Preparation and Investigation of some Physical Properties and Photocatalytic Activity of a co-Catalyst CoO-V₂O₅

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Abstract

The current work involves preparation of neat vanadium oxide and cobalt oxide. Likewise, various percentage from their mixed forms were prepared in various ratios: 25%, 50%, 75% and 100% from CoO were precipitated with fixed weight of V₂O₅ 2.0 g. In addition, the above percentage from V₂O₅ were precipitated with fixed weight of CoO, 2.0 g. The individual metal oxides and their mixtures with various ratios were prepared from the solution of sulphate and nitrate of both metals respectively by co-precipitation process by adding sodium carbonate. All metals carbonates transfer to corresponding oxides by calcification at 773 K. Spectroscopic studies for metal oxides and their mixtures with various ratios using X-rays diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR). Some of physical properties of these oxides and their mixtures were studies such as pore size, density, porosity and electrical conductivity. The activity of these materials were investigated by following photocatalytic oxidation of 1-octanol over a suspension of these materials. The obtained results showed that photocatalytic oxidation of alcohol was achieved when using each of catalyst, light, air and 1-octanol.

Keywords: Vanadium oxide, cobalt oxide, dye removal, photocatalytic activity.

1. Introduction

When a semiconductor photocatalyst is exposed to a proper photo or thermal energy that is equal to or greater than it band gap (E₅), then an electron is excited from its valence band (VB) into its conduction band (CB). This process would produce a pair of conduction band electrons (e^-CB) and valence band holes(h^+VB)^1,2. These produced e^-CB and h^+VB are incorporated in redox reactions with the pre-adsorbed species at the surface of the photocatalyst to produce some reactive species such as OH^-, H₂O₂, O₂^- and O. These reactive species are contributed in photocatalytic reactions at the surface. The main draw back for the efficiency of the photocatalytic process according to the above mentioned mechanism is the back electron transfer for e^-CB to recombine with h^+VB in what is called recombination reaction^3,4. Recombination reaction that occurs normally in case of use neat
photocatalyst can reduce the efficiency of the photocatalytic reactions. This process can be presented in the following equations\(^5,6\):

To overcome this drawback, many attempts and researches have been directed by the researchers over the world in order to enhance the activity of the photocatalysts. These involve modification of physical and chemical properties of the photocatalyst. This aim can be achieved by doping the surface with some reactive metals, doping surface with some non-metals, sensitization of the surface with a suitable photosensitizer (dye), and composite semiconductors\(^7,8\). This method involves using two semiconductors with different band gaps, so that irradiation of smaller \(E_g\) semiconductor would transfer electron from its VB into the CB of the second semiconductor\(^9-11\). In this case there will be a sufficient charge separation which leads to reduce the rate of back electron transfer and hence reduce the rate of recombination reaction to high extent\(^12\). Generally, photocatalytic reactions involving both homogeneous and heterogeneous photocatalytic reactions can be used in wide range of applications under both solar radiation and artificial radiation\(^13\). Important examples of these potential applications involve, photocatalytic oxidation of many organic materials, photocatalytic treatment of polluted wastewaters, dye sensitized solar cells, fuel energy storage systems, photochromic devices\(^14-16\). Cobalt oxide (Co\(_3\)O\(_4\)) or it can be written as (CoO, Co\(_2\)O\(_3\)) as it can exist as a spinel oxide has excellent catalytic properties such as high photo-stability, non-toxicity and it can be recovered from reaction mixture which allow it to be used in many industrial and scientific uses. Co\(_3\)O\(_4\) have many industrial uses such as electrochemical devices, colored glasses industries\(^17,18\), and in hydrogenation reaction. The main weaknesses of this oxide, it may lose some of its activity due to recombination reaction and at high temperatures over than 700 °C\(^19\). In terms of vanadium oxide, there are different form of vanadium oxides such as Vanadium (II) oxide (vanadium monoxide, VO), Vanadium (III) oxide (vanadium trioxide V\(_2\)O\(_3\)), Vanadium (IV) oxide (vanadium dioxide VO\(_2\)), and vanadium pentoxide V\(_2\)O\(_5\)). Among these oxides, vanadium oxide (vanadium pentoxide), V\(_2\)O\(_5\) is the most stable form of different oxides forms of vanadium, and therefore most common. This oxide is used in many applications such as in industrial chemical reactions. It is also used in optical uses such as making of laser crystals. In nano-fiber and nanowire uses an it also used in the manufacture of some alloys and ceramics materials. Generally, supported V\(_2\)O\(_5\) catalyst has a great importance for wide range of applications especially for the selective oxidation of hydrocarbons, as well as for the selective catalytic reduction of nitrogen oxides\(^19,20\). The present study describes modification of V\(_2\)O\(_5\) surface by doping with CoO using different ratios of CoO on V\(_2\)O\(_5\) surface. The photocatalytic activity of the prepared photocatalyst would be investigated by following photocatalytic oxidation of 1-octanol to 1-octanal over a suspension of the prepared photocatalyst. The chemistry of the process of surface modification of V\(_2\)O\(_5\) with CoO is shown in Figure 1.
Figure 1. Schematic of the surface modification of V₂O₅ with CoO.

2. Materials and Methods

2.1. Chemicals

The used chemicals in this study were 1- Octanol (99.5%) Merck Company, 1- Octanal (99.5%, Merck Company), Co(NO₃)₂·6H₂O (99%, E. Merck Darmstant), VOSO₄ (99%, Aldrich), Na₂CO₃ (99%, Aldrich), C₆H₆N₃O₄ (99%, B.D.H), KOH (85%, BDH), HCl (37%, BDH), C₂H₅OH (99.8%, Merck) and n- C₆H₁₄ (99%, Merck).

2.2. Preparation Method of Catalyst

2.2.1. Preparation Vanadium Oxide

Vanadium oxide was prepared by dissolving 3.2 gm of vanadium sulphate in 500 mL of D.W. Then sodium carbonate (1 M) was added with heating of a mixture at 333K with continuous stirring and adding sodium carbonate gradually until the pH reaches pH=8.5. Then mixture was continuously stirred for two hours and the obtained precipitate was separated by filtration. Then it was washed with water for several times. Then the obtained precipitate was dried in oven at 373 for 16 hours.

Cobalt oxide was prepared by co-precipitation method for cobalt carbonate and cobalt nitrate. According to this method 7.7 gm of cobalt nitrate dissolved in 500 mL of D.W. Then sodium carbonate (1 M) was added drop wisely under heating at 333K with continuous stirring until the pH of mixture reaches 8.5. Then the obtained mixture was stirred for further two hours at the same temperature. The obtained solid was separated by filtration and washed with water for some times. The solid then dried in oven at 337 K for overnight.

Table 1. Adding of different weights of cobalt nitrate into a fixed amount of vanadium sulphate to prepare different weight ratios of the mixture.
A composite of V$_2$O$_5$/CoO was prepared by doping different ratios of CoO over a fixed amount of V$_2$O$_5$ (2.0 g). This was achieved using co-precipitation method for carbonate salts for these two metals in their nitrate salts. In this context, different weights of cobalt nitrate were added into different weights of vanadium sulphate in a 500 mL of DW (see Table 1). To this mixture sodium carbonate (1 M) was added drop wisely with continuous stirring at 333 K and the pH of mixture reaches 8.5. After that reaction mixture was kept under stirring for further two hours at the same temperature. Then the obtained solid was separated by filtration and washed with D.W for several times and then dried in oven at 373 K for 16 hours (see Figure 2).

A composite of V$_2$O$_5$/CoO was prepared by doping different ratios of CoO over a fixed amount of V$_2$O$_5$ (2.0 g). This was achieved using co-precipitation method for carbonate salts for these two metals in their nitrate salts. In this context, different weights of cobalt nitrate were added into different weights of vanadium sulphate in a 500 mL of DW (see Table 1). To this mixture sodium carbonate (1 M) was added drop wisely with continuous stirring at 333 K and the pH of mixture reaches 8.5. After that reaction mixture was kept under stirring for further two hours at the same temperature. Then the obtained solid was separated by filtration and washed with D.W for several times and then dried in oven at 373 K for 16 hours (see Figure 2).

| Wt. of cobalt nitrate (g) | Wt. of vanadium sulphate (g) | Wt. ratio of a mixture | Wt. percentage ratio |
|--------------------------|-------------------------------|-----------------------|---------------------|
| 1.9                      | 3.2                           | 2.0:0.5               | 25%                |
| 3.8                      | 3.2                           | 2.0:1.0               | 50%                |
| 5.7                      | 3.2                           | 2.0:1.5               | 75%                |
| 7.7                      | 3.2                           | 2.0:2.0               | 100%               |

Figure 2. A schematic overview is summarizing of the synthesis method of V$_2$O$_5$/CoO.
2.2.2. Photoreaction apparatus
Photocatalytic experiments were performed using a homemade photoreactor which is composed of reaction cell of 25 mL made up of Pyrex glass and provided with quartz window that is received a UV radiation from mercury vapor lamp (middle pressure). This unit also provided with water circulating system to adjust reaction at a desired temperature. The block diagram of reaction unit is shown in Figure 3.

![Figure 3. Apparatus of photoreaction unit that was used in this study.](image)

2.3. Preliminary experiments for investigation of photocatalytic activity of the prepared materials
A series of experiment were performed in dark and light over V$_2$O$_5$, CoO and V$_2$O$_5$/CoO with different loading ratios. These experiments were performed using 6.2 mol. Of –octanol. These experiment were carried out at 298 K under atmospheric and inert conditions. Photocatalytic oxidation 1-propanol yields 1-octanal, concentration of the produced corresponding aldehyde was estimated using 2,4-dinitophenyl hydrazine reagent. This process is commonly used in determination of concentration of aldehydes those are produced from oxidation of their corresponding primary alcohols. This process depends mainly on formation of the derivative of 2,4-dinitro phenyl hydrazone of the corresponding aldehyde followed by adding alcoholic KOH to produce a wine color of formed aldehyde derivative which can be measured at 480 nm. A suitable calibration curve using standard concentrations of octanal is used in order to evaluate concentration of the produced aldehyde. Periodically, 1.0 mL of reaction mixture was withdrawn and subjected to centrifugation to separate the solid catalyst. The obtained supernatant liquid was added to a mixture of pure hexane/ethanol (3.7: 5 mL), to this mixture 2.0 mL of 2,4-dinitro phenyl hydrazine (0.2 g. Of this reagent was dissolved in 50 mL of ethanol with adding of 2 mL of concentrated HCl. The obtained mixture was kept a side for 30 minute at 328 K, then it cooled to room temperature and diluted to 25 mL with alcoholic KOH (prepare by dissolving 11.8 g of KOH in 50 mL of D.W, then it diluted into 200 mL with ethanol). Then the absorbance was measured at 480 nm.

3. Results and Discussion
3.1. Catalyst characterization
3.1.1. X-rays diffraction for the prepared materials(XRD).
XRD patterns of both neat and coupled V$_2$O$_5$ and CoO were investigated using XRD-Phillips X-ray diffraction with CuK$\alpha$ radiation ($1.542$ Å, $40$ KV, $30$ MA), in the $2\theta$ range, 10-80 degrees. XRD6000, Shimadzu, Japan. The obtained patterns are shown in Figure 4.

**Figure 4.** XRD patterns for neat V$_2$O$_5$ (a), neat CoO(b) and the coupled V$_2$O$_5$/CoO oxides.

From above XRD patterns for neat oxides, it can be seen a characteristic XRD patterns for each oxide (Figure 4a and 4b).

XRD patterns of cobalt oxide show main peaks at 31, 36, 37 and 45 which correspond to the (220), (311), (222), (400) planes, respectively, of face-centered cubic CoO (Joint committees powder diffraction standards-JCPDS card no. 43-1003). XRD patterns of V$_2$O$_5$ show peaks at 2theta 30, 36, 43, and 62 these peaks are corresponded to the (101), (400), (011) and (301) diffraction planes(Orthorhombic). These are in accordance rhombohedral structure of the V$_2$O$_5$ phase. The XRD patterns of coupled oxides (V$_2$O$_5$/CoO) show the XRD patterns for both oxides (Figure 4c). The crystal structure of each oxide doesn't effect significantly upon mixing and the XRD patterns for each oxide in the mixture are almost similar to that of neat oxide. Also from these patterns it can be noted that, there are some shifting in the values of 2theta for each peak in the mixture in comparison with the individual XRD patterns for each oxide before mixing. This is a good evidence for occurrence of combination between these two oxides to yield the coupled oxide(V$_2$O$_5$/CoO).

XRD patterns for co-oxide are almost containing the main peaks for both neat oxides with changes in peaks positions and their intensities. This observation confirms combination in both oxides under applied conditions to yield co-CoO/V$_2$O$_5$.

### 3.1.2. Fourier transform infra-red spectroscopy(FTIR)

Functional groups of the surface of both neat and coupled oxides were investigated using Perkin Elminer Spectrophotometer Company. Before run, samples were mixed with potassium bromide (KBr) to prepare a disk to run the spectrum. The spectra are presented in.
Figure 3. The band with a peak at 3860 cm\(^{-1}\) is due to the presence of co-ordinated/entrapped water. Two high-frequency intense bands at 567 and 663 cm\(^{-1}\). These two bands, are as metal-oxygen low spin vibrations in octahedral sites\(^ {25,26} \). The peak around 663 cm\(^{-1}\) corresponds to bending modes of vibration of cobalt oxide. The peaks around 2443 cm\(^{-1}\) and 2550 cm\(^{-1}\) is due to OH bond stretching vibration modes.

FTIR spectra of the samples were studied in the sort of 400-4000 cm\(^{-1}\) wavenumber which identifies the chemical bonds in addition to functional groups in the compound. The large broad band at 3060 cm\(^{-1}\) and 3050 are ascribed to the O-H and C-H groups. FTIR spectra of V\(_2\)O\(_5\) NPs exhibited three characteristic vibration modes: V-O vibrations at 659 cm\(^{-1}\), the V-O-V symmetric stretch around 561 cm\(^{-1}\) and the V-O-V asymmetric stretch at 815 cm\(^{-1}\), 941 cm\(^{-1}\) (see Figure 5). As obviously seen, the bands appearing, between 950 and 1020 cm\(^{-1}\) were assigned to a vanadyl stretching modes (\(\delta\) V-O\(^{2+}\)). Bands between 700 and 900 cm\(^{-1}\) were ascribed to the bridging V-O-V stretching. The peaks around 2300-2600 cm\(^{-1}\) are due to OH vibration modes. The peak around 1193 cm\(^{-1}\) is attributed to OH bending modes. FTIR spectra for coupled oxides showed some peaks which represent in almost the peaks of each single oxide around 516 cm\(^{-1}\), 532 cm\(^{-1}\), and 622 cm\(^{-1}\) are related to characteristic metal-oxygen bond for each oxide in the composite. The peaks around 1400 cm\(^{-1}\)-1600 cm\(^{-1}\) is attributed to OH bending modes. The peak around 3400 cm\(^{-1}\) are related to OH stretching vibration modes\(^ {28} \).

Figure 5. FTIR spectra for CoO(a), V\(_2\)O\(_5\) (b) and coupled oxide in a ratio of 1:1 (c).

3.2. Physical properties of the neat and coupled oxides

3.2.1. Porosity of the materials

The porosity of each of neat CoO, V\(_2\)O\(_5\) and the coupled oxides with different ratios. Porosity of these materials was evaluated using impregnation method. The obtained results are presented in Figure 6.
Figure 6. The variation in porosity of V\textsubscript{2}O\textsubscript{5} under different loading ratios of CoO.

From the results that are presented in Figure 6, it is clear that the porosity of V\textsubscript{2}O\textsubscript{5} increases with increase in CoO ratio until reaching to (2.0:2.0) ratio. This probably related to release coordinated water, CO\textsubscript{2} gas as well as other volatile materials. All these released species can produce more vacancies which leads to increase its porosity. Beside that different crystal structure of the oxides (Face - Centered Cubic of CoO and Orthorhombic for V\textsubscript{2}O\textsubscript{5}) can lead to randomly aggregation between these two oxides which can lead to formation of more vacancies\textsuperscript{29}.

3.2.2. Density of neat V\textsubscript{2}O\textsubscript{5} and that doped with CoO
Change in density of neat V\textsubscript{2}O\textsubscript{5} oxide and that coupled with CoO in different ratios was investigated by calculating the obtained densities for each case. From the obtained results it was found that, the density of neat V\textsubscript{2}O\textsubscript{5} was more than that for coupled form. For the coupled oxides the density was lowered and reaching a minimum value at a coupling ratio of (2.0:2.0). This probably arises from random arrangement of the particles of the two oxides in the mixture with leads to increase in the apparent volume with fixation of the final mass of the mixture\textsuperscript{29}. The obtained results are presented in Figure 7.
3.2.3. Electrical conductivity for neat and coupled V$_2$O$_5$

Electrical conductivity of V$_2$O$_5$ was investigated with change in the ratio of doped CoO. The obtained results are presented in Figure 8.

![Figure 7](image1.png)

**Figure 7.** Change in density of coupled oxides with increasing of CoO ratio in the prepared coupled oxides.

![Figure 8](image2.png)

**Figure 8.** Change in electrical conductivity of V$_2$O$_5$ with change in doped CoO ratio.
From the results that are presented in Figure 8, it can be seen that the conductivity was increased with increase of the ratio of CoO in the mixture to reach maximum value at a ratio of (2.0:2.0). This feature can be attributed to the overlapping between the lattices of the two-coupled oxides.

3.3. Preliminary experiments for the photocatalytic activity of the coupled oxides
The activity of the prepared materials was investigated via investigating photocatalytic oxidation of 1-octanol to the corresponding 1-octanal over a suspension of these materials under dark as well as irradiation with UV radiation from medium pressure mercury lamp. Different reaction conditions were performed at 298 K for a period of five hours for each experiment under dark and irradiation with UV radiation. Reactions also were performed under inert and air atmospheric conditions. Similar effects and functionality have been discussed elsewhere for a range of other surface-modified nanoparticles, e.g., ZnONPs and TiO2NPs. The formation of 1-octanal was detected using spectrophotometric method, by following formation of aldehyde group which appears around 295 nm. All conditions that are listed in Table 2 don’t give any product except reaction number five which shows formation of 1-octanal. This means that photocatalytic oxidation of 1-octanol into the corresponding 1-octanal require presence of the catalyst, UV light and air.

### Table 2. Different reaction conditions for performing of the photocatalytic oxidation of liquid 1-octanol

| Expt. no | Reaction conditions                     | results  |
|----------|----------------------------------------|----------|
| 1        | CoO/V2O5 in dark+air+1-octanol         | No product |
| 2        | CoO/V2O5 in dark+N2+ 1-octanol         | No product |
| 3        | 1-octanol +light+air+1-octanol         | No product |
| 4        | CoO/V2O5 +light +N2+ 1-octanol         | No product |
| 5        | CoO/V2O5 +light+air+ 1-octanol         | Product formation |

4. Conclusions
From the obtained results in this study, it can be concluded that combination of CoO with V2O5 leads to change of some surface and bulk properties. Combination of CoO with V2O5 leads to enhance porosity of the co-oxides in comparison with that for neat V2O5. While both of density and electrical conductivity of coupled oxide were reduced in comparison with that for neat V2O5. In terms of crystal structure, from XRD patterns for coupled oxides, it is clear that combination of these oxides under these conditions don’t alter its crystalline structure in spite of appearing new diffraction peaks for CoO/V2O5 in comparison with that for neat oxide. The prepared materials also showed a photocatalytic activity, this was confirmed by photocatalytic oxidation of 1-octanol into its corresponding aldehyde.

5. References
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