Simultaneous Determination of Multiple Chemical Residues in Agricultural and Livestock Products and Dietary Risk Assessment Based on Optimized Pretreatment Method

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Abstract An improved QuEChERS pretreatment combined with HPLC-MS/MS and ICP-MS was developed for simultaneous quantification of pesticide residues, veterinary drug residues and heavy metals in cereals, fruits, vegetables, meat and aquatic products. All analytes displayed good linearity with $R^2 > 0.999$; recoveries ranged from 86.2% to 108.5%, and intra-/inter-day precisions (RSD) were less than 4.5%. A total of 320 real market samples were tested, with an overall qualification rate of 97.19%. This optimized approach is rapid, cost-effective and high-throughput, applicable to routine large-scale monitoring in food inspection laboratories, and provides fundamental data for regional food safety risk management.

Key words Food safety; Multi-residue detection; Modified QuEChERS; Matrix effect; Dietary risk assessment; Market surveillance

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Food safety is an important public health issue closely related to human life quality and social stability[1]. With the continuous expansion of modern agricultural cultivation scale, intensive livestock breeding and standardized food processing industry, the use of pesticides, veterinary drugs and chemical additives has become increasingly common in the food supply chain. However, unreasonable application, irregular medication interval and environmental migration inevitably lead to residual chemical pollutants in agricultural products, livestock products and daily diet. Long-term low-dose dietary exposure may cause cumulative toxic effects, including endocrine disturbance, liver and kidney damage, and potential carcinogenic risks, which seriously threaten consumer health and restrict the high-quality development of the food industry.

The QuEChERS pretreatment technique[2] has been widely used in food residue detection due to its advantages of simplicity, rapidity, low reagent consumption and environmental friendliness. However, the classic QuEChERS formula still has deficiencies in removing complex interfering substances in high-oil and high-pigment food samples, resulting in significant matrix effects in actual detection. In order to solve the above problems, this study optimized the extraction solvent ratio, salt-out system and purification adsorbent dosage to establish an improved pretreatment method suitable for multi-category food samples[3]. Combined with instrumental quantitative analysis, a synchronous detection system for various pesticide residues, veterinary drug residues and heavy metal pollutants was constructed[4].

Furthermore, based on the monitoring data of actual commercial food samples in local market, this study carried out dietary exposure risk assessment for different age groups, systematically analyzed the pollution characteristics and potential health risks of food chemical residues in the region[5]. The research results aim to provide technical support for grassroots food safety supervision, risk early warning and daily batch detection, and provide reference data for formulating targeted food safety control strategies[6].

1 Materials and methods

1.1 Instruments and reagents High performance liquid chromatography tandem mass spectrometry (HPLC-MS/MS, Thermo Fisher Scientific, USA) and inductively coupled plasma mass spectrometry (ICP-MS, Agilent, USA) were used for quantitative detection of organic residues and heavy metal elements, respectively. High-speed refrigerated centrifuge, vortex oscillator, ultrapure water purification system and analytical balance were applied for sample pretreatment and preparation.

Pesticide standard substances, veterinary drug standard substances and heavy metal standard stock solutions were all purchased from national standard reagent center with purity higher than 98%. Acetonitrile, methanol and formic acid were chromatographic grade; anhydrous magnesium sulfate, sodium chloride, PSA adsorbent and C_{18} adsorbent were all analytical grade. Ultrapure water (18.25 MΩ · cm) was used throughout the experiment.

1.2 Sample collection A total of 320 batches of common food samples were randomly collected from local supermarkets, farmers' markets and catering distribution centers, covering typical food matrices including vegetables, fruits, cereals, pork, beef and aquatic products. All samples were sealed, numbered and stored at 4 ℃ for short-term preservation and −20 ℃ for long-term pres-

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ervation. The sample information, including sampling location, variety and production batch, was recorded in detail to ensure the representativeness and comprehensiveness of monitoring data.

1.3 Traditional and optimized QuEChERS pretreatment methods

1.3.1 Traditional QuEChERS method. 5.0 g of homogenized food sample was accurately weighed into a centrifuge tube, and 10 ml of acetonitrile solution was added to shake vigorously for 3 min. Then, anhydrous magnesium sulfate and sodium chloride were added for salting-out extraction. After high-speed centrifugation, the supernatant was taken and purified by traditional single adsorbent, and finally filtered through a 0.22 μm of organic filter membrane for machine detection.

1.3.2 Optimized pretreatment method. Based on the traditional extraction system, the solvent proportion and purification conditions were optimized. The mixed extraction solvent was configured according to the optimized ratio to improve the extraction efficiency of polar and weakly polar residues. The composite adsorbent system of PSA and C_{18} was adjusted according to different food matrices to remove protein, fat and pigment interference effectively. The centrifugation speed and time were optimized to reduce impurity precipitation and improve supernatant purity. The optimized pretreatment process significantly reduced matrix effect and improved the stability of recovery rate for different kinds of food samples.

1.4 Instrument detection conditions For organic pesticide and veterinary drug residues, HPLC-MS/MS multi-reaction monitoring (MRM) mode was adopted. Mobile phase was acetonitrile-0.1% formic acid aqueous solution with gradient elution procedure. The column temperature, flow rate, injection volume and ion source parameters were set according to optimized conditions to realize rapid separation and accurate qualitative and quantitative analysis of multiple residues.

For heavy metal pollutants, ICP-MS standard mode was used to detect trace elements. The tuning solution was used to optimize instrument sensitivity, resolution and stability, and the standard curve method was adopted for quantitative determination.

2 Results

2.1 Optimization results of sample pretreatment conditions

In this study, key pretreatment parameters including extraction solvent ratio, salting-out dosage, centrifugation condition and compound adsorbent proportion were systematically optimized to reduce matrix interference and improve extraction efficiency. Compared with the traditional single acetonitrile extraction system, the optimized mixed solvent system exhibited higher extraction recovery for both polar and non-polar residual targets.

For high-fat matrices such as meat and aquatic products, the optimized combination of C_{18} and PSA composite adsorbent effectively removed lipid impurities and macromolecular protein interferents. For high-pigment vegetable and fruit samples, the improved purification system significantly reduced pigment retention in the supernatant, which greatly weakened the matrix suppression effect during mass spectrometry detection.

Centrifugation parameters were finally determined as 10 000 r/min for 8 min at 4 ℃. Under this condition, the impurity precipitation was complete, and the loss of target residues was minimized. The overall pretreatment time for a single sample was shortened by nearly 30% compared with the traditional method, which significantly improved the throughput capacity for large-scale food safety monitoring. Matrix effect comparison between traditional QuEChERS and optimized pretreatment in four food matrices was shown as Table 1.

Table 1 Matrix effect comparison between traditional QuEChERS and optimized pretreatment in four food matrices %

Matrix	Traditional	Optimized
Meat	−32.5	−7.8
Vegetable & fruit	+36.2	+8.5
Cereal	−27.3	−6.3
Aquatic product	+31.7	+7.2

Note: Positive value represents matrix enhancement effect; negative value represents matrix suppression effect.

2.2 Method validation performance The linearity, limit of detection (LOD), limit of quantification (LOQ), recovery and precision of the optimized multi-residue detection method were fully verified. All target pollutants showed excellent linear regression relationships within the corresponding concentration ranges, with correlation coefficients (R^2) greater than 0.999 (Table 2).

The LOD and LOQ of the method were significantly lower than the maximum residue limits specified by national food safety standards and EU food safety regulations (Table 2), indicating that the method possessed high sensitivity for trace residual detection in complex food matrices.

The average recoveries of low, medium and high spiked levels ranged from 86.2% to 108.5%, and the relative standard deviations (RSD) of intra-day and inter-day precision were all less than 4.5% (Table 2). The results demonstrated that the established method had good accuracy, stability and repeatability.

In terms of matrix effect evaluation, the optimized pretreatment strategy effectively inhibited matrix enhancement and matrix suppression phenomena. The matrix effect values of most detection targets were controlled within ±10% (Table 2), which was far superior to the detection performance of the traditional QuEChERS method.

2.3 Actual sample detection results A total of 320 batches of commercially available food samples covering vegetables, fruits, cereals, livestock meat and aquatic products were analyzed using the optimized method. The overall qualified rate of local food samples was 97.19%, indicating that the overall food safety status of the regional market was stable and controllable.

Trace residual pollutants were detected in 9 unqualified batches. Among them, a small amount of pesticide residues were found in individual leafy vegetable samples, trace veterinary drug residues existed in partial pork samples, and micro heavy metal

accumulation was detected in a few aquatic product samples. No severe excessive contamination or compound heavy residual pollution was observed in all tested samples.

The pollution characteristics showed that residual pollutants were mainly low-concentration trace residues, which were closely related to agricultural planting habits, breeding drug administration and environmental background pollution in the region.

Table 2 Linear regression parameters, LOD, LOQ, recovery and precision of target pollutants ($n=6$)

Analyte group	R^2	LOD//mg/kg	LOQ//mg/kg	Average recovery//%	Intra-day RSD//%	Inter-day RSD//%	Matrix effect//%
Pesticides	0.999 2 – 0.999 8	0.001 – 0.012	0.003 – 0.038	87.1 – 107.2	1.8 – 3.9	2.1 – 4.2	–8.2 – 7.6
Veterinary drugs	0.999 1 – 0.999 7	0.002 – 0.015	0.005 – 0.050	86.2 – 108.5	1.5 – 3.6	1.9 – 4.4	–9.1 – 8.3
Heavy metals	0.999 3 – 0.999 9	0.001 – 0.008	0.003 – 0.025	88.3 – 106.1	1.2 – 3.1	1.6 – 3.8	–7.5 – 6.9

Note: All validation experiments were carried out at low, medium and high three spiked concentration levels. Matrix effect between –10% and +10% indicates negligible matrix interference.

2.4 Dietary exposure and health risk assessment Based on the actual detection data and resident dietary consumption parameters, the estimated daily intake (EDI) and hazard quotient (HQ) of chemical residues for different age groups were calculated. The results showed that the dietary exposure levels of pesticide residues, veterinary drug residues and heavy metals in local residents were all far below the acceptable daily intake (ADI) threshold stipulated by national and international food safety organizations.

The HQ values of all pollutants were less than 1.0 (Table 3), indicating that the long-term chronic dietary health risk caused by food residual pollutants was at an acceptable level for ordinary residents.

However, it was noteworthy that children and adolescent groups had relatively higher exposure risk per unit body weight due to their single dietary structure and lower body weight. Although the current residual level would not cause acute poisoning, long-term cumulative low-dose exposure still had potential hidden health risks, which required continuous monitoring and risk prevention.

Table 3 HQ values of three pollutants for different resident groups

Resident group	Pesticide	Veterinary drug	Heavy metal
Children	0.36	0.31	0.27
Adolescent	0.22	0.19	0.16
Adult	0.11	0.09	0.08

Note: $HQ<1$ indicates acceptable long-term chronic dietary health risk. Children have relatively higher exposure risk per kilogram body weight.

3 Discussion

3.1 Advantages of the optimized multi-residue detection method In this study, the improved QuEChERS pretreatment system balanced the extraction efficiency of different types of polar and non-polar pollutants. The composite adsorbent formula targeted the removal of typical interferents in different food matrices, which effectively solved the common technical problems in routine food safety testing. Compared with previous published studies, this method achieved lower matrix effect, higher detection stability and wider matrix applicability.

Moreover, this method realized synchronous screening of pesticides, veterinary drugs and heavy metal pollutants, which greatly improved the detection efficiency of grassroots testing institutions and was more suitable for practical supervision and monitoring work.

3.2 Analysis of regional food residue pollution characteristics According to the sample monitoring results, the unqualified rate of local food products was low, and the pollution level was dominated by trace residues. The trace pesticide residues in vegetable samples were mainly derived from conventional pest control in agricultural cultivation. Individual veterinary drug residues in meat products were related to non-standard drug withdrawal periods in livestock breeding. The trace heavy metal enrichment in aquatic products was affected by regional water environmental background values.

The pollution characteristics reflected that the current food safety risks in the region were not sudden severe pollution events, but long-term subtle residual problems formed in the industrial chain. This kind of low-concentration residual pollution is easily ignored in daily supervision, but it is the key source of long-term dietary cumulative health risks.

4 Conclusions

In this study, an improved and optimized QuEChERS pretreatment method combined with HPLC-MS/MS and ICP-MS detection techniques was successfully established for the simultaneous determination of multiple pesticide residues, veterinary drug residues, and heavy metal contaminants in complex agricultural and livestock food matrices. By optimizing the extraction solvent ratio, salting-out conditions, centrifugation parameters, and composite adsorbent dosage, the matrix interference caused by fat, protein, and pigments in different food samples was effectively eliminated. The established method exhibited excellent linearity, high sensitivity, satisfactory recovery, and good precision, fully meeting the technical requirements for national standard detection and international food safety monitoring.

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cing ecological redlines and managing the intensity of urban development, is the core pathway to enhancing ecological resilience in Zhaoqing so as to maintain its function as an ecological barrier for the Greater Bay Area.

4 Conclusion

Between 2000 and 2024, Zhaoqing City's ecological resilience index surged from 0.214 3 to 0.629 0, marking an increase of 193.5%. This growth manifested in three distinct stages. Concurrently, Zhaoqing City's ecological security framework transitioned from being predominantly pressure-driven to response-oriented. Specifically, the proportion attributed to the pressure index declined from 39.7% to 26.5%, whereas the response index's share escalated from 27.5% to 41.6%. An analysis of obstacle factors revealed that although there were notable improvements in solid waste utilization and per capita carbon emissions, the SO_2 removal rate (16.0%) and soot removal rate (14.5%) emerged as primary constraints. This underscores that atmospheric pollutant removal capacity is a critical bottleneck. Further environmental factor analysis pinpointed forest coverage as the paramount determinant influencing the ecological resilience index ($R^2 = 0.71$, $P < 0.01$), accounting for 71% of its spatial variation. In contrast, temperature exerted a marginal influence ($R^2 = 0.14$, $P < 0.05$), while precipitation and sunshine duration were statistically insignificant. To bolster Zhaoqing City's ecological resilience index, it is imperative to prioritize forest protection and stringent air pollution control measures.

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