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Modified QuEChERS-LC-MS/MS for the simultaneous detection of eight prohibited dyes in complex food matrices: method validation and application to real samples

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The illicit use of industrial dyes in food products remains a critical food safety concern due to their potential toxicity and carcinogenic properties. This study developed and validated a robust method for the simultaneous determination of eight prohibited organic dyes (Auramine O, Rhodamine B, Chrysoidine G, Methylene Blue, Brilliant Green, Sudan Yellow, Toluidine Red, and Sudan Red G) in various food matrices using a modified QuEChERS extraction technique coupled with liquid chromatography-tandem mass spectrometry (LC-MS/MS). The extraction procedure was optimized to maximize recovery efficiency, effectively remove matrix interferences, and enhance analytical sensitivity. The LC-MS/MS system operated in multiple reaction monitoring (MRM) mode, utilizing a C18 column and a gradient mobile phase to achieve efficient separation of dyes with diverse physicochemical properties. The method demonstrated high linearity (R^2 > 0.9991), with limits of detection (LOD) ranging from 0.048 to 2.868 μg kg^{-1} and limits of quantification (LOQ) from 0.160 to 9.561 μg kg^{-1}. Recoveries ranged between 76.8% and 113.1%, with repeatability (RSD_r) < 15% and intermediate precision (RSD_{iR}) < 22% across complex matrices such as chili sauce, wine, sausage, durian, dried chili, and potato chips. The proposed method is rapid, accurate, and highly selective, making it suitable for current routine food safety monitoring programs.

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Sustainability spotlight

The fraudulent use of industrial dyes in food undermines public health and economic stability. This work presents a validated, resource-efficient LC-MS/MS method for detecting eight banned dyes, including Auramine O in durian. By enabling precise and rapid surveillance, this method supports the enforcement of food safety regulations, protects consumer health, and contributes to a more sustainable and transparent global food trade, directly supporting United Nations Sustainable Development Goals 3 and 12.

1 Introduction

Color is a primary sensory attribute determining consumer preference and quality perception of food products. To meet market demands for vibrant and stable coloration at low costs, unscrupulous producers have increasingly resorted to the fraudulent application of industrial organic dyes. However, this economic motivation disregards severe public health implications. Many synthetic industrial dyes, specifically Auramine O, Rhodamine B, Chrysoidine G, Methylene Blue, Brilliant Green, Sudan Yellow, Toluidine Red, and Sudan Red G, pose significant toxicological risks, including allergenicity, genotoxicity, and potential carcinogenicity, particularly linked to hyperactivity in children and long-term chronic diseases.[1–4]

In response to these risks, regulatory bodies worldwide have enforced strict compliance measures. In Vietnam, the Ministry of Health issued Circular 24/2019/TT-BYT to strictly manage food additives, permitting a limited list of 51 colorants while explicitly banning industrial dyes.[5] Despite these regulations, the unauthorized presence of these hazardous compounds remains a pervasive issue in the food supply chain. For instance, Doan *et al.* recently alarmed the public by detecting Chrysoidine G in 19 grilled meat samples in Hanoi, with concentrations reaching up to 462 μg kg^{-1}.[6] This adulteration is not isolated to Vietnam but is a global concern, with similar alerts reported in the Philippines,[7] China,[8] and India regarding the rampant abuse of non-permitted dyes in various food matrices.[9]

To enforce these zero-tolerance policies, robust analytical methods are imperative. Historically, high-performance liquid

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chromatography (HPLC) coupled with UV/Vis or diode array detectors (DAD) has been widely employed.[10–13] Gas chromatography-mass spectrometry (GC-MS) has also been utilized.[14,15] However, these conventional techniques often suffer from limited sensitivity for trace-level detection or require cumbersome derivatization processes. Consequently, liquid chromatography-tandem mass spectrometry (LC-MS/MS)[16,17] and high-resolution Orbitrap mass spectrometry[18,19] have emerged as the gold standards due to their superior selectivity and sensitivity. Despite these analytical advances, a critical research gap remains. Most existing methods focus on simple liquid matrices (beverages) or single classes of dyes. The challenge intensifies when analyzing complex solid food matrices, such as durian (high sugar/creaminess), sausages (high fat/protein), and chili powders (high pigments/waxes). In these matrices, co-extracted interferences can cause severe signal suppression or enhancement in MS detectors, compromising accuracy and method longevity.[20,21] Traditional extraction methods often fail to achieve a balance between comprehensive analyte recovery and effective cleanup, leading to either dirty extracts or loss of target compounds.

To address this bottleneck, the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) technique offers a versatile platform for multi-residue analysis.[22,23] However, standard QuEChERS protocols are often insufficient for the simultaneous extraction of structurally diverse dyes (cationic, neutral, and acidic) from complex biological matrices. Therefore, this study aims to develop and validate a modified QuEChERS protocol coupled with LC-MS/MS for the simultaneous quantification of eight banned industrial dyes. By optimizing the solvent partitioning and dispersive solid-phase extraction (d-SPE) composition, this research provides a rapid, high-throughput, and accurate method suitable for routine surveillance of challenging food matrices, contributing to enhanced food safety control and the protection of public health.

2 Materials and methods

2.1 Chemicals and reagents

All chemicals and solvents used were of analytical or HPLC grade. Acetonitrile (ACN) and methanol (MeOH) (≥99.9% purity) were supplied by Merck (Germany). Formic acid (HCOOH, ≥98%) and ammonium acetate (CH_3COONH_4, ≥98%) were used as mobile phase additives. Salts for the QuEChERS extraction, including sodium chloride (NaCl, ≥99%), anhydrous sodium sulfate (Na_2SO_4, ≥99%), anhydrous magnesium sulfate ($MgSO_4$, ≥98%), and sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$, ≥99%), were purchased from Merck (Germany). Dispersive solid-phase extraction (d-SPE) sorbents, including primary secondary amine (PSA) and octadecyl (C18) bulk sorbents, were obtained from Agilent Technologies (USA). Double-distilled water was prepared in-house using a purification system meeting TCVN 4851:1989 (ISO 3696:1987) standards.

Certified reference standards of solvent yellow 34 hydrochloride (Auramine O, 83.1%), Rhodamine B hydrochloride (97.0%), Chrysoidine G hydrochloride (98.9%), Methylene blue hydrate (92.6%), Brilliant green hydrogen sulfate (95.0%), Sudan yellow (98.6%), toluidine red (96.5%), and Sudan red G (97.1%) were procured from Dr Ehrenstorfer (Germany) and Sigma-Aldrich (USA).

2.2 Preparation of standard solutions

Individual stock solutions (1000 mg L^{-1}) were prepared by dissolving accurately weighed amounts of each reference standard in appropriate solvents. Specifically, ACN was used for Auramine O, Brilliant Green, and Sudan Yellow; Meoh for Rhodamine B, Chrysoidine G, and Methylene Blue; Dichloromethane (DCM) for Toluidine Red; and Toluene for Sudan Red G to ensure complete solubility. All stock solutions were stored at −20 °C ± 4 °C in amber glass vials and were stable for up to 12 months.

Intermediate standard mixtures (Color mix A, 10–100 mg L^{-1}) were prepared by diluting aliquots of stock solutions with ACN. Working standard solutions (Color mix B, 0.3–3.0 mg L^{-1}) were freshly prepared by further dilution of Color mix A with ACN. These working solutions were stored at −20 °C and used for spiking samples and constructing calibration curves.

2.3 Instrumentation and LC-MS/MS conditions

Chromatographic separation was performed on a Waters ACQUITY UPLC H-Class system (Waters Corp., Milford, MA, USA). The analytes were separated on an ACQUITY UPLC BEH C18 column (50 mm × 2.1 mm, 1.7 μm particle size) maintained at 35 °C. The mobile phase consisted of (*A*) 0.1% formic acid in water and (*B*) 0.1% formic acid in acetonitrile. The gradient elution program was set as follows: 0–0.5 min, 20% B; 0.5–1.5 min, linear ramp to 75% B; 1.5–2.5 min, linear ramp to 98% B; 2.5–5.0 min, hold at 98% B; 5.0–5.2 min, return to 20% B; followed by equilibration at 20% B for 1.3 min. The flow rate was constant at 0.3 mL min^{-1}, and the injection volume was 10 μL.

Mass spectrometric detection was carried out on a Xevo TQ-S micro triple quadrupole mass spectrometer (Waters Corp.) equipped with an electrospray ionization (ESI) source operating in positive mode (ESI^+). Data acquisition was performed in Multiple Reaction Monitoring (MRM) mode. The optimized source parameters were capillary voltage, 0.5 kV; cone voltage, 20 V; source temperature, 150 °C; desolvation temperature, 500 °C; cone gas flow, 20 L h; and desolvation gas flow, 1000 L h^{-1}. Detailed MRM transitions, including precursor ions, product ions, and collision energies for each dye, are summarized in Table 1. Data acquisition and processing were conducted using MassLynx V4.2 software (SCN985).

2.4 Sample preparation procedure

2.4.1 Sample collection and pre-treatment. A total of six representative food matrices prone to illegal dye contamination were selected for method validation: chili sauce (M1), red wine (M2), sausage (M3), durian pulp (M4), dried chili powder (M5), and potato chips (M6). Samples were collected from local

Table 1 Optimized MRM parameters for the target organic dyes

Component	Retention time (min)	Precursor ion (*m/z*)	Product ion (*m/z*)	Dwell time (s)	Cone volt (V)	Collision energy (V)
Auramine O	1.61	268.096	147.062[a]	0.017	12	28
			122.021	0.017	12	24
Rhodamine B	1.86	443.223	399.125[a]	0.017	16	42
			355.107	0.017	16	62
Chrysoidine G	1.40	213.032	76.945[a]	0.017	32	18
			120.918	0.017	32	20
Methylene blue	1.45	284.100	268.200[a]	0.017	52	26
			252.050	0.017	52	52
Brilliant green	2.02	385.280	341.250[a]	0.017	66	40
			297.210	0.017	66	48
Sudan yellow	2.61	226.260	77.030[a]	0.017	30	20
			121.060	0.017	30	19
Toluidine red	2.72	308.000	156.000[a]	0.017	20	15
			128.000	0.017	20	30
Sudan red G	2.75	279.032	122.936[a]	0.017	22	18
			107.975	0.017	22	32

[a] Quantitation ions.

markets in Ho Chi Minh City, Vietnam. Solid samples were homogenized using a laboratory blender, while liquid samples were thoroughly mixed. Blank samples (free of target analytes) were confirmed prior to validation studies. All samples were stored at −20 °C until analysis.

2.4.2 Modified QuEChERS extraction. The sample preparation was carried out using a modified QuEChERS procedure based on the method described by our previous works.[24,25] Briefly, a 3.00 ± 0.05 g portion of homogenized sample was weighed into a 50 mL polypropylene centrifuge tube. For dry samples (*e.g.*, chili powder), 3 mL of distilled water was added, and the mixture was vortexed for 1 min to ensure hydration. Extraction was initiated by adding 10 mL of a mixture ACN: MeOH (4 : 1, v/v). The tube was vortexed vigorously for 1 min and then shaken on a mechanical shaker for 15 min to facilitate analyte partitioning.

Subsequently, a salt mixture comprising 1.00 g $CH_3COONa \cdot 3H_2O$ and 4.0 g anhydrous $MgSO_4$ was added. The mixture was immediately vortexed for 2 min to prevent agglomeration of $MgSO_4$ and then centrifuged at 4500 rpm for 5 min. An aliquot (2 mL) of the upper organic layer was transferred to a 15 mL d-SPE clean-up tube containing 100 mg C18, 100 mg PSA, and 1.00 g anhydrous $MgSO_4$. The tube was vortexed for 1 min and centrifuged at 4500 rpm for 5 min. Finally, the purified extract was filtered through a 0.22 μm PTFE membrane filter into an amber vial for LC-MS/MS analysis. Fig. 1 illustrates the schematic diagram of the modified QuEChERS-LC-MS/MS analytical workflow for organic dyes in food samples.

2.5 Method validation

The proposed method was validated in accordance with the guidelines of AOAC International.[26] The validation parameters

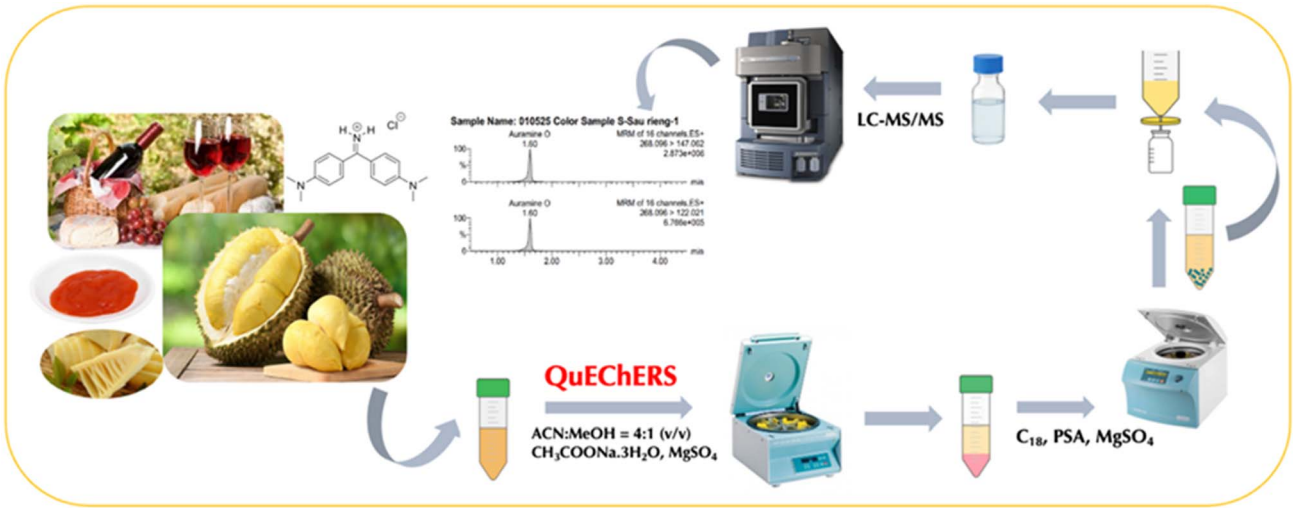

Fig. 1 Schematic diagram of the modified QuEChERS-LC-MS/MS analytical workflow for organic dyes in food samples.

included system suitability, selectivity, linearity, limit of detection (LOD), limit of quantification (LOQ), recovery, repeatability, and intermediate precision.

2.5.1 System suitability. To assess the performance and stability of the LC-MS/MS system, six replicate injections of a mixed standard solution (Standard S3, 5.0 μg L^{-1}) were performed under the optimized chromatographic conditions. The system was considered suitable if the relative standard deviation (RSD) for peak area (RSD_a) was less than 2% and for retention time (RSD_t) was less than 1%.

2.5.2 Selectivity. Selectivity was evaluated by analyzing blank samples of each matrix ($n = 7$) to ensure the absence of interfering peaks at the retention times of the target analytes.[27] Selectivity was confirmed by spiking blank samples with the mixed standard solution at low (1 μg kg^{-1}), medium (2 μg kg^{-1}), and high (5 μg kg^{-1}) concentration levels. The method was deemed selective if no significant interference was observed in the blank chromatograms and if the retention times and mass transitions of the analytes in the spiked samples matched those of the standards.

2.5.3 Linearity and calibration curves. Matrix-matched calibration curves were constructed to compensate for matrix effects. Blank samples (3.00 ± 0.05 g) of each matrix were spiked with appropriate volumes of the working standard solution (Color mix B) to obtain calibration standards at five concentration levels. Specifically, for Auramine O, Rhodamine B, Chrysoidine G, Methylene Blue, Brilliant Green, and Sudan Yellow, the calibration points were 1, 2, 5, 10, and 20 μg kg^{-1}. For Toluidine Red and Sudan Red G, the levels were 10, 20, 50, 100, and 200 μg kg^{-1}. Each calibration level was prepared in triplicate. Linearity was evaluated by linear regression analysis of peak area *versus* concentration, with a coefficient of determination (R^2) ≥ 0.99 required for acceptance.

2.5.4 Limits of detection (LOD) and quantification (LOQ). The LOD and LOQ were determined based on the standard deviation of the response and the slope of the calibration curve. Seven independent replicates of blank samples spiked at the lowest calibration level (1 μg kg^{-1} for the first group and 10 μg kg^{-1} for the second group) were analyzed. The LOD was calculated as 3 times the standard deviation (SD) of the replicate analyses (LOD = 3 × SD), and the LOQ was calculated as 10 times the SD (LOQ = 10 × SD). The estimated LOQ values were verified by analyzing samples spiked at these levels, ensuring a signal-to-noise ratio (S/N) ≥ 10.

2.5.5 Recovery. Accuracy was assessed through recovery studies. Blank samples of each matrix (M1–M6) were spiked with the target analytes at three concentration levels: low (1 and 10 μg kg^{-1}), medium (2 and 20 μg kg^{-1}), and high (5 and 50 μg kg^{-1}), corresponding to the LOQ, 2 × LOQ, and 5 × LOQ levels, respectively. Seven replicates were prepared and analyzed for each spiking level. The recovery percentage was calculated by comparing the measured concentration to the theoretical spiked concentration. Acceptable recovery ranges were defined based on AOAC guidelines (typically 70–120% for these concentration levels).

2.5.6 Precision (repeatability and intermediate precision). Precision was evaluated in terms of repeatability (intra-day precision) and intermediate precision (inter-day precision). Repeatability was determined by analyzing seven replicates of spiked samples at three concentration levels (same as recovery studies) within a single day. Intermediate precision was assessed by repeating the analysis on three different days over a period of two weeks. Precision was expressed as the relative standard deviation (RSD) of the replicate measurements. The acceptance criteria were $RSD_r < 15\%$ and $RSD_{iR} < 22\%$, consistent with AOAC recommendations for residue analysis.

2.5.7 Estimation of measurement uncertainty. The estimation of measurement uncertainty is a fundamental requirement to ensure the metrological quality and reliability of the analytical results. By providing a quantitative range (U) in which the true value of the analyte is expected to lie, researchers can make more informed and statistically sound decisions regarding food safety and regulatory compliance.

In this study, the measurement uncertainty for the determination of prohibited dye residues was estimated using the "top–down" approach, in accordance with the EURACHEM/CITAC Guide. This approach is highly effective as it utilizes empirical method validation data to account for the collective impact of various error sources rather than calculating each individual component separately. The expanded measurement uncertainty (% U) is calculated using the following formula:

$$\%\ U = k \times \%\ u_c = k \times \sqrt{\%\ u_{RSD_{iR}}{}^2 + \%\ u_{bias}{}^2}$$

where: % u_c: the combined standard uncertainty of the analytical method. % U: the expanded measurement uncertainty, representing the interval within which the result is expected to lie with a confidence level of approximately 95% (using a coverage factor $k = 2$). % $u_{RSD_{iR}}$: the uncertainty component associated with method precision, specifically the within-laboratory reproducibility. This value is derived from the relative standard deviation (RSD) of repeated measurements across different days and concentration levels. % u_{bias}: the uncertainty component related to method trueness, estimated through recovery studies. It accounts for the systematic deviation of the measured value from the reference or spiked value.

2.6 Quality assurance of test results

To ensure the reliability of the analytical results, strict quality assurance (QA) measures were implemented. A quality control (QC) sample was analyzed with every batch of samples to monitor instrument drift and method performance. The accuracy of the method was further verified through participation in a proficiency testing scheme organized by the National Institute for Food Control (NIFC). The specific test involved the determination of Auramine O in fresh bamboo shoots (Program Code: H25.57). The method performance was evaluated based on the z-score, with $|z| \leq 2$ indicating satisfactory performance.

3 Results and discussion

3.1 Optimization of LC-MS/MS conditions

The precise optimization of LC-MS/MS parameters was critical for the simultaneous detection of eight target industrial dyes

with diverse physicochemical properties. Initial experiments were conducted *via* direct infusion of individual dye standards (0.3 mg L^{-1} in ACN) into the ESI^+ source to identify the most stable precursor ions ($[M + H]^+$ or $[M]^+$) and their corresponding product ions. For each analyte, two characteristic transitions were selected (one for quantification and one for qualification) to ensure robust identification, strictly adhering to international confirmation criteria (*e.g.*, EU Decision 2021/808).[28] The optimized precursor and product ion transitions, along with their respective cone voltages and collision energies, are summarized in Table 1.

Regarding chromatographic separation, a C18 column was chosen for its proven efficacy in retaining both moderately polar and non-polar compounds. To achieve adequate resolution within a short analysis time, a gradient elution program using water and acetonitrile, both acidified with 0.1% formic acid, was developed. The incorporation of formic acid was essential to enhance the ionization efficiency of the basic dyes in ESI^+ mode and to suppress peak tailing. As shown in Fig. 2, all target analytes were well-separated and eluted with good peak symmetry within 6.5 minutes. This optimized high-throughput capability is crucial for routine food safety monitoring, where large numbers of complex samples need to be processed rapidly.

The use of ESI^+ mode was critical, as the majority of the target dyes, including Auramine O, Rhodamine B, and Brilliant Green, are basic or cationic and thus readily protonate in the presence of an acid (formic acid in the mobile phase). While some Sudan dyes are non-ionic, their detection in ESI^+ mode is often possible through adduct formation or low-level protonation.[29] Our findings are consistent with several previous studies that successfully employed ESI^+ for the analysis of these dyes.[30,31] For instance, similar ESI^+ conditions were reported by Li *et al.* (2013) for illegal dyes in chili products,[8] and by Phuong *et al.* (2020) for 16 dyes in foodstuffs.[32] The selection of two MRM transitions per analyte, with appropriate ion ratios, provides strong evidence for analyte confirmation, significantly reducing the risk of false positives often encountered in complex food matrices.

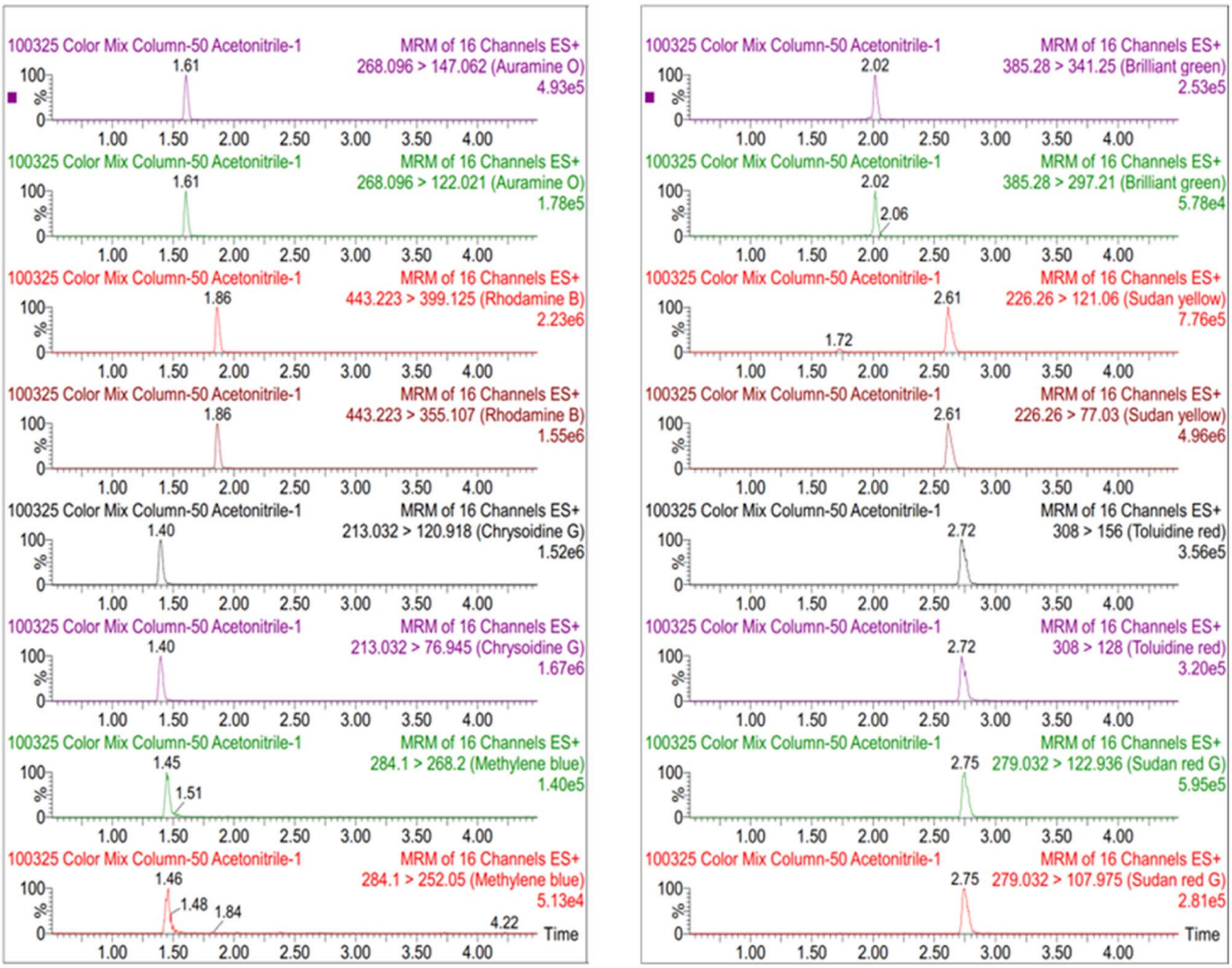

Fig. 2 Representative LC-MS/MS chromatograms of eight target dyes at 2 μg L^{-1} in a mixed standard solution. LC-MS/MS conditions: C18 column, gradient mobile phase (0.1% HCOOH in water/ACN), 0.3 mL min^{-1} flow rate, 10 μL injection.

The choice of a short UPLC C18 column (50 mm) allowed rapid separation, reducing overall run time per sample. This high-throughput capability is crucial for routine food safety monitoring where large numbers of samples need to be processed quickly. While some studies have used longer columns for achieving superior resolution for very complex mixtures,[33] our optimized 50 mm column, combined with the carefully tailored gradient, provided sufficient resolution for the eight target dyes without unduly prolonging analysis time. This balance between resolution and speed is a key advantage for practical applications. The acidic mobile phase, while promoting ESI^+ ionization, also helps in maintaining the stability of certain dyes, preventing degradation during chromatographic separation, a factor that has been highlighted in previous research concerning dye analysis.[33,34]

The meticulous optimization of LC-MS/MS parameters, including ESI^+ mode operation, MRM transition selection, and chromatographic gradient, proved highly effective. The established conditions ensured optimal ionization, high sensitivity, and excellent chromatographic separation for all eight target organic dyes within a short run time. The use of dual MRM transitions, in conjunction with specific retention times, provides a robust foundation for unambiguous identification and accurate quantification in complex food matrices, laying the groundwork for reliable method validation.

3.2 Optimization of sample preparation

The complexity of food matrices, encompassing diverse components such as proteins, fats, carbohydrates, and pigments, necessitates a robust and efficient sample preparation method. The optimization of the QuEChERS procedure was critical to achieving high analyte recoveries while simultaneously minimizing matrix interferences. This section details the systematic approach taken to select the most appropriate extraction solvent, buffering salts, and d-SPE clean-up sorbents.

3.2.1 Selection of extraction solvent. The initial and most crucial step in QuEChERS is the choice of extraction solvent, which must effectively partition the target analytes from the matrix while minimizing co-extraction of interfering compounds. Various solvents and solvent mixtures were evaluated for their extraction efficiency, specifically acetonitrile (ACN), methanol (MeOH), and their combinations. As presented in Table 2 and detailed in Table S1, pure ACN yielded moderate recoveries for most dyes, but significantly lower for some, notably chrysoidine G and toluidine red. Pure MeOH generally resulted in lower overall recoveries compared to ACN, particularly for less polar dyes. However, a mixture of ACN : MeOH (4 : 1, v/v) consistently provided the highest and most comprehensive recoveries across all eight target dyes, ranging from 54.8% to 99.8%.

The observed trend underscores the varied polarity of the target dyes and the need for a mixed solvent system. ACN is a popular choice in QuEChERS due to its ability to precipitate proteins and extract a broad range of analytes while being immiscible with saline aqueous solutions, facilitating phase separation.[35,36] However, for more polar or strongly matrix-bound dyes, the addition of a protic solvent like MeOH is often beneficial. MeOH can disrupt hydrogen bonding and dipole–dipole interactions between analytes and the matrix, thereby enhancing extraction efficiency, particularly for compounds like chrysoidine G and toluidine red, which might have stronger interactions with certain food components.[37] The synergistic effect of ACN : MeOH (4 : 1, v/v) likely stems from ACN's protein precipitation and general extraction capabilities combined with MeOH's ability to "push" more challenging, potentially more polar or matrix-bound, dyes into the solvent phase. This finding is in agreement with several studies that have reported improved recoveries for diverse dye panels when using ACN-MeOH mixtures compared to single solvents.[38,39] For instance, researchers analyzing illegal dyes in various matrices have frequently adopted similar binary solvent systems to broaden the extraction spectrum.[25,40]

3.2.2 Selection of partitioning salts. The salting-out step is integral to QuEChERS, promoting phase separation and further isolating analytes from the aqueous matrix. This study investigated the influence of different salt combinations, including $CH_3COONa \cdot 3H_2O$, $MgSO_4$, NaCl, and Na_2SO_4. As detailed in Table 3 and S2, the salt mixture composed of $CH_3COONa \cdot 3H_2O$ and $MgSO_4$ (SA2: 1 : 4 ratio) consistently delivered superior recoveries for the majority of analytes (38.3–98.6%) compared to other combinations. While NaCl : Na_2SO_4 (SA3, SA4) also performed reasonably well, the $CH_3COONa \cdot 3H_2O/MgSO_4$ system demonstrated broader applicability and higher recoveries for several problematic dyes.

Table 2 Recovery of target dyes under different extraction solvents[a]

		Recovery (%)			
No	Analytes	Solvent (S_1)	Solvent (S_2)	Solvent (S_3)	Solvent (S_4)
1	Auramine O	48.8–80.5	51.2–81.7	53.6–76.5	54.8–90.8
2	Rhodamine B	48.4–84.6	72.5–86.3	69.8–88.9	79.0–99.6
3	Chrysoidine G	20.1–74.5	9.00–50.0	15.4–80.6	25.6–90.7
4	Methylene blue	39.9–77.7	28.3–79.9	30.2–76.1	41.9–88.2
5	Brilliant green	32.2–81.2	55.0–80.5	53.5–83.4	65.7–94.1
6	Sudan yellow	37.9–97.5	34.9–88.5	44.4–92.5	55.3–99.8
7	Toluidine red	24.4–80.1	12.3–45.3	33.8–70.1	64.7–92.6
8	Sudan red G	39.2–82.9	27.1–63.6	44.5–78.9	64.4–95.7

[a] Note: S_1: ACN; S_2: MeOH; S_3: ACN : MeOH = 1 : 1; S_4: ACN : MeOH = 4 : 1.

Table 3 Recovery of target dyes with different QuEChERS salt mixtures[a]

	Recovery (%)			
Analytes	Salt mixture (SA_1)	Salt mixture (SA_2)	Salt mixture (SA_3)	Salt mixture (SA_4)
Auramine O	48.8–91.4	89.5–98.6	66.3–91.1	76.5–92.6
Rhodamine B	36.3–81.6	66.3–97.5	48.0–96.5	54.9–96.4
Chrysoidine G	15.8–49.6	69.2–90.7	33.6–76.1	26.9–78.9
Methylene blue	21.5–84.6	69.6–87.5	60.9–82.6	63.5–80.5
Brilliant green	48.7–87.7	64.3–96.6	56.5–98.5	70.0–98.6
Sudan yellow	39.1–70.7	62.7–86.3	34.1–88.0	51.4–80.9
Toluidine red	22.8–61.8	38.3–97.9	18.6–79.9	28.1–77.7
Sudan red G	27.0–61.0	40.5–92.8	27.5–73.1	31.9–73.4

[a] Note: SA_1: $CH_3COONa \cdot 3H_2O : Na_2SO_4 = 1:4$; SA_2: $CH_3COONa \cdot 3H_2O : MgSO_4 = 1:4$; SA_3: $NaCl : Na_2SO_4 = 1:4$; SA_4: $NaCl : MgSO_4 = 1:4$.

The superior performance of the $CH_3COONa \cdot 3H_2O/MgSO_4$ combination can be attributed to several factors. $MgSO_4$ serves primarily as a strong dehydrating agent, effectively removing residual water from the organic phase, which helps in concentrating the analytes and reducing matrix effects exothermic hydration also aids in breaking down matrix components.[41–43]

Critically, $CH_3COONa \cdot 3H_2O$ functions as a buffering agent, maintaining the pH of the aqueous phase in a mildly alkaline range, typically around 9.0–10. This controlled pH is crucial for the stability of pH-sensitive dyes, preventing their degradation or unwanted ionization that could lead to losses during extraction. Specifically, for certain basic dyes and some other dyes in this panel, a mildly alkaline environment can enhance their stability or promote more efficient extraction compared to acidic conditions.[44] This finding aligns with modifications in QuEChERS methodology to incorporate buffering salts suitable for stable analytes or preferentially extracted at alkaline pH, or for matrices with inherent acidic/basic properties.[45] The ability of the $CH_3COONa \cdot 3H_2O/MgSO_4$ system to prevent reversed partitioning of analytes back into the aqueous phase and to mitigate potential hydrolysis of certain dyes contributes significantly to its effectiveness.[46]

3.2.3 Optimization of d-SPE clean-up. Following the initial extraction and phase separation, a dispersive solid-phase extraction (d-SPE) step is essential to remove co-extracted matrix components that can interfere with LC-MS/MS analysis, such as lipids, waxes, and pigments. The choice and amount of d-SPE sorbents were optimized by testing various combinations of C18, PSA, and $MgSO_4$. As shown in Table 4 and S3, the clean-up kit comprising 100 mg C18, 100 mg PSA, and 1 g $MgSO_4$ (K3) provided the most balanced performance, achieving high recoveries (84.2–99.8%) while ensuring effective matrix removal.

Each sorbent in the d-SPE mixture plays a specific role. PSA is a weak anion exchanger highly effective at removing organic acids, sugars, and some pigments, which are prevalent in fruit and vegetable matrices.[47] C18 is a non-polar sorbent that efficiently retains and removes fatty acids, waxes, and lipids, which are abundant in high-fat matrices like sausages, durian pulp, and potato chips.[47,48] The inclusion of $MgSO_4$ in the d-SPE tube further aids in removing any remaining water, ensuring a dry extract and preventing adverse effects on chromatographic performance and MS ionization.[49]

The optimization process revealed that while increasing the amounts of C18 and PSA generally improved matrix cleanup, excessive amounts could lead to some loss of target analytes, particularly those with chemical similarities to the removed interferences. Therefore, the chosen quantities of 100 mg C18 and 100 mg PSA represented a crucial balance, maximizing cleanup efficacy without compromising analyte recovery. This optimized d-SPE strategy is particularly vital for challenging matrices, where high levels of co-extractives can cause significant signal suppression, ion source fouling, and column degradation.[50] This multi-sorbent approach aligns with the best practices for QuEChERS cleanup of complex food samples, as demonstrated in various studies targeting pesticide residues or food contaminants.[51–53]

The systematic optimization of the QuEChERS extraction procedure was pivotal in achieving the desired analytical performance. The synergistic effect of the ACN : MeOH (4 : 1, v/v) solvent mixture, coupled with the buffering capacity of $CH_3COONa \cdot 3H_2O$ and the dehydrating properties of $MgSO_4$, maximized analyte recoveries while effectively managing matrix co-extractives. Furthermore, the optimized d-SPE clean-up using a balanced combination of C18, PSA, and $MgSO_4$ efficiently

Table 4 Recovery of target dyes with different d-spe clean-up sorbents[a]

	Recovery (%)			
Analytes	Sorbent (K_1)	Sorbent (K_2)	Sorbent (K_3)	Sorbent (K_4)
Auramine O	74.7–97.6	73.8–97.3	97.3–99.3	91.0–98.0
Rhodamine B	69.6–82.4	70.3–97.1	94.9–98.5	85.9–97.7
Chrysoidine G	65.1–93.8	62.9–96.0	84.2–98.3	73.3–95.6
Methylene blue	78.4–98.7	75.2–98.0	92.2–98.5	87.1–94.1
Brilliant green	76.0–91.1	81.0–93.2	94.6–98.5	94.7–97.9
Sudan yellow	79.4–87.2	78.5–92.4	96.1–99.8	89.4–96.5
Toluidine red	51.4–91.2	53.1–94.8	94.9–97.9	69.9–93.9
Sudan red G	58.9–87.5	60.2–94.5	92.9–96.3	73.2–94.6

[a] Note: K_1: $C_{18} : PSA : Na_2SO_4 = 100:100:1000$ (mg); K_2: $C_{18} : PSA : Na_2SO_4 = 50:50:1000$ (mg); K_3: $C_{18} : PSA : MgSO_4 = 100:100:1000$ (mg); K_4: $C_{18} : PSA : MgSO_4 = 50:50:1000$ (mg).

Table 5 System suitability test results for the LC-MS/MS method

Analytes	Time retetion (min)	Peak area	RSD_t (%)	RSD_a (%)
Auramine O	1.59	106 070	0.00	1.66
Rhodamine B	1.85	126 406	0.00	1.48
Chrysoidine G	1.38	57 065	0.30	1.11
Methylene blue	1.42	56 770	0.36	1.52
Brilliant green	2.00	56 927	0.26	1.75
Sudan yellow	2.59	610 403	0.16	0.81
Toluidine red	2.70	422 406	0.15	1.70
Sudan red G	2.72	150 720	0.00	1.75

removed interfering matrix components without significant loss of target analytes. These optimized conditions collectively form a robust and versatile sample preparation strategy essential for the accurate determination of diverse organic dyes in challenging food samples.

3.3 Method validation

Following the optimization of both chromatographic and sample preparation conditions, the developed method was rigorously validated to assess its performance characteristics in accordance with AOAC International guidelines.[26] This section details the evaluation of system suitability, specificity and selectivity, linearity, limits of detection and quantification, accuracy (recovery), and precision (repeatability and intermediate precision).

3.3.1 System suitability. System suitability tests are fundamental for ensuring the proper functioning of the LC-MS/MS system prior to sample analysis. Six consecutive injections of a mixed standard solution (S3, 5.0 μg L^{-1}) were performed. As presented in Table 5 and Table S4, the relative standard deviations (RSDs) for both peak areas and retention times for all target analytes were well within the acceptance criteria (RSD for peak area < 2%; RSD for retention time < 1%). Specifically, peak area RSDs ranged from 0.00% to 0.36%, and retention time RSDs ranged from 0.81% to 1.75%.

These results confirm the excellent stability and precision of the LC-MS/MS system, demonstrating its readiness for accurate and reproducible quantitative analysis.[54] The consistent retention times are particularly important for reliable peak identification, while low peak area variability indicates stable signal response, minimizing quantification errors. This level of system performance is comparable to that reported in other validated LC-MS/MS methods for trace contaminants in food.[55]

3.3.2 Selectivity. Selectivity is crucial for ensuring that the method can accurately identify and quantify target analytes without interference from the complex food matrix or other co-extracted compounds. Analysis of seven replicate blank samples from various food matrices (*e.g.*, chili sauce) confirmed the absence of interfering peaks at the retention times and MRM transitions of the target analytes (Fig. 4). Further, blank samples spiked at low, medium, and high concentrations exhibited clean chromatograms with distinct peaks for the target dyes, showing clear separation from matrix components (Fig. 2 and 3). The peak areas in spiked samples increased proportionally with the spiking level, clearly differentiating from the blank chromatograms (Table S5).

The method's high specificity is further supported by monitoring two specific MRM transitions for each analyte. This dual-transition approach, requiring a specific ion ratio for confirmation, significantly reduces the probability of false positives, especially in highly complex matrices.[24,55] The combination of chromatographic separation and mass spectrometric detection effectively resolves potential co-eluting interferences. This high degree of selectivity is paramount for detecting banned substances present at trace levels in foods, where complex matrix components often pose a significant challenge findings are consistent with other comprehensive multi-residue methods that leverage LC-MS/MS for enhanced selectivity.[56]

3.3.3 Linearity and matrix effects. Linearity was assessed by constructing matrix-matched calibration curves for each of the six food matrices to account for potential matrix interferences. The calibration range for Auramine O, Rhodamine B, Chrysoidine G, Methylene Blue, Brilliant Green, and Sudan Yellow was 1-20 μg/kg, while for Toluidine Red and Sudan Red G, it was 10–200 μg kg^{-1}. All calibration curves exhibited excellent linearity, with correlation coefficients (R^2) ranging from 0.9991 to 0.9999. The high R^2 values demonstrate a strong linear relationship across the entire calibration range for all target analytes, consistent with the requirements of AOAC International guidelines.[26]

However, matrix effects (ME) remain a significant challenge in LC-MS/MS analysis, often leading to signal suppression or enhancement.[57,58] In this study, the ME was quantitatively evaluated by comparing the slopes of the matrix-matched calibration curves with those obtained in pure solvent (see more Table S6). As summarized in Table S6, varying degrees of signal suppression were observed across all matrices The ME values ranged from −5.7% (brilliant green in wine) to −24.0% (methyllene blue in potato chips).

According to SANTE 11312/2021 guidelines, matrix-matched calibration is required if the signal suppression or enhancement exceeds 20%.[59] Given that several matrices, particularly durian (M4), dried chili (M5), and potato chips (M6), exhibited suppression levels reaching −24.9%, the use of solvent-based calibration would lead to significant systematic errors. Consequently, the implementation of independent matrix-matched calibration curves for each specific food category was essential. This approach effectively neutralized the disparate matrix interferences and ensured the highest level of analytical accuracy and reliability for each individual sample type throughout the validation and real-sample analysis.

3.3.4 Limits of detection (LOD) and quantification (LOQ). The LOD and LOQ determine the lowest concentrations at which analytes can be reliably detected and quantified, respectively. Based on a signal-to-noise ratio (S/N) of 3 : 1 for LOD and 10 : 1 for LOQ, the method achieved LODs ranging from 0.049 to 2.868 μg kg^{-1} and LOQs from 0.160 to 9.561 μg kg^{-1} across the various food matrices (Table 6). These values were confirmed by analyzing spiked blank samples.

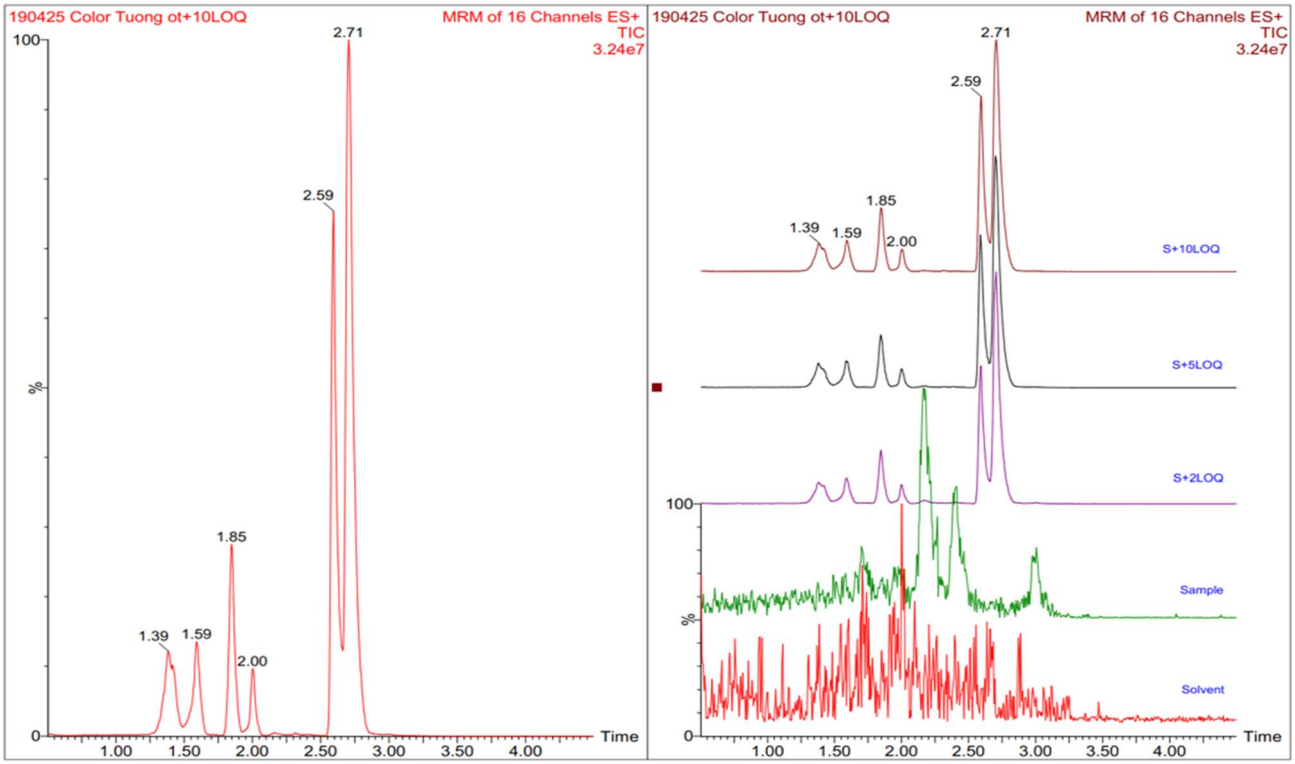

Fig. 3 Representative LC-MS/MS chromatograms of eight target dyes spiked in a chili sauce matrix. (Illustrates the method's selectivity and specificity in a complex food matrix (chili sauce), comparing the blank chili sauce (unspiked) with samples spiked at various concentrations. Clear detection of analytes with minimal matrix interference is observed. LC-MS/MS conditions: C18 column, gradient mobile phase (0.1% HCOOH in water/ACN), 0.3 mL min^{-1} flow rate, 10 μL injection).

Table 6 Summary of method validation parameters (LOD, LOQ, recovery, and precision) for target dyes in various food matrices

Sample matrix	LOD (μg kg^{-1})	LOQ (μg kg^{-1})	Repeatability RSD_r (%)	Intermediate precision RSD_{iR} (%)	Recovery (%)	Expanded uncertainty U ($k = 2$, 95%)
Chili sauce (M1)	0.049–1.579	0.165–5.264	1.66–8.28	2.34–13.0	82.7–110.5	9.50–17.0
Wine (M2)	0.048–1.871	0.160–5.236	1.46–6.45	2.48–13.4	87.3–113.1	10.9–13.9
Sausage (M3)	0.089–1.972	0.297–6.573	1.34–8.41	2.46–10.6	82.1–110.0	8.77–19.2
Durian (M4)	0.115–2.760	0.383–9.199	0.53–9.27	1.84–11.1	80.4–111.2	7.37–15.8
Dried chili (M5)	0.074–1.724	0.245–5.747	0.86–6.40	1.28–11.9	76.8–108.3	11.3–18.6
Potato (M6)	0.175–2.868	0.583–9.561	1.02–9.24	2.55–13.8	80.7–108.8	13.5–18.3

These low LOD and LOQ values underscore the high sensitivity of the developed method, enabling the detection of these illegal dyes at trace levels, which is essential for enforcing regulatory limits (often zero-tolerance).[30] The sensitivity achieved is comparable to, or in many cases superior to, other reported methods for similar analytes in complex food matrices. For example, some studies reported LOQs for Sudan dyes in chili products in the range of 84–111 μg kg^{-1} using HPLC-DAD,[60,61] whereas our LC-MS/MS method achieves much lower LOQs, highlighting the significant advantage of tandem mass spectrometry. This high sensitivity makes the method particularly suitable for robust food safety monitoring programs.

3.3.5 Recovery and precision (repeatability and intermediate precision). Accuracy, expressed as recovery, and precision, indicated by repeatability (intra-day) and intermediate precision (inter-day), were evaluated by spiking blank samples at LOQ, 2 × LOQ, and 5 × LOQ levels (corresponding to 1–5 μg kg^{-1} for most dyes and 10–50 μg kg^{-1} for Toluidine Red and Sudan Red G). As summarized in Table 6, mean recoveries ranged from 76.8% to 113.1% across all matrices and spiking levels, meeting the AOAC recommended range of 70–120%.[26] The repeatability (RSD_r) values were consistently below 15% (ranging from 0.53% to 9.27%), and intermediate precision (RSD_{iR}) values were below 22% (ranging from 1.28% to 13.8%) across all tested matrices and spiking levels (see more detail in Table S7).

The acceptable recovery rates demonstrate the method's accuracy and its ability to effectively extract and quantify the target dyes from challenging matrices without significant loss or overestimation. The low RSD values for both repeatability

and intermediate precision highlight the method's reliability and reproducibility, both within a single analytical run and across different days or operators.[62–64] These results are well within the internationally recognized performance criteria for residue analysis in food, underscoring the method's suitability for routine application in regulatory laboratories.[65] The robust performance across diverse matrices, from high-fat sausage to sugary durian and spicy chili powder, confirms the versatility and applicability of the optimized QuEChERS procedure.

3.3.6 Quality assurance of test results. To further ensure the reliability and external validity of the developed method, participation in a proficiency testing (PT) scheme was undertaken. The method was successfully applied to analyze Auramine O in fresh bamboo shoot samples provided by the National Institute for Food Control (NIFC) under program H25.57. As presented in Table S8, the obtained *z*-score for Auramine O was 0.35, which falls well within the acceptable range of $|z| \leq 2$.[66] This successful participation in an independent PT scheme provides strong, objective evidence of the method's accuracy and the laboratory's competency. It serves as an external validation of the internal validation data, confirming that the method yields reliable and comparable results to other accredited laboratories. This rigorous quality assurance step is vital for any analytical method intended for regulatory enforcement and food safety monitoring.[67,68]

The comprehensive validation demonstrated that the developed modified QuEChERS-LC-MS/MS method fulfills all critical performance criteria according to AOAC guidelines. Excellent system suitability, high specificity and selectivity, broad linearity across relevant concentration ranges, and sensitive detection limits were achieved. Crucially, the method exhibited satisfactory recovery rates (76.8–113.1%) and high precision ($RSD_r < 15\%$, $RSD_{iR} < 22\%$) across all diverse food matrices and spiking levels. Furthermore, successful participation in an independent proficiency testing scheme affirmed the external validity and robustness of the method. Collectively, these results confirm that the method is reliable, accurate, and fit-for-purpose for the simultaneous determination of the eight banned organic dyes in complex food matrices, providing a powerful tool for food safety surveillance.

3.3.7 Comparison of method performance. The performance characteristics of the proposed modified QuEChERS-LC-MS/MS method were compared with previously reported literature to evaluate its efficiency and sensitivity. As summarized in Table 7, our method achieved remarkably low LOD and LOQ values across six diverse and complex food matrices. Specifically, the LOQs for all analytes ranged from 0.160 to 9.56 μg kg^{-1}, which is significantly more sensitive than the methods reported by Vu Lan Phuong *et al.*[69] (LOQ = 15 μg kg^{-1}) and Doan Thu Huyen *et al.* (LOQ = 30 μg kg^{-1}).[70]

The robustness of the current method is further highlighted by its ability to maintain high recovery (76.8–113.1%) and excellent precision ($RSD_{iR} < 13.8\%$) in challenging samples such as durian (high sugar/fat) and chili sauce (high pigment). While some previous studies achieved satisfactory results for a single matrix like chicken meat[71] or grilled meat,[70] this study successfully broadened the application to a wide array of structurally different prohibited dyes (cationic, neutral, and acidic). The high-throughput capability, with a total run time of only 6.5 minutes, offers a substantial advantage for routine food safety monitoring compared to existing protocols.

The integration of a modified QuEChERS extraction with a finely tuned d-SPE cleanup (C18 and PSA) proved to be a superior strategy for eliminating matrix interferences, as evidenced by the satisfactory matrix effect values and expanded uncertainty (% *U*) ranging from 7.37% to 19.2% across all matrices. Consequently, this method represents a powerful and reliable tool for the simultaneous determination of illegal colorants in complex food supply chains.

Table 7 Comparison of analytical performance between the proposed method and previously reported LC-MS/MS methods for the determination of illegal dyes

Analytes	Method	Samples	LOD (μg kg^{-1})	LOQ (μg kg^{-1})	RSD_r (%)	RSD_{iR} (%)	Recovery (%)	References
Auramine O Rhodamine B Chrysoidine G Methylene blue Brilliant green Sudan yellow Toluidine red Sudan red G	LC-MS/MS	Food	0.048–2.868	0.160–9.561	0.53–9.27	1.28–13.8	76.8–113.1	This work
Auramine O	LC-MS/MS	Chicken meat	0.06	0.2	<10	—	85.1–93.3	71
Auramine O	LC-MS/MS	Food	0.1	0.5	2.10–6.10	—	80.1–99.4	72
Auramine O Rhodamine B Chrysoidine G	LC-MS/MS	Food	5	15	2.30–11.1	—	83.0–104	69
Sudan red G	LC-MS/MS	Raisins, chili powder	—	10	4.60–13.0	—	91.8–94.0	73
Chrysoidine G	LC-MS/MS	Grilled meat	10	30	5.30–10.7	—	90.0–96.0	70
Auramine O Rhodamine B Chrysoidine G	LC-MS/MS	Food	0.05–0.60	0.30–3.0	5.70–15.8	7.80–15.6	90.0–112.4	8

Table 8 Results of market surveillance for prohibited dyes in food samples

Sample matrix	No of samples	Contaminated samples	Concentration (mg kg^{-1})
Jackfruit	2	0	ND[a]
Spices	4	0	ND
Wine/liquor	7	0	ND
Dried potatoes	2	0	ND
Candy	7	0	ND
Chili powder	3	0	ND
Sausages	4	0	ND
Fermented pork	5	0	ND
Watermelon seeds	3	0	ND
Roasted chicken	2	0	ND
Durian	26	8	Auramine O (1.82–22.6)
Total	65	8	–

[a] ND: not detected.

3.4 Application to real samples

The validated method was applied to analyze real-world food samples collected from local markets in Ho Chi Minh City, Vietnam, to assess the prevalence of the target illegal dyes. A total of 65 samples across various food types, including chili sauce, red wine, sausage, durian, dried chili, and potato chips, were screened. The results of this survey are summarized in Table 8 and visually represented in Fig. 5.

The analysis revealed a significant finding with Auramine O, which was detected in 8 out of 26 durian samples (30.8%), with concentrations ranging from 1.82 to 22.6 μg kg^{-1}. Importantly, none of the other 39 food samples (chili sauce, red wine, sausage, dried chili, potato chips) showed any detectable levels of the eight target dyes. The detection of Auramine O in nearly one-third of the durian samples is a grave concern, highlighting a prevalent food adulteration practice that directly impacts consumer health and. Auramine O, an industrial dye used for textiles and paper, is classified as a potential human carcinogen (Group 2B) by the International Agency for Research on Cancer (IARC) and is strictly prohibited in food products worldwide.[25,74] Its presence in durian is most likely intended to enhance the fruit's yellow color, mimicking ripeness or desired intensity, thereby increasing its market appeal and potentially its price. This practice is particularly insidious as durian is a popular fruit, consumed widely both domestically and internationally.

This finding aligns with recent reports from other regions facing similar challenges. For instance, the General Administration of Customs of China (GACC) has frequently issued warnings regarding Auramine O contamination in durian and other agricultural products from Southeast Asian countries, leading to rejections of exported goods.[75] Studies in other matrices like bamboo shoots and curry powder have also

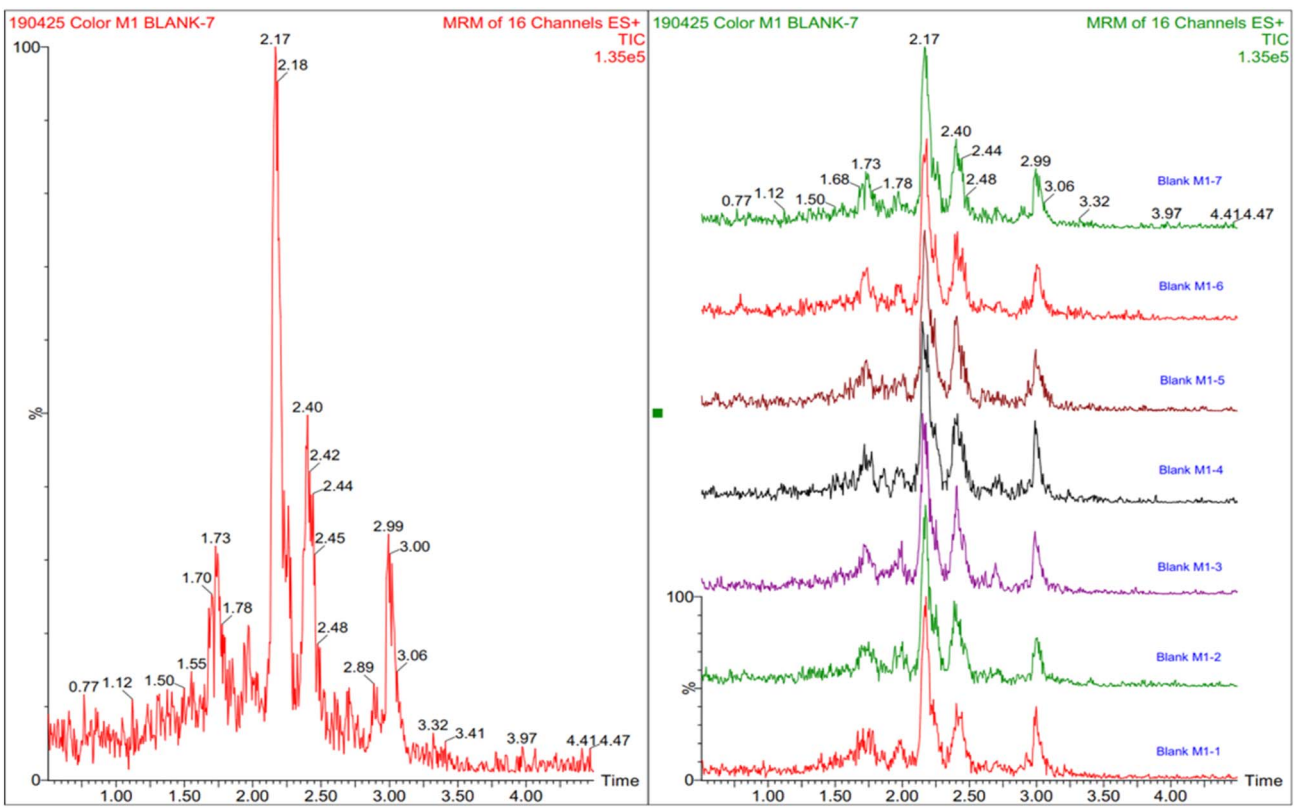

Fig. 4 Representative LC-MS/MS chromatograms of blank chili sauce matrix. (Displays chromatograms from unspiked chili sauce samples (M1), processed through the entire modified QuEChERS extraction and clean-up procedure. LC-MS/MS conditions: C18 column, gradient mobile phase (0.1% HCOOH in water/ACN), 0.3 mL min^{-1} flow rate, 10 μL injection).

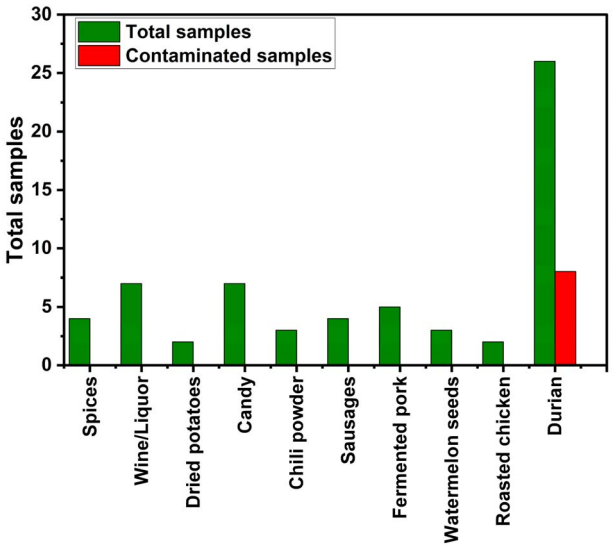

Fig. 5 Comparison of total samples and contaminated samples by food category.

reported Auramine O contamination, indicating its widespread misuse. The concentrations detected in our study (up to 22.6 μg kg^{-1}) are comparable to, or in some cases higher than, levels reported in other contaminated foods. These levels, while seemingly low, are unacceptable for a prohibited substance and pose a cumulative health risk over time, especially for regular consumers. The absence of other target dyes in the remaining food categories is reassuring but does not necessarily imply their complete absence from the market. It suggests that if contamination exists, it might be at concentrations below the LOQ of the current method, or that the specific batches analyzed were uncontaminated. However, the targeted detection of Auramine O in durian points to a specific and pressing issue within this product chain.

The implications of these findings are substantial. For consumers, it raises serious health concerns and erodes trust in food safety systems. For the Vietnamese food industry, particularly its agricultural export sector, such adulteration can lead to significant economic losses due to trade barriers and damage to reputation. Countries like China, a major importer of durian, impose strict limits (often zero-tolerance) for such contaminants.[76] Therefore, continuous and rigorous monitoring programs are urgently needed, focusing on high-risk commodities like durian, to protect both public health and the economic viability of the food sector. The developed method provides a robust and sensitive tool for these efforts.

4 Conclusion

This study successfully developed and rigorously validated a sensitive, accurate, and robust method for the simultaneous determination of eight prohibited organic dyes (Auramine O, Rhodamine B, Chrysoidine G, Methylene Blue, Brilliant Green, Sudan Yellow, Toluidine Red, and Sudan Red G) in diverse and challenging food matrices. The optimized method integrates a modified QuEChERS extraction with LC-MS/MS, addressing critical analytical challenges such as matrix complexity and the need for high-throughput analysis. The developed method demonstrated excellent analytical performance, including high linearity (R^2 > 0.999), low limits of detection (0.048–2.868 μg kg^{-1}) and quantification (0.160–9.561 μg kg^{-1}), satisfactory recoveries (76.8–113.1%), and high precision (RSD_r < 15%, RSD_{iR} < 22%). Furthermore, the method's reliability was externally verified through successful participation in a proficiency testing scheme, confirming its fitness for purpose in routine regulatory applications. The application of this method to real market samples yielded a significant finding: Auramine O contamination was detected in 30.8% of durian samples (ranging from 1.82 to 22.6 μg kg^{-1}). This alarming discovery underscores the pervasive issue of food adulteration with banned industrial dyes and highlights a specific threat to consumer health and international trade, particularly for agricultural exports. In conclusion, this research provides a powerful and indispensable analytical tool for food safety authorities to effectively monitor and control the presence of illegal dyes in the food supply chain. The insights gained from the market surveillance emphasize the urgent need for heightened vigilance and stricter enforcement to protect public health and maintain the integrity of the food industry, especially in the context of global trade for key agricultural products like durian.

Author contributions

Van-Phung Binh, conceptualization, data curation, formal analysis, validation, Thi- Son Tra Nguyen: formal analysis, validation, data curation, visualization, Van-Cuong Nguyen and Tran Quang Hieu: conceptualization, methodology, supervision, writing – original draft, writing – review & editing.

Conflicts of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Supplementary information (SI) is available. See DOI: https://doi.org/10.1039/d6fb00064a.

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