Original Article

Impact of Titanium Dioxide Particles on Adsorption Properties of HZSM-5 Zeolite for Toluene Treatment

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Abstract

Introduction: Today, a variety of catalysts were used for the removal of the volatile organic compound in work environments. Zeolites are frequently used as catalyst and catalyst base because of their desirable characteristics. HZSM-5 attracts the attention of air pollution researchers in recent years. The aim of this study is to investigate the adsorption properties of HZSM-5/titanium dioxide (TiO_2) catalyst. **Materials and Methods:** A dynamic system was used for producing 42 ppm of toluene in 250C, 5% humidity, and environment pressure. For determination of adsorption capacity, 42 ppm of toluene with the flow of 0.5 L/min passed through the reactor containing 1 g of the HZSM-5/TiO_2 catalyst which was coated within 3%, 5%, and 8% of TiO_2 using impregnation method. For determination of the catalyst characteristics, different analyses of X-ray diffraction, Brunauer Emmett Teller (BET), Fourier transform infrared, and scanning electron microscope were used. **Results:** Adsorption capacity was 22.3, 23.81, 38.06, and 28.88 mg/g for HZSM-5, HZSM-5/Tio_2 3%, HZSM-5/Tio_2 5% and HZSM-5/Tio_2 8%, respectively. The specific surface was 298.8 m^2/g for HZSM-5 and 212.8, 189.3, and 185.1 m^2/g for 3%, 5%, and 8% HZSM-5/TiO_2 catalyst, respectively. Results indicated that the breakthrough time increases by increasing in weight percent of TiO_2. Adsorption isotherm was identified as type I, based on International Union of Pure and Applied Chemistry (IUPAC) classification. **Conclusion:** The integration of TiO_2 particles with HZSM-5 created a photocatalyst with desirable properties that include high adsorption capacity and long breakthrough time to maintain a high amount of toluene vapors, which lead to better removal efficiency.

Keywords: Adsorption properties, HZSM-5, titanium dioxide particles, toluene

Introduction

Volatile organic compound (VOC) emission is a major issue with harmful effects on human health and environment.[1-3] VOCs such as toluene are the main group of air pollutants in the work environment. These compounds also are one of the most important resources of photochemical reactions. VOCs can easily enter to air from different industrial and environmental resources because of their high vapor pressure.[4] VOCs also are the main resource of formation of photochemical smog in the urban area.[5] Among the vast variety of VOC components, toluene is one of the most dangerous ones that are using in a lot of chemical industries.[6] So far, a number of methods have developed for VOC removal such as thermal oxidation,[7] catalyst oxidation,[8] absorption,[9] and nonthermal plasma.[5,10] In the recent decade, adsorption is an effective and practical way for VOC removal and recovery. More than 10% of the industrial abatement units are based on adsorption techniques, and it is growing to meet the strict legislation on VOC control. In the recent decade, adsorption is an effective and practical way for VOC removal and recovery. More than 10% of the industrial

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abatement units are based on adsorption techniques, and it is growing to meet the strict legislation on VOC control.[11]

Today, foundations such as zeolites, activated carbon, or diatomite with high surface area provide more time for photocatalytic reactions of catalyst and ultraviolet (UV) by the absorption of volatile compounds on the surface and their pores and thus increase the removal efficiency.[12] Zeolites are a common and popular candidate for removal gaseous and vaporous pollutants as the catalyst and effective adsorbent because of their high surface area, high adsorption capacity, chemical, thermal stability, controllable acidity, and pore structure.[4,13,14] Zeolites also are aluminosilicate minerals with a cage (framework) structure with three dimensions.[15] HZSM-5 zeolite with Mordenite Framework Inverted (MFI)-type structure is one of the famous zeolites, which is frequently used as a support for many heterogeneous catalysts and photocatalysts for removal of the pollutants such as VOCs.[16] Modifying the molecular sieves by chemical or thermal methods or both can improve features such as specific surface area and the functional groups and thus increase its efficiency in the refining processes.[17] Zeolite combination with another catalyst such as metals and photocatalyst materials showed better pollutant oxidation and improving performance efficiency for VOC treatment.[18] Photocatalytic oxidation by having advantages such as an activity at ambient temperature and low pressure, low cost, and low power consumption in comparison with other methods has been introduced as one of the promising alternatives for treating a wide range of VOCs in the recent years.[19-21]

Titanium dioxide (TiO_2) has become apparent that organic compounds can be oxidized to carbon dioxide (CO_2) by hydroxyl radicals generated on the TiO_2 surfaces.[22] In the photocatalytic process, a catalyst with the proper band gap such as zinc oxide or TiO_2 and light source (usually UV) is used to convert organic compounds into benign and odorless constituents – water vapor (H_2O) and CO_2.[21,23,24] TiO_2 (with three crystalline phases: anatase, rutile, and brookite), a white powder that has a photocatalytic and superhydrophilic, is used in water and wastewater treatment, air pollution, and buildings.[12] Features such as low price, availability, chemical stability, and lack of toxicity make the TiO_2 as the most appropriate photocatalyst.[25] TiO_2 is the most common photocatalyst with high stability, nontoxicity, low cost, high photocatalytic activity under UV source, and chemical inertness.[6,26,27] The literature review indicates that numerous researches carried out studies on the different aspects of heterogeneous catalyst and HZSM-5 zeolite. Radwan *et al*. compared the different preparation methods for HZSM-5/TiO2 photocatalyst.[26] Mesopore structured ZSM-5 zeolitic materials with acidic sites for n-heptane and toluene adsorption and diffusion.[16] Selective Adsorption of silica coated ZSM-5 for p-chloronitrobenzene and o-chloronitrobenzene studied by Guo *et al*.[28] Huang studied the promotional effect of HZSM-5 on the catalytic oxidation of toluene over MnOx/HZSM-5 catalysts.[18] The main goal of this research is to investigate the adsorption properties and capacity of different percentages of TiO_2 heterogeneous photocatalyst for removal toluene vapors from the air stream.

Materials and Methods

Photocatalyst preparation

HZSM-5 zeolite was synthesized in the laboratory and after granulation sieved with 10/20 mesh. The granules were placed in the oven at 100°C for dehumidification, then calcinated for 3 h at 450°C, and finally, were dried for 24 h in the desiccator.[29] Impregnation method was used for photocatalyst preparation. Ammonium titanium oxalate monohydrate with chemical formulation $(NH_2)_2 TiO (C_2O_4)_2.H_2O$ with molecular weight 294 was used for coating TiO_2 on zeolite. First, this salt was dehydrated at 108°C for 2 h. After weighing 3.5% and 8%, 1 g of zeolite was added to TiO_2 solution and then stirred for 1 h at 70°C. Photocatalyst was dried in the oven for 12 h at 120°C. Any additional compound on photocatalyst evaporated after calcination at 500°C for 3 h.[29]

Photocatalyst characterizations

Physical characterization of HZSM-5 zeolite and HZSM-5/TiO2 photocatalyst including structure and morphology was identified by scanning electron microscope (SEM). Crystal structure was identified by X-ray diffraction (XRD) and Brunauer Emmett Teller (BET) test for illustrating the isotherm, specific surface area, pore size, and total volume distribution. To determine the elemental composition of photocatalyst, Energy Dispersive X ray (EDX) spectra were used.

Photocatalyst testing

Toluene (purchased from Merck Company) vapors produced in a dynamic system in 42 ppm and follow rate of 0.5 L/min, then introduced to the reactor containing 1 g of HZSM-5/TiO_2 (3%, 5%, and 8% TiO_2). The co-axial photocatalyst reactor included a stainless steel bar as center of the reactor with 25 cm length and 6 mm external diameter and cylindrical tube of quartz glass with 25 cm length and 1 cm internal diameter. The concentration of toluene in inlet and outlet of reactor, regularly monitored using tiger pho check. The experimental setup is shown in Figure 1.[30]

When the first concentration of vapors was released from the reactor, it was recorded to calculate adsorption capacity and breakthrough point. For determining the adsorption capacity for all four catalysts, the following equation was applied:

$$BC = \frac{C_{in} + T_{bk} + Q}{g}$$

where

BC = Adsorption capacity (mg/g)

C_{in} = Inlet toluene concentration (ppm)

T_{bk} = Breakthrough point

Q = Flow rate (L/min)

g = Weight of adsorbent (g)

Results

X-ray diffraction analysis

The XRD spectra of HZSM-5 showed general formula $H_{49.61}Al_{1.78}O_{216}Si_{94.22}$ and hydrogen tecto-aluminosilicate hydrate chemical formula and proved H exchange in zeolite structure. The XRD pattern spectrum 2 θ parameter is similar between zeolite of this study and standard zeolite. Figure 2 illustrates the two sharp peaks in 2 θ = 7°–9° and 2 θ = 23°–25° which conforms to the standard pattern of HZSM-5 zeolite. This zeolite also showed a high amount of Si element.

The EDX spectra [Figure 3] showed the element composition of HZSM-5/TiO_2 5% as an example. From the EDX graphs investigation, the elements including AL, O, Na, Si, Ca, Ti, and Ag were found on the surface of the photocatalyst. Among them, Al, Si, O, and Ti had the highest amount and the sharpest peaks.

The BET-specific surface area

The adsorbed and desorbed lines of HZSM-5/TiO_2 photocatalyst showed a type II of sorption isotherm according to IUPAC classification [Figure 4]. The specific surface area was determined by the BET method, showing that HZSM-5 was 289 m^2/g, HZSM-5/TiO_2-3% was 213 m^2/g, 5% was 189 m^2/g, and 8% was 185 m^2/g. The textural properties of samples are shown in Table 1.

Scanning electron microscope image

Figure 5 illustrates the SEM of HZSM-5/TiO2 photocatalyst at 3% weight percent in 2 μm and 500 nm.

The adsorption test of the Photocatalyst

The adsorption tests were carried out according to the procedure presented in the Photo catalyst test section. The different sample adsorption experimental results are shown in Table 2. As indicated in the experimental results in Table 2, by increasing the percentage of TiO_2, the adsorption capacity is increased, but the specific surface area was decreased.

Discussion

With consideration of the importance of adsorption capacity and adsorption properties of absorbent materials such as Zeolites for VOCs treatment, the aim of this study is investigation of HZSM-5 and HSZM-5/TiO_2 adsorption properties for toluene vapors removal from air stream. As mentioned in the result section, the XRD pattern of self-prepared zeolite was compatible with standard pattern.[31]

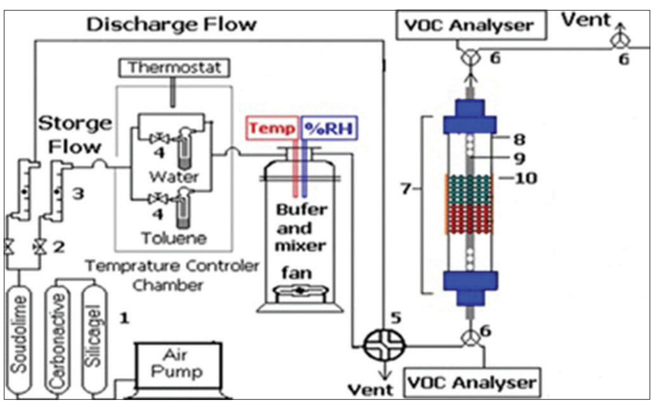

Figure 1: Schematic diagram of experimental setup: (1) air purifier filter; (2) two-way valve; (3) airflow meter; (4) bubbler; (5) four-way valve (6) three-way valve; (7) plasma reactor; (8) quarts tube; (9) high voltage electrode; (10) catalyst[22]

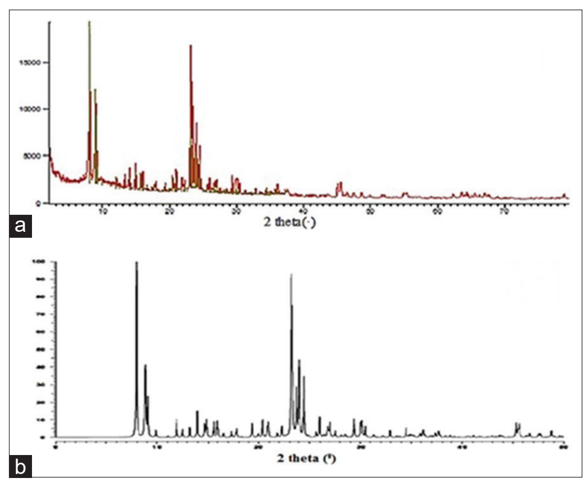

Figure 2: (a) X-ray diffraction spectra on HZSM-5 zeolite; (b) X-ray diffraction standard pattern for HZSM-5 zeolite

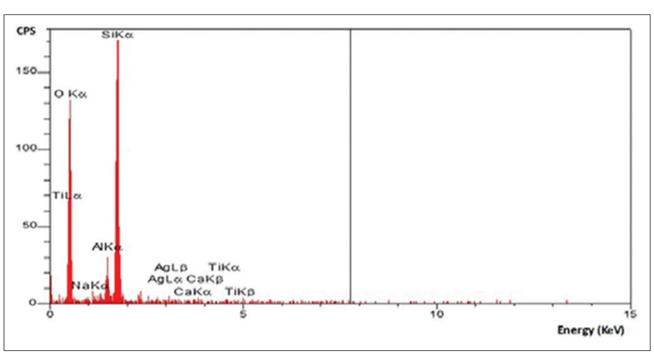

Figure 3: X-ray energy dispersive spectra of HZSM-5/titanium dioxide-5%

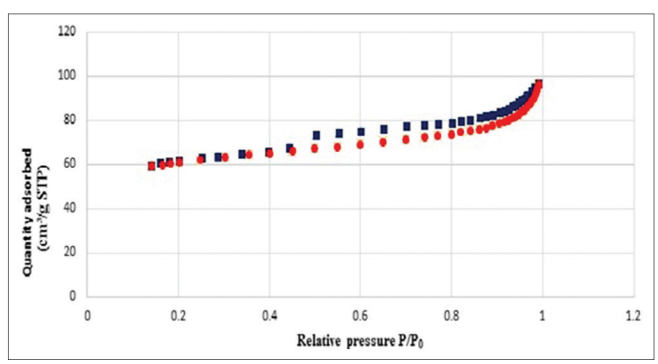

Figure 4: The adsorbed and desorbed line of HZSM-5/titanium dioxide-3%

Table 1: Textural properties HZSM-5 and photocatalyst from BET test

Sample	Total surface area (m^2/g)	Micro pore surface area (m^2/g)	Total pore volume (cm^3/g)	Micro pore volume (cm^3/g)
HZSM-5	298	202.56	0.9	0.103
HZSM-5/TiO_2-3%	213	143.13	0.218	0.08
HZSM-5/TiO_2-5%	189	147.1	-	0.079
HZSM-5/TiO_2-8%	185	124.4	-	0.067

TiO_2: Titanium dioxide

Table 2: Different sample adsorption experimental results

Photocatalyst	$T_{bk\ (min)}$	Bed saturation time (min)	Adsorption capacity (mg/g)	Total time (min)
HZSM-5	286	148	22.63	434
HZSM-5/TiO_2-3tw	301	281	23.81	582
HZSM-5/TiO_2-5tw	481	255	28.88	620
HZSM-5/TiO_2-8tw	365	180	38.06	661

TiO_2: Titanium dioxide

The Si/Al ratio in the current zeolite was 52 that declares the high acidity power of this zeolite. High Si/Al ratio increases the hydrophobicity of zeolite which prevents water condensation in the zeolite cavities and consequently improves its adsorption properties for desired pollutants.[32] The specific surface area of HZSM-5 and HZSM-5/TiO2 at 3, 5, and 8 weight percent was examined by BET test, as indicated in Table 1. HZSM-5 showed the highest specific surface area, and this parameter decreased by adding Tio_2 particles on the zeolite. The large surface area came from the porous surface of zeolite, and most of the TiO2 was coated on the external surface of zeolite.[33] One gram of HZSM-5 adsorbed 22.63 mg of toluene vapors at 0.5 L/min flow rate and 25°C that was more than its capacity for adsorption of thiophene in Weitkamp *et al*. study,[34] and this obtained 23.81, 28.88, and 38.06 mg/g for HSZM-5/TiO_2-3%, 5%, and 8%, respectively. Based on these results, adding TiO_2 particle to the HZSM-5 zeolite improved adsorption capacity and it was maximum for 5% of TiO_2. It has been reported that loading TiO_2 on zeolites takes place on or and then, it can said that adding TiO_2 on the zeolite caused better adsorption of toluene vapors on the acid sides of the zeolite. Then, pollutant degradation will be better when external UV source applies in the system.[35,36] This photocatalyst showed a good capability for capturing toluene vapors, which is compatible with Migliardini *et al*. study. They reported that high adsorption capacity of toluene can be due to its large molecular size, as for a large molecule, the overlap of the adsorption field from neighboring walls enhances the interaction energy.[37] Furthermore, the adsorption capacity was the lowest for HZSM-5 zeolite and the highest for 5% TiO_2 sample, and this percentage can be the best percentage for capturing toluene vapors from the air. It indicates that sample with better surface area does not necessarily present better adsorption capacity or longer breakthrough time.[38] The adsorption capacity rising can be due to the increase in saturation time, as mentioned

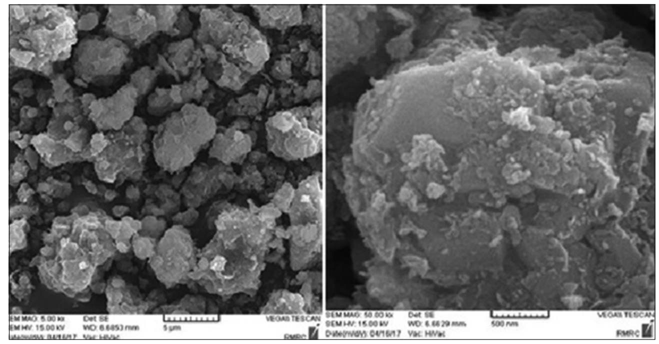

Figure 5: Scanning electron microscope images of HZSM-5/titanium dioxide (3% titanium dioxide)

in Kim and Ahn study.[39] In this case, the BET surface area and pore surface area were decreased with an increase in the percentage of TiO_2 particles, whereas the adsorption capacity showed the reverse procedure. In other words, the adsorption capacity does not appear to depend on the BET surface area or micropore surface area.[39] The breakthrough time for all samples illustrated in Table 2, breakthrough curves divided into three stages. In the first stage, complete adsorption of toluene happened and the concentration of the pollutant in the reactor exhaust was close to zero. In the second stage, the outlet concentration increased slowly and reached the breakthrough point that the pollutant started to exit from the reactor. Then, outlet concentration increased significantly. In the third stage, both inlet and outlet concentration were equal.[38] All of the four samples showed a long breakthrough time for capturing toluene molecules inside themselves that indicate the desired capability of this photocatalyst for adsorption toluene vapors. Introduction of the TiO_2 to HZSM-5 increases the breakthrough time. The longest breakthrough time was 481 min (HZSM-5/TiO_2-5%) and the shortest was 286 min (HZSM-5). It can be concluded that the longer breakthrough time creates a better dynamic adsorption capacity and sufficient time for interaction between toluene vapors and photocatalyst structure, which is compatible with the result of the Zhang *et al*. study.[38]

Conclusion

Adsorption characteristics and adsorption capacity of TiO_2/HZSM-5 in different percentages of TiO_2 for toluene vapor removal from airstream were investigated. With the addition of TiO_2 particles, adsorption and textural properties of HZSM-5 were changed. In the TiO_2/HZSM-5 photocatalyst-specific surface area, micropore surface area was lower than those in HZSM-5 alone. Adsorption capacity, breakthrough time, saturation, or equilibrium time was increased in photocatalyst, which may be because of TiO2 particle interaction with toluene vapors. Among the different TiO_2 weight percentages, TiO_2/HZSM-5-5% showed the highest adsorption capacity and longest breakthrough time. High adsorption capacity and long breakthrough time indicate that this photocatalyst can absorb the high amount of toluene vapors for a long time and provide enough time for the catalytic and photocatalytic reaction for removal and degradation of pollutant.

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Nil.

Conflicts of interest

There are no conflicts of interest.

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