Facile Cloud Point Extraction for the Separation and Determination of Phenolic Acids from Dandelion

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ABSTRACT: It is significantly crucial to develop a robust pretreatment for the quantitative analysis of herbs. However, the traditional strategies are time-consuming, tedious, and not eco-friendly. In this work, cloud point extraction (CPE) is engineered for the simultaneous separation and enrichment of ferulic acid (FA), chlorogenic acid (CLA), and caffeic acid (CA) from dandelion prior to its determination by high-performance liquid chromatography (HPLC). A famous nonionic surfactant of Triton X-114 was selected as an extractant of CPE, and parameters affecting the extraction, such as surfactant concentration, salt content, pH value, temperature, and incubation time, were investigated carefully. Furthermore, the well-designed CPE with ultrasonic assistance combined with HPLC was developed for the detection of the target analytes in dandelion. The established method having a good linearity in the range of 0.15−26.2 mg L\(^{-1}\) with \(R^2\) more than 0.9979 and the spiked recoveries ranging from 81 to 96% was applied to test real samples of dandelion. The contents of CA in samples were consistent with those assayed by the method (Chinese Pharmacopoeia 2015). The proposed method afforded good analytical performances, shorter pretreatment time (65 min), and less organic solvent consumption (less than 1.0 mL). It was proved that the developed method presented a facile, inexpensive, efficient, and environment-friendly pretreatment and can be used for the quantitative analysis of CLA, CA, and FA in dandelion. As expected, the proposed method would be a promising potential for the quality analysis of herbal medicines.

1. INTRODUCTION

Cloud point extraction (CPE), a promising extraction method, has been improved unceasingly over the past decade. Compared with the traditional pretreatments, CPE has the advantages of facile operation, higher efficiency, safety, and eco-friendliness.\(^1,2\) It mainly utilizes the solubilization of the surfactant and the cloud point phenomenon to achieve phase separation from the extracting solution.\(^3,4\) Usually, the hydrophilic phase of the surfactant expands in water to form a long and flexible vermiform micelle. Thus, the analytes interacting with micellar systems can be concentrated into the surfactant-rich phase in a small volume.\(^5,6\) Once the concentration of the surfactant exceeds the critical point of micelles, the analytes will transfer to the interior of micelles and be tightly bound to the hydrophobic groups in micelles.\(^7,8\) When the systems are heated above the cloud point temperature (CPT), the solution divides into two phases: the hydrophobic ingredients are extracted into the surfactant-rich phase and the hydrophilic constituents retain in the aqueous phase.\(^9−11\) Triton X-100, Tween-80, Triton X-114, and Genapol X-080 are commonly used as nonionic surfactants.\(^12\) The CPT of Triton X-114 is lower than that of others, hence CPE can be performed under mild conditions.\(^13\) Triton X-114 is usually applied for the extraction of the sensitive analytes.\(^14,15\) Based on the conventional CPE, dual-CPE (dCPE) has been proposed.\(^16\) While samples are pretreated with dCPE, the target analytes are purified and the influences of interfering components are ulteriorly reduced.\(^17,18\) Nong et al. coupled dCPE with high-performance
liquid chromatography (HPLC) to simultaneously extract, separate, and determine trace sulfonamide in urine and water samples. Therefore, it is interesting and significant to explore the applications of CPE.

At present, CPE has been widely applied for the separation and enrichment of trace substances and active components in traditional Chinese medicine by combining with other technologies. Chen et al. proposed a novel method for separating the hydrophobic compounds of psoralen and isopsoralen from *Psoralea corylifolia* L. using Genapol X-080 as the extract surfactant of CPE. Xing et al. developed a CPE with Triton X-114 with satisfied recovery for testing the camptothecin content in the fruit, bark, and leaves of *Camptotheca acuminata*. Tang et al. designed a microwave-assisted CPE using Triton X-100−NaCl−HCl as the extracting matrix, which was employed to extract and synchronously separate flavonoids and alkaloids from *Crotalaria sessiliflora* L. It is unconfused that the eco-friendly CPE represents the inherent advantages over conventional liquid−liquid extraction with toxic organic solvents. Besides, CPE is considered to be a miniaturized technique, which is facile to be implemented and does not require sophisticated and expensive instruments. To date, CPE has been extensively employed in the pretreatment of samples concerned with herbs.

Dandelion is a common Chinese herbal. Ferulic acid (FA), chlorogenic acid (CLA), and caffeic acid (CA) with the properties of antiinflammation and disinfection are the representative components of dandelion. It is crucial to test the representative components for the quality assessment of dandelion. The traditional sample pretreatment of dandelion mainly including liquid−liquid extraction, liquid−solid extraction, and microwave-assisted extraction mostly requires lots of expensive and toxic organic solvents, and the operation process is time-consuming. Therefore, it is necessary to develop a facile, inexpensive, and eco-friendly pretreatment for the quality assessment of dandelion. In this work, CPE using a nonionic surfactant of Triton X-114 was used to simultaneously separate and enrich FA, CLA, and CA from dandelion prior to its determination by HPLC. First, phenolic acids were extracted by ultrasonication with water (5% Triton X-114, w/v). Then, the target analytes were isolated via phase separation, which can be facilely performed at 60 °C within 30 min. There are several advantages for the proposed CPE: (1) ultrasound and the surfactant are synergistic to promote the extraction of target components; (2) phenolic acids are separated and enriched simultaneously with less than 1.0 mL of the organic solvents; and (3) the CPE including extraction and separation can be accomplished within 65 min at 60 °C. Then, the well-designed CPE (Scheme 1) with ultrasonic assistance combined with HPLC was developed for the detection of FA, CLA, and CA in real samples of dandelion. It was proved that the proposed method presented a facile, inexpensive, efficient, and environment-friendly pretreatment and can be used for the quantitative analysis of CLA, CA, and FA in dandelion. As expected, the suggested method can provide a prospective alternative for the quality control of dandelion or its related products.

2. RESULTS AND DISCUSSION

2.1. Optimization of CPE. In this work, Triton X-114 is chosen as a surfactant for the formation of the surfactant-rich phase due to its excellent physicochemical characteristics: low CPT (23−26 °C), lower hydrophilic lipophilic balance value

![Molecular structure of CLA, CA, FA, and Triton X-114.](https://doi.org/10.1021/acsomega.1c01768)
(12.4), high density of the surfactant-rich phase, and low toxicity, which facilitates the phase separation by facile operation. Figure 1 reveals that the phenolic acids consist of a benzene ring, hydroxyl groups, and a carboxyl group and Triton X-114 has a benzene ring and hydroxyl group. It is considered that the interactions such as hydrogen bonding, π−π stacking, and hydrophobic effects occur between analytes and Triton X-114, which could primarily contribute to the extraction. Consequently, Triton X-114 is selected as the surfactant. There are many factors influencing the extraction efficiency of CPE, mainly including surfactant concentration, ratio of salt, pH value, temperature, and incubation time. In order to achieve the optimal operation conditions, it is necessary to investigate carefully the parameters of CPE. The effects on extraction were evaluated by the extraction efficiency which is defined in eq 1:

\[
\text{the extraction efficiency (\%)} = \frac{C_S V_S}{C_0 V_0} \times 100\%
\]  

(1)

where \(C_S\) represents the analyte’s concentration in the surfactant-rich phase of volume, \(V_S\), while \(C_0\) represents the analyte’s concentration in the initial sample–surfactant mixture of volume, \(V_0\).

2.1.1. Effect of the Surfactant Concentration. The surfactant concentration is a critical factor for CPE and directly affects the extraction efficiency. When the concentration is low, the extraction would be incomplete; however, the increase of the concentration may disturb the subsequent determination. The relationship between the concentration of the surfactant and the extraction recovery was studied carefully, and the results are shown in Figure 2a. The recovery increased with the growth of Triton X-114 concentration from 2 to 5% (w/v), the recovery reached a plateau at a concentration of 5% (w/v), and the recovery maintained balance, although the concentration increased up to 8% (w/v). The results might be related to the micelle formation; more micelles form in the solution with increasing concentration of the surfactant and more analytes transfer into the micelle phase. Furthermore, the extraction efficiency of FA, CLA, and CA was similar and more than 90%. Therefore, we selected the surfactant concentration of 5% (w/v) for the next study.

2.1.2. Effect of the Salt Concentration. Salt concentration is another important factor in the CPE process. In general, the phase separation is accomplished by heating the mixture over CPT. Owing to the presence of the electrolyte, the CPT of surfactants is reduced, which improved the phase separation, and the use of low temperature may prevent the analyte degradation. In addition, the nonelectrolyte becomes less soluble in the presence of the relatively high concentration of the electrolyte according to the salting-out concept, and hydrophobic interactions between micelles can be enhanced by adding a salt to the micellar solution. In view of that, NaCl is readily available, inexpensive, and more frequently used in the procedure of CPE. In this work, we chose NaCl to promote the extraction efficiency, and the results are demonstrated in Figure 2b. The recovery was greatly enhanced with the increase of salt concentration from 5 to 10% (w/v). Once the salt concentration exceeded 10% (w/v), the recovery presented...
insignificant changes except that of CA. However, the reproducibility of the extraction declined obviously when the salt concentration was more than 20% (w/v). As is well known, hydration spheres form around the ionic salt molecules, reducing the solubility of analytes, which conducive to the phase separation.30 Therefore, the efficiency of extraction was strengthened via adding NaCl into the solution. Nevertheless, the salting-out effect arises under higher salt concentration, which affects the precision of extraction.26,31 Based on overall consideration of the results, 10% (w/v) of salt concentration was selected in this work.

2.1.3. Effect of the Extraction Time. CPE is an ongoing process and carried out in two immiscible solutions; the separation of incompatible substances requires equilibrium time. With the increase of heating time, the more complete the two-phase separation, the easier to obtain the oil phase.15,20 The incubation time for phase separation is also a key factor for CPE, and the different incubation times were studied ranging from 10 to 60 min, as shown in Figure 2c. When the incubation time was less than 30 min, the recovery boosted with the time extension. There was a slight variation for the extraction efficiency though it was more than 30 min. The results indicated that no obvious effects on the extraction recovery occurred with the growth of time from 30 to 60 min. It is considered that the phase separation almost completes within 30 min. Based on the extraction efficiency, 30 min was fixed as the working condition.

2.1.4. Effect of Temperature. Temperature is a critical parameter for CPE because the phase separation only occurs when the temperature is higher than CPT.32 Generally, the temperature is set to 20 °C above the CPT.13 In this work, the effects of temperature toward extraction were studied carefully in the range of 30–80 °C, and the results are shown in Figure 2d. When temperature was below 60 °C, the recovery presented an overall upward trend with the increase of temperature, whereas the growth rate of recovery decreased and tended to stabilize gradually with the rising of temperature from 60 to 80 °C. The extraction recovery was enhanced because the nonionic surfactant becomes more hydrophobic with the increase of temperature. The dehydration of the surfactant improved when the temperature increased, which contributes to the repulsion and aggregation of micelles.2,25,32 Meanwhile, more analytes are retained in the surfactant-rich phase. Hence, the solubilization capacity of micelles is accelerated with the rise of temperature, leading to an increase in the extraction of analytes. Considering the extraction efficiency and the convenience of experimental operation, 60 °C was chosen as the optimum condition.

2.1.5. Effect of pH. It is well-known that the pH of the sample solution influences significantly the extraction efficiency because the analyte’s existing forms (ionic or neutral compound) depend on the pH of the solution during the extraction process.19,22 As shown in Figure 1, FA, CLA, and CA have similar chemical structures and similar functional groups, meaning that their chemical properties are also alike. The pKa values of FA, CLA, and CA are 4.58, 4.58, and 3.91, respectively (https://www.chemicalbook.com). Under acidic conditions, they should exist in the form of neutral molecules, but they would present ions in the alkaline solution. The effects of pH (2.0, 3.5, 6.8, 8.0, and 9.0) on the extraction efficiency were investigated, and the outcomes are displayed in Figure 3. The different extraction recoveries of target analytes were performed with different pHs in the extraction solution; the extraction efficiency increased with the decrease in the pH of the extraction solution and the maximum extraction efficiency was achieved at a pH less than 3.5. The solubility of CA, FA, and CLA declines in acid solutions where they are present in the neutral form, which facilitates the target analytes to transfer to the surfactant-rich phase.22 Hence, the extraction recovery was reinforced greatly at pH 3.5. Nevertheless, they prevail in the form of ions in alkaline solutions, boosting their hydrophilicity, which is favorable to their solubility and impedes their retention in hydrophobic surfactant micelles.19 While the pH of the solution approached neutral, a moderate extraction efficiency was obtained. Due to the similar structure and properties of CA, FA, and CLA, parallel results were produced with the pH changes of the solution. In view of the results, the CPE was performed at pH = 3.5. In summary, the selected conditions for CPE are detailed in Section 4.4.

2.2. HPLC Analysis. In order to separate completely and accurately quantify the target analytes, the factors such as the composition of the mobile phase and detector wavenumber were evaluated carefully. It can be seen from Figure 1 that FA, CLA, and CA own acid groups and they tend to be ionic in alkaline solutions, which is a disadvantage for chromatographic separation. It could be favorable to promote the retention and column efficiency while they are present in the form of neutral molecules in acid solutions. Therefore, acetic acid was added to the mobile phase and the percent of 0.1–2.0% (v/v) was tested. Furthermore, different mobile phases consisting of methanol/water and acetonitrile/water were examined, and the velocity of the mobile phase (0.7, 0.8, and 1.0 mL min⁻¹) was also investigated separately. In addition, the maximum absorption wavelength of FA, CLA, and CA is around 320 nm based on the results of full-wavelength scanning, as given in Figure S1. Ultimately, methanol/acetonitrile/acetic acid (1.0%) (12.5:12.5:75, v/v) with 0.8 mL min⁻¹ and a detector wavelength of 320 nm were selected as the optimal parameters with a running time of 8 min, and the well resolutions for the analytes were obtained even in the real sample, as shown in Figure 4. The developed method qualifies for the testing of FA, CLA, and CA in dandelion.

2.3. Analytical Performances of the Methods. Under optimum conditions for CPE and HPLC analysis, the analytical method was developed, and the linear range, repeatability, precision, and recovery were evaluated. The analytical performances of the proposed method were investigated carefully, as listed in Section 4, and the calibration curves were constructed by plotting the peak area versus the
concentrations of compounds. As can be seen in Table 1, the proposed method provided a good linearity in the range of 0.15–26.2 mg L\(^{-1}\) with the coefficients of correction (\(R^2\)) more than 0.9976 and relative standard deviation (RSD) less than 2.0%. The limits of detection (LODs), based on the signal being three times as large as the baseline noise (S/N = 3, where S is the signal of the analyte and N is the baseline noise), for CLA, CA, and FA were 0.008, 0.005, and 0.007 mg L\(^{-1}\), respectively.

The stability of the developed method was tested in terms of intraday and interday precisions by analyzing triply a standard sample in 0, 1, 3, 5, and 7 days. At the optimum condition, the intraday and interday precisions were expressed with RSD of the retention time and peak area for CLA, CA, and FA. The RSD variations of the retention time for analytes were in the range of 1.2–3.0%, and the good precisions of peak area with RSD were all less than 5.0% for intraday and interday.

Furthermore, the repeatability tests of the proposed method were validated by measuring parallelly the same sample of dandelion based on the optimum procedure. Five equivalent powders of dandelion, each of which was about 0.05 g, were extracted by CPE (Section 4.4) and determined with HPLC (Section 4.5), and the RSDs of analyte contents were in the range of 3.5–5.0%. The data revealed that the developed method owned remarkable repeatability.

Besides, the recovery tests were performed at three spiked levels to evaluate the accuracy of the proposed method (Table 2). The standard substances of analytes, which were equivalent to 0.5, 1.0, and 2.0 times of the original content in dandelion, respectively, were added to approximately 0.05 g of the extracted samples, and then, the resulting samples were extracted and tested at the optimum condition. The recoveries of CLA, CA, and FA at three concentration levels were in the range of 80–100% with RSDs of 2.1–4.5%, indicating that the developed method is reliable for determining the analytes in dandelion.

### Table 1. Analytical Performances of the Proposed Method (n = 3)

| analytes | linearity range (mg L\(^{-1}\)) | \(R^2\) | RSD (%) | LOD (mg L\(^{-1}\)) |
|----------|---------------------------------|--------|---------|-------------------|
| CLA      | 0.25–26.2                       | 0.9989 | 1.3     | 0.008             |
| CA       | 0.15–16.3                       | 0.9985 | 1.5     | 0.005             |
| FA       | 0.21–22.5                       | 0.9979 | 1.6     | 0.007             |

### 2.4. Comparison Study

The developed method was compared with other works concerning determination of phenolic acids. Compared to the microwave-assisted extraction of CLA from blueberries,\(^\text{33}\) the developed CPE required a shorter time as well as less consumption of organic solvents in this work. It can be noted that small amounts of organic solvents and less time were hired by dispersive liquid–liquid microextraction for the extraction of phenolic acids from procumbens,\(^\text{34}\) but the proposed CPE afforded a facile procedure. The analytical performances of the proposed method were comparable or in some cases have distinct advantages over the other reported studies. The LOD of CLA was far less than that (1.64 mg L\(^{-1}\)) of HPLC-diode array detection (DAD) for the quantification of CLA in medicinal plants.\(^\text{35}\) The LOD of CA was comparable to that (0.06 mg L\(^{-1}\)) of HPLC-DAD for the determination of CA in Physalis angulata L.\(^\text{36}\) but more than that (0.016 μg L\(^{-1}\)) of micro-SPE-HPLC-DAD for the analysis of CA in the medicinal extracts of plants.\(^\text{37}\) The LOD of FA corresponded to that (0.0015 mg L\(^{-1}\)) by liquid chromatography–mass spectrometry for pharmacokinetics of CA in the Naoxintong capsule.\(^\text{38}\) In addition, the LODs of CLA, CA, and FA were less than that (0.027–0.068 mg L\(^{-1}\)) of HPLC-DAD for dandelion.\(^\text{39}\) In comparison with the previous works, the developed method is qualified for the quantification analysis of CLA, CA, and FA in dandelion.

### 2.5. Analysis of Real Samples

To validate the method, three batches of dandelion samples were measured with the developed method. Dandelion powder (0.05 g) was extracted by ultrasonication (Section 4.3), the resulting samples were treated by CPE (Section 4.4) to enrich the analytes, and the contents of analytes were determined by HPLC-DAD. Figure 4 shows the results of chromatographic analysis. It can be observed that the chromatogram affords a good separation of CLA, CA, and FA (Figure 4a). Figure 4b illustrates a visible spectrum after dandelion samples were extracted by the proposed method, and the analyte signals are enhanced significantly compared with those of without CPE (Figure 4c). However, FA could not be detected even if dandelion samples were extracted by ultrasonication (Figure 4c). Figure 4d reveals the chromatogram of the control assay for the solution containing Triton X-114 (5%, w/v), and there are no interferences for the quantitative analysis of analytes. The analytes of CLA, CA, and FA were identified by the retention time, and their spectrum from the sample chromatogram...
average contents of the three analytes in dandelion were different due to the difference in the growth place of the raw materials. The contents of CA were in the range of 0.016–0.045%, indicating that the herbs of dandelion were qualified based on Chinese Pharmacopoeia 2015. In addition, the contents of CLA and FA were in the range of 0.03–0.044 and 0.001–0.007%, respectively, which were consistent with the literature. In order to further verify the feasibility of the devised method, the contents of CA in the dandelion samples were tested using the assay recorded in Chinese Pharmacopoeia 2015, and the consistent results were achieved even though lower levels of CA were detected (Table S1). The results confirmed that the proposed method was feasible for the quantitative analysis of CLA, CA, and FA in dandelion.

3. CONCLUSIONS

In this work, a robust and efficient pretreatment with ultrasonic assistance and CPE was developed to measure CLA, CA, and FA in the herb of dandelion. The satisﬁed extraction efﬁciency was obtained under the optimum conditions of Triton X-114 (5%, w/v), salt concentration (10%, w/v), pH (3.5), and incubation for 30 min at 60 °C. CPE with ultrasonic assistance was combined to HPLC-DAD for the detection of analytes in dandelion. The established method having a good linearity with \( R^2 \) more than 0.9979 and the spiked recoveries ranging from 81 to 96% was applied to test real samples of dandelion. The contents of CA in samples were consistent with those assayed by the method recorded in Chinese Pharmacopoeia 2015. The pretreatment can be accomplished in 65 min with less than 1.0 mL of the organic solvents. It was proved that the proposed method presented a facile, inexpensive, efficient, and eco-friendly pretreatment and can be used for the quantitative analysis of CLA, CA, and FA in dandelion. As expected, the proposed method affords a promising potential for the quality analysis of herbal medicines.

4. MATERIALS AND METHODS

4.1. Reagents and Materials. CLA, CA, and FA were purchased from the National Institute for the Control of Pharmaceuticals and Biological Products (Beijing, China). The nonionic surfactant Triton X-114 ([1,1,3,3-tetramethylbutyl]-phenyl-polyethylene glycol) was provided by Sigma-Aldrich (Shanghai) Trading Co., Ltd (Sigma, Shanghai, China). Acetonitrile was of chromatographic grade and obtained from Sigma-Aldrich (Shanghai) Trading Co., Ltd (Merck, Shanghai, China). Methanol, sodium chloride, ethanol, hydrochloric acid, and sodium hydroxide were supplied by Tianjin Chemical Reagent Co. Ltd (Tianjin, China). Distilled water (Wahaha, Hangzhou, China) was used in all experiments. All other reagents used in this work were of analytical grade.

4.2. Instruments. All tests were performed on c2695 HPLC systems equipped with a vacuum degasser, an auto sampler, a binary pump, and a DAD w2998 (Waters, USA). The samples of dandelion were pulverized by a pulverizer (Zhongyi, Beijing, China). A thermostatic bath (Langyi, Shanghai, China), a centrifuge (Anting, Shanghai, China), a pH meter (pH 2000, Shanghai, China), an ultrasonic cleaner (Kunshan, Suzhou, China), and a vortex mixer (Jianing, Zhejiang, China) were used during the process of CPE.

4.3. Sample Pretreatment. Samples of dandelion (Taraxacum mongolicum Hand.-Mazz.) were purchased from the local market (Zhengzhou, China). All samples were crushed and passed through no. 3 sieve. The dried dandelion powder (about 0.05 g) was accurately weighed and transferred into a 15 mL centrifuge tube, and then, 8.0 mL of Triton X-114 solution (5%, w/v) was added. After the resultant mixture was thoroughly mixed, ultrasonic extraction was performed for 30 min at room temperature. At last, the solution was centrifuged at 6000 rpm for 5 min and the supernatant was collected for the next step.

4.4. Cloud Point Extraction. CPE was carried out as the following procedure. 8.0 mL of the supernatant of the sample extract or resulting standard solution of FA, CA, and CLA containing Triton X-114 (5%, w/v) was placed in a 15 mL centrifuge tube. Then, the solution pH was adjusted to 3.5 by hydrochloric acid and sodium chloride was added into the solution with a ratio of 10% (w/v). After ultrasonication for 5 min, the mixture was incubated in a water bath at 60 °C for 30 min. Furthermore, the phase separation was accelerated by centrifugation at 4000 rpm for 5 min. At last, the nether water phase was removed, and the upper oil phase was kept and diluted with methanol to 1.0 mL. All assays were performed in triplicate.

4.5. HPLC Analysis. All HPLCs were performed with a C18 column (4.6×150 mm, 5 μm) at 30 °C. Methanol/acetonitrile/acetic acid (1%) (12.5:12.5:75, v/v) was selected as the mobile phase with a velocity of 0.8 mL min⁻¹. The diode array detector was set at a wavenumber of 320 nm for all analytes. The sample solution obtained by CPE was filtered by a 0.45 μm membrane, and 20 μL of the filtrate was injected into the HPLC system for analysis. This method was validated by the investigation of linearity, stability, precision, repeatability, and recovery of the sample.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c01768.

UV–vis spectra of FA, CA, and CLA; UV spectra (obtained by DAD) of the corresponding chromatogram peaks of FA, CA, and CLA from the dandelion sample; and content of CA in three batches of dandelion from different areas (n = 3, %) (PDF)
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Notes
The authors declare no competing financial interest.

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