

original article

Photocatalytic removal of gaseous toluene by titanium dioxide coated on nickel foam: Influence of relative humidity and toluene concentration

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ABSTRACT

Aims: The aim of this study is to determine the impact of relative humidity (RH) and contaminant concentration on photocatalytic conversion of gaseous toluene by using TiO₂ coated on nickel foam.

Materials and Methods: TiO₂ nanoparticles were prepared by the sol-gel process and coated on nickel foam. Structural and morphological characteristics of nanoparticles were determined using Scanning electron microscope, X-ray diffraction analysis. Photocatalytic conversion of gaseous toluene at the different levels of RH and toluene concentration was measured under ultraviolet-A radiation by gas chromatograph with Flame Ionization Detector.

Results: After being fixed the contaminant concentration, 30% level of RH had the most impact on the photocatalytic efficiency, 10, 0.5 and 80% levels of humidity had, in turn, the most impact. Results also showed that TiO₂ nanoparticles coated on nickel foam at the concentration of 20 ppm had the most efficiency of photocatalytic conversion. After that, the most efficiency was recorded at the 10 and 50 ppm concentrations, respectively.

Conclusion: Based on the results, the photocatalytic conversion of gaseous phase toluene by TiO₂ coated nickel foam is increased with the increase of RH to a certain level; beyond that the conversion efficiency is decreased gradually due to the saturation of photocatalyst surface and decrease in nanoparticle activity. Furthermore, photocatalytic conversion of gaseous toluene is decreased with the increase of toluene concentration.

Key words: Concentration, nickel foam, photocatalyst, relative humidity, TiO₂, toluene

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INTRODUCTION

Volatile organic compounds (VOCs) are the most important air contaminants being found in indoor and outdoor environments. Emission of VOCs, due to the negative impact on air quality, make undesirable effects over human health and the environment.^[1] Among VOCs, toluene, is an obvious pollutant and the largest contributor to air

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pollution in industrial areas and also indoor environments.^[2] Toluene (methylbenzene; Chemical Abstract Service No. 108-88-3) is an aromatic hydrocarbon that is widely used as an industrial feedstock and as a solvent. It is also a hydrocarbon and a solvent that is rapidly absorbed through the respiratory and gastrointestinal tracts and to a lesser extent, through the skin.^[3] Exposure to toluene affects the central nervous system which causes to decrease thought and memory ability, muscles power and disturb body balance. Exposure to toluene at the noisy environments makes some levels of hearing loss and color vision ability. Toluene is excreted in the exhaled air in its unchanged form and in the urine as the metabolite hippuric acid.^[4,5] Hence, it is necessary to use suitable methods in order to eliminate or reduce toluene concentration in the air. The applied technologies to eliminate it include thermal and catalytic oxidation processes.^[6] Photocatalytic oxidation is used, in the presence of light (visible/ultraviolet [UV]), as a developed technology to eliminate VOCs at the enclosed air condition especially when concentration is at the low level.^[7] Among semiconductors, titanium dioxide (TiO_2) is the most effective photocatalyst that is used widely because of chemical and physical stability, high light efficiency, low cost, being inert and nontoxic and corrosion resistance.^[8]

TiO_2 as a photocatalyst is used to produce electron (e^-) and holes (h^+) and subsequently, to provide Redox (reduction and oxidation) reactions. Electrons and holes can be stimulated through UV radiation which leads to production of radical OH^- and O_2^- ions and convert the desirable contaminant to the CO_2 and H_2O .^[9] There are some problems in the optimized use of photocatalytic oxidation technology. Some of these problems consist of being photocatalytic oxidation amount less than expected levels, environmental toxicity and reaction of intermediate materials and catalyst deactivation.^[10] Operational variables like environmental relative humidity (RH) and ratio of contaminant concentration play important roles in the photocatalytic oxidation process of the VOCs. RH has significant effects on efficiency of photocatalytic oxidation so that many studies have investigated this subject. These studies reported the effect of RH as an inhibiting or promoting factor.^[11-16] A study by Yang *et al.* found that RH has not significant effect on the photocatalytic oxidation of gaseous formaldehyde, because formaldehyde does not need to free radicals of OH. They also showed that there is not competitive adsorption in the low concentrations between formaldehyde and water steam.^[17] Zhang *et al.* concluded in a study that, in photocatalytic conversion of VOCs, photocatalytic, reactions are formed between Photocatalyst and absorbed water film on the Photocatalyst surface. As a result, VOCs, first, should be absorbed on water film and then penetrate into catalyst surface.^[18] The effect of contaminant concentration on photocatalytic efficiency of TiO_2 was another subject studied by researchers. In some cases, it has been proved that the increase in pollutant concentration in

operational condition increased photocatalytic conversion by sample containing TiO_2 ^[19,20] on the other hand, it has been found that the increase in pollutant concentration lead to a decrease in the photocatalytic activity of TiO_2 .^[21]

In this study, the TiO_2 nanoparticles was produced by the sol-gel process and then coated on the nickel foam. In addition to special physical, chemical and thermal characteristics, the porous metal foam of nickel provide a wide surface to pollutant encounter with Photocatalyst. In order to study the photocatalytic activity in the operational condition under study, toluene was used as model pollutant from VOCs and all reactions were performed under UV-A radiation. In this study, the efficiency of toluene photocatalytic conversion was investigated at various RH levels and pollutant concentration in a batch reactor with controlled environmental condition.

MATERIALS AND METHODS

Materials

The prerequisite solution to prepare TiO_2 was provided by the sol-gel process. For this, 17.59 ml titanium isopropoxide ($\text{Ti}(\text{O}i\text{Pr})_4$) and 7.95 ml three ethanolamine were solved in 70.74 ml dry ethanol. After 1 h-vigorous stirring of the solution at room temperature, the solution obtained from dry ethanol and deionized water was added to the stirred solution in the second stage.^[22] Obtained solution was stirred for 2 h at room temperature and then was left in the dark place for 24 h. TiO_2 nanoparticles were obtained from calcination of this sol in the anatase and rutile phases. Nickel foam with following characteristics was used as substrate (0.8 cm thickness, surface area = 35.3 cm^2 2 mm cells diameter, porosity = 70% and pore per inch = 30, by Nano poshesh felez Co., Iran). In order to purify nickel foam from possible contamination, it was placed for 15 min in ultrasonic device. Then it was exposed to 550°C for 30 min. Afterward, the nickel foam was overwhelmed inside sol. It was then dried at room temperature in order to calcination, furnace temperature raised up to 550°C in according to a thermal planning during 8 h and 34 min. Sample on nickel foam substrate calcinated in this temperature for 45 min. In order to prevent from cracking of TiO_2 film and making a homogenous catalyst surface on the substrate of nickel foam, the furnace temperature reached to room temperature (25°C) gradually.

Characterization

In order to identification of phases in the samples, X-ray diffraction (XRD) (Bruker, D8 ADVANCE, Germany), wavelength: 1.5406 \AA (Cu K_α), voltage: 40 kV, current: 40 mA) with Cu K_α radiation were used. After preparing the gold and doing its deposition, microstructure and morphology of TiO_2 coated on foam nickel, was studied using a Scanning electron microscope (SEM, XL 30, Philips).

Procedure and analysis

In order to test photocatalytic efficiency of TiO₂ nanoparticles, a static reactor made of stainless steel with volume of 7500 cm³ was used. UV-A light was emitted inside the reactor through a quartz window with an area of 120 cm². Figure 1 shows the schematic view of reactor designed for experiment. TiO₂ sample coated on nickel foam was placed inside the reactor in 6 cm distance from UV-A lamp, with 15 W power. The intensity of light received from UV-A lamp in sample surface was 11.6 W/m². Air containing toluene concentration was circulated inside the reactor by a fan with speed of 0.07 fpm. In the pretest stages, the optimized time to reach final efficiency was removed and toluene concentration became constant in the reactor chamber. The duration of this process was determined 270 min. In order to study the effects of operational variables (RH and toluene concentration) on photocatalytic efficiency of TiO₂, RH was studied in the 5 levels; i.e., 0, 10, 30, 50 and 80% ($\pm 3\%$) at room temperature. Threshold limit for occupational exposure to toluene in the air is 20 ppm for 8 h of daily working.^[23] To determine the effect of toluene concentration, three levels of gaseous phase toluene including 1/2 (10 ppmv), 1 (20 ppmv) and more than 2 (50 ppmv) equal to TWA were studied at room temperature. Toluene concentration in the gaseous phase was measured using a gas chromatograph with flame ionization detector (GC-FID, VARIAN 3800, USA). After turning on the UV-A source, once every 20 min, 100 ml of air into the reactor was injected to the GC-FID. The peak area represents the concentration of toluene. Temperature of injector, column and detector were 200, 75 and 250°C respectively (NIOSH Method 2549). To study the effect of humidity level and Toluene concentration level, nitrogen was used as carrier gas. After determining the concentration, the toluene removal efficiency percent was calculated by the following equation:

$$\%R = \frac{C_0 - C_1}{C_0} \times 100 \quad (1)$$

where %R, C₀ and C₁ are the percentage of photocatalytic conversion, initial concentration and secondary concentration after photocatalytic reaction, respectively.

RESULTS

Morphology and structure of materials

The deposition of TiO₂ nanoparticles on the surface of nickel metal foam was studied carefully by use of different experiments. The Figure 2 shows the XRD pattern for TiO₂ sample coated on nickel foam. This sample has been calcified at 550°C.

Therefore, the crystalline structure of both Anatase and Rutile phases was determined in the ratios of 61.1% and 38.9%, respectively. The particles sizes were, in turn, 18 nm and 24 nm in anatase and rutile phases. Microstructure and morphology of TiO₂ coated on nickel foam after calcification

at 550°C temperature is illustrated in Figure 3. SEM analysis indicates that substrate surface of nickel foam is completely coated by TiO₂ nanoparticles. In spite of existing of some micro cracks in different parts of specimen, the surface of substrate was totally coated by nanoparticles due to the amount of TiO₂ located on foam substrate.

The effect of RH and toluene concentration

The percentage of photocatalytic conversion of toluene by TiO₂ nanoparticles coated on nickel foam was determined by placing

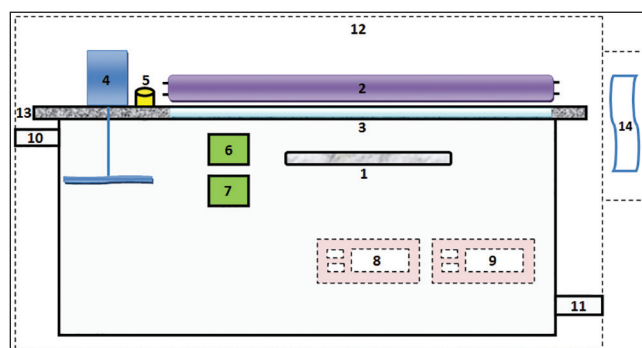


Figure 1: Schematic diagram of photoreactor for toluene photocatalytic degradation. (1) Titanium dioxide coated on Ni foam; (2) ultraviolet-A lamp; (3) quartz glass cover; (4) motor for air circulation; (5) Sampling point; (6) % relative humidity (RH) sensor; (7) temperature sensor; (8) %RH display; (9) temperature display; (10) input for air cleaning; (11) output for air cleaning; (12) general dark room of reactor; (13) Al door of reactor; (14) fan for cooling of reactor space

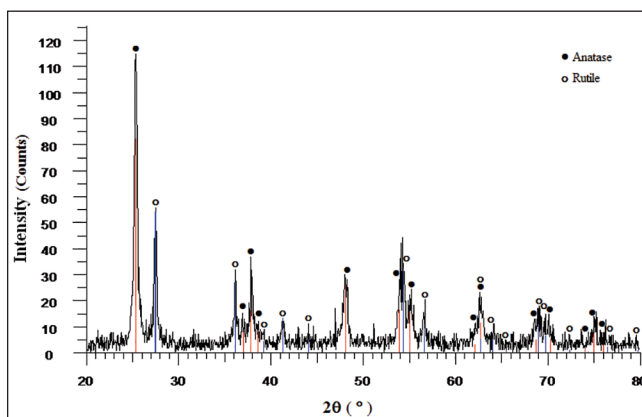


Figure 2: X-ray diffraction patterns of titanium dioxide coated on Ni foam substrate

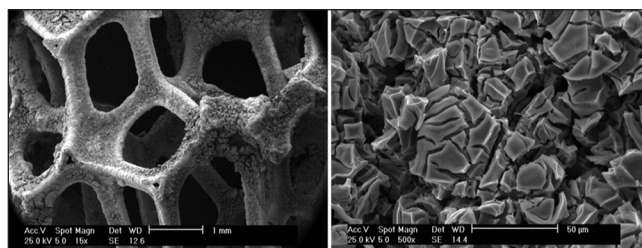


Figure 3: Scanning electron microscope images of titanium dioxide coated on Ni foam

the specimen with contaminant in a static reactor at room temperature. The final concentration of toluene was calculated 20 min after injecting water into the reactor when the expected humidity was constant and also 90 min after injecting toluene into the static reactor without using UV-A light. Table 1 shows the final concentration of toluene when surface adsorption at different levels of humidity and concentration were constant. These concentrations are considered as the basic level required for photocatalytic oxidation. The influence of different levels of RH was investigated in 100 ppmv of toluene. The highest surface adsorption of toluene, in various levels of RH, was at 10% level. The lowest level was also at 50%. The surface adsorptions of toluene at 10, 20 and 50 ppm toluene were 35%, 40% and 54%, respectively.

Figure 4 shows the toluene photocatalytic conversion using nickel foam coated on TiO₂ at a different level of RH. At 150 min, the level of 0% (3% actual) had the highest photocatalytic conversion of toluene. However making some changes continued up to 270 min and then was fixed, photocatalytic conversion of toluene at 270 min were considered. So, the greatest photocatalytic conversion of toluene was 52.5% at 30% RH level. After this, the highest photocatalytic conversions were recorded 51.11%, 36.2%, 28.4% and 22.4% at RH levels of 0%, 10%, 50%, 80% respectively. Figure 5 shows the

Table 1: Actual concentration of toluene in the reactor, after adsorption in different levels of RH and toluene concentration

Levels of %RH	0	10	30	50	80
RH					
Levels of %RH	0	10	30	50	80
Actual %RH	3	13	33	53	79
Actual C (ppmv)	77.7	62.4	73.2	79	77.9
Concentration					
Levels of C (ppmv)	10	20	50		
Actual C (ppmv)	6.5	12	23.3		

Actual %RH: Actual percent of RH in about 60 min after injecting the water into the reactor, Actual C (ppmv): Actual concentration (ppmv) of toluene in about 90 min after injecting the toluene into the reactor), RH: Relative humidity

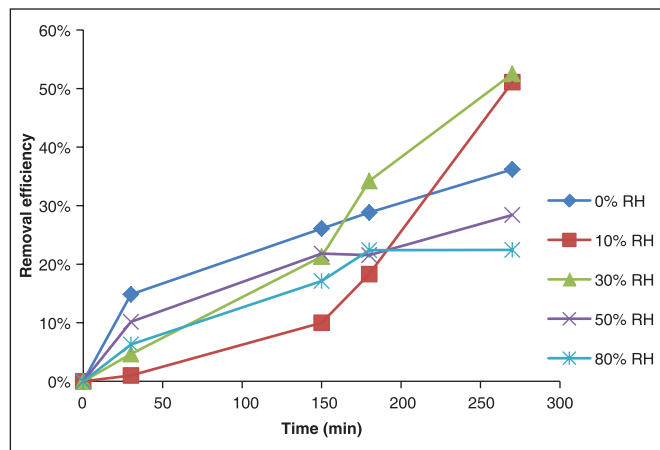


Figure 4: Gaseous toluene degradation rates of titanium dioxide films coated on foam nickel substrate in different levels of relative humidity and 100 ppm toluene

photocatalytic activity of balanced gaseous toluene conversion based on concentration by using coated TiO₂ on nickel foam. These amounts were measured based on room temperature, at 10% humidity and total air flow rate of 0.05 fpm. According to Figure 5, the greatest photocatalytic conversion of toluene obtained 6.8 at 6.5 ppm (initial level of 10 ppm) concentration. After this, the efficiency of toluene photocatalytic conversion was obtained 3.45% and 0.42% at 12 ppm and 23.3 ppm (initial levels of 20 and 50 ppm), respectively. Comparing humidity with concentration level, the differences between efficiency might be related to the blockage of more oxidation sites of photocatalytic by subsidiary productions of toluene conversion reaction.

DISCUSSION

In the present study, photocatalytic conversion of toluene was investigated by means of TiO₂ nanoparticles coated on foam nickel at 5 RH levels, and 3 toluene concentration levels. Using photocatalytic technology, the highest efficiency of toluene photocatalytic conversion, was obtained about 70% during adsorption and photocatalytic conversion. The obtained efficiency was lower than studies of Hu *et al.*^[22,24,25] These researchers reported 70% removal efficiency of acetaldehyde by a TiO₂ film that coated on nickel foam at 550°C. One of the most crucial and effective agents affecting contaminants photocatalytic conversions is the contact time between the contaminants and photocatalytic. While Hu *et al.* achieved 70% removal efficiency of toluene at 360 min, while in the present study the efficiency of 52% was achieved during 270 min after turning the UV-A lamp up. The static reactive applied in this study was 7.5 times in volume greater than the case of Hu *et al.* which normally caused differences in encountering contaminants with photocatalytic specimen. Furthermore, there were some other differences between our study and theirs such as the contaminants type and

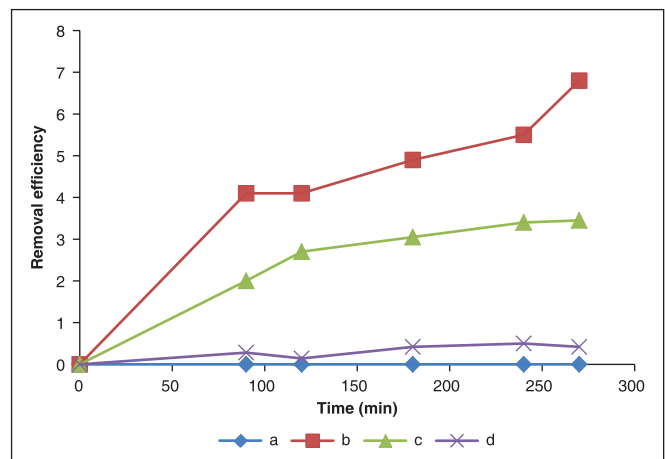


Figure 5: Gaseous toluene degradation rates according to the concentration (a) Ni foam without titanium dioxide (TiO₂) in 10 ppm; Ni foam with TiO₂ in (b) 10 ppm; (c) 20 ppm; and (d) 50 ppm toluene

concentration, UV-A radiation resource etc. These factors could affect the photocatalytic efficiency subsequently. It was revealed that the greatest surface adsorption was 27.6% and 26.8% at 90 min after injecting toluene into the reactor at 10% and 30% humidity levels, respectively. Afterward, the most surface adsorption was 22.3%, 22.1%, and 21% at 0%, 80% and 50% relative humidities, respectively. By increasing the RH of the environment up to 30%, the surface adsorption of toluene decreased. This result is similar to the findings of Zhang *et al.*^[18] They reported the following results about surface adsorption of chlorobenzene on photocatalytic surface: While injecting chlorobenzene after water vapor, its surface adsorption significantly reduced along with increasing RH. The reason of this phenomenon could be due to more saturation of the surface by RH at higher levels of humidity. Evaluating the photocatalytic efficiency of TiO₂ nanoparticles coated on metal nickel foam at different RH demonstrated the highest efficiency of toluene removal at 30% humidity level. Moreover, it was recognized that changes of toluene photocatalytic conversion increased gradually up to 30% RH level, and then decreased as the RH goes up. Hang and Ye studied the photocatalytic conversion of toluene under the humidity levels ranged from 0% to 75% and reported that the highest efficiency of toluene conversion at 75% and 50% and the least at 1%.^[26] However, Dezhai *et al.* and Yang and Ollis reported the inhibiting effect of RH on toluene photocatalytic conversion.^[11,27] Our results indicated that efficiency variation diagram of toluene photocatalytic conversion has a turning point (30% RH level) so that the efficiency changes, in turn, increase and decrease before and after this level. In fact, our survey showed the impact of RH on photocatalytic efficiency of TiO₂ nanoparticles coated on nickel foam, as a promoting and also an inhibiting factor. In photocatalytic procedure, the contaminant is oxidized by OH and O₂ radical ions through UV-A adsorption on TiO₂ particle surfaces. Existence of environmental RH causes to make free hydroxyl radical. Subsequently, it leads to aggravate photocatalytic process. If the RH of the environment raises to high levels, it may lead to the saturation of contaminants surface. It causes less contaminants to be in contact with photocatalytic surface. Subsequently, the contaminants conversion efficiency would decrease. Investigating the influence of toluene concentration on photocatalytic activity showed that the highest percentage of photocatalytic conversion was at 10 ppm while the lowest one at 50 ppm concentration. In this research, the obtained efficiency was balanced based on toluene contaminant concentration. In contrast to Yu and Lee who considered the elevation of contaminant concentration, as the main factor in increasing the efficiency of photocatalytic conversion,^[19] we found opposite results exactly. Our results are as the same as Zhang's findings.^[21] It can be concluded that an increase in contaminant amount would decrease photocatalytic efficiency. Therefore, due to more surface adsorption of toluene on higher concentration levels, greater saturation of photocatalytic surface would be occurred and more oxidation sites of catalyst would be blocked by subsidiary productions

of toluene conversion reaction. Therefore in this condition the efficiency of photocatalytic conversion decreased.

CONCLUSION

In the present study, after preparing TiO₂ nanoparticles by using the sol-gel technique, and coating it on nickel foam, the influence of RH and contaminant concentration was examined on toluene photocatalytic conversion. According to the obtained results, by increasing the RH to a specific level (30%), the efficiency of photocatalytic conversion improved. But in higher RH, it didn't work. It may be because of contaminants surface saturation and declining its activity. This project proved that increasing the contaminant level, had an inhibitor effect on photocatalytic activity. Moreover, enhancing the concentration amount caused to decline toluene photocatalytic conversion. Based on the results of this survey, it is highly recommended that the efficient level of operational variations such as RH and contaminant concentration would put as the main goal to attain. Apparently, it will be done for using the photocatalysts in the laboratory and industrial studies in according to photocatalysts amount. It is also suggested applying photocatalysts sample in the efficient conditions.

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