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Degradation of Acetic Acid to decrease COD by Photocatalytic Process in Wastewater: TiO_2 catalyst with UV Process

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ABSTRACT

This study was conducted to decrease the concentration of acetic acid in wastewater of acetic acid plants through the photocatalytic oxidation method. This process employed commercial Titanium dioxide powder (TiO_2) as a photocatalyst, utilizing a UV lamp as the light source within a batch reactor system for advanced oxidation. Various experimental parameters were modified, including the concentration of acetic acid, the amount of catalyst, the volume of waste, temperature, and reaction time. The residual acid concentration and COD values were recorded as results of the process. The percentage of acetic acid remaining in the solution was determined by using a gas chromatography (G.C) device. Experiments were conducted with different volumes, from 200 ml to 35 ml, and utilized varying amounts of photocatalyst: 0.01 g, 0.005 g, 0.0025 g, and 0.001 g. Additionally, the experiments were carried out over two-time intervals of 2 hours and 5 hours. The wastewater concentration contained 3% by mass of acetic acid, and the average COD value was 13300. After experiments, it was found that the optimal conditions for removing acetic acid were a volume of 35 ml and 0.0025 g of catalyst used for 2 hours. In this condition, the percentage of acetic acid decreased from 3% to 0.2%, which is a 93% decrease, and the COD decreased from 13,300 to 2,800, which is a 79% decrease.

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1. Introduction

The development of industries worldwide has increased wastewater and toxic organic pollutants, leading to significant environmental issues and health hazards. Therefore, the treatment of polluted water has become a major challenge. Environmental pollutants directly impact human health, and hazardous and toxic wastewater pollutants have caused severe environmental problems.

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Additionally, much wastewater, including acetic acid, has been exhausted from various material plants, such as cellulose acetate, isophthalic acid, dimethyl terephthalate, terephthalic acid, and esters [1].

Three main processes are used to control the release of pollutants into the environment: biological, physical, and chemical treatment technologies. Among them, chemical treatment technology, particularly the photocatalytic oxidation reaction, stands out as the most practical chemical process for solving environmental problems. This is because it is capable of decomposing a wide range of dangerous organic and inorganic pollutants [2]. In photocatalytic reactions, the degradation reaction is influenced by two important elements - the light source and photocatalysts. Among all semiconductor materials, titanium dioxide is considered the most promising photocatalytic material that is currently suitable for industrial and commercial applications. Titanium dioxide nanoparticles have been prevalent in the past few decades due to their exceptional advantages such as high photocatalytic activity, self-cleaning, antibacterial properties, physical and chemical stability, non-toxicity, and longer lifespan [3]. The availability and affordability of TiO_2 photocatalyst have made it a popular choice for water and wastewater treatment. This powerful technology employs photocatalytic decomposition to break down organic compounds, with the resulting products being CO_2, H_2O, and mineral salts. It is an effective method for eliminating organic pollutants [4].

Photocatalytic degradation, a chemical treatment technology that utilizes visible-ultraviolet light and oxygen, has caught the attention of researchers for its effectiveness in cleaning hazardous pollutants from water. Compared to conventional purification techniques, photocatalytic degradation offers several advantages, including the ability to degrade a wider range of organic pollutants, lower cost, and milder operating conditions [5]. Photocatalytic degradation of various organic pollutants including alcohols, phenols, carbonyls, carboxylic compounds, aromatics, and halocarbons has been widely studied using TiO_2 [6].

The TiO_2 photocatalytic oxidation reaction works in three main steps: (a) light absorption, (b) creation and separation of pairs of electrons (e-) and holes (h+), and (c) oxidation-reduction reaction on the TiO_2 surface. When TiO_2 is exposed to light, e- and h+ are produced in the conduction band (CB) and valence band (VB) of TiO_2, respectively [7]. When excited electrons (e-) interact with oxygen molecules (O2) on the TiO_2 surface, they produce superoxide radicals (O-2). Similarly, positive holes (h+) can react with water molecules (H2O) to produce hydroxyl radicals ($OH\cdot$). These radicals, O-2 and $OH\cdot$, are strong oxidizers in the photocatalytic oxidation process. They can effectively break down pollutants in the environment [8].

Kraeutler and Bard conducted the first research on the photo-degradation of carboxylic acetic acid using Pt/TiO_2 as a photocatalyst in 1970. This process, known as the "photo-Kolbe reaction", results in the production of CH_4 and CO_2 as the primary products. This method shows potential for removing toxic organic acid pollution through an environmentally friendly process, while also yielding several valuable products. [9].

Falconer and Muggli conducted a study on the breakdown of adsorbed acetic acid. They discovered that this process occurs through two parallel pathways. One pathway does not require oxygen and results in the production of methane and CO_2. The other pathway involves oxygen in the TiO_2 lattice and leads to the formation of ethane, CO_2, and H_2O [10].

It has been reported that TiO_2 can remove various carboxylic acids from water, including aliphatic, aromatic, polycarboxylic, and chlorobenzoic acids, as well as salicylic acid [11] [12] [13] In addition, titanium dioxide has been studied for the photocatalytic degradation of higher aliphatic carboxylic acids, such as branched C_4 and C_5 acids [14].

~~The research focuses on~~ In 2013 by utilizing of semiconductor materials as photocatalysts to remove organic and inorganic pollutants in both aqueous and gaseous phases, a study was done. Among the photocatalysts, titanium dioxide (TiO_2) is the cheapest and most effective option due to its resistance to corrosion, stability, non-toxicity, and high oxidation power [15]. Most catalytic reactions occur under environmental conditions. In many cases, the photocatalyzed reaction using titanium dioxide (TiO_2) can completely oxidize pollutants to CO_2. This method is affordable and remains stable even when in contact with various compounds [16]. Conductor photocatalytic oxidation is the best method for removing organic pollutants from wastewater in an environmentally friendly way [17]. Due to its high chemical stability and low degradability, acetic acid is a very resistant compound that is difficult to decompose using conventional methods. However, photocatalytic degradation is an attractive method for breaking down persistent pollutants such as acetic acid. This method is relatively low-cost and can be performed under mild operating conditions [18]. Furthermore, the photodecomposition of acetic acid produces mainly gaseous products such as CH_4, CO_2, and minor concentrations of H_2 [19]. In this way, converting water pollutants into synthesis gas and/or fuel is beneficial from both ecological and economic perspectives.

TiO_2 is a semiconductor photocatalyst that shows great promise due to its stability in aqueous environments, tolerance to acidic and alkaline media, ease of recycling, and ability to effectively purify air and water. However, the efficiency of the photocatalytic system is limited by the rapid recombination of e-/h+ pairs, which reduces its overall effectiveness [20]. TiO_2 has a relatively wide band gap, with 0.3 eV for the rutile phase and 2.3 eV for the anatase phase, which falls in the UV region. Consequently, only 5% of sunlight photons can activate TiO_2 [21]. In this study, the effect of UV light on the degradation of acetic acid as wastewater of petrochemical plants that produce acetic acid has been detected. Moreover, the effect of titanium oxide as a photocatalyst in the presence of UV light has been demonstrated. Finally, the effect of contact time in catalytic solution on the parameter of COD has been shown.

2. Experimental section

2.1. Method and materials

The photocatalytic activity of the photocatalyst was evaluated by measuring its ability to degrade acetic acid (AA) under atmospheric conditions. The experiment was conducted in a reactor system with a UV lamp as the light source and titanium dioxide (TiO_2) as the photocatalyst. The experiment utilized a Hach Company UVP shortwave pen UV lamp to activate a photocatalyst suspension. The mixture was continuously stirred with a magnetic stirrer during the experiment. The photoreactor is a sealed container that is illuminated by a UV lamp through a Pyrex glass. To maintain a constant temperature, this container is surrounded by a water jacket. The absorption reaction was initially conducted in the absence of light, with continuous stirring at room temperature for one hour until adsorption equilibrium was achieved. After that, the light was turned on. To investigate the effect of parameters on the ~~production~~ degradation of acetic acid, solution volume, catalyst amount, reaction time, UV light radiation intensity, and

temperature were measured. The intensity of the lump was fixed. On the other hand, due to the various density of catalytic particles in solutions, There were different intensities when light contact to acid molecules.

The process of photolysis reaction of AC was conducted for a duration of 5 hours without any catalyst. This was done to demonstrate the photocatalytic performance of the newly synthesized catalyst. The remaining concentration of acetic acid in the solution was measured using a gas chromatograph.

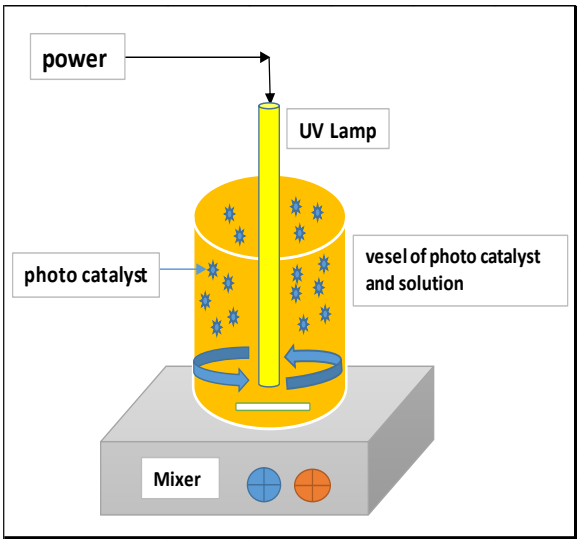

Fig. 1. Schematic diagram of photocatalytic system.

2.2. Photocatalyst and pollutant

The commercial TiO_2 P-25 from Merck Company used as the photocatalyst in this research has an 80:20 anatase to rutile phase ratio, with an average particle size of 21 nm and a bulk density of 850 kg/m3. The concentration of acetic acid in the sample was 3wt% with 4.8 of PH. Additionally, the COD of the sample was measured and found to be 13300 ppm.

2.3. The Effect of reaction time and volume

The data were carried out in a 200 ml constant volume with the presence of UV light to study the impact of UV radiation in the absence of a catalyst. In addition, the results of experiments was determined for 6 hours and was analyzed at 3, 5, and 6-hour intervals The results obtained have been illustrated in Figure 2. A.

Based on the results, it is evident that the percentage of remaining acid in the solution decreases with time. This indicates that increasing the duration of exposure to the light emitted by the lamp has resulted in a greater amount of acetic acid being decomposed [22].

When a mixture is irradiated by UV wavelength, OH radicals are formed from the collision of electrons and holes (h+) [22].

Water molecules (Equations 1 and 2) are directly involved in the decomposition of organic compounds [23].

Passing time accelerates acetic acid molecule degradation due to increased radical enhancement and collision probability [23].

$$h^+ + H_2O \longrightarrow H^+ + \cdot OH \quad (1)$$

$$h^+ + -OH \rightarrow \cdot OH \quad (2)$$

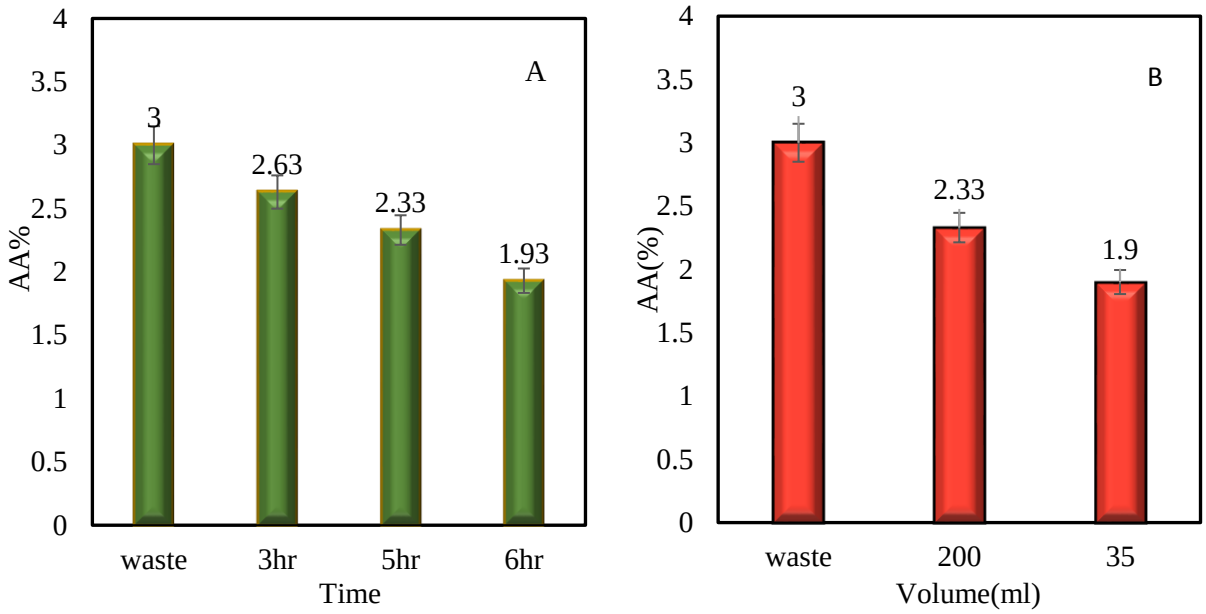

Fig. 2. A Change in the percentage of residual acid in a fixed volume of 200 ml, without catalyst, with UV light, 1. B Change in acid percentage with the volume change, time constant 5 hours, without catalyst, with UV lamp.

To study the effect of UV lamp intensity on AA decomposition, experiments were conducted at two different volumes - 200ml and 35ml. The power of the UV lamp was analyzed for the decomposition of acetic acid, in a reaction time of 5 hours, without using any catalyst. The results are presented in Figure 2. B, which shows the percentage of acetic acid remaining after irradiation. The bar chart depicts the relationship between the acetic acid volume and its degradation rate. It can be concluded that a larger amount of acid was decomposed in a smaller volume where more photons were absorbed in the same irradiation time. The concentration of acetic acid gradually decreased under direct UV light without a catalyst present. When the solution volume was 200 ml, the degradation amount remained below 35 ml due to the effective UV energy that dissociated acetic acid molecules [24].

2.4 Influence of catalyst dosage

It is significant to conduct experiments to determine the optimal amount of catalyst as the photo reactor geometry, intensity of source radiation, and optical path in the suspension all play a crucial role in determining the amount needed [25]. Increasing the amount of the catalyst increases the rate and efficiency of photocatalytic carboxylic acid degradation due to its larger surface area for adsorption [26]. The optimal amount of catalyst necessary for photocatalytic reactors depends on the initial concentration and type of pollutant, as well as the operating conditions of the system. Therefore, the optimal amount varies based on the specific photocatalytic system in use, as well as the type and initial concentration of the carboxylic acid being treated [27]. Figure 3 reports the impact of the Titanium dosage on the disappearance of acetic acid. Solutions with 3 wt% acetic acid concentration were used to examine the effect of catalyst loading by varying the amount of TiO_2 from 0.01 to 0.001g. The degradation of acetic acid increased as the catalyst amount decreased, and the highest degradation of acetic acid (53% after 5 hours) was achieved at a catalyst dose of 0.0025. However, acetic acid's

decomposition rate decreased when the catalyst's dosage exceeded the optimal level. This could be due to the increase in the solution's opacity, which reduced the intensity of radiation transmission and the absorption of the pollutant on the catalyst surface for decomposition [27]. Based on the results depicted in Figure 2B, a volume of 35 ml was chosen to investigate the impact of catalysts on the reaction. To determine the optimal quantity of catalyst, experiments were performed using this volume, with the UV lamp set to a fixed time of 5 hours. The findings of these experiments are presented in Figure 3A.

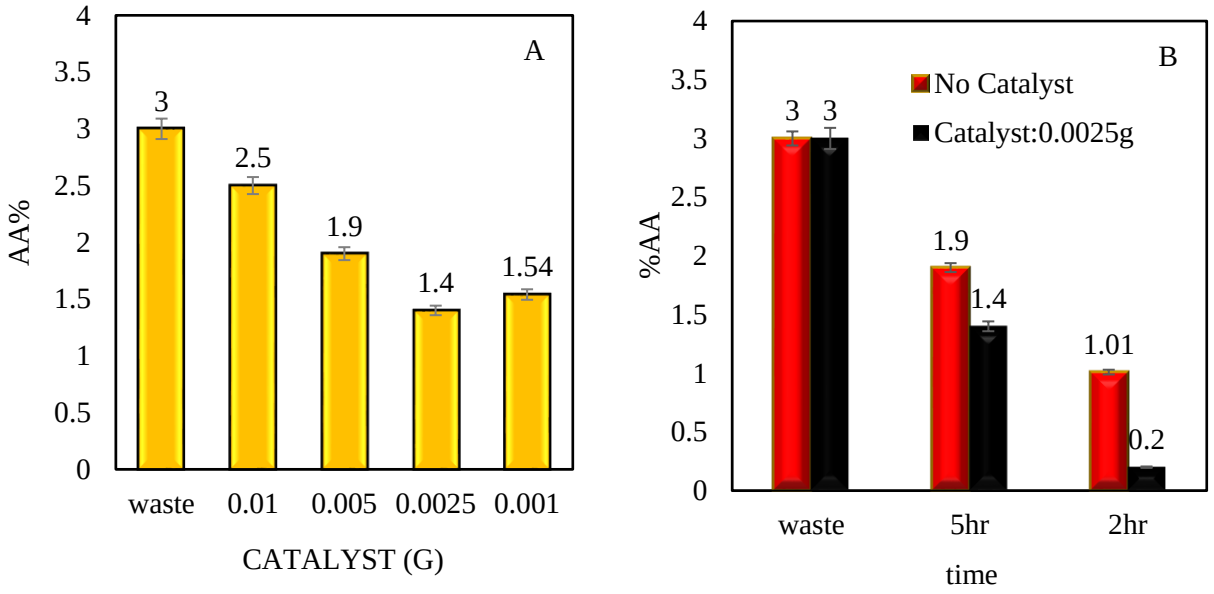

Fig. 3. (A). Changes in acid percentage with different amounts of catalyst in a fixed volume of 35 ml and a time of 5 hours. (B). The effect of the resident time on acetic acid degradation 0.0025g and a *fixed volume of 35 ml.*

Based on the results and the graph, it is evident that reducing the amount of catalyst in a volume of 35 ml has an impact on the percentage of remaining acid. Initially, the amount of acid decreased from 0.001 to 0.0025 grams of catalyst, but it then increased upon further reduction of the catalyst amount to 0.001. Therefore, the lowest amount of residual acid was obtained with 0.0025 grams of catalyst in a volume of 35 ml and a time of 5 hours.

The catalyst surface becomes more porous and absorbs more light, increasing the number of catalytic particles [28]. An increase in catalyst dose from 0.01g to 0.0025g resulted in optimal degradation due to the balanced catalyst and UV light ratio. The presence of TiO_2 nanoparticles in the reaction solution can significantly enhance photocatalytic degradation of acetic acid molecules, especially when the nanoparticles are well dispersed in the solution [29]. Enhancing the percentage of acetic acid by decreasing the catalyst from 0.0025 to 0.001.

As the amount of catalyst increases, the obstruction of light in the path of photons that reach the surface of the catalyst decreases. This leads to an increase in the amount of light that penetrates the acetic acid solution, which in turn results in a decrease in the decomposition of acetic acid. As a result, the formation of hydroxyl radicals and active superoxide decreases simultaneously. Therefore, there is an optimal value for the catalyst in which the maximum degradation efficiency is obtained [30].

Figure 3B demonstrates how the degradation of acetic acid increases significantly when the residence time decreases from 5 hours to 2 hours, while constant power and a fixed amount of catalyst (0.0025 g) are present.

The bar charts show the effect of catalysts on wastewater at two different times. After 5 hours of the UV process, the sample of catalytic and non-catalytic wastewater was degraded. The catalytic substrate degraded from 3% to 1.4%, whereas the non-catalytic substrate degraded from 3% to 1.9%. The reason for this difference is that time has a positive effect on the degradation of acetic acid [22]. A photocatalytic reaction is a process in which the highly oxidizing OH radical decomposes organic compounds absorbed onto a semiconductor's surface. Electrons and positive holes produced by light energy have energy larger than the band gap. The rate-determining step for a catalytic reaction may be delayed if the absorption rate of the reactant or desorption rate of the product is slow. In the case of TiO_2 and UV reactions, the rate-determining step is the absorption of reactants to the TiO_2 surface [31] .

Upon repeating the test after 2 hours, the percentage of catalytic and non-catalytic substrates sharply decreased to 0.2% and 1.01%, respectively. The addition of a catalyst decreased the percentage of residual acid in both instances. It can be seen from the graph that reducing the reaction time from 5 to 2 hours significantly decreased the residual acid percentage in the solution with the catalyst. According to the gas chromatography results, it can be inferred that at the beginning of the reaction, acetic acid transformed into various acidic compounds, including formic acid, propionic acid, methyl acetate, and additional unidentified acidic compounds that were not detected by gas chromatograph analysis. The table displays the results of gas cromatography results conducted under different conditions, reported as mass percentages. The liquid phase during the photocatalytic degradation of acetic acid produced methanol (CH_3OH), formic acid (CH_2O_2), and methyl acetate (CH_3COOCH_3). Some organic intermediates, such as methanol, can decompose into CO_2 and H_2 [32].

2.5 The effect of time on COD

The significant COD parameter for hydrocarbon degradation is the same as that analyzed for acetic acid. The experiments were conducted using constant volume, catalyst, and reaction time. The results are presented in Figure 4. The bar chart illustrates that optimizing conditions reduces COD levels. The suitable volume for COD reduction is 35 ml with a catalyst weight of 0.0025 g and a residence time of 2-5 hours (Figure 4). As depicted in Figure 4, the COD level reduced significantly from 13300 to 6420 after 5 hours of adding 0.0025 g of TiO_2 catalyst. This reduction occurred due to the degradation of hydrocarbons and acetic acid. Moreover, diminishing the resident time to 2 hours resulted in a further decrease in COD level, reaching 2800. The reason for that is the intermediate products may be formed during the degradation process. When the resident time is 5 hours, COD and acetic acid concentration are completely photodegraded. The acetic acid disappeared under the UV/TiO_2 reaction [33]. In a reaction system, catalytic holes are the initiators of the reaction, which can be written as follows:

$$CH_3COOH + h^+ \rightarrow CH_3 \cdot + CO_2 + H^+ \quad (3)$$

During the decomposition of acetic acid to carbon dioxide, intermediates such as acetate, methoxy, formaldehyde, and formate are formed. (reactions 4-6)

$$CH_3COOH \longrightarrow CH_3COO \quad (4)$$

$$CH_3COO \rightarrow CH_3O + CO_2 \quad (5)$$

$$CH_3O \rightarrow CH_2O \rightarrow HCOO \rightarrow CO_2 \quad (6)$$

When formaldehyde, methoxy, and formate intermediates (reaction 5) are formed, they

stick to the catalyst's surface, preventing further absorption of acetic acid and slowing down the rate of carbon dioxide formation. At high concentrations, the formaldehyde acid is absorbed preferentially on TiO_2 because of the basic surface sites. The concentration of formaldehyde increases as the acid reacts with the holes, and when the acid concentration decreases, formaldehyde is absorbed more [34]. Based on Equations 3 to 6, it can be concluded that in the presence of hydroxyl radicals, multiple reactions may occur in the system being studied. These reactions include the mineralization of acetic acid or the formation of organic intermediates in the liquid phase, which can further decompose. As a result of these reactions, the main product is likely to be CO_2 [35]. Therefore, when the resident time is extended due to the presence of more CO_2 and byproducts hydrocarbons (as seen in equations 3-6), the COD parameter is higher compared to situations where the contact time is low.

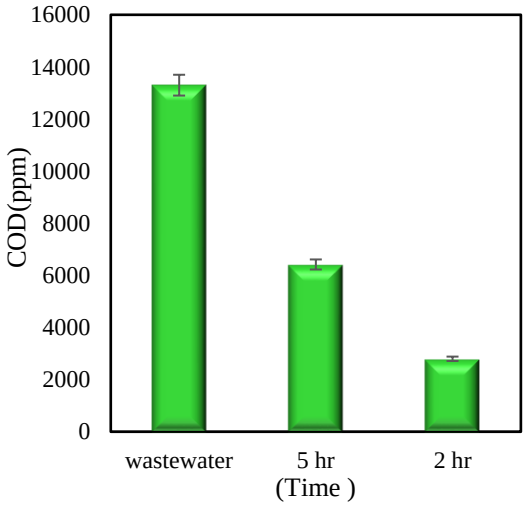

Fig. 4. Effect of Time on COD.

3. Conclusion

In this study, the photocatalytic decomposition of acetic acid in wastewater of a Petrochemical Company under UV irradiation was investigated. The results showed that the maximum amount of acid decomposition occurred in a volume of 35 ml with a catalyst amount of 0.0025 grams, using a constant UV light source. In addition, the effect of UV light alone and with the catalyst was studied. The addition of the catalyst led to better reaction progress. The turbidity, amount of catalyst, and power of the UV source relative to the volume of the effluent are important factors in the decomposition of acetic acid. Reducing the reaction time from 5 hours to 2 hours results in the decomposition of acetic acid into other acidic compounds like formic acid and methyl acetate. The residence time in the reactor is another important parameter, and when other oxidizing ions like oxygen or air are present, the produced products are decomposed into lighter compounds. In optimal conditions, the percentage of acid decreased by 93%, from 3% to 0.2%, and COD decreased from 13300 to 2800, a reduction of 79%.

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