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Ultrasonic Extraction of Carbofuran Residues from Radishes[1]

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Ultrasonic extraction of pesticides was first reported by JOHNSON and STARR (1967). This initial report indicated that the use of an ultrasonic cleaner had promise and a second publication (JOHNSON and STARR 1970) confirmed the effectiveness of this method. Ultrasonic extraction of several organochlorine insecticides from soil was as efficient as Soxhlet extraction and superior to roller and blender methods.

JOHNSON and STARR (1972) reported that the Polytron homogenizer was also generally superior to other extraction procedures for the removal of organochlorine insecticides from soil. MOYE et al. (1977) also reported that the Polytron homogenizer was equal in extraction efficiency to Soxhlet extraction of a sandy soil fortified with certain carbamate and organophosphate pesticides.

This laboratory is investigating the extraction of "field incurred" pesticide residues. This paper reports the extraction efficiency utilizing the Polytron homogenizer for carbofuran residues in radishes using various blending times and blending speeds.

MATERIALS and METHODS

Radishes (Red Globe variety) were grown from seed in an environmental growth chamber. Mature radishes were treated by soil application at a rate of 0.2 lb/acre with Furadan 4 Flowable formulation containing ^{14}C-carbofuran (uniformly ring labelled, New England Nuclear Corporation).

Three days after carbofuran application, the radishes were harvested, the tops removed and the roots rinsed with water to remove adhering soil. Radishes were chopped, thoroughly mixed and 100 g portions were weighed into 32-oz glass jars. Radish tissues were blended using a Model PT-10-35 Polytron homogenizer with a PT 35K non-saw tooth generator in 200 ml acetone for various times (at maximum speed) and for various speeds (for 1.0 min periods). The blended slurry was

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poured into a sintered glass filter funnel and the acetone-water extract was collected by vacuum filtration.

The total amounts of radioactivity in the extract and in the tissue residue were determined. Aliquots of extracts were evaporated to dryness in small cellophane pouches, combusted in an automatic sample oxidizer (IN/US, Model 4101) and the ^{14}C determined by liquid scintillation counting. Portions of the tissue residue were also oxidized and subjected to scintillation counting. All combustion samples were done in duplicate. The combustion efficiency of the automatic oxidizer and ^{14}C carryover between samples were monitored routinely. These parameters, as well as liquid scintillation counting efficiencies, were constant during analyses.

RESULTS and DISCUSSION

The results of extraction efficiency (% Extracted) vs blending time at maximum speed are presented in Table 1. Blending time is shown in the far left column. The "a" and "b" terms represent the two combustion and counting replications for the extract and for the residue and are included to show the variability of the data. The far right column indicates the final temperature of the solvent immediately after the blending was completed. The results indicate that, in general, all blending times of 0.50 min and longer at maximum speed are of equal efficiency in extracting carbofuran treated radishes. The 0.25 min time may be slightly less effective removing only 52% of the ^{14}C from the radishes. These blending times resulted in extraction efficiencies ranging from 54% to 63%.

One advantage of shorter blending times is the factor of solvent temperature. During the first 0.50-1.0 min, the temperature rose 8°C. During the second and third minutes of blending, temperature rises were 6° and 8°, respectively. Only after 10 min of blending were further temperature increases noted. The extraction efficiency at 0.25 min was relatively good and no temperature increase was detected; this indicates that extraction efficiency is not dependent upon elevated solvent temperatures. It would, therefore, be advantageous to keep the blending time short in order to minimize potential problems related to elevated solvent temperatures.

The results of extraction efficiency (% Extracted) vs blending speed (1.0 min blending time) are presented in Table 2. The speed designations are the rheostat designations on the Model PT-10-35 basic assembly. The blender did not run at rheostat settings of "1" or "2" and, therefore, data are not included.

TABLE 1

POLYTRON TIME STUDY: Carbofuran Treated Radishes at 3 Days After Application

Blending Time (min)[1]	Extract (cpm)			Tissue Residue (cpm)			Total Activity (avg)	% Extracted (avg)	Temp (°C)
	a[2]	b	avg	a	b	avg			
0.	--	--	--	--	--	--	--	--	26
0.25	135180	130815	132997	123316	120722	122019	255016	52.2	26
0.50	151860	152955	152407	83361	93408	88384	240791	63.3	34
0.75	165795	150045	157920	190135	109057	109096	267016	59.1	34
1.0	153315	146625	149970	111287	105354	108320	258290	58.1	34
1.5	140145	130545	135345	105847	102770	104308	239653	56.5	36
2.0	149895	152085	150990	92326	96153	94239	245229	61.6	40
2.5	149055	155205	152130	105404	108793	107098	259228	58.7	48
3.0	142875	132915	137895	111326	111324	111325	249220	55.3	48
4.0	138765	136605	137685	102839	103696	103268	240953	57.1	48
5.0	133755	135365	135060	111475	114997	113236	248296	54.4	47
7.0	142755	138735	140745	105396	110309	107853	248598	56.6	48
10.0	148275	142905	145590	94486	95564	95025	240615	60.5	50

[1]Samples were blended at maximum speed using a Polytron homogenizer for the times indicated.

[2]"a" and "b" are combustion and counting replications.

TABLE 2

POLYTRON SPEED STUDY: Carbofuran Treated Radishes at 3 Days After Application

Blending Speed[1]	Extract (cpm) a[2]	Extract (cpm) b	Extract (cpm) avg	Tissue Residue a	Tissue Residue b	Tissue Residue avg	Total Activity (avg)	% Extracted (avg)
3	27702	28840	28271	22537	24586	23562	51833	54.5
4A[4]	28017	28087	28052	23335	23265	23300	51352	54.6
4B	30975	29767	30371	22189	22779	22484	52855	57.5
5A	30975	31202	31089	24497	24670	24584	55673	55.8
5B	32112	--	32113	22439	22037	22238	54350[3]	59.1
6A	32497	31325	31911	18833	18005	18419	50330	63.4
6B	33915	32830	33372	24201	24732	24466	57838	57.7
7	33600	34545	34072	21224	20204	20714	54786	62.2
8A	35752	35367	35560	22389	20958	21673	57233	62.1
8B	34755	32672	33714	21396	21994	21695	55409	60.8
9	33320	34037	33679	22884	22786	22835	56514	59.6
10	33302	35385	34344	22218	23652	22935	57279	60.0

[1]Samples were blended on a Polytron homogenizer for 1.0 min using dial settings 3 to 10 as indicated.

[2]"a" and "b" are combustion and counting replications.

[3]The average value for the extract is taken from counting replicate "a" only.

[4]"A" and "B" are duplicate samples.

Duplicate samples were run at settings of 4, 5, 6 and 8 and are presented to show variation between samples. The "percentage extracted" data indicate that the Polytron is somewhat less efficient and consistent at the lower speeds (3, 4 and 5); the efficiency seems to increase and stabilize in the speed range of 7 to 10.

The data in Table 1 and in Table 2 were derived from different applications of ^{14}C-carbofuran in formulated material. It should be pointed out the radish tissues appeared to be well disrupted under all the experimental conditions utilized. Furthermore, it is known from other work done at this laboratory (WHEELER et al. 1977) that 55-60% extraction efficiency represents maximum removal of ^{14}C from radishes treated with ^{14}C-carbofuran in commercial formulation; exhaustive Soxhlet extraction of radish tissues yielded no significant additional ^{14}C.

Thus, the Polytron homogenizer is an efficient blending device for extracting carbofuran from radishes. It is likely that this effectiveness would extend to other pesticides and substrates. This homogenizer was efficient for relatively short blend times, but exhibited greater effectiveness at higher rather than lower blend speeds.

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REFERENCES

JOHNSON, R. E. and R. I. STARR: J. Econ. Entomol. 60, 1679 (1967).

JOHNSON, R. E. and R. I. STARR: J. Econ. Entomol. 63, 165 (1970).

JOHNSON, R. E. and R. I. STARR: J. Agr. Food Chem. 20, 48 (1972).

MOYE, H. A., S. WITKONTON and G. CASH: Extension of multi-residue methodology: I. Determining multiclass pesticide residues in soil by gas chromatography. II. Dynamic fluorogenic labelling detector for carbamates. EPA-600/1-77-029, Environmental Health Effects Research Series, 168 pp.

WHEELER, W. B., N. P. THOMPSON, P. ANDRADE and R. T. KRAUSE. Final report of Food and Drug Administration Contract No. 223-74-2223 (1977).