

RESEARCH PAPER

Effect of Nano-TiO₂ Immobilized on Activated Carbon, Zeolite Y and ZSM-5 on the Removal of Styrene Vapors from Polluted Air

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ABSTRACT

TiO₂ is a very well-known and well-researched material due to the stability of its chemical structure, biocompatibility, and its physical, optical and electrical properties. In this study, the synergistic effect of nano-TiO₂ immobilized on activated carbon, zeolite Y and ZSM-5 on the removal of styrene vapors from polluted air was investigated. The prepared catalysts were characterized by SEM, BET and XRD analysis. The enhanced photocatalytic activity of AC, ZSM-5 and Y zeolite for the degradation of the styrene was attributed to the synergistic effect of TiO₂. Photodegradation experiments were carried out at ambient temperature, different styrene concentrations (20, 100 and 300 ppm) and inflow of 0.5 l/min. The results indicate that adding TiO₂ to the adsorbents increased the removal efficiency of styrene and among these catalysts; AC-TiO₂ had a better performance. The high catalytic activity of AC-TiO₂ should be attributed to the interaction between AC and TiO₂, which provides the appropriate ability to adsorb reactants by AC and catalytic oxidation of styrene by TiO₂. Better performances with this promising material – TiO₂ deposited onto AC, ZSM-5 and Y zeolite – compared with bare TiO₂ could be explained by the vicinity of photocatalytic and AC, ZSM-5 and Y zeolite adsorption sites. According to the results obtained from the adsorption capacity and the breakthrough and removal efficiency of catalysts, it can be concluded that coated adsorbents (AC, ZSM-5 and Y zeolite) with TiO₂ can enhance the removal efficiency of styrene.

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INTRODUCTION

With the development of industry and the subsequent awareness of the adverse health effects from air pollution exposure, separation and treatment of organic pollutants in air has become an increasingly important aspect of air pollution control (1, 2). At present, the use of air purifiers

has become popular in removing air pollutants. Traditional air purifiers use filters or adsorption materials for this purpose (3, 4). These methods just transfer the pollutants to another phase instead of removing them, and additional disposal steps are subsequently required (5). An alternative technique, which is a promising, effective and green technology for pollution control, is

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heterogeneous photocatalytic oxidation (PCO) (6-9). PCO actually oxidizes pollutants to H₂O and CO₂ (1, 2) and is cost-effective in comparison with other similar conventional technologies because semiconductors are inexpensive (10). The energy source of photocatalytic processes is natural/UV light; therefore, this method is low in energy consumption but highly effective in degrading most organic compounds (11). Different kinds of photocatalysts such as pure or doped metal oxides (e.g., TiO₂, CdS, ZnO, Fe(III)-doped TiO₂) and chalcogenides have been developed (4, 8, 11). Among these, titanium dioxide has been the dominant photocatalyst because of its strong oxidizing power, especially for aromatic compounds, high photocorrosion resistance, and its nontoxic, stable and inexpensive characteristics, all of which facilitate removing harmful organic compounds in the air and water (9, 12, 13). However, TiO₂ photocatalyst alone has low efficiency due to its small specific surface area (2, 8), and recombination of electron-hole pairs (10, 14). In addition, its performance is not quite well under visible light (6). On the other hand, the link between the fine particle of TiO₂ and the carrier cannot be fast enough and the carrier has a negative effect on photochemical catalyst (15). For these reasons, many efforts have been devoted to minimizing these restrictions and improving the performance of the dominant photocatalyst TiO₂. In this regard, TiO₂ nanoparticle has to be supported (immobilized) on noble metals (e.g., Au, Ag, Pt) nanoparticles, activated carbon and zeolite (e.g., Y, ZSM-5). However, both TiO₂ and metal nanoparticles are unstable and tend to assemble in larger structures, which will reduce the photocatalytic performance (6). Instead, carbon structures can be used for the distribution of TiO₂ particle and increased sensitivity to visible light as well as hindering the recombination of electron-hole pairs (6, 11, 15). Meanwhile, different literatures have reported enhanced photocatalytic activity of TiO₂ supported on zeolites compared with bare TiO₂ (7, 14, 16). However, few, if any, studies have considered supporting TiO₂ on activated carbon and zeolites in order to remove air pollutants. Given the above-mentioned benefits of using TiO₂ supported on activated carbon and zeolite in the photocatalytic process, the present work considered how effective this could be in removing gaseous styrene, as important volatile organic compounds (VOCs) widely used in the

production of synthetic rubber, resins, polyesters and plastics and a proven carcinogenic, mutagenic, and teratogenic agent (6, 9, 17). Therefore, the main purpose of this study was to determine the synergistic effect of nano-TiO₂ immobilized on activated carbon, zeolite Y and ZSM-5 on the removal of styrene from air pollution.

MATERIALS AND METHODS

Materials

Zeolite Y and ZSM-5 were produced in China and purchased from Iran zeolite store. Activated carbon was obtained from Merk CO. These materials were used either as an adsorbent or as TiO₂ support in this study with 20-40 mesh. Degussa Nano-TiO₂-P25 was from purchased from Germany. Styrene (purity≥99.98%) was obtained from Merck, Germany.

Preparation of AC-TiO₂, Y-TiO₂ and ZSM-5-TiO₂ catalysts

In this study, the weight percentage of TiO₂ nanoparticles in the adsorbent coating was selected at 5 %. Many studies reported this to be an appropriate portion (18). For the preparation of AC-TiO₂ catalyst, at first a specific mass of TiO₂ (0.1 gr) was weighed by scale (precision 0.0001), then it was poured into the Arlon containing deionized water and stirred in an ultrasonic generator (Starsonic 18-35.Italy) for 30 min. In this stage, TiO₂ nanoparticles were completely separated and this provided a homogenous suspension. After this stage, a specific amount of activated carbon (1.9 gr) was added to the suspension. Preparation of Y-TiO₂ and ZSM-5/TiO₂ catalysts, were carried out as described above.

Characterization of AC-TiO₂, Y-TiO₂ and ZSM-5/TiO₂ catalysts

The AC-TiO₂, Y-TiO₂ and ZSM-5/TiO₂ catalysts were characterized for surface morphology, specific surface area and X-ray diffraction. The surface morphology of the catalysts was analyzed using the Field Emission Scanning Electron Microscopy (FESEM) technique (Model of MIRA3TESCAN-XMU made, Manufacturing Corporation of TSCAN America). Evaluations for BET specific surface area of the catalysts were determined by Quantachrome Chem BET Instrument. The crystalline of the TiO₂ loaded on the AC, Y and ZSM-5 was identified using the X-ray diffraction (XRD) analysis (X-PERT PRO MPD instrument).

Experimental set-up and analytical method

The experimental set up in this study is shown in Fig. 1. The system consisted of three sections. 1) Input section 2) Adsorb and removal section 3) Sampling system.

Styrene was injected by a syringe pump (manufacturing Co Hitachi Ltd) in three different concentrations (20, 100 and 300 ppm in air). Styrene vapors were produced by bubbling method in which pure air was passed into a styrene generator (composed of a water bath and a wash bottle filled with styrene), and the produced styrene vapor was mixed with pure air as balance gas in the mixing chamber.

Photocatalyst removal section was carried out in a photoreactor containing 2 gr of catalyst. The reactor designed for this study was a cylindrical reactor of quartz glass (to pass UV rays). Illumination was provided by an 8-W UVA lamp that was placed in the center of reactor and three 6-W UVA lamps that were located outside the reactor in a distance of 5 cm from the reactor. According to similar studies, it was found that nanoparticles of TiO₂ and ZnO in the presence of UVA with wavelength of 356 nm have the highest photocatalytic function (19). The length, internal diameter and thickness of this reactor were 280 mm, 20 mm and 2 mm respectively, and it had an inlet at the top and an outlet at the bottom in opposite directions.

The sampling section consisted of a sampling port at the outlet and at the inlet of the reactor. A portable photoionization VOC detector (Phocheck

Tiger, model of 5000, Ion Science Ltd., UK), with a detection limit of 1 ppb – 10,000 ppm, was used to detect the concentration of styrene. In order to ensure the measurement accuracy, some concentrations were also measured by GC (Philips PU4410) equipped with FID detector and Phocheck Tiger.

Finally, the performance index of the catalysts was calculated using adsorption capacity and removal efficiency as follows relations: (20)

$$RE = \frac{C_i - C_o}{C_i} \times 100 \tag{1}$$

Where C_i and C_o are the concentrations of the input and output gaseous styrene, respectively.

$$BC = \frac{Q \cdot C \cdot Tbk}{M_{adsorbent}} \tag{2}$$

where BC is the mass of styrene fed to the adsorber divided by mass of adsorbent at the breakthrough, mg/g; Q is the air flow rate to adsorber, m³/h; C is the concentration of styrene, mg/m³; tbk is the breakthrough time (the time of being observed styrene in the exit gas stream), h; and M adsorbent is the mass of adsorbent, gr.

RESULT AND DISCUSSION

Characterization of the prepared catalysts

The crystal phase of the prepared AC-TiO₂, Y-TiO₂ and ZSM-5/TiO₂ catalysts was analyzed by X-ray diffraction (XRD). X-ray diffraction analysis was performed to assay the phase composition and crystalline nature of the prepared catalysts. It

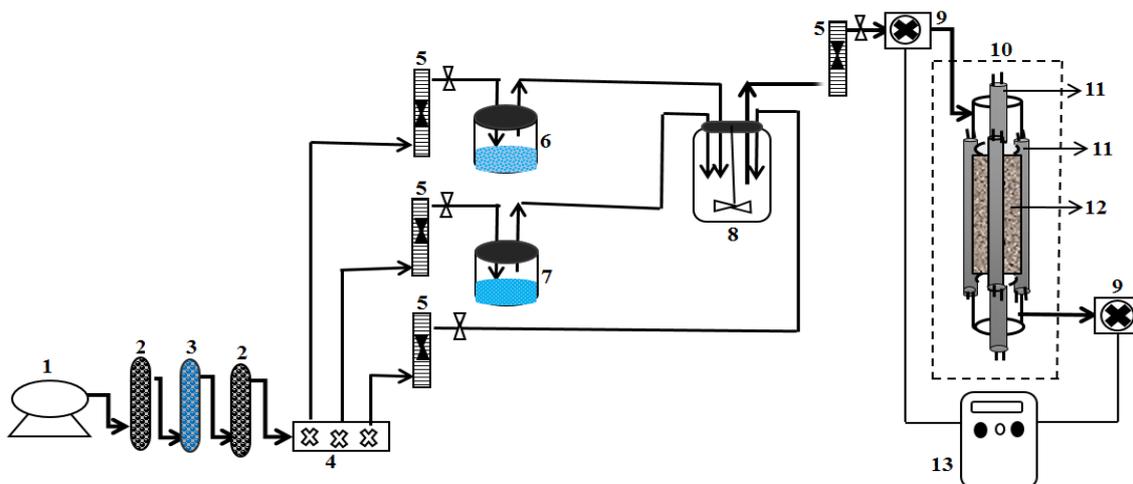


Fig. 1. Experiment schematic (1) Air Pump; (2) Active carbon; (3) SILICAGEL; (4) Branch wall boxes; (5) Rotameter; (6) Styrene container; (7) Water container; (8) Mixing tank; (9) Sampling port; (10) Photocatalytic reactor; (11) UV lamps; (12) Catalyst; (13) Analyzer



is shown in Fig. 2. We observed various diffraction peaks of TiO₂ at 2θ=25.3°, 27.57, 36.23, 37.8, 48.08°, 54.38°, 62.7°, given by AC-TiO₂ catalyst and diffraction peaks of TiO₂ at 25.3°, and 48.08° by Y-TiO₂ and ZSM-5/TiO₂ catalysts.

Fig. 3 shows the SEM images of Y-TiO₂, ZSM-5/TiO₂ and AC-TiO₂ catalysts. Images show the surface morphology of the normal paper. It is

obvious that the surface of ZSM-5 zeolite, Y-zeolite and activated carbon was covered by TiO₂ nanoparticles. Indeed, TiO₂ appears well deposited onto the surface of catalysts.

The BET specific surface areas and pore volumes of catalysts are summarized in Table 1. The specific surface areas of ZSM-5 zeolite, Y-zeolite and activated carbon decreased after loading TiO₂ on

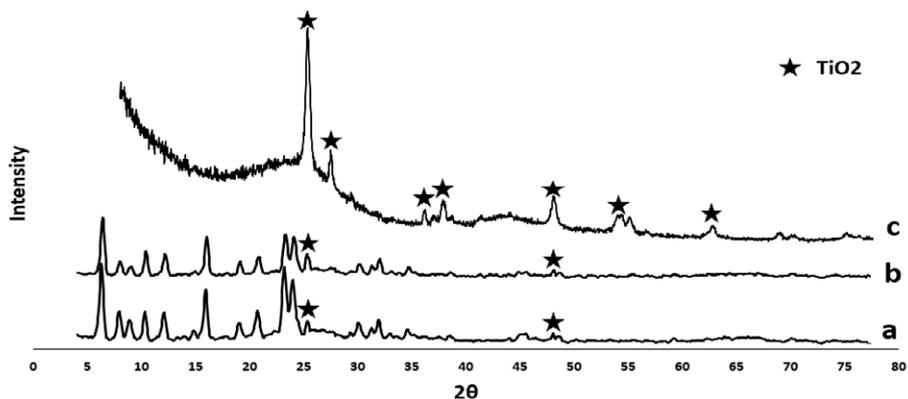


Fig. 2. XRD patterns of: (a) ZSM-5/TiO₂, (b) Y-TiO₂, (c) AC-TiO₂

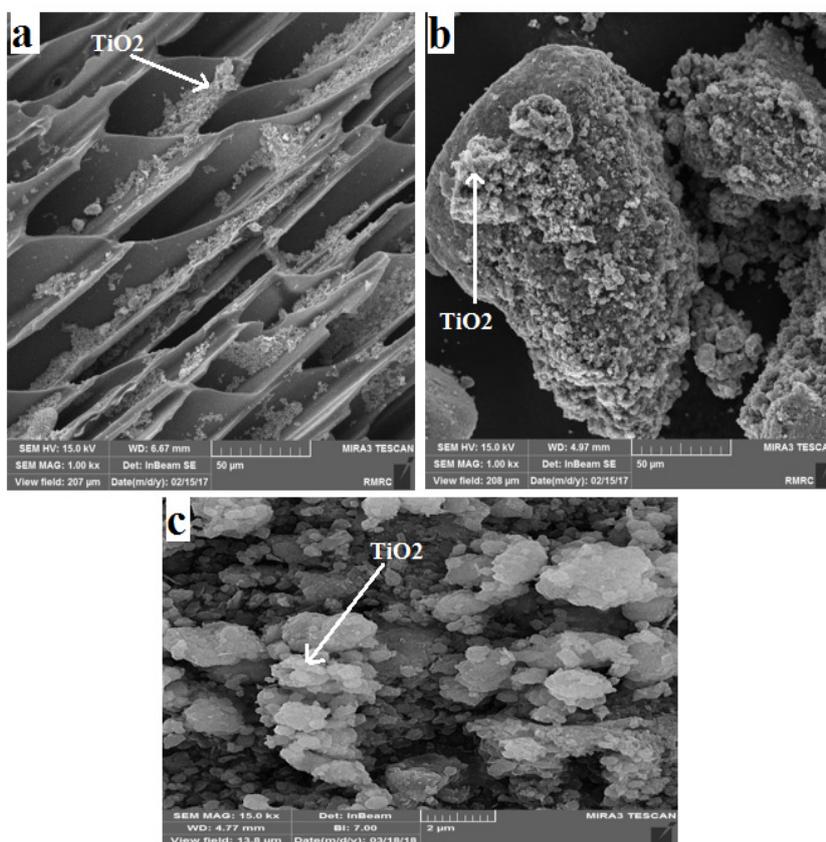


Fig. 3. SEM image from (a) AC-TiO₂, (b) Y-TiO₂, (c) ZSM-5/TiO₂

them. The decrease of specific surface areas and pore volume and the average pore diameter of catalysts after loading TiO₂ indicate that some TiO₂ particles got into pore of catalysts and blocked some channels.

The synergic effect of TiO₂ nanoparticle on AC, ZSM-5 and Y zeolite

Fig. 4 shows the removal efficiency of styrene by bare TiO₂, AC, Y-zeolite, ZSM-5-zeolite, ZSM-5/TiO₂, Y-TiO₂ and AC-TiO₂ at a flow rate of 0.5 l/min and inlet concentration of 20 ppm. When using the catalyst bare-TiO₂, styrene is detected for the first time after 10 min of reaction. After 100 minutes, styrene reached a constant level of 16.5 ppm at the reactor outlet. When AC, Y-zeolite, and ZSM-5-zeolite adsorbents are used, styrene is recognized 43.5, 26.25 and 4.75 hours after reaction, respectively, and again after 77, 47 and 16.5 hours, styrene reached a constant amount of 19.4 ppm at the outlet. Conversely, when AC-TiO₂, Y-TiO₂ and ZSM-5-TiO₂ are used, styrene is observed after 46.75, 28 and 5.66 h of reaction respectively, and after 70, 47 and 8 hours, styrene is fixed at the concentration of approximately 3, 4.55 and 8 ppm. All the supported TiO₂ show better

catalytic activity toward styrene degradation and the photocatalytic activity in the presence of TiO₂-adsorbents is much higher than that shown in the presence of bare TiO₂. In fact, when AC, Y-zeolite, and ZSM-5-zeolite adsorbents are used separately in the reactor, styrene is just transported from gaseous phase to the solid phase and after a while, the adsorbent is saturated. Therefore, the amount of styrene gas at the inlet and outlet parts becomes equal. However, in adsorbents which are coated with TiO₂, styrene is adsorbed by the adsorbent, and then at high concentrations, the pollutant sets around TiO₂ particle, and the adsorption and removal of styrene take place at the same time. This better performance of adsorbents with TiO₂ leads to longer breakthrough time for styrene at the outlet of reactor and higher removal efficiency. In fact, the synergetic effect between adsorption and photocatalytic oxidation led to efficient styrene degradation by AC-TiO₂, Y-TiO₂ and ZSM-5/TiO₂. This finding is confirmed by the results of many studies result. (21, 22) Some researchers have suggested that when compared with bare TiO₂, TiO₂ supported on adsorbent provides a higher specific surface area and is more capable of adsorbing the compounds on the effective

Table 1. Properties of catalysts surface

Sample	ZSM-5	ZSM-5/TiO ₂	Y	Y-TiO ₂	AC	AC-TiO ₂
characteristic						
BET (m ² /gr)	356.4	333.5611	562.882	539.0821	735.36	701.056
The average pore diameter (Å)	25.0185	21.378	29.180	21.2259	36.659	33.962
Total volume (cm ³ / gr)	0.20168	0.18951	0.3112.	0.27911	0.398	0.382

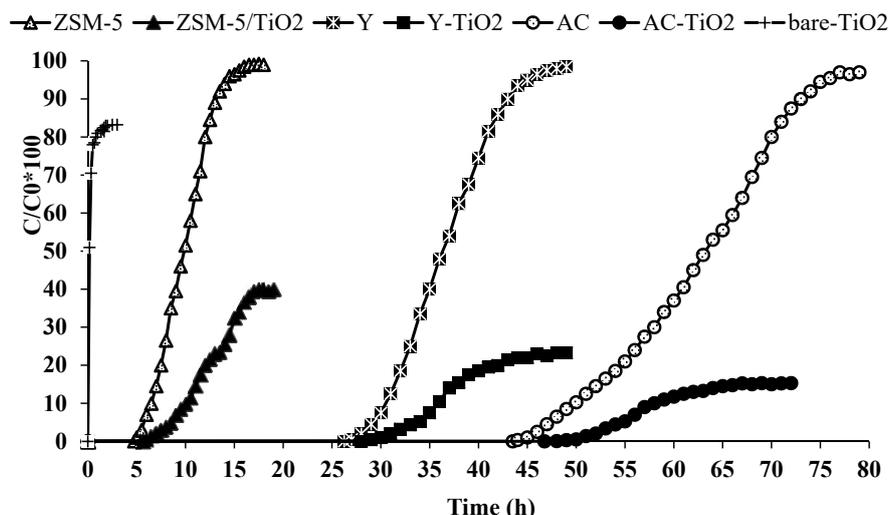


Fig. 4. Photocatalytic removal efficiency of catalysts (C=20 ppm, Q=0.5 l/min)



adsorption site and hence has enhanced removal efficiency. (1, 22)

The effect of concentration on photocatalytic process

The effect of various initial styrene concentrations (20, 100 and 300 ppm) on the photocatalytic removal efficiency of ZSM-5/TiO₂, Y-TiO₂ and AC-TiO₂ catalysts were investigated and illustrated in Fig. 5. The results show that, in

all catalysts increasing the styrene concentration from 20 to 300 ppm decreases the removal efficiency. This finding is related to the kinetic effect and blocking the active sites. Increasing the concentration to 300 ppm makes the adsorption of more styrene molecules possible on the photocatalyst surface and these molecules will block more active sites on the catalyst surface and lead to a reduction of the hydroxyl radical generation on the photocatalyst surface. This

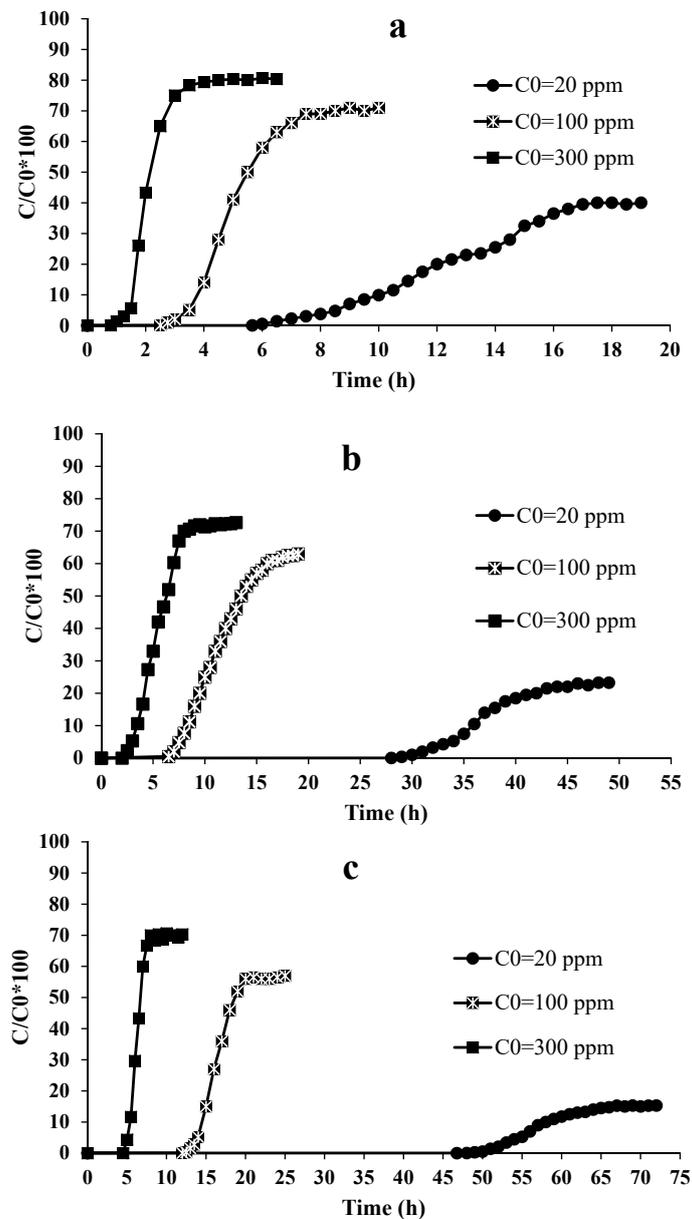


Fig. 5. photocatalytic removal efficiency of (a) ZSM-5-TiO₂, (b) Y-TiO₂ and (c) AC-TiO₂ at different concentration

phenomenon brings about the deactivation of the catalyst and reduces the production of active species on the photocatalyst surface leading to the decrease of photocatalytic removal efficiency of styrene at higher concentrations (23, 24). Also at a low concentration, the adsorption rate of the pollutants over the surface of catalysts is obviously slower than the oxidation reaction rate. Therefore, the adsorption of the gas-phase pollutants is the major factor that determines the removal rate. As the concentration of the pollutants increase, the adsorption and reaction rate get closer, and both of these are important for the overall removal rate. When the concentrations of the pollutants increase to a high level (ppm), the adsorption is much quicker than the photooxidation, and the photoactivity should be the rate controlling factor.

Comparison of removal efficiency of AC-TiO₂, Y-TiO₂ and ZSM-5-TiO₂

The results of this study showed that at each styrene concentration, the overall photodegradation catalyst efficiency varied as: AC-TiO₂ > Y-TiO₂ > ZSM-5/TiO₂, respectively. The result of photocatalytic degradation of styrene using AC-TiO₂, Y-TiO₂ and ZSM-5-TiO₂ is shown in Fig. 6. The difference of elimination of catalysts is due to the structure and specific surface area of catalysts and subsequently adsorption onto catalyst surface. The high catalytic activity of AC-TiO₂ should be attributed to the interaction between AC and TiO₂, which provides the appropriate ability to adsorb reactants by AC and catalytic oxidation of styrene by TiO₂. Also the surface area of the AC

is larger than Y-zeolite and ZSM-5-zeolite, causing more styrene molecules to be adsorbed and to be in contact with TiO₂ and subsequently the photocatalytic removal efficiency of styrene is increased.

Adsorption capacity

The adsorption capacity of the catalysts and adsorbents was investigated at different inlet concentrations (20, 100, and 300 ppm) and the results are presented in Table 2. In all adsorbents and adsorbents coated with TiO₂, with increasing the inlet concentration of styrene, the breakthrough time decreases while the adsorption capacity increases. Studies show that of the higher the concentration, the more mass of pollutants per unit of time removed from air current, so the breakthrough time and saturation of adsorbent reduce (25, 26). The results also demonstrated that the adsorption capacity and breakthrough of adsorbents coated with TiO₂ was higher than those of bare adsorbents in all samples. Comparing the performance of bare adsorbents with that of coated adsorbents indicates that the latter could remove much more styrene from the waste air stream than the former under similar experimental conditions.

One of the effective factors in adsorption capacity is the specific surface area of the material. In this study, the correlation between specific surface area of the adsorbent and the adsorption capacity of styrene showed the following relation in terms of adsorption capacity AC > Y-Zeolite > ZSM-5 Zeolite. Also the results indicated the

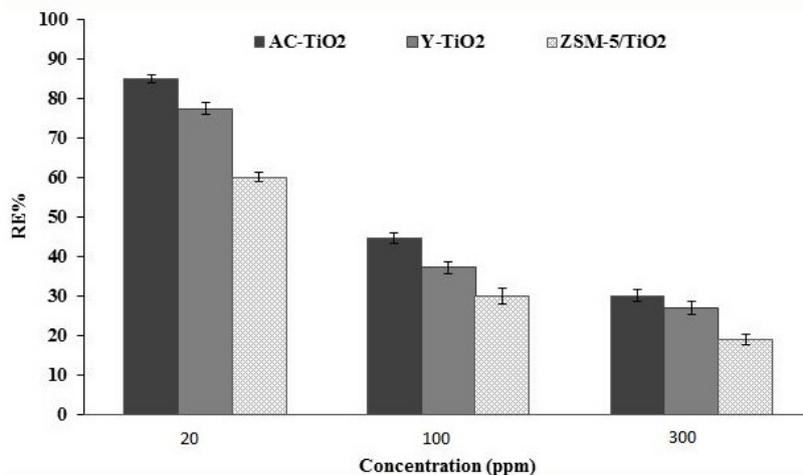


Fig.6. Comparison of removal efficiency of AC-TiO₂, Y-TiO₂ and ZSM-5-TiO₂

Table 2. Results of the breakthrough and catalyst adsorption capacity

Sample	Flow rate (l/min)	Concentration (ppm)	Breakthrough (h)	Adsorption capacity (mg/g)
AC	0.5	20	47	59.974
		100	10.25	65.399
		300	4	76.564
AC- TiO ₂	0.5	20	55	70.182
		100	14	89.325
		300	5.16	98.882
Y	0.5	20	29	37.005
		100	6.75	43.067
		300	2.5	47.852
Y-TiO ₂	0.5	20	34	43.385
		100	7.66	48.916
		300	2.75	52.63
ZSM-5	0.5	20	5.75	7.337
		100	2	12.760
		300	0.83	15.95
ZSM-5/TiO ₂	0.5	20	9.75	12.441
		100	3.5	22.331
		300	1.5	28.71

high surface area and adsorption capacity of photocatalyst can exert a positive influence on their photocatalytic performance. The greater styrene adsorption capacity of coated adsorbents with TiO₂ compared to bare adsorbents must play a role in the photoactivity of TiO₂. Some researchers have determined that with the presence of TiO₂, the hydroxyl (OH[•]) concentration is increased and the degradation process takes a shorter time to complete (27). This can increase the breakthrough time. The results also demonstrated that photocatalysts with high adsorptive capacity enhanced the subsequent photocatalysis reactions and led to a positive synergistic effect.

CONCLUSION

In conclusion, we have demonstrated that bare TiO₂ had a low photocatalytic activity in removal of styrene, but by the loading of TiO₂ on adsorbent (AC, Y and ZSM-5 zeolite) the photocatalytic performance enhances. Among catalysts, AC-TiO₂ showed a much stronger photocatalytic degradation of styrene molecules than did Y-TiO₂ and ZSM-5/TiO₂. Factors like the high surface area of AC and the interaction between AC and TiO₂ may cause enhanced photocatalytic activity of AC-TiO₂. According to our result and those of other studies, high adsorption capacity and excellent mass transfer properties of these catalysts make these materials ideal candidates for industrial applications.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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