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Dissipation kinetics, dietary risk assessment of indoxacarb residue in/on sapota using LC-MS/MS under South Gujarat condition

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Abstract
A simple, sensitive and reproducible analytical method, an efficient triple quadrupole mass spectrometer used for the quantification of indoxacarb residues in sapota fruit under South Gujarat conditions was validated using LC-MS/MS. The validated method was linear (0.001-0.25 µg/ml) having correlation coefficients (R^2) value lyinges 0.998 to 0.999. The LOD and LOQ were 0.001 and 0.004 µg/g, respectively. Modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction technique involved for extraction of indoxacarb residues with 1% acetic acid in acetonitrile provided acceptable recoveries in the range of 98.63 ± 6.20 to 100.50 ± 1.21 per cent at 0.01, 0.05 and 0.10 µg/g spiking level with precision relative standard deviation (RSD) range from 1.20 to 6.29 per cent. Initial deposit on sapota fruits following two application (15 days interval between application) of indoxacarb 14.5% SC at 145 and 290 g a i/ha was 0.203 µg/g and 0.243 µg/g and dissipated to 0.009 and 0.015 µg/g, respectively, on 10 days after application during 2019-20 and 2020-21. Half-life were 2.15 and 2.51 days, safe waiting period (SWP) on the basis of Codex MRL (0.02 µg/g) limit were ranged from 7.17-9.05 days for RD and 9.42-10.27 days for 2 x RD respectively, for the year 2019-20 and 2020-21. As per European Union (EU) and United States (US) MRL values (1 µg/g) at RD and 2 x RD the residues of indoxacarb were found below MRL on day 0 during both years under South Gujarat conditions. Dietary risk assessment (Risk quotient <1) was performed on the basis of field trial suggested that application of indoxacarb at recommended dose 14.5% SC at 145 g a i/ha is safe for the consumers.

Keywords: Dissipation, LC-MS/MS, indoxacarb, Sapota fruit, dietary risk assessment

Introduction
Sapota [*Manilkara achras* (Mill.) Forsberg] is an evergreen fruit tree species from the family Sapotaceae a native of Mexico and Tropical America and introduced in India probably during 1898 in '*Gholwad'* village of Maharashtra State (Sulladmath and Reddy, 1990) [17]. It is commonly known by different names in India as *Chiku* or *Chikoo* in Hindi, Marathi and Gujarati. Among the 41 varieties grown all over India, Kalipatti is an excellent variety of sapota and is popularly cultivated in Gujarat due to its excellent taste, aroma and soft mellow flesh with less number of seed, high productivity, continuous fruiting throughout the year. Gujarat region, bud borer, *Anarsia achrasella* Bradley; chiku moth, *Nephopteryx eugraphella* (Ragonot); seed borer, *Trymalitis margarias* Meyrick; Fruit fly, *Bactrocera* spp. and midrib folder, *Banisia myrsusaleselearalis* Walker are major insect pests and found throughout the year. There is a number of insecticides are available in market and as well used by farmers to control the pests damage. The extensive use of pesticides on one hand improved the agricultural productivity many folds, but on the other hand, they are posing a severe threat to the ecology and environment with widespread pollution. Which may pose a serious threat to environmental quality and indeed can lead to acute and chronic effects on human life and biodiversity (Jamil *et al*., 2004) [7]. Information regarding indoxacarb persistence dissipation behavior and is not available particularly for Heavy Rainfall Zone (AES-III) of South Gujarat.
Indoxacarb is a non-systemic, reduced risk and synthetic organophosphate replacement insecticide that belongs to oxadiazine group used for managing lepidopteron larvae and sucking insects infesting many vegetables and commercial crops (United States Environmental Protection Agency 2000) [20]. The indoxacarb 14.5% SC (suspension concentrate) formulation was registered in India for managing lepidopteron larvae infesting cotton, fruits and vegetables. It mainly acts on the nervous system by blocking the voltage dependent sodium channel in insect nerve cells resulting in paralysis and death of larvae (McCann *et al*. 2001)

[10]. The consumption of food grains, vegetables and fruits contaminated with indoxacarb leads to the acute onset of methemoglobinemia; a condition which reduces the effectiveness of red blood cells to exchange oxygen with organs can be noticed (Prasanna *et al*. 2008).

Material and Methods

a. Materials and reagents

The commercial formulation insecticide product King Doxa® (Indoxacarb 14.5% SC) was purchased from local pesticide shop, Navsari, Gujarat, India. Certified reference materials of indoxacarb having purity 95% was produced from Sigma-Aldrich Pvt. Ltd. (Bangalore, India). MS-grade acetone, acetonitrile, n-hexane, magnesium sulphate, sodium chloride, sodium sulphate, methanol and water were purchased from Merck (Darmstadt, Germany). LC-MS grade ammonium formate and formic acid (99.5+%, Optima™) were purchased from Fisher Chemical (Fair lawn, NJ, USA). Primary secondary amines (PSA) were purchased from Supelco Sigma Aldrich (Germany). Stock solution of indoxacarb (14.15 mg) in methanol: water (80:20 v/v) concentration of 268.85 µg/ml was prepared and stored at -20 ^{0}C. The stock solutions were diluted to formulate the intermediate and then working standards were prepared for further analysis.

b. Apparatus

Samples were processed using a heavy-duty variable speed homogenizer (SRK instruments, Gujarat), centrifuge (Eppendorf, Germany) and Turbovap (Caliper life Science, PerkinElmer, USA). The indoxacarb was analysed on LC-MS/MS-TSQ Quantum Access Max® equipped with UHPLC having Dinonex Ultimate 3000 RS Pump (Thermo Fisher, USA).

c. Field experiment

The field experiment on sapota var. Kalipatti was conducted following good package of practices. The experimental field was located at the Fruit Research Station, Gandevi, Navsari Agricultural University, Navsari, India with GPS position of 20° 81' N latitude and 73° 02' E at an altitude of 7.6 meters above the Mean Sea Level (MSL) during winter 2019-2020 and 2020-2021. Indoxacarb (14.5% SC) insecticide was tested as two foliar sprays at 15 days interval, first spray being applied at marble size fruit stage. In accordance with Central Insecticide Board and Registration Committee (CIBRC), the treatments of recommended dose (RD) 145 g a i/ha and double to recommended dose (2RD) 290 g a i/ha. Were evaluated against the control (water spray).

d. Sample collection and preparation

From each treatment across the three replications, approximately 2 kg sapota fruits were harvested at 0 day (2 hrs. after application), 1, 3, 5, 10 and 20 days after the last spray of insecticide. the collected samples were kept in air tight container and brought to the laboratory for pesticide residues analysis.

e. Sample extraction and cleanup

The samples were processed and analysed in Food Quality Testing Laboratory. NMCA, NAU, Navsari, Gujarat, India. Each sample were analysed as per modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method for fruits and vegetables (AOAC, 2007, Sharma, 2013) [2, 15]. The sapota fruit samples were cut and homogenized by heavy-duty variable homogenizer and a representative sample (15 ±0.1 g) was taken in 50 ml capacity polypropylene centrifuge tubes. In sample, 1% acetic acid in acetonitrile (15 ml) was added and kept in a deep freeze at -20^0 C for 10-20 minutes. The mixture of anhydrous $MgSO_4$ (6.0 g) and sodium acetate (1.5 g) added and shake for 1 min and vortex for 30 seconds. The content was subjected to centrifugation for 3 minutes at 3500 rpm to separate organic layer. The supernatant (6 ml) was transferred to 15 ml capacity centrifuge tube containing 300 mg PSA and 900 mg anhydrous $MgSO_4$ and vortex for 30 seconds and then centrifuged again or 2 minutes at 3500 rpm and take 2 ml supernatant (aliquot) in test tube and evaporated it to dryness with TurboVap at 40°C. Finally make up the volume to 2 ml using methanol: water (80:20 v/v) for LC-MS/MS. The samples were filtered through syringe filters (0.22 µm, pore size) before injected for quantification on LC-MS/MS.

f. Instrumental determination (LC-MS/MS)

The quantitative analysis of indoxacarb was performed on Thermo Scientific Dionex UltiMate-3000 made QqQ detector (MS/MS) with column Accucore® C_{18} (100 mm X 2.1 mm i.d 2.1 µm) with a flow rate 0.3 ml/min. at 30°C. An elution gradient was used with solvent A (Water + 0.1% Formic acid + 5Mm Ammonium Formate) and B (Methanol + 0.1% Formic acid + 5Mm Ammonium Formate) with gradient profile (t (min) % A). The QqQ MS/MS parameters of indoxacarb were optimized in positive ionization mode with capillary voltage 4500 V, vaporizer temperature was 350 ^{0}C sheath gas (N_2) 48 arbitrary unit, aux gas (N_2) 18 arbitrary unit, ion transfer capillary temperature 325^{0}C and tube lens 89 V. The masses were monitored and optimized using standard parameters; Precursor ion 528.0 m/z and product ion 150.06 m/z (Collision energy; 25eV) and 203.02 m/z (Collision energy; 38 eV) as depicted in Figure 1. Chromatogram of a indoxacarb standard with retention time (RT) 10.20 min is presented in Figure 1. The data were processed using LCQUAN™ 2.9 QF1 software (Thermo Scientific).

g. Analytical method validation

The performance of the method was developed and validated as per SANTE guidelines (SANTE. 2019) [14] by studying different parameters that include the following aspects.

1. Linearity studies

The linearity of indoxacarb in methanol: water (80:20 v/v) were obtained using six calibration standards ranging from 0.001-0.25 µg/g. The linearity relationships among the ratios of the peak area and the concentrations were observed.

2. LOD and LOQ

Five repetitive responses on specific instruments for indoxacarb at different concentration (particularly in linear dynamic range) was recorded to establish the limit of detection (LOD) and the limit of quantification (LOQ) of the analytical method using the following formula, LOD (µg/g) = (mean of standard deviation/slope) × 3 and LOQ (µg/g) = (mean of standard deviation/slope) × 10 (Patil et.al, 2018).

3. Accuracy and precision

The accuracy and precision were evaluated through recovery study for indoxacarb. Three concentration levels of fortification for indoxacarb (0.01, 0.05 and 0.10 µg/g) in sapota fruit matrix was used with five replications (n=5). The consistency of the recovery study results reflects the precision, which can be represented by the relative standard deviation (RSD %).

4. Dietary risk assessment

The residue obtained from sapota fruit subjected to spray of indoxacarb at recommended dose (RD) 145 g a.i./ha and double to recommended dose (2×RD) 290 g a.i./ha collected on 0 days (2 hrs. after application) were used to work out the risk quotient (RQ). The estimated daily intake (EDI) of indoxacarb was calculated by multiplying the product of pesticide concentration (µg/g) with average food consumption rate (100 g/day) divided by the mean body weight of different group of Indian consumers (kg) (ICMR, 2011) [6]. The long-term risk assessment of intakes compared to pesticide toxicological data was assessed by calculating the risk quotient (RQ), divided by EDI by the relevant acceptable daily intake (ADI) expressed in mg/kg body weight (bw). ADI value of indoxacarb is 0.006 mg/kg body weight (bw) (EFSA, 2020). Numerically, the RQ value is more than 1 indicates the chemical poses risk to the consumers (Kansara *et al.*, 2021) [8].

h. Statistical analysis

The dissipation of indoxacarb in sapota fruit was calculated by using Single First Order dissipation model using the equation $C_t = C_0 e^{-kt}$ where C_t is the insecticide concentration at time t; C_0 is the initial concentration and k is the dissipation constant. The residue data were subjected to statistical analysis according to Hoskins (1961) to compute the residual half-life (DT_{50}) and Waiting period.

Results and Discussion

Method validation

The linearity studies of indoxacarb at different levels (0.001, 0.0025, 0.005, 0.01, 0.025, 0.05, 0.1 and 0.25µg/g) in 80:20 v/v methanol: water on LC-MC/MS. The correlation coefficient R^2 (n=5) values of indoxacarb were 0.998 to 0.999 for sapota matrix. The obtained values were in accordance with the acceptable limit of $R^2 \geq 0.99$ as per SANTE guideline 2019 [14]. The matrix effect of indoxacarb for methanol: water (80:20 v/v) and sapota fruit, was found to be in the range of 0.86 to 5.96 per cent. Here, matrix effect is found in the range between -20 per cent and 20 per cent which doesn't enhance and suppressed the response so matrix effect is ignored (Sakthiselvi *et al.*, 2020) [13]. The LOD and LOQ values of indoxacarb in sapota fruit worked out were 0.001 µg/g and 0.004 µg/g respectively (Table 1). The observed LOQ values of indoxacarb were lower than maximum residue limit (MRL) values fixed for sapota i.e., Codex MRL (0.01 µg/g), European Union maximum residue level (1 µg/g) and US MRL (1 µg/g). It indicates that the respective instruments were sensitive enough to abide the internationally acceptable standard. The average recovery of indoxacarb was ranged from 98.63 to 100.50 per cent in sapota fruit, with relative standard deviation ranged from 1.20 to 6.29 per cent when spiked the samples (0.01, 0.05 and 0.1 µg/g level of sampling) (Table 1). All the results of recoveries and RSDs were within the acceptable criteria of SANTE guidelines (SANTE, 2019) [14] i.e. Recovery (70–120%) and RSD (<20%). The validation of the developed method by fortification indoxacarb in cabbage showed good accuracy and precision (Sun *et al.*, 2012) and Urvashi *et al.* (2012) [18, 19] by employing standardized QuEChERS technique in cabbage at PAU, Ludhiana.

2. Residue analysis

The persistence and dissipation pattern of indoxacarb pertaining to recommended (145 g a.i./ha) and double to recommended dose (290 g a.i./ha) in sapota fruit from the open field experiment are presented in Table 2. The initial deposits of indoxacarb residues in sapota fruits during year 2019-20 and 2020-21 were 0.049 ± 0.011µg/g and 0.065 ± 0.020 µg/g, respectively (0 day after 2hr). The initial residues of indoxacarb in recommended dose steadily and progressively dissipated up to 95.65 per cent and 93.72 per cent within the 10 days during year 2019-20 and 2020-21, respectively. (Table 2). In first order kinetics, during the year 2019-20 and 2020-21, the coefficient of determination (R^2) of deltamethrin in RD were 0.956 and 0.977, respectively. The half-life (DT_{50}) was 2.15 days and 2.51 days and DT_{90} was 7.13 days and 8.34 days, respectively during 2019-20 and 2020-21. The present finding on DT_{50} of indoxacarb @ recommended dose is differing with the results reported with DT_{50} for cabbage 2.8 to 4.6 days in two enantiomers (Sun *et al.*, 2012) and 2.88 days (Urvashi *et al.*, 2012) [18, 19], in cucumber and tomato 3.0 days and 5.9 days, respectively (Wang *et al.*, 2013) [21], tomato 2.37 days (Anita *et al.*, 2018) [1], pomegranate 7.4 days (Mohapatra *et al.*, 2019) [11], Pigeonpea 1.13 days (Naik *et al.*, 2020), tomato 3.12 days (Sakthiselvi *et al.*, 2020) [12, 13].

The indoxacarb residues in treatment receiving doble to recommended dose (2 × RD) in sapota fruits during 2019-20 and 2020-21 were presented in Table 2. The highest indoxacarb residues 0.540 ± 0.084 µg/g and 0.313 ± 0.105 µg/g were found at 0 day (2 hrs. after application) and continued to dissipated up to 96.04 per cent and 94.63 per cent within the 10 days during the year 2019-20 and 2020-21, respectively. In first order kinetics, the coefficient of determination (R^2) of indoxacarb in 2 x RD were 0.991and 0.943 respectively, While, in case of half-life (DT_{50}) were 2.16 days and 2.37 days and DT_{90} were 7.17 days and 7.89 days, respectively, during 2019-20 and 2020-21 and found below determination limit after 20 DAA of indoxacarb during both years. The present finding on DT_{50} of indoxacarb for 2 x RD is differing from the results reported with DT_{50} in cabbage 1.92 days (Urvashi *et al.*, 2012) [19], rice at 1.5 RD is 5.83 days (Li *et al.*, 2016), tomato 2.48 days (Anita *et al.*, 2018) [1], pomegranate 8.4 days (Mohapatra *et al.*, 2019) [11], Pigeonpea 1.23 days (Naik *et al.*, 2020) [12], tomato 3.21 days (Sakthiselvi *et al.*, 2020) [13]. This variation might be the resultant of the prevailing environmental factors and processes *viz.*, temperature, relative humidity, volatilization and photo-degradation at field conditions which are not consistent in different geographical regions.

3. Dietary risk assessment

The cumulative dietary risk quotient (RQ) calculated based on the collective pesticide residues for indoxacarb was less than 1 from 0 day (2hr) at RD and DRD (Table 3). It signifies that sapota fruit collected from the field was safe for consumption. It indicates that the indoxacarb will not cause any adverse effect to consumer after consumption of sapota fruit. Therefore, the consumption of sapota fruit laced with indoxacarb at recommended dose with an observed waiting period of 10 days are safer as their RQ values are lower than 1 at 0-day (2 hr).

Table 1: Method validation studies of indoxacarb in sapota fruits

Parameter	Particular		Indoxacarb
Linearity (n=5)	Calibration range		0.01 to 0.25 (μg/g)
	Regression equation		y = 865715x + 635.50
	R^2 {R^2 ≥0.99}		0.998 to 0.999
	Matrix effect {-20% to 20%}		0.86 to 5.96 %
Sensitivity (n=5)	LOD (μg/g)		0.001
	LOQ (μg/g)		0.004
Accuracy (n=7)	% Recovery {70-120}	F level (μg/g) 0.01 to 0.10	98.63 to 100.50 %
Precision (n=7)	%RSD {≤ 20%}	F level (μg/g) 0.01 to 0.10	1.20 to 6.29 %

N: number of replication; R^2: Correlation coefficient; LOD: Limit of detection; LOQ: Limit of quantification; F level: Fortification level, RSD: Relative standard deviation; value given in parenthesis {} are the standard acceptance criteria as per SANTE 2019

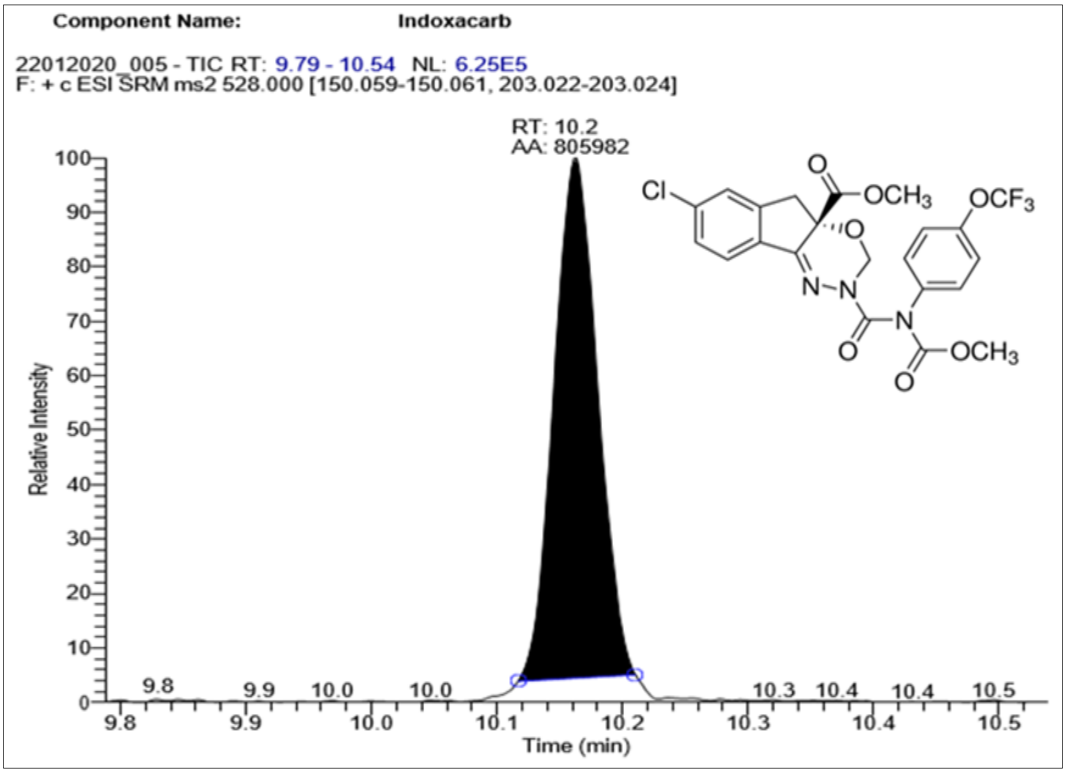

Fig 1: Chromatogram of matrix match standard of indoxacarb on LC-MS/MS

Table 2: Persistence and dissipation behaviour of indoxacarb in sapota fruits

DAA	2019-20				2020-21			
	Dose (RD @ 145 g a.i./ha)		Dose (2XRD @ 290 g a.i./ha)		Dose (RD @ 145 g a.i./ha)		Dose (2XRD @ 290 g a.i./ha)	
	Residue (μg/g ±SD)	Dissipation (%)	Residue (μg/g ±SD)	Dissipation (%)	Residue (μg/g ±SD)	Dissipation (%)	Residue (μg/g ±SD)	Dissipation (%)
0	0.203±0.018	0.00	0.540±0.084	0.00	0.243±0.067	0.00	0.313 ±0.105	0.00
1	0.130±0.015	36.14	0.364±0.056	32.56	0.148±0.033	38.92	0.219 ±0.076	30.13
3	0.035±0.006	82.75	0.157±0.070	71.01	0.058±0.023	76.27	0.120 ±0.053	61.74
5	0.011±0.001	94.54	0.084±0.044	84.38	0.026±0.011	89.50	0.060 ±0.028	80.87
10	0.009±0.001	95.65	0.021±0.007	96.04	0.015±0.005	93.72	0.017±0.005	94.63
20	BQL	-	BQL	-	BQL	-	BQL	-
Reg. eq.	$y = 0.133e^{-0.323x}$		$y = 0.477e^{-0.321x}$		$y = 0.1728e^{-0.276x}$		$y = 0.2919e^{-0.292x}$	
R^2	0.956		0.991		0.977		0.943	
DT_{50}	2.15 days		2.16 days		2.51 days		2.37 days	
DT_{90}	7.13 days		7.17 days		8.34 days		7.89 days	
WP	Codex	7.17 days	Codex	10.27 days	Codex	9.05 days	Codex	9.42 days
	EU	1 day	EU	1 day	EU	1 day	EU	1 day
	US	1 day	US	1 day	US	1 day	US	1 day

MRL: Codex: 0.02 μg/g (Ref: www.fao.org) EU: 1.00 μg/g (ref: www.efsa.europa.eu) US: 1.00 μg/g (ref: www.fas.usda.gov)

Table 3: Health risk assessment in sapota for indoxacarb

Group	Age group (year)	Body weight (kg)	2019-20				2020-21			
			RD		2 x RD		RD		2 x RD	
			EDI	RQ	EDI	RQ	EDI	RQ	EDI	RQ
Children	1-3	12.9	0.0016	0.0262	0.0042	0.0698	0.0019	0.0314	0.0024	0.0404
	4-6	18	0.0011	0.0188	0.0030	0.0500	0.0014	0.0225	0.0017	0.0290
	7-9	25.1	0.0008	0.0135	0.0022	0.0359	0.0010	0.0161	0.0012	0.0208
Boy	10-12	34.3	0.0006	0.0099	0.0016	0.0262	0.0007	0.0118	0.0009	0.0152
Girl	10-12	35	0.0006	0.0097	0.0015	0.0257	0.0007	0.0116	0.0009	0.0149
Boy	13-15	47.6	0.0004	0.0071	0.0011	0.0189	0.0005	0.0085	0.0007	0.0110
Girl	13-15	46.6	0.0004	0.0073	0.0012	0.0193	0.0005	0.0087	0.0007	0.0112
Boy	16-18	55.4	0.0004	0.0061	0.0010	0.0162	0.0004	0.0073	0.0006	0.0094
Girl	16-18	52.1	0.0004	0.0065	0.0010	0.0173	0.0005	0.0078	0.0006	0.0100
Man	Sedentary work	60	0.0003	0.0056	0.0009	0.0150	0.0004	0.0068	0.0005	0.0087
	Moderate work	60	0.0003	0.0056	0.0009	0.0150	0.0004	0.0068	0.0005	0.0087
	Heavy work	60	0.0003	0.0056	0.0009	0.0150	0.0004	0.0068	0.0005	0.0087
Woman	Sedentary work	55	0.0004	0.0062	0.0010	0.0164	0.0004	0.0074	0.0006	0.0095
	Moderate work	55	0.0004	0.0062	0.0010	0.0164	0.0004	0.0074	0.0006	0.0095
	Heavy work	55	0.0004	0.0062	0.0010	0.0164	0.0004	0.0074	0.0006	0.0095

Conclusion

QuEChERS method adopted for extraction with acetonitrile and quantification on LC-MS/MS (indoxacarb) is accurate, precise and sensitive enough as per SANTE guidelines (2019) [14] for estimation of indoxacarb from sapota fruit. Indoxacarb followed first order dissipation kinetics (R^2) in sapota fruit with the half-life (DT_{50}) ranges from 2.15 – 2.51 days, 90 per cent dissipation time (DT_{90}) ranges from 7.89 -8.34 days. The waiting period of deltamethrin ranges from 7.17 – 9.05 days in recommenced dose (145 g a.i./ha) under South Gujarat conditions. The dietary risk quotient (RQ) was found < 1 which signifies that sapota fruits collected from the field was safe for consumption of Indian population.

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