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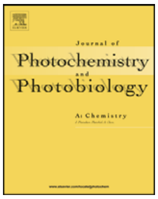

Nanoparticles TiO_2-photocatalyzed oxidation of selected cyclohexyl alcohols

Omima S. Mohamed, Saleh A. Ahmed, Mostafa F. Mostafa, Aboel-Magd A. Abdel-Wahab*

Chemistry Department, Faculty of Science, Assiut University, Assiut 7111, Egypt

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ABSTRACT

The present article embraces the photocatalytic oxidation (PCO) of three selected cyclohexyl alcohols namely cyclohexylmethanol (**1**), 2-cyclohexylethanol (**2**) and 1-cyclohexylethanol (**3**) using nanoparticles titanium dioxide (TiO_2-P25) as a semiconductor photocatalyst in acetonitrile under aerated conditions. The photocatalytic experiments showed moderate conversion ratios (55.2%, 51.8% and 60.3%, respectively). The primary photocatalytic oxidation products are the corresponding aldehydes or ketones and acids. The photocatalytic oxidation products were identified mainly by GC and GC/MS techniques using authentic samples. Formation of electron–hole pair at the surface of the semiconductor nanoparticles followed by oxidation reaction was the suggested mechanism. A first-order kinetic model was observed for the photocatalytic oxidation of the investigated alcohols and the rate constants were calculated.

1. Introduction

A wide range of oxygen-containing molecules including alcohols, aldehydes, ketones, epoxides and carboxylic acids are manufactured by chemical plants and utilized as starting materials for producing, in particular, plastics and synthetic fiber materials. For producing oxygen-containing materials, such as carboxylic acids like adipic acid, nicotinic acid and pyromellitic acid, nitric acid has been widely used as a useful chemical oxidizing agent [1,2].

Although a few compounds can undergo direct reactions when they are exposed to light, a wide range of other organic compounds can be destroyed in the photocatalytic processes [3,4]. Photocatalytic processes can take place either in a homogeneous medium or in a colloidal (heterogeneous) suspension of semiconductor particles, e.g. titanium dioxide [5]. Photocatalytic effects of semiconductors have been of much interest from the view point of solar energy utilization [6]. Photocatalytic oxidation (PCO) has been applied not only to produce useful chemicals [7], but also to convert pollutants in waste streams to innocuous or less harmful forms [8–10]. The PCO process is non-toxic and environmentally friendly, as well as an important goal of the "green chemistry" concept [11,12].

Detailed applications of the photocatalytic oxidation of alcohols have been extensively reviewed [13–22]. Fox et al. [23] reported that photoinduced oxidation of primary, secondary and tertiary alcohols by several tungstates of varying structural complexity ($WO_2(OR)_2$, $PW_{12}O_{40}{}^{3-}$ or $(WO_3)_n$) requires strong alcohol precomplexation before photoactivation and occurs via a rapid two-electron transfer. PCO of propan-2-ol to propanone using suspensions of titanium dioxide irradiated with filtered UV was also studied [13]. Fox and Abdel-Wahab [18], studied the PCO of multifunctional organic molecules and the effect of an intramolecular aryl thioether group on the semiconductor-mediated oxidation/dehydrogenation of primary alcohols in dry acetonitrile.

Also, the PCO of ethanol, benzyl alcohol, cinnamyl alcohol, *n*-hexyl alcohol, isopropyl alcohol, cyclopentanol and cyclohexanol afforded the corresponding aldehydes or ketones by using oxygen gas and visible light 488 nm (85 mW) illumination from an Ar^+ laser, the process is catalyzed by H_2PtCl_6 and $CuCl_2$ [14]. The photocatalytic dehydrogenation of aliphatic alcohols as methanol, ethanol, ethylene glycol and 2-methylpropan-2-ol in aqueous suspensions of platinized titanium dioxide was reported also [15]. The regioselectivity in the semiconductor-mediated photooxidation of 1,4-pentanediol was studied in 1989 by Fox et al. [16].

Recently, PCO of aryl alcohols [21] showed a high oxidation rate of benzyl alcohol to benzaldehyde and 1-phenylethanol to acetophenone (92% and 95%, respectively). Also, Pillai and Salhe-Demessie [19] studied the PCO of benzyl alcohol, 2-phenylethanol and 1-phenylethanol. However, the photocatalytic oxidation of cyclohexylalkanols remained intact.

The high selectivity photocatalytic oxidation of alcohols to carbonyl compounds are one of the most important chemical transformations in industrial chemistry. Carbonyl compounds such as ketones and aldehydes are the precursors for many drugs,

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* Corresponding author. Tel.: +2 10 0104909; fax: +2 88 2342708.
E-mail address: wahab_47@hotmail.com (A.A. Abdel-Wahab).

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vitamins, and fragrances and are also important intermediates for many complex syntheses.

As a continual interest in our laboratory on PCO at irradiated semiconductors [17,18,21,22], we present here the heterogeneous photocatalytic oxidation of selected cyclohexyl alcohols (**1**–**3**) by using TiO_2 nanoparticles, as a superior photocatalyst, mainly for synthetic and mechanistic purposes.

2. Experimental

2.1. Materials

Titanium dioxide (P25 Degussa-Hűls AG, ca. 80% anatase and 20% rutile, powder) with surface area of 37.7 $m^2 g^{-1}$, average particle size 21 nm and pore diameter 31.0 Å was dried at 120 °C for 24 h before use. Oxygen was dried by passage through a drying calcium chloride tube. Acetonitrile (Aldrich, HPLC grade) was distilled before use. Cyclohexylmethanol (**1**) [24,25], 2-cyclohexylethanol (**2**) [26] and 1-cyclohexylethanol (**3**) [27] were synthesized as reported and were distilled before use. The authentic samples of cyclohexanecarboxaldehyde (**4**) [28,29], cyclohexanecarboxylic acid (**5**) [30], cyclohexaneacetaldehyde (**6**) [28], cyclohexylacetic acid (**7**) [31], cyclohexyl methyl ketone (**8**) [32] were synthesized as described in the literature. Cyclohexanol (**9**), cyclohexanone (**10**) and 1,4-cyclohexadione (**11**) were purchased from Aldrich Chemical Company and were distilled before use. The chemical structures of the synthesized authentic samples were determined by both spectroscopic (IR, ^{1}H NMR) and analytical (GC, GC/MS) techniques.

2.2. Apparatus

All the photocatalytic reactions were carried out in a cell made of vycor glass (internal diameter 2.5 cm and length 29.5 cm), fitted with a reflux condenser capped with $CaCl_2$ drying tube and externally irradiated with a 450 W medium pressure mercury lamp (ACE glass, immersion type) with a pyrex well which has maximum emission at 296.7–578 nm (4.18–2.15 eV). The system was covered with aluminium foil which served as light reflector to decrease light loss and the apparatus was set up in a metallic cabinet. The distance between sample and irradiation source was 5 cm.

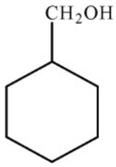
IR spectra were recorded using Shimadzu IR-470 spectrophotometer, using thin film and NaCl disks. The electronic absorption spectra were recorded using UV-2101 PC, UV–vis scanning spectrophotometer-Shimadzu. ^{1}H NMR spectra were carried out using 90 MHz Varian 390 instrument in $CDCl_3$ and TMS as an internal standard. GC analyses were performed employing Perkin Elmer, Auto system XLGC using capillary column packed with 5% diphenyl–95% dimethyl polysiloxane, 30 m length, 0.32 mm internal diameter and 0.25 μm film thickness. The initial temperature is 40 °C and the final temperature is 280 °C, rate of heating is 20 °C/min. The injector temperature is 300 °C and the detector is a flame ionization detector heated at 250 °C. GC/MS analyses were carried out using GC model: GC 2000 Thermo, capillary column DB-5 (5% phenyl–95% methyl polysiloxane), 30 m length, 0.25 mm internal diameter, 0.25 μm film thickness, temperature of column range from 50 to 300 °C (10 °C/min), and injector temperature 250 °C, attached with mass spectrometer model SSQ 7000 produced by Finnigan. All melting points were determined on a Gallen-Kamp melting point apparatus.

2.3. General procedure

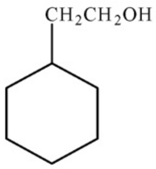
A 75 ml (0.1 M) solution of the investigated alcohols (**1**–**3**) was prepared in acetonitrile, 75 mg of titanium dioxide (P25 Degussa-Hűls AG) was added to the solution, the mixture was transferred to the reaction vessel and sonicated in an ultrasonic bath for 15 min. The stirred suspension was bubbled with a stream of oxygen gas at a flow rate 30 ml/min and then illuminated for 20 h using 450 W medium pressure mercury lamp. The reaction progress was monitored by GC. After irradiation, the titanium dioxide powder was removed by filtration using a medium porosity frit and the solvent was evaporated by using a rotatory evaporator. The reaction mixture was analyzed by using GC and GC/MS techniques, and then the products were identified and estimated with convincing match qualities compared with the standard mass spectra in the mass spectral libraries and with their retention time matched with authentic samples.

3. Results and discussion

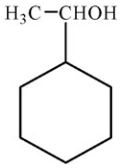
As indicated in Section 1, it became of special interest to investigate the photocatalytic oxidation of selected cyclohexylalkanols (cyclohexylmethanol, 2-cyclohexylethanol and 1-cyclohexylethanol, **1**, **2** and **3**, respectively), to through some light on the effect of the nature of the alcohol on the efficiency of oxidation, oxidation products and potential use of PCO for synthetic purposes.



Cyclohexylmethanol (1)



2-Cyclohexylethanol (2)



1-Cyclohexylethanol (3)

3.1. PCO of cyclohexylmethanol (**1**) in acetonitrile

Cyclohexylmethanol (**1**) as a primary alcohol, gave upon PCO the corresponding cyclohexanecarboxaldehyde (**4**) which converted via further oxidation to cyclohexanecarboxylic acid (**5**). Also, formation of cyclohexanol (**9**) and cyclohexanone (**10**) is not unexpected through decarboxylation [33] of cyclohexanecarboxylic acid (**5**) to cyclohexane (**12**). According to the results of Larsen et al. [34,35] cyclohexane finally produce cyclohexanone (**10**) under the prevailing conditions (Scheme 1; Table 1, Exp. 1). Isolation of cyclohexanol (**9**) supports this route.

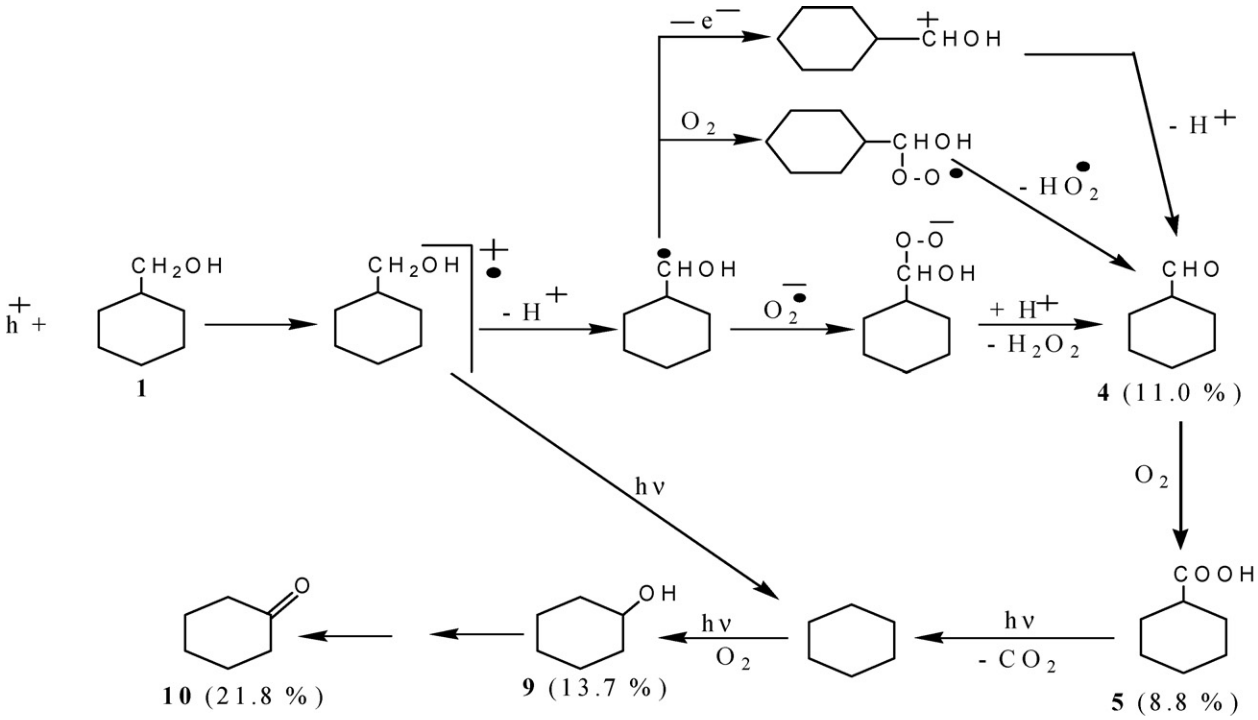
It is interesting to mention that, upon increasing the time of illumination to 50 h, cyclohexanol (**9**) disappeared, the concentration of cyclohexanone increased (**10**), 1,4-cyclohexadione (**11**) was formed and the amount of cyclohexanecarboxylic acid (**5**) increased on the expense of cyclohexanecarboxaldehyde (**4**) and other products (**13**, **14**) (Table 1, Exp. 2).

3.2. PCO of 2-cyclohexylethanol (**2**) in acetonitrile

2-Cyclohexylethanol (**2**) was oxidized to the corresponding aldehyde cyclohexaneacetaldehyde (**6**) which upon further oxidation converted to cyclohexaneacetic acid (**7**). Decarboxylation [32] of cyclohexylacetic acid (**7**) to methylcyclohexane is suggested as the precursor for cyclohexylmethanol (**1**) [36,37] (Scheme 2). As discussed in Scheme 1, cyclohexylmethanol (**1**) can give cyclohexanecarboxaldehyde (**4**), cyclohexanecarboxylic acid (**5**), cyclohexanol (**9**) and cyclohexanone (**10**) [34,35] (Table 1, Exp. 3).

3.3. PCO of 1-cyclohexylethanol (**3**) in acetonitrile

Also, PCO of 1-cyclohexylethanol (**3**) as a secondary alcohol, gave the corresponding ketone cyclohexyl methyl ketone (**8**), which



Scheme 1. Suggested mechanism for PCO of **1** in acetonitrile.

transformed by further oxidation to 2-acetylcyclohexanone (**15**). As discussed before the side chain cleavage of 1-cyclohexylethanol (**3**) gave cyclohexane which photocatalytically oxidized to cyclohexanol (**9**) and cyclohexanone (**10**) [34,35] (Scheme 2; Table 1, Exp. 4).

From the data listed in Table 1, Exp. 5, it is clear that increasing the reaction time resulted in the disappearance of cyclohexanol product (**9**), increase in the concentration of cyclohexanone (**10**) and formation of the 1,4-cyclohexandione (**11**) and 2-(1-hydroxyethyl)-cyclohexanone (**16**) [22].

The percentage conversion of cyclohexylmethanol (**1**) is greater than that of 2-cyclohexylethanol (**2**) (55.2% and 51.7%, respectively, Table 1). This is in agreement with the values of their heats of formation (−85.4 and −91.3 kcal/mol, respectively) [38].

The effect of different interactive parameters, viz., oxygen, light and TiO_2 has been experimental investigated. No oxidation products were detected upon irradiation of cyclohexylmethanol (**1**) in the absence of catalyst and/or light. Furthermore, experiments carried out in the absence of oxygen showed also no significant evidence of products formed. All these results confirm the essential role of oxygen, TiO_2 and light in the PCO process [17,21].

Also, a polar, nonhydroxylic solvent (CH_3CN) represents the best opportunity for controlling oxidative reactivity at the interface between a liquid reaction mixture and a solid irradiated photocatalyst [17,39].

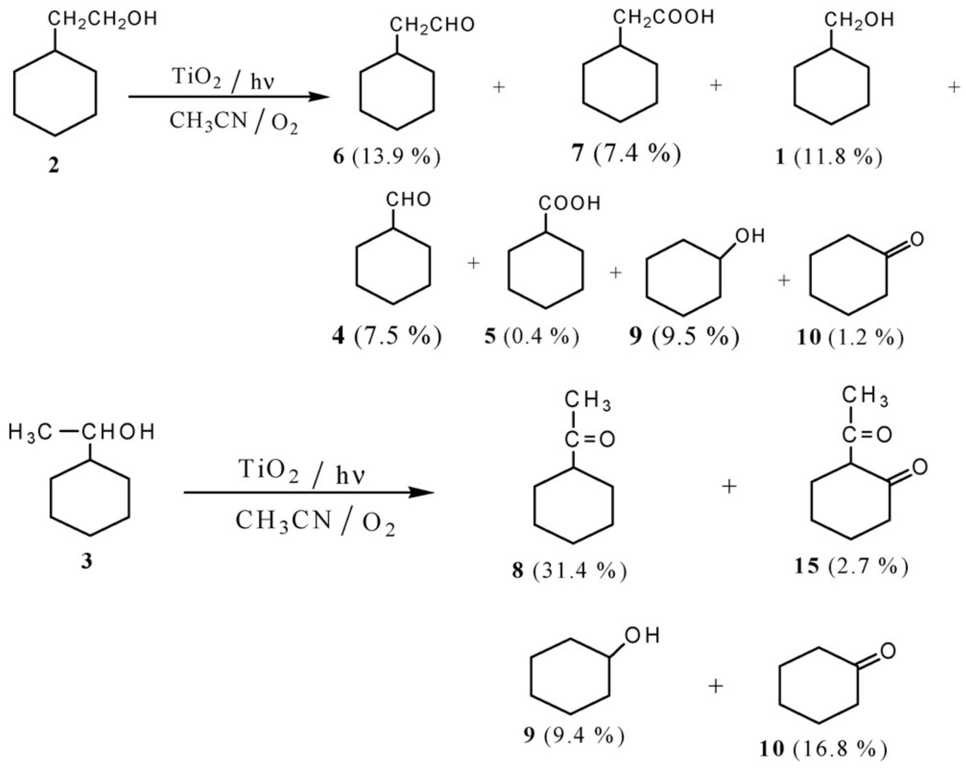
In addition, the UV absorption spectra of the target compounds (**1**–**3**) have been inspected to eliminate the possibility of direct excitation. The maximum absorption of the investigated alcohols (**1**–**3**) ranges between 277.1 nm for **2** and 283.5 nm for **1**, a range that will be cut-off by Pyrex glass [17]. As the maximum emission of the used 450 W medium pressure mercury lamp ranges between 296.7 and 578.0 nm (4.18–2.15 eV), the obtained products are therefore originated through initial band gap excitation of TiO_2 (3.23 eV) [40,41], but not through direct excitation of the alcohol.

When titanium dioxide is irradiated by UV, it generates h^+_{VB}/e^-_{CB} pairs, as a result of optical excitation, and these species migrate to the solid surface (Eq. (1)), which must be trapped in order to avoid recombination, i.e. charge separation is essential for any photocatalytic electroprocess:

$$TiO_2 \xrightarrow{h\nu} [TiO_2^*] \rightarrow e^-_{CB} + h^+_{VB} \qquad (1)$$

Table 1
PCO of the cyclohexyl alcohols **1**–**3** in acetonitrile

Exp.	Substrate	Time (h)	Conv. (%)	Products (no, yield %)
1	Cyclohexylmethanol (**1**)	20	55.2	Cyclohexanecarboxaldehyde (**4**, 11.0), cyclohexanecarboxylic acid (**5**, 8.8), cyclohexanol (**9**, 13.7) and cyclohexanone (**10**, 21.8)
2	Cyclohexylmethanol (**1**)	50	78.7	Cyclohexanecarboxaldehyde (**4**, 3.5), cyclohexanecarboxylic acid (**5**, 25.9), cyclohexanone (**10**, 44.5), 1,4-cyclohexadione (**11**, 1.2), and other products (**13**, 2.2 and *m*/*e* = 86) and (**14**, 1.5 and *m*/*e* = 100)
3	2-Cyclohexylethanol (**2**)	20	51.8	Cyclohexaneacetaldehyde (**6**, 13.9), cyclohexylacetic acid (**7**, 7.4), cyclohexylmethanol (**1**, 11.8), cyclohexanecarboxaldehyde (**4**, 7.5), cyclohexanecarboxylic acid (**5**, 0.4), cyclohexanol (**9**, 9.5) and cyclohexanone (**10**, 1.2)
4	1-Cyclohexylethanol (**3**)	20	60.3	Cyclohexyl methyl ketone (**8**, 31.4), cyclohexanol (**9**, 9.4), cyclohexanone (**10**, 16.8) and 2-acetylcyclohexanone (**15**, 2.7)
5	1-Cyclohexylethanol (**3**)	75	81.0	Cyclohexyl methyl ketone (**8**, 46.5), cyclohexanone (**10**, 24.2), 1,4-cyclohexadione (**11**, 2.3), 2-acetylcyclohexanone (**15**, 5.5) and 2-(1-hydroxyethyl)-cyclohexanone (**16**, 2.6)



Scheme 2. PCO of **2** and **3** in acetonitrile.

The surface of the semiconductor becomes so strongly oxidizing power, a photo-generated hole (+2.4 V vs. SCE in acetonitrile) [16] that can initiate the oxidation of many organic materials at the solid–liquid interface by converting dissolved oxygen, water or hydroxyl ions to hydroxyl radicals $^{\bullet}OH$ and others, such as $O_2^{\bullet-}$ and $HOO^{\bullet}$. The use of nonaqueous reaction medium rules out the participation of hydroxyl radicals generated by water trapping of the hole, in the oxidation process [42]. From the product distribution (Table 1) and reaction products profiles (Schemes 1 and 2), the reaction mechanism of TiO_2-sensitized photooxidation of the target alcohols (**1**–**3**) in aerated acetonitrile has been proposed as indicated in Schemes 1 and 2. The photo-generated electron can be trapped by adsorbed oxygen to form superoxide or other negatively charged adsorbed oxygen species:

$$O_2(\text{solution}) \rightleftharpoons O_2(\text{ads}) \tag{2}$$

$$O_2(\text{ads}) + e^- \rightarrow O_2^{\bullet-}(\text{ads}) \xrightarrow{H^+} {}^{\bullet}OOH \xrightarrow[+H^+]{+e^-} H_2O_2 \tag{3}$$

On the other hand, the adsorbed alcohol species in dry acetonitrile are the likely traps for holes forming the radical cation, by single electron transfer, which rapidly deprotonated producing α-hydroxy radical of reasonable stability [16].

Two fates are available to such radical. (i) A second electron oxidation: the latter intermediate is extremely easily oxidized [16] by loss of a second electron generating a protonated carbonyl intermediate. This intermediate gives the proton to the environment forming the corresponding carbonyl compound (two electrons mechanism). (ii) Trapping of O_2 and/or $O_2^{\bullet-}$: an alternative mechanism is the trapping of the α-hydroxy radical by oxygen forming the peroxy radical or by superoxide ($O_2^{\bullet-}$) forming peroxy anion, decomposition of which would be expected to occur rapidly to produce the carbonyl products [16] (Scheme 1).

Also, we infer that the mechanism involved in photocatalytic oxidation of **2** and **3** (Scheme 2) is parallel to that proposed in the PCO of **1** (Scheme 1).

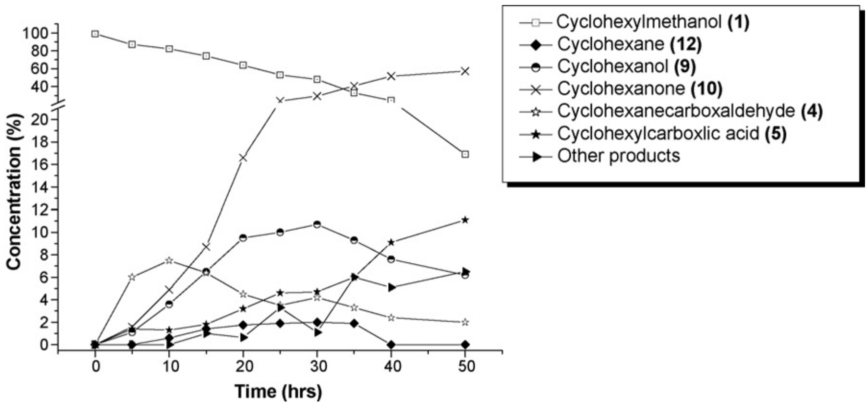


Fig. 1. Kinetic studies of PCO of cyclohexylmethanol (**1**) in acetonitrile.

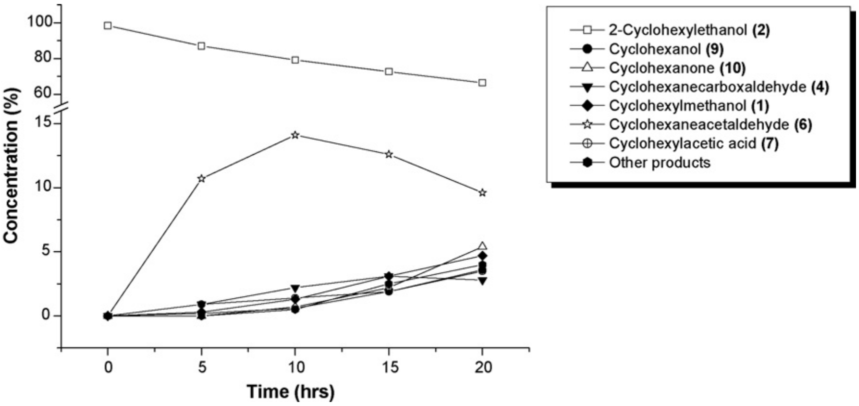


Fig. 2. Kinetic studies of PCO of 2-cyclohexylethanol (**2**) in acetonitrile.

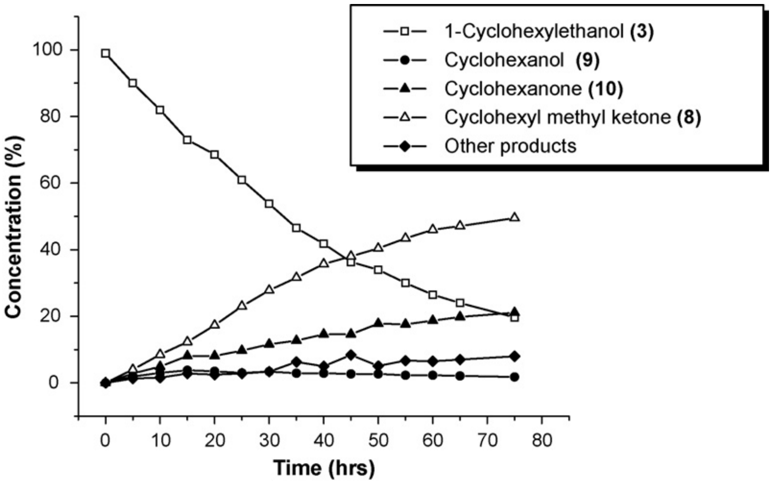


Fig. 3. Kinetic studies of PCO of 1-cyclohexylethanol (**3**) in acetonitrile.

4. Kinetic studies

The kinetics of photocatalytic oxidation of cyclohexylmethanol (**1**), 2-cyclohexylethanol (**2**) and 1-cyclohexylethanol (**3**) in acetonitrile were followed up by GC analyses (Figs. 1–3, respectively). As shown in Fig. 1, by an increase of irradiation time the concentration of cyclohexylmethanol (**1**) decreases. Concentration of the first oxidation product cyclohexanecarboxaldehyde (**4**) increased during the first 10 h and afterwards decreased, due to further oxidation to cyclohexanecarboxylic acid (**5**), cyclohexane (**12**) [33], cyclohexanol (**9**), cyclohexanone (**10**) [34,35]. Similarly, the kinetic study of PCO of 2-cyclohexylethanol (**2**), Fig. 2, as with cyclohexylmethanol (**1**) indicated that by increase of irradiation time the concentration of 2-cyclohexylethanol (**2**) decreased along with an increase in the concentration of the first oxidation product cyclohexaneacetaldehyde (**6**) during the first 10 h and afterwards decreased, due to further oxidation to cyclohexylacetic acid (**7**), methyl cyclohexane [33], cyclohexylmethanol (**1**), cyclohexanecarboxaldehyde (**4**)

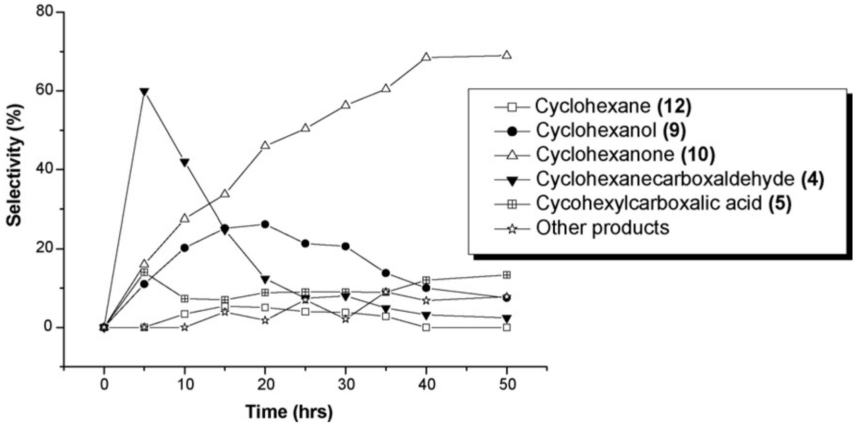


Fig. 4. Selectivity of PCO products of **1** in acetonitrile.

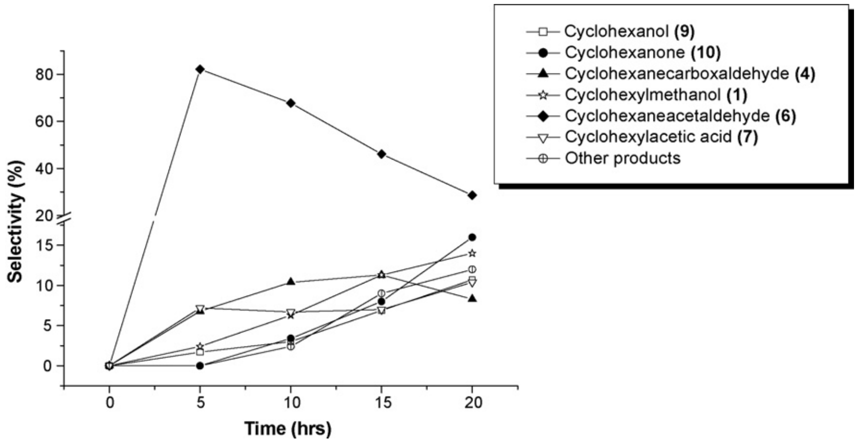


Fig. 5. Selectivity of PCO products of **2** in acetonitrile.

cyclohexanecarboxylic acid (**5**), cyclohexane (**12**) [33], cyclohexanol (**9**) and cyclohexanone (**10**) [34,35]. Unlikely, Fig. 3, showed that the concentration of the first PCO products cyclohexyl methyl ketone (**8**), progressively increased with time, because further oxidation of ketones, oxidative cleavage is much more difficult than aldehydes.

The kinetics of disappearance of the investigated alcohols (**1**–**3**) from an initial concentration in acetonitrile are shown in Figs. 1–3 [43].

Selectivities of photocatalytic oxidation (Eq. (4)) of the target cyclohexyl alcohols (**1**–**3**) are shown in Figs. 4–6, respectively. The selectivity of products are defined as follows (Eq. (4)) [19]:

$$\text{Selectivity of product } P\,(\%) = \frac{\%\,\text{formation of the product } P}{\%\,\text{of total conversion}} \times 100 \qquad (4)$$

It appears that the selective oxidation of **1** to cyclohexanecarboxaldehyde (**4**) is 60% after 5 h and then decreased due to the further oxidation to cyclohexanecarboxylic acid (**5**). Also, the selectivity of PCO of 2-cyclohexylethanol (**2**) is maximum (82%) after 5 h. On the contrary of **1** and **2** the selective oxidation of 1-cyclohexylethanol (**3**) to cyclohexyl methyl ketone (**8**), is 40% after 5 h although progressively increased with time, as shown in Fig. 6. This observed high regioselectivity of PCO of **2** compound with **1** (80%:60%) can be rationalized as a reflective contribution from preferential adsorption effects. In **2**, the bulky cyclohexyl group is away from the α-carbon atom to the hydroxyl group (Schemes 1 and 2). Similarly, PCO of **3** is regioselectivity controlled as it has a value (40%) lower than in **1** and **2** [16].

The semilogarithmic plots of the concentration data gave straight lines for cyclohexylmethanol (**1**), 2-cyclohexylethanol (**2**), 1-cyclohexylethanol (**3**) in acetonitrile as shown in Fig. 7. This indicates that the photocatalytic oxidation of the investigated alcohols in TiO_2 suspension can be described by the first-order kinetic model; $\ln C = -kt + \ln C_0$, where C_0 is the initial concentration and C is the concentration of alcohols at time t. This is in agreement with the results of Cinar et al. [43] on the PCO of *m*-cresol. The rate constants (k) of the PCO of **1**–**3** under the applied experimental conditions, were calculated and found to be $(22.3 \pm 3.7) \times 10^{-3}$, $(21.8 \pm 2.1) \times 10^{-3}$ and $(19.3 \pm 1) \times 10^{-3}\ h^{-1}$, respectively.

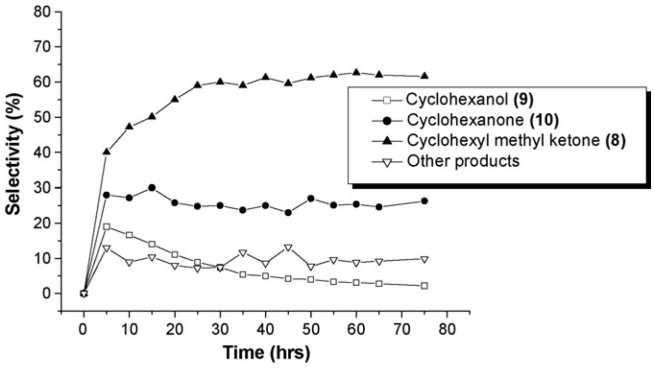


Fig. 6. Selectivity of PCO products of **3** in acetonitrile.

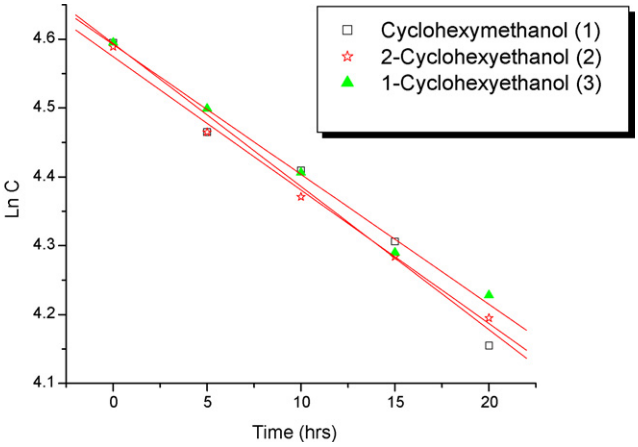


Fig. 7. First-order PCO of **1**–**3** in acetonitrile.

5. Conclusions

Photocatalytic oxidation of three cyclohexyl alcohols (**1**–**3**) using TiO_2 (P25) catalyst, 450 W medium pressure Hg lamp in aerated acetonitrile has been carried out for selective oxidation to the corresponding carbonyl compounds. The PCO mechanism of the investigated alcohols (**1**–**3**) has been proposed. PCO kinetics was rationalized on the bases of regioselectivity in **1**–**3**. Also, PCO of **1**–**3** was found to follow a first-order mechanism and rate constants were calculated. In addition, PCO of alcohols using the cheap, robust, non-toxic TiO_2 catalyst and oxygen could be a green alternative for synthesis of carbonyl compounds.

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