Application of a Sustainable Bioderived Solvent (Biodiesel) for Phenol Extraction

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ABSTRACT: Replacement of volatile organic compound solvents by greener or more environmentally sustainable solvents is becoming increasingly important due to the increasing health and environmental concerns. In the present work, a bioderived solvent, soybean oil methyl ester, which is better known as biodiesel and is a nonvolatile organic compound, was used as a solvent to replace the fossil solvent (kerosene) for phenol extraction. First, biodiesel was selected as an optional solvent to replace kerosene based on Hansen solubility parameter calculation results. Second, the effects of solvent concentration, equilibrium pH of the aqueous phase, temperature, extraction time, etc. on phenol extraction were examined. The results show that biodiesel has strong extraction ability on phenol extraction than that of kerosene. An acidic environment decreases the phase disengagement time. Phenol extraction reached equilibrium in 30 s of contact time at room temperature. McCabe–Thiele diagram calculation results show that the phenol extraction efficiency can reach 98% in three theoretical stages at an A/O ratio of 10:1 (Cyanex923 + biodiesel). Finally, the extraction mechanism indicated that biodiesel could reduce the intermolecular hydrogen bond forces in the extractant so as to improve the extraction efficiency.

INTRODUCTION

The organic fossil solvents used in industrial applications have been mainly volatile organic compounds (VOCs), including alcohols, ethers, and aliphatic compounds, as well as organic derivatives of halogenated hydrocarbons. Most of these organic compounds are volatile, toxic, flammable, and detrimental to the environment.1 Due to the extensive attention to environmental pressure and the increasing pressure on the management and control of VOCs, it is particularly important to choose more green and environmentally friendly organic solvents.

Bioderived solvents are renewable resources that can be extracted from plants and algae. They are green solvents, less toxic, and biocompatible.2 Recently, bioderived solvents have been applied to various areas, including organic synthesis,3 catalysis,4 biotransformation,5 separations of rare earths,6 and extraction.7 To date, although biodiesel has been used as a solvent in extraction,8 there is still lack of complete study on the application of bioderived solvents in the solvent extraction.

Phenol is widely used in petrochemical industries and other chemical industries.7 As a result, phenol is extensively present in the effluents of these manufacturing plants10 and are inevitably introduced into waste water. Solvent extraction is an effective method for recovering phenol.11 Impregnated resin12 with Cyanex923, a solid extractant, was used for extraction, and the kinetics of extraction was studied. The common liquid extractants used in industry include 2-octanol,13 TBP + kerosene,14 Cyanex923 + kerosene,15 etc.

In this work, biodiesel was selected as a solvent to replace kerosene in the solvent extraction of phenol from aqueous solution. The influence of solvent concentration (kerosene and biodiesel), temperature, A/O ratio, and equilibrium pH on phenol extraction was investigated. Moreover, the equilibrium data was regressed by an empirical model and the necessary theoretical stages of extraction were calculated by McCabe–Thiele diagrams. Finally, the related extraction mechanism was also discussed.

RESULTS AND DISCUSSION

Graphical Estimation of the Normalized Hansen Solubility Parameter (HSP) Values of the Extractant. The physical properties of extractants and solvents are shown in Table 1.16 The main component of kerosene is aliphatic kerosene.

According to eqs 9–14, HSP of several extractants could be estimated using the graphical method proposed by Teas et al.17 that the three components of the HSP are used as reference axes in a diagram. Equation 1 is an example for the coordinates.
on the dispersion axis. The ternary HSP diagram of solvents and extractants is shown in Figure 1.

\[
\%_{\text{dispersion}} = \frac{\delta_{\text{d}}}{\delta_{\text{d}} + \delta_{p} + \delta_{h}} \times 100
\]  

(1)

In Figure 1, the position of water is in the lower left corner of the diagram, whereas the solvents and extractants are at the top. The normalized HSP values of the solvents and extractants are far from the ones of water is refer to the low solubility. As the distance between biodiesel and phenol is shorter than the distance between kerosene and phenol, the capability of biodiesel for dissolving phenol could be better than that of kerosene. Although there is a certain distance between the biodiesel and kerosene, the position between Cyanex923 + biodiesel and Cyanex923 + kerosene is very close, which means kerosene has the potential to be replaced by biodiesel. A bioderived solvent, biodiesel, is selected as a solvent for phenol extraction in this work.

**Effects of Phase Contacting Time on Extraction.** The data on phase contacting time\textsuperscript{12} is required to analyze and design an extraction process especially on an industrial scale. In this paper, the effects of phase contacting time on phenol extraction in biodiesel and kerosene solvents are studied. The results are shown in Figure 2. The phenol (2500 mg/L) was extracted with 92 vol % biodiesel + 8 vol % Cyanex923 and 92 vol % kerosene + 8 vol % Cyanex923 at an A/O ratio of 20:1, respectively. It can be found that the phenol extraction efficiency increased with the increase of contact time and reached equilibrium in 30 s. The contact time of subsequent extraction experiments was set as 50 s.

**Effect of Temperature on Phenol Extraction.** The effect of temperature on phenol extraction was studied in the range of 298−328 K (Figure 4). It was found that \( D \) increased with the decrease of the temperature.

The enthalpy change of the extraction, \( \Delta H \), could be calculated from the slope of the log \( D \) versus 1000/T by the van’t Hoff equation\textsuperscript{18} (eq 2).

\[
\log D = -\frac{\Delta H}{2.303RT} + C
\]  

(eq 2)

where \( R = 8.314 \) is the universal gas constant and \( C \) is a constant for the system.

The relationship between log \( D \) and 1000/T in different solvents is shown in Figure 4, and the calculated \( \Delta H \) values are

| chemical formula | av MW (g/mol) | \( \rho \) (g/cm\(^3\)) | solubility (g/L, 298 K) |
|------------------|--------------|----------------|------------------------|
| kerosene \( \text{CH}_3(\text{CH}_2)_8\text{CH}_3(97 \text{ wt } \%) \), \( \text{C}_6\text{H}_5(\text{CH}_2)_4\text{CH}_3(3 \text{ wt } \%) \) | 142.17−254.30 | 0.800 | insoluble |
| Cyanex923 \( (\text{CH}_3(\text{CH}_2)_7\text{PO})_3 \) | 340−350 | 0.879 | <0.1 |
| biodiesel \( \text{CH}_2(\text{CH}_3)_2\text{CH}═\text{CH}(\text{CH}_2)_3\text{COOCH}_3 \) | 296.49 | 0.874 | insoluble |

**Table 1. Physical Properties of Extractants and Diluents**

**Figure 1.** Ternary HSP diagram of solvents and extractants. (The ellipse represents the area where the HSP of the extractants is expected).

**Figure 2.** Effect of extraction time on phenol extraction by different extractants (stirring speed = 400 rpm, PhOH = 2500 mg/L, A/O ratio = 20:1, pH = 7; \( T = 298 \text{ K} \)).

**Figure 3.** Variation in phenol extraction efficiency with respect to the volume concentration of solvents (PhOH = 2500 mg/L, A/O ratio = 10:1, pH = 7; \( T = 298 \text{ K} \)).

higher than 92%, the extraction of phenol has obvious difference. Compared to that of kerosene being used as a solvent, biodiesel has high extraction efficiency. This could be explained as the biodiesel has strong extraction ability on phenol extraction than kerosene.

**Effect of Temperature on Phenol Extraction.** The effect of temperature on phenol extraction was studied in the range of 298−328 K (Figure 4). It was found that \( D \) increased with the decrease of the temperature.

The enthalpy change of the extraction, \( \Delta H \), could be calculated from the slope of the log \( D \) versus 1000/T by the van’t Hoff equation\textsuperscript{18} (eq 2).

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where \( R = 8.314 \) is the universal gas constant and \( C \) is a constant for the system.

The relationship between log \( D \) and 1000/T in different solvents is shown in Figure 4, and the calculated \( \Delta H \) values are
listed in Table 2. \(\Delta H\) could be calculated to \(-9.459\) or \(-11.22\) kJ/mol, which was similar to the bond energy of the hydrogen bond. The results show that \(\Delta H < 0\) in phenol extraction, which indicated that the extraction process is exothermic. Therefore, the extraction experiment is suitable at room temperature.

**Effect of pH on Phenol Extraction.** Phenol is a weak acid that dissociates into PhO\(^{-}\) and H\(^{+}\), with the dissociation acid equation defined as eq 3.

\[
\text{PhOH} \leftrightarrow \text{PhO}^- + \text{H}^+ \quad \text{pK}_a = 9.98 
\]  
(3)

The form of phenol in varying pH values is different. Li et al. find that phenol has UV–vis absorption at 270, 287, and 300 nm, which depends on the pH of solution. All experiments in this work are conducted in acidic and neutral environment wherein phenol only has absorption at 270 nm.\(^{19}\) When the pH is less than or equal to 7, the form of most phenol existing in water phase is PhOH. When the pH is more than 7, the form of most phenol existing in water phase is PhO\(^{-}\). It is worth noting that the pH of the original phenol solution is 7.

To determine the effects of pH on phenol extraction behavior, the extractants (92 vol % biodiesel + 8 vol % Cyanex923, 92 vol % kerosene + 8 vol % Cyanex923, and 100% biodiesel) were used for phenol extraction at various pH values. The results of extraction efficiency and phase disengagement time (PDT) are shown in Figures 5 and 6, respectively.

In Figure 5, it was observed that the efficiency in all three extractants decreased with the increase of pH. The reason is that phenol was extracted in the molecular form and alkaline environment restrained phenol extraction of the mixed solvents. The phenol extraction capacity followed the sequence: 92 vol % biodiesel + 8 vol % Cyanex923 > 92 vol % kerosene + 8 vol % Cyanex923, and 100% biodiesel). Using biodiesel as a solvent (92 vol % biodiesel + 8 vol % Cyanex923), the extraction efficiency could reach about 80%.

PDT is a critical parameter in determining the usefulness of a solvent extraction system. The result of PDT at various pH values is shown in Figure 6. The PDT of three extractants increases with the increase of pH, and the acidic solution environment enhances the separation of organic phase from aqueous phase. The PDT of 100% biodiesel is generally longer than those of other extractants, but the PDT shortened when mixed with Cyanex923 in proportion. Based on the results above, it can be known that the acidic solution environment is beneficial to phenol extraction by different extractants and solvents.

**Isotherms of Phenol Extraction.** The phenol extraction isotherms were obtained using different kinds of extractants with the same initial phenol concentration in aqueous phase (Figures 7 and 8). The results show that the maximum phenol loading of 92 vol % biodiesel + 8 vol % Cyanex923 system reached to 36 500 mg/L, which is higher than that of the 92 vol % kerosene + 8 vol % Cyanex923 system (21 100 mg/L). Moreover, the phenol extraction efficiency of these extractants decreased with the increase of the A/O ratio. A/O of 10:1 was selected in subsequent experiments from the view of industrial application.

**Extraction Stages Estimated by the McCabe–Thiele Method.** McCabe–Thiele diagram is an import method to determine the theoretical stages for the liquid–liquid solvent extraction process.\(^{20}\) Figure 9 shows the McCabe–Thiele diagram constructed for phenol extraction by different solvent systems at pH 7 and 298 K (92 vol % biodiesel + 8 vol % Cyanex923 and 92 vol % kerosene + 8 vol % Cyanex923). Using 92 vol % kerosene + 8 vol % Cyanex923 as the

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**Table 2. Thermodynamic Parameters of Extractants for Phenol Extraction**

| extractants                     | slope | \(\Delta H\) (kJ/mol) |
|---------------------------------|-------|-----------------------|
| 92 vol % biodiesel + 8 vol % Cyanex923 | 0.586 | -11.22                |
| 92 vol % kerosene + 8 vol % Cyanex923  | 0.494 | -9.458                |

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**Figure 4.** Effect of temperature on phenol extraction with different extractants (PhOH = 2500 mg/L, A/O ratio = 10:1, pH = 7).

**Figure 5.** Variation of efficiency with respect to the pH value (PhOH = 2500 mg/L, A/O = 10:1, T = 298 K).

**Figure 6.** Variation of the PDT with respect to the pH value (PhOH = 2500 mg/L, A/O = 10:1, T = 298 K).
extractant, four theoretical extraction stages would suffice to remove the phenol from 2000 to 10 mg/L at an A/O ratio of 10:1. In contrast, only three theoretical extraction stages are required for the 92 vol % biodiesel + 8 vol % Cyanex923 system.

**Extraction Mechanism.** To illustrate the extraction mechanism of the extraction reaction, the extracted complex was characterized with Fourier transform infrared (FTIR) spectra. The characteristic spectra of organic phase at different loading conditions are shown in Figure 10.

The bands from 1260 to 1110 cm\(^{-1}\) are attributed to the P=O stretching vibration of Cyanex923.\(^ {21}\) The P=O stretching vibration peak of pure TRPO was located at 1153 cm\(^{-1}\), but the P=O stretching vibration peak of Cyanex923 had shifted from 1153 to 1166 cm\(^{-1}\) after diluting to 8 vol % with kerosene and from 1153 to 1170 cm\(^{-1}\) after diluting to 8 vol % with biodiesel. The results suggested that Cyanex923 molecules interacted with each other, possibly because the O atom on the P=O bond formed a hydrogen bond with the H atom on the carbon chain of another Cyanex923 molecule. When biodiesel was added as a diluent, the hydrogen bond of Cyanex923 might be weakened. Therefore, the stretching vibration peak of P=O is moved to a higher wavenumber and is better than that of kerosene. After extraction, the P=O stretching vibration peak of 92 vol % kerosene + Cyanex923 has shifted from 1166 to 1149 cm\(^{-1}\) and the P=O stretching vibration peak of 92 vol % biodiesel + Cyanex923 has shifted from 1166 to 1146 cm\(^{-1}\). This indicated the existence of the hydrogen-bonding interactions between P=O groups and phenol molecules. FTIR spectra of extractants and complexes, showed in Figure 11, indicated that phenol was extracted due to the O−H stretching vibration peak of phenol at 3648 cm\(^{-1}\). The C=O stretching vibration peak of biodiesel at 1744 cm\(^{-1}\) has not shifted after extraction. Therefore, biodiesel improves the extraction efficiency by reducing the intermolecular hydrogen bond forces in the extractant.
CONCLUSIONS

A biosolvent (biodiesel) and Cyanex923 have been shown to be effective for the extraction of phenol. According to the HSP calculated, kerosene had the potential to be replaced by biodiesel as a solvent in phenol extraction. The phenol extraction efficiency decreased with the increasing solvent concentration of both kerosene and biodiesel. Biodiesel had strong extraction ability for phenol extraction than that of kerosene. Phenol extraction reached equilibrium in 30 s of contact time, and room temperature was optional. PDT was decreased in the acidic solution environment and increased in the alkaline environment. From the calculation results of the McCabe–Thiele method, phenol extraction could reach 98% using three theoretical stages for 92 vol % biodiesel + 8 vol % Cyanex923 with the initial concentration of 2000 mg/L phenol at an A/O ratio of 10:1. Biodiesel, used as a diluent, could reduce the intermolecular hydrogen bond forces in the extractant, which improved phenol extraction.

It was found that the new solvent selected in this study, biodiesel, has the potential to not only improve the phenol extraction ability but also will pave the way for the application of bioderived solvents in the solvent extraction.

MATERIALS AND METHODS

Reagents and Instrument. Kerosene, sodium hydroxide (NaOH), and 98 wt % sulfuric acid (H₂SO₄) were purchased from Tianjin Guangfu Chemical Reagent Co. Ltd. (Tianjin, China). Phenol was purchased from Tianjin Fuchen Chemical Reagents Co. Ltd. (Tianjin, China). Biodiesel was obtained from Guangzhou Fufei Chemical Corporation (Guangzhou, China). Cyanex923 was purchased from Shanghai Cytech Company (Shanghai, China). All reagents were of analytical purity grade and used without any further purification.

Absorbance was measured using a LebTech-UV-9100 UV–vis spectrophotometer (Beijing, China). A 98-I-C heating Jacket (China) was used for maintaining a constant temperature of the aqueous phase and an HD2004W motor stirrer (Shanghai, China) was used for the phenol extraction. Institute-DRIFT-TENSOR (Germany) was used for infrared scanning.

Stock Solution Preparation. Phenol (2500 mg/L; pH 7), 10 wt % H₂SO₄, and 5 M NaOH were prepared by diluting them with deionized water. The same volume of Cyanex923 was diluted by kerosene and biodiesel, respectively. The percentage of extractants is the percentage of volume, for example, 8 vol % Cyanex923 + 92 vol % biodiesel.

Liquid–Liquid Extraction Experiment Procedure. At room temperature (298 K), a fixed volume of phenol was taken into a separating funnel and a fixed volume of the extractant was added. The mixture was briefly shaken for 10 min and kept aside for 30 min, after which the aqueous phase and organic phase got separated. The aqueous phase was collected, and the residual phenol was measured on a UV–vis spectrophotometer.
at 270 nm. Accordingly, the concentration of phenol in the organic phase was calculated by mass balance. The reduction in phenol concentration was deduced as eq 4. The distribution ratios could be calculated from eq 5.

Phenol extraction

\[ E = \frac{C_i - C_f}{C_i} \times 100\% \]  

(4)

Distribution ratio

\[ D = \frac{V_x}{V_o} \times \frac{C_i - C_f}{C_i} \]  

(5)

where \( C_i \) is initial phenol concentration in the aqueous phase, \( C_f \) is phenol concentration after extraction, \( V_x \) is the volume of the aqueous phase, and \( V_o \) is the volume of the organic phase.

Determination of Organic Solvent by Hansen Solubility Parameter (HSP). Materials with similar HSP values have high affinity for each other. The solubility of organic polymer substances can be predicted according to the HSP. The basic equation governing the assignment of Hansen parameters is that the total cohesion energy, \( E \), must be the sum of the individual energies that make it up.

\[ E = E_d + E_p + E_h \]  

(6)

where \( E_d \) is the dispersion cohesive energy, \( E_p \) is the polar cohesive energy, and \( E_h \) is the electron exchange parameter.

Dividing this by the molar volume gives the square of the total solubility parameter component of HSP.

\[ \frac{E}{V} = \frac{E_d}{V} + \frac{E_p}{V} + \frac{E_h}{V} \]  

(7)

\[ \delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \]  

(8)

where \( V \) is the molar volume, \( \delta_d \) is the D component of HSP, \( \delta_p \) is the P component of HSP, \( \delta_h \) is the H component of HSP, and \( \delta \) is the total solubility parameter component of HSP.

The HSP of solvents, solutes, and other organic compounds can be calculated using the group contribution method described by Hansen with the group contribution values collected by Barton as follows

\[ \delta_i = \sum F_i / \sum V \]  

(9)

\[ \delta_p = (\sum F_p^2)^{1/2} / \sum V \]  

(10)

\[ \delta_h = (\sum -U_{m\text{me}})^{1/2} / \sum V \]  

(11)

Table 3 summarizes values of the parameters used in eqs 9–11 for some of the most common organic groups needed to calculate the HSP by means of the group contribution method. As organic molecules were mainly based on hydrocarbons, the HSP of the solvent and extractant can be calculated by the group contribution method. The HSP of the mixed extractant (ME) can be estimated by the group contribution method in the following simplified way

\[ \delta_{EME} = \left( x \sum F_{d\text{extractant}} + (1 - x) \right) \sum F_{d\text{solvent}} \]  

\[ / \left( x \sum V_{\text{extractant}} + (1 - x) \left( \sum V_{\text{solvent}} \right) \right) \]  

(12)

\[ \delta_{p\text{ME}} = \left( x \sum F_{p\text{extractant}}^2 + (1 - x) \right) \sum F_{p\text{solvent}}^2 \]  

\[ / \left( x \sum V_{\text{extractant}} + (1 - x) \left( \sum V_{\text{solvent}} \right) \right) \]  

(13)

\[ \delta_{h\text{ME}} = \left( x \sum -U_{\text{m\text{extractant}}} + (1 - x) \right) \sum -U_{\text{m\text{solvent}}} \]  

\[ / \left( x \sum V_{\text{extractant}} + (1 - x) \left( \sum V_{\text{solvent}} \right) \right) \]  

(14)

The \( x \) (%) in eqs 12–14 is represented as the volume concentration of the extractant.

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Notes

The authors declare no competing financial interest.

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