Study and optimization of styrene waste gas purification process in bioscrubber

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Abstract. In order to explore the removal effect of styrene waste gas by bioscrubber with multi-sided hollow spheres as filler, the residence time, circulating liquid pH and gas-liquid ratio were taken as experimental factors, and the removal rate of styrene was taken as the response value. The experiment was designed according to Box-Behnken, and the response surface analysis method was adopted to establish the corresponding predictive regression model and conduct the analysis of variance. The optimization module of Design-Expert software was used to optimize the optimal combination of all process parameters: the residence time was 43.18 s, the pH of circulating liquid was 7.61, and the gas-liquid ratio was 3.95. Under this condition, the removal rate of styrene was 88.52%. The influence of different fillers on the purification performance of biological tower was compared and analyzed, so as to provide the basis and basis for the application of efficient VOCs processing technology in bioscrubber.

1. Introduction

Styrene is a kind of widely used organic chemical material [1]. In the process of chemical production and use, there may have the styrene gas emission [2]. Styrene, as a typical volatile organic compounds (VOCs) which are difficult to be biodegraded, is recognized as one of the eight odor gases [3], which has a huge potential safety hazard to the ecological environment and human health [4]. Because of its potential harm, environmentalists and scholars at home and abroad have attached great importance to the removal of styrene. How to properly deal with styrene waste gas has become a research hotspot.

In recent years, due to the advantages of low cost, high removal efficiency and no secondary pollution, biological methods have attracted more and more attention [5,6,7,8]. Biological method mainly includes three technologies: biological filtration, biological trickling filtration and biological washing. Among them, biofilter and biotrickling filter have been successfully applied to the treatment of styrene waste gas [9,10,11,12]. Liu J.W. [13] used the fungal biofilter to treat styrene waste gas, and the results showed that the fungal biofilter had a good treatment effect on styrene, with the maximum removal capacity of 66.78 g/(m³·h); the production of CO₂ and the removal of styrene showed a linear relationship, indicating...
that styrene was completely mineralized by the micro biodegradation. Yang B.R. [14] used multi-sided hollow spheres and activated carbon fiber as filler of biotrickling filter to purify styrene waste gas, and the results showed that the maximum removal load of biotrickling filter could reach 136.4 g/(m³·h); the system had strong adaptability to the large change of pH of spray liquid and intermittent operation. Compared with biofilter and biotrickling filter, the biological washing method has the advantages of easy control of reaction conditions and less blockage of filler. Liu Y.H. [15] used bioscrubber to treat waste gas containing phenol. It was found that the average removal efficiency of long-term operation was 97%, and the removal load was about 30 g/(m³·h). When the phenol load was more than 50 g/(m³·h), the liquid phenol would accumulate, which affected the stable operation of the system. Therefore, the high-load degradation of phenol gas needs further study.

In this experiment, the bioscrubber is used to purify styrene waste gas. The influencing factors and removal effect of styrene waste gas by bioscrubber with multi-sided hollow spheres as filler are analyzed and studied. On the basis of single factor experiment, the best process parameters of styrene waste gas removal are optimized by response surface method.

2. Materials and methods

2.1 Experimental equipment and process

See Figure 1 for the experimental equipment of removing styrene waste gas by bioscrubber, which is mainly composed of waste gas generation system, bioscrubber and biochemical reactor. The bioscrubber is composed of organic glass with a diameter of 100mm and a height of 800mm, with a total volume of 6.28L. The filler adopts a multi-sided hollow sphere with a diameter of 25mm, and the total height of the filler layer is 90mm. After passing through the air pump, the air is divided into two parts of gas; one part of gas enters the gas generating bottle, which makes the styrene gas volatilize from the pure styrene bottle; the other part of gas enters the buffer bottle, and the two parts of gas mix evenly in the buffer bottle to obtain a certain concentration of styrene waste gas for simulation. In the experiment, the gas-liquid countercurrent operation is adopted. The waste gas enters from the bottom of the tower, and mass transfer adsorption will be carried out with the microorganism on the filler and the circulating liquid containing activated sludge which is lifted to the top of the tower for spray by the water pump; part of the waste gas is degraded here, but most of the styrene gas is absorbed and returned to the biochemical reactor by the circulating liquid, and is metabolized and degraded by the microorganism in the activated sludge.

Figure 1. Schematic diagram of experimental equipment.
2.2 Culture and acclimation of activated sludge
The activated sludge used in this experiment is taken from an aerobic tank for wastewater treatment of a pharmaceutical factory in Shijiazhuang. The circulating liquid is aerated intermittently by air pump, i.e. daily aeration for 20 h and stopping aeration for 4 h to keep the dissolved oxygen concentration at 2-6 mg/L and the pH value at about 6-7. Replace 0.5 L nutrient solution every 24 h, keeping \( \frac{m(C):m(N):m(P)}{} = 100:5:1 \). The formula of nutrient solution is: glucose 2500 mg/L, urea 100 mg/L, mono-potassium phosphate 50 mg/L, magnesium sulfate 250 mg/L, calcium chloride 300 mg/L and ferrous sulfate 25 mg/L. Sludge settling velocity (SV), mixed liquid suspended solids (MLSS), sludge volume index (SVI) and chemical oxygen demand (COD) are used as parameters to investigate the growth of activated sludge. In order to acclimate activated sludge, liquid styrene is added into the circulating liquid as the only carbon source, and its evaluation index is the same as that of the culture stage.

The changes of SV, MLSS, SVI and daily residual COD in the culture stage are shown in Figure 2. It can be seen from Figure 2 that after 14 days of culture, SV increases to about 16%, MLSS increases to 1100 mg/L, SVI stabilizes at 140-150 mL/g, and daily residual COD decreases to 45 mg/L. In the culture stage, it can be observed that the sludge flocs gradually increase, the settling performance is significantly improved, and the supernatant becomes clear from turbid, which indicates that the biomass in the activated sludge increases rapidly, the microbial activity is frequent and active, and the metabolic activity is also enhanced.

The changes of SV, MLSS, SVI and daily residual COD in acclimation stage are shown in Figure 3. It can be seen from Figure 3 that SV, MLSS and SVI fluctuate greatly in the early stage of acclimation, and then gradually stabilize after 10 days. Then MLSS steadily increases to 1280 mg/L, and the daily residual COD gradually decreases until the completion of acclimation on the 16th day. The color of activated sludge becomes lighter, the flocs are fine and the settling performance is good.

2.3 Analysis method
The mass concentration of styrene is determined by 7890B-5977 gas chromatography-mass spectrometer of Agilent Co, and the column is HP-5MS quartz capillary chromatographic column. Gas phase conditions: the inlet temperature is 200 °C, the carrier gas is helium, the split ratio is 5:1, and the column flow (constant current mode) is 1.2 mL/min. Heating procedure: the initial temperature is 30 °C, maintained for 3.2 min, and the temperature is raised to 200 °C at 11 °C/min, maintained for 3 min. Mass spectrometry conditions: the scanning mode is full scanning, the scanning range is 35~270 amu, the ionization energy is 70 eV, and the interface temperature is 280 °C.

3. Results and discussion
3.1 Effect of inlet concentration on styrene removal capacity
The inlet concentration of styrene directly affects the degradation performance of the bioscrubber. Therefore, under the conditions of residence time of 37.5 s, pH of 7 and gas-liquid ratio of 4, the experiment is divided into three stages according to the inlet concentration of styrene: low, medium and
high, i.e. the inlet concentration of styrene is 200-400, 600-1000 and 1400-1600 mg/m$^3$, respectively. The effect of the inlet concentration on the purification of styrene waste gas by bioscrubber is investigated. The results are shown in Figure 4.

![Figure 4. Effect of inlet load on styrene removal rate.](image1)

![Figure 5. The relation between inlet load and removal load.](image2)

It can be seen from Figure 4 that with the increase of styrene inlet concentration from 194 mg/m$^3$ to 1656 mg/m$^3$, the removal rate decreases from 85.00% to 40.00%; with the increase of inlet load, the removal load gradually increases from 15.63 g/(m$^3$·h) to 80.05 g/(m$^3$·h), and when the inlet load further increases to 140.58 g/(m$^3$·h), the removal load decreases to 58.10 g/(m$^3$·h).

In the low concentration stage, the removal rate of styrene is relatively stable, and the average removal rate of styrene waste gas in the bioscrubber can reach 86.65%. On the 8th day, the inlet concentration of styrene is increased to 655.37 mg/m$^3$, and the removal rate is reduced to 60.59%. After operation for a period of time, the removal rate can increase to 83.60%, indicating that microorganisms have strong adaptability to the change of inlet concentration. Although the inlet concentration of styrene is increased a lot, it still has a good treatment effect of styrene, and can maintain a high removal rate under medium concentration conditions. On the 23rd day, the average removal rate of styrene is only 62.31%, which indicates that the purification effect of the system for the treatment of high concentration styrene needs to be improved. Therefore, the appropriate inlet concentration of styrene is 200-400 mg/m$^3$.

3.2 Relation between inlet load and removal load
The removal capacity refers to the pollutant degradation amount of per unit volume of filler in unit time, which is an important parameter to evaluate the degradation performance of the reactor. In this experiment, the styrene removal capacity of the reactor is studied under the condition of styrene inlet load of 0-60.00 g/(m$^3$·h). The results are shown in Figure 5.

As can be seen from Figure 5, as the inlet load increases, the removal load increases. When the inlet load is low, the removal load has a linear relationship with the inlet load, and styrene can be removed in almost 100%. However, when the styrene inlet load gradually increases, the curve gradually deviates from the 100% removal curve. When it increases to a certain value, the removal capacity of styrene tends to be stable and no longer increases. At this time, the degradation performance of the bioscrubber reaches the best. The maximum removal capacity of styrene in this reactor is 40.23 g/(m$^3$·h) under the condition of inlet load of 0-60.00 g/(m$^3$·h).

3.3 Single-factor experimental analysis

3.3.1 Effect of the residence time on styrene removal capacity. Gas residence time is the time for gas to pass through the filler layer, which is mainly determined by the gas flow rate. It is a very important control parameter affecting mass transfer during the operation of the experiment. Therefore, under the condition of controlling the mass concentration of inlet air as 400 mg/m$^3$, pH as 7, and gas-liquid ratio as 4, the gas flow rate is selected as 100 L/h, 67 L/h, and 50 L/h respectively, and the corresponding residence time is 25.0 s, 37.5 s and 50.0 s to study the effect of residence time on styrene removal rate.
After continuous operation for 6 days under the conditions of 25.0 s, 37.5 s and 50.0 s respectively, the results are shown in Figure 6.

![Figure 6. Effect of the residence time on styrene removal rate.](image)

![Figure 7. Effect of circulating liquid pH on styrene removal rate.](image)

It can be seen from Figure 6 that the shorter the residence time, the lower the styrene removal rate. This is because when the inlet concentration of styrene is the same, the shorter the residence time is, the larger the inlet load is. Because of the short contact time between the waste gas and the filler, the limited gas-liquid mass transfer results in the decrease of styrene degradation rate. The average removal rate of styrene is 72.16%, 84.64% and 85.33% when the residence time is 25.0 s, 37.5 s and 50.0 s, respectively. When the residence time decreases from 37.5 s to 25.0 s, the removal rate decreases obviously, but it is stable; when the residence time is 50.0 s and 37.5 s, the removal rate has little difference. In order to increase the total amount of waste gas removed, the best residence time of the bioscrubber is 37.5 s.

3.3.2 Effect of circulating liquid pH on styrene removal capacity. The pH of circulating liquid directly affects the activity of microorganism, and then affects the purification efficiency of bioscrubber. Under the condition that the mass concentration of inlet styrene is 400 mg/m³, the residence time is 37.5 s, and the gas-liquid ratio is 4, the effect of circulating liquid pH on the styrene removal rate is shown in Figure 7.

It can be seen from Figure 7 that the removal efficiency is better when the pH of circulating liquid is about 7. When the pH is about 5, the removal efficiency is reduced to 72.88%. When the pH is about 9, the removal efficiency is still down to 81.79%. Therefore, the best pH of circulating liquid is 7.

3.3.3 Effect of gas-liquid ratio on styrene removal capacity. Gas-liquid ratio is an important factor affecting the performance of bioscrubber. It refers to the volume of styrene waste gas absorbed by circulating liquid per unit, which reflects the driving force and absorption rate in the absorption process. The main function of the circulating liquid is to wet the biofilm on the surface of the filler, which facilitates absorption and degradation of styrene and provides necessary water and nutrients for the life activities of microorganisms. In the experiment, the mass concentration of inlet air is 400 mg/m³, pH is 7, and residence time is 37.5 s. The spray amount of liquid is changed to investigate the relationship between the gas-liquid ratio and styrene removal rate. See Figure 8 for the experimental results.

![Figure 8. Effect of gas liquid ratio on styrene removal rate.](image)

It can be seen from Figure 8 that when the air inlet concentration is fixed, changing the gas-liquid
ratio of the bioscrubber has no significant effect on the purification efficiency. When the spray amount of liquid is insufficient, due to the decrease of humidity in the bioscrubber, the normal life activities of microorganisms are affected, resulting in the decrease of removal rate. In addition, the performance of the bioscrubber will be slightly reduced by increasing the spray amount of liquid, because styrene belongs to volatile organic compounds and is difficult to dissolve in water. When the spray amount is too large, the contact between styrene gas and biofilm is hindered, resulting in the decrease of removal rate. Therefore, the best gas-liquid ratio is 4.

3.4 Response surface experimental optimization

3.4.1 Design and results of response surface experiment. According to the central composite design principle of Box-Behnken, the pH, residence time and gas-liquid ratio of circulating liquid are selected as independent variables, and the factor level design is shown in Table 1. Take the styrene removal rate as the response value to optimize the degradation effect, and the results are shown in Table 2.

Table 1. Box-Behnken test design factor level and coding table.

| Factor             | Level |
|--------------------|-------|
|                    | -1    | 0   | 1   |
| circulating liquid pH | 5    | 7   | 9   |
| EBRT/s             | 25.0  | 37.5 | 50.0 |
| gas liquid ratio   | 3     | 4   | 5   |

Table 2. Box-Behnken test design and result.

| Test number | Factor | Removal rate/% |
|-------------|--------|----------------|
|             | A      | B   | C   |
| 1           | 0      | 1   | -1  | 83.42 |
| 2           | 0      | 0   | 0   | 84.95 |
| 3           | 0      | 0   | 0   | 85.78 |
| 4           | 1      | 0   | 1   | 79.27 |
| 5           | 0      | -1  | -1  | 70.48 |
| 6           | 1      | -1  | 0   | 70.46 |
| 7           | 0      | 0   | 0   | 88.61 |
| 8           | 0      | -1  | 1   | 71.24 |
| 9           | 0      | 0   | 0   | 87.62 |
| 10          | -1     | 0   | 1   | 70.31 |
| 11          | 1      | 0   | -1  | 80.57 |
| 12          | -1     | -1  | 0   | 60.46 |
| 13          | 0      | 1   | 1   | 82.43 |
| 14          | 1      | 1   | 0   | 82.16 |
| 15          | -1     | 0   | -1  | 67.43 |
| 16          | -1     | 1   | 0   | 71.45 |
| 17          | 0      | 0   | 0   | 86.47 |

3.4.2 Establishment and test of regression model. By fitting the data in Table 2, the quadratic polynomial regression equation is obtained as

\[ Y = 86.69 + 5.35A + 5.85B + 0.17C + 0.18AB - 1.04AC - 0.44BC - 9.03A^2 - 6.53B^2 - 3.27C^2. \]

The analysis of variance of quadratic regression equation is shown in Table 3.

Table 3. Analysis of variance for quadratic regression equations.

| Source | Sum of | df | Mean Square | F Value | p-value |
|--------|--------|----|-------------|---------|---------|
| Model  | 1130.87| 9  | 125.65      | 93.92   | <0.0001 ** |
A 229.09 1 229.09 171.22 <0.0001 **
B 274.01 1 274.01 204.80 <0.0001 **
C 0.23 1 0.23 0.17 0.6922
AB 0.13 1 0.13 0.094 0.7678
AC 4.37 1 4.37 3.26 0.1137
BC 0.77 1 0.77 0.57 0.4741
A² 342.99 1 342.99 256.36 <0.0001 **
B² 179.43 1 179.43 134.11 <0.0001 **
C² 44.90 1 44.90 33.56 0.0007 **
Residual 9.37 7 1.34
Lack of Fit 0.91 3 0.30 0.14 0.9287
Pure Error 8.46 4 2.11
Cor Total 1140.24 16

Note: **P<0.01, extremely significant difference; *P<0.05, significant difference.

In the analysis of variance of regression equation, the significance of independent variables on response value is determined by F-test. The smaller the probability p-value is, the better the significance of independent variables is. It can be seen from Table 3 that the p-value of the model is less than 0.0001, P<0.05, which indicates that the model has high significance, and the lack of fit item is 0.9287>0.05, which indicates that the model is not lack of fit and can be used for the analysis and prediction of the performance of styrene degradation. The model has reached an extremely significant level, the adjustment coefficient R²=0.9918, so the model can explain 99.18% change of response value. The correction coefficient is R² adj=0.9812, which can be seen that the fitting degree of the model is relatively good, so it can be used for prediction within the design range. Therefore, the order of factors affecting the removal rate of styrene waste gas is: residence time>pH of circulating liquid>gas-liquid ratio.

3.4.3 Analysis of response surface graph and optimization of optimal conditions. The three-dimensional response surface graph can directly reflect the interaction of various factors and the influence on the response value. The three-dimensional stereogram of the quadratic regression equation of the interaction of various factors is shown in Figure 9.

![Response surface graph analysis](image)

(a) circulating liquid pH and EBRT  (b) circulating liquid pH and gas-liquid ratio

(c) EBRT and gas-liquid ratio

Figure 9. Response surface graph analysis.
According to the optimization experiment, in the selected experimental range, the best process conditions for the purification of styrene waste gas by the multi-sided bioscrubber are: the residence time is 43.18 s, the pH of the circulating liquid is 7.61, and the gas-liquid ratio is 3.95. Under this condition, the theoretical removal rate is 88.82%, and the result of three parallel tests on the theoretical value is 88.52%, which is close to the theoretical value itself, indicating that the optimization result of response surface analysis is reliable.

4. Effects of different fillers on the performance of bioreactors

The filler is the basic component of gas-liquid contact and mass transfer in the bioscrubber. The liquid flows from top to bottom on the filler surface. The gas flows in a continuous phase from bottom to top, reversely with the liquid, carrying out mass transfer and heat transfer between gas and liquid. Fillers have a great influence on the performance of VOCs degradation in bioreactor. Table 4 lists the filler types, heights and treatment effects of different bioreactors with different structures for treatment of different target pollutants.

Table 4. The effects of different fillers on the performance of bioreactors have been reported.

| configuration of reactor | Targeted pollutant | Filler type | Filler height or volume | Removal load /g • m⁻³ • h⁻¹ | Removal rate /% | reference | Years |
|-------------------------|--------------------|-------------|-------------------------|-------------------------------|----------------|-----------|-------|
| biotrickling filter     | styrene            | The spherical ceramsite | 6.4L | 30 | 90 | 1 | 2006 |
|                         | styrene            | the mycelium pyrolytic carbon | 300mm 2.4L | 153.1 | 95 | 4 | 2013 |
|                         | styrene            | Pall ring and polyurethane foam filler | 1800mm | 2 | 92 | 12 | 2012 |
|                         | styrene            | Polyhedral hollow spheres with activated carbon fibers | 800nm | 136.4 | 90.1 | 14 | 2015 |
|                         | methyl tert-butyl ether | Polyurethane pellet | 300mm | 13.47 | 75 | 18 | 2019 |
|                         | H₂S                | the loaded activated carbon light ceramic corrugated filler | 950mm | 25 | 99 | 19 | 2018 |
|                         | phenol             | Polyhedral ball | 1000mm | 30 | 97 | 15 | 2004 |
| bioscrubber             | benzene            | the multi-sided hollow sphere filler | 300mm | 27.99 | 94.15 | 16 | 2019 |
|                         | styrene            | plastic step ring | 1000mm | ------ | 75 | 17 | 2016 |
|                         | styrene            | inert filler | ------ | ------ | 64.8 | 20 | 2008 |

It can be seen from the above table that different filler types can affect the removal effect of bioreactor. In the biotrickling filter, the biological suspension modified filler, the loaded activated carbon light ceramic corrugated filler and the mycelium pyrolytic carbon have good adsorption performance and high removal efficiency. The mycelium pyrolytic carbon is porous structure, with abundant and concentrated micro and mesoporous pores with uniform pore diameter in the interior and surface, and
the mass density is small, which greatly increases the specific surface area of mycelium pyrolytic carbon and results in strong adsorption performance, so its removal rate can reach more than 95%. For the bioscrubber, the filler used for styrene removal includes plastic step ring and inert filler, and the removal rate is less than 80%, which is not as efficient as the multi-sided hollow sphere filler used in this experiment. Therefore, in the actual operation process, the appropriate filler type should be selected according to the concentration and flow of waste gas and purification requirements to be achieved.

5. Conclusions
(1) Under the conditions of residence time of 37.5 s, pH of 7 and gas-liquid ratio of 4, when the inlet load of styrene is 200-400 mg/m³, the bioscrubber can reach an average removal rate of 86.65% of styrene waste gas, so the appropriate inlet concentration of styrene is 200-400 mg/m³.
(2) As the inlet load increases, the removal load increases. When the inlet load is low, there is a linear relationship between the removal load and the inlet load. However, when the inlet load of styrene continues to increase, the removal capacity of styrene tends to be stable and will not increase any more. The maximum removal capacity of styrene in this reactor is 40.23 g/(m³·h) with the inlet load of 0-60.00 g/(m³·h).
(3) On the basis of single factor test, the optimal process conditions of purification of styrene waste gas by the bioscrubber optimized by response surface method are as follows: the residence time is 43.18 s, the pH of circulating liquid is 7.61, and the gas-liquid ratio is 3.95. Under this condition, the result of verification experiment is 88.52%, which is close to the predicted value, so the optimization result of response surface analysis is reliable.
(4) In the actual operation process, the appropriate filler should be designed according to the waste gas concentration and purification requirements.

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