

Solvent extraction process development and on-site trial-plant for phenol removal from industrial coal-gasification wastewater

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Abstract

A phenol removal process was developed for the coal-gasification wastewater. Based on extraction principles and experimental results, an extracting solvent was selected in consideration of phenol removal, solvent recovery and COD removal for the coal-gasification wastewater. The extraction process conditions were studied, and a flowsheet for phenol removal was proposed. An on-site trial-plant of 2 t/h wastewater was set up for testing and industrial verification. The results of the on-site trials showed that more than 93% of the phenols and 80% of COD in the wastewater were removed. The operating cost of the proposed process was approximately balanced by the economic return of the recovered phenols.

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1. Introduction

Phenols are toxic organic compounds listed as priority pollutants by the US environmental protection agency (EPA) [1]. They are lethal to fish at low concentrations (5–25 mg/L) and bring objectionable tastes to drinking water [2]. The effluent from the coal-gasification process contains high concentration of phenols. At such high concentration of phenols, e.g. more than 3000 mg/L, microorganisms cannot survive. This makes biological treatment difficult [3]. Furthermore, in the coal-gasification wastewater, non-volatile dihydric and trihydric phenols appear at concentrations of hundreds or even thousands of milligram per liter [4,5]. These dihydric and trihydric phenols are extremely resistant to biological oxidation [4]. Therefore, a pretreatment to remove phenols in the coal-gasification wastewater is particularly crucial.

Solvent extraction is one of the most important techniques used in high phenol-concentrated wastewater pretreatment [6,7]. Through solvent extraction, most phenols in the wastewater can be recovered as a by-product with economic benefit. COD value

of the effluent wastewater can be dramatically reduced. This makes biochemical treatment easier.

A large gas-making plant in northern China uses lignite as a raw material to make coal gas through the Lurgi process. The plant yields 2,420,000 Nm³ gas per day, and provides civil gas for a city with a population of 4 million. In the process of coal-gasification, however, there is an effluent of 100 t/h toxic wastewater from the plant, containing over 5000 mg/L of phenols along with COD as high as 20,000 mg/L. The present work was to develop a phenol removal technique for the wastewater from the plant.

In this work the performance of phenol removal from the wastewater with four alternative solvents, namely diisopropyl ether (DIPE), butyl acetate, methyl isobutyl ketone (MIBK) and 30% tributyl phosphate (TBP)–kerosene were investigated. In the comprehensive consideration of phenol removal, solvent recovery and COD removal, MIBK was selected as the extracting solvent for the coal-gasification wastewater. Then, the extraction process conditions such as pH, temperature, solvent ratio and the number of extraction stages were studied. Finally a phenol removal process was proposed and an on-site trial-plant of 2 t/h wastewater was set up. The results of the on-site trials showed that this proposal process paved a sound pretreatment ground for the biochemical treatment of the coal-gasification wastewater.

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Nomenclature

a_p	specific surface area
c	concentration of volatile phenols
c_o	concentration of total phenols in original wastewater
c_T	concentration of total phenols
d	diameter of column
D	distribution coefficient
g	gravity acceleration
i	number of extraction segment
L_R	flow ratio for two phase
n	number of the extraction stages
Q	energy quantity
R	solvent to water ratio
t	temperature
u_0	intrinsic velocity
u_{df}	flooding velocity of disperse phase
u_{cf}	flooding velocity of continuous phase
u_c	operating velocity of continuous phase
V_c	volume flow of continuous phase
x	concentration of phenols in the water phase
x_F	concentration of phenols in the feed stream
y	concentration of phenols in the solvent phase

Greek letters

α	boundary tension
ε	voidage of packing
ρ_c	density of continuous phase
$\Delta\rho$	density difference of two phase
Φ	diameter
Φ_f	flooding holdup

Table 2

Constituents analysis for the extracted substances

No.	Constituents	Number	Constituents
1	Phenol	15	Catechol
2	2-Propyl phenol	16	2,4-Dimethyl phenol
3	3-Ethyl-5-methyl phenol	17	2,6-Dimethyl phenol
4	2-Ethyl-5-methyl phenol	18	2,5-Dimethyl phenol
5	<i>o</i> -Methyl phenol	19	3,5-Dimethyl phenol
6	<i>m</i> -Methyl phenol	20	2-Methyl hydroquinol
7	<i>p</i> -Methyl phenol	21	Isobutenyl-methyl ketone
8	Hydroquinol	22	1,2-Dimethyl piperidine
9	Resorcinol	23	4-Methyl resorcinol
10	2-Methyl resorcinol	24	5-Methyl resorcinol
11	3-Methyl catechol	25	2-Ethyl phenol
12	4-Methyl catechol	26	3-Ethyl phenol
13	2,2,6,6-Tetramethyl-4-piperidine	27	<i>N</i> -Methyl aniline
14	4-Hydroxyl-4-methyl-2-pentanone	28	α -Naphthol
15	<i>N,N</i> -Diformyl diaminocyclohexane	29	β -Naphthol
14	4-Amino-4-methyl-2-pentanone	30	2-Methyl-1-naphthol

2.2. Experimental procedure

Batch extraction experiments were performed as follows. Mixture of the wastewater and extracting solvent at certain solvent ratio was placed in the thermostatic bath at the proper temperature, violently stirred for 15 min and then left to equilibrate for 2 h before the raffinate was separated. Concentrations of volatile phenols and total phenols of the raffinate were then determined as well as COD value. The solvent dissolved in the raffinate was recovered by simple distillation until its concentration was less than 50 mg/L. The pH of the wastewater was adjusted by addition of H₂SO₄ or NaOH and monitored with a pH meter.

2.3. Analytical methods and apparatus

The concentration of volatile phenols was measured by a 721 model visible UV spectrophotometer at wavelength of 460 nm in the 4-aminoantipyrine spectrometric method (EPA420.1). The concentration of total phenols was determined by the direct bromination method (EPA320.1). In this method, an excessive brominating agent was added directly to the system and it would react with the existing phenols, then the iodometric method (EPA345.1) is used to measure the excessive brominating agent that helped to calculate the total phenols. This direct bromination method was especially appropriate for measuring total phenols in wastewater containing non-volatile phenols. The determination of COD in the wastewater was carried out by the potassium dichromate oxidation method (EPA410.4). The constituents of the extract were analyzed by

2. Experiment and analysis

2.1. Materials

Coal-gasification wastewater was provided from the mentioned plant. Some relevant indices of water quality are shown in Table 1. The constituents of the phenols are especially complicated, as shown in Table 2. DIPE, butyl acetate, MIBK and TBP (chemically pure grade, Shanghai Lingfeng Chemical Reagents Company, China) were used in the laboratory experiments. Kerosene was a commercial product with boiling range from 150 to 250 °C. MIBK (technical grade, Shell Chemical, USA) was used in the on-site trials.

Table 1
Some water quality indices of coal-gasification wastewater

Water quality index	Original wastewater
pH	9.8
Volatile phenols (mg/L)	2750
Non-volatile phenols (mg/L)	2660
Total phenols (mg/L)	5410
COD _{Cr} (mg/L)	21364

a GC/MS-QP2010 Model gas chromatograph/mass spectrograph (Shimad, Japan) with a 30 m × 0.25 mm × 0.25 μm DB-5MS capillary column. The content of the solvent dissolved in the raffinate was analyzed by a GC-6820 Model gas chromatograph (Agilent, USA) equipped with a flame-ionization detector and a 30 m × 0.35 mm × 0.5 μm DB-FFAP capillary column.

3. Selection of solvent

3.1. Distribution coefficient

Many solvents such as benzene, tributyl phosphate and butyl acetate, etc. have been used as solvents for extraction of phenols [5,9]. According to the extraction principles, the performance of a solvent is determined with its distribution coefficient between solvent phase and water phase. The higher the distribution coefficient of an extracting solvent is, the better its performance is. Therefore, the extracting solvent can be cursorily selected by the distribution coefficient. Moreover, as an industrial extracting solvent, the density and other physical properties are very important, together with its cost. Shown in Table 3 are four selected solvents suitable as industrial extracting solvents and their distribution coefficients of phenol [4,8,9].

3.2. Phenol removal

The experimental results on phenol extraction from the coal-gasification wastewater with DIPE, butyl acetate, 30%TBP–kerosene and MIBK are shown in Fig. 1. The perfor-

Table 3
Distribution coefficient of phenol in selected solvent (25 °C)

Solvent	<i>D</i>
DIPE	36.5
Butyl acetate	71
MIBK	100
30% TBP–kerosene	171.9

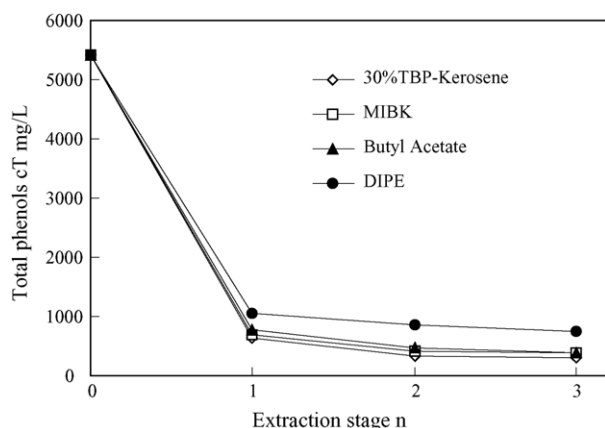


Fig. 1. Phenol removal performance with different solvents from the coal-gasification wastewater.

Table 4
Boiling points and recovery methods of the alternative solvents

Solvent	Boiling point, (°C)	Recovery method
DIPE	68	Distillation
Butyl acetate	126	Distillation
MIBK	116	Distillation
TBP	289	Reverse extraction with alkali
Kerosene	150–250	Reverse extraction with alkali

mance of phenol removal, apart from DIPE, is similarly good. The poor performance of DIPE is attributed to its poor distribution coefficient on the non-volatile dihydric and trihydric phenols, as reported in some research [4]. The results also show that using MIBK, 30%TBP–kerosene or butyl acetate, the concentration of total phenols in the wastewater can be reduced from 5410 mg/L to around 300 mg/L after three stages of batch extraction.

3.3. Solvent recovery

To make solvent recycling possible, solvent recovery has to be studied carefully. Normally, for phenol extraction the low-boiling point solvents can be recovered by distillation, while the high-boiling point solvents can be reversely extracted with alkali. In Table 4 the boiling points [10] and the recovery methods of the four selected solvents are shown.

It is noticed from Table 4 that the boiling points of butyl acetate (126.3 °C) and MIBK (115.9 °C) are much lower than that of phenol (182 °C). They can be easily separated with distillation. The boiling point/boiling range of 30%TBP–kerosene is 289 °C and 150–250 °C, which should be recovered by reverse extraction with alkali rather than distillation. Phenols are Lewis acids whose hydrophilicity will be highly reinforced in alkali solutions. When the solvent rich in phenols is extracted by alkali solution, the phenols will reversely enter into the water phase in the form of phenolic salts, and the solvent may be recovered. Between the two recovery methods, distillation is a mature technique in which the recovered solvent is purer, while reverse extraction is an energy-saving method except that the recovered solvent contains more impurities and the by-product phenols exist in the ionic state. In gas-making plants, distillation is a widely accepted industrial separation operation. Therefore, it is chosen for solvent recovery.

3.4. COD removal

The main objective of the wastewater pretreatment is to recover useful substances from the water, to reduce the COD value in the wastewater, and to relieve the biochemical treatment as much as possible. Thus, in the selection of the solvent, the COD value after extraction is extraordinarily important and has to be concerned.

The wastewater COD values after phenol extraction and solvent recovery with the four solvents are presented in Table 5. It is shown that MIBK is the best solvent among the four candidates in reduction of the effluent's COD value.

Table 5
Wastewater COD_{Cr} after phenol extraction and solvent recovery

Solvent	COD _{Cr} ^a (mg/L)	COD _{Cr} ^b (mg/L)
DIPE	14760	5923
Butyl acetate	12100	4790
MIBK	33420	3744
30%TBP–kerosene	4900	4900

^a The values after extraction.

^b The values after extraction and solvent recovery.

In accordance with the above investigation, MIBK is selected as the extracting solvent for the phenol extraction and removal process from the coal-gasification wastewater after overall performance considerations.

4. Process conditions

pH value, temperature, solvent ratio and the number of extraction stages are the main process conditions that affect the solvent extraction operation. In this section we investigate how these conditions work on the phenol extraction and removal process.

4.1. pH value

For the coal-gasification wastewater, pH value typically lies between 9.0 and 10.5. At such high values of pH a significant fraction of phenols is ionized, further discouraging extraction. In some research [7] it has been found that at pH >8 the distribution coefficient for phenol extracted with MIBK begins to drop, and little phenol can be extracted at pH >12. The effect of pH on extraction of phenols with MIBK from the coal-gasification is investigated. It is shown in Fig. 2 that the performance of phenol removal reduces as the pH value increases. When pH >9, the concentration of volatile phenols in the raffinate increases quickly. To ensure the phenol removal performance, the pH value of the wastewater should be adjusted. To cut down the operating cost, it is proposed that the pH value should be set between 7.5 and 8.5.

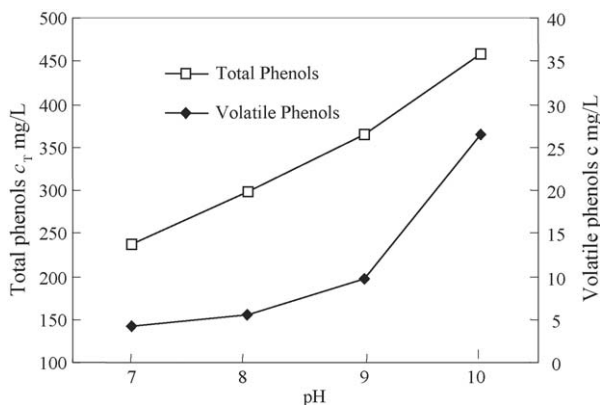


Fig. 2. Effect of pH on extraction of phenols with MIBK ($c_0 = 5410$ mg/L, $R = 1:1$, $n = 1$).

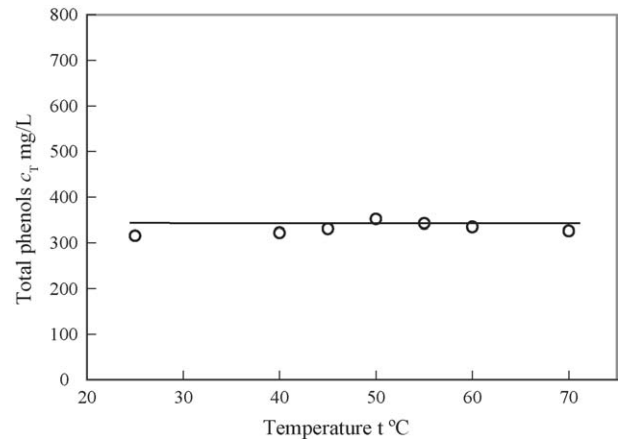


Fig. 3. Effect of temperature on extraction of phenols by MIBK from wastewater ($R = 1:1$, $n = 1$, pH 8.0).

4.2. Temperature

With the interaction of hydrogen bonds between phenols and MIBK, the phenol extraction performance of MIBK is enhanced. Extraction at low temperature is preferred, since the interaction of hydrogen bonds is much stronger at low temperature. However, it is found from our experiments that the effect of temperature on the extraction performance of MIBK is limited when the temperature ranges from 25 °C to 70 °C, as shown in Fig. 3. In consideration of industrial cooling problems, the extraction temperature is set between 40 and 60 °C.

4.3. Solvent ratio R

In the extraction process, the solvent-to-water ratio R is an important process parameter. It influences the number of the extraction stages, the concentration of phenols in the raffinate, and the energy consumption of solvent recovery. Generally speaking, the number of the extraction stages and the concentration of phenols in the raffinate decrease as the solvent ratio increases, while the energy consumption of solvent recovery increases. Thereby, to save energy the solvent ratio should be reasonably small. Shown in Fig. 4 is the depen-

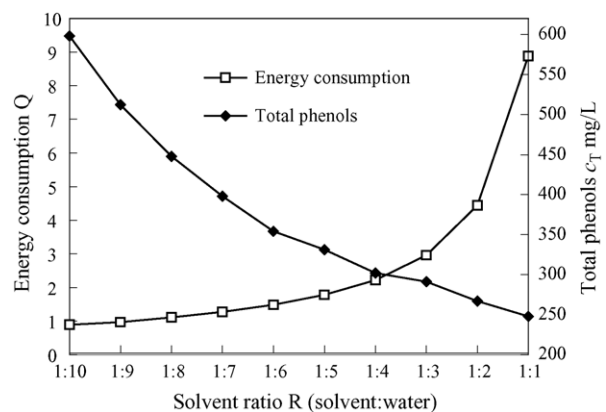


Fig. 4. Solvent ratio on phenol removal and energy consumption of solvent recovery.

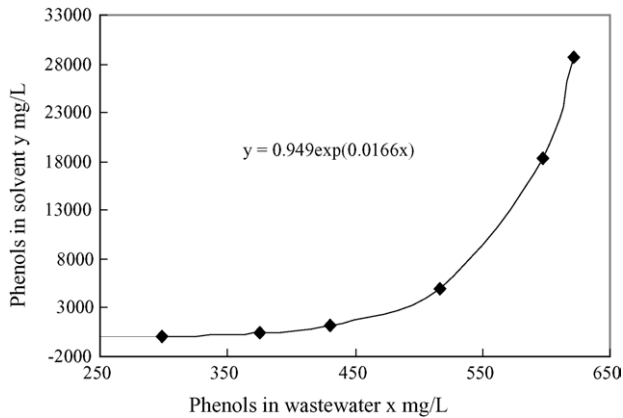


Fig. 5. Phenol extraction equilibrium with MIBK in coal-gasification wastewater (25 °C, pH 8.0).

dence of solvent ratio on extraction effect and energy consumption.

The two curves cross at the solvent ratio 1:4. It is shown that at $R > 1:3$, the energy consumption of solvent recovery increases rapidly, and at $R < 1:6$, the concentration of total phenols in the raffinate gradually increases. To control the concentration of total phenols in about 300 mg/L and save energy, the solvent ratio is best to be set at 1:3–1:6.

4.4. Extraction stage

To determine the number of the extraction stages, the equilibrium data for phenols distribution between MIBK and coal-gasification wastewater is measured. The equilibrium equation is fitted, as shown in Fig. 5.

According to the multi-stage counter-current extraction in non-miscible system, the mass balance equation is as the following:

$$y_{i+1} = \frac{1}{R}x_i + \left(y_i - \frac{1}{R}x_F \right) \quad (1)$$

The fitted extraction equilibrium equation for the wastewater is as follows:

$$y_i = 0.949 \exp(0.0166x_{i+1}) \quad (2)$$

Calculating from Eqs. (1) and (2), when solvent ratio is set at 1:4, at the first stage the concentration of phenols has been reduced to 608 mg/L. After four-stage counter-current extraction the total concentration of phenols in the wastewater could be reduced from 5410 to 329 mg/L.

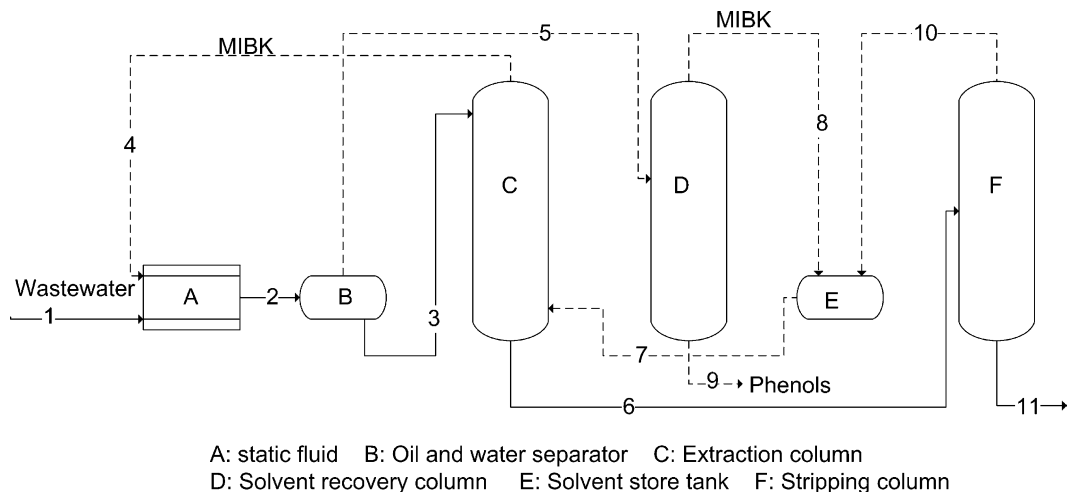
5. Process development and design

5.1. Process development

From the above experiments and analysis, MIBK is selected as the extracting solvent for the treatment of coal-gasification wastewater. The further study of the process conditions indicates that when the pH value between 7.5 and 8.5, temperature ranging from 40 to 60 °C, solvent ratio set at 1:3–1:6, the total concentration of phenols in the wastewater can be reduced from 5410 mg/L to around 300 mg/L, and the volatile phenols less than 10 mg/L after four-stage counter-current extraction. A phenol removal process from wastewater was proposed, as shown in Fig. 6.

In the proposal process, the extraction part consists of a static fluid mixer and an extraction column. As described in above, the concentration of phenols can dramatically reduce from about 5000 to 600 mg/L in the first stage extraction. Here the static fluid mixer and the oil-and-water separator remove most of the phenols and serves as the first stage in the extraction process. The extraction column is operated in counter-current extraction. Distillation is used to separate phenols from the solvent. The recovered solvent is used circularly. And stream stripping is used for recovering the solvent dissolved in the raffinate.

The details of the proposal process are described as the following: The coal-gasification wastewater was drawn from the plant and its pH value was adjusted with H_2SO_4 to 8.0. Together with the extracting solvent overflowing from the extraction column, the wastewater was pumped into the static fluid. Then



A: static fluid B: Oil and water separator C: Extraction column D: Solvent recovery column E: Solvent store tank F: Stripping column

Fig. 6. Flow diagram of the proposal phenol removal process for the coal-gasification wastewater.

the mixture entered an oil–water separator. After separation, the wastewater stream was pumped into the extraction column and extracted with counter-current MIBK coming from the solvent tank. The raffinate was then pumped into the stripping column for the dissolved solvent recovery and then released to the biological treatment unit. The solvent phase overflowing from the extraction column, going through the static fluid mixer and the oil–water separator, was then pumped into the distillation column. The recovered solvent distilled from the top of the column was then used circularly. The separated phenols were collected as a by-product for further refining.

5.2. On-site trial-plant design

An on-site trial-plant of 2 t/h wastewater was set up based on the proposed phenol removal process as shown in Fig. 6. The trial plant was reconstructed partly with the existing installation, while the extraction column, the solvent recovery column and the static fluid mixer was redesigned and fabricated.

Here a Φ 40 mm \times 2 m static fluid mixer and a Φ 500 mm \times 2 m oil-and-water separator were used to serve as the first stage extraction. The residence time was designed for 30 min. The solvent recovery column was reconstructed with the existing distillation column of 400 mm diameter. To meet the specification of separation, the number of theoretical stages for the solvent recovery column should be over 12. It was packed with 10 m high SM-125 corrugate plate packing. The stripping column was also redesigned from an existing distillation column of 300 mm diameter and 10 m high. 10 theoretical distillation stages were designed to recover the solvent in the raffinate. Thus, the packing layer of the stripping column was designed as 7 m high. The extraction column was 10 m high and 300 mm in diameter, packed with 8 m high of FG-II grid packing whose Height of Equivalent Theoretical Plate (HETP) was of 1–2 m. It was expected to function of four extraction stages. The diameter was calculated as 300 mm, with the following equations [11] when the wastewater was 2 t/h and the extraction solvent ratio was 1:4.

$$u_0 = 0.849 \left(\frac{4g\alpha\Delta\rho}{\rho_c^2} \right)^{0.25} \quad (3)$$

$$\phi_f = \frac{2}{3 + \sqrt{1 + 8/L_R}} \quad (4)$$

$$u_{cf} = \varepsilon u_0 (1 - \phi_f)^2 (1 - 2\phi_f) \quad (5)$$

$$u_{df} = L_R u_{cf} \quad (6)$$

$$u_c = 0.5 u_{cf} \quad (7)$$

$$d = \sqrt{\frac{4V_c}{\pi u_c}} \quad (8)$$

The detailed sizes and internals for the main equipment of the trial-plant are shown in Tables 6 and 7.

Table 6
Sizes and internals for main equipment

Equipment	Diameter (mm)	Height (m)	Internals
Static fluid mixer	40	2	–
Oil–water separator	500	2	FG-II grid packing
Extraction column	300	10	FG-II grid packing
Solvent recovery column	400	12	SM-125 corrugate plate packing
Stripping column	300	10	SM-125 corrugate plate packing

Table 7
Characteristics of the internals

Internal	FG-II grid packing	SM-125 corrugate plate packing
Diameter, Φ (mm)	500, 300	400, 300
Voidage, ε (%)	120	125
Specific area, a_p (m ² /m ³)	96	98
HETP (m)	1–2	0.7

Data are provided by the packing manufacturer.

6. Operating results of the on-site trial-plant

The results of the on-site trials are presented in Table 8. It showed that with the treatment of the proposed process, the concentration of phenols in the wastewater could be reduced from 5123 mg/L to around 329 mg/L, and the COD less than 4000 mg/L. With the proposed process, 99.7% of the volatile phenols was eliminated, more than 93% of the total phenols was removed, and about 80% of the COD was removed from the wastewater.

Shown in Table 9 are the data on the performance of the extraction column. The second column of design values were calculated when the extraction column was designed at 300 mm in diameter and 10 m high packed with 8 m FG-II grid packing, while the third column shows practical effects in the trial operation. With the comparison it was found that the HETP of the

Table 8
Comparison of water quality between original and treated wastewater

Water quality index	Original wastewater	Treated wastewater	Percent
pH	9.8	7.8	–
Total phenols (mg/L)	5123	329	93.6
Volatile phenols (mg/L)	2665	8.25	99.7
COD _{Cr} (mg/L)	20644	4070	80.3

Table 9
Comparison of performance for the extraction column between design and trial operation

Performance	Design value	Operating value
Number of stages	4	3
HETP (m)	1–2	2.7
u_{df} (m/s)	0.01608	–
u_{cf} (m/s)	0.00402	–
Holdup (Φ_f)	0.423	–

Table 10
Operation cost evaluation for 1 m³ wastewater treatment

Item	Consumption ^a	Unit price ^b (yuan)	Operation cost (yuan)
H ₂ SO ₄ (kg/h)	0.5	0.6	0.3
Solvent (kg/h)	0.35	18	6.3
Cooling water (t/h)	0.983	0.13	0.13
Steam, 0.5 Mpa (t/h)	0.118	35	4.13
Steam, 2.5 Mpa (t/h)	0.10	40	4.0
Electricity (kw h)	1.19	0.2	0.24
Subtotal	–	–	15.10
Phenols ^c (kg/h)	–5.25	2.8	–14.7
Sum	–	–	0.4

^a Consumption was estimated from the on-site trial.

^b Price was estimated by the plant.

^c Phenols were recovered for sales.

FG-II grid packing was much greater than expected, for the number of stages of the column was around 3 rather than the expected 4. It indicated that in this extraction column the mass transfer performance was not as perfect as it was theoretically calculated. The operating flooding velocity fluctuated in the range of 20% of the designed value. The operating holdup of disperse phase was not determined due to the restriction of the on-site trial-plant.

In Table 10 it shows that the operating cost evaluation of the proposed process. The operation cost for 1 m³ wastewater treatment was about 15.1 yuan, while the economic return from the by-product, phenols was 14.7 yuan. They were approximately balanced in economic benefit.

7. Conclusions

To remove phenols and relieve the biochemical treatment burden, the present work developed a phenol removal process for the coal-gasification wastewater. Based on the theoretical study and experiments, MIBK was selected as the extracting solvent for the treatment of the coal-gasification wastewater of 5000 mg/L phenols and 20,000 mg/L COD. With the investigation of the process conditions, a phenol removal process was developed,

which paved a sound pretreatment ground for the biochemical treatment.

The on-site trial-plant of 2 t/h wastewater set up for industrialized verification was constructed with the extraction column, the solvent recovery distillation column, the dissolved solvent stripping column, and the static fluid mixer. The operation results of the trial-plant showed that more than 93% of the phenols in the wastewater were recovered, which was a by-product with economic benefit. The operating cost of the proposed process was approximately compensated with economic return of the recovered phenols.

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