Research Article

Effect of Pretreatment on Detection of 37 Pesticide Residues in Chrysanthemum indicum

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This study aimed to develop a method, followed by gas chromatography-mass spectrometry, for detecting 37 pesticides in Chrysanthemum indicum (C. indicum) and investigating the decrease in the matrix-induced enhancement effect. The influence of QuEChERS extraction and matrix solid-phase dispersion (MSPD) on the recovery and matrix effect (ME) was compared. The cleanup sorbents, volume and type of solvent, and treatment time were optimized. The accuracy (as recovery), precision (as relative standard deviation, RSD), linearity, limit of quantitation, and limit of detection were determined. The recoveries at the three levels using mixed standard solution ranged between 76% and 120% with RSD ≤ 15%, and 76% and 120% with RSD ≤ 11% for MSPD and QuEChERS extraction, respectively. The results suggested that the ME for 21 pesticides was in the range of 80%–120% after MSPD and 15% after QuEChERS extraction. QuEChERS extraction was simpler and faster than MSPD. This methodology was applied in the analysis of 27 C. indicum samples; phorate was most frequently detected (63.0% of the sample).

1. Introduction

Chrysanthemum indicum (Ye Ju Hua) is a medicinal herb with anti-inflammatory, analgesic, antioxidant, antibiotic, and other pharmacological effects. It is widely distributed and planted in China. It is very popular in Asian countries, such as China, Korea, and Japan, as traditional medicine, herbal tea, and functional foods [1].

With the implementation of standardized cultivation of Chinese herbal medicines, many herbs, including C. indicum, have established planting bases. Pest control is very important to improve the yield and quality of herbs. Good agricultural practices (GAP) attach great importance to the protection of the ecological environment in the production of Chinese medicinal materials and have strict usage norms and safety standards for the control of pesticide residues, such as selecting the rational pesticide varieties, application period, and mix pesticides. During the growth period of C. indicum, the flowers and aerial parts of C. indicum can have some diseases, such as spot blight, blight, and downy mildew; the pests are mainly aphids and beetles. Omethoate, dicofol, metalaxyl, carbendazim, chlorothalonil, methyl triazine, mancozeb, carbamic acid powder, and chlordimeform are relatively common pesticides used to protect flowers and plants and maintain the production yield. The pesticides commonly used in Chrysanthemum indicum are low-toxic and high-efficiency pesticides, but it cannot be ruled out that their pesticides will...
not exceed the standard. Therefore, some residues of pesticides should remain in herbal products [2]. The residues of pesticides are closely related to the soil environment.

Pesticide residues in herbs are pesticide precursors, degradants, toxic metabolites, and impurities that remain in the medicinal parts after the use of pesticides in the growing environment, planting, processing, and storage [3]. According to the chemical structure, pesticides are divided into organophosphorus, organochlorine, pyrethroid, and carbamate. Organochlorine pesticides use benzene or cyclopentadiene as raw materials, such as hexachlorocyclohexane (BHC) and dichlorodiphenyltrichloroethylene (DDT). These pesticides are chemically stable and easily accumulate in living organisms. The long-term accumulation of DDT in the human body can cause immune system dysfunction and genetic and developmental toxicity; DDT can even be carcinogenic and teratogenic. Organophosphorus pesticides are currently widely used types of pesticides, including dichlorvos, dimethoate, and phosphorothioate, thionazin, phorate, sulfotep, diazinon, disulfoton, endrin, \( \alpha \)-BHC, \( \beta \)-BHC, \( \delta \)-BHC, \( \alpha \)-endosulfan, \( \beta \)-endosulfan, \( \delta \)-endosulfan, and \( \delta \)-endosulfan sulfate, which are also widely used in medicine. However, in previous studies, the interactions of pesticides with the matrix were observed in the detection of pesticide residues in \( C. \ indicum \) using GC-MS. In this study, 37 types of pesticides were analyzed by GC-MS, and the MSPD and QuEChERS pretreatment methods were compared in terms of purification and ME. We aimed to establish a simple, rapid, and efficient method to analyze pesticide residues in \( C. \ indicum \).

2. Materials and Methods

2.1. Reagents and Material. The HPLC grade waters were purified using the Milli-Q system (Millipore, USA). Acetonitrile, acetone, hexane, and ethyl acetate were obtained from Merck Company (Germany). Sorbent kits were purchased from the Agela Technologies (Tianjin, China), and QuEChERS commercial extraction bag (magnesium sulfate, NaCl 1 g, and sodium citrate dibasic) and purification bag (primary secondary amine (PSA) + graphitizing of carbon black (GCB), PSA + octadecylsilane chemically bonded silica (C18)) were purchased from the ANPEL Laboratory Technologies (Shanghai, China). PSA, GCB, C18 amino (NH2), silica, and florisil from the ANPEL Laboratory Technologies (Shanghai, China) were used as sorbents.

Analytical grade \( \alpha \)-BHC, \( \gamma \)-BHC, heptachlor epoxide, aldrin, \( \beta \)-BHC, \( \delta \)-BHC, \( \alpha \)-endosulfan, \( \beta \)-endosulfan, dieldrin, endrin, \( mp `\)-DDE, \( \beta \)-endosulfan sulfate, quinazolop ethyl, and pentachloronitrobenzene were obtained from ANPEL Laboratory Technologies (Shanghai, China) (Supplementary Table 1). Alachlor, heptachlor exo-epoxide, pendimethalin, tetraclifton, famphur, \( \alpha, \alpha, \alpha \)-triethylphosphorothioate, thionazin, phorate, sulfotep, diazinon, disulfoton, dimethoate, rosette, metalaxyl, chlorpyrifos, methyl parathion, fenfuran, bromophos, parathion, quinalphos, procymidone, and profenofos were purchased from Sigma-Aldrich (Germany) (Supplementary Table 1). Each stock standard solution was prepared at various concentrations in acetone and stored in the dark at a temperature less than –20°C. Pesticides were divided into groups A and B according to their characteristics and retention time. The mixed standard working solution of each group was prepared in acetone (about 10 mg/L) and stored in the dark at a temperature less than –20°C.
2.2. Samples. A total of 27 Chrysanthemum indicum samples were collected from four hospitals (Guangzhou, China), nine pharmacies (Guangzhou, China), four medical markets (Guangzhou, China; Guangxi, China; Hebei, China; and Anhui, China), and two planting bases (Hubei, China).

2.3. Pretreatment Methods. The pretreatment methods were based on the previously reported methods, and the cleanup sorbent and cleanup solvent were optimized. The main steps of the MSPD method included mixing the sample with the cleanup sorbent, filling the column, extracting, concentrating, and reconstituting (Supplementary Figure 1). The main steps of QuEChERS included two parts, namely, extraction and cleanup.

2.4. Gas Chromatography-Mass Spectrometry Analysis. The analysis was performed using gas chromatography-mass spectrometry (GC-MS). An Agilent 7890B GC system was equipped with Agilent 5977A MSD. The chromatographic separation was performed using the Agilent DB-1701 column with a length of 30 m × 1D 0.25 mm × and film thickness of 0.25 μm. The flow of carrier gas helium was 1.3 mL/min. The oven temperature was as follows: 50°C (1 min) and 30°C min\(^{-1}\) to 160°C, 4°C min\(^{-1}\) to 200°C, 3°C min\(^{-1}\) to 230°C (2 min), 2°C min\(^{-1}\) to 250°C, 20°C min\(^{-1}\) to 270°C, and 5°C min\(^{-1}\) to 300 (5-min hold), with a total run time of 48.5 min. The injector temperature was 230°C. Injection mode was splitless mode. The MS was operated in the electron ionization mode with a transfer line temperature of 250°C and an ion source temperature of 230°C.

2.5. Method Validation. The method validation was performed using the following parameters, namely, accuracy (expressed as recovery), precision (expressed as RSD), linearity (expressed as \(R^2\)), limit of detection (LOD), and limit of quantification (LOQ).

The accuracy was expressed as recovery. Three different levels were analyzed (0.4 mg/kg, 2 mg/kg, and 10 mg/kg), with three replicates for each level. The linearity was studied by analyzing the mixed standard solution at five concentration levels. The range of analyzed concentrations was 0.02–10.0 mg/L.

2.6. Matrix Effect. Initially, two pretreatment methods were evaluated in terms of matrix effect (ME) by comparison between the areas of the standard in the extract and the standard in the solvent, shown by the following equation: ME (%) = (area of the standard in the matrix/area of the standard in the solution) × 100. When the ME value is close to 100%, there are no influences by the matrix. When the ME value is out of the range 80%–120%, it means that the matrix effect is significant. [25]

2.7. Qualitative and Quantitative Detection. The qualitative analysis of pesticide residues referred to the method for the determination of pesticide residues (Chinese Pharmacopoeia 2015, fourth edition). During sample testing, if the retention time of the detected peak was consistent with the reference, the qualitative ions appeared in the mass spectrum after subtracting the background. Moreover, the relative abundance of the sample was consistent with the reference (relative abundance >50%, deviation allowed up to ±20%; relative abundance 20%–50%, deviation allowed ±25%; relative abundance 10%–20%, deviation allowed ±30%; and relative abundance <50%, deviation allowed ±50%). Then, the presence of the pesticide in the sample was determined. The internal standard method was used for the quantitative analysis, and the internal standard was heptachlor epoxide.

3. Results and Discussion

3.1. Pesticide Selection and Grouping. A total of 37 representative pesticides were selected for analysis, including pesticides and their metabolites used in planting bases and pesticides restricted or prohibited in China and other countries, which are not easily metabolized or highly toxic. Based on the relevant literature and the actual planting situation of the base, this study summarized pesticides commonly used in the control of C. indicum diseases and pesticides with more domestic dosage forms; 37 pesticides were used as the detection indicators. These pesticides were divided into two groups (groups A and B) based on the following principle: (1) retention time: pesticides were grouped with close retention times and overlaps to avoid mutual interference between compounds and (2) physical and chemical properties: group A mainly included organochlorine pesticides, while group B mainly included organophosphorus pesticides (Table 1).

3.2. Chromatographic Analysis. The ions for each compound were obtained by GC-MS analysis in full-scan (Figure 1) and selective ion monitoring (SIM) modes (Table 1). The standard solutions at a concentration of 10 mg/L of pesticides were prepared in acetone. The sensitivity of some pesticide reference materials was related to chromatographic conditions. The examination of the injector temperature, transfer line temperature, and flow revealed that the injector temperature had a great influence on endosulfan sulfate, quizalofop ethyl, profenofos, and famphur.

3.3. Optimum Method of MSPD. The MSPD processing method was as follows: 0.5 g C. indicum sample was grounded for 3 min in the agate mortar with 1 g of single sorbent or 0.5 g of mixed sorbents. It was filled in the column (10 mL tube, 15.8 mm × 88 mm) with anhydrous sodium sulfate at a 2 cm height of the extraction column, which was prerinsed with 4 mL of the extraction solvent. Before the liquid level reached the top of anhydrous sodium sulfate, 25 mL of extraction solvents (n-hexane : acetone = 4 : 6) was added to the extract. The elution solvent was concentrated to
Table 1: Qualitative and quantitative ions of group A and group B pesticides.

| Name                | Retention time (T/min) | Qualitative ions | Qualitative ions | Name                | Retention time (T/min) | Quantitative ion | Qualitative ions |
|---------------------|------------------------|------------------|------------------|---------------------|------------------------|------------------|------------------|
| α-BHC               | 12.446                 | 181              | 183 217 219      | γ-BHC               | 13.971                 | 181              | 183 111 219      |
| γ-BHC               | 13.971                 | 181              | 111 219          | Triethylphosphorothioate | 5.366                 | 198              | 121 97 93       |
| Heptachlor epoxide  | 14.664                 | 100              | 272 274 270      | Thionazin           | 10.668                 | 97               | 96 107 143      |
| Aldrin              | 15.69                  | 66               | 263 91 265       | Phorate             | 11.82                  | 75               | 121 97 260      |
| Alachlor            | 16.575                 | 45               | 160 188 146      | Sulfotep            | 11.973                 | 322              | 97 202          |
| β-BHC               | 17.175                 | 181              | 183 219 109      | Pentachloronitrobenzene | 12.912                 | 267              | 142 214 249     |
| δ-BHC               | 18.121                 | 181              | 183 219 217      | Diazinon            | 13.305                 | 137              | 179 152 199     |
| Heptachlor exo-epoxide | 18.98                 | 253              | 255 81 351       | Disulfoton          | 14.058                 | 88               | 89 97           |
| Pendimethalin       | 19.586                 | 252              | 162 253 281      | Dimethoate          | 15.676                 | 87               | 93 125 79       |
| α-Endosulfan        | 19.972                 | 195              | 241 237 239      | Ronnel              | 16.116                 | 285              | 287 125 109     |
| p,p′-DDE            | 21.111                 | 246              | 318 248 316      | Metalaxyl           | 17.355                 | 206              | 132 160 146     |
| Dieldrin            | 21.757                 | 79               | 81 82 263        | Chlorpyrifos        | 17.481                 | 197              | 97 199 314      |
| Endrin              | 22.743                 | 263              | 81 265 261       | Methyl parathion    | 17.661                 | 109              | 125 263         |
| m,p′-DDD            | 25.141                 | 235              | 237 165          | Fenthion            | 18.287                 | 278              | 125 109 169     |
| β-Endosulfan        | 25.301                 | 195              | 237 207 241      | Bromophos           | 18.467                 | 331              | 329 125 333     |
| Endosulfan sulfate  | 29.937                 | 123              | 272 183 237      | Heptachlor exo-epoxide | 18.985                 | 253              | 255 81 351      |
| Tetrachlorfuran     | 33.773                 | 159              | 111 227 229      | Parathion           | 19.456                 | 109              | 97 291 139      |
|                      |                        |                  |                  | Quinalphos          | 20.232                 | 146              | 157 118 156     |
|                      |                        |                  |                  | Procyomidone        | 22.239                 | 96               | 283 67 285      |
|                      |                        |                  |                  | Profenofos          | 22.46                  | 139              | 97 207 206      |
|                      |                        |                  |                  | Fampur              | 30.968                 | 218              | 125 93 217      |
|                      |                        |                  |                  | Quizalofop ethyl    | 39.729                 | 299              | 372 163 243     |

Figure 1: Continued.
dryness, followed by the addition of 1 mL of n-hexane for dissolution, and filtered for GC-MS analysis. The results of the purification effect of a single sorbent on *C. indicum* indicated that GCB had a better effect on pigment removal; both GCB and C18 had strong adsorption on some pesticides, such as heptachlor epoxide and endrin (Figure 2). When using the NH2+C18 mixed sorbent, the recovery of pesticides in group B was mostly in the range of 75%–125%, but the recovery of 9 of 17 pesticides in group A was less than 75%; the recovery of 2 pesticides was higher than 125% (Figure 2(c)). When using the PSA + NH2 mixed sorbent, the recovery of only three pesticides was less than 75% (Figure 2(a)). Therefore, PSA + NH2 mixed sorbent was chosen as the adsorbent filler.

Furthermore, the ratio and amount of PSA and NH2 and the time for mixing of the cleanup sorbent with the sample powder also had a significant influence on the purification effect. Based on the recovery, a two-factor and four-level orthogonal experiment was designed to evaluate the optimal ratio of PSA and NH2, and the results were verified. The amount of the absorbent had a great influence on the recovery of organochlorine pesticides (group A) but had little effect on organophosphorus pesticides (group B). According to the orthogonal test results, an appropriate increase in the amount of cleanup sorbents increased the recovery; when the sorbent combination was 200 mg PSA + 200 mg NH2, the best purification effect was achieved and verified. The recoveries of 37 pesticides ranged from 76.24% to 118.76% with RSD <10.0% (Figure 3). As shown in Figure 4(b), the repeated mixing of the adsorbent and the sample by grinding improved the extraction and purification effect. If the grinding time was too long, some pesticides, such as

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**Figure 1**: Total ion chromatograms of groups A (a), group B (b), and *C. indicum* sample (c) in the GC-MS full-scan mode.
Figure 2: Effect of different cleanup sorbent combinations on recovery.
quizalofop ethyl, were decomposed to reduce the recovery. Overall, it was most suitable to mix the sample with the adsorbent for 2 min.

Pretreatment of pesticide residues in herbs is a process of extracting and enriching trace components. The ideal extraction solvent should be able to extract pesticides as characteristically as possible. Different types of samples have different requirements for extraction solvents; the choice of extraction solvent is a very important step in the pretreatment method. Therefore, in this study, four kinds of solvent combinations were used for extraction, which were acetonitrile (A), n-hexane: ethylacetate (9:1) (B), n-hexane: acetone (9:1) (C), acetone:acetonitrile (3:7) (D), acetone: ethyl acetate (1:1) (E), and n-hexane: acetone (4:6) (F). The extraction solvents A, B, and C had a large interference with \( \gamma \)-BHC (recovery >140%), while the extraction effect of B and C on dimethoate was very poor (undetectable). When using E as the extraction solvent, the recovery of most pesticides was less than 75%. The recovery of 16 pesticides, including heptachlor epoxide, was less than 75% using solvent combination D, while the extraction effect of F on dieldrin and endrin was poor. In summary, n-hexane: acetone (4:6) (F) was a suitable extraction solvent for C. indicum samples. The amount of the extraction solvent was also one of the factors affecting the recovery. Increasing the amount of the extraction solvent increased the recovery to a certain extent. However, excessive use not only caused unnecessary waste but also led to an increase in the number of transfers during the recovery of the solvent. In this method, 15 mL of the extraction solvent was sufficient to elute completely, and the pesticide recovery was in the range of 75%–125% (Figure 4(a)).
3.4. Optimum Method of QuEChERS. Despite the simplicity of the experiment, the effectiveness of the QuEChERS method depends on the nature of the target analyte, matrix composition, equipment, and analytical techniques available in the laboratory. Therefore, when developing the QuEChERS protocol, several parameters that affect the extraction efficiency need to be considered and optimized. [14]. The initial steps of QuEChERS extraction were as follows: 1.0 g of the sample was accurately weighed and placed in a 50 mL centrifuge tube. Then, 10 mL of the extraction solvent was precisely added and shaken at 1,000 rpm for 4 min. Furthermore, the extraction package was added, shaken at 1,000 rpm for 4 min, and centrifuged at 5,000 rpm for 5 min. The supernatant was transferred to a purification tube, shaken at 1,000 rpm for 4 min, and centrifuged at 5,000 rpm. The supernatant was filtered for GC-MS analysis.

The cleanup sorbents, volume and type of solvent, and treatment time were optimized. Six kinds of cleanup sorbents were explored (Supplementary Table 2), of which No. 1, No. 5, and No. 6 had higher adsorption to profenofos (Figure 5(a)). The interference of No. 4 and No. 6 with 1,0-BHC was relatively large (Figure 5(a)). When the No. 2 cleanup sorbent was used, the recovery of pesticides ranged between 82.07% and 119.95%, indicating that it had a good purification effect.

Next, the extraction effect of acetonitrile (AcN) and a solution with different concentrations of HAc (0.1%, 0.2%, and 0.4%) was investigated. Thus, 0.1% HAc was added to AcN to increase the recovery of pesticides (76.92%–115.41%). The recovery of a few pesticides, such as metalaxyl and disulfoton, reduced when the acid concentration was too high (Figure 5(b)). Different volumes (8, 10, and 15 mL) of the extraction solvent had a certain influence on the extraction effect; 10 mL was found to be sufficient.

The increase in extraction time had a great influence on the recovery of some organophosphorus pesticides. When the extraction time was 2 min, the recovery of disulfoton and methyl parathion was 66.69% and 138.47%, respectively (Figure 5(c)). The purification time had minimal effect on the recovery rate (Figure 5(d)). After adding the extraction solvent to the sample, the shaking time was 1 min. After adding the adsorbent, the same shaking was performed for 1 min.

3.5. Method Validation. The optimal conditions for the application of MSPD and QuEChERS to detect 37 pesticides in C. indicum were as follows. (1) The optimal conditions of MSPD were that 0.5 g C. indicum sample was taken in the agate mortar, 0.2 g PSA and 0.2 g NH2 were added, and the mixture was grounded for 2 min. The column was filled, followed by the addition of anhydrous sodium sulfate to an extraction column of about 2 cm height, and prerinised with 4 mL of n-hexane : acetone (4 : 6). Before the liquid level reached the top of anhydrous sodium sulfate, 15 mL of n-hexane : acetone (4 : 6) was added to the extract, the elution solvent was concentrated to dryness, and 1 mL of n-hexane was added for dissolution. The filtrate was used for GC-MS analysis. (2) The optimal conditions of QuEChERS were to accurately weigh 1.0 g sample in a 50 mL centrifuge tube. Then, 10 mL of 1% HAc–AcN was added and shaken at 1,000 rpm for 1 min.

The extraction bag (4 g MgSO4 and 1 g NaCl) was added, shaken at 1,000 rpm for 1 min, and centrifuged at 5,000 rpm for 5 min. The supernatant was taken, transferred to a purification tube, shaken at 1,000 rpm for 1 min, and centrifuged at 5,000 rpm. The supernatant was taken and filtered for GC-MS analysis.

Different parameters, such as accuracy, precision, linearity, LOD, and LOQ, were determined (Supplementary Tables 3 and 4). The recovery and precision of the method for all pesticides at three levels (0.4 mg/kg, 2 mg/kg, and 10 mg/kg) in three replicates were determined. The recoveries at the three levels using mixed standard solution ranged between 76% and 120% with RSD ≤15%, and 76% and 120% with RSD ≤11% for MSPD and QuEChERS extraction, respectively (Supplementary Tables 3 and 4).

3.6. Matrix Effect. In the analysis of pesticide residues, the type and content of the matrix affect the recovery. During the gas chromatography injection process, the matrix reduces the decomposition of thermally unstable pesticides and reduces the injection of polar pesticides at the injection port. That is to say, the sample matrix increases the amount of pesticide to be analyzed that enters the column from the inlet. The calibration using pure solvent standard solutions is that ME can cause deviations in pesticide residue analysis results and recovery calculations. Several methods are used to compensate for ME, such as matrix purification. The most effective method is calibration with matrix-matched standard solutions or calibration with analytical protection agents [26, 27].

Blank matrices of two extraction methods were prepared to compare the effects of the two pretreatment methods on ME, and a mixed standard solution was added to the matrix. The peak area of the standard in the solvent and the peak area of the matrix mixed standard were measured separately to calculate ME. After MSPD treatment, the ME value of 21 pesticides exceeded the range of 80%–120%, while after QuEChERS treatment, the ME value of only 15 pesticides exceeded 80%–120% (Figure 6).

In the C. indicum matrix, most of the pesticides exhibited matrix enhancement effects, especially organochlorine pesticides. MSPD and QuEChERS methods compensated the matrix enhancement effects to a certain extent using matrix-matched standard solutions, which was more obvious in the QuEChERS method.

3.7. Comparison of MSPD and QuEChERS. When selecting the cleanup sorbent for the MSPD treatment method, the cleanup sorbent containing GCB (including GCB + C18, GCB + PSA, and GCB + florisil) had a good removal effect on pigments and a strong adsorption capacity for pesticides, resulting in low recovery. Most of the improvements associated with the QuEChERS method include the optimization of amount and combinations of solvents and salts, according to the chemical nature of target analytes. These
Figure 5: Effect of extraction conditions on pesticide recoveries in groups A and B (a) pesticide recovery for different cleanup sorbents; (b) pesticide recovery for different extraction solvents; (c) pesticide recovery for different extraction times; and (d) pesticide recovery for different purification times.

Figure 6: Matrix effects of different pretreatment methods.
Table 2: Detection rates of pesticides in 27 Chrysanthemum indicum samples (∗ indicates that the limit is exceeded).

| Name                        | Detection rate/% | Content /mg·kg⁻¹ |
|-----------------------------|------------------|------------------|
| Phorate                     | 62.96            |
| Profenofos                  | 59.26            |
| Aldrin and dieldrin         | 47.04            |
| o,o,o-Triethylphosphorothioate | 33.33        |
| Dimethoate                  | 29.63            |
| Thionazin                   | 14.81            |
| Methyl parathion            | 14.81            |
| Procymidone                 | 14.81            |
| Fenfluthrin                 | 11.11            |
| BHC                         | 11.11            |
| Disulfoton                  | 11.11            |
| Pentachloronitrobenzene     | 7.41             |
| Alachlor                    | 7.41             |
| Endrin                      | 7.41             |
| Sulfotep                    | 7.41             |
| Quinalphos                  | 3.7              |
| Pendimethalin               | 3.7              |
| Diazinon                    | 3.7              |
| Parathion                   | 3.7              |

4. Conclusions

In this study, two sample pretreatment methods (MSPD and QuEChERS) were evaluated for detecting pesticide residues in *C. indicum*, reducing the influence of matrix to improve the accuracy of detection. The results of the comparison of the two sample processing methods (MSPD and QuEChERS) show that QuEChERS is better than MSPD, but the price is also more expensive than MSPD. Compared with the extraction solvents commonly used in laboratories, such as n-hexane, acetone, and acetonitrile, acetonitrile is less toxic. QuEChERS method selects acetonitrile as the extraction solvent. Adding acetic acid according to the nature of the sample is beneficial to the recovery of pesticides. Anhydrous magnesium sulfate has a strong dehydration capacity, which can meet the rapid dehydration needs of QuEChERS. PSA can absorb impurities such as sugars and fatty acids in the sample to achieve the purpose of purification. Both methods have their own advantages. If we want to apply the method to actual sample detection, we need a simpler and faster method. A GC-MS method for detecting pesticide residues in *C. indicum* was developed. The accuracy, precision, linearity, and LOQ results showed that the proposed method was feasible and applicable to detect 37 pesticide residues in *C. indicum* samples. The calibration using matrix-matched standard solutions effectively compensated for the ME of pesticide residue analysis in *C. indicum*. As an important food and dual-use product, *C. indicum* is widely used by consumers in their daily lives, especially in China. Therefore, it is necessary to establish a faster and more effective method for analyzing pesticide residues in *C. indicum*, a matrix to ensure a healthy product to its consumers.

Data Availability

All the data used during the study appear in the submitted article.

Conflicts of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Authors’ Contributions

LU Xiao-ying completed data analysis and drafted the manuscript. OUYANG Yan-qin designed and executed the research. ZENG WEI-ya, LIN Cui-qing, XIAO Lu-hua, and LUO Gui-hua participated in the experiment. ZHAN Ruoting and YAN Ping conceived the study, participated in its design and coordination, and helped to draft the manuscript. All the authors read and approved the final manuscript.
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Supplementary Materials

Some figures and tables are included in the supplementary file. (Supplementary Materials)

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