Aldicarb in Edible Potato Crops: Agronomic Interest and Residues in Tubers during Growth and after Cooking

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The efficacy of Temik 10G (microgranule at 10% aldicarb) in potato crops against aphis (*Myzus persicae* Sulz.) and root-lesion nematodes (*Pratylenchus* sp.) was observed in relation with the residues present in the tubers. Different rates were used (from 10 to 100 kg/ha) up to 250 kg/ha to determine the stability of the toxic residues after the tubers were cooked in boiling water and in a microwave oven.

(1) INTRODUCTION

Aldicarb [2-methyl-2-(methylthio)propionaldehyde *O*-(methylcarbamoyl)oxime] is a systemic insecticide manufactured by Union Carbide under code UC21149 and the brand name Temik.

Although aldicarb residues and metabolization were studied earlier (Andrawes et al., 1971; Awasthi et al., 1979; Cairns et al., 1984; George et al., 1975; Smelt et al., 1977), no practical data were available in Belgium where Temik 10G (microgranule at 10% active ingredient) is used in sugar beet crop at a rate of 10 kg/ha of formulated product (1 kg of ai/ha) to contend with aphis, vectors of beet yellow virus. At this rate, a minor efficacy of the product is also known against nematodes. In potato crops, its utility is discussed. Pests of potatoes like *Ligus pabulinus* L., *Psylliodes affinis* Payk, or *Scutigerella immaculata* Payk, against which Temik could be useful, are rarely prejudicial in Belgium.

Only cyst nematodes (*Globodera rostochiensis* Woll. and *Globodera pallida* Stone) could justify its use but at a rate of 3 kg of ai/ha applied on the whole surface. However, in some trials, after an application of 1 kg of ai/ha in the line, an increase of the yield has been observed. A hypothesis for this phenomenon is an efficacy of aldicarb against some nematodes present from the beginning of the crop, like *Pratylenchus*. To verify this proposal and to determine the residues, a study was established. Biological observations on nematodes and aphis were done during the growing season, and yields were measured at harvest. High rates of treatment (10 and 25 kg of ai/ha) have also been applied to ensure residues in potatoes for the determination of aldicarb stability after cooking. Two modes of cooking were applied: in boiling water and dry in a microwave oven.

(2) EXPERIMENTAL PROCEDURES

(2.1) Field Treatment. Temik, granule at 10% aldicarb, was applied on May 4, 1988, in the line with a microgranulator on a plantation of potatoes, variety Bintje, one of the most important used in Belgium, at rates of 1, 3, 5, 10, and 25 kg of ai/ha in four replicated plots of 8 m × 2.8 m (density of planting, 0.7 m × 0.4 m). Four plots were not treated as control. Soil was a sandy loam containing 1.9% w/w organic matter. Rainfalls were also registered.

(2.2) Biological Observations. *(2.2.1) Evolution of Aphis Populations.* At each date of Table I, aphides (*Myzus persicae* Sulz.) were counted on 200 leaves from the two central lines of each plot.

(2.2.2) Penetration of Pratylenchus Species in the Roots. During the first weeks of growing, two observations were made. Plants were taken off each parcel. After washing, 3 or 10 g of radicles (for the first and the second taking, respectively) were extracted according to Coolen and d'Herde's (1972) method. Results are expressed in numbers of nematodes per gram of radicle (Table II).

(2.2.3) Yield Measurements. At harvesting, tubers from the two central lines were weighed after washing.

(2.3) Residue Analysis. *(2.3.1) Sampling Procedure.* Samples of potatoes from five plants were taken from every replicated

Table I. Evolution of the Populations of *M. persicae*

treatment	rate, kg/ha	av no. of aphides on 200 leaves: June 28, 1988	July 1, 1988	July 12, 1988	July 19, 1988
control		196	148	34	18
Temik 10G gypsum	10	0	0	2	2
Temik 10G gypsum	30	0	0	0	0
Temik 10G gypsum	50	0	0	0	0
Temik 10G gypsum	100	0	0	0	0
Temik 10G gypsum	250	0	0	0	0
lsd[a] (α = 0.05)		62	38	12	8
lsd (α = 0.01)		86	52	18	12
% SD		125	103	145	167

[a] Student's *t*-test.

Table II. Evolution of *Pratylenchus* Species Populations in the Root Tissues in Relation with the Treatments

treatment	rate, kg/ha	av no. of *Pratylenchus* species/g of root: May 30, 1988[a]	June 20, 1988[b]
control		6.0	8.25
Temik 10G gypsum	10	1.3	0.50
Temik 10G gypsum	30	1.0	1.00
Temik 10G gypsum	50	0.3	0.25
Temik 10G gypsum	100	0.3	0.75
Temik 10G gypsum	250	0.8	0.25
lsd[c] (α = 0.05)			3.9
lsd (α = 0.01)			5.3
% SD			140

[a] Analysis of 3 g of roots by parcel; the results are expressed in grams. [b] Analysis of 10 g of roots by parcel; the results are expressed in grams. [c] Student's *t*-test.

Table III. Treatments and Yields

treatment	rate, kg/ha	yield, kg/ha
control		56 585
Temik 10G gypsum	10	59 754
Temik 10G gypsum	30	68 906
Temik 10G gypsum	50	64 687
Temik 10G gypsum	100	64 576
Temik 10G gypsum	250	63 013
lsd[a] (α = 0.05)		6 569
lsd (α = 0.01)		9 085
% SD		6.84

[a] Student's *t*-test.

0021-8561/92/1440-0139$03.00/0

Table IV. Average and Confidence Limit Residues of Aldicarb Sulfone in Potatoes at Rates from 1 to 25 kg of ai/ha

weeks after treatment	mg/kg of aldicarb sulfone at rate of					cum rainfall on the plantation L/m²
	1 kg of ai/ha	3 kg of ai/ha	5 kg of ai/ha	10 kg of ai/ha	25 kg of ai/ha	
7	0.35 ± 0.05	1.32 ± 0.45	1.95 ± 0.75	5.25 ± 1.20	16.12 ± 8.2	105.4
9	0.18 ± 0.04	0.51 ± 0.06	1.35 ± 0.24	2.54 ± 0.81	8.50 ± 1.33	132.7
11	0.19 ± 0.02	0.45 ± 0.09	0.69 ± 0.23	1.12 ± 0.24	6.77 ± 2.09	181.6
13	0.09 ± 0.03	0.47 ± 0.13	0.47 ± 0.04	0.74 ± 0.11	2.87 ± 0.85	230.2
15	0.08 ± 0.02	0.14 ± 0.02	0.53 ± 0.07	0.53 ± 0.16	3.33 ± 0.76	237.3
20	0.03 ± 0.003	0.20 ± 0.09	0.29 ± 0.09	0.60 ± 0.11	1.94 ± 0.29	318.8

plot, first on the beginning of the formation of tubers (7 weeks after planting) and during the growth each 14 days later according to the recommended method of sampling for the determination of pesticide residues (Codex Alinorm, 1979). They were frozen at −20 °C until analysis.

After 15 weeks of growing, potatoes were made fane by a treatment with ammonium glufosinate (BASTA), and a last sampling was done at harvesting (20th week); they were not frozen and analyzed immediately.

(2.3.2) Analytical Procedure. The method used for the determination of aldicarb residues is based on the method published by the Ministry of Welfare, Health and Cultural Affairs (1985) of The Netherlands. For the present study, to enhance the recovery (announced at 75–125%) and the reproducibility, the extraction procedure has been modified.

Potatoes (frozen or not) from each plot were cut with a Hobart food cutter. A total of 50 g of the homogeneous sample was mashed and blended in a Waring blender with 150 mL of acetone–dichloromethane (50:50). This mixture was centrifuged at 4000*g* (5 min) and the supernatant collected on anhydrous sodium sulfate. The residue of centrifugation was re-extracted twice with 2 × 50 mL of the same solvent mixture. The supernatants were combined and evaporated to dryness for oxidation by potassium permanganate. The determinations were conducted in duplicate.

The residue was dissolved in 2 mL of acetone followed by the addition of 5 mL of buffer solution (13.6 g of potassium dihydrogen phosphate and 2.36 g of sodium hydroxide in 1000 mL of water) and 20 mL of permanganate solution (0.2% in water). The mixture was allowed to stand for 15 min at room temperature. The mixture was transferred to a separating funnel with 20 mL of water and extracted twice with 50 mL of dichloromethane. The dichloromethane phases were combined after filtering on anhydrous sodium sulfate and evaporated to dryness. The residue was dissolved in 2 mL of acetone for gas chromatography.

The chromatographic conditions were also modified. The glass column (1.75 m, i.d. of 3 mm, 5% OV-225 on Chromosorb W-HP 150–180 μm) was replaced by a capillary fused silica column (12 m, coated with methyl silicone) with a split–splitless injection. The temperatures were, for the injector, 175 °C and, for the detector, 275 °C. The oven program was 60 °C for 30 s, 120 °C for 10 min. (Program rate between 60 and 120 °C, 10 °C/min; post value 220 °C for 10 min.) The flame photometric detector has been replaced by a nitrogen–phosphorus ionization detector (NPD) in sulfur mode.

With this procedure, the recovery is between 85% and 95% in the range 0.05–10 mg/kg, and the limit of quantification is 0.01 mg/kg.

(2.3.3) Cooking Procedure. To be sure of the cooking action on the residue level, analyses were performed on individual potatoes. Each raw potato was peeled, and two aliquots of ±50 g exactly weighed were taken: one for the analysis on raw material and the other for analysis on the same potato after cooking.

Two modes of cooking were used. (1) Potatoes were cooked in boiling water for 20 min at 100 °C. After draining and cooling, the samples were analyzed as described in the above method. (2) Potatoes were cooked in a microwave oven (700 W), to avoid any dilution or solubilization in water, for 7 min at high power. After cooling, the samples were analyzed.

For each treatment and in the four replicates by treatment, six samples were analyzed.

(2.3.4) Expression of the Results. Results are expressed in milligrams of aldicarb sulfone per kilogram of potato (ppm). Each sample has been analyzed twice and each extract injected twice. The amount of aldicarb sulfone has been calculated by comparison with standard references of approximately the same value injected before and after two samples. Results given in the tables state the average values issued from the 24 results from the 4 replicates of the same treatment.

Confidence limits (CL) are expressed as the bounds of uncertainty about the average caused by the variability of the experiment (Bauer, 1971)

$$CL = \bar{X} \pm ts/n^{1/2}$$

where $\bar{X}$ is the average, t is Student's t value for a degree of confidence of 90%, s is the standard deviation, the square root of the variance $V = (X - \bar{X})^2/n - 1$, and n is the number of results.

(3) RESULTS AND DISCUSSION

(3.1) Biological Results. *(3.1.1) M. persicae.* As shown in Table I, a multiplication period of population of *M. persicae* was observed in the beginning of the growing, with a maximum during the last days of June, followed by a quick decrease. Temik 10G, even a low rate, has protected the crops, but its use is not justified regarding the aphis population in this trial.

(3.1.2) Pratylenchus Species. Table II gives the influence of Temik 10G on *Pratylenchus* species. The populations of *Pratylenchus* species in the root tissues, examined on May 30 and June 20, 1989, were clearly reduced by the treatments.

(3.1.3) Yield of the Crop. The application of Temik 10G at rates of 3, 5, and 10 kg of ai/ha induces a significative to highly significative increase of the yield (see Table III). Application at 1 kg of ai/ha also increases the yield, but by comparison with the untreated parcel the difference is not significative.

(3.2) Residues. *(3.2.1) During Growth.* Evolution of aldicarb residues in tubers of potatoes from the 7th week after planting to the harvest (20th week) expressed in milligrams per kilogram (ppm) is detailed in Table IV. Rainfalls are also mentioned.

At each treatment rate, a decrease of the concentration of aldicarb sulfone (sum of aldicarb, its sulfoxide and its sulfone) is clearly stated through the 7th week, owing to the growth of the tubers, but there are always residues present at harvest, even at usual rates, 1 and 3 kg of ai/ha, with respectively 0.03 ± 0.003 and 0.2 ± 0.09 mg/kg. In the control potatoes, residues were not detected. At higher rates, put in place to have incurred residues for cooking tests, higher levels of residues were found.

In addition, Table IV states the confidence limits of the results for a degree of confidence of 90%. These confidence limits take into account the variability due to the treatment, the sampling, and the analysis. These confidence limits are generally closed except at high residues level.

The influence of rainfall cannot be clearly stated. However, the relative stability of the residues between the 13th and the 15th weeks could be explained by a period of dryness, and for all the growing season the rainfall amounts are 50 mm lower than normal.

(3.2.2) After Cooking. Residues of aldicarb sulfone in

Table V. Residues of Aldicarb Sulfone in Potatoes before and after Cooking in Boiling Water and in a Microwave Oven

treatment rate, kg of ai/ha	mg/kg of aldicarb sulfone			
	in boiling water		in a microwave oven	
	before cooking	after cooking	before cooking	after cooking
3	0.23 ± 0.14	0.16 ± 0.11	0.35 ± 0.28	0.24 ± 0.14
5	0.21 ± 0.08	0.14 ± 0.08	0.45 ± 0.38	0.28 ± 0.12
10	0.48 ± 0.05	0.19 ± 0.05	0.54 ± 0.23	0.40 ± 0.13
25	1.79 ± 0.80	1.47 ± 0.59	1.34 ± 0.18	1.27 ± 0.20

peeled tubers of potatoes before and after cooking, in boiling water or in a microwave oven, are detailed in Table V.

It is clearly stated that the cooking does not destroy the aldicarb sulfone present in the tubers. There is a slight decrease of the aldicarb concentration after cooking, generally more significative after cooking in boiling water, but in any case, and particularly at the adequate biological rate (3 kg of ai/ha), there are always aldicarb residues in the tubers—0.2–0.3 mg/kg before cooking and 0.1–0.2 mg/kg after cooking. These concentrations are higher than the maximum residue limits authorized in Belgium (0.05 mg/kg) but in accordance with the Codex alimentarius LMR (0.5 mg/kg).

(4) CONCLUSIONS

Regarding the results, the use of granules of aldicarb in the line of the planting of edible potato crop, variety Bintje, cannot be encouraged in spite of the yield increase observed.

At the lower rate of 1 kg of ai/ha, the biological efficacy is limited to secondary pests, and at the rate of 3 kg of ai/ha, efficient against cyst nematodes, the residues at harvest are 0.2 mg/kg, which could be too high for a toxic compound classified "extremely hazardous" with an LD_{50} of 0.93 mg/kg by WHO (1990) in an important food.

Even after cooking, in boiling water or in a microwave oven, some of these residues are present.

ACKNOWLEDGMENT

We gratefully acknowledge the laboratory assistance of Josiane Potvin.

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Received for review May 15, 1991. Accepted September 27, 1991.

Registry No. Aldicarb, 116-06-3; aldicarb sulfone, 1646-88-4; aldicarb sulfoxide, 1646-87-3.