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Performance of batch vacuum distillation process with promoters on coke-plant wastewater treatment

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ABSTRACT

This paper presents a simple study of laboratory-scale vacuum distillation process with promoters (VD_P) on coke-plant wastewater, which has the advantage that the chemical reaction and pollutants separation can be concurrent in one pot. The main parameters that evaluate the efficiency of VD_P are conductivity, chemical oxygen demand (COD) and ammonia nitrogen compounds (NH_3-N). The blank experiment, vacuum distillation (VD) process was also investigated for comparison. The effects of some operating factors, such as, vacuum degree of system, the temperature of wastewater and temperature gradient (ΔT) on the treatment efficiency were investigated in detail. And satisfactory results were obtained in the VD_P system with caustic soda and slaked lime as promoters. It was found that high COD removal of 99.7% can be obtained in 180 min for caustic soda promoted system (the detailed condition: NaOH 2.0 g, vacuum degree −85 kPa, ΔT 11 °C). Meanwhile, near 100% NH_3-N removal can be obtained simultaneously in this VD_P process.

1. Introduction

During the past decades, as global environmental pollution has aggravated increasingly, much attention has been focused on environmental awareness and health concerns. Consequently, numerous restricted legislations have been prompted on emissions of various contaminants. Typically, coke-plant wastewater, generated from the coal coking, coal gas purification, and byproduct recovery processes of coke plants [1], represents a major problem; it can create serious environmental pollution after discharged into the receiving aquatic environment directly. Stringent limits for coke-plant effluent have been set in many countries [2], such as China (chemical oxygen demand (COD) < 150 mg l^{-1} and ammonia nitrogen compounds (NH_3-N) < 25 mg l^{-1} for effluent discharge standard) [3]. In particular, a number of small coking plants have been shut down in Shaanxi Province since 2003, as their effluents failed to meet the legal restrictions. Hence, there is an urgent demand for development of efficient technologies to address this issue.

On the other hand, it is very difficult to treat coking wastewater due to its extremely diverse features that vary from one factory to another [3–5], and high concentrated of refractory and biologically inhibitory pollutants are present in this stream, such as phenolic compounds and ammonia. This is one of main characteristics of coking wastewater [6]. Meanwhile, phenols account for most of the chemical oxygen demand of wastewater, which are toxic, mutative and carcinogenic [6–8], and large amounts of ammonia nitrogen compounds will result in serious eutrophication to receiving aquatic environment. To solve these problems, until now, domestic coke-plant wastewater is mainly treated by caustic soda and steam stripping, followed by biological process [2,4,5]. Unfortunately, these complicated processes involve long residence time and considerable space, cannot remove effectively the COD and high-strength NH_3 [1–3], especially in small coke-plant where above sufficient conditions are difficult to be equipped. Furthermore, one of the most shortcomings is that repetitive heating and cooling carried out in aforementioned processes result in considerable energy waste. As a result, taking into account the feature of coking wastewater (it is a type of high temperature liquid), these issues call for new processing technologies with high efficiency.

For many years, vacuum distillation (VD) as an important physical technique for separating and purifying the materials has been widely applied in seawater desalination [9], petroleum refining [10] and organic synthesis [11], but little attention is paid to vacuum distillation for wastewater disposal because of high energy requirement, despite some existing plants have utilized it for treatment of waste emulsions [12,13]. In our study, a combined technology, vacuum distillation assisted with various promoters (VD_P), such as caustic soda (NaOH) and slaked lime ($Ca(OH)_2$), is proposed for the treatment of coking wastewater in laboratory-scale. During the VD_P process, the important feature of wastewater with relative high temperature (average 70 °C for this effluent) can be effectively uti-

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lized, so expensive energy cost can be saving as much as possible. Moreover, volatile components, such as phenols and NH_3, may form the corresponding salts in the mother liquor then separated from condensate maximally [14], so that the purified water with minimum pollutants is obtained which can be recycled in coke-plant or discharged directly.

Herein, the aim of the present work is to investigate the feasibility of VD_P in actual coking wastewater treatment in laboratory-scale. The operation conditions including operating pressure, temperature of wastewater, and the dosages of various promoters were investigated so as to remove most of inorganic and organic pollutants simultaneously from the coking wastewater. Three major parameters, conductivity values, COD and NH_3-N were discussed to evaluate the performance of variant VD_P systems.

2. Materials and methods

2.1. Actual wastewater and materials

The raw wastewater discharged from a coke plant, located in Shaanxi Province, China, was used as feed in this work. It should be pointed out that the chemical composition of coking wastewater is very complex, which typically consists in a mixture of several liquid streams from the cooling step after coking coals, the liquid-stripping step of the coke oven gas, and process water added during gas treatment and byproduct recovery [15]. Here, the wastewater mainly collected from the stage of byproduct recovery, which was deep brown in color and of obnoxious odors, was not pretreated. Some important characteristics of this wastewater are summarized in Table 1. Moreover, the organic pollutants in the wastewater were also analyzed by GC–MS (see Fig. S1, Supporting Information), which indicated the volatile phenolic compounds were main organic pollutants in this stream. Thereinto, based on the data of Table S1 (Supporting Information), it was clear that phenol accounted for most of the COD of this wastewater, followed by cresol. According to the exhibited results, it can be reasonably concluded that this wastewater is characteristic of high COD, NH_3-N and salinity.

2.2. The VD_P experiment for wastewater treatment

All the VD_P tests were carried out using a classical lab-scale vacuum distillation system that was represented in Fig. S2 (see Supporting Information), main part of which was composed of one three-neck flask, water-cooled condenser and vacuum pump provided with a pressure control device, and the flask was surrounded by thermostatic water bath for temperature control. For blank experiments, VD processes were carried out with 150 mL raw wastewater at different vacuum degree and bath temperature to find the optimal operation conditions. Before distillation the wastewater was firstly heated to the desired temperature so as to simulate the actual hot coking wastewater, and then the temperature of vapor was also recorded. All the correlative operating details for various VD processes are shown in Table S2 (Supporting Information), including vacuum degree of system, the vapor temperature, and wastewater temperature (simulated actual temperature of coking wastewater).

Table 1
Characteristics of influent used in this study for VD_P process.

Parameter	Average value
Temperature (°C)	70
pH	6.4
Conductivity ($\mu S\,cm^{-1}$)	17110
COD ($mg\,l^{-1}$)	7045
NH_3-N ($mg\,l^{-1}$)	408
Cyanides ($mg\,l^{-1}$)	7.8
Phenol ($mg\,l^{-1}$)	1230

For VD_P experiments, 150 mL of wastewater was filled into the flask, and then the reactor was agitated after the promoter added into wastewater. Subsequently, continuous vacuum distillation experiment was operated and the condensate was drawn at regular intervals and analyzed by pH, conductivity, COD, NH_3-N and ultraviolet absorption during each experiment. Here, in all VD_P experiments a stable vacuum degree and bath temperature to distillation process were monitored according to the results of blank experiments.

The COD and NH_3-N removal efficiency at any distillation time t, R R_{COD} and $R_{NH_3\text{-N}}$ are calculated as follows:

$$R = \frac{c_0 - c_t}{c_0} \times 100\%$$

where c_0 is the initial COD or NH_3-N concentration of raw wastewater and c_t is the COD or NH_3-N concentration in the condensate at any time t.

2.3. Analytical methods

The COD for wastewater was measured utilizing a colorimeter according to APHA standard methods [16]. The analytical determination of phenol was carried out using UV–vis spectrophotometer (UV-7504, China). The measurement of total NH_3-N concentration in wastewater was conducted by Nessler's reagent colorimetric method on a UV-7504 spectrometer. The pH value was examined using an Orion 290 pH meter, and the conductivity was utilized to estimate the salinity of condensate indirectly, which almost changed linearly with salinity [17]. Measurements of above test parameters were performed in duplicate and the results obtained as mean values. GC–MS was used for analysis of organic compounds in coking wastewater after liquid–liquid extraction with CH_2Cl_2, and the detailed procedures and analysis methods have been reported by Lai et al. [4].

3. Results and discussion

3.1. Treatment efficiency of the VD system

The vacuum degree of system and temperature gradient (ΔT) display significantly influence on the vacuum distillation process for separation of liquid mixtures [12,13]. In this context, there is a serious need for investigating the interplay of these parameters so as to find the optimal treatment condition. In order to reflect clearly the effect of wastewater temperature on water quality, the ΔT is defined as the difference between the temperature of vapor and the hot wastewater in our study.

3.1.1. The influence of operating vacuum degree

In this work, based on the actual variation of raw wastewater, system vacuum degree at −90, −85, −75 and −63 kPa, and the corresponding simulated wastewater temperature of 42, 55, 65 and 77 °C, respectively, were investigated. This resulted in a lowest ΔT of about 7 °C achieved to all the VD processes, which allowed the continuous distillation just running in our experiment conditions. Thus, it can reflect the effect of operating vacuum degree on condensate quality straightforward, and reduce energy consumption.

The evolution of condensate quality along with distillation time is shown in Fig. 1. It can be observed clearly in Fig. 1a and 1b that both the COD and NH_3-N values increase in various extent as the vacuum degree of system decreases from −90 to −63 kPa, especially at the initial stage of VD process. However, this trend weakened gradually as operation proceeded. As we know, the lower the system vacuum degree, the higher the evaporation temperature.

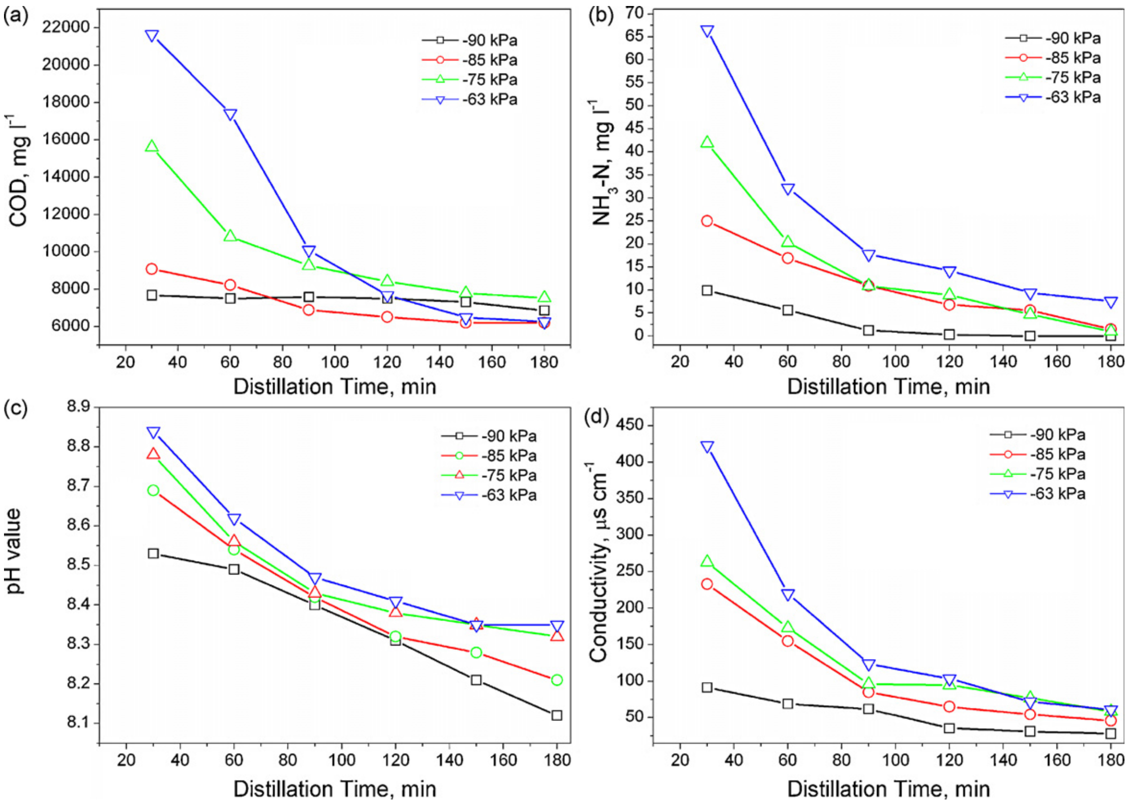

Fig. 1. Effects of operating vacuum degree on the condensate quality. (a) COD evolution; (b) NH_3-N evolution; (c) pH and (d) conductivity.

This favors the evaporation of volatile compounds like ammonia and phenolic compounds, leading to the enhancement of above pollutants in the condensate, and similar phenomenon was also described by Gutiérrez et al. [12]. On the other hand, the boiling is very violent at the start phase of VD process because vapor–liquid equilibrium is not established at the time. This situation can greatly stimulate the entrainment of wastewater into the vapor phase, and result in much poorer quality for distillates at the initial stage than that of at other stages. However, this phenomenon weakened gradually with operation time prolonging. Above argument is strongly reinforced by the conductivity data, which has often been a suitable indicator of the salinity of aqueous solution [17,18]. In view of the enormous contributions of non-volatile inorganic salts to the liquid salinity [18], it is acceptable that the conductivity value can not only evaluate the variation of inorganic pollutants in distillates indirectly, but also plays as an estimator of entrainment behavior. It is self-evident that the non-volatile inorganic salts are hardly distilled into condensate in VD process. As can be seen from Fig. 1d, the trend of conductivity is similar to the trend of COD and NH_3-N, which also decrease gradually with operation time prolonging in all cases, indicating the severe entrainment behavior is concentrated at the initial stage that weakens rapidly as time increasing. So the quality of condensate gradually was improved as time prolonging. Furthermore, it is apparent that the entrainment behavior is also increased with system vacuum degree decreasing. However, all the conductivity values of condensate were much lower than that of raw wastewater (17110 μS cm^{-1}) throughout operating period, which implied that inorganic pollutants can be reduced well in the condensate. Moreover, in all cases the cyanides were not detected in the condensate.

On the other hand, it should be noted that the operating vacuum degree shows much more influence on the COD than on the NH_3-N. This can be ascribed to the discrepancy of volatility between ammonia and phenol. Thus, an excessive high COD value for condensate was present in all cases, which was even higher than that of raw wastewater (7045 mg l^{-1}) during most period of VD process. It may be reasonable to speculate that the evaporation of volatile phenols was much superior to that of water in VD process, indicating phenols can be hardly separated from water effectively by VD regardless of the operating pressure. Conversely, to relative low-volatile ammonia nitrogen compounds, VD exhibited satisfied separation performance of ammonia and water. The NH_3-N value of condensate at various stages was much lower than that of raw wastewater (408 mg l^{-1}), although it also increased with operating pressure increasing. In addition, a similar trend is observed from the pH of condensate (Fig. 1c), which declines gradually with operating time increasing. Because aqueous ammonia solutions show weak alkaline while aqueous phenol solutions show weak acidic, the variation of pH may result from complicated concentration fluctuation of these pollutants in the condensate.

The occurred phenomenon in above VD processes was also confirmed by GC–MS results, which was consistent with the aforementioned COD testing. The determined GC–MS profiles of condensates collected at 30 and 180 min, respectively, and raw wastewater are shown in Fig. 2. The operating parameters were fixed at vacuum degree of −85 kPa and wastewater temperature of 55 °C in this experiment. The similar composition for condensates collected at 30 min {condensate (30)}, condensate (180) and raw wastewater can be observed clearly in Fig. 2, indicating the VD can hardly remove the volatile organic pollutants from water even if prolonging distillation time.

On the basis of above discussion, it can be concluded that VD has excellent property on separation of relative low-volatile ammonia and inorganic salts from wastewater. For example, the NH_3-N values of condensate were all lower than 25 mg l^{-1} when the operating vacuum degree was above −85 kPa, meeting the effluent discharge standard of China. Unfortunately, the removal of organic pollutants was not satisfactory in VD processes. Thus, the followed study on

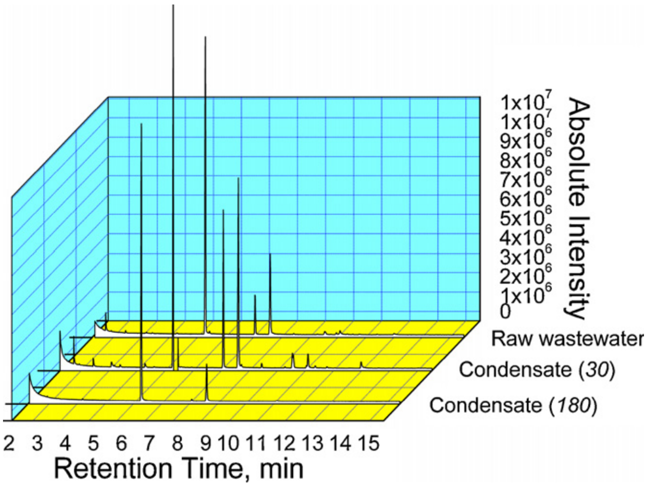

Fig. 2. GC of raw wastewater and condensate at various times in VD process (operating vacuum degree: −85 kPa; influent temperature: 55 °C).

the treatment efficiency of VD process was performed at the operating vacuum degree of −85 kPa in considering the treatment cost.

3.1.2. *The influence of temperature gradient*

Special attention should be also paid on another important operation index, evaporation rate (ER) that determines considerably the space-time efficiency of VD process. Previous studies have revealed that both operating pressure and temperature gradient can affect the ER [12,13], especially the latter. This is coincident with our results (Fig. 3), where the ER is expressed as the volume of liquid sample evaporated per unit time. To be clear, the ΔT of 7, 9, 11 and 13 °C was obtained at a constant vacuum degree (−85 kPa) but at different wastewater temperatures (55, 57, 59 and 61 °C, in turn). As shown in Fig. 3, the effect of operating vacuum degree on ER is limited; it increases slowly with operating vacuum degree decreasing, whereas a minor enhancement of ΔT results in a sharp rising of ER. The ER at ΔT of 13 °C is almost 3.5 times faster than that of 7 °C, while the ER only increases from 20.2 to 25.5 mL h^{-1} with the operating vacuum degree varying from −85 to −63 kPa. It should be pointed out that near 95 vol.% wastewater can be transferred into condensate at ΔT of 11 and 13 °C after processing, just leaving the semisolid mixtures in the reactor, which can be treated easily by several technologies, such as incineration, landfill and cement solidification technology [19–21].

On the other hand, temperature gradient displays much complicated effect on condensate quality (Fig. 4). As can be seen from Fig. 4a, COD reduces a little as ΔT increases from 7 to 13 °C at the beginning of VD process, this trend strengthens with time prolonging, particularly at the final stage. Meanwhile, COD decreases gradually with time increasing in all cases, which drastically reduces in the case of two relative high ΔT values (11 and 13 °C). Whereas, it is obvious in Fig. 4b that the NH_3-N value does not reduce regularly with ΔT increasing throughout operating period. For example, NH_3-N value obtained at ΔT of 9 °C was slightly less than that obtained at ΔT of 11 °C during most of the time. Furthermore, the NH_3-N value does not always reduce with time prolonging in the case of two relative high ΔT, which increases markedly at the final stage of VD process. However, the NH_3-N values detected during the whole process always met the limit set by the National Discharge Standard of China.

Presumably, the reasons to above complicated phenomena can be summed up as follows: (i) as discussed early, the higher ΔT value, the higher ER. Then it will be logical to assume that the growth of ER for water is higher than that of pollutants in the wastewater during the same period, so the concentration of pollutants in the condensate relatively decreases with the ΔT increasing. Moreover, considering the high volatility of phenols, these components in wastewater are largely vaporized into gas phase during the VD process, hence the remarkably reduction of COD in the later stages is also partially due to the significantly decreasing of these pollutants in the mother liquor at the time. (ii) Previous work has described that the high ΔT value facilitates the violent boiling like operating pressure [13], which inevitably results in serious entrainment behavior at the initial stages of VD process. Thus, the complicated concentration fluctuation of pollutants in the condensate can be attributed to the confliction between the ER of liquid and the entrainment behavior. To volatile organic pollutants, lots of them were vaporized into gas phase continuously, so the trend that the COD decreased with the ΔT value increasing was clearer as the entrainment behavior weakened gradually. However, to relative low-volatile ammonia, lots of them retained in the wastewater during the VD process, so the concentrated mother liquid with ammonia at the final stages can affect significantly the NH_3-N of condensate by the entrainment behavior, especially in the two high ΔT conditions. This is consistent with the conductivity results (Fig. 4d), where a similar trend can be observed as compared with the NH_3-N results. The reason to this situation can be due to close volatility of ammonia and other inorganic pollutants, so that both the ΔT and the entrainment behavior play similar role in determining the concentration of these components in the condensate. Likewise, the sharp dropping of pH in the condensate at the final stages (Fig. 4c) may be also due to the presence of acidic inorganic salts in condensate at that time.

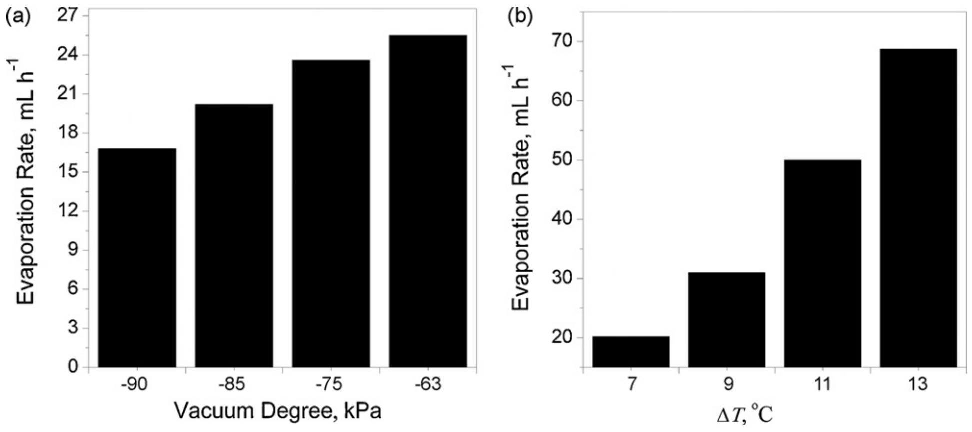

Fig. 3. Effect of vacuum degree (a) and temperature gradient (b) on evaporation rates.

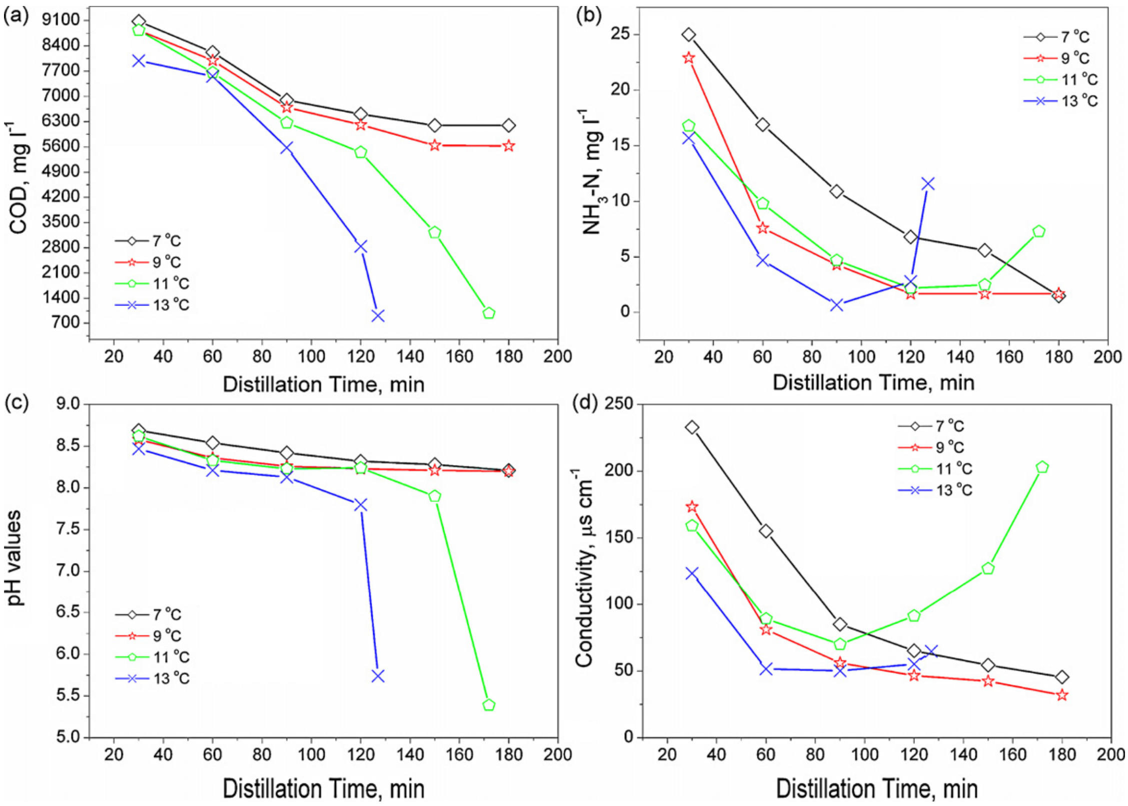

Fig. 4. Effects of temperature gradient on the condensate quality. (a) COD evolution; (b) NH_3-N evolution; (c) pH values and (d) conductivity values.

From what has been discussed above, we may finally draw the conclusion that the enhancement of ΔT displays dual effects on the condensate quality, although it facilitates the high ER. Thus, in order to avoid serious entrainment behavior, the ΔT cannot be fixed at too high value.

3.2. *Treatment efficiency of the VD_P system*

In above blank experiments, a satisfied COD removal is never able to obtain regardless of operating parameters, although the ammonia nitrogen compounds and other inorganic pollutants can be removed well from condensate. For example, even though at the relative optimal operating conditions (operating vacuum −85 kPa and ΔT 11 °C), the lowest COD still reached to 980 mg l^{-1} (corresponding to the COD removal of 86%), which was much higher than that set by National Discharge Standard of China. According to the previous GC–MS data, the main organic pollutants in the condensate were enormous quantities of volatile phenols. Thus, depending on the physicochemical properties of these compounds, to search some efficient promoters that can inhibit the high volatility of phenols is very crucial during the VD process. Herein, considering the following reaction of phenol in the aqueous solution:

$$C_6H_5\text{-}OH + OH^- \rightarrow C_6H_5\text{-}O^- + H_2O \qquad (1)$$

it is reasonable to infer that the addition of OH^- into wastewater favors the formation of phenolate ion, the volatility of which is much lower than that of phenol molecule. As a result, phenols can be separated from condensate when some basic agents are com-

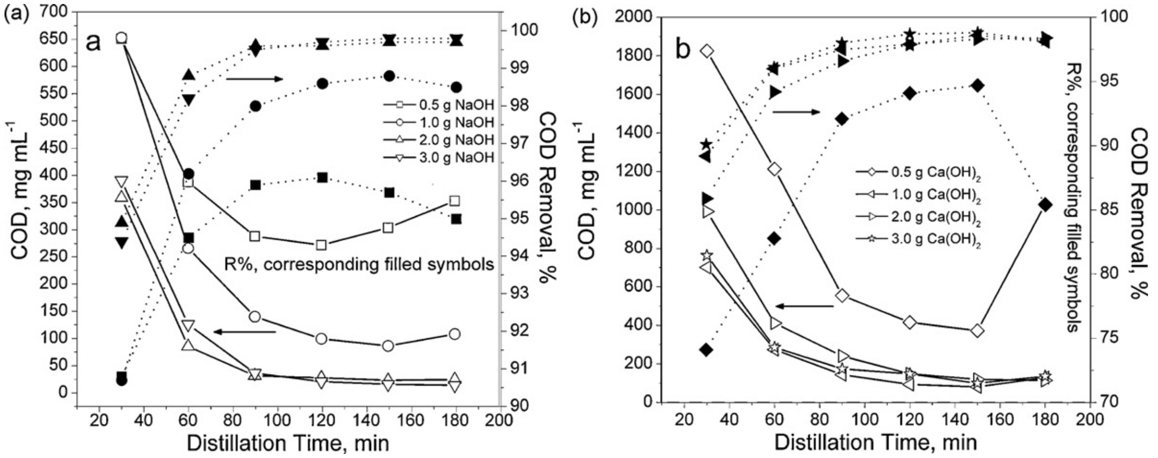

Fig. 5. Effects of different promoters dosages on condensate COD and COD removal in VD_P processes (operating vacuum degree: −85 kPa; ΔT: 11 °C, the blank symbols correspond to COD and the filled symbols correspond to COD removal).

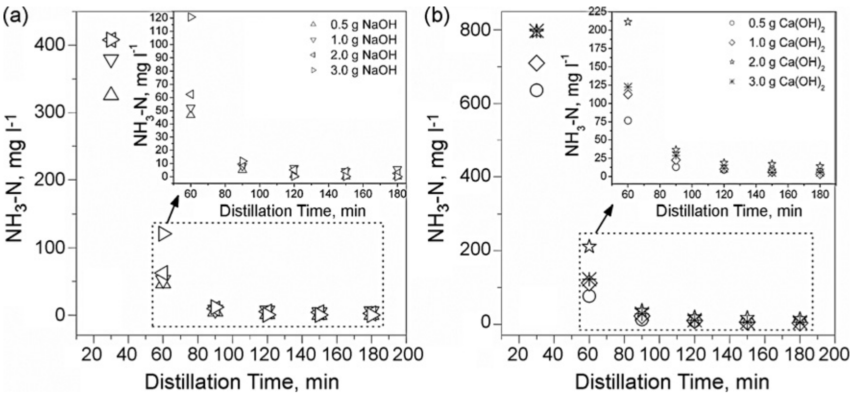

Fig. 6. Effect of different promoters dosages on condensate NH_3-N in VD_P processes.

bined with VD system, which remain in mother liquor as phenolate ions that can be recovered easily [7]. Here, two basic agents, caustic soda and slaked lime were selected to improve the efficiency of VD_P process, which have been traditionally utilized as a primary material in wastewater treatment [22,23].

At the constant operating conditions (operating vacuum degree −85 kPa and ΔT 11 °C), the effects of variant basic agents dosages on condensate COD were investigated in our study, and the result is shown in Fig. 5. As expected, it is clearly seen in Fig. 5a that COD is significantly reduced after NaOH added into VD system. For example, the condensate COD collected at initial 30 min was only 651 mg l^{-1} (corresponding to the COD removal of 90.8%) in the case of 0.5 g NaOH promoted VD_P process, which was much lower than the minimum COD obtained in the blank experiment at the same conditions. This proves that the presence of OH^- ions in wastewater can improve efficiently COD removal of VD_P process. Moreover, COD decreased remarkably as NaOH dosages increased from 0.5 to 2.0 g during operation period, whereas, a close change trend in COD can be observed as NaOH dosages increased from 2.0 to 3.0 g. This indicated that the further addition of NaOH dosages in wastewater may be not an effective way to enhance the removal of COD greatly when the dosage reached to 2.0 g during VD_P processes. However, it should be emphasized that in the case of two high NaOH dosages, all the COD values were below 150 mg l^{-1} after 60 min, which were lower than the limit set by the National Discharge Standard of China. Furthermore, the COD trend during the VD_P process can be also supported by the corresponding UV–vis absorbance (Fig. S3 displays the UV absorption spectra of condensate samples treated by 2.0 g NaOH, Supporting Information). Fig. S3 shows that only the sample collected at initial 30 min displays relative strong characteristic absorption peaks of phenolic compounds [24], and the characteristic peaks are not clear in other samples. In addition, much similar COD trend in the case of $Ca(OH)_2$ promoted VD_P process can be also observed in Fig. 5b. However, the treatment efficiency of $Ca(OH)_2$ is obviously lower than that of NaOH under the same condition. Even though in the case of 3.0 g slaked lime, COD was still above 150 mg l^{-1} during the most of operating time.

On the other hand, the effects of variant basic agents dosages on the NH_3-N can be observed clearly in Fig. 6. It can be seen that much high NH_3-N values appear at the initial stages for both NaOH and $Ca(OH)_2$ promoted systems, although they all decrease rapidly with operation prolonging. The reason may be that more hydroxyl ions in solution impel the reaction equilibrium of Eq. (2) towards the left side so that wastewater turns to be enriched with ammonia molecules, the volatility of which is much higher than that of NH_4^+ ion.

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \quad (2)$$

However, it should be pointed out that the NH_3-N concentration was below 25 mg l^{-1} since the operation was carried out for 90 min in the NaOH promoted VD_P process, suggesting that the ammonia molecules derived from the negative effects of basic agents were not great influence on NH_3-N removal efficiencies with operating time prolonging.

4. Conclusions

In terms of removal efficiencies on COD and NH_3-N, caustic soda would be a suitable promoter for VD_P process in our work, and the optimal dosage was 2.0 g in considering the removal efficiencies and treatment cost comprehensively. The quality of condensate met the limit set by the National Discharge Standard during most of distillation period; it can be discharged or reused directly. In summary, the experimental results in our work confirm that the VD_P process is feasible for treating coking wastewater with high COD, NH_3-N and salinity, and it makes the best use of waste heat of water, thus avoiding large energy consumption during traditional treatment process and improving the environmental pollution. However, researches on recovering some valuable substances from the residues would be further studied so as to improve the value of VD_P process in practice.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.cej.2010.03.053.

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