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Study on the effect of grain storage and processing on deltamethrin residues in post-harvest treated wheat with regard to baby food safety requirements

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Abstract

The effects of storage intervals and of milling procedures on dissipation of deltamethrin residues in post-harvest treated wheat grain were studied with the aim of obtaining scientific data on compliance of the processed products with the safety requirements concerning baby foods. The insecticide formulation was applied on stored wheat at recommended rate of active ingredient of 0.5 mg kg⁻¹ and at the higher rate of 4 mg kg⁻¹ providing the highest protective effect. The loss of residues and their distribution in different fractions of the milled grain were studied after various storage intervals, from 7 to 270 days after treatment. Eight fractions - bran, semolina, 3 types of groats and 3 types of flour were collected after milling of grain and analyzed for pesticide residues. The residues were determined by a GC method characterized by limit of determination 0.005 mg kg⁻¹ - low enough for enforcement of the maximum residue level of 0.01 mg kg⁻¹ established by European Commission Directive for any pesticide in cereal-based foods. Deltamethrin applied post-harvest on wheat as a grain protectant is characterised by low rate of degradation on the grain under practical storage conditions. 180 days after treatment at an application rate 0.5 mg kg⁻¹ the residues were between 0.03 and 0.2 mg kg⁻¹ in the various types of flour. 270 days after treatment at rate 4 mg kg⁻¹ the residues in flour were in the range 0.4 - 1.5 mg kg⁻¹.

Keywords: deltamethrin, wheat grain, post-harvest treatment, residues, processed products, baby food

Introduction

Food safety is one of the most serious problems of the modern society. Contamination of plant production with pesticide residues is a source of risk for human health. Obtaining a

knowledge of the effects of processing on the fate of residues is one of the basic pre-conditions for refinement of pesticide risk assessment and especially of potential risk to consumers of high vulnerability (Regulation (EC) No 396/2005). Special attention has to be paid to the health protection of infants and young children as they represent the most vulnerable group of population. Infants and young children have been shown to be very sensitive to certain toxic effects (Matsumura F. 2004, National Research Council 1993). Furthermore, babies and young children are exposed to relatively higher doses of toxicants per unit body weight, compared to adults because of their proportionally higher consumption of food. Based on FDA data, children receive 3 - 5 times more cereals than adults per body weight. For better protection of the health of infants and young children a general MRL of 0.01 mg kg⁻¹ for any individual pesticide in processed cereal-based foods and baby foods has been established by EC (European Commission 2003^a, European Commission 2003^b).

Post-harvest treatment for protection of raw grain against stored-product pests has been reported as a source of contamination of cereals (JMPR 1991, JMPR 1992). A summary of residue monitoring data sorted by frequency of incidents showed that grain protectants used on cereals accounted for most of the positive findings (Hamilton et al. 1997). Studies on the effect of storage on insecticides residues following post-harvest treatments have generally shown that residues decline rather slowly (Holland et al. 1994). For refining of dietary intake estimates of pesticide residues a generation of food processing studies data has been required. Milling is one of the 10 processing methods identified as deserving main attention (World Health Organization 1997). Studies on the fate of pesticide residues on grain subjected to milling and processing showed that the bulk of the insecticides deposited on the grain remained in the epidermis and was removed from the bran during milling. Most residues were found in the outer portion of grains (Hajslova, 2000). The concentration on the bran was from

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3 1 ~~2 to 3.2 times the concentration on the whole wheat~~ (Desmarchelier 1979^a, Desmarchelier
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5 2 1979^b). Bran/grain concentration factors in the range of 2 to 6 were reported in many studies
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7 3 (JMPR 1988, JMPR 1989, JMPR 1991, JMPR 1992). Generally the concentration of
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9 4 insecticide in flour was 10% or less of the concentration on the whole wheat.
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15 6 Deltamethrin has been used as grain protectant for more than 30 years. The MRL of 1 mg kg⁻¹
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17 7 is established for wheat grain intended for general nutritional uses and this value
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19 8 accommodates post-harvest treatment of the commodity (European Commission 2002). The
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21 9 MRL established by EC (European Commission 2003^a, European Commission 2003^b). for
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23 10 residues in baby food is 100 times below the deltamethrin MRL for food of general
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25 11 consumption. The enforcement of this very stringent limitation of residues in processed
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27 12 cereal-based baby food imposes the necessity of careful tuning of all technological parameters
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29 13 in order to select procedures that ensure baby food production in compliance with the safety
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31 14 requirements from potentially contaminated raw materials.
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39 16 For the determination of residues at ultra-trace levels considerable attention has to be paid to
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41 17 the problems of assurance the high quality of analytical methods both in respect of
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43 18 quantitative parameters (sensitivity, trueness, precision, and range) and of qualitative
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45 19 assessment (identification and confirmation). Implementation of analytical methods capable
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47 20 to determine the pesticide studied at or below 0.01 mg kg⁻¹ was mandatory in the study.
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53 22 The study was aimed at assessment of the risk of baby food MRL violation due to utilization
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55 23 of contaminated wheat grain for processing and production of baby food. The objectives of
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57 24 this work were: to study the influence of the wheat grain storage intervals and of milling on
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59 25 the decline and distribution of residues of deltamethrin applied post-harvest on wheat grain; to
60

1 establish the residue balance in processed products; to provide scientific data on compliance
2 of the processed post-harvest treated wheat grain products with the safety requirements
3 concerning baby foods.

4 5 **Experimental**

6 All processes applied to the study for processing effects assessment were planned in a way to
7 correspond as closely as possible to those that occur in actual practices.

8 9 *Wheat treatment, sampling and processing.*

10 High grain quality wheat (*Triticum estivum*) cultivar “Momchil” was used in the experiments.
11 The initial grain moisture was $14.0 \pm 0.5\%$. The commercial insecticide formulation was Decis
12 2.5 EC containing 25 g.l^{-1} active ingredient (a.i). It was applied at two rates – the
13 recommended rate of 20 ml.t^{-1} and the rate assessed to give highest effect in stored grain
14 protection – 160 ml.t^{-1} (Obretenchev, D., personal communication). 40 kg wheat grain were
15 spread in about 1 cm thick layer and sprayed with 40 ml of working solution. The application
16 was accomplished by means of a hand-operated spraying device used in the practice. In order
17 to achieve uniform distribution of the pesticide the working solution was divided into three
18 portions. After uniform spraying of each portion the bulk of the grain was mixed by
19 quartering, then spread again and the spraying procedure was repeated consecutively with the
20 portions left. Finally the treated wheat grain was mixed thoroughly and kept in open
21 containers in a storehouse at conditions simulating the practical grain storage. Untreated grain
22 was stored under the same conditions as control variant. The room temperature was $20 \pm 2^{\circ}\text{C}$
23 and the air moisture - $70 \pm 5\%$.

1 A sample of 0.5 kg of treated grain was taken 1 hour after treatment, when the grain became
2 dry, for determination of the initial concentrations of pesticide in the whole grain. The
3 following samplings were made after 7, 90, 180 and 270 days storage periods. 3 kg of the
4 treated grain and of untreated control were taken on particular sampling dates and submitted
5 to milling in laboratory mill, simulating the industrial processing.

6
7 Milling was carried out in a fractionating laboratory mill Buhler model MLU 202 M/s
8 (Buhler, Uzwil, Switzerland). After milling the following fractions were collected: bran,
9 semolina, 3 fractions of groats, and 3 fractions of flour. These processed products fully
10 resemble the industrial processing products. The collected fractions as well as the whole grain
11 were subjected to analysis.

12
13 *Chemicals*

14 *Pesticide standards.*

15 Analytical standard of deltamethrin, purity 99.9% Pestanal was obtained from Sigma-Aldrich
16 Laborchemicalien GmbH (Seelze, Germany). Stock solution (1.0 mg.ml^{-1}) was prepared in
17 ethyl acetate and stored in a freezer at -23°C . Working standard solutions at concentration
18 rates of 10 ng.ml^{-1} to 500 ng.ml^{-1} were prepared by diluting of stock solution with ethyl
19 acetate and stored at 4°C in the dark.

20
21 *Solvents and reagents.*

22 All organic solvents were of residue analysis grade and were obtained from Merck KGaA
23 (Darmstadt, Germany). Double distilled water was used when needed in experiments. Solid
24 phase extraction (SPE) sorbent Bond Elute SAX 250 mg-PSA 250 mg was from Varian Inc.
25 (Lake Forest, CA 92630 USA).

Instrumentation.

Hewlett-Packard gas chromatograph Model 5890 Series II equipped with ^{63}Ni electron capture detector (ECD) was used. Split-splitless injector operated in the splitless mode. Capillary column ATTM-5ms, (30 m $\times$ 0.25 mm $\times$ 0.25 μm) was obtained from Alltech Associates, Inc. (Lokeren, Belgium). Nitrogen of purity 99.9995 % was used for gas chromatography. Vacuum manifold (Varian BV) was used for SPE.

Analytical method

Extraction. The weight of the samples taken of different products varied depending on the expected levels of the residue concentrations. The sample: solvent ratios varied because of the different swelling capacity of the matrices. The whole wheat grain laboratory samples were homogenized by mixing, analytical samples of 20 g were weighed, 40 ml ethyl acetate and 10 g anhydrous sodium sulfate were added and samples were extracted by shaking for 30 min on a shaking machine Labortechnik HS 501 (IKA Werke GmbH & Co. KG, Staufen, Germany). Analytical samples of 20 g were weighed of bran and semolina, 10 g anhydrous sodium sulfate and 100 ml of the solvent were added for extraction. 40 g analytical samples of flour and groats were extracted with 80 ml of the solvent after adding of 10 g anhydrous sodium sulfate. The raw extracts were filtered through paper filter, 5 ml aliquot of each extract was taken in a graduated tube and the volume of the extract was adjusted at 4 ml by evaporation under gentle stream of air. 1 ml methanol was added to the extracts. The samples dissolved in the resulted 5 ml mixed solvent ethyl acetate-methanol (4:1) were submitted to further SPE cleanup and pre-concentration.

1 *Sample clean-up.*

2 The mixed mode SAX-PSA sorbent combines the hydrogen bonding and weak anion-
3 exchange capability of primary/secondary amines of PSA sorbent with the strong anion-
4 exchange capability of quaternary amines of SAX sorbent. This SPE product possesses
5 powerful ability to remove polar interferences such as fatty acids, other organic acids, various
6 sugars. A pre-packed cartridge SAX-PSA was washed with 2 ml methanol and conditioned
7 with 2 ml ethyl acetate-methanol (4:1). The samples were passed through the sorbent at a rate
8 of 1 ml.min⁻¹. The eluate was collected in a graduated tube. After the passage of the sample
9 the elution of the pesticides continued with ethyl acetate-methanol (4:1). A total volume of 10
10 ml of the eluate was collected. 1 µl was injected in GC system and if the obtained detector
11 response was out of the calibration range, the sample volume was adjusted by dilution or
12 evaporation under gentle stream of air in order to obtain analyte concentrations appropriate to
13 quantitative determination.

15 *Gas-chromatographic determination.*

16 GC-ECD operating conditions: carrier gas nitrogen at flow rate 4 ml.min⁻¹; make up nitrogen
17 30 ml.min⁻¹; injector temperature 270°C; detector temperature 320°C. Oven temperature
18 programme: 150°C, hold 1 min; rate 30°C/min to 290°C final temperature, hold 8 min.
19 Injection volume was 1 µl for all samples. Quantification was performed by using external
20 standards. The recovery studies were performed by using blank samples of untreated wheat
21 grain. The weights of blanks were the same as the weights of the analytical samples of treated
22 grains. The fortification was done by addition of calculated volumes of working standard
23 solutions in order to obtain 3 concentration levels of the insecticide studied: at MRL (0.01 mg
24 kg⁻¹), below MRL (0.005 mg kg⁻¹) and ten times MRL (0.1 mg kg⁻¹). Five parallel samples
25 were prepared for each fortification level. After waiting time of 1 h for evaporation of the

ethyl acetate from standard solutions the fortified samples were analyzed according to the method described above.

Results and discussion

Method validation

The method was validated for each matrix studied.

The ECD response for deltamethrin was linear in the range of 0.02 ng – 0.5 ng and the correlation coefficient r^2 was 0.9897 (five-point calibration curve). The concentrations of the analytes in the samples were calculated by using a single-level calibration method. This approach is recommended in case the detector response is variable with time (Document SANCO/10476/2003). In our studies it was confirmed that the single-level calibration gave more accurate results than multi-level calibration when the analyte concentrations in the samples were adjusted in an interval $\pm 10\%$ of the calibration level used.

The mean recoveries and the repeatability expressed as relative standard deviation (RSD), determined at three fortification levels (number of samples $n=5$) are presented in table 1. The limit of quantification (LOQ), determined as the lowest concentration of the analyte that yield signal-to-noise ratio of 10:1 was established at 0.005 mg.kg^{-1} . The LOQ was specified at this concentration level because it ensured compliance with the European Commission criteria for quantitative residue methods (Document SANCO/10476/2003).

Processing.

The laboratory fractionating mill is a conventional device used for simulated fractionation that corresponds closely to the fractionation under the real industrial process. The results of residue analysis obtained from processing experiments carried out on stored wheat grain,

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1 treated post harvest with 0.5 mg kg⁻¹ deltamethrin are presented in table 2 and of grain, treated
2 with 4 mg kg⁻¹ deltamethrin – in table 3. Deltamethrin residues were persistent under the
3 storage conditions studied. No decrease was observed of the average residue levels on the
4 whole grain and in the processed fractions after storage periods of 180 and 270 days. The
5 measured slight increase of deltamethrin residues after 180 days storage period in the higher
6 dose variant is within the estimated uncertainty of the method. This result confirms that no
7 measurable degradation of deltamethrin residues on stored wheat grain occurred during the
8 storage period. These findings correspond with the results of deltamethrin residues evaluation
9 by FAO (Food and Agriculture Organization 1983). The data reported by several countries
10 confirmed that deltamethrin was stable on stored wheat grain for more than a year. Some
11 increase of the concentrations on grain after 6 – 9 months of storage was also observed (Food
12 and Agriculture Organization 1983).

13
14 The distribution of deltamethrin residues in fractions from processed wheat is presented in
15 tables 2 and 3. The bulk of the insecticide deposited on the grain remained in the bran. The
16 concentrations of deltamethrin in the bran were from 3.3 to 4.7 times as high as those present
17 on the whole wheat grain. A considerable part of the insecticide was distributed in the
18 semolina fractions. The residues determined in semolina were 1.8 – 2.7 times as high as those
19 on the whole grain. Generally the residue levels in flour were 3 – 7 times lower than the
20 concentration on the whole grain. The reduction of residues in flour and groats is mostly due
21 to the removal of the insecticides with bran and partly with semolina fractions.

22
23 In order to determine the percentage loss of residues during processing the residual amounts
24 (mg) in the separate fractions were calculated by multiplication of the weight of each fraction
25 by the corresponding residue concentration. The balance of the residual amounts in all

fractions compared to the amount on whole grain before milling, determined in samples taken after 180 days storage (table 4), shows that deltamethrin undergoes negligible loss of residues (0.7 %) during milling. Taking into consideration that volatilization is one of the basic processes acting on pesticide residues during grinding, screening etc., the negligible losses could be explained by the very low vapor pressure of deltamethrin - $V.p.(25^{\circ}C)=1.25 \times 10^{-5}$ mPa (Tomlin 2000).

Conclusions

The analytical method used was sensitive enough for reliable determination of deltamethrin in wheat at concentration 0.01 mg kg^{-1} . The storage time and milling of wheat grain, treated post-harvest against stored-product pests with deltamethrin at recommended application rate, are not effective enough to reduce the residues in milled products to levels at or below the general MRL of 0.01 mg kg^{-1} established by EC for any individual pesticide in processed cereal-based foods and baby foods. The bulk of the insecticide deposited on the grain is removed with the bran and partly with the semolina fractions during milling but yet the flour and groats fractions, used directly for preparing of baby foods, could contain deltamethrin in quantities which do not comply with the infants and young children safety requirements. The results of this study indicate that processed products (flour, groats) of wheat, treated post-harvest with deltamethrin at the recommended rate are in compliance with the safety requirements for general population. The increase of the application rate beyond the recommended rate should be avoided because it resulted in exceeding the MRL of 1 mg.kg^{-1} for general population uses.

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Table 1.

Fortification level, mg.kg^{-1}	Recovery $\pm$ RSD, %
0.005	97.0 ± 7.1
0.01	89.6 ± 16.6
0.1	97.6 ± 15.3

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Table 2.

Matrix	Mean residue, $\text{mg.kg}^{-1} \pm \text{RSD}\%$			
	Days after treatment			
	0	30	90	180
Whole grain	0.53±9.7	0.52±9.4	0.52±5.0	0.51±7.5
Bran		2.4±5.2	2.5±5.3	2.3±5.3
Semolina		1.2±0.5	1.2±6.7	1.1±3.3
Groats 1 st fraction		0.15±5.6	0.10±11.5	0.16±10.5
Groats 2 ^d fraction		0.08±7.4	0.09±9.7	0.09±3.0
Groats 3 rd fraction		0.14±2.7	0.22±4.6	0.16±4.7
Flour 1 st fraction		0.05±11.0	0.06±10.3	0.03±3.6
Flour 2 ^d fraction		0.09±2.8	0.05±4.6	0.05±11.9
Flour 3 rd fraction		0.16±13.8	0.07±11.4	0.23±3.3

Table 3.

Matrix	Mean residue, $\text{mg.kg}^{-1} \pm \text{RSD } \%$				
	Days after treatment				
	0	7	90	180	270
Whole grain	2.4±4.1	2.1±3.8	2.2±4.6	3.2±4.7	3.5±0.6
Bran		9.9±0.7	9.0±9.4	10.5±4.7	13.4±7.9
Semolina		3.9±1.9	4.8±1.5	8.7±5.7	5.3±4.7
Groats 1 st fraction		0.22±2.6	0.42±4.7	0.41±13.2	0.19±3.7
Groats 2 ^d fraction		0.13±2.2	0.41±3.9	0.40±5.6	0.13±0.6
Groats 3 rd fraction		0.19±6.0	0.49±4.5	1.25±2.6	0.43±0.7
Flour 1 st fraction		0.69±2.9	1.9±3.0	1.9±4.4	1.55±2.1
Flour 2 ^d fraction		0.58±3.9	1.1±9.2	1.5±2.3	0.39±1.6
Flour 3 rd fraction		0.77±8.8	1.4±5.9	1.6±0.3	0.45±6.3

Table 4

Product	Deltamethrin distribution	
	Amount (mean), mg	%
Bran	1.2	75.0
Semolina	0.24	15.7
Groats 1 st fraction	0.03	1.9
Groats 2 ^d fraction	0.02	1.3
Groats 3 rd fraction	0.01	0.7
Flour 1 st fraction	0.04	2.6
Flour 2 ^d fraction	0.02	1.3
Flour 3 rd fraction	0.02	1.3
Total	1.52	99.3
Whole grain	1.53	100

Captions to tables

Table 1. Mean recoveries and standard deviations of deltamethrin in wheat (n=5)

Table 2. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 0.5 mg/kg (n=3)

Table 3. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 4 mg/kg (n=3)

Table 4 Residue balance of deltamethrin in processed products after storage intervals of 180 days (application rate 0.5 mg/kg)

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Captions to tables

Table 1. Mean recoveries and standard deviations of deltamethrin in whole grain (n = 5)

Table 2. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 0.5 mg.kg⁻¹ (n = 3)

Table 3. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 4 mg.kg⁻¹ (n = 3)

Table 4 Residue balance of deltamethrin in processed products after storage intervals of 180 days (application rate 0.5 mg.kg⁻¹)