



Oxidation of Acetone by CO₂/H₂O₂ over MgO/ γ -Al₂O₃ Catalyst in Liquid-Phase

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Abstract

This article reports the oxidation of acetone by H₂O₂ in the presence of CO₂. It describes the role played by carbon dioxide on the course of the reaction. The reaction was catalyzed by (MgO/ γ -Al₂O₃). The prepared materials were characterized by XRD, FTIR, TGA, TEM, and SEM. Analysis of the reaction products by gas chromatography coupled with mass spectrometry and gas chromatography showed that acetic acid was the main product of the reaction. The improvement in the conversion and selectivity of acetic acid by the use of the oxidizing system (CO₂/H₂O₂) compared to the use of CO₂ alone or H₂O₂ alone is due to the role of the percarbonate entity (HCO₄⁻), formed by the reaction between the carbon dioxide and hydrogen peroxide. Thanks to CO₂/H₂O₂ as a soft oxidizing agent, a conversion of 15.13% and a selectivity in acetic acid of 100% were obtained. This simple, safe, and environmentally friendly method could be an alternative green route for acetic acid production from acetone.

Subject Areas

Chemical Engineering & Technology

Keywords

Liquid-Phase, Carbon Dioxide, Acetone, Hydrogen Peroxide, Oxidation

1. Introduction

Several volatile organic compounds (VOCs) are atmospheric pollutants that contribute to the greenhouse effect. They can undergo secondary reactions leading to ozonation of air, the arising of smog, a grave threat to both human health and the environment, etc. [1]-[3]. The primary sources of VOCs include the burning

of fossil fuels, the manufacture of biofuel and waste incinerators, diesel engines, petroleum refineries, and other industrial processes [4] [5]. More than 300 substances are listed as pollutants, including alcohols, alkanes, halogenated aromatic compounds, polycyclic aromatic hydrocarbons, ketones, and aldehydes [6] [7]. One such example of an oxygenated volatile organic molecule that is employed in many sectors and has a significant impact on both human health and the environment is acetone [8]. Acetone is widely used as a raw chemical in industrial synthesis, which causes it to contribute significantly to VOC emissions [9]. When it comes to acetone removal, catalytic oxidation has been seen as one of the most promising approaches [10]. Undoubtedly, catalysts are classified as noble metal catalysts or non-noble metal catalysts and are essential to the catalytic performance of VOC purification [11] [12]. The noble metal catalysts, primarily platinum (Pt), palladium (Pd), gold (Au), and silver (Ag), exhibit exceptional catalytic performance at low temperatures. Even so, they are expensive and quickly become inactive through sintering and agglomeration [13] [14]. As an alternative to precious metal catalysts, transition metal oxides and their composites have gained traction throughout time because of their inexpensive cost, strong thermal stability, superior anti-toxicity, and catalytic oxidation activity [15] [16]. Because of their great surface area, low cost, and high natural abundance, metal oxides (NiO, Co₃O₄, MgO, ZrO₂, TiO₂, ZnO₂, Al₂O₃, MnO, etc.) have been thought to be effective catalysts for the catalytic oxidation of VOCs. However, low temperatures are necessary to optimize their catalytic activity [17]-[22]. By improving the catalytic performance of metal oxide catalysts, it is essential to achieve a balance between catalytic activity and economic efficiency [23] [24]. It will therefore be of tremendous scientific and technological interest to create low-cost and efficient catalysts for the chemical breakdown of VOCs. The products of the total oxidation of VOCs are H₂O and CO₂ [25] [26]. In addition, the physical-chemical properties of the catalysts and their catalytic performance are primarily determined by factors including specific surface area, metal valence, reactive oxygen species, and oxygen vacancies. The morphology of the catalyst also affects the distribution of the active sites, which ultimately influences the catalytic activity. Thus, creating an appropriate structure can significantly improve the catalytic qualities [27] [28]. Therefore, the development of high-activity and high-stability catalysts is essential to lessen negative environmental effects [29]. As a possible transition metal, magnesium oxides (MgO) display excellent catalytic activity for the oxidation of several VOCs [30]. Because of its remarkable oxygen mobility and large oxygen storage capacity, aluminum has also been employed extensively as a catalytic activator for the oxidation of VOCs [31] [32]. Mg and Al oxides exhibit excellent compatibility and can form a solid solution. Because of Mg and Al synergy, the MgO catalytic activity with Al₃O₂ oxygen storage and release capacity enhances the overall catalytic oxidation performance [33] [34]. Because of the cooperative effects between the elements and the changes in the structural or redox characteristics, the oxide mixture generally exhibits better physico-chemical and catalytic properties than the individual ox-

ides [35] [36]. Therefore, the mixed oxides from hydrotalcite-type precursors consisting of Mg-Al-M (M: Mn, Co, Cu, Fe) are active catalysts in the oxidation of different VOCs thanks to the unique properties of these oxides, such as large surface area, strong thermal stability, good redox properties, and high dispersion of active sites [37]-[40]. Systems based on MgO/Al₂O₃ catalysts are considered efficient catalysts in the oxidation of VOCs. [41] [42]. A process using environmentally friendly reagents such as H₂O₂ and O₂ in the oxidation is highly appreciated. However, O₂ is rarely used because it is kinetically inert and requires harsh conditions of temperature and/or pressure to be activated. H₂O₂ is considered a green oxidant because it only produces H₂O as a by-product; it is a very operative oxidant in liquid-phase reactions. However, its cost limits its application on an industrial scale. Increasing its efficiency can compensate for its cost [43]-[48]. The solution proposed in this work is to use it in a mixture with CO₂. The aim of this study was to develop a facile method to prepare MgO/ γ -Al₂O₃, which is simple, low-cost, and has high activity in synthesis of acetic acid from acetone by a simple, green, and safe method. Catalyzed the H₂O₂/CO₂ mixture and carried out the partial oxidation as a mild oxidant without the use of solvent. The prepared catalyst was extensively characterized using IR, XRD, TGA, SEM, and TEM.

2. Materials and Methods

2.1. Materials

All the reagents used in this work, magnesium nitrate hexahydrate Mg(NO₃)₂·6H₂O (Merck, 99.99%), γ -aluminum oxide γ -Al₂O₃ (Merck, 190 m²/g), acetone CH₃-COCH₃ (Sigma Aldrich, 99.5%), acetic acid CH₃COOH (BDH, 99.8%), hydrogen peroxide H₂O₂ (AVONCHEM, 30%), and sodium hydroxide NaOH (WINLAB, 98%) are of analytical grade and were used as received without further purification.

2.2. Preparation of MgO/ γ -Al₂O₃ Catalysts

The precursor of the (MgO/ γ -Al₂O₃) mixed oxide catalyst with a Mg/Al mass ratio of (1:1) was prepared by the co-precipitation method. To an aqueous molar solution of Mg(NO₃)₂·6H₂O, an aqueous molar solution of NaOH (1 M) is added dropwise (PH 11). The mixture is stirred and kept at 85 °C during the progression of precipitation. After completion of the precipitation, the desired mass of γ -Al₂O₃ was added with stirring while stopping the heating. The stirring was extended for an extra 1 h. Then the resulting co-precipitate was filtrated, washed twice with distilled water, and dried overnight at 120 °C. The precursor binary metal salt was calcined at 450 °C in a stainless steel reactor with an air flow of 6 L/h. It was first heated at 250 °C for 2 hours, then at 450 °C for 1 hour.

2.3. Characterization of MgO/ γ -Al₂O₃ NPs

Powder X-ray diffraction (XRD) patterns were carried out using a D2 phaser

(Bruker, Germany) X-ray diffractometer, the wavelength of X-ray (1.540 \AA). Fourier transform infrared (FTIR) spectra were recorded in a Perkin-Elmer spectrum BX (Perkin Elmer, USA) using the KBr disk method in the range of 400 to 4000 cm^{-1} . Thermogravimetric Analysis (TGA) was studied employing PerkinElmer Thermogravimetric Analyzer 7. The oxide was heated from room temperature to 800°C , and the weight loss was observed. Transmission electron microscopy (TEM) was carried out using a Jeol TEM model JEM-1101 (JEOL, Tokyo, Japan), which was used to determine the shape and size of nanoparticles. Scanning electron microscopy (SEM) was carried out using a Jeol SEM model JSM 6360A (JEOL, Tokyo, Japan). This was used to determine the morphology of nanoparticles.

2.4. Catalytic Oxidation

The oxidation reactions were carried out in a 100 mL stainless steel autoclave equipped with a magnetic stirrer, vent, and manometer. The temperature of the autoclave was modified by a heating jacket. Usually, a mixture of 10 mL of acetone, 20 mL of H_2O_2 (30% in aqueous solution), and 0.5 g of catalyst was magnetically stirred at the desired temperature and CO_2 pressure. The mixture was heated to 75°C , and then pressurized to 5.5 bar under stirring. After 5 h of reaction, the mixture was cooled and analyzed with a gas chromatograph (GC) equipped with a flame ionization detector (FID) using a (Rtx-5-length 30 m , ID 0.53 mm) capillary column. The qualitative analysis of the products was carried out occasionally by gas chromatography-mass spectrometry (GC-MS) using a Thermo Trace GC Ultra gas chromatograph (AI 3000) equipped with a TR-5 MS-SQC capillary column ($30 \text{ m} \times 0.25 \text{ mm}$ internal diameter, phase thickness $0.25 \text{ }\mu\text{m}$) was used with He as the carrier gas (at a flow rate of 1 mL/min).

3. Results and Discussion

3.1. Catalyst Characterizations

3.1.1. XRD Analysis

Figure 1 shows X-ray diffraction patterns for a $(\text{MgO}/\gamma\text{-Al}_2\text{O}_3)$ catalyst. In general, for this type of sample, three main crystalline phases, MgO, $\gamma\text{-Al}_2\text{O}_3$, and MgAl_2O_4 , are observable after calcination at 450°C . The peaks at $2\theta = 31.10^\circ$, 37.69° , 39.67° , 45.94° , 60.96° , and 67.40° are assigned to $\gamma\text{-Al}_2\text{O}_3$ (JCPDS file No.: 01-079-1558) [49]; while those at $2\theta = 36.5^\circ$, 43.0° , 62.3° , 74.7° , and 78.6° are assigned to MgO (JCPDS file No. 87-0653) [50]. Additionally, the MgAl_2O_4 spinel phase was formed, as indicated by the peaks at $2\theta = 19^\circ$, 31° , 37° , 45° , 56° , 59° , and 65° (JCPDS file No. 21-1152) [51]. Indicating that $\gamma\text{-Al}_2\text{O}_3$ is finely dispersed in the MgO structure, possibly resulting in the formation of Al-O-Mg linkages. Thus denoting the combined configuration. In actuality, the catalyst XRD pattern agreed well with previous research.

3.1.2. FTIR Analysis

Figure 2 presents the FTIR spectra of the $(\text{MgO}/\gamma\text{-Al}_2\text{O}_3)$ nanostructure. The

stretching vibration of O-H manifests as the broad bands at 3400 - 3500 cm^{-1} . This band is due to the overlapping of the OH stretching vibration of adsorbed water [52]. The weak intensity peaks at 1630 - 1650 cm^{-1} are attributed to the O-H bending modes due to H_2O molecules adsorbed the surface of $\text{MgO}/\gamma\text{-Al}_2\text{O}_3$ [53]. In addition, it can be attributed to the OH not completely calcined hydroxides [54]. As for the presence of nitrates that could remain on the catalysts after calcination, the results showed that traces of them were still present on the catalyst. In fact, small, sharp absorption bands at 1383 cm^{-1} and 1425 cm^{-1} are related to NO_3 groups. They are the main peaks to prove the remained nitrate groups, suggesting the presence of a small amount of $\text{Mg}_2(\text{OH})_3\text{NO}_3$. The small, sharp band with a low intensity observed at 620 cm^{-1} can be attributed to carbonates and/or nitrates. The adsorption bands at 2350 and 2422 cm^{-1} can be due to CO_2 physisorbed [55].

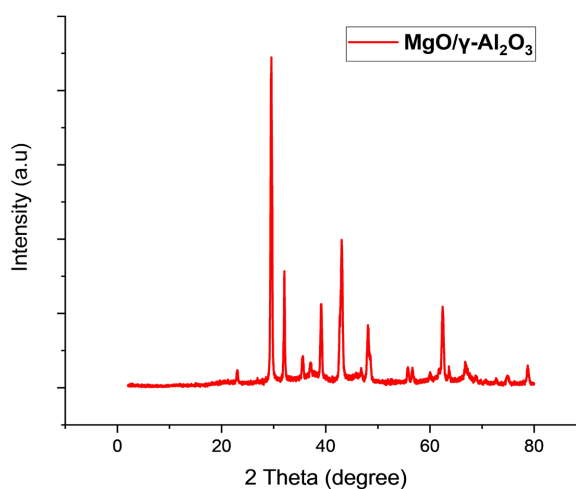


Figure 1. XRD spectra of $\text{MgO}/\gamma\text{-Al}_2\text{O}_3$.

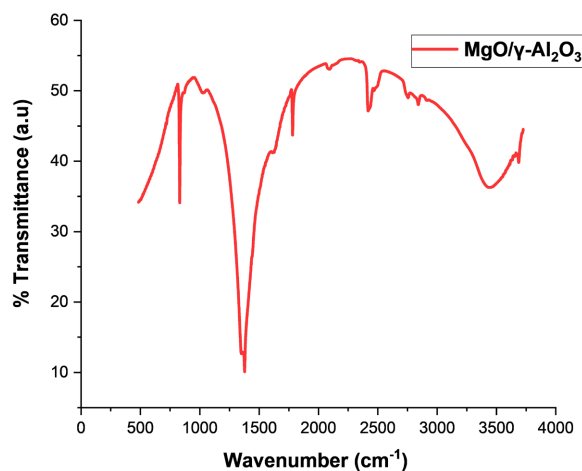


Figure 2. FTIR spectra of $\text{MgO}/\gamma\text{-Al}_2\text{O}_3$.

3.1.3. TGA Analysis

The thermal stabilities of the target substance, the ($\text{MgO}/\gamma\text{-Al}_2\text{O}_3$) catalyst, were monitored using the TGA technique. As can be seen, three degradation steps can

be identified in **Figure 3**. Weight loss up to 250°C corresponds to desorption of physically adsorbed water and crystalline water; weight loss in the region of 350 - 490°C corresponds to the decomposition of magnesium hydroxide; and weight loss at about 500 - 600°C was due to the decomposition of magnesium salts and finally producing magnesium oxide MgO [56] [57]. In addition, as there was no discernible weight loss, this finding verified that a stable composite was successfully created in the calcined catalyst up to 700°C.

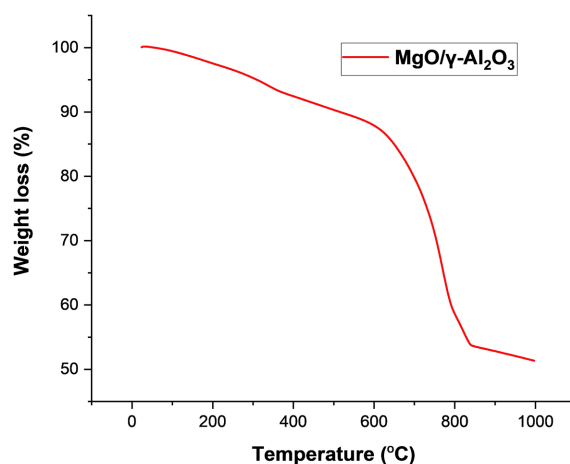


Figure 3. TGA curves of MgO/γ-Al₂O₃.

3.1.4. TEM Analysis

For better understanding of the structural and morphological characteristics of the synthesized (MgO/γ-Al₂O₃) nanoparticles, the products have been further examined by TEM, as shown in **Figure 4**. It can be seen from the image two kinds of particles, spherical and hexagonal. The size of spherical particles was in the range of 10 - 100 nm. The hexagonal particle size was in the range of 50 - 80 nm.

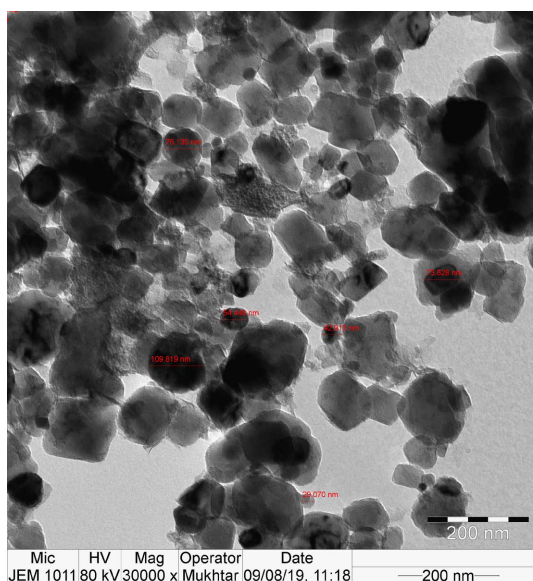


Figure 4. TEM micrograph of MgO/γ-Al₂O₃.

3.1.5. SEM Analysis

The surface morphology and elemental composition of (MgO/ γ -Al₂O₃) mixed oxide was studied by scanning electron microscopy (SEM), as shown in **Figure 5**. It is clearly seen that the MgO/ γ -Al₂O₃ product is composed of an agglomeration of irregular and hexagonal-like morphologies with spherical flakes. White spot agglomerates of the MgO nanoparticles were due to the hygroscopic nature of the material [58] [59]. As can be shown, there is additional proof of the catalyst nanostructure and catalyst-support compatibility in the nearly identical particle sizes acquired from both TEM and SEM.

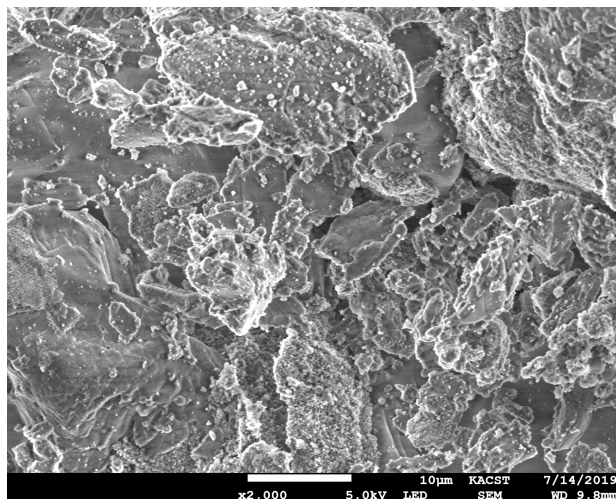
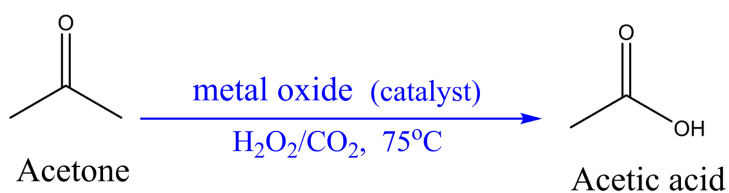


Figure 5. SEM images of MgO/ γ -Al₂O₃.

3.2. Catalytic Activity

The catalytic activity of the (MgO/ γ -Al₂O₃) was evaluated for the reaction of the oxidation of acetone. The experiments were carried out under a (CO₂/H₂O₂) pressure of 5.5 bar at 75 °C during 5 h. The reaction products were analyzed by GC and GC-MS. The results showed that acetic acid was obtained as a major product (**Scheme 1**).



Scheme 1. Main product obtained by oxidation of acetone with H₂O₂ in the presence of CO₂ over MgO/ γ -Al₂O₃ catalyst.

3.2.1. Effect of H₂O₂/CO₂ Oxidizing System

By oxidizing acetone with CO₂, H₂O₂, and a combination of H₂O₂ and CO₂, the impact of the content of the oxidizing system on the conversion and selectivity was investigated. The results (**Table 1**) show that in the case of oxidation by CO₂ alone and H₂O₂ alone, the conversions obtained are 0.18% and 5.20%, respec-

tively. It is interesting that there was an important 15.13% increase in conversion when acetone was oxidized utilizing a mixture of H₂O₂/CO₂. The improvement in the oxidation of acetone by the H₂O₂/CO₂ oxidizing system is the result of two factors. The first is the efficient activation of H₂O₂ by (MgO/ γ -Al₂O₃), and the second is the development of a percarbonate species, a powerful oxidant formed by the reaction of CO₂ with H₂O₂.

Table 1. Effect of the composition of the oxidizing system on the conversion and selectivity of the product for the reaction catalyzed by (MgO/ γ -Al₂O₃). Reactions conditions: T = 75 °C; P (CO₂) = 5.5 bar; (H₂O₂/acetone: 2) volume ratio; Rt = 5 h, and m (cat) = 0.5 g.

Oxidant	Conversion (%)	Selectivity of Acetic acid (%)
CO ₂	0.18	90
H ₂ O ₂	5.20	92
CO ₂ /H ₂ O ₂	15.13	100

3.2.2. Effect of Reaction Temperature

The oxidation of acetone was carried out at different temperatures while the CO₂ pressure was set at 5.5 bar. When utilizing a CO₂/H₂O₂ oxidizing agent system, the results (Figure 6) demonstrated that the temperature has an important effect on the process. The conversion increased quickly from 8.4 to 15.1% when the temperature rose from 35 °C to 75 °C. As for the selectivity of the product, it is obvious that the acetic acid selectivity was between 90.1% and 100%. Based on the aforementioned findings, it seems that the reaction's temperature has an important effect on the conversion and product selectivity.

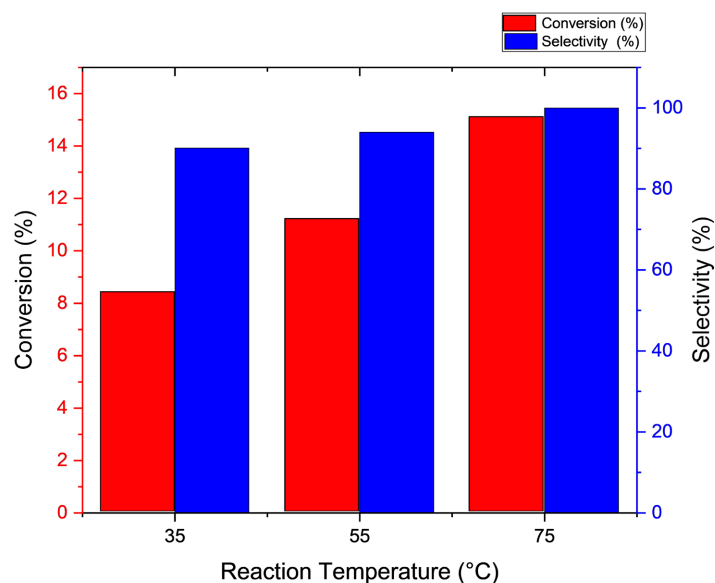


Figure 6. Effect of reaction temperature on the conversion and product selectivity over MgO/ γ -Al₂O₃ catalyst. Reactions conditions: T = 75 °C; P (CO₂) = 5.5 bar; (H₂O₂/acetone: 2) volume ratio; Rt = 5 h, and m (cat) = 0.5 g.

3.2.3. Effect of CO₂ Pressure

As seen in **Figure 7**, the conversion of acetone was significantly improved from 9.1% to 15.13% upon increasing P (CO₂) from 1.5 to 5.5 bars. As for acetic acid selectivity, it can be seen that CO₂ pressure has no significant influence. Hence, these results reflect the importance of CO₂ pressure control.

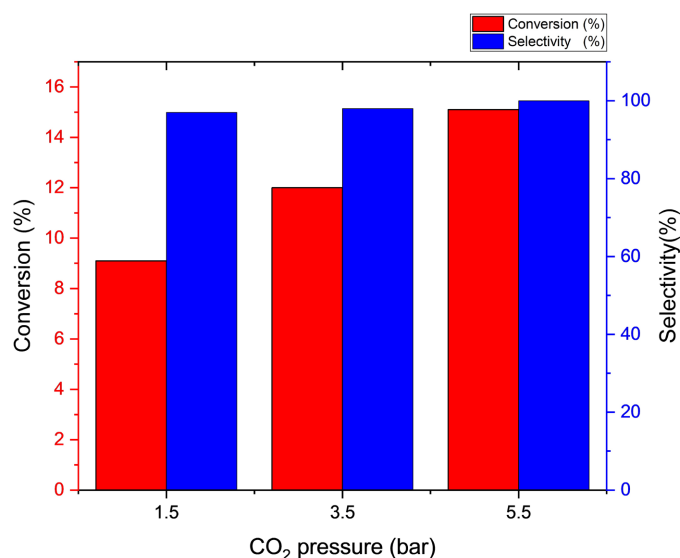


Figure 7. Variation of conversion and selectivity as a function of CO₂ pressure over MgO/ γ -Al₂O₃ catalyst. Reaction conditions: T = 75 °C; (H₂O₂/acetone: 2) volume ratio; Rt = 5 h, and m (cat) = 0.5 g.

3.2.4. Catalyst Recycling

Reusability and activity are seen to be the most crucial metrics for industrial catalyst selection [60]. In liquid phase oxidation processes, solid catalyst stability against leaching of the active species under turnover circumstances and the catalytic nature are essential characteristics that need to be thoroughly examined [61]. The reusability of the MgO/ γ -Al₂O₃ catalyst in the oxidation of acetone (10 mL) was examined. The experiments were carried out at 75 °C for 5 h under (CO₂ = 5.5 bar, H₂O₂ = 20 mL). After the first run, the catalyst was separated from the reaction mixture by centrifugation, washed with acetone several times, and dried in an oven (80 °C for 24 h) to remove the impurities deposited on the catalyst. As can be seen in **Figure 8**, after three runs, a reduction in the acetone conversion from 15.13% to 14.2% was observed; however, the acetic acid selectivity remained almost constant, confirming the stability and reusability of the catalyst.

4. Conclusion

The aim of this work was to fabricate a catalyst (MgO/ γ -Al₂O₃) for the conversion of acetone to acetic acid using a solvent-free method and (H₂O₂/CO₂) as an oxidant. Hence, the (MgO/ γ -Al₂O₃) catalyst was prepared by using the co-precipitation method and then characterized using XRD, FTIR, TGA, TEM, and

SEM. Our systematic experiments involved varying critical parameters such as oxidation system, temperature, pressure, and repeated use of catalyst to optimize acetic acid selectivity and acetone conversion. The results of this study demonstrated a 15.13% acetone conversion to acetic acid with a selectivity of 100%.

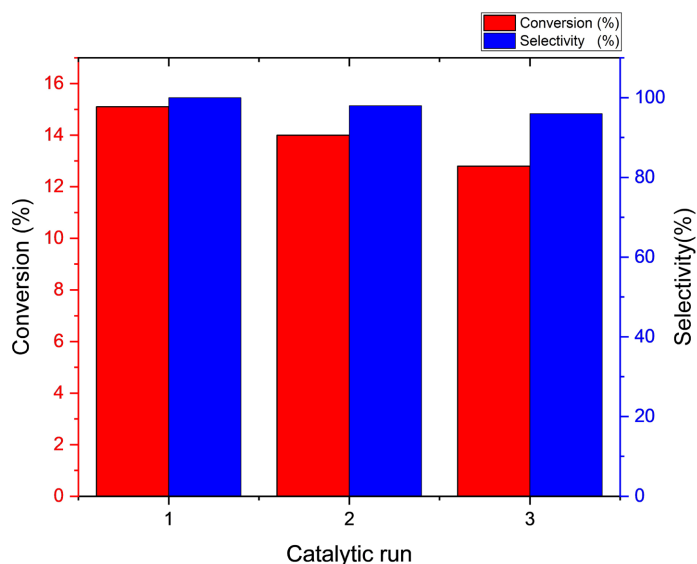


Figure 8. Reusability of (MgO/ γ -Al₂O₃). Reactions conditions: T = 75 °C; P (CO₂) = 5.5 bar; (H₂O₂/acetone: 2) volume ratio; Rt = 5 h, and m (cat) = 0.5 g.

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Conflicts of Interest

The authors declare no conflicts of interest.

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