

Comparison between the performances of a fluidized-bed reactor and a fixed-bed reactor for the oxidation of benzene to maleic anhydride

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Abstract

The selective oxidation of benzene to maleic anhydride (MAN) was studied to compare the performances of fluidized-bed and fixed-bed reactors. The gas-phase catalytic oxidation of benzene was carried out in laboratory-scale fluidized-bed and fixed-bed reactors with a vanadium pentoxide catalyst supported by silica gel. The influences of parameters such as temperature, space time, and air-to-benzene molar ratio on the reaction selectivity were investigated at normal atmospheric pressure. Similar operating conditions were provided in the experiments carried out in both reactors. Because of the limitations of the experimental sets-up and the recommended operating conditions given in the literature, some differences could be seen between the reactors. It was observed that the conversion of benzene to MAN increased with increasing temperature in both reactors. It was further found that both the conversion of benzene to MAN (x_1) and the total conversion of benzene (x_T) also increased with increasing air-to-benzene molar ratios. This study demonstrates the availability of the 2 reactors for the oxidation reaction of benzene to MAN.

Key Words: Gas-phase oxidation, benzene, maleic anhydride, fixed-bed reactor, fluidized-bed reactor

1. Introduction

The oxidation of organic compounds such as benzene in the vapor phase is an industrially important reaction since the main product, maleic anhydride (MAN), and the side product, phthalic anhydride, are very valuable intermediates. MAN is also referred to as 2-5 furandione, cis-butenedionic anhydride, toxic anhydride, and maleic acid anhydride. This multifunctional chemical intermediate finds applications in almost all fields of industrial chemistry. The principal use of MAN is in the manufacture of alkyd and unsaturated polyester resins, surface coatings, plasticizers, lubricating oil additives, agricultural chemicals, textile chemicals, paper reinforcement, food additives, and pharmaceuticals. Furthermore, due to its double bond and anhydride function, MAN is a versatile intermediate for the production of copolymers of MAN, such as, for example, ethylene glycol and vinyl monomer. Recently, potential new uses of MAN have been found in its conversion to 1,4-butanediol and

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in the manufacturing of tetrahydrofuran (McKetta, 1977; EPA, 1995). MAN is produced industrially by the oxidation of suitable hydrocarbons in the gas phase. Benzene was formerly used as the predominant starting material, but, in the last decade, the oxidation of C_4 hydrocarbons has become increasingly important. MAN obtained from phthalic anhydride production might account for 2% of the total MAN produced (Marx et al., 2011). In the oxidation of C_4 hydrocarbon processes, there are 3 types of process that have been developed or are in the development stages: the fixed-bed process, the fluidized-bed process, and the transport-bed process (Lohbeck et al., 1992; Hakimelahi et al., 2006; Uraz and Atalay, 2007; Marin et al., 2010). The most widely used catalysts in the production of MAN include vanadium pentoxide (V_2O_5), molybdenum trioxide (MoO_3), and sodium oxide (Na_2O). The catalysts proposed in the literature have difficult production processes and expensive supports such as alumina, magnesium, zirconium, beryllium, and titanium. It was seen that the catalysts supported by silica gel and superacidic catalysts can also be used in the oxidation processes (Bond et al., 2000). The catalytic performances are sometimes affected by the crystal phase of titania support, anatase, and rutile, because of differences in the dispersion of vanadia and the formation of reduced vanadium ions. The preparation methods and conditions are the critical factors for the catalytic performances and the structure of supported vanadium oxide. Impregnation, flash hydrolysis, chemical vapor deposition, grafting, and chemical liquid deposition are the preparation methods (Satsuma et al., 2002). The catalyst generally used for the selective oxidation of benzene to MAN consists of vanadium-phosphorus-oxide (VPO). The most important catalytic component in benzene oxidation is V_2O_5 . A catalyst for this purpose is usually modified by the addition of MoO_3 . Overbeek developed a preparation procedure that was cheaper and much more controllable than the preparation procedures of the VPO catalyst. However, the newly developed supported VPO catalysts did not show reasonable catalytic performance (yield below 20%). In general, titania-supported catalysts are much more active than their silica counterparts. However, silica-supported catalysts are much more selective, resulting in better overall yields (Overbeek, 1994; Fernandez et al., 2010).

The aim of this study was to compare the performances of fluidized-bed and fixed-bed reactors by testing the gas-phase oxidation of benzene to MAN on the catalyst prepared by using the knowledge and procedures found in literature. In the same context, the influences of parameters such as temperature, space time, and benzene-to-air molar ratio on the reaction were investigated and characterization of the catalyst was performed.

2. Experimental section

2.1. Catalyst preparation

In this study, a vanadium pentoxide (V_2O_5) catalyst supported by silica gel was prepared to investigate the oxidation of benzene to MAN. The composition of the catalyst is given in Table 1.

Table 1. Composition of the catalyst.

Type of carrier and % by weight	Catalyst	
		Silica gel (72)
Composition of active ingredient, wt%	V_2O_5	53.6
	MoO_3	35.7
	NiO	10.7

The catalyst was prepared by following the procedures described by Marg (1971). Silica gel was initially treated with 72 mL of HCl, 14.4 mL of HNO_3 , and 57.6 mL of H_2O in a porcelain basin. V_2O_5 was then

slurried with water and kept at around 80 °C by heating on a hot plate. Powdered oxalic acid (16.25 g) was added to the slurry with a stirring till. When the V_2O_5 was completely dissolved, it gave a deep-blue-colored solution (solution A). Ammonium molybdate was dissolved in water (solution B) and nickel nitrate was dissolved in water (solution C). A 36-mL mixture of the solutions (prepared by the mixing of solutions A, B, and C) was kept in a sand bath for 10-12 h at 100 °C. The acid was then decanted off, and the gel was again treated with 36 mL of the mixed acid and heated for 2 h with frequent mixing. After the decanting of the acid from the solution, the water was drained off and the gel was dried in an oven. The gel was then spread out on silica trays and left in a muffle furnace. The temperature was gradually raised over the course of 5 h to 700 °C, and the furnace was maintained at this temperature for 2 h.

After the catalyst was prepared, it was ground and a sieve analysis was performed in order to achieve a uniform size distribution of 150 μm . For the fixed-bed reactor, the catalyst was pelletized and then placed into the reactor.

2.2. Apparatus

Two different experimental set-ups were used.

2.2.1. Fixed-bed reactor

A fixed-bed reactor made of Pyrex glass was used for the gas-phase oxidation reaction. A glass tube was fitted into an electrical heated furnace, the temperature of which was controlled by a temperature controller. The reactor was 2 cm in diameter and 21 cm in length. The catalyst was placed onto the catalyst bed and the temperature was controlled with a resistant thermometer connected to a temperature controller. Glass wool was used under the catalyst bed to prevent the sliding of the granular catalyst to the condenser. At the top of the reactor, glass packing was placed to obtain a homogeneous flow of air by preventing channeling. A schematic drawing of the catalytic reactor is shown in Figure 1.

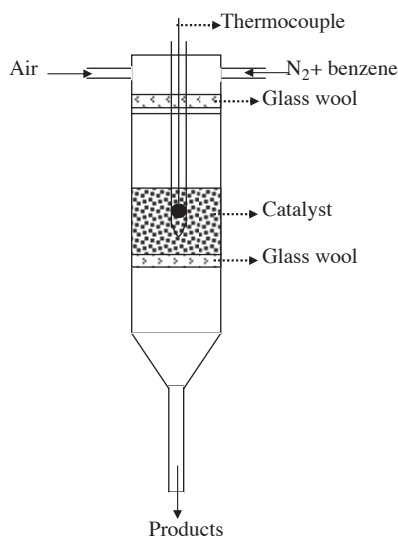


Figure 1. Schematic diagram of the fixed-bed catalytic reactor.

2.2.2. Fluidized-bed reactor

A schematic diagram of the experimental set-up used is shown in Figure 2. A fluidized-bed reactor (Figure 3) with a 25.4-mm inner diameter and a length of 420 mm was used for the reaction. It was made of AISI-316 SS. The catalyst was placed into the reactor and 2 mesh sieves (300 mesh) were placed at the bottom and at the exit of the reactor to support and to prevent the escape of the powder catalyst. An electrical heater, wound around the reactor, was used for heating. For insulation of the reactor, glass wool was used. The reaction temperature was controlled through a temperature controller (PID) connected to an iron-constantan-type Cole-Palmer probe thermocouple. The powers of the heaters and the geometrical parameters of the reactor are listed in Table 2.

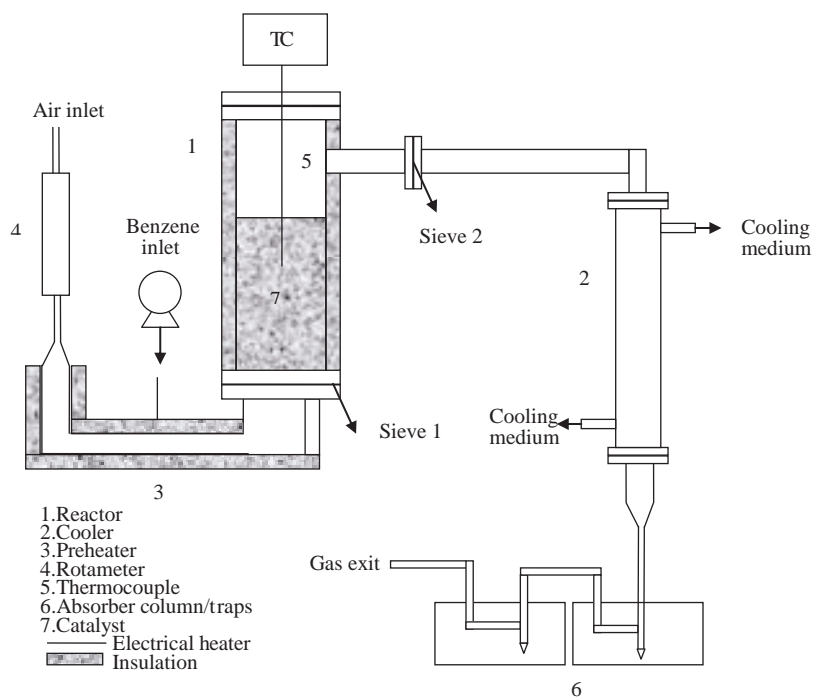


Figure 2. Schematic diagram of the fluidized-bed reactor.

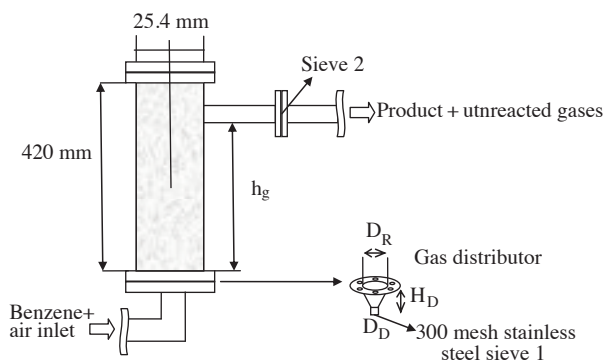


Figure 3. Schematic representation of the reactor.

Table 2. The powers of heaters and geometrical parameters of the reactor.

Reactor material	AISI 316 SS
Heater power supply	250 W
Preheater power supply	300 W
H_R	420 mm
D_R	25.4 mm
r_R	355 mm
Volume of reactor	1050 mm
Catalyst particle diameter	90 μm
Gas dist. dimensions ($H_R \times D_R \times h_R$)	56 \times 30 \times 15 mm

2.3. Experimental procedure

2.3.1. Fixed-bed reactor

The catalyst to be tested was loaded into the reactor and a leakage test was performed. The catalyst was then kept at 350 °C in an air stream for 2 h. Benzene was then charged to the graduated cylinder. The traps were filled with ice and salt and the reactor was turned on. The temperatures of the vaporizer and the reactor were set to the desired values, the air-flow rate was adjusted, the peristaltic pump was turned on, and the flow rate of benzene was adjusted. At the same time, the flow rate of nitrogen was adjusted and the time was recorded. When the reactor temperature reached the desired value, the pressure of nitrogen supplied from the gas cylinder was regulated. The exit-gas flow-rate measurement and carbon dioxide measurements were recorded at specified time intervals. CO₂% measurements were obtained using a CO₂ measuring device (Dräger Polytron IR CO₂), which measured the amount of CO₂ in the exit-gas stream during the experiment duration as both percentage and ppm values. During the experiment, air and nitrogen flow rates were kept at the desired values. At the end of the experiment, after 2 h, the air and nitrogen valves were closed, the heater and peristaltic pump switches were turned off, and the reactor was disconnected and taken out.

2.3.2. Fluidized-bed reactor

One of the most important design parameters in a fluidized-bed reactor is the minimum fluidization velocity (Engelhard Corporation, 2000). This value was calculated theoretically as 0.0154 m s⁻¹. The experiments were carried out at higher air-flow rates than this value and satisfied the minimum fluidization velocity.

Before starting the experiment, the sieved catalyst was charged into the reactor and leakage control was performed. The catalyst was then kept at 350 °C in an air stream for 2 h. The temperatures of the reactor and the preheater were set to the desired values, the circulation of the cooling fluid (tap water) was begun, and the benzene was charged to the graduated cylinder. After the temperature reached the desired value, benzene was supplied to the air stream at the desired flow rate. The time and the liquid level in the measuring cylinder were then recorded, and the flow rate of air was adjusted by the valve on the calibrated rotameter and sent to the preheater and then to the reactor. The effluent passed through the cooler and then in through the traps. The flow rates of exit gas in all experiments and CO₂% in some experiments were measured at specified time intervals. The uncondensed gas was purged into the atmosphere. At the end of the experiment (after 2 h), the pump was switched off and the preheater and reactor were cooled down. The air was allowed to flow for a while and was then stopped.

For both reactors, the product mixture obtained in each experiment and collected in the traps was washed with 2 mL of acetone and then analyzed with a Hewlett Packard 5890 Series II gas chromatograph with a flame ionization detector (FID) and HP integrator. The conditions for analysis of the gas chromatography (GC) are given in Table 3.

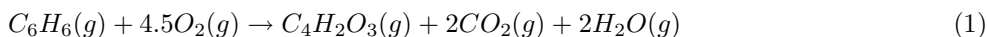
Table 3. The conditions for analysis on gas chromatograph.

Column	HP-5 (cross linked 5%Ph Me silicone)
Column dimensions	25 m × 0.32 m × 0.52 μm
Carrier gas	Nitrogen
Injection temperature	270 °C
Detector	FID
Detector temperature	300 °C
Sample amount	0.4 μL
Oven temperature	100 °C
Total analysis time	34.5 min

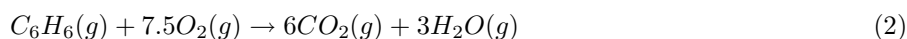
3. Evaluation of the experiments

The conversion of benzene was calculated by considering 2 possible main reactions that could take place in the reactor.

Partial oxidation:



Total oxidation:



Condensed and collected components were analyzed by GC. The weight percent of MAN was calculated with the help of GC analysis. At the end of each experiment, the collected products in the traps were weighed. The produced mole number of MAN and total benzene fed into the reactor were then calculated. The conversion of benzene for reaction 1 (x_1) was calculated using the following equation:

$$x_1 = \left[\frac{\text{Consumed benzene in reaction 1}}{\text{Benzene fed}} \right]. \quad (3)$$

Since the total flow rate of the exit gas and CO₂ % were measured during the experiments, the mole number of the CO₂ was also calculated. The conversion of benzene for reaction 2 was calculated using following equation:

$$x_2 = \left[\frac{\text{Consumed benzene in reaction2}}{\text{Benzene fed}} \right]. \quad (4)$$

The total conversion of benzene (x_T) was calculated using the following equation:

$$x_T = \left[\frac{\text{Total consumed benzene}}{\text{Benzene fed}} \right]. \quad (5)$$

Partial selectivity is defined as the ratio of the benzene conversion in reaction 1 to that in reaction 2. Total selectivity is defined as the ratio of benzene conversion for reaction 1 to the total conversion.

$$\text{Partial selectivity} = \left[\frac{x_1}{x_2} \right] \quad (6)$$

$$\text{Total selectivity} = \left[\frac{x_1}{x_T} \right] \quad (7)$$

4. Results and discussion

In this study, with a vanadium pentoxide (V_2O_5) catalyst supported by silica gel, selective oxidation of benzene to MAN was studied in a laboratory-scale fixed-bed reactor and fluidized-bed reactor. During the experiments, the effects of temperature, space time, and benzene-to-air molar ratio on the reaction selectivity were investigated.

The operating conditions are given in Table 4.

Table 4. Operating conditions in the experiments with pressure of 1 atm.

Parameter	Range	
	Fixed-bed	Fluidized-bed
Reactor temperature, T_R ($^{\circ}\text{C}$)	300-375	350-400
Space time ($W/F_{B0} = \text{g s mol}^{-1}$)	(1×10^6) - (1.42×10^6)	(0.915×10^6) - (2.96×10^6)
Catalyst amount (g)	2	15
Air-to-benzene ratio (mol:mol)	40.82:163.39	27.62:90.17
Catalyst-to-benzene ratio (w:w)	1.97:3.79	2.46:17.83

4.1. Fixed-bed reactor

Some of the results obtained by the experiments performed with the fixed-bed reactor are plotted in Figures 4-6. The influence of temperature on conversion at a constant space time ($1.33 \times 10^6 \text{ g s mol}^{-1}$) and at a constant air-to-benzene ratio (41.37) can be seen in Figure 4. It is clear that both total conversion of benzene (x_T) and

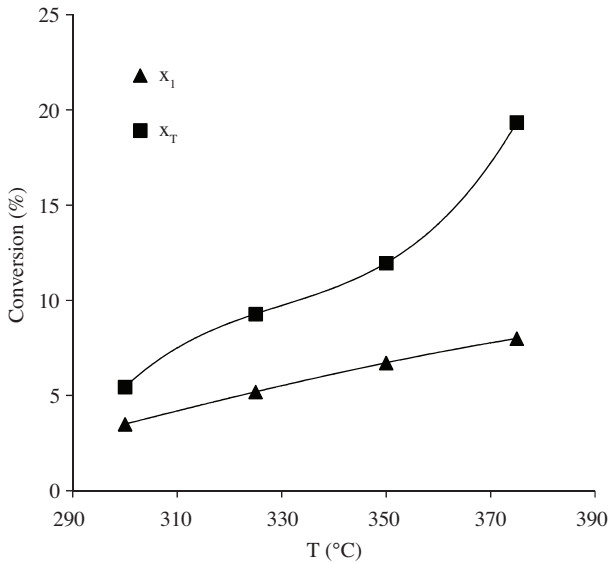


Figure 4. Conversion versus temperature at constant space time of $1.33 \times 10^6 \text{ g s mol}^{-1}$ and constant air-to-benzene ratio of 41.37.

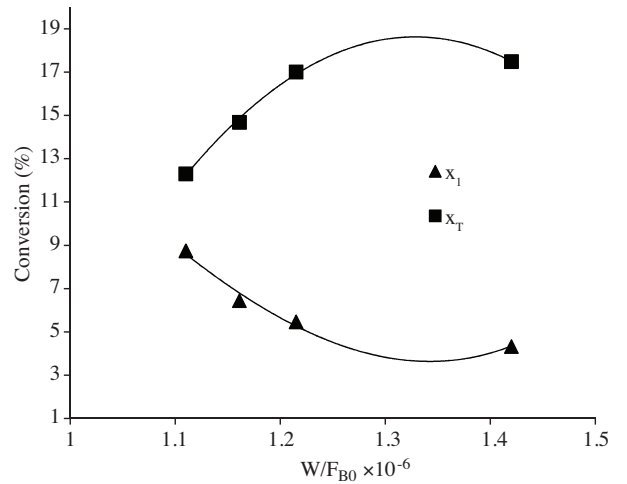


Figure 5. Conversion versus space time at constant temperature of 350°C and constant air-to-benzene molar ratio of 115.74.

conversion of benzene to MAN (x_1) increased with the increase in temperature. As the temperature increased, the difference between the total conversion and the conversion of benzene to MAN (x_1) became larger. This finding might emphasize that the increase in temperature favored the total oxidation rather than the selective oxidation.

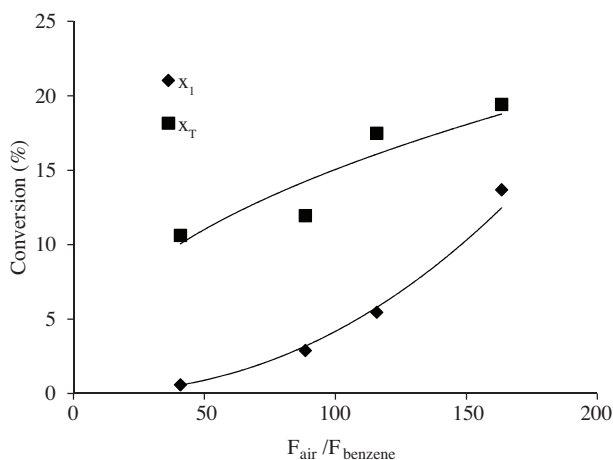


Figure 6. Conversion versus air-to-benzene molar ratio at constant temperature of 350 °C and constant space time of $1.33 \times 10^6 \text{ g s mol}^{-1}$.

Figure 5 was prepared to show the influence of space time on conversion. It was drawn at a constant temperature (350 °C) and at a constant air-to-benzene molar ratio (115.74). As can be seen easily, the total conversion of benzene increased and the conversion of benzene to MAN decreased with increasing space time. This finding is in accordance with the results cited in literature (Sugiyama et al., 1999). It can thus be said that when the benzene flow rate is increased, the reaction tends to go in the direction of total oxidation. Longer space time might cause the further conversion of MAN produced by selective oxidation.

Figure 6 was plotted to see the effect of the air-to-benzene molar ratio on the conversion at a constant temperature (350 °C) and at a constant space time ($1.33 \times 10^6 \text{ g s molB}^{-1}$). The minimum flow rate of air corresponding to the stoichiometric amount of air was $2.54 \times 10^{-4} \text{ mol min}^{-1}$ in the first reaction. It can be seen that the conversion of benzene, both through MAN and through CO_2 , increased generally with increased air-flow rate, although low conversion values were obtained in reaction 1. As the flow rate of air increases, it can be passed through the region in which the external mass transfer effect can be neglected. The results presented in Figure 6 show that the reaction was still under the mass transfer effect.

4.2. Fluidized-bed reactor

In this part of the study, the oxidation of benzene was studied in a fluidized-bed reactor using the same catalyst. Effects of the parameters were compared for experiments performed in the temperature range of 325-400 °C and at nearly constant space-time values. It can be seen in Figure 7 that the conversion of benzene to MAN increased with the increasing temperature of the catalyst supported by the silica gel. Due to the insignificant conversion between 350 and 370 °C, the temperature range was extended to 400 °C, although with this extension, the conversion values were lower than those of the fixed-bed reactor.

The total flow rate of the exit gas in all of the experiments and the $\text{CO}_2\%$ in some experiments were

measured. Since there were not $\text{CO}_2\%$ values in all experiments, the conversion of benzene for reaction 2 (x_2) and the total conversion of benzene (x_T) could not be calculated for the fluidized-bed reactor.

Figure 8 shows the influence of space time on the conversion. It was plotted at a constant temperature ($400\text{ }^\circ\text{C}$) and at a constant air-to-benzene molar ratio (50). As can be seen from the graph, the conversion of benzene to MAN decreased with increasing space time. It can thus be said that when the benzene flow rate is increased, the reaction tends to the direction of MAN production.

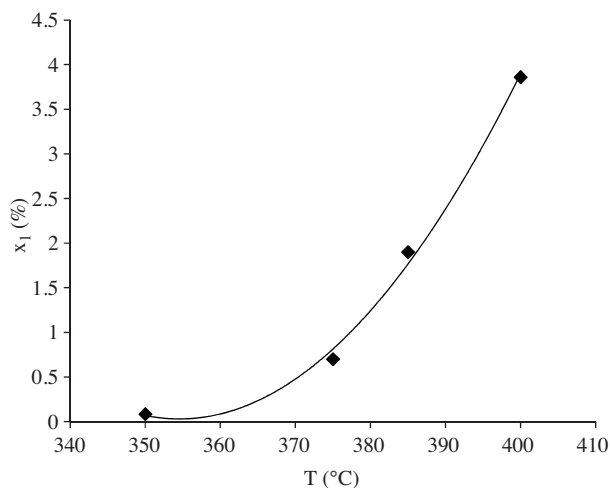


Figure 7. Conversion versus temperature for constant W/F_{B0} of $1.38 \times 10^6 \text{ g s molB}^{-1}$ and air-to-benzene molar ratio of 72.99.

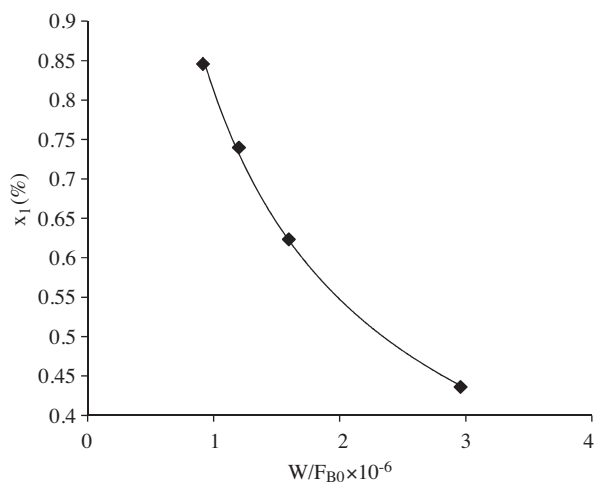


Figure 8. Conversion versus space time at constant temperature of $400\text{ }^\circ\text{C}$ and constant flow rate of air of $673.43 \text{ mL min}^{-1}$.

If the experiments that were carried out at a constant temperature ($400\text{ }^\circ\text{C}$) and at a constant space-time value ($W/F_{B0} = 1.63 \times 10^6 \text{ g s molB}^{-1}$) are investigated, it is seen in Figure 9 that conversion increased with an increasing air flow rate.

4.3. Catalyst characterization

4.3.1. Infrared studies

To understand whether there was any loss in the activity of the catalysts, fresh and used catalysts were characterized. The catalyst was reused for each experiment. The experiments were not always begun with fresh catalyst. Infrared (IR) spectroscopy analyses of fresh and used catalysts were performed on a Shimadzu IR-470. When the IR spectra of fresh and used catalysts were investigated (Figures 10 and 11), the same peaks are detected for both catalysts. It can thus be said that the catalyst might not lose its activity. In Figure 10, at 1600 cm^{-1} , there is a peak attributed to the SiO_2 compound in the catalyst, and at 3600 cm^{-1} , there is an OH stretching bond for the same reason.

4.3.2. Nitrogen adsorption studies

The nitrogen adsorption isotherms of the catalysts are shown in Figure 12. The specific surface areas (BET) of the catalysts were characterized by multipoint physical adsorption of N_2 using a Micromeritics ASAP 2010 instrument at 78.23 K . The different surface characteristics measured with the nitrogen adsorption, such as

BET, Langmuir surface areas, micropore area, and micropore volume of the catalysts, are shown in Table 5. The adsorption isotherms for the catalysts may be classified as type V according to the Brunauer, Deming, and Teller classification. Such isotherms represent the presence of microporosity or mesoporosity. As seen in Table 5, the pores decreased in used catalysts.

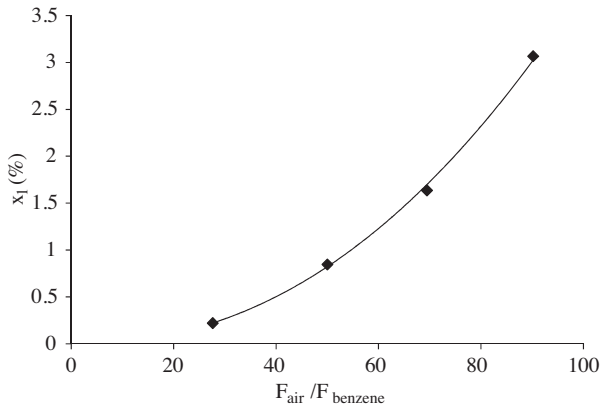


Figure 9. Conversion versus benzene-to-air molar ratio at constant temperature of 400 °C and constant space time of $1.633 \times 10^6 \text{ g s molB}^{-1}$.

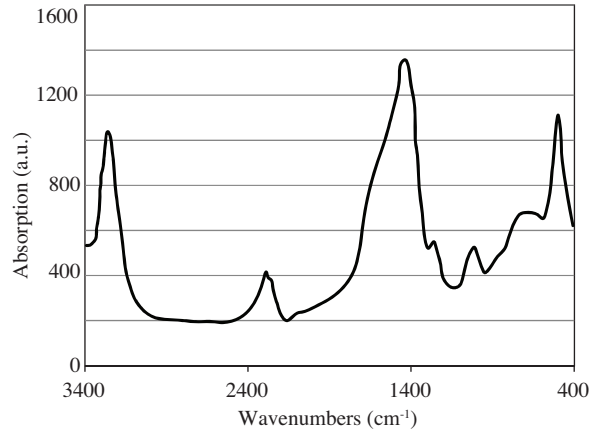


Figure 10. The results of IR spectroscopy of fresh catalyst.

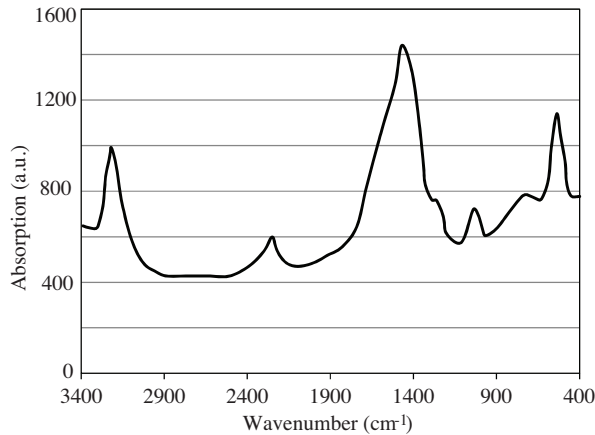


Figure 11. The results of IR spectroscopy of used catalyst.

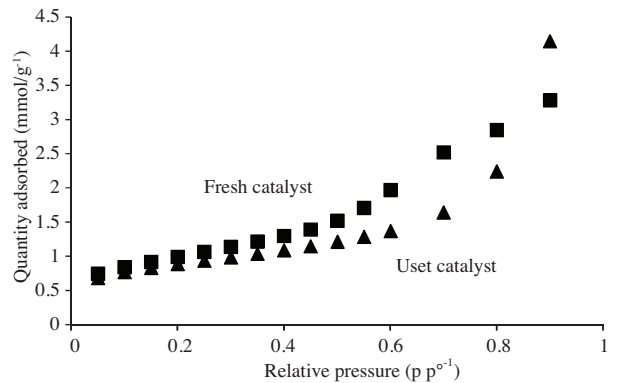


Figure 12. Nitrogen adsorption isotherm of fresh and used catalyst.

Table 5. Results of the nitrogen adsorption studies of the catalysts.

Catalysts	BET surface area ($\text{m}^2 \text{g}^{-1}$)	Langmuir surface area ($\text{m}^2 \text{g}^{-1}$)	T-plot external surface area ($\text{m}^2 \text{g}^{-1}$)	Micropore area ($\text{m}^2 \text{g}^{-1}$)	Total pore volume ($\text{cm}^3 \text{g}^{-1}$)	Micropore volume ($\text{cm}^3 \text{g}^{-1}$)
Fresh catalyst	48.82	450.24	77.22	90.115	0.025929	0.000687
Used catalyst	45.46	347.46	57.31	69.72	0.023709	0.005664

4.3.3. X-ray diffraction studies

The X-ray diffraction (XRD) analyses of fresh and used catalysts were performed on a Phillips X'Pert Pro multipurpose X-ray diffractometer operating with Cu K α radiation over the 2θ range of 5° - 70° and a position-sensitive detector with a 0.033 step size. The XRD diagram of the fresh and used catalysts is seen in Figure 13. According to the results, there were no remarkable changes in the catalyst structure after the reaction. It can be also said that the catalyst showed an amorphous structure. The crystal structure may be affected by the high calcination temperature.

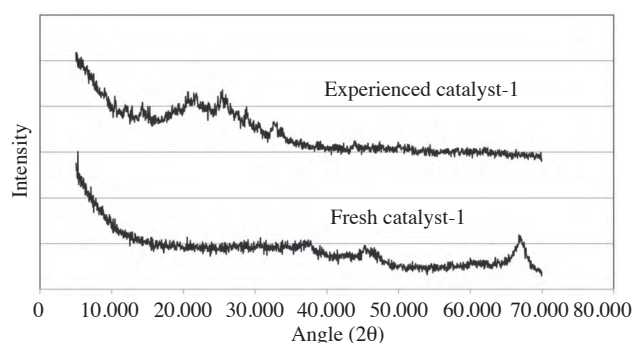


Figure 13. The result of XRD analyses of fresh and used catalyst 1.

4.3.4. Thermogravimetric analysis studies

Characterization of the catalysts was also carried out to measure the weight loss as a result of the increase in sample temperature. Thermogravimetric analysis (TGA) results are given in Figures 14 and 15.

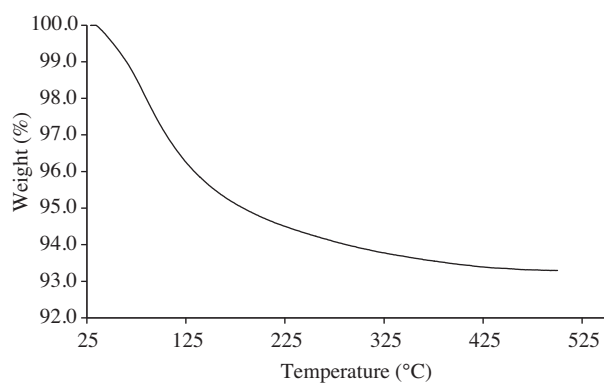


Figure 14. TGA of fresh catalyst.

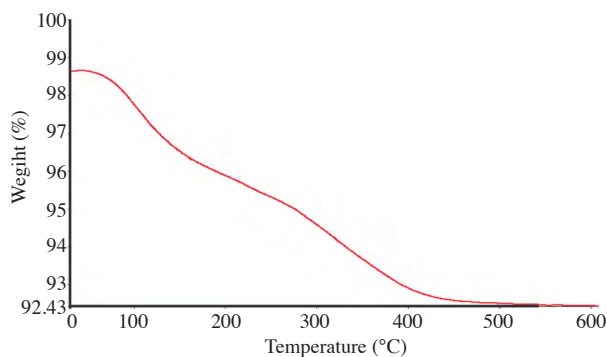


Figure 15. TGA of used catalyst.

The thermogravimetric curve in Figure 14 seems to indicate an overall weight loss of 7% for the fresh catalyst with the removal of physisorbed water within the temperature range of 25-100 °C and weight loss at 100-500 °C. Generally, a small amount of weight loss was observed for the catalysts and there was no significant weight loss in the fresh catalyst. The other thermogravimetric curve in Figure 15 seems to indicate an overall weight loss of 6% for the used catalyst with the removal of physisorbed water within the temperature range of 30-100 °C and weight loss at 100-500 °C. A small amount of weight loss was observed for the used catalyst and there was no significant weight loss in the used catalyst.

The performances obtained with the 2 different reactors under the same conditions were comparable. This work has demonstrated the possibility of using these reactors for the oxidation reaction of benzene to MAN. Oxidation of benzene to MAN was obtained with both reactors. The V_2O_5 catalyst supported by silica gel is suitable for the oxidation of benzene to MAN in a fixed-bed reactor and in a fluidized-bed reactor. In the experiments carried out in the fixed-bed reactor, higher MAN conversions were obtained. However, in the experiments carried out in the fluidized-bed reactor, almost 3%-4% of the conversions were obtained. If a comparison between the performances of the fixed-bed reactor and the fluidized-bed reactor is made, it can be said that the fixed-bed reactor had higher performance than the fluidized-bed reactor. According to the catalyst deactivation, there was no difference seen between the fixed-bed reactor and fluidized-bed reactor. As a result, it can be said that the fixed-bed reactor could be used for oxidation of benzene to MAN by using a V_2O_5 catalyst supported by silica gel. It can be concluded that a fixed-bed reactor is a more suitable reactor for the oxidation of benzene to MAN using a V_2O_5 catalyst supported by silica gel.

5. Conclusions

- SiO_2 is a suitable support for the oxidation of benzene to MAN.
- Conversion of benzene to MAN and total oxidation increased with increasing temperature.
- When space time is increased, the reaction tends to the direction of MAN production.
- As the flow rate of air increases, it can be passed through the region in which the external mass transfer effect can be neglected. The results show that the reaction is still under the mass transfer effect.
- The fixed-bed reactor had a higher performance than the fluidized-bed reactor for the oxidation of benzene to MAN using a V_2O_5 catalyst supported by silica gel.
- The fixed-bed reactor could be used for the oxidation of benzene to MAN with a V_2O_5 catalyst supported by silica gel.
- The adsorption isotherms for both catalysts may be classified as type V, indicating the presence of microporosity or mesoporosity.
- After reaction, the surface and the micropore area of both catalysts decreased due to the possible plugging of the pores by metal oxide crystallites.
- Generally, a small amount of weight loss was observed and there was no significant weight loss in either fresh or used catalysts.

Nomenclature

F	flow rate (mol s^{-1})	M, m, wt	weight (g)
F_{B0}	flow rate of benzene (mol s^{-1})	MW	molecular weight (g mol^{-1})
GC	gas chromatography	T	temperature ($^{\circ}\text{C}$)
MAN	maleic anhydride	W	weight of catalyst (g)
		W/F_{A0}	space time (g s mol^{-1})
		x	conversion (%)

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