that the solvent has a substantial influence for the produce spreading. In which the reaction is carried out in the absenteeism of whichever solvent, the low quantity for formation of the benzaldehyde was experiential condition. Present study, catalytic oxidation of benzyl alcohol is working as a model reaction toward examine that the polar aprotic solvents, such as, acetonitrile, DMF and DMSO and nonpolar solvents, such as, hexane, and toluene (Table 2).

The oxidation of the solvents remained used in the reaction, the benzyl alcohol to benzaldehyde decreases in the following order: no solvent > DMF > DMSO > toluene > *n*-hexane > acetonitrile (Table 2). DMF, DMSO and the toluene were also used as aprotic polar solvents; nevertheless the yield of benzaldehyde is selfsame low in these three solvents. DMF, DMSO and the toluene with a low boiling point has underprivileged solubility of benzyl alcohol to benzaldehyde. Moreover, that the somewhat high solubility of H_2O_2 in acetonitrile and the *n*-hexane also fast-tracks the oxidation of cobalt oxides. When using acetonitrile and the *n*-hexane, the catalyst gave comparatively good catalytic movement. In the event of *n*-hexane, appreciable yields are observed owing to the competition from hydrogen transfer with the solvent. Acetonitrile can activate the peroxide through forming a perhydroxyl anion (OOH^-) that nucleophilically attacks the nitrile to engender a peroxy-carboximidic acid intermediate, in which is a good oxygen transfer agent. Comparatively good solubility power of acetonitrile in both the organic substrate and the liquid phase H_2O_2 makes it as a good solvent. The high-quality of the solvent is significant in the circumstance of liquid phase oxidation reaction, because the solvent had a inordinate inspiration on the catalytic commotion and in the proportion delivery of yields [12, 13].

3.4.5 Catalytic reactions and mechanism

In addition, cobalt oxides based on the nanoparticles remained originate to be good heterogeneous catalysts

Table 2 Yield (%) of benzaldehyde for the catalytic oxidation of benzyl alcohol using Co_3O_4 catalysts

Co_3O_4 sample code	Yield (%)				
	Acetonitrile	<i>n</i> -Hexane	Toluene	DMSO	DMF
Sample A	80	78	74	69	62
Sample B	92	84	78	76	72

Reaction conditions: oxidation of benzyl alcohol by using oxidant (H_2O_2) 5 mmol, different solvents, 5 mmol, 0.5 g catalyst, refluxed at 80 °C for 6 h

fashionable that the acetonitrile medium, for the oxidation of benzyl alcohol by using H_2O_2 as an oxidant on the catalytic reaction temperature of $80\text{ }^\circ\text{C}$, and the reaction time 8 h. In the catalytic rejoiner assortment, individual compound formation benzaldehyde remained that the found as an oxidation creation.

Current research designated that the acetonitrile enthusiastically dissolved in H_2O_2 lengthways within that the benzyl alcohol by way of polar and the having a self-same high dielectric constant. Catalytic reaction starting (induced) to solvent acetonitrile container activate hydrogen peroxide via the forming a perhydroxyl anion (OOH^-) that the nucleophilically attacks that the nitrile to produce a peroxyoximidic acid intermediate in which is a good oxygen transmission mediator. Together that the organic substrate for the oxidant H_2O_2 dissolves in acetonitrile forming as a uniform solution [35]. Typical research influence that the critical factor to control that the morphology for the catalysts and the dispersion of cobalt (active sites) of oxygen into the reaction arrangement by the oxidant arrangements Co-OOH^- species. These observations disclose that the phenyl ring and the OH group of benzyl alcohol interact with the metal (Co^{3+}) ion of Co_3O_4 and the inner active sites remains intact. The communication of the phenyl ring and the OH group through the outer metal ions of Co_3O_4 is also evident on the adsorption of phenyl ring on Co_3O_4 . Nonetheless, that the catalyst surface gets regenerated by the action of the oxidant with the catalyst, in which indications to the desorption of the product

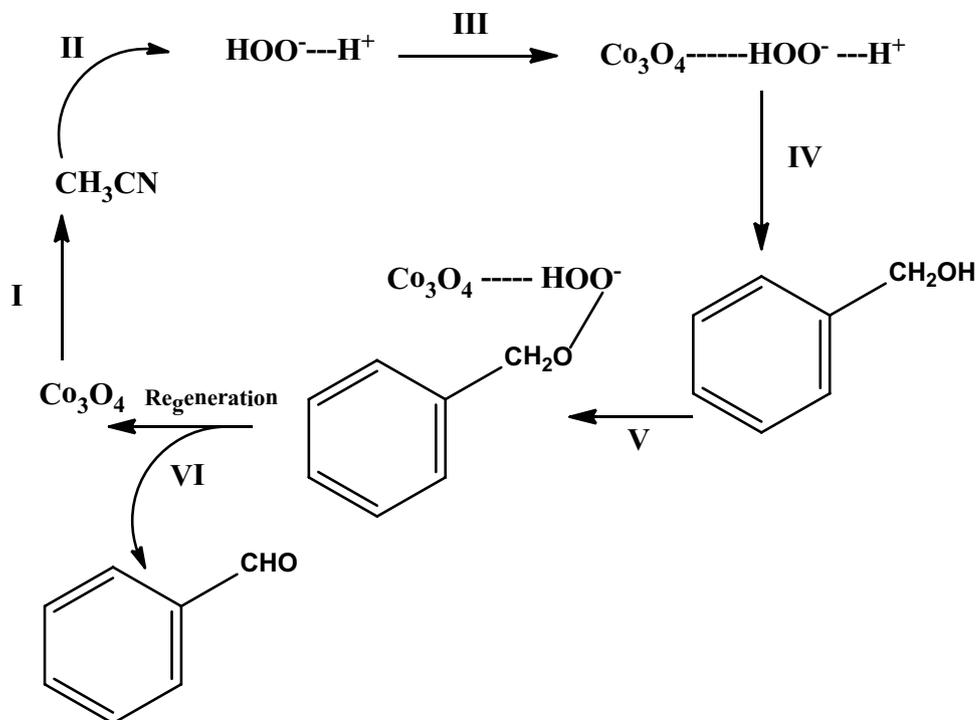
particles interact into catalytic reaction, thus [14, 15] preferring additional oxidation (Scheme 1). The oxidation of benzyl alcohol to benzaldehyde on cobalt oxide catalysts is proposed for mechanism and the several authors have investigated for catalytic properties were reported [16–19]. The catalytic oxidation of BzOH should be attributed to the increase in the active sites; which may be due to the decomposition of H_2O_2 to H_2O , and thereby it increases the catalytic activity. Therefore, it is likely that the benzyl alcohol, acetonitrile and H_2O_2 interact with Co_3O_4 effectively and the product is formed.

3.4.6 Reusability tests

Designed that the recyclability examinations for the reactions were achieved the under the identical response for conditions by way of designated overhead. Every one period that the catalyst is out-of-the-way from the reaction solution on the termination for the catalytic reaction, washed through the ethanol and heated at $120\text{ }^\circ\text{C}$ for 3 h.

In recent centuries (In 2016), cobalt oxides is originate toward obligate no considerable beating of movement in the (Fig. 6) recover assessments for the proved that it consumed a stability structure during catalysis and the environmental consideration. Furthermore, catalytic oxidation of alcohol originate that the incorporation combination that the cobalt oxides after several reprocesses likewise slightly reduced the catalytic activity studies. Nonetheless, particular changeover metal oxides

Scheme 1 Schematic diagram of the formation process of the Co_3O_4



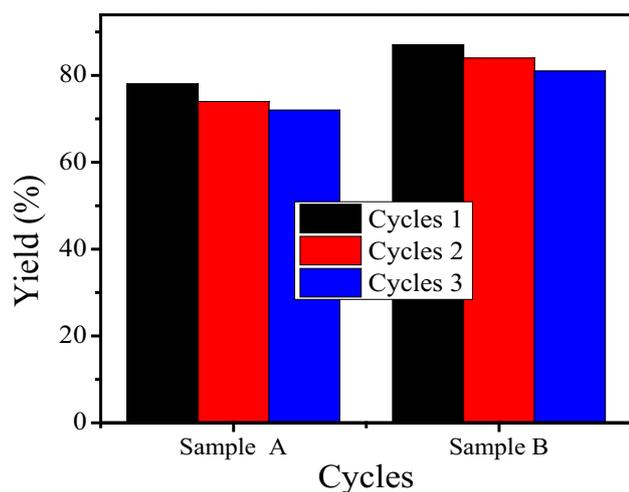


Fig. 6 Recycle for Co_3O_4 nanoparticles samples A and B

catalysts comparatively that the (cobalt oxides) good stability and gives good recycle presentation. Meanwhile, that the cobalt oxides catalyst is intelligent to dissolve BzOH to BzH through the high activity, highly recyclable, remarkably stable and the overhead completely, biologically friendly, they are hereby are promising for the industrial applications.

4 Conclusion

In decision, that the important characteristic belongs to Co_3O_4 was deliberated. The addition for fuel re-ins that the formation for cobalt oxide nanoparticles within as a comparable and the unchanging the shape and the size. Consequently, that the determined that the promising constituents of the manufacturing the applications. The consequence as an good arrangement (for structural and magnetic) catalytic presentation for the oxidation for benzyl alcohol within respectable in the conversion for almost 97% and the very high benzaldehyde selectivity's through the using H_2O_2 for the oxidant. Then, the substance can be recycled for numerous periods deprived for the important forfeiture fashionable that the conversion and the selectivity for carbonyl compounds is obtained.

Compliance with ethical standards

Conflict of interest The authors are no conflict of interest.

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