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Comparison of Blenders for the Extraction of Carbofuran from Radishes[1]

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Extraction, although an essential link in the chain of pesticide analysis, is rarely evaluated. Most of the methods utilized to determine pesticide recoveries through an analytical scheme cannot in fact assess extraction efficiency. Work has been performed at this laboratory to more quantitatively determine extraction efficiencies and to identify significant variables in the extraction process (WHEELER et al. 1978, 1979). Typically, the extraction process involves the mixing of an extracting solvent with the substrate to be extracted utilizing one of several commonly used blenders. Analytical methodology protocols of various agencies rarely define the type of blending apparatus that should be used or the operational parameters for that apparatus. It has been assumed that the various blenders will produce adequate, or at least similar extractions. This paper will compare the effectiveness of three commonly used blenders to extract ^{14}C-labeled carbofuran from radishes.

MATERIALS AND METHODS

Radishes (Red Globe variety) were grown from seed in a greenhouse. Mature radishes were treated by soil application at a rate of 2.0 lb/acre with Furadan 4 Flowable formulation mixed with ^{14}C-carbofuran (uniformly ring labeled).

Three, 7 and 14 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 jars. Blenders and jars used were: 1) a Polytron Ultrasonic Homogenizer, Model PT-10-35 with a PT 35K non-sawtooth generator using 32-oz glass orange juice jars (Tropicana Products, Inc.); 2) a Lourdes blender Model VM with type MJA blades using 32-oz mason jars; and 3) a Waring Blendor and standard 1-qt Waring Blendor jars.

Samples were blended with 200 mL of methanol under the conditions described in Table 1. Blending was started at a low speed to avoid spillage and then increased. The blended slurry was then poured into a sintered glass filter funnel and the methanol-water

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extract was collected by vacuum filtration. The blender blades and jar were rinsed with 100 mL of methanol and this solvent was poured through the crop marc under vacuum and combined with the first filtrate. Extractions were replicated three times at each harvest interval.

TABLE 1

Blender Operating Parameters

	Initial		Final	
Blender	Speed	Time (min)	Speed	Time (min)
Polytron	5*	0.5	10*	1.0
Lourdes	3000 rpm	0.5	7000 rpm	2.0
Waring	Low	0.5	High	2.0

*Rheostat dial setting

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 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. Counts per minute determined by liquid scintillation counting were converted to disintegrations per minute using appropriate quench curves.

To evaluate the identity of extracted ^{14}C, thin layer chromatography (TLC) was performed on Polytron homogenizer extracts. Extracts were concentrated using a rotary evaporator, the aqueous portion remaining was partitioned with dichloromethane and the organic soluble portion was streaked on silica gel plates. The plates were developed using ether:benzene (3:1) and ether:hexane (3:1), the radioactive areas were detected using x-ray film autoradiography and the ^{14}C present in each zone was measured by scraping and liquid scintillation counting. Compounds were tentatively identified by comparing R_f values of radioactive radish extracts to R_f values of authentic compounds in the same TLC system and to literature values.

RESULTS AND DISCUSSION

The mean extraction efficiencies of the Polytron, Lourdes and Waring blenders are presented in Figure 1. At 3, 7 and 14 days the Polytron homogenizer extracted 78, 61 and 54%, respectively, of the ^{14}C present at harvest, the Lourdes blender extracted 80, 60 and 51% respectively and the Waring Blendor extracted 78, 54 and 48% respectively. Analysis of variance and

the Duncan's New Multiple Range Test indicated that the mean extraction percentages for the three blenders at 3 days were all equivalent (0.05 confidence level). At 7 and 14 days, the Polytron and Lourdes were not significantly different while the Polytron was significantly different than the Waring Blendor (0.05 confidence level). Of the ^{14}C extracted by the Polytron, 85% was parent compound at 3 days, 66% at 7 days and 49% at 14 days. One other major metabolite, tentatively identified as 3-hydroxycarbofuran by TLC constituted 7% of the extractable ^{14}C at 3 days, 15% at 7 days and 20% at 14 days.

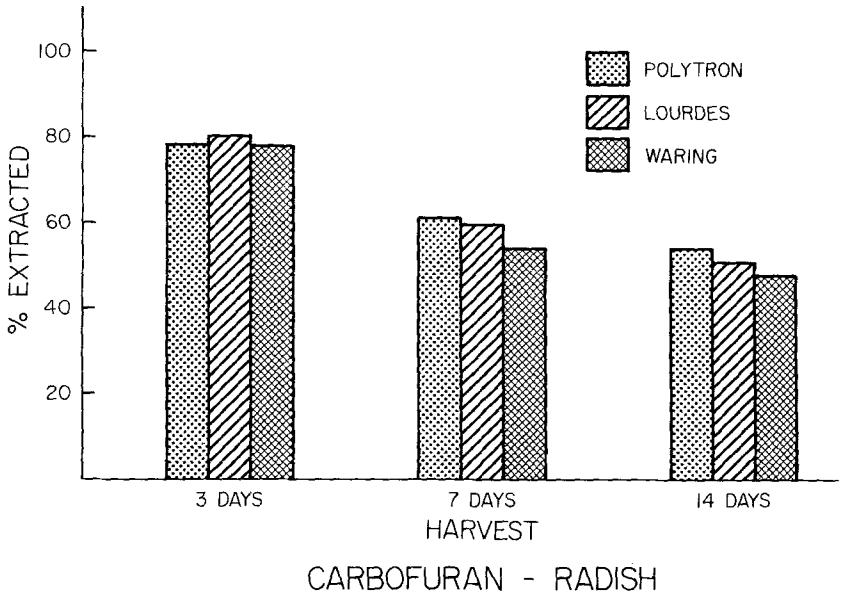

FIGURE 1. Percentage ^{14}C-extracted by Polytron, Lourdes and Waring blenders at intervals of 3, 7 and 14 days post-application of carbofuran to radishes.

Although statistical analyses indicate the superior ability of the Polytron and Lourdes blenders to extract carbofuran from radishes under these conditions, from a practical viewpoint the differences are not large; the least efficient blender extracted 90% of that extracted by the most efficient blender. It might also be possible to increase the efficiency of the Waring Blendor by changing its use parameters (e.g. lengthening the blending time). The blending parameters used for the Polytron and Lourdes have been shown to be in the optimum range for these devices (WHEELER et al. 1977, 1979). To confirm this, Soxhlet extraction

(chloroform:methanol/9:1) of the tissue marc remaining after blending with the Polytron homogenizer removed no substantial ^{14}C (mean value less than 0.7% of the total present).

If one assumes that similar results would be realized with other pesticide-crop combinations, (unpublished work performed in this laboratory shows this assumption valid for carbaryl and methomyl applied to radishes and extracted using the Polytron and Lourdes blenders) then the residue analyst can be confident that no major analytical errors would be introduced at the blending step by the use of any of these three devices.

To the best of our knowledge, these data represent the first direct comparison of the efficiency of three commonly used blenders for the extraction of "weathered" residues of a pesticide which had been applied in a commercial formulation.

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