

High phenol-containing coking wastewater treatment with environmentally benign alkali-enhanced extractant

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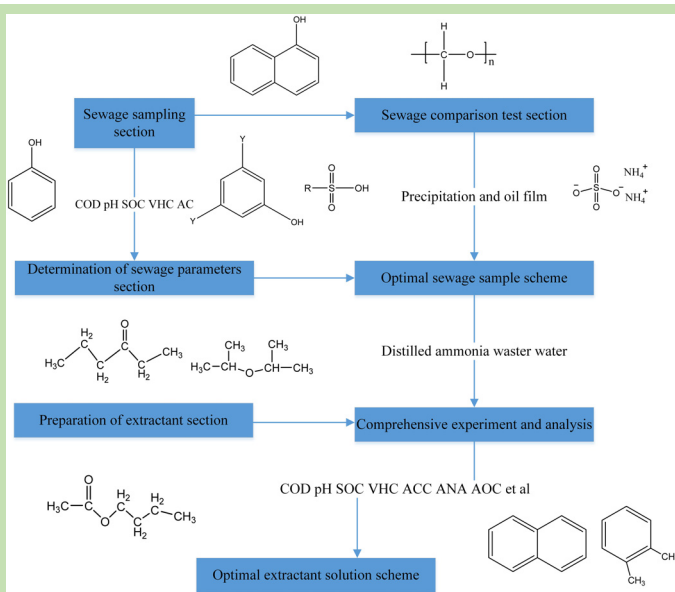
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Abstract: This work hereby reports an eco-friendly efficient extractive wastewater dephenolization process with particular emphasis on cutting secondary organic pollutants. A collection of coking waste water samples containing high phenol concentration was treated by an innovative phenol extraction method. To avoid experimental errors and facilitate water treatment, certain parameters were selected for determining the chemical composition of waste water samples. The distilled ammonia waste water was found to be ideal due to lack of oil film or ammonium sulfate tar precipitation during the dephenolization. In contrast to distilled ammonia waste water, the residual ammoniated water not only was the cause of extreme water pollution, it also consumed large quantity of

acid resulting in high cost, thus creating economic issues for this process. For environmental aspects, while determining the phenol removal efficiency other influencing factors, such as determination of cyanides, sulfur ions, ammonia nitrogen content, chloride ions, sulfate ions, total volatile phenols, total salts, sewage oil content, along with total hardness, COD_{cr}, electrical conduction and pH for comparative test and analysis of alkali-enhanced extractant were also determined. The diffusion behavior of organic molecules and reasons which determine changes in parameter before and after the phenol extraction were studied. Repeatedly washing the used BQ complex extractant 5 times with 3wt% sodium hydroxide solution at 23°C, the relatively optimal regenerated extractant can be prepared. The VHC value of the waste water processed by regenerated extractant was 265.45 mg/L. The regenerated BQ complex extractant was found to have a level of COD_{cr}, sewage oil content, pH, dephenolization efficiency values as 3638.34, 188.86, 6.18, 83.76%, respectively, which meanted very little secondary organic pollutants.

Key words: phenol; removal; extractant; alkali



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环保性碱液强化萃取剂处理高含酚量的焦化废水

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摘要: 本工作研究了一种注重于减少二次有机污染的废水高效环保脱酚工艺, 采用了一种创新的方法采集含酚焦化废水的样品, 通过检测特定参数确定废水样品的化学成分, 避免实验误差且增强了水处理效果. 蒸氨废水是一种理想的焦化废水样品, 脱酚过程中不会产生酸焦油油膜和硫酸铵沉淀. 相对于蒸氨废水, 残余氨水不仅会导致严重的水污染还会消耗更多的酸, 导致成本增高. 从环保角度研究萃取剂, 除了关注其脱酚效率还需要研究其他影响参数如总氮量、S⁻含量、氨氮量、Cl⁻含量、SO₄²⁻含量、挥发酚含量、总含盐、污水含油量、总硬度、COD_{Cr}、电导率和 pH 值等. 考察了有机微粒的扩散特征和导致脱酚前后参数变化的原因. 以温度 23℃、浓度为 3wt% 的氢氧化钠溶液反复洗涤用过的 BQ 络合萃取剂 5 次可得再生萃取剂, 用其处理过后废水挥发酚含量为 265.45 mg/L. 这款优良的萃取剂的 COD_{Cr}, 污水含油量, pH, 脱酚效率依次分别为 3638.34, 188.86, 6.18 和 83.76%, 造成的二次有机污染较少.

关键词: 酚; 脱除; 萃取; 碱液

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1 INTRODUCTION

Coking waste water effluents from oil refineries and chemical industries contains high concentration toxic and carcinogenic phenols posing threat to human life^[1,2]. Gupta et al.^[3] highlighted multiple organ failure as a result of phenol poisoning. Phenol water pollution is also reported to be detrimental to the environment resulting in permanent damage to aquatic life and other organic matter^[4], thus it is vital to install control measures and stop the release of phenols in to water^[5]. Environment protection agencies across the globe formulated specific regulations to protect environment from phenols due to its potential toxic effects upon humans^[6]. Also the recovered phenols from waste water is a valuable resource and used as intermediate in multiple chemical industries^[7], paving way towards sustainable development.

Different authors have reported wastewater treatment for dephenolization which can broadly be categorized in three types: (a) reactive degradation, (b) membrane separation and (c) physical separation. Mohammadi et al.^[8] reported photo-oxidative phenol degradation in conjunction with membrane separation. Li et al.^[9] used advanced anaerobic biofilter method for treatment of coal gasification wastewater, along with effluent recirculation to enhance phenol removal.

Masuda et al.^[10] used enzymatic polymerization precipitation method using *Coprinus cinereus* peroxidase. These reactive degradation methods are relatively innovative, but the equipment cost is often too large to make it economically viable and the operational procedure in each case is very complex. Filtration membrane methods is another wastewater treatment method, where the core component is a porous membrane filter. Tung et al.^[11] and Zeng et al.^[12] independently reported efficient economical ultrafiltration membranes with phenol separation efficiency ranging from 26%~45.6%. Supported ionic liquid (ILs) membranes is another group of highly efficient filtration membranes, where IL is immobilized inside the pores of thin microporous solid support^[13,14]. Another type is liquid emulsion membrane where the diffused solutes pass through the organic phase and is considered to be the most effective membrane with efficiency up to 97%^[15-17]. Finally, physical wastewater treatment methods such as steam distillation, extraction and adsorption were also adopted by some researchers. For example, Pratarn et al.^[18] used fluidized bed reactor to investigate the combined effect of adsorption and oxidation for phenolic wastewater treatment. Apart from the above-mentioned methods, extraction is another popular method due to its process simplicity

and low operation cost. Selection of a proper extractant is the core of the whole extraction process. Extractants such as coconut oil and palm oil^[19,20] were reported to remove phenol pollutants from wastewater. But their dephenolization is inefficient and complex pretreatment of extractant is required. It is even worse that residual chemical remains of the extracted water is often higher which means the secondary organic pollution^[21]. In this work, the comparative analysis of alkali-enhanced dephenolization extractant is reported. Firstly, the analysis parameters for dephenolization from a microscopic point of view were optimized. Secondly, the diffusion process of organic molecules was also described. Thirdly, factors controlling variation in parameter values before and after dephenolization were explained for the test items. Lastly, an alkali-enhanced extractant process was developed with optimal dephenolization performance.

2 EXPERIMENTAL

2.1 Materials and Equipment

Sulfuric acid (98%), sodium hydroxide (Yunnan Anning Chemical Industry, China), reagent barrels, separating funnels (1000 mL, Hirschmann Co., Ltd., Germany), electronic balance (Denver Co., Ltd., USA), four cylinders of 1000 mL capacity, high-speed mixers (Huasco Plastics Machinery Co., Ltd., China), pH meter (2.00–16.00 pH, Guangdong Huigu Co., Ltd., China), BQ complex extractant (Beijing B.Q.G.Y Technologies Co., Ltd., China), portable volatile phenol tester (Beijing Zhi Yun Da Technology Co., Ltd., China), infrared spectrometer (Qingdao Jinghong Co., Ltd., China), COD fast measuring instrument (Hash Co., Ltd., USA), thermometers, deionized water, multi-parameter controller (Supelco Co., Ltd., Germany).

2.2 Sample Process and Sewerage Determination

Coking waste water samples containing high concentration phenol are provided by Kunming Steel Coal Coking Co. Ltd, specialized in production of commercial gas, ammonium sulphate and light

benzene. The factory has a tar recovery system to produce the ammonium sulphate as well as two towers of tubular furnace for continuous tar processing. Simultaneously, the technical naphthalene is produced by the continuous distillation, washing and purification in separate towers with in-built tubular furnaces. Consequently, the residual ammonia waste water and distilled ammonia waste water containing phenol, cyanogen, oil and a large number of other organic substances are produced.

As shown in Fig.1, sampling spots were the entrances of the regulating tanks for the industrial waste water and its discharge outlets to several treatment sections. The chemical composition of waste water samples was the prime necessary datum for further analysis and discussion. A portable volatile phenol tester was used to determinate phenol content which can provide valuable data for the performance evaluation of installed extraction system. Infrared spectrometer and pH detector was used to further determine the composition of waste water. The parameter Chemical Oxygen Demand (COD_{cr}) was an important criterion to judge the organic pollution level, which were determined by COD fast measuring instrument.

The determination of chemical composition of sewage water can provide necessary data for the performance evaluation of extraction process. The residual ammonia water samples were taken from the entrances of the five regulating tanks in 30 test tubes, divided in to 5 groups with 6 test tubes in each, and sample collection was done at an interval of 1 h. The distilled ammonia waste water samples were also collected by the same method from the plate-type condenser and atomizing cooler condensate. Next the whole set of 60 test tubes were allowed to settle undisturbed for 30 minutes. It was followed by determination of the primary indices of both sewages. There were four prime indices, volatile phenol content (VHC), COD, sewage oil content (SOC) and pH, the values of these indices are shown in Table 1.

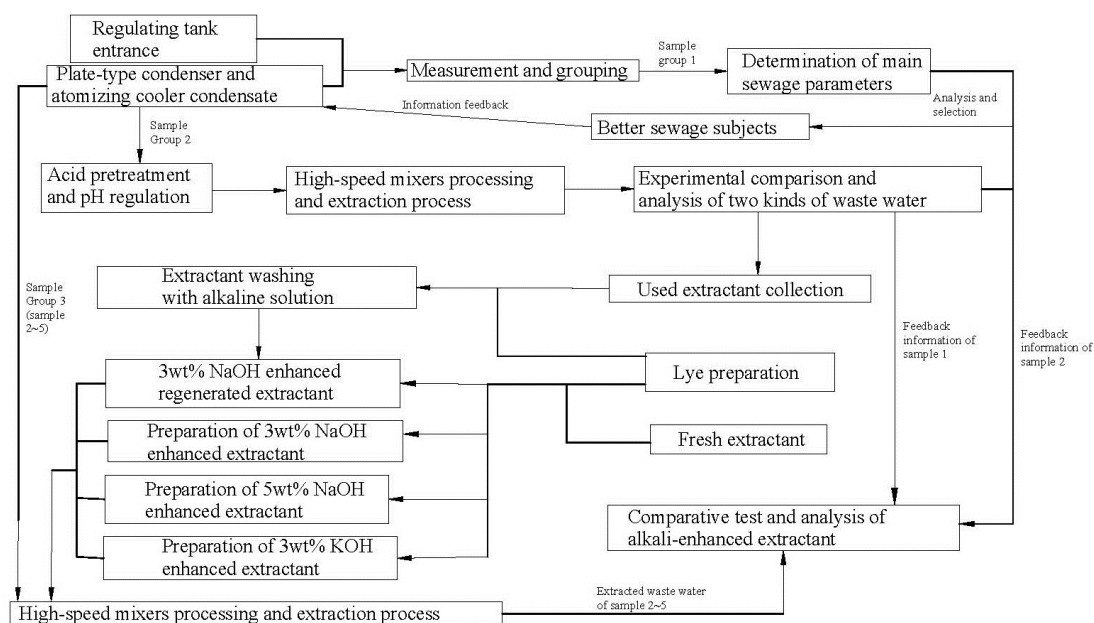


Fig.1 Flow chart of experimental investigation

Table 1 Primary indices of the two kinds of waste water samples

Primary Indices	COD _{cr} /(mg/L)	SOC/(mg/L)	VHC/(mg/L)	pH
Residual ammonia water	7331.83	95.88	1721.52	8.58
Distilled ammonia waste water	7259.27	59.78	1634.67	6.34

2.3 Extraction Treatment Experiment

It is advised to pretreat sewage samples before the extraction process, because the BQ complex performs better in acidic environment by specification. Accordingly, 2 liters of waste water samples were taken in reagent barrels for pretreatment. Sulfuric acid was taken in beakers and stirred with glass rods, and pH was determined at this moment with the help of pH meters. The pH was maintained between the range of 1.5~2.5, by dropwise addition of NaOH solution, while approximately 2 liters NaOH solution was stored in other reagent barrels. In order to avoid errors in data deionized water was used for preparation of reference solutions.

After pretreatment, the pretreated waste water samples taken in beakers, as mentioned above were grouped into five experimental groups and placed on high-speed mixers for 10 min, followed by settling for 20 min. The samples were allowed to cool to 23°C regularly checking with thermometer.

Each experimental group needed 100 mL BQ complex extractant and 400 mL waste water (ratio 1:4), carefully measured using graduating cylinders. Then the mixtures were vigorously mixed in beakers using

glass rods. It was again followed by stirring the mixtures by placing them on high oscillating mixtures for 20 min. After the solutions were homogeneously mixed, the whole mixture was poured into separating funnels of 1000 mL capacity and allowed to settle for 10 min. This extraction process of each experimental group was repeated 5 times using fresh extractant to remove as much contaminants from waste water as possible. Finally, the extracted solution and used extractants are stored respectively in storage bottles.

The composition of the other two kinds of extracted waste water samples taken in the other ten beakers were determined. The average value for each parameter is shown in Table 2. Some test items are added for improving the accuracy of the experiment. For example, acid consumption capacity (ACC) is used to determine the organic pollution of the extracted waste water.

2.4 Comparative Alkali-enhanced Extractant tests

As demonstrated in Table 2, the extracted residual ammonia water as the primary waste water sample for the comparative test is cumbersome. There is an obvious increase of sewage oil content (SOC) in the extracted residual ammonia water. Moreover,

contrast to the distilled ammonia waste water, the residual ammonia water needed more acid for the pretreatment before the extraction, which was not convenient. Furthermore, the extracted residual ammonia water contained the oil film of the acid tar oil and precipitation ammonium salt. As shown in Fig.1, that it is advised to choose distilled ammonia waste water as test sample for comparative test of alkali-enhanced extraction and regenerative extraction.

Table 2 Primary indices of the extracted two kinds of waste water samples

Primary indices	Whether or not precipitation occurred	Whether or not oil film produced	COD _{cr} / (mg/L)	SOC/ (mg/L)	VHC/ (mg/L)	pH	ACC/ (g/L)
Extracted residual ammonia water	Yes	Yes	8863.25	368.64	343.42	6.18	0.73
Extracted distilled ammonia waste water	No	No	8160.34	223.86	323.42	6.23	0.092

Multiple theories have been put forward about the effect of concentration of washing solution, concentration of anti-extracting agent, pH and volume ratio which significantly alter the course of extraction and reverse extraction^[21]. The washing liquid concentration and extractant concentration are estimated using the following expression (1):

$$K_A = \frac{\eta \rho_B V_B}{100 V_A} \alpha, \quad (1)$$

where, K_A is quality score of detergent volume (mg/L), η is experimental parameter for determining the washing efficiency (%), ρ_B is the phenol content in organic solution (mg/L), V_B is the volume of organic solution (L), V_A is detergent volume (L), α is washing times.

As for reverse extraction process the expression is:

$$K_A = \frac{\eta \rho_B V_B (VHC_1 - VHC_2)}{100 V_A} \alpha, \quad (2)$$

where, VHC_1 is phenol content in distilled ammonia waste water (mg/L), VHC_2 is phenol content in the extracted distilled ammonia waste water (mg/L)

As for the ordinary extraction process:

$$K_A = \frac{\eta \rho_B V_B VHC_1}{100 V_A} \alpha. \quad (3)$$

Measuring cylinders, glass rods, beakers and high-speed mixers were used to prepare 12wt% and 3wt% NaOH solutions, as shown in Fig.1. The former solution was used for regenerative extraction while the latter one was used for alkali-enhanced extraction. The used extractant (stated earlier) and 12wt% sodium hydroxide solution were mixed in beakers according

to ratio of 1:1 for extracting agent and waste water. The mixtures were processed by high-speed mixers for 10 min. The mixtures were then poured into a separating funnel (1000 mL capacity) and were allowed to settle. Storage bottles were used to store the regenerated extractant. Another batch of four regenerated extractants were prepared by the same procedures and washed repeatedly with fresh sodium hydroxide (3wt%) solution 5 times to further their dephenolization efficiency.

Four experimental control groups with modified extractants were prepared in comparison to the earlier test groups containing original BQ complex extractant. Twenty beakers of the distilled ammonia waste water were processed by the same pretreatment procedure as mentioned above. Detailed information about the concentration and volume ratio is important for preparation of modified extractant.

Each alkali-enhanced extraction was carried out using the same strategy, i.e. scrubbing one part extractant with five parts of fresh alkali hydroxide solution. 5 conical storage bottles were used to store the regenerated extractant and other 15 bottles were used to store alkali-enhanced extractants.

For improving the accuracy of the comparative experiment, the ratio of extractant to waste water sample was maintained at 1:1, to which five parts of alkali-enhanced extractant were also added with the 3wt% sodium hydroxide solution as fresh extractant. The beakers containing extractant and waste water sample are stirred using high-speed mixers for 10 minutes. The mixtures were then poured into 1000 mL separating funnel and allowed to settle.

To study the effect of anion and cation of the alkali solution on the creation of the alkali-enhanced extraction, 3wt% potassium hydroxide solutions were used to make five parts of alkali-enhanced extractants as match group. The alkali concentration as a strengthening effect, the 5wt% sodium hydroxide

solutions were used to prepare other five pieces of alkali-enhanced extraction as match group.

In every group, five parts extractants were prepared, following the same strategy as the distilled ammonia waste water samples. It is important that multi-parameter controller should be used to investigate more indices for better comparison. The indices included those shown in the Table 2, the content of cyanide (AOC), sulfur ions amount (SIA), ammoniacal-nitrogen amount (ANA), total salt content hydrology (SCH), electrical conduction (EC), chloride ions amount (CIA), sulfuric acid ion amount (SAIC), total hardness (TH). The tags used for the new BQ complex extractant, 3wt% NaOH enhanced extractant, 5wt% NaOH enhanced extractant, 3wt% KOH enhanced extractant, 3wt% NaOH enhanced regenerated extractant are A, B1, B2, C, D, respectively. In the same way, the extracted waste water samples were numbered as 1, 2, 3, 4, 5, respectively.

Dephenolization efficiency (λ) can be calculated by the following expression (4), and shown in Fig.2.

$$\lambda = \frac{VHC_1 - VHC_2}{VHC_1} \times 100\%. \quad (4)$$

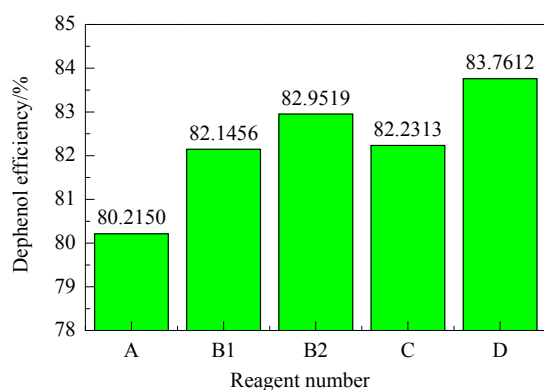


Fig.2 Dephenolization efficiency of different extractants

3 DISCUSSION AND ANALYSIS

It is vividly demonstrated by the Table 2 that BQ complex extractant is an effective method to reduce the volatile phenol content of the residual ammonia water and distilled ammonia waste water samples. In addition, the sewage oil content of two waste water samples increased after the extraction procedure because of the BQ complex extractant. As an oily

chemical, some of the BQ complex extractant will remain in water, but it is worth noting that the sewage oil content of the extracted residual ammonia water is higher than that of the extracted distilled ammonia waste water sample.

Table 1 shows that the sewage oil content of the residual ammonia water is higher than that of the distilled ammonia waste water, because the residual ammonia water contains much more tar. According to the similar phase distribution laws, the tar and the BQ complex extractant are both the nonpolar organic solvent and oily reagent, so the BQ complex extractant will be easily dissolved in the tar particles. On the contrary, the distilled ammonia waste water was processed using sand filter and ammonia steaming process, which helped remove the tar particles. As a result, the sewage oil content of the extracted residual ammonia water is higher than that of the extracted distilled ammonia waste water.

Sulfuric acid is used to adjust the pH value of the waste water. Extracted waste water was weakly acidic as pH=6.2. But the residual ammonia water contains much tar and ammonium ions that reacted with the sulfuric acid, leading to precipitation and oil film formation in the extracted residual ammonia water. Sulfuric acid reacted with tar particles leading to oil film formation that is a kind of waste and difficult to be decomposed. The theory mentioned above also explains the phenomenon that the acid consumption capacity of the residual ammonia water was nearly 8 times more than that of the distilled ammonia waste water.

From the Table 3, it can be seen that the pH value, ANA, SCH, EC remained nearly constant. Volatile phenol content showed that the regenerated extractant had the best ability for dephenolization. Alkali-enhanced extractants were better than the new BQ complex extractant for the reason that the new BQ complex extractant contained small organic molecules creating obstacles for extraction. 5wt% sodium hydroxides enhanced extractant were better than the 3wt% NaOH enhanced extractant. In an alkaline solution with more hydroxyl groups, some small organic molecules in the extractant can easily dissolve

in water, which result into an improve extraction efficiency. In a certain range, the stronger the hydroxyl group, the easier for the small organic molecules to dissolve. Repeated washing is an effective way to remove small organic molecules as well as improve

the extraction performance. The performance of 3 wt% sodium hydroxide enhanced extractant was nearly the same to 3wt% potassium hydroxid enhanced extractant. The cation type has little effect on the extraction efficiency.

Table 3 Primary indices of the extracted residual ammonia water samples in comparative test

Entry No.	Reagent	COD _{cr} / (mg/L)	SOC/ (mg/L)	VHC/ (mg/L)	pH	ANA/ (mg/L)	AOC/ (mg/L)	SIA/ (mg/L)	SAIC/ (mg/L)	SCH/ %	TH/ (mg/L)	EC/ (mc/cm)	CIA/ (mg/L)
Origin	None	7259.27	59.78	1634.67	6.34	331.24	15.68	32.49	96.13	0.69	28.26	10.17	979.24
1	A	8160.34	223.86	323.42	6.23	331.34	14.62	31.23	96.93	0.68	28.11	10.18	979.34
2	B1	7462.34	206.35	291.86	6.24	331.22	14.54	31.24	96.83	0.69	28.14	10.16	979.44
3	B2	7363.45	194.55	278.68	6.25	331.14	14.61	31.28	96.83	0.68	28.12	10.19	979.34
4	C	7468.35	205.89	290.46	6.24	331.32	14.56	31.22	97.23	0.69	28.22	10.18	979.14
5	D	3638.34	188.86	265.45	6.18	326.23	13.64	31.21	97.64	0.67	26.43	11.37	979.34

The theory of small organic molecular content can also explain the role of SOC value. As shown in Fig.3, the SOC values decreased gradually, but still higher than the original water sample. The dissolution of small organic molecules in the extractant which caused increase in the SOC values after extraction is necessary, but the SOC value in the extracted waste water can be controlled using the hydroxyl groups to act as the extractant.

As shown in Fig.3, the use of regenerated extractants reduced COD_{cr} of the distilled ammonia waste water samples, but the use of other extractants increased it. The new BQ complex extractant caused the highest value of the COD_{cr} of the extracted the distilled ammonia waste water. The small organic molecules in the regenerated extractant were removed thoroughly after repeated washing with alkaline NaOH. The removal of phenols is more than the dissolution of small organic molecules, which contributed to the decline of COD_{cr}. But, for the 3wt% sodium hydroxide enhanced extractants and other extractant, it has an opposite effect. The removal effect of phenol compared to the dissolution of small organic molecules is too small, so the overall COD_{cr} value rose instead of decline. As shown in Fig.2, alkali-enhanced regenerated extractant has the highest dephenolization efficiency of 83.76%.

Table 3 shows further information about the parameters used. AOC was supposed to decrease after extraction because of the volatilization of cyanides during mixing and dilution of sulfuric acid, resulting in dissolution of cyanide compounds in the extractant.

Upon mixing and dilution of sulfuric acid to regulate the pH, the SIA of extracted distilled ammonia waste water decreased, because of evaporation of sulfur ions in the form of hydrogen sulfide. EC has increased slightly due to lower non-conductivity resulting from the reduced COD_{cr} and OC content.

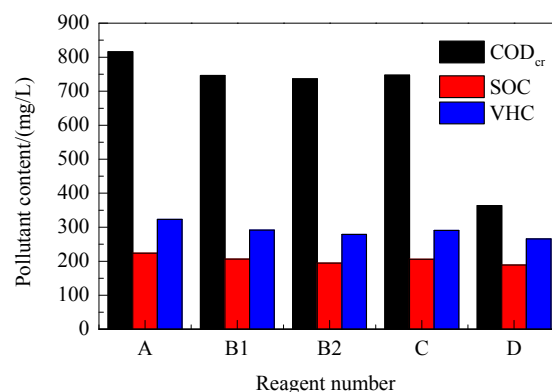


Fig.3 Comparison diagram of main parameters for performance evaluation of different extractants

As for other indices such as pH, ANA, CIA, TH, SCH remained almost constant irrespective of the type of extractant used for dephenolization. The extraction effect of the new BQ complex for removal of phenol was very limited, and it caused an increase in the COD and SOC values resulting in secondary organic pollution.

4 CONCLUSIONS

(1) Repeated washing (5 times) of the regenerated BQ complex extractant with 3wt% sodium hydroxide solution can achieve alkali-enhanced regenerated extractant having optimal extraction performance with lowest VHC.

(2) In strong alkaline environment, the OH^- radicals furthered the dissolution of organic molecules in water, whereas the type of cation in lye usually had little effect. In a some cases, the higher the lye concentration, the better the extraction performance.

(3) When BQ complex extractant and sulfuric acid were used for dephenolization, the distilled ammonia waste water sample showed better results during the experiment because it could avoid the formation of acid tar oil film or ammonium sulfate precipitation. Moreover, the distilled ammonia waste water sample was shown to reduce the consumption of valuable acid during the extraction process.

(4) pH, ANA, CIA, EC, TH, SCH were nearly constant during the extraction process, while the AOC, SAIC, SIA values were shown to have some changes. Extractants was shown to inevitably increase the SOC of the extracted waste water, but the alkali-enhanced regenerated extractant can reduce COD_{cr} and SOC compared to the other extractants which meant reduction in the secondary organic pollutants.

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