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Application of extraction and adsorption to the alkylpyrazine removal from wastewater

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ABSTRACT

Application of extraction and adsorption process to the alkylpyrazine removal from wastewater was investigated. The detailed extraction process was studied. It was found that the alkylpyrazine content in the water phase after extraction was decreased to 0.3% or less from about 3.0% (wt.). Extractant was recovered by concentrating the water phase and then the wastewater generated from concentration process was disposed by adsorption treatment in order to further lower the level of alkylpyrazine in wastewater. Adsorption of simulated wastewater containing 2,3-dimethylpyrazine on the different resins was also investigated. Among the resins of various grades used, the XDA-200 had the best properties in decontamination of aqueous 2,3-dimethylpyrazine solutions. The chemical oxygen demand (COD) of authentic wastewater generated from extraction process after adsorption treatment with XDA-200 was less than 1,000 mg/L and its removal percentage was above 90%. It was shown that regeneration of the adsorbent bed could be effectively performed with ethanol. Based on the extraction and adsorption experimental results, more than 98% of COD in the wastewater was removed.

Keywords: Alkylpyrazine; Wastewater treatment; Extraction; Adsorption

1. Introduction

Pyrazine derivatives are well known and important two-nitrogen-containing six-membered ring aromatic heterocyclic compounds. Nitrogen-containing compounds serve as important intermediates for the manufacture of fine chemicals. Recently, a large number of alkylpyrazine derivatives have been found to possess diverse pharmacological properties, which has caused an increasing interest by researchers in this area [1–4].

Nowadays, the direct amination of ethanolamine using ammonia is a new technology for co-production of ethylenediamine, piperazine and triethylenediamine [5–7]. The process has been successfully developed in Xi'an Modern Chemistry Research Institute. However, the amination process is also a source of wastewater. Depending on the process conditions up to 0.6 t liquid wastes is generated per ton of the ethylenediamine, piperazine and triethylenediamine. The wastewater contains 2–3% alkylpyrazine along with chemical oxygen demand (COD) as high as 60,000 mg/L. Taking into account the refractory and pungent

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character of the alkylpyrazine, the importance of decontamination of these effluents is obvious [8].

The conventional treatment of the high concentration organic wastewater mainly includes advanced oxidation process [9–12], chemical and electrochemical techniques [13–15], extraction process [16], adsorption procedures [17–21] and biological treatment [22,23]. Amongst all the treatments proposed, adsorption using sorbents is one of the most popular methods since proper design of the adsorption process will produce high-quality treated effluents. It is now recognized as an effective, efficient and economic method for water decontamination applications. However, the adsorption process exhibits some essential disadvantages, ex. the high organic matter content in wastewater directed to the adsorption step involves either a frequent regeneration of the adsorbent bed or the demand of its high capacity.

Therefore, the present work aimed at developing a hybrid process (extraction–adsorption) for the treatment of effluents from the amination process of ethanolamine and ammonia. Extraction technique would allow removal of a considerable part of the organic pollutants, then adsorption treatment would further lower the alkylpyrazine concentration of the treated effluent to the level acceptable by wastewater treatment plant. Solvent extraction is a commonly used method for separation and enrichment of pyrazine compounds [24,25]. The solvent, such as hexane, ether, benzene, methylene chloride, chloroform, etc. were mainly used in the extraction process, but these solvents were easy to loss and would cause secondary pollution. Ren et al. [26] evaluated the extraction efficiency of sodium hydroxide solution as extractant in the separation of 2-methyl pyrazine and water azeotropic system. Results show that the content of 2-methyl pyrazine is increased to 96.1% from 45.4% after extraction, while sodium hydroxide solution could be easily recovered and reused. However, in this case study, the extraction efficiency for lower 2-methyl pyrazine concentration or other alkylpyrazine was not investigated. In this paper, we describe the results of our studies on the extraction removal of alkylpyrazine using sodium hydroxide solution and followed by alkylpyrazine adsorption on polymeric resins.

2. Material and methods

2.1. Material

Deionized water was used for preparation of all solutions and for dilution of wastewater samples. Methanol, ethanol, 2,3-dimethylpyrazine and sodium hydroxide were of analytical quality. All wastewater were obtained from the 200 t/a ethylenediamine pilot process. The chemical constitution in wastewater was determined by gas chromatograph/mass spectroscopy (GC/MS). For the main composition of wastewater see Table 1.

Table 1
The main composition of wastewater

Composition	Contents (wt.%)
Pyrazine	9.7
2-methylpyrazine	19.5
2-ethylpyrazine	5.7
2-methyl-3-ethylpyrazine	29.7
2,3-dimethylpyrazine	12.3
2-methyl-3-propylpyrazine	12.3
2-butyl-3-methylpyrazine	1.3

The resins, XDA-200, HPD-950 and D113, were applied in adsorption process and their characteristics are compiled in Table 2.

Table 2
The properties of the adsorbent resins

	Resin type		
	XDA-200	HPD-950	D113
Matrix	St-DVB	St-DVB	AA
Specific surface (m^2/g)	887.69	1108.51	–
Pore volume (cm^3/g)	0.51	0.31	–
Pore size (nm)	36.16	85.67	–
Moisture content (%)	57.2	54.3	50.1
Manufacture	Sunresin	Bon co.	Sunresin

2.2. Experimental

2.2.1. Extraction experiments

Based on the above, sodium hydroxide solution was used as extractant for wastewater in extraction process. Extraction experiments were performed by mixing wastewater and sodium hydroxide solution at stirring condition. The mixtures were stirred continuously at 20–25°C for 30 min or more to reach equilibrium and transferred into separation funnel standing for 2 h to obtain the organic phase and water phase. Then, the water phase was concentrated to recover the sodium hydroxide solution. The content of alkylpyrazine in investigated mixtures was determined by using gas chromatography. GC-2010 gas chromatograph equipped with CAM-5 packed column and a thermal conductivity detector (TCD) was used. GC-2014 gas chromatograph equipped with DB-5 packed column and a flame ionization detector (FID) was also used. Each sample was diluted by ethanol. The values determined by TCD were corrected by FID according to the content of ethanol in the organic phase of each sample.

2.2.2. Adsorption experiments

The resins, XDA-200, HPD-950 and D113 were applied in adsorption experiments for simulated wastewater containing 2,3-dimethylpyrazine. Prior to use, all the adsorbent resins were pretreated. All the adsorption experiments were carried out under the dynamic conditions using column method. Among resins mentioned above, that with good properties was applied in adsorption experiments of wastewater generated from extraction process. Alkylpyrazine content in investigated mixtures was determined by using gas chromatography followed as the method above. COD was also used as the index for evaluating the treatment efficiency of wastewater in this study, which was measured by the potassium dichromate oxidation method.

The experimental apparatus for the column adsorption test is shown in Fig. 1. The adsorption column was glass tube of 1.2 cm I.D. and 28.5 cm high. It was equipped with a water jacket for temperature control. The adsorption column was filled with the resins (different particle size fractions) to an adsorption bed volume (BV) of 25 cm^3. It was fed with model solution or wastewater at different flow rates (1–10 BV/h). The effluent COD was regularly measured. Regeneration solution was applied fair current. For regeneration of XDA-200, methanol or ethanol was used. After regeneration the sorbent was rinsed using deionized water.

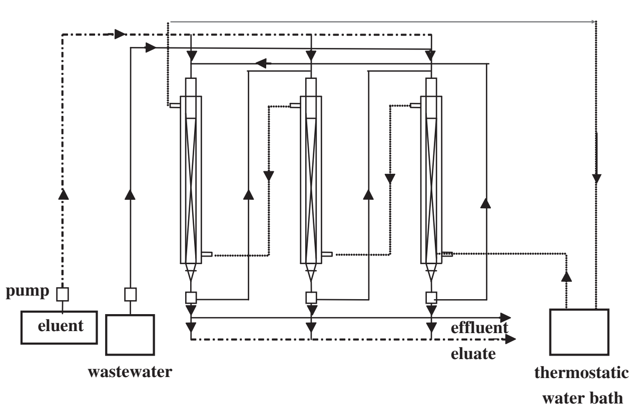

Fig. 1. Experimental apparatus for column adsorption.

3. Results and discussion

3.1. Extraction

Extraction temperature, extraction ratio, concentration of sodium hydroxide solution and concentration of wastewater are the main process conditions that affect the extraction operation. In this section, we investigate how these conditions work on the alkylpyrazine extraction and removal process. The extraction results for wastewater containing alkylpyrazine were shown in Figs. 2–4 and Table 3.

With the interaction of hydrogen bonds between alkylpyrazine and water, extraction at high temperature is preferred, since the interaction of hydrogen

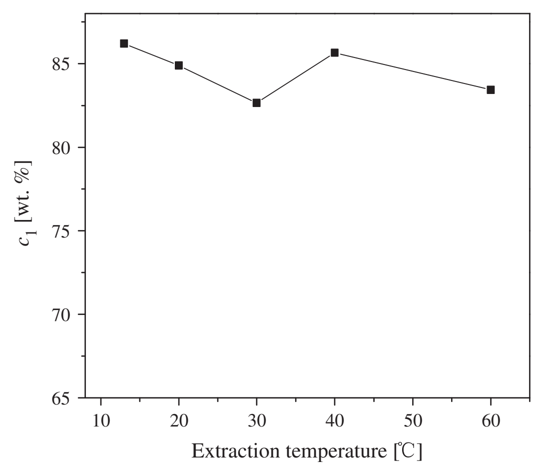

Fig. 2. The effect of extraction temperature for extraction ($c_0 = 3.9\%$, $R = 1$, $c_E = 40\%$).

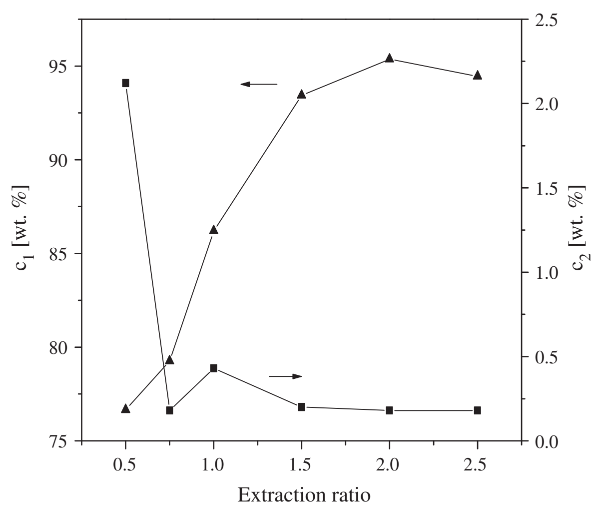

Fig. 3. The effect of extraction ratio for extraction ($c_0 = 3.9\%$, $T = 20$℃, $c_E = 40\%$).

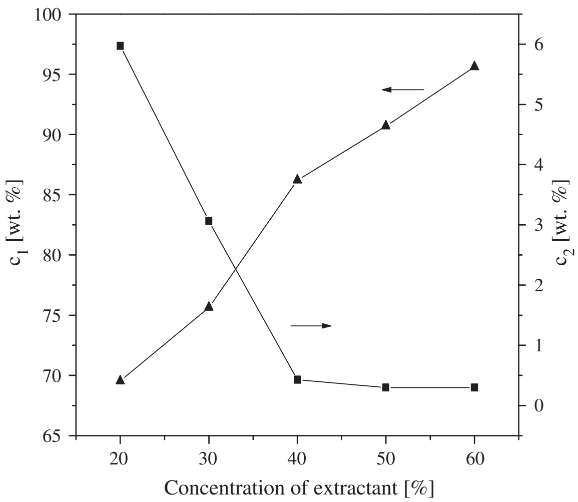

Fig. 4. The effect of the concentration of extractant for extraction ($c_0 = 3.9\%$, $R = 1$, $T = 20$°C).

Table 3
The effect of wastewater concentration for extraction process

c_0 (wt.%)	Content of AP after extraction		Removal percentage of AP (%)
	c_1 (wt.%)	c_2 (wt.%)	
2.4	86.0	0.3	75.0
3.9	85.4	0.3	84.6
6.0	84.9	0.3	90.0
10.7	86.2	0.4	92.5

Extraction process conditions: $c_E = 40\%$ (wt.) $T = 25$°C; $R = 1$.

bonds is much weaker at high temperature. However, it is found from our experiments that the effect of temperature on the extraction performance of sodium hydroxide solution is limited when the temperature ranges from 13 to 60°C, as shown in Fig. 2. In consideration of industrial cooling problems, the extraction temperature is set between 40 and 60°C.

In the extraction process, extraction ratio and concentration of sodium hydroxide solution are two important factors that influence the extraction efficiency and the concentration of alkylpyrazine in the water phase. Fig. 3 shows the influences of extraction ratio for extraction process. To control the concentration of total alkylpyrazine about 0.4% (wt.) in the water phase, the extraction ratio is best to be set at 1–1.5. The effect of concentration of sodium hydroxide solution on extraction process is shown in Fig. 4 that the performance of alkylpyrazine removal enhanced as the concentration of sodium hydroxide solution increases. To ensure the alkylpyrazine removal performance, it is proposed that the concentration of sodium hydroxide solution should be set 40% (wt.) or more.

Furthermore, considering the wastewater concentration fluctuations, the effect of wastewater concentration for extraction was also investigated. The results were presented in Table 3. It showed that the influences of wastewater concentration could be ignored. However, the removal percentage of alkylpyrazine will be lower with the decrease of the concentration of wastewater. Therefore, the extraction process for removal of alkylpyrazine is much more suitable for higher concentration wastewater.

For the recovery of sodium hydroxide solution, distillation is a mature technique in which the water phase is concentrated to recover the 40% (wt.) sodium hydroxide solution. However, the water from the concentration process still contains about 0.6% (wt.) alkylpyrazine. Application of adsorption would be much more efficient for removal of alkylpyrazine in this concentration range.

3.2. Adsorption

3.2.1 Adsorption of simulated wastewater

The simulated wastewater containing 2,3-dimethylpyrazine which is the typical representative component in wastewater was applied to investigate the adsorption properties of different adsorbent resins. Sorption curves of 2,3-dimethylpyrazine on the resins plotted in Fig. 5 enabled the comparison of

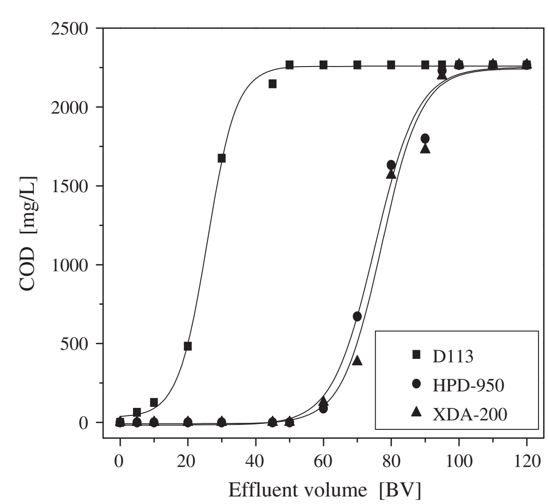

Fig. 5. Adsorption properties of different resin for 2,3-dimethylpyrazine ($c_0 = 0.1\%$, $T = 25$°C, flow rate: 4 BV/h).

Table 4
The COD of effluent at different adsorption conditions

Ex.	Adsorption temperature (℃)	Flow rate (BV/h)	COD (mg/L)	Removal percentage of COD (%)
1	10	1.5	220	99.0
2	22	1.8	312	98.6
3	28	3.1	720	96.7
4	38	3.3	1,100	95.1

The COD of the simulated wastewater is about 22,000 mg/L, the volume of the treated wastewater is 9 BV.

properties of various adsorbents and the choice of the most efficient one. The XDA-200 and HPD-950 resins made of styrene–divinylbenzene copolymer showed higher affinity to 2,3-dimethylpyrazine than the D113 one with methyl acrylate–divinylbenzene copolymer matrix. The poly (styrene–divinylbenzene) resins were known as efficient adsorbent for adsorption of various organic compounds, among them aromatic ones. Taking into account the results concerning 2,3-dimethylpyrazine adsorption on various grades resins, further adsorption experiments under dynamic conditions for simulated wastewater or authentic wastewater were carried out using XAD-200 resin alone.

The experiments were carried out with 1% (wt.) 2,3-dimethylpyrazine simulated wastewater, at different adsorption temperature and flowing rates. Table 4 presents the effect of different temperature and flowing rates on COD removal efficiency. It can be seen that the removal percentage of COD is still no less than 95% when the temperature of the simulated wastewater increased to 38℃, the flowing rates increased to 3.0 BV/h.

3.2.2 Wastewaters

The wastewater from the concentration process of sodium hydroxide solution contains about 0.6% (wt.) alkylpyrazine and the COD of this wastewater is up to 14,400 mg/L. Adsorption for this wastewater was performed in order to investigate the properties of the chosen adsorbent XDA-200 during adsorption and regeneration steps.

The COD curve for the adsorption step made it possible to estimate the volume of eluate up to breakthrough point. The COD curves obtained at 25℃ with different process conditions are presented in Figs. 6 and 7. It was found that the COD of effluents increased with the increase of effluent volume and flow rates, the removal rate of COD is only 50% when the effluent volume is 9 BV. In order to improve the removal efficiency, two-step adsorption process was used in the following experiments. Fig. 7 shows the treatment effect of the different adsorption methods. The results showed that the COD of the effluents after two-step adsorption process is only 1,000 mg/L when the effluent volume is 9 BV. The removal percentage of COD is above 90%.

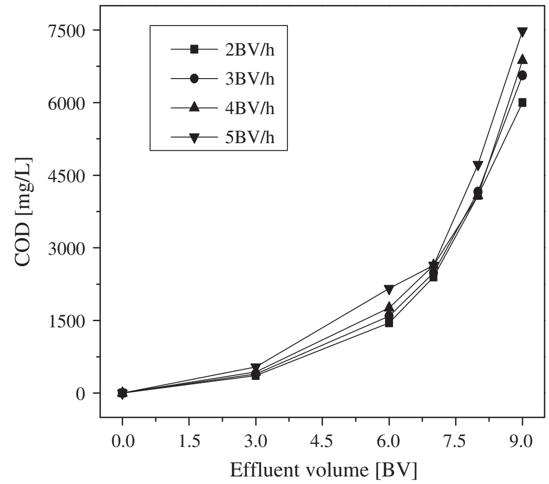

Fig. 6. The effect of flow rates for adsorption ($c_0 = 0.6\%$, $T = 25$℃, flow rate: 4 BV/h).

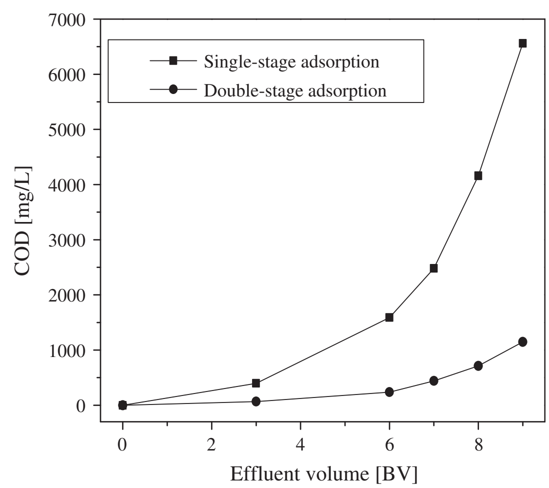

Fig. 7. The treatment effect of different adsorption methods ($c_0 = 0.6\%$, $T = 25$℃, flow rate: 4 BV/h).

The regeneration curves obtained at 1.5 BV/h elution flow rates are presented in Fig. 8. Fig. 8 shows that the regeneration step could be carried out efficiently with ethanol or methanol. However, the content of alkylpyrazine seen from Fig. 8 was determined by gas chromatography equipped with FID, so the

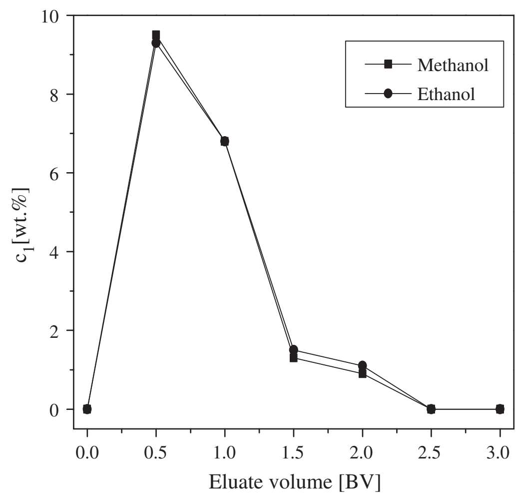

Fig. 8. Elution effect of different eluent (regenerant: methanol, ethanol, flow rate: 1.5 BV/h).

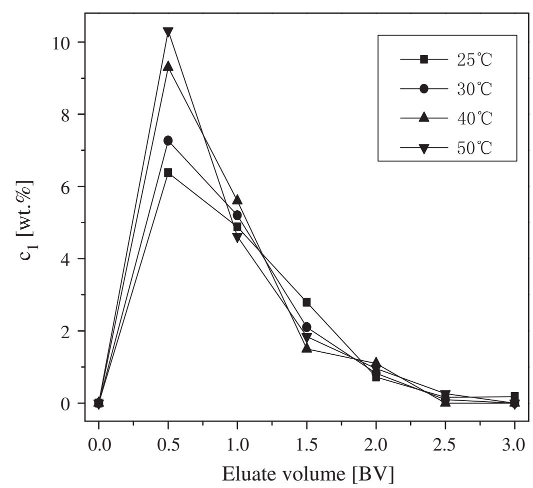

Fig. 10. The effect of temperature for desorption (regenerant: ethanol, flow rate: 1.5 BV/h).

content of water in eluent was not known. In order to obtain the regeneration efficiency curve of the XDA resin with ethanol, concentration of alkylpyrazine in the accumulative eluates at different eluate volume was determined by gas chromatography equipped with TCD. It was seen from Fig. 9, that about 2.5 BV ethanol as the eluant could nearly regenerate the XDA resin bed completely.

In addition, the regeneration process could also be carried out efficiently with hydrochloric acid solution due to the chemical reaction between alkylpyrazine and hydrochloric acid. Taking into account the simplicity of the process, ethanol was selected as the eluent to regenerate the adsorbent bed.

The regeneration curves obtained at different temperatures are shown in Fig. 10. Elution effect was enhanced with increase in temperature, but the optimized temperature is 40℃ because the ethanol will lose greatly at higher temperature.

Moreover, adsorption–regeneration cycle experiments were performed in order to investigate the properties of the XDA-200 resin after processing multiple batches of wastewater. The COD and its removal percentage of the wastewater after adsorption at different batches are shown in Fig. 11. The results showed that the COD of different batches treated wastewater were less than 1,000 mg/L and their removal percentages were above 95%.

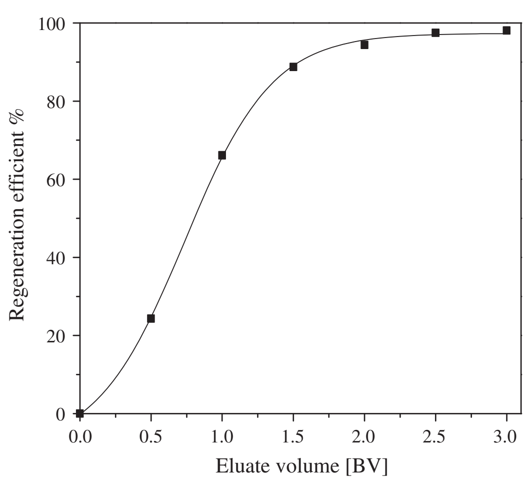

Fig. 9. Regeneration efficiency curve of the XDA resin with ethanol (regenerant: ethanol, flow rate: 1.5 BV/h).

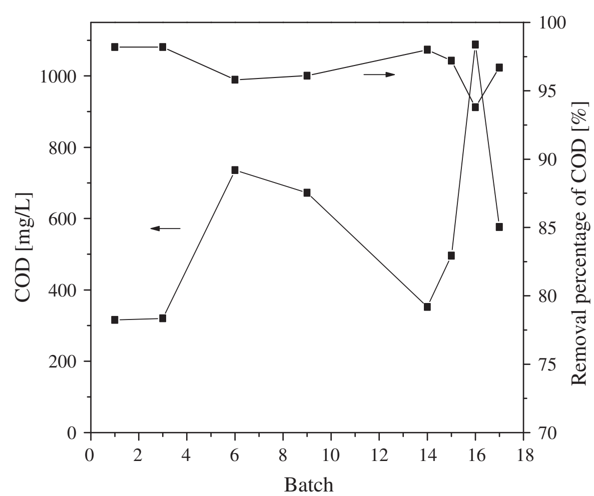

Fig. 11. The values of COD of different batches of treated water ($c_0 = 0.6\%$, flow rate: 4 BV/h, $T = 25$℃).

Table 5
The performance of XDA-200 resin before and after used

	Specific surface (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)	Moisture content (%)
Before	887.69	0.51	36.16	57.4
After	812.43	0.65	45.22	58.3

Finally, the structure performance of the resin after 17 batches adsorption–regeneration was characterized and compared with the original resin. It is seen from Table 5, that the structure performance has no significant changes. Based on the above results, we can draw a conclusion that the XDA-200 resin is really a good adsorbent for alkylpyrazine removal from wastewater.

In addition, the ethanol was recovered by distillation of the regenerations, while the 10% (wt.) alkylpyrazine solution was obtained.

3.3. Proposed process

Extraction and adsorption results obtained with alkylpyrazine wastewater suggest that the hybrid system could be used for efficient decontamination of the effluents from the amination process of ethanolamine and ammonia. The scheme of such hybrid process is presented in Fig. 12.

The details of the proposal process are described as the following: the alkylpyrazine wastewater is extracted with 40% (wt.) sodium hydroxide solution, while the extraction ratio is 1. Then, 85% (wt.) alkylpyrazine is obtained by split-phase operation. The water phase is concentrated to recover the 40% (wt.) sodium hydroxide solution, while wastewater from the concentration process is further treated by adsorption process. Adsorption process is performed with adsorption column filled with the resins to an adsorption BV of 0.6 m^3. After treating 4.8 m^3 of wastewater, the adsorption column is regenerated with 1.8 m^3 of ethanol. Finally, the ethanol is recovered by distillation of the regenerations.

Table 6 shows the operating cost evaluation of the proposed process. Because the ethanol and 40% (wt.) sodium hydroxide solution can be used repeatedly, so the main costs are from the recovery process of ethanol and 40% (wt.) sodium hydroxide solution. The operation cost for 1 m^3 wastewater treatment was about 74.2 yuan, while the economic return from the alkylpyrazines was 71.4 yuan. They were approximately balanced in economic benefit.

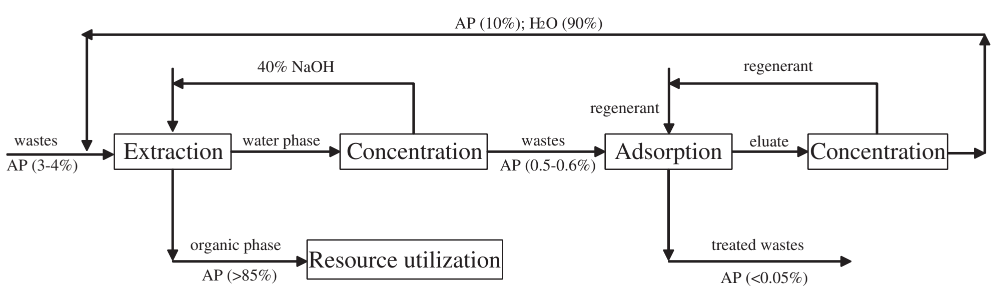

Fig. 12. The proposed hybrid extraction–adsorption process for treatment of wastewaters containing alkylpyrazine (AP).

Table 6
Operation cost evaluation for 1 m^3 wastewater treatment

Item		Consumption[a]	Unit price[b] (yuan)	Operation cost (yuan)
Steam, 0.5 Mpa (t)	40%(wt.) NaOH Ethanol	0.618	120	74.2
Alkylpyrazine[c] (kg)		−34	2.1	−71.4
Sum		–	–	2.8

[a]Consumption was estimated from simulation calculation.
[b]Price was estimated by the plant.
[c]Alkylpyrazines were recovered for sales.

4. Conclusions

Extraction experiments proved that the alkylpyrazine could be effectively removed from the wastewater using sodium hydroxide solution as extractant. With the investigation of the extraction process conditions, the optimized alkylpyrazine removal process was developed. The results showed that more than 70% of the alkylpyrazine in the wastewater was recovered, which was a by-product with economic benefit.

The adsorption experiments were carried out under column methods. The best sorption properties were found in the case of the XAD-200. Using this adsorbent, made of cross-linked styrene–divinylbenzene copolymer, the removal of alkylpyrazine from wastewater containing alkylpyrazine (0.6% wt.) was carried out by the column method. Ethanol was used to regenerate the sorbent bed. The removal curves of COD and the regeneration curves were determined. It could be found that above 90% of COD could be removed and the COD of effluents was less than 1,000 mg/L.

With the investigation of the extraction and adsorption process, a hybrid process for the alkylpyrazine removal was developed, which paved a sound pretreatment ground for the depth treatment. However, in order to obtain the biggest economic benefit from the recycling by-products, the resources development of recovered alkylpyrazine remains to be researched in the following work.

Acknowledgement

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List of symbols

BV — volume of resin bed
AP — alkylpyrazine
c_0 — content of AP in wastewater (wt.%)
c_1 — content of AP in organic phase (wt.%)
c_2 — content of AP in water phase (wt.%)
c_E — concentration of sodium hydroxide solution (wt.%)
R — extraction ratio (extractant: water)
T — temperature (℃)

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