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Retraction

Retracted: Electrochemical-Based Extraction, Separation, and Purification of Coumarin Compounds from *Trifolium chinensis*

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This article has been retracted by Hindawi following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of one or more of the following indicators of systematic manipulation of the publication process:

(1) Discrepancies in scope
(2) Discrepancies in the description of the research reported
(3) Discrepancies between the availability of data and the research described
(4) Inappropriate citations
(5) Incoherent, meaningless and/or irrelevant content included in the article
(6) Peer-review manipulation

The presence of these indicators undermines our confidence in the integrity of the article's content and we cannot, therefore, vouch for its reliability. Please note that this notice is intended solely to alert readers that the content of this article is unreliable. We have not investigated whether authors were aware of or involved in the systematic manipulation of the publication process.

Wiley and Hindawi regrets that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity.

We wish to credit our own Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

References

[1] Y. Zhu, W. Wang, R. Ruan, and J. Chen, "Electrochemical-Based Extraction, Separation, and Purification of Coumarin Compounds from *Trifolium chinensis*," *Journal of Chemistry*, vol. 2022, Article ID 6466271, 6 pages, 2022.

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Research Article

Electrochemical-Based Extraction, Separation, and Purification of Coumarin Compounds from *Trifolium chinensis*

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In order to deal with the problems of the unstable transformation process and volatile palatability of coumarin compounds in *C. chinensis*, a method for the extraction, separation, and purification of coumarins in Chinese medicinal materials based on electrochemistry was prepared. First, an electrochemical distance-independent localization mechanism is used to determine node locations and routing mechanisms, and an extraction method is designed to achieve automatic and accurate real-time collection, aggregation, and transmission of ever-growing data. Environmental information for *C. chinensis* is collected. On the basis of this method, according to the chemical properties of coumarin and the extraction rate of coumarin as an index, the two-phase solvent extraction method and macroporous adsorption resin method were used to separate and purify it, and the best separation was determined. The purification process achieves efficient purification of coumarin. The experimental results show that the retention rate of coumarin by ethyl acetate is 34.5–56.8% higher than that of the other three extractants. When the optimal process determined in this study is adopted, the coumarin adsorption rate is greater than 98% and the recovery rate is greater than 85%. The purity of samples with 50% coumarin content can be increased to more than 97%, which verifies the correctness and advancement of the separation and purification process in this study.

1. Introduction

The Internet of Things (IOT) combines electronic devices, computer peripherals, information-sharing technologies, modern networks, and wireless communications, among others. It can collaboratively monitor and record real-time experimental data of various devices through various integrated microsensors. Data are transmitted wirelessly in an ad hoc multihop network and sent to user terminals, identifying the global connections of global bodies, global computers, and human life [1]. With the continuous development and maturity of the Internet of Things technology, it will effectively solve the way that people obtain important ingredients in medicinal materials through manual grinding in the traditional pharmaceutical industry. In various control systems, the temperature sensor, humidity sensor, pH sensor, light sensor, ion sensor, biosensor, CO_2 sensor, and other equipment of the IoT system can detect physical properties such as temperature, relative humidity, pH value, light utilization rate, and soil. Deficient nutrients and CO_2 concentrations are automatically controlled when released by various devices or as automatic controls, so as to ensure a good and suitable separation and purification environment when extracting components of medicines [2].

Melilotus is an annual or biennial herb of the family Leguminosae. The root system is developed; the stem is erect and has many branches. There are three-leaf compound leaves; the leaflets are elliptic, and the margins are sparsely toothed. It has racemes axillary and butterfly shaped corolla. The pods are oval, with obvious reticulation. The seeds are kidney-shaped, yellow-green, and multiply. The development and utilization of *C. chinensis* have a wide range of development and utilization, and it is called "baby grass." The main development and utilization aspects include forage feed, soil improvement/fertilization, nectar plants, windbreak and sand fixation, water and soil conservation, spices, and medicinal purposes. Among them, the main medicinal ingredient is coumarin. How to determine the content of coumarin in *C. chinensis* and its separation and purification is becoming a hot topic in the world today.

In this study, an IoT-based coumarin extraction method was developed. By placing sensors with different functions in the area to be measured, it can monitor small climate changes, including temperature, humidity, moisture, and other growing environment information of *C. chinensis*. Accordingly, the growth of *C. chinensis* requires corresponding changes to the environment in a timely manner. The method can reduce the error of manual operation and manual measurement in the actual production process, reduce the cost of extraction, separation, and purification of *C. chinensis*, can realize the automatic and accurate real-time collection, aggregation, and transmission of *C. chinensis* growth environment information to the greatest extent, and design the structure, as shown in Figure 1. On the basis of this extraction method, according to the actual characteristics of coumarin in *C. chinensis*, it was separated and purified by the two-phase solvent extraction method and macroporous adsorption resin method, and finally, the efficient purification of coumarin was realized.

1.1. Literature Review. According to the research of Lu S. et al., coumarins, scientific name: 1,2-benzo α-pyrone, molecular weight 146.15, melting point 68–70°C, boiling point 303°C, volatile, composed of molten benzene. It is composed of α-pyridone ring and is a white solid, which is easily soluble in organic solvents such as ethanol and chloroform [3]. Coumarin has been shown to be important in plant metabolites and is found in essential oils such as cinnamon bark oil, cinnamon leaf oil, and lavender oil. Coumarin is also found in fruits (such as bilberries and cloudberries), green tea, and other foods such as chicory. Coumarin mainly comes from Rutaceae, Umbelliferae, Compositae, and Leguminosae, and some of them come from microorganisms [4]. Yusof et al. found that in plants, coumarin compounds exist in the form of free or glycosides and are mainly distributed in leaves, flowers, stems, and fruits [5]. The research of Sperotto et al. pointed out that the determination of coumarin content generally adopts the process of first separation and then quantification according to different concentrations of standard products. The principle of determination is mainly based on the UV absorbance of coumarin at a specific wavelength. Commonly used diagnostic procedures include thin layer chromatography scans, high performance liquid chromatography-mass spectrometry (HPLC-MS), supercritical fluid chromatography (SFC), and gas chromatography (GC) [6].

Thin layer chromatography was first proposed by Mishra et al., which is an adsorption chromatography that separates by successive adsorption, desorption, resorption, and redesorption. The thin layer scanning method mainly determines the content according to the absorption intensity of ultraviolet fluorescence [7]. Louisa et al. used thin layer chromatography to extract and isolate dicoumarin and determined its content by UV spectrophotometry. Thin layer chromatography is simple and low cost. However, the number of theoretical plates is relatively low, which will result in a certain quantitative deviation [8]. A liquid chromatography-mass spectrometry (LC-MS) method was prepared, which has the characteristics of small injection volume, high sensitivity, and fast analysis speed. It has long been the most common mainstream method, analytical method. LC-MS plays an irreversible role in the evaluation of coumarin quality and value. Fast and efficient analysis of complex in-process systems is without complete chromatographic separation. In recent years, this method has been widely used in biological, plant, food, and environmental samples [9]. Selivanova et al. used high performance liquid chromatography on the water extract of red sandalwood, quantitatively determined the content of coumarin using a photodiode array detector, and determined the active components of licorice in different traditional Chinese medicines by the HPLC-MC method, which mainly included Coumarin compounds [10]. Nguyen et al. compared classifications at different levels of the separation effect. In addition, in order to improve the resolution of the target compounds, a three-phase mobile phase gradient method (such as water: methanol: acetonitrile) was also used. The three-phase mobile phase gradient method (0.01 % phosphoric acid/water, acetonitrile, and methanol) was used to separate coumarin, furocoumarin, and methoxyflavonoids. There are separate isolated bases for each use [11]. With the development of analytical equipment, high performance liquid chromatography (UHPLC) is increasingly used for the separation of coumarins, as these techniques can improve synthesis, separation, and shorten detection time. For example, Dukare et al. separated and determined 22 coumarin derivatives in cosmetics using a UPLCBEHC18 column (2.1×100 mm, 1.7 mm). A total of 22 coumarin derivatives were separated in 5 minutes, saving time [12].

2. Research Method

2.1. Environmental Information Collection for Extraction of Coumarin Compounds from C. chinensis. The sensor nodes in the environmental information collection method for coumarin extraction are deployed in the monitoring area and constitute the basic unit of the Internet of Things. Sensor nodes can communicate with each other and form a node network through self-organization. Adjacent nodes in the network form clusters, and the cluster head node collects the data collected by each node in the cluster and then sends the compressed data to the sink node after data fusion. The sink node is responsible for the task of communication between the task management node and the sensor node and is generally served by a sensor node with strong energy or a router with wireless gateway capability [13].

The task management node is the data and instruction management center of the wireless sensor network (WSN), which generally consists of several servers [14]. The user configures and manages the sensor network through the task management node, publishes monitoring tasks, and collects monitoring data. The task management node can analyze and store the collected information and can issue instructions to the sensor node in real time. The position of each sensor node in the monitoring area can be obtained through GPS positioning or the node's own positioning algorithm. They collect information according to the instructions sent

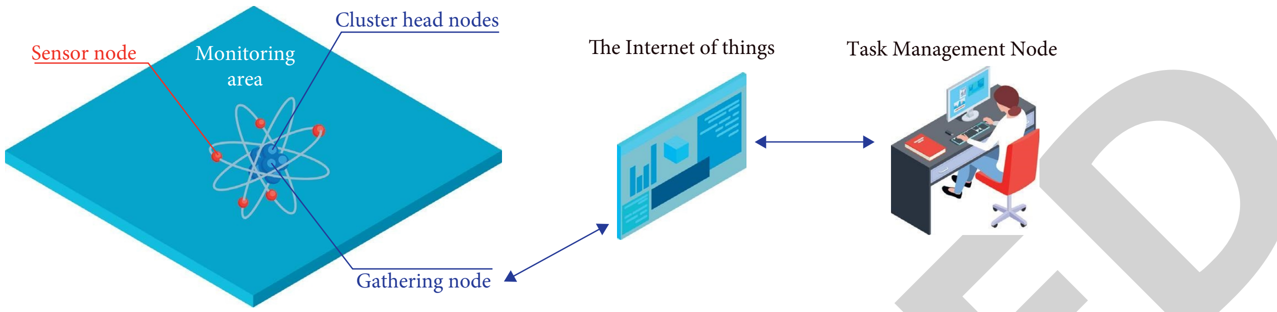

FIGURE 1: Environmental information collection, separation, and purification method framework for the extraction of coumarin compounds from *Trifolium chinensis*.

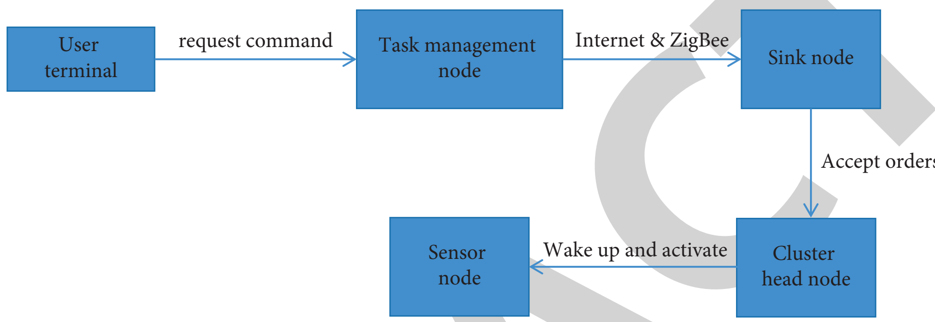

FIGURE 2: Workflow of the environmental information collection method for coumarin extraction.

by the task management node and send the collected information to the cluster head node. The cluster head node fuses the data and then sends it to the corresponding sink node. The data received by the sink node will be screened and sorted and then sent to the task management node through the Internet or communication satellites [15]. The working process of the environmental information collection method for coumarin extraction is shown in Figure 2.

The specific process is as follows. (1) The user sends a request command to query various indicators of the coumarin extraction environmental information through the task management node and transmits it to the sink node through the Internet and ZigBee network. (2) The sink node selects the cluster to be queried according to the specific requirements of the request command. After receiving the command, the cluster head wakes up and activates all nodes in the cluster to collect, store, and communicate data. The node collects data in time according to the command requirements and sends it to the corresponding cluster head node after digital-to-analog conversion. The cluster head fuses all the incoming data and sends the fused data back to the sink node. (3) The gathering node will screen and sort the obtained data and send it to the task management node through the external network. The task management node will feedback the collected data to the user and will store it in the coumarin extraction environment information database to provide a basis for future analysis and decision-making [16].

2.2. Separation and Purification of Coumarin. The components in the solid substance are dissolved in the solvent by the extraction method, and after solid-liquid separation, a liquid mixture containing multiple components is obtained, which is called the extraction solution, and then, various separation methods are used to separate the chemical components in the extraction solution. In this study, the two-phase solvent extraction method is mainly used for separation, and the macroporous adsorption resin method is used for purification [17]. According to the properties of coumarin and applicability of various separation and purification methods to coumarin, a separation and purification process route was drawn up for the ethanolic extract of coumarin, as shown in Figure 3.

The extract concentrate (extraction stock solution) of *C. chinensis* has been concentrated under reduced pressure to a certain concentration, add the extractant in a certain proportion, shake and stand for 30 minutes, take the organic phase, and repeat for several times. The organic phases were combined, concentrated under reduced pressure, and dried under vacuum at 50°C. The extracted product was weighed, and the coumarin content was measured by HPLC to calculate the coumarin content and coumarin retention rate in the extracted product. The extraction effect was evaluated by the coumarin content and coumarin retention rate in the extracted product. The definition formula is shown in the following formula [18]:

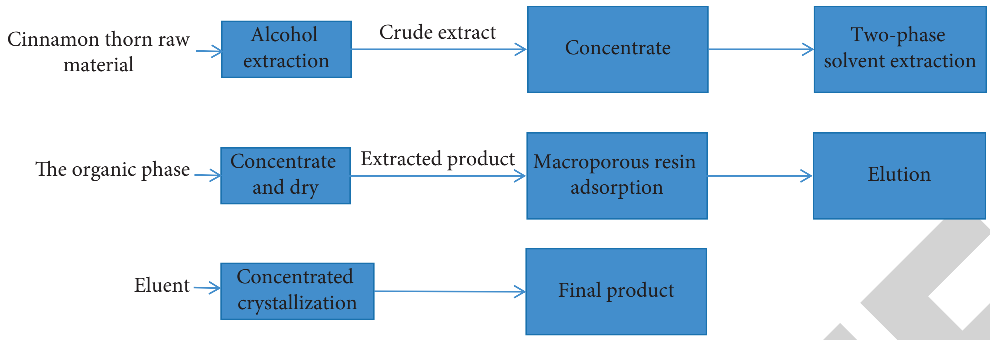

Figure 3: Extraction, separation, and purification process route of coumarin compounds in *C. chinensis*.

$$\text{The content of coumarin in the extract}\,(\%) = \frac{\text{Coumarin quality}}{\text{Extracted product quality}} \times 100. \tag{1}$$

$$\text{Coumarin retention rate}\,(\%) = \frac{\text{Coumarin quality} - \text{coumarin content} \times \text{extracted product quality}}{\text{The quality of coumarin in the extraction solution}} \times 100. \tag{2}$$

For the extraction effect, the highest content of coumarin in the extraction product and the high retention rate of coumarin are the best results, and these two indicators should be considered comprehensively in the evaluation [19].

The macroporous adsorption resin experiment is divided into the following steps. (1) Pretreatment: soak the unused resin in 95% ethanol for 24 hours, wash it with distilled water until there is no alcohol smell, and then use 5% hydrochloric acid and 5% hydrogen peroxide, respectively. Soak in sodium solution for 3 hours, wash with distilled water to neutral pH, and take part of the wet resin for use; the remaining resin is vacuum-dried at 333 K to constant weight for use. (2) Regeneration: the used resin is dynamically washed with 95% ethanol on a chromatography column. (3) Static adsorption and desorption: precisely weigh 0.5 g (dry weight) of the pretreated macroporous adsorption resin and add 50 ml of the extracted product to prepare an aqueous solution, Cinnamon officinalis sample solution, shake for 24 h at a shaking speed of 130 r/min in a shaker, measure the coumarin concentration in the supernatant, and calculate the resin adsorption capacity and adsorption rate. Discard the supernatant, wash the resin with water and dry it naturally, add 50 ml of 95% ethanol, desorb under the same conditions, measure the coumarin concentration in the desorption solution, and calculate the resin desorption rate. (4) Dynamic adsorption and desorption of resin: weigh 20 ml of pretreated wet screening macroporous adsorption resin and put it into a glass column (15×450 mm), use HPD300 resin to absorb coumarin, accurately weigh 0.5 g of HPD300 resin, and add 50 ml. The coumarin concentration was 2.0447 mg/ml in the sample solution, which was shaken in a shaker (298 K, 130 r/min), and the coumarin concentration in the supernatant was regularly detected [20].

3. Result Analysis

The equipment used in the experiment is given in Table 1.

Depressurize the *C. chinensis* extract to an extraction stock solution of 0.2 g/ml (raw material quality/volume of solution) and add different extractants at a ratio of 1 : 3 (volume of extract: volume of stock solution): chloroform, ethyl acetate ester, petroleum ether, and n-butanol, shake, after standing for 30 min, take the organic phase, and repeat 3 times. The organic phases were combined, compressed under reduced pressure, and dried under vacuum at 50°C. The coumarin content in the extracted product was determined by HPLC, and the coumarin retention rate was calculated [21]. The results are shown in Figure 4.

As can be seen from Figure 4, chloroform, ethyl acetate, and petroleum ether are used as extractants to extract coumarin, and the content of coumarin in the extraction product is similar, 34.5–56.8%, and serious emulsification will occur when the other three extractants are used as extractants. Ethyl acetate is recognized as a good extractant, with low cost, good biodegradability, and high extraction rate of lactones. Therefore, combined with the method of collecting environmental information for the extraction of coumarin compounds from *Trifolium chinensis* based on the Internet of Things [22], this study comprehensively considers the factors such as the good extraction effect, low toxicity, and good economy when ethyl acetate is used as the extraction agent, and ethyl acetate should be selected. Esters were used as extractants to separate and purify coumarin compounds in *C. chinensis*, and the dynamic breakthrough curve of HPD300 adsorption of coumarin obtained at the same time is shown in Figure 5.

It can be seen from the figure that 20 ml of wet HPD300 resin can handle 30 BV of sample solution without leakage under the optimized sample concentration (3.0 mg/ml) and flow rate (3.0 BV/h). When the effluent volume exceeds 30 BV, it begins to leak slowly. When the effluent volume is 33 BV, the coumarin concentration in the effluent reaches 1/10 of the sample concentration, which is called the breakthrough point [23]. When the effluent volume exceeds 33 BV, the leakage rate begins to increase sharply. When the

Table 1: Laboratory equipment table.

Name	Specification	Origin
Chinese herbal medicine grinder	FW135	Tianjin
Rotary evaporator	RE-52AA	Shanghai
Circulating water type multipurpose vacuum pump	SHB-B	Zhengzhou
Ultrasonic cleaner	KQ3200	Kunshan
Instant thermostatic heating magnetic stirrer	DF-101S	Shanghai
Shelf balance	JPT-10C	Shanghai
Electronic balance	BP211D	Shimadzu
Low speed centrifuge	LDA-2	Beijing

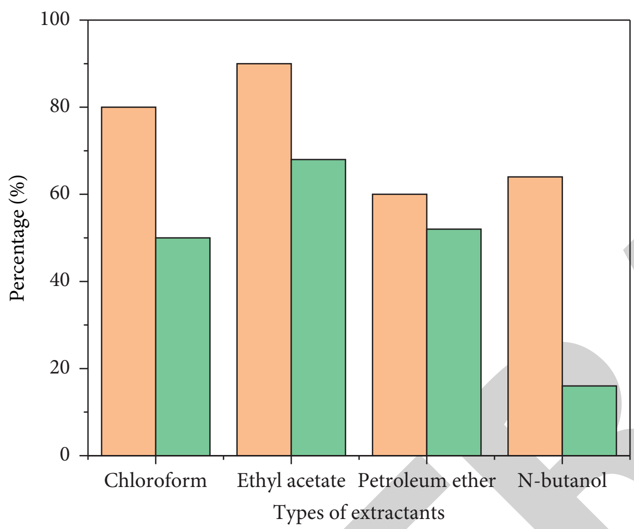

Figure 4: Effects of different extraction solvents on liquid-liquid extraction.

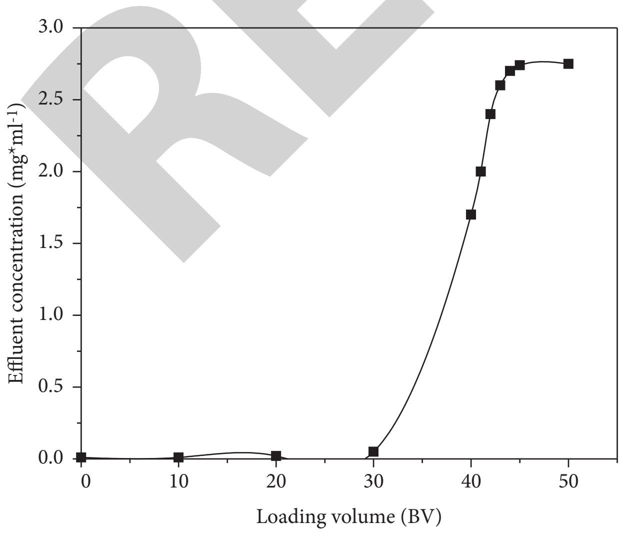

Figure 5: Dynamic breakthrough curve of coumarin adsorption by HPD300 resin.

effluent liquid agent reaches 42 BV, the effluent concentration is basically the same as that of the sample solution, and the resin reaches adsorption saturation, which is the saturation point of the resin [24, 25]. The experimental results show that under this process condition, 20 ml wet HPD300 resin can process 33 BV sample solution, its working adsorption capacity is 95.82 mg/ml wet resin, the coumarin adsorption rate is greater than 98%, and the recovery rate is greater than 85%. The purity of the samples with bean content of 50% increased to more than 97%, which verifies the correctness and advancement of the separation and purification process in this study.

4. Conclusion

As an important part of the new generation of information technology, the Internet of Things is used in many aspects such as data collection, transmission, processing, and business management, providing people with a new way to obtain and process information. Aiming at the characteristics of large amount of data collection in the collection method of coumarin extraction environmental information, combined with the advantages of Internet of Things technology, this study designed a coumarin extraction environmental information collection method and a coumarin separation and purification method based on the Internet of Things. The GEAR routing mechanism and the distance-independent positioning algorithm are adopted, thereby avoiding the energy consumed by nodes due to self-organizing network calculations. Meanwhile, the application of geographic location routing can more accurately locate the position of crops, which can effectively prolong the service life of the method. The macroporous adsorption resin method for the separation and purification of coumadin was determined. The experimental results show that 20ml wet HPD300 resin can treat 33BV sample solution under this process condition, and its working adsorption capacity reaches 95.82 mg/ml wet resin. The adsorption rate of coumarin was more than 98% and the recovery was more than 85%. The purity of the sample with 50% coumarin content can be increased to more than 97%, which verifies the correctness and advancement of the separation and purification technology in this paper.

There are still deficiencies in the research in this study. For the areas not covered, I would like to propose the following suggestions. (1) The pharmacological effects of the extracted and isolated high-purity coumarin products can be studied, and the anti-inflammatory and analgesic properties of the products can be studied. The pharmacological effects of detumescence and other aspects provide a basis for its development into a new drug. (2) Industrial scale-up experiments can be carried out on the extraction and separation of coumarin, so as to realize industrialization and bring economic effects as soon as possible and social benefits.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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