



Total Oxidation of Isopropyl Alcohol in Liquid Phase, Under Atmospheric Pressure and at Low Temperature Over Low Cost and Stable Nickel Oxide Based Catalyst Prepared Via Precipitation Approach

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Abstract

Total catalytic oxidation of toxic and carcinogenic isopropanol alcohol (IPA), widely used as a solvent and cleaning fluid, is still one of the most recognized low cost and efficient techniques to remediate indoor environmental pollutants. Our Study aims to the design of a low cost, high and stable nickel oxide-based catalyst for the total oxidation of IPA into organic compounds of carbon dioxide and water under conditions close to those of a normal environment. In a first step, the preparation and characterization by various physicochemical methods of Nickel oxide has been done. Nickel oxide has been crystallized in a face-centered cubic structure with an average grain size at 35.5 nm. FTIR analysis confirmed the XRD results by exhibiting well-defined peaks related to Ni-O bonds which indicate the high quality of the synthesized samples. Textural properties show the good texture of NiO based-catalyst with a specific surface area of 3 m²/g, an average pore diameter of 189 Å and a pore volume of 0.09 cm³/g. The DTG/DTA investigations showed the resistance of nickel oxide to the thermal treatment with a measured weight loss of 1.761 % which could be assigned to the dehydration of the sample and a second weight loss related to the loss of carbon dioxide. Scanning electron microscopy shows that the NiO nanoparticles consist of irregularly shaped grains interconnected with each other.

Nickel oxide prepared by the precipitation method shows good catalytic activity towards the decomposition of the toxic isopropanol in its liquid phase at low temperature with a total destruction duration of isopropanol not exceeding 2h whatever the temperature with formation of acetone as secondary byproduct. Acetone could be formed in an oxidizing environment by oxidative hydrogenation. In this case, the active site would be a pair of redox sites. Moreover, the kinetic study of the isopropanol oxidation reaction, in liquid phase, showed that this reaction follows a successive

mechanism from isopropanol to acetone. The apparent activation energy of nickel oxide was determined to be 4.56 KJ.mol⁻¹. This low value explains the relatively high catalytic activity of NiO based catalyst. It is clear that the low value of the apparent energy of activation is related to the high values of the catalytic activity of nickel oxide.

Keywords: VOCs, Catalytic Oxidation, Isopropanol, Nickel Oxide, Acetone, Catalytic Activity

Introduction

Nowadays, air pollution remediation has gained not only substantial interest and worldwide concerns but also becomes one of the most important public challenges of the 21st century. Volatile organic compounds (VOCs) are liquid complexes of carbon-containing gases such as alcohols, butane, hexane, isopropanol, aldehydes, organic acids, etc. Rapid industrialization and urbanization contribute to the ever-increasing emission of VOCs into the indoor and outdoor atmosphere. These volatile organic compounds are a crucial classes of indoor air pollutants with higher concentrations in common household implements such as printing, dying, wood and surface finishing processes, combustion byproducts and cooking. Meanwhile, these VOCs contaminate the outdoor air due to their widespread uses in industries such as solvents along with its release rates from vehicular exhausts and petroleum refineries. Due to their boiling points ranging from 50 °C to 260 °C at room temperature and under atmospheric pressure [1], high diffusivity, toxicity and volatility, VOCs have the ability to get changed phases and getting emitted to the atmosphere even at ambient conditions and consequently resulted in serious threats to the human health and environment safety through the the formation of ground-level ozone, aerosol and smog [2-4].

Thereby, both of research communities and industries gathered their efforts in developing practical environment recovery technologies in order to effectively remove VOCs including absorption, adsorption, membrane separation, and condensation techniques. Catalytic oxidation has appeared as the low cost and most effective technique for the oxidation of VOCs into CO₂, water, and other relatively less harmful compounds [5-6]. This technology aims to the complete destruction of VOCs instead of its transformation to to another phase as it has occurred in condensation and adsorption approaches. In this technique, the VOCs have been destroyed in the presence of a suitable catalyst at lower temperatures (250°C-500 °C).

Noble metal based catalysts have been widely used in complete VOC oxidation at low temperatures [7-9] thanks to their high catalytic activity. However, its high cost and low thermal stability restricted their use only to industry. In contrast, metal oxide-based catalysts have appeared as promising candidates for VOC removal owing to its outstanding advantages namely its high catalytic performance, low cost and strong resistance to poisoning [5,6,10-13]. Isopropyl alcohol is one of the toxic and carcinogenic VOCs and is widely used as a solvent and cleaning fluid especially for dissolving lipophilic pollutants and cleaning electronic equipments [14].

It is also used as a highly flammable liquid in fragrance Lamp. The exposure to higher concentrations of IPA was harmful to human health.

By doing a literature survey, Dehmani et al [15,16] have prepared Cr₂O₃, Fe₂O₃ and ZnO based metal oxide catalysts by the simple and cheaper precipitation method. Then, they have investigated the Total Oxidation of Isopropanol at low temperature over these catalysts. They reported their good catalytic activities and their use resulted in the total oxidation of isopropyl alcohol into acetone and carbon dioxide as oxidation byproducts. Among all the studied metal oxides, NiO is an inexpensive, environmentally friendly and relatively active in VOC combustion [17,18]. Its high catalytic activity was related to its p-type conductivity with an electron deficiency in the [19]. This facilitate the removal of electrons from the metal cations and led to the formation of active species such as O⁻. There are no work published before on the total oxidation of isopropyl alcohol over NiO based catalyst. This work aims to test the catalytic efficiency of p-type Nickel oxide based catalyst in complete oxidation of isopropanol in the liquid phase at low temperature. At first, we report the low cost and eco-friendly synthesis of Nickel oxide based catalyst via the precipitation approach. Then, a structural, morphological

and optical investigation of the synthesized catalyst has been developed. Finally, we test the total catalytic oxidation of isopropanol in liquid phase at different temperatures.

Experimental Details

Preparation of the adsorbent

A fixed mass of dehydrated nickel nitrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ was dissolved in one volume of distilled water. Then, a molar solution of NH_4OH was added to this mixture by dropwise (7ml/min). The obtained mixture was filtered under vacuum. The obtained powder was washed several times with hot distilled water and then dried overnight in an oven at 100°C . Finally, the resulted powder was get calcined at 500°C for 3h [20].

Catalyst characterization

XRD: The structural variations and phases identification of the NiO based catalyst was analyzed using the X-ray diffractometer (X'PERT MPD-PRO) with a diffracted beam monochromator and Ni filtered $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The used voltage was 45 kV and the intensity was 40 mA. The 2θ angle was scanned between 4° and 30° , and the counting time was 2.0 s at each angle step (0.02°).

FTIR: The structural changes, that were took place during the degradation of the studied samples, were collected in the Fourier Transform Infrared Spectrometer (FTIR) (Shimadzu, JASCO 4100). The samples were prepared in KBr discs from very well dried mixtures of about 4% (w/w). FTIR spectra were recorded by accumulation of at least 64 scans with a resolution of 4 cm^{-1} per sample.

SEM: The morphological study of synthesized sample's surface was recorded using Scanning Electron Microscopy (SEM) by Brand EIF Quanta 200 apparatus equipped with energy dispersive X-Ray spectroscopy (EDX).

BET: The surface area and porosity analysis of the NiO based catalyst were carried out by nitrogen adsorption-desorption measurements at $T = -196^\circ\text{C}$ by using a Micromeritics ASAP 2010 apparatus. The specific surface area and the average pore size distributions were evaluated according to the standard Brunauer-Emmett-Teller (BET) and the Barrett-Joyner-Halenda (BJH) models applied to the adsorption branch of the isotherm.

DTG/DTA measurements: In order to investigate the resistance of the elaborated nickel oxide based catalyst to the thermal

treatment, a differential thermogravimetric analysis (DTA) has been developed. Then, a measurement of the mass of carbon loss and adsorbed water has been done.

Catalytic Tests

To a 250 ml ground-necked flask used as a reactor, 0.15 ml of isopropanol (reagent), 120 mg of catalyst, and 200 ml of distilled water are successively added. The temperature of the water bath which is the reaction temperature was adjusted to the desired value (40°C , 60°C and 80°C) before immersing the reactor flask. The air, which is the oxidizing reagent, was then admitted into the reactor through a lateral tube at a flow rate of about 40 ml/min (more than enough to keep the solution constantly saturated with oxygen). The reaction mixture is stirred continuously during the whole test in order to facilitate the accessibility of the catalyst grains to the reagent and to ensure a work in chemical regime. It should be remembered that each catalytic test is performed with a blank catalyst sample. The quantitative evolution of the products and reactants during the reaction is followed by gas chromatography.

Key Findings

X-Ray Diffraction Analysis

The phase purity, crystallinity, and crystal structure of the NiO based product were evaluated from the XRD patterns. Figure 1(a) shows the XRD diffractogram of Nickel oxide based powder. It's clearly seen, three main reflection peaks corresponding to (111), (200), and (220) planes, which were attributed to the cubic phase of NiO according to the standard data (JCPDS no. 47-1049) [21]. The same positions and intensities of the observed peaks in the diffraction planes were also noticed in literature [22-24]. There are no traces neither of nickel hydroxide nor of salt impurity phase could be observed which clearly indicates the good crystallinity of the elaborated NiO based powder [22-24]. The average crystallite size of the sample was calculated from the X-ray line broadening of the peaks at the (200) and (220) planes using the Debye-Scherrer equation (Eq.1) [25-26];

$$d = 0,94 * \frac{\lambda}{\beta * \cos(\theta)} \quad (\text{Eq.1})$$

With ; λ is the X-ray wavelength (1.54 \AA), β represent the broadening of the line at half themaximum intensity (38.6 and 43.28°) and θ is the Bragg angle.

The average grain size for the sample is 35.5 nm which indicates a high particle size of the prepared Nickel oxide.

FTIR analysis

In order to evaluate the functional groups of synthesized nanoparticles, the chemical structures of NiO were performed through FTIR spectroscopy. Figure 1(b) exhibits the FTIR spectra of NiO NPs. The spectra of NiO NPs show several bands. The broad band observed at about 3420cm^{-1} was attributed to the OH stretching vibration due to chemisorbed water. In addition, the bands that noted below 1000cm^{-1} were attributed to Ni-O bonds vibration [25-26].

Textural analysis

Textural properties such as specific surface area, pore diameter and pore volume have significant effects on the catalytic performance of the synthesized metal-oxide based catalyst.

These physical properties of nickel oxide were investigated by the Nitrogen adsorption/desorption measurements and the obtained isotherm was shown in Figure 1(c). It's obvious that the obtained nitrogen adsorption/desorption isotherm was of IV type with a hysteresis loop of the type of H3 [27-28]. The analytical calculation resulted in a specific surface area of $3. \text{m}^2/\text{g}$, an average pore diameter of 189 \AA and a pore volume of $0.09 \text{ cm}^3/\text{g}$.

TGA/DTA Analysis

The TGA and DTA of Nickel oxide based catalyst are illustrated in Figure 1(d). The curves depicted in Figure 1-d show a weight loss of 1.761% with an endothermic peak between $300 \text{ }^\circ\text{C}$ and $400 \text{ }^\circ\text{C}$ which is assigned to the sample dehydration. The second loss was attributed to the loss of CO_2 .

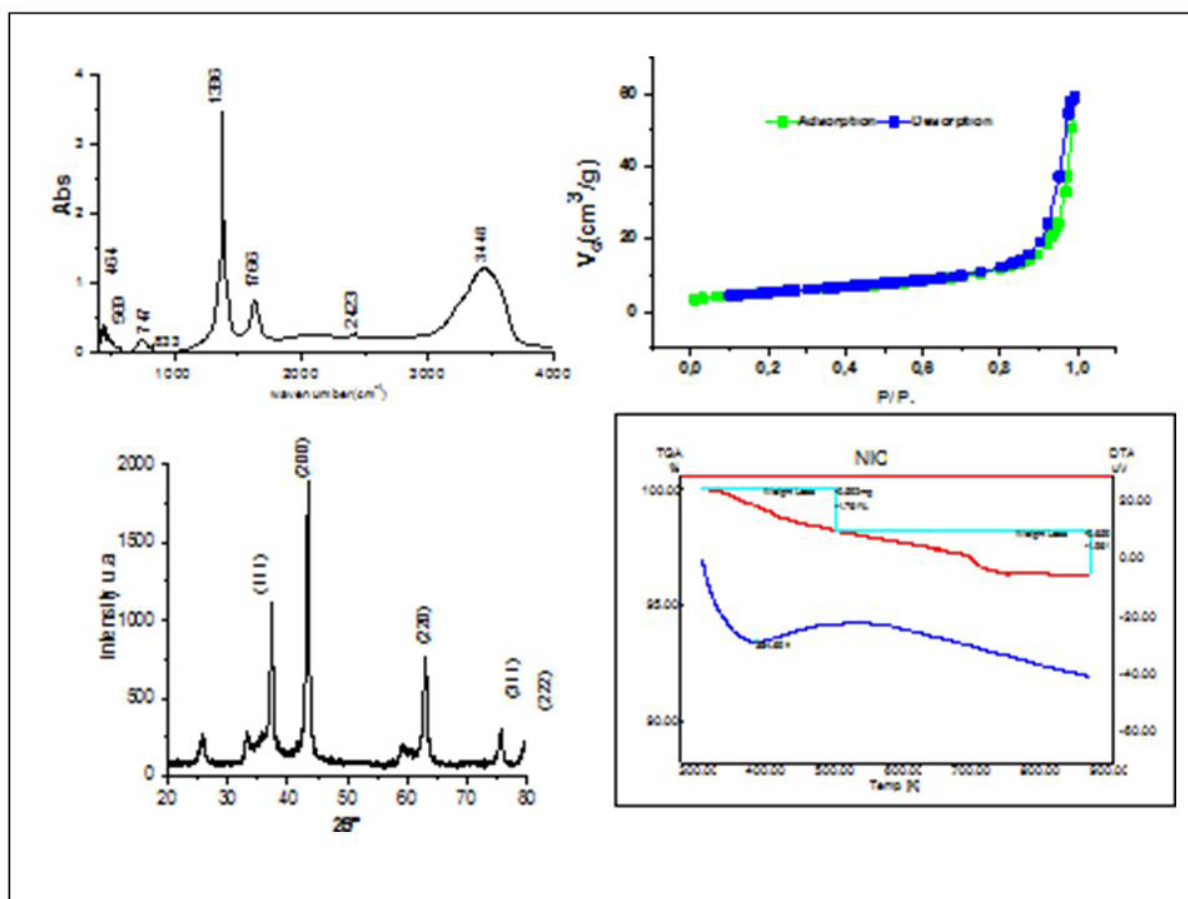


Figure 1: Characterization of nickel oxide by a) XRD, b) FTIR, c) BET, d) DTA/GTA

Morphological investigation

Surface morphology of nanoparticles is a very exclusive tool in order to investigate its microstructure. The scanning electron micrograph of Nickel oxide nanoparticles were shown in Figure 2 with two different scales. As obviously shown in Figure 2 (a) and Figure 2(b), the surface of NiO nanoparticles was covered

with irregularly shaped grains interconnected with each other. It's also evident that this surface consisted of lamellar fibers with distinct rods. The EDX spectrum of NiO depicted in Figure 2 (c). EDX spectrum confirms the presence of nickel and oxygen elements with atomic percentages of 47.7% and 50.1% for Nickel and Oxygen respectively [29].

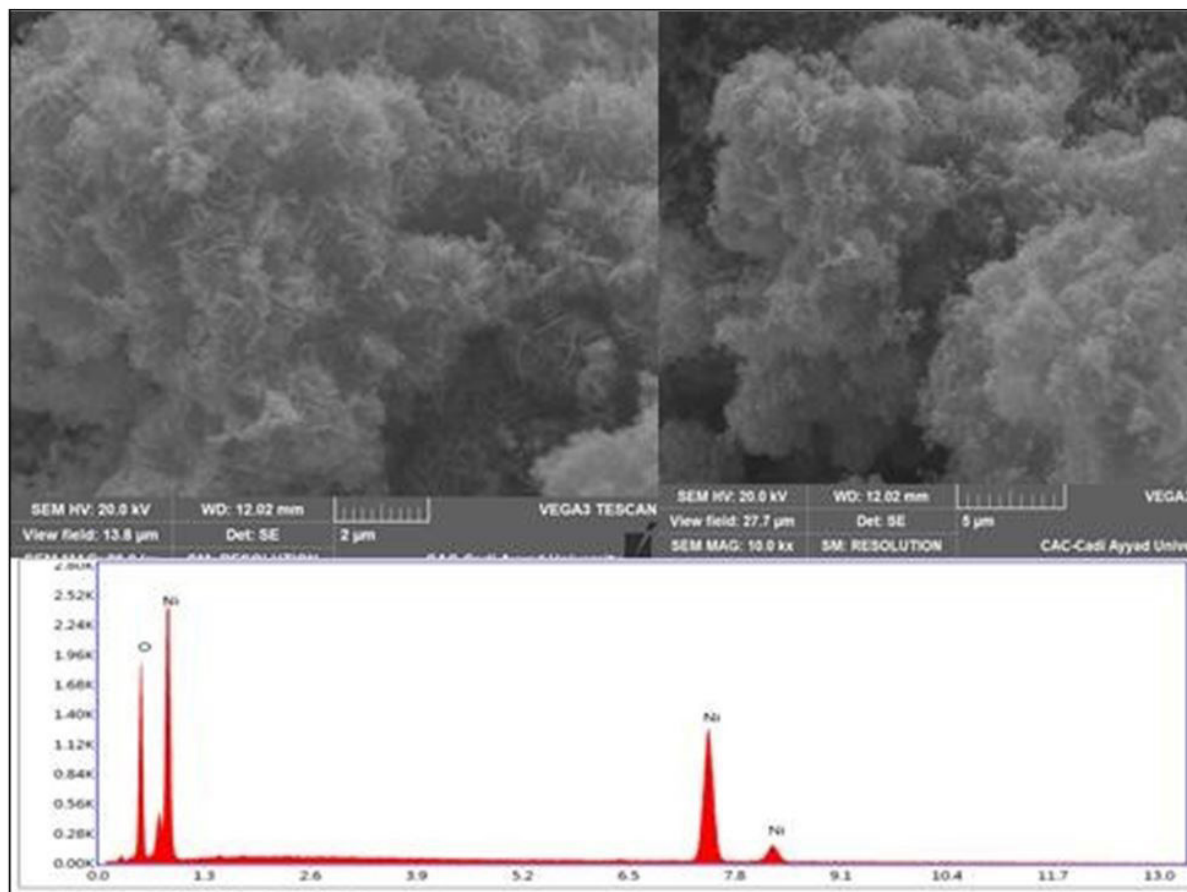


Figure 2: SEM images of the nickel oxide samples at different scales (a) 3 micro mètre, (b) 5 micro mètre. (c) EDX spectrum of nickel oxide

Catalytic oxidation tests

The Catalytic oxidation tests of isopropanol alcohol were carried out at different temperatures including 40 °C, 60 °C and 80 °C over the nickel oxide based catalyst in liquid phase. Figure 3 shows the variation of moles number of isopropanol and its global transformation rate with respect to the time. It is noticeable that, under normal operating conditions of standard pressure and temperature close to room temperature, this NiO catalyst allows the complete transformation of isopropanol into CO₂ after 60 min to 120 minutes [30]. This time decreases with increasing temperature. Thus, the analysis of the curves leads us to deduce that nickel oxide has a better catalytic potential in the total oxidation of isopropanol. Indeed, at any reaction temperature, the specific activity is higher for nickel oxide than

for other catalysts quoted in literature. This result was supported by the results obtained by Kulkarni et al [31] conducted on gas phase oxidation over various transition metal oxides. The difference may be linked to the nature of the metal oxide and its textural properties. In general, the dehydration of isopropanol to propene was usually related to the presence of acidic sites (Brønsted or/and Lewis). Whereas, the dehydrogenation of isopropanol into acetone was related to the presence of basic sites [32]. However, the formation of propene observed on basic zeolite involving many controversies [33,34]. The formation of alkene from alcohol on catalysts with only basic sites was also supported by Alali et al [30]. Therefore, it is concluded that the catalytic oxidation of isopropanol allows us know the nature of the metal oxide surface sites either basic or acid sites

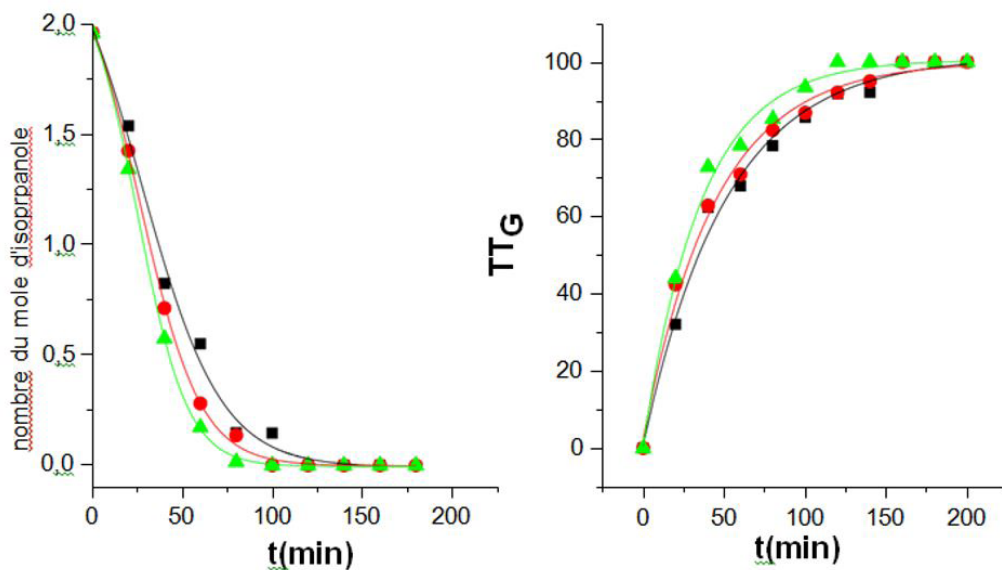


Figure 3: variation of moles number of isopropanol and its global transformation rate as a function of time

The kinetics of the total catalytic oxidation reaction of isopropanol was determined by studying the variation of the global transformation rate of isopropanol (TTG) with the following conversion formula (Eq.2);

$$TTG = (n_0 - n) / n_0 \tag{Eq.2}$$

Also, the rate of transformation of isopropanol into acetone (TTact) was determined by the following relation (Eq.3);

$$TTact = nact / n_0 \tag{Eq.3}$$

With n and nact are the number of moles of isopropanol at time t and acetone respectively and. n0 is the number of isopropanol moles at a time t = 0.

Indeed, the rate of transformation into carbon dioxide (TTCO2) could be calculated via the relation between TTG and TTact as follows;

$$TTCO2 = (TTG) - TTact \tag{Eq.4}$$

Figure 4 displays the evolution curves of the acetone TTact and TTCO2 with respect to TTG at different temperatures for the elaborated NiO based catalyst.

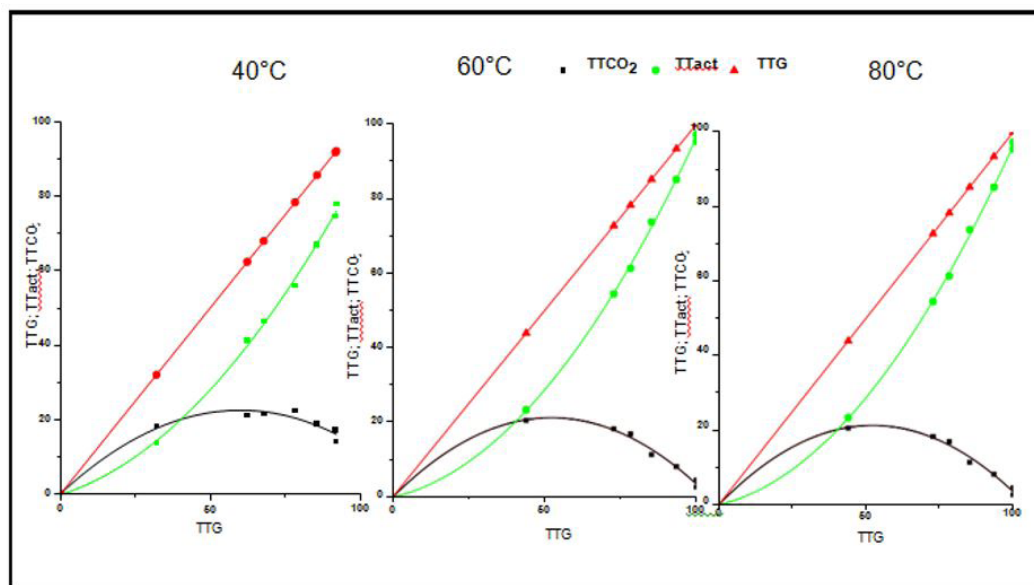


Figure 4: Variation of TTG, TTact and TTCO2 as a function of TTG

These curves show that the oxidation of isopropanol into carbon dioxide is complete at all working temperatures of 40 °C, 60 °C, and 80 °C for a reaction time not exceeding 110 min. The secondary formed product is the acetone which reached its maximum for all temperatures. Note that the remarkable initial selectivity is extrapolated to time 0 (100%), which drops to 0 when the conversion is close to unity [16,35,36]. This implies that the conversion of acetone to CO₂ over the time was verified in this study. In addition, the absence of isopropylene as a reaction byproduct indicates the absence of acidic sites on the nickel oxide surface. It was concluded that the oxidation of isopropanol into acetone proceeds according to a redox mechanism [8,37], with alternation of oxidation and reduction steps of the catalyst according the general scheme [15,38,39].

Isopropanol + oxidized catalyst → reduced catalyst + oxidation byproduct (Eq.5)

Furthermore, the conversion of acetone into carbon dioxide over the time was discussed above, this suggests a successive reaction scheme as follows [11, 13, 18, 40]:

Isopropanol → Acetone → Carbon dioxide (Eq.6)

Apparent activation energy

The isopropanol conversion curves with respect to time at different temperatures could be used to calculate the kinetic parameters such as the constant rate k and the apparent activation energy (E_a) of the catalytic oxidation reaction. The specific activity (a_s) could be expressed in Eq.7;

$$a_s = A \exp(-E_a/RT)^f \quad (\text{Eq.7})$$

where A is the pre-exponential Arrhenius factor and f is a function of the concentrations of the active constituents and the mass of the catalyst. Then, at constant concentrations and variable temperature, a linear expression as a function of the inversed temperature can be written as follows (Eq.8) [20, 41]:

$$\text{Log}(a_s) = \text{Log} A + \text{Log} f - E_a/RT \quad (\text{Eq.8})$$

Figure 5 illustrates the linear transformation of the Arrhenius law $\ln(a_s)$ as a function of $(1/T)$.

It clearly yields a straight line with a regression factor close to 1. The slope of this straight line gives the value of the apparent activation energy which is determined to be 4.56 KJ.mol⁻¹ for the nickel oxide. This low value explains the relatively high activity values.

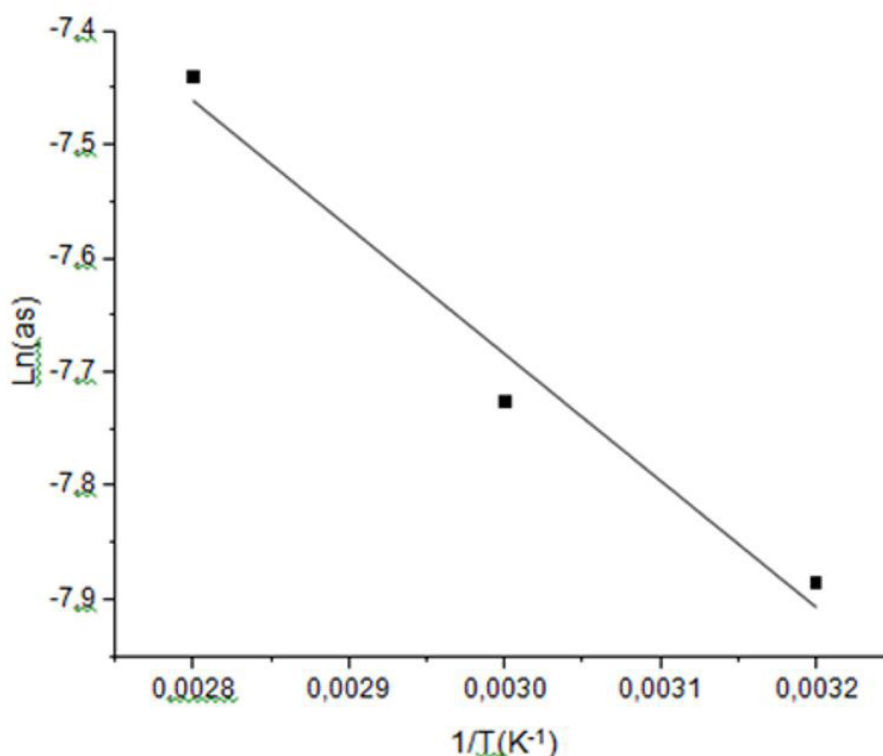


Figure 5: Variation of $\ln(a_s)$ as a function of $1/T$

Conclusion

Nickel oxide based photocatalyst has been successfully prepared via a simple and cheaper precipitation approach. The oxidation of isopropanol (IPA) over this synthesized catalyst has been investigated. In view of structure, nickel oxide has been crystallized in a face-centered cubic structure with an average grain size at 35.5 nm. FTIR spectroscopy confirms the XRD results by showing well defined peaks related to Ni-O bonds. Textural properties show a good texture of elaborated NiO based catalyst with a specific surface area of 3. m²/g, an average pore diameter of 189 Å and a pore volume of 0.09 cm³/g. The DTG/DTA investigation shows the resistance of nickel oxide to the thermal treatment with a measured weight loss of 1.761% which could be assigned to the dehydration of the sample and a second weight loss related to the loss of carbon dioxide. Scanning electron microscopy shows that the NiO nanoparticles consist of irregularly shaped grains interconnected with each other. Nickel oxide prepared by the precipitation method shows good catalytic activity towards the decomposition of toxic isopropanol in its liquid phase at low temperature and with acetone as a mediator byproduct.

Acetone could be formed in an oxidizing environment by oxidative hydrogenation. In this case, the active site would be a pair of redox sites. Moreover, the kinetic study of the oxidation reaction of isopropanol, in liquid phase, showed that this reaction follows a successive mechanism from isopropanol to acetone. The apparent activation energy of nickel oxide was determined to be 4.56 KJ.mol⁻¹. This low value explains the relatively high catalytic activity of NiO based catalyst.

Data Availability

All data underlying the findings of this study are fully available without restriction.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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