

Multiresidue Pesticides Analysis in Norbixin Color Additive using LCMS-8050 and GCMS-TQ8040 NX

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User Benefits

- ◆ The method involves study of 72 pesticide residues for their LOQ on both LC-MS/MS and GC-MS/MS, based on SMPR set for recovery, repeatability (RSD_r) and within-laboratory reproducibility (RSD_R).
- ◆ A modified QuEChERS extraction procedure has been employed for quantifying pesticides at trace levels in complex matrix like norbixin using Ultra-fast technologies of LCMS-8050 and GCMS-TQTM8040 NX.
- ◆ LCMS Method Package for Residual Pesticide Ver.3 and GCMS Smart Pesticides DatabaseTM Ver.2 from Shimadzu Corporation enables ease of optimizing instrumental method.

1. Introduction

Norbixin is the yellow-(red) orange carotenoid, which in combination with bixin, constitutes for 80 % of red-orange annatto dye which is extracted from the pericarp of the seeds of *Bixa Orellana* (Fig. 1). The annatto pigment has global economic significance, as it is one of the most widely used natural dyes to color food, cosmetics and pharmaceutical products.



Fig. 1 *Bixa Orellana* seeds and its extract

Owing to its large culinary uses and other diverse applications, use of chemical pesticides for its production in large quantities is imperative. Dye extraction process may result in concentration of pesticides and in turn contribute to adverse impact on human health when incorporated in various preparations. Hence quantitation of residual pesticides in norbixin colour additive becomes very important. As the oleoresin is a complex matrix for extraction, it is required to develop a rugged, sensitive and efficient method for residual pesticide analysis.

This study reports a highly sensitive method for simultaneous quantification of multiple pesticides in complex matrix of norbixin using modified QuEChERS^[1] with triple quadrupole liquid chromatography (LC-MS/MS) and gas chromatography (GC-MS/MS) system.

2. Materials and Methods

For this study, customized reference standard mixture of 72 most commonly observed pesticides in colour additives were procured from Restek Corporation.



Fig. 2 Shimadzu LCMS-8050

The calibration standards were analyzed from 0.05 to 10 µg/L for LC-MS/MS and from 1 to 50 µg/L for GC-MS/MS. Linearity was plotted by external standard method and using weighted regression of $1/C^2$. Sample was spiked at 3 levels i.e., 5, 10 and 25 µg/kg. Recovery samples were prepared in 6 replicates at each level. Shimadzu LCMS-8050 with NexeraTM X2 (Fig. 2) and GCMS-TQ8040 NX (Fig. 3), manufactured by Shimadzu Corporation Japan, were used for quantitation. LabSolutions InsightTM was used for data processing, which helped in evaluating validation parameters with ease.

2.1. Sample preparation

This study uses single extraction procedure for GC-MS/MS and LC-MS/MS. For extraction, modified QuEChERS method approach was adopted. Sodium chloride (AR grade), anhydrous magnesium sulphate ($MgSO_4$) (AR grade) salts were used in optimized proportion to get maximum recoveries of pesticides. Acetonitrile was used as extraction solvent.

After extraction, clean up was performed using optimum combination of C-18, GCB (Graphitized carbon black), PSA (Primary secondary amine) and anhydrous $MgSO_4$ to minimize matrix interference, reduce instrument contamination and achieve lower LOQs.

After clean up, the aliquot of acetonitrile was divided in two parts. For LC-MS/MS, one part was diluted 8 times using methanol : water (50:50 v/v) to obtain 40 times dilution of final spike sample followed by filtration through 0.22µm nylon filter. For GC-MS/MS, remaining aliquot was reconstituted in ethyl acetate such that final sample was diluted 2.5 times.

All samples were analysed as per conditions shown in Table 1 and 2 for LC-MS/MS and GC-MS/MS, respectively.



Fig. 3 Shimadzu GCMS-TQTM8040 NX

2.2. Analytical Conditions

Table 1 Instrument configuration and Analytical Conditions: LC-MS/MS

System Configuration	
LC-MS/MS	: LCMS-8050
Auto-sampler	: Nexera X2 SIL-30AC
Column	: Shim-pack™ Scepter C18-120 (100 mm × 4.6 mm I.D., 5 µm, P/N: 227-31020-04)
LC	
Flow rate	: 0.6 mL/min
Mobile phase A	: 2 mM Ammonium formate in water + 0.02 % Formic acid
Mobile phase B	: 2 mM Ammonium formate in methanol + 0.02 % Formic acid
Gradient program	: B Concentration → 10 % (0.0 min to 1.0 min) → 60 % (3.0 min) → 100 % (11.0-13.0 min) → 10 % (13.20 to 16 min)
Run time	: 17 min
Injection volume	: 20 µL (Co-injection with 30 µL water)
Column oven temp	: 40 °C
MS	
Ionization mode	: ESI
Nebulizing gas flow	: 3 L/min
Interface temp.	: 300 °C
Heating gas flow	: 8 L/min
Drying gas flow	: 8 L/min
DL temp.	: 150 °C
Heating block temp.	: 400 °C

Table 2 Instrument configuration and Analytical Conditions: GC-MS/MS

System Configuration	
GC-MS/MS	: GCMS-TQ8040 NX
Auto-injector	: AOC™-20i + s
Column	: SH-Rxi-5Sil MS (30 m × 0.25 mm I.D., df = 0.25 µm)
Liner	: Topaz Liner, Splitless Single Taper w/Wool
GC	
Injector temp.	: 250 °C
Column oven temp	: 80 °C (2 min), 20 °C/min to 180 °C (0 min), 5 °C/min to 300 °C (3 min)
Run time	: 34 min
Injection mode	: Splitless (High pressure at 250 kPa)
Injection volume	: 1 µL
Carrier gas	: He
Linear Velocity	: 40.4 cm/sec (Constant mode)
MS	
Ionization mode	: EI
Ion source temp.	: 230 °C
Interface temp.	: 280 °C
Solvent cut time	: 5.0 min
Loop Time	: 0.3 sec

3. Result and Discussion

Validation parameters like linearity, recovery and precision were studied against criteria set by Standard Method Performance Requirement (SMPR) (Refer Table 3). Results obtained on LC-MS/MS and GC-MS/MS are shown in Table 4 and Table 5, respectively.

Table 3 SMPR

Analytical range	LOQ to 100 times LOQ
Recovery %	60-120
RSD _R %	≤30
RSD _r %	≤20

3.1. Linearity study

For linearity study, matrix match calibration standards were used. Calibration curve ranged from 0.05 to 10 µg/L for LC-MS/MS and from 1 to 50 µg/L for GC-MS/MS. All calibration standards were found within 80 to 120 % accuracy as per SANTE guidelines^[2].

The linearity graphs of few representative pesticides are shown in Fig. 4 and Fig. 5.

3.2. Recovery study

Six spiked samples of each 5, 10 and 25 µg/kg were analyzed, and their mean recovery was evaluated against SMPR. Except Methoxyfenozide, all pesticides showed good recovery within the range of 60 to 120 % at LOQ level (Refer Tables 4 and 5). As mentioned previously, spiked samples were diluted 40 times for LC-MS/MS and 2.5 times for GC-MS/MS, respectively.

3.3. Precision study

For precision, repeatability and within-laboratory reproducibility studies were carried out.

RSD_r: Repeatability experiment was performed by injecting 6 replicates of spiked samples at 5 µg/L, 10 µg/L and 25 µg/L concentration levels. The %RSD for 6 injections at their respective LOQ levels was found to be less than 20 % (Refer Tables 4 and 5).

RSD_R: Reproducibility experiment for recoveries was performed on 6 different spiked samples at 5 µg/L, 10 µg/L and 25 µg/L concentration levels. The %RSD of 6 spiked samples at their respective LOQ level was found to be less than 30 % (Refer Tables 4 and 5).

Trend graphs for recovery and precision data obtained on LC-MS/MS and GC-MS/MS are shown in Fig. 6 and 7, respectively. Out of 72 pesticides analyzed, only Methoxyfenozide showed 125 % recovery at 10µg/kg, which was higher than SMPR requirement. In GC-MS/MS, Captan could be detected in the form of it's degradant i.e. Tetrahydrophthalamide (THPI) at 25µg/kg.

This method successfully achieved 5µg/kg LOQ for all pesticides on LC-MS/MS. On GC-MS/MS, 5µg/kg, 10µg/kg and 25µg/kg LOQs were achieved for 45, 10 and 1 pesticides, respectively. Refer to summary Tables 4 and Table 5. Representative chromatograms of pesticides at their LOQ levels are shown in Fig. 4 and Fig. 5.

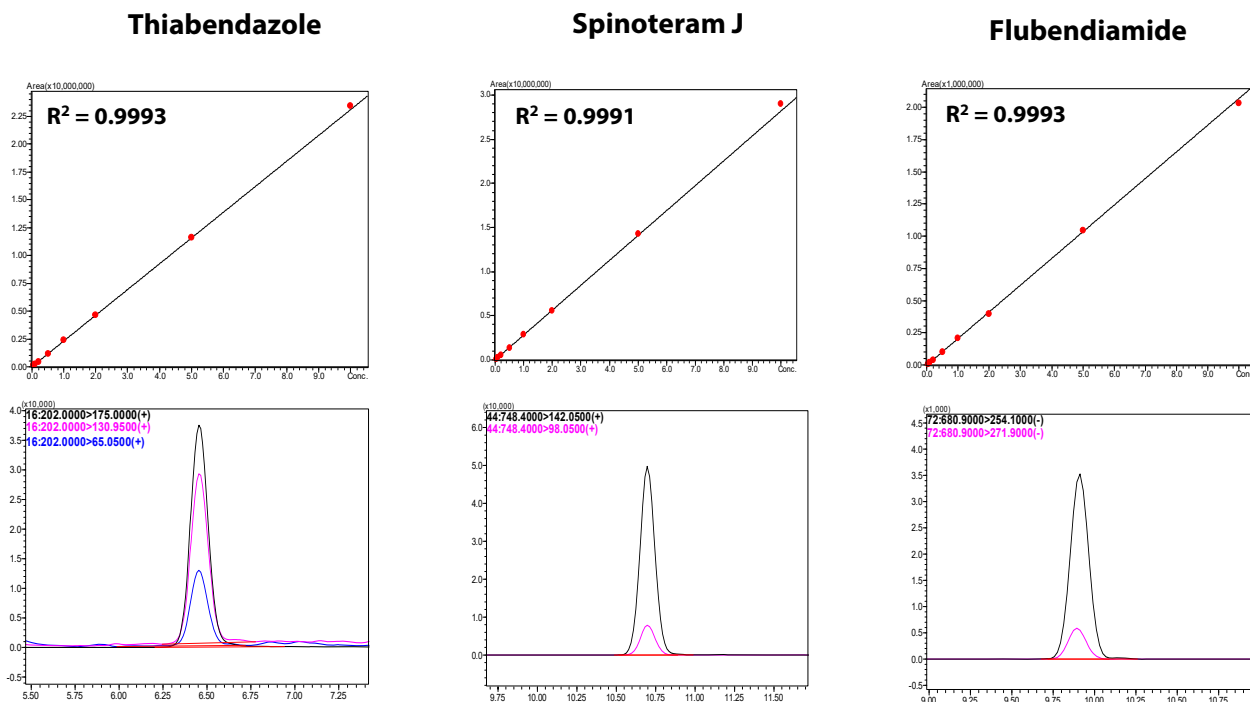


Fig. 4 Representative linearity graphs and chromatograms at LOQ level of LC-MS/MS pesticides

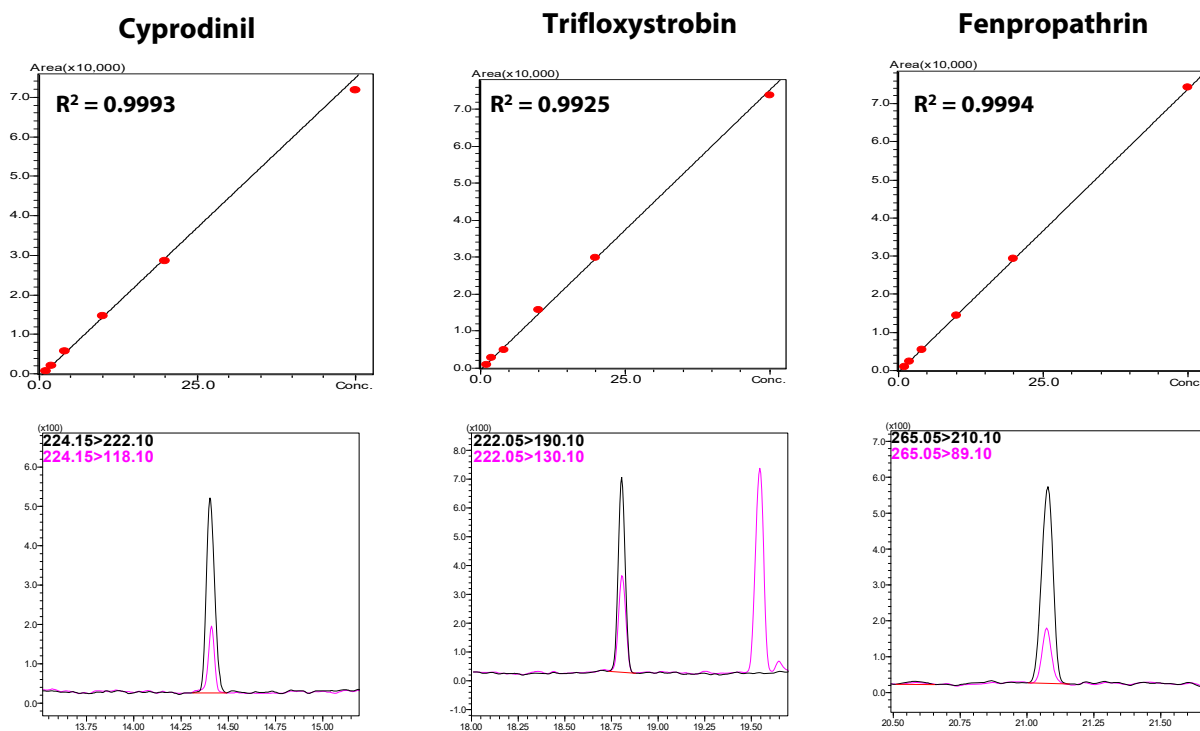


Fig. 5 Representative linearity graphs and chromatograms at LOQ level of GC-MS/MS pesticides

Table 4 Summary results of LC-MS/MS analysis

ID	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE	Determination Coefficient (R ²)	LOQ		Precision		
						mg/kg	% Accuracy at LOQ	Recovery at LOQ (%)	%RSD _R (n=6)	%RSD _F (n=6)
1	Methamidophos	4.12	142.00>94.05	-15	0.9997	0.005	99.60	91.15	3.78	1.72
2	Acephate	4.41	183.90>143.00	-10	0.9969	0.005	110.60	99.00	3.84	2.59
3	Omethoate	4.57	214.00>124.90	-22	0.9989	0.005	104.30	93.75	4.53	2.51
4	Dinotefuran	4.70	203.05>87.00	-15	0.9941	0.005	111.40	102.34	7.35	5.63
5	Methomyl	5.15	163.00>88.00	-9	0.9996	0.005	101.60	107.00	4.23	3.74
6	Thiamethoxam	5.13	292.00>211.00	-12	0.9958	0.005	99.70	110.22	4.73	2.03
7	Imidacloprid	5.44	256.00>209.00	-16	0.9936	0.005	103.10	77.77	10.61	7.62
8	Clothianidin	5.55	250.00>169.00	-13	0.9915	0.005	116.30	107.56	6.24	7.71
9	flupyradifurone	5.64	288.95>125.95	-20	0.9980	0.005	92.10	105.53	6.40	2.59
10	Carbendazim	5.80	192.00>160.05	-18	0.9989	0.005	102.00	79.82	6.12	3.26
11	Acetamiprid	5.70	225.00>128.00	-20	0.9983	0.005	102.50	106.73	6.71	4.66
12	Dimethoate	5.83	230.00>198.90	-10	0.9986	0.005	100.10	106.66	5.29	2.84
13	Sulfoxaflor	5.80	277.95>174.10	-8	0.9952	0.005	107.10	114.89	11.60	4.70
14	Thiacloprid	5.99	253.00>126.05	-20	0.9996	0.005	100.60	107.35	4.30	2.42
15	Thiabendazole	6.46	202.00>175.00	-25	0.9993	0.005	102.20	90.42	5.35	1.33
16	Carbaryl	7.52	202.00>145.00	-11	0.9977	0.005	108.20	108.79	5.78	2.83
17	Imazalil	7.75	297.00>158.95	-21	0.9981	0.005	108.20	90.21	8.46	4.99
18	Flutriafol	7.84	302.10>70.05	-17	0.9992	0.005	103.80	107.02	2.53	4.36
19	Metalaxyl	8.16	280.10>220.10	-14	0.9997	0.005	98.50	105.78	3.86	2.21
20	Chlorantranilprole	8.42	483.80>285.70	-16	0.9978	0.005	94.30	104.15	5.10	4.70
21	Azoxystrobin	8.63	404.00>371.95	-15	0.9996	0.005	101.00	103.80	2.80	2.44
22	Mandipropamid	8.94	412.00>328.00	-15	0.9984	0.005	103.00	108.09	5.09	4.72
23	Boscalid	9.06	343.00>272.05	-30	0.9927	0.005	82.90	112.39	7.31	11.19
24	Fluxapyroxad	9.13	382.00>362.05	-14	0.9980	0.005	91.50	107.68	2.18	5.14
25	Linuron	9.21	249.00>160.00	-16	0.9982	0.005	93.80	106.75	9.47	15.41
26	Dimethomorph	9.21	388.00>301.00	-21	0.9994	0.005	99.30	109.37	4.30	3.74
27	Permethrin	8.86	391.00>304.00	-22	0.9987	0.005	103.40	110.49	10.53	17.25
28	Malathion	9.38	331.00>126.90	-13	0.9977	0.005	93.60	117.73	10.15	6.61
29	Pyrimethanil	9.53	200.10>107.10	-25	0.9979	0.005	100.90	111.12	10.75	8.86
30	Bifenazate	9.50	301.10>198.10	-10	0.9990	0.005	98.30	117.92	7.52	3.85
31	Fluopyram	9.51	396.90>207.90	-21	0.9998	0.005	99.30	109.34	5.07	4.32
32	Spirotetramat	9.58	374.10>216.00	-33	0.9985	0.005	105.80	99.19	4.17	3.70
33	Fenhexamid	9.65	302.10>97.20	-24	0.9908	0.005	83.40	76.82	14.34	13.74
34	Fenbuconazole	9.83	337.00>124.95	-28	0.9972	0.005	108.20	103.04	13.03	6.16
35	Pyriproxyfen	9.83	338.95>69.95	-22	0.9891	0.005	108.40	104.64	13.58	19.17
36	Cyazofamid	9.89	325.00>107.90	-16	0.9976	0.005	108.50	105.37	6.47	5.87
37	Diflubenzuron	10.15	311.00>158.10	-14	0.9953	0.005	94.70	103.64	10.70	9.27
38	Tebuconazole	10.42	308.10>69.95	-24	0.9971	0.005	96.30	88.07	18.43	12.07
39	Spinetoram J	10.70	748.40>142.05	-30	0.9991	0.005	103.80	91.31	4.85	1.58
40	Propiconazole	10.65	342.00>158.90	-27	0.9968	0.005	110.80	96.50	7.94	9.88
41	Diazinon	10.85	305.00>169.10	-21	0.9983	0.005	103.90	97.48	7.10	3.91
42	Pyraclostrobin	10.86	388.00>194.00	-13	0.9997	0.005	97.80	105.70	3.70	3.94
43	Cyprodinil	10.98	226.10>93.10	-37	0.9943	0.005	113.30	92.08	6.01	6.62
44	Indoxacarb	10.95	528.00>202.90	-40	0.9950	0.005	91.20	103.41	2.90	11.09
45	Difenoconazole (isomer)	11.07	406.00>250.90	-25	0.9990	0.005	98.80	103.82	4.97	3.73
46	Spinetoram L	11.18	760.40>142.10	-29	0.9982	0.005	95.10	90.71	6.84	4.77
47	Trifloxystrobin	11.21	409.00>186.00	-20	0.9987	0.005	99.30	105.24	4.54	2.84
48	Triflumizole	11.31	346.10>278.00	-10	0.9994	0.005	98.60	104.58	3.25	5.04
49	Profenofos	11.71	372.80>302.80	-19	0.9971	0.005	110.20	111.96	6.21	4.95

Table 4 Summary results of LC-MS/MS analysis (Continued)

ID	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE	Determination Coefficient (R ²)	LOQ	% Accuracy at LOQ	Recovery at LOQ (%)	Precision	
						mg/kg			%RSD _R (n=6)	%RSD _F (n=6)
50	Buprofezin	11.86	306.20>201.05	-13	0.9976	0.005	109.30	102.17	4.50	2.06
51	Piperonyl-butoxide	12.10	356.10>177.00	-20	0.9990	0.005	99.00	105.33	3.81	2.12
52	Etozazole	12.39	360.10>141.10	-15	0.9994	0.005	102.50	109.25	7.29	3.22
53	Fenpropathrin	12.41	367.00>125.10	-17	0.9962	0.005	106.50	111.98	5.37	7.21
54	Quinoxifen	12.46	308.00>197.00	-31	0.9997	0.005	99.00	106.38	7.56	3.91
55	Spirodiclofen	12.56	411.10>313.05	-14	0.9902	0.005	117.40	108.38	6.49	5.91
56	Pyridaben	12.96	365.20>147.20	-25	0.9994	0.005	102.10	103.09	3.77	1.44
57	Bifenthrin	13.53	440.20>181.15	-17	0.9975	0.005	109.20	118.15	8.67	2.63
58	Flonicamid	5.14	273.95>228.15	8	0.9949	0.005	111.70	108.15	6.33	11.82
59	Fludioxonil	9.17	247.10>180.15	28	0.9975	0.005	103.20	91.64	18.53	19.51
60	Fipronil	9.85	434.90>330.00	16	0.9946	0.005	110.40	100.54	9.33	8.84
61	Flubendiamide	9.91	680.90>254.10	27	0.9993	0.005	100.00	114.81	6.70	10.15
62	Novaluron	11.10	491.00>470.90	13	0.9952	0.005	110.50	104.58	15.21	19.83

Table 5 Summary results of GC-MS/MS analysis

ID	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE	Determination Coefficient (R ²)	LOQ	% Accuracy at LOQ	Recovery at LOQ (%)	Precision	
						mg/kg			%RSD _R (n=6)	%RSD _F (n=6)
1	Propamocarb	7.22	188.15>58.10	12	0.9988	0.01	100.24	117.82	2.50	0.26
2	Tetrahydrophthalimide (THPI) as Captan deg.	8.06	151.10>79.00	18	0.9940	0.025	108.10	77.46	24.68	17.20
3	Diazinon	10.89	304.10>179.20	19	0.9978	0.005	99.82	75.70	8.72	4.91
4	Pyrimethanil	11.08	198.10>118.10	30	0.9984	0.005	104.52	76.02	5.13	3.04
5	Metalaxyl	12.49	234.10>146.20	20	0.9976	0.005	95.69	86.61	8.19	5.74
6	Linuron	13.16	248.00>61.00	16	0.9994	0.01	99.45	76.64	7.63	11.14
7	Malathion	13.16	157.95>125.00	9	0.9986	0.005	99.05	82.78	4.84	5.44
8	Chlorpyrifos	13.39	313.95>257.90	17	0.9974	0.005	105.54	83.98	12.54	11.59
9	Cyprodinil	14.41	224.15>222.10	24	0.9969	0.005	97.90	75.91	9.39	8.45
10	Fipronil	14.64	367.00>213.00	29	0.9971	0.005	103.15	80.83	9.63	10.23
11	Triflumizole	15.06	278.05>73.10	8	0.9700	0.01	81.32	74.75	19.80	18.95
12	Thiabendazole	15.16	174.10>65.00	28	0.9822	0.005	103.26	78.00	10.67	19.61
13	Flutriafol	15.99	219.10>123.10	21	0.9945	0.005	111.12	87.45	12.42	4.92
14	Profenofos	16.33	339.00>268.90	15	0.9979	0.01	96.56	84.84	8.74	12.02
15	Fludioxonil	16.53	248.05>127.10	27	0.9959	0.005	109.75	75.29	4.38	5.06
16	Myclobutanil	16.78	179.05>152.00	9	0.9903	0.005	113.74	80.70	5.67	4.22
17	Buprofezin	16.82	172.10>57.10	21	0.9949	0.005	104.77	75.56	7.23	13.44
18	Chlorfenapyr	17.06	247.00>227.00	14	0.9824	0.01	81.81	71.85	12.95	17.79
19	Trifloxystrobin	18.81	222.05>190.10	5	0.9925	0.005	107.32	82.26	13.02	6.73
20	Propiconazole-1	18.85	172.95>109.00	25	0.9987	0.005	96.01	84.37	8.91	13.24
21	Quinoxifen	18.90	237.00>208.10	27	0.9987	0.005	102.04	75.14	5.35	5.50
22	Propiconazole-2	19.07	172.95>109.00	25	0.9973	0.005	106.50	80.86	11.47	9.79
23	Fenhexamid	19.11	177.00>113.00	17	0.9930	0.005	112.79	71.02	10.07	8.15
24	Fluopicolide	19.17	209.00>182.00	19	0.9990	0.005	101.13	83.83	7.84	5.94
25	Tebuconazole	19.58	250.10>125.10	21	0.9956	0.005	105.02	78.48	10.11	12.67
26	Piperonyl-butoxide	19.84	176.05>131.10	13	0.9977	0.005	103.47	80.31	5.39	2.62
27	Iprodione	20.48	314.00>245.00	12	0.9990	0.005	96.40	69.39	17.74	13.68
28	Fluxapyroxad	20.70	381.10>159.10	16	0.9983	0.005	103.10	86.18	5.18	5.99
29	Bifenthrin	20.74	181.05>165.10	22	0.9963	0.005	107.30	84.43	5.33	4.87
30	Bifenazate	20.96	300.10>258.10	9	0.9897	0.005	100.51	61.30	11.86	9.26

Table 5 Summary results of GC-MS/MS analysis (Continued)

ID	Compound Name	Ret. Time (min)	Target MRM (m/z)	CE	Determination Coefficient (R ²)	LOQ mg/kg	% Accuracy at LOQ	Recovery at LOQ (%)	%RSD _R (n=6)	%RSD _D (n=6)
31	Chlorantranilprole	20.99	278.00>249.00	20	0.9927	0.005	89.41	63.24	13.18	12.74
32	Etoxazole	21.03	330.10>57.10	24	0.9941	0.01	107.66	88.50	11.16	4.68
33	Fenpropathrin	21.07	265.05>210.10	12	0.9994	0.005	96.32	77.59	11.35	8.58
34	Pyriproxyfen	22.24	136.10>78.00	24	0.9925	0.005	106.67	88.83	11.43	4.31
35	Lambda-Cyhalothrin	22.62	208.05>181.10	9	0.9983	0.005	100.58	88.73	5.68	4.71
36	Spirodiclofen	23.83	312.00>109.10	21	0.9863	0.005	87.86	74.66	15.32	6.05
37	Permethrin-1	24.10	162.95>127.00	9	0.9947	0.005	94.03	79.40	9.89	4.36
38	Permethrin-2	24.35	162.95>127.10	9	0.9968	0.005	104.90	79.42	4.44	5.73
39	Pyridaben	24.36	147.15>117.10	24	0.9961	0.005	108.30	85.03	8.22	9.19
40	Fenbuconazole	25.08	198.10>129.10	12	0.9986	0.005	103.44	83.32	4.42	3.68
41	Cyfluthrin-1	25.17	226.05>206.10	15	0.9932	0.01	104.25	75.65	12.42	15.97
42	Cyfluthrin-2	25.37	226.05>206.10	15	0.9798	0.01	85.41	85.47	13.12	10.49
43	Cyfluthrin-3	25.48	226.05>206.10	15	0.9894	0.01	98.45	86.09	12.40	9.76
44	Cyfluthrin-4	25.58	226.05>206.10	15	0.9975	0.01	95.49	88.12	9.80	6.57
45	Boscalid	25.85	140.10>76.00	24	0.9980	0.005	102.56	81.28	3.45	3.54
46	Cypermethrin-1	25.78	162.95>127.00	9	0.9950	0.005	108.15	85.96	7.36	7.41
47	Cypermethrin-2	26.00	162.95>127.00	9	0.9957	0.005	93.46	78.84	9.92	15.88
48	Cypermethrin-3	26.09	162.95>127.00	9	0.9986	0.005	101.76	72.57	7.35	10.27
49	Cypermethrin-4	26.18	162.95>127.00	9	0.9954	0.005	90.17	81.45	8.22	2.72
50	Pyraclostrobin	27.68	164.05>132.10	12	0.9989	0.005	98.49	83.86	3.69	4.20
51	Difenoconazole-1	28.35	323.05>264.90	18	0.9984	0.005	102.08	83.68	9.86	8.59
52	Difenoconazole-2	28.46	323.05>264.90	18	0.9977	0.005	100.08	76.02	10.03	5.15
53	Indoxacarb	28.79	264.05>176.00	15	0.9969	0.005	103.80	74.27	12.26	6.14
54	Azoxystrobin	29.26	344.10>329.00	21	0.9956	0.005	105.91	78.64	5.13	10.39
55	Dimethomorph-1	29.48	301.05>165.10	15	0.9950	0.005	101.80	84.57	6.02	4.92
56	Dimethomorph-2	30.05	301.05>165.10	15	0.9969	0.005	104.76	86.63	4.97	5.63

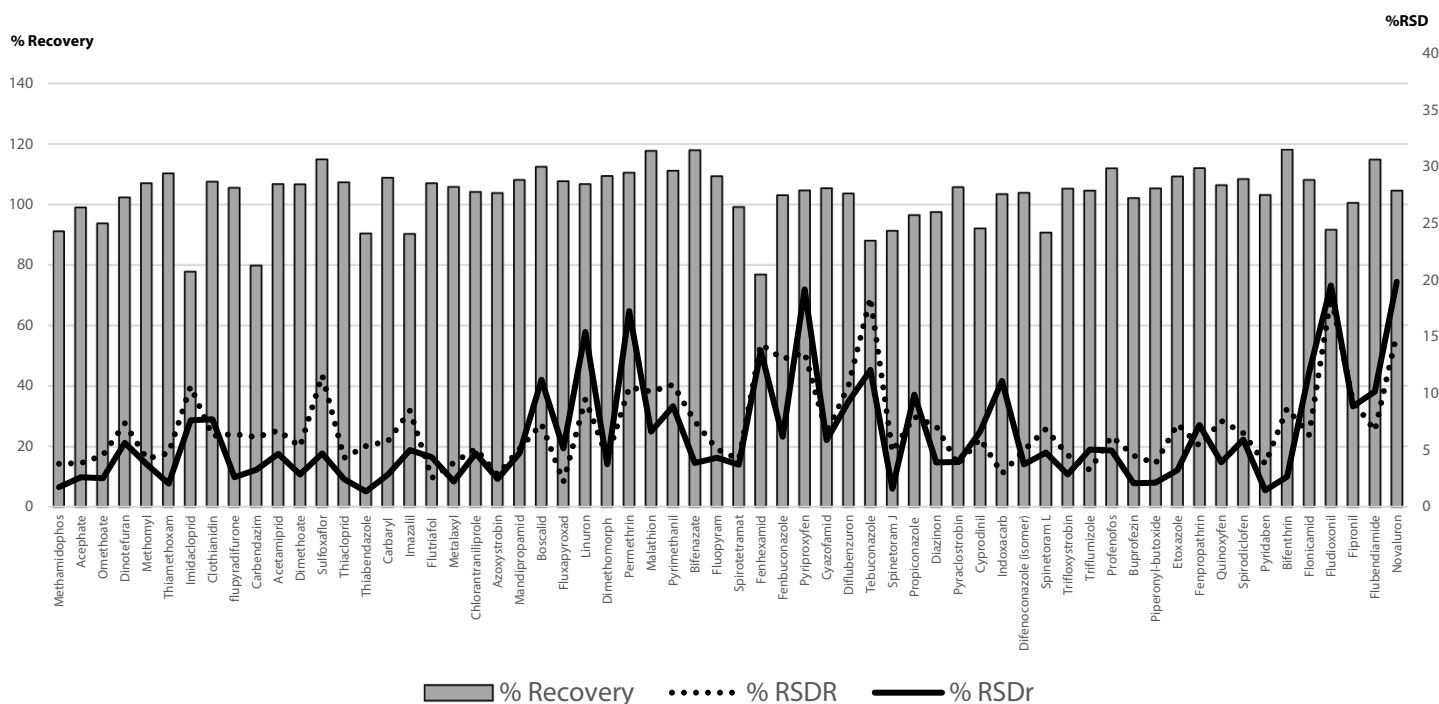


Fig. 6 Trend graph of summary results on LC-MS/MS

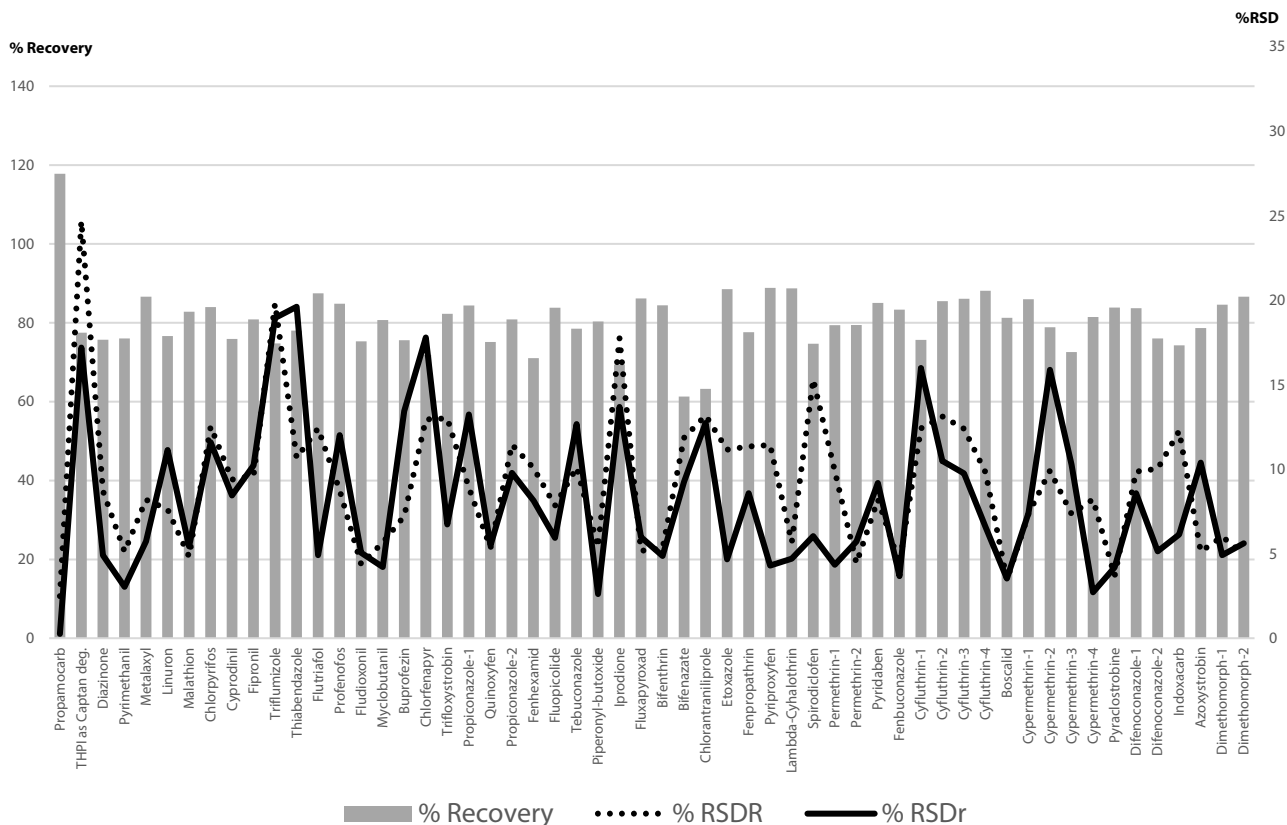


Fig. 7 Trend graph of summary results on GC-MS/MS

4. Conclusion

This study shows that the modified QuEChERS method combined with LC-MS/MS and GC-MS/MS systems is a reliable and efficient tool to quantify residual pesticides in norbixin sample. Although oleoresin is a complex matrix, the modified QuEChERS method significantly reduces interference. Also, highly sensitive Shimadzu LC-MS/MS and GC-MS/MS allows trace level detection even after multifold dilution of sample. This helps in reducing contamination and enhancing ruggedness resulting in reproducible detection of analytes.

5. References

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06-SAIP-F-04-EN

First Edition: May. 2022