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Effectiveness of the standard evaluation method for hydraulic nozzles employed in stored grain protection trials

Eficiencia de la evaluación estándar de boquillas hidráulicas utilizadas en experimentos de protección de granos almacenados

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Abstract: In stored grains, smaller depositions and great variation with respect to theoretical insecticide doses are frequently found. The objective of this work was to study the effectiveness of the standard method (ISO 5682/1-1996) employed to evaluate hydraulic nozzles used in stored corn and wheat grain protection experiments. The transversal volumetric distribution and droplet spectrum of a model TJ-60 8002EVS nozzle were determined in order to calibrate a spraying system for an application rate of 5 L/t and to obtain theoretical concentrations of 10 and 0.5 mg/kg of fenitrothion and esfenvalerate, respectively. After treatment, the corn and wheat grains were processed and deposition was analyzed by gas chromatography. The type of grain did not have any influence on insecticide deposition and was dependent upon insecticide only. The insecticide deposits on the grains only reached 42.1 and 38.2% of the intended theoretical values for fenitrothion and esfenvalerate concentrations, respectively. These results demonstrate the ineffectiveness of the standard evaluation method for hydraulic nozzles employed in stored grain protection experiments.

Key words: Application technology. Volumetric distribution. Insecticide deposition. Chromatography.

Resumen: En los granos almacenados, depósitos inferiores y gran variación en relación a las dosis teóricas de insecticidas aplicadas son frecuentemente encontrados. El objetivo del presente trabajo fue estudiar la eficiencia del método estándar (ISO 5682/1-1996) utilizado para la evaluación de boquillas de pulverización usadas en experimentos de protección de granos de maíz y trigo. La distribución volumétrica transversal y el espectro de gotas del modelo TJ-60 8002EVS se determinaron con la finalidad de calibrar un sistema de pulverización para aplicar un volumen de aplicación de 5 L/t y obtener una concentración teórica de 10 y 0,5 mg/kg de fenitrotion y esfenvalerato, respectivamente. Después del tratamiento, los granos de maíz y trigo fueron procesados y los depósitos de los insecticidas analizados mediante técnica de cromatografía gaseosa. El tipo de grano no tuvo influencia en el depósito de los insecticidas y dependió apenas del insecticida. Los depósitos en los granos solamente alcanzaron valores de 42,1 y 38,2% de la concentración teórica pretendida de fenitrotion y esfenvalerato, respectivamente. Estos resultados demuestran la ineficiencia del método estándar para la evaluación de boquillas hidráulicas empleadas en experimentos de protección de granos almacenados.

Palabras clave: Tecnología de aplicación. Distribución volumétrica. Depósito de insecticida. Cromatografía.

Introduction

In a storage facility, grains are usually treated on a conveyor belt, where hydraulic nozzles are mounted for this purpose. Under these conditions, smaller depositions and great variations with regard to theoretical insecticide doses are frequently found (Vardell *et al.* 1973; Rowlands 1975; Desmarchelier *et al.* 1987; Redlinger *et al.* 1988; White and Sinha 1990; Acda *et al.* 1994). In order to improve the quality of sprays generated by hydraulic nozzles, a number of systems have been developed under laboratory conditions, but with little success when it comes to solving the above-mentioned problems. Great variation in insecticide deposition on the mass of grains may favor the occurrence of two important biological phenomena. The first is associated with subtoxic amounts of the insecticide, which may stimulate population growth of the pest (hormoligosis) (Kuenen 1958; Luckey 1968; Morse 1998), while the second is associated with high amounts of the chemical compound (resistance), which may favor the survival of highly resistant individuals whose biological performance in the absence of the insecticide may be just identical to the susceptible strain, resulting in practical complications in the management of this phenomenon (Oliveira *et al.* 2005). In Brazil, failure in the chemical control of *Sitophilus oryzae* (L., 1763), *Sitophilus zeamais* Motsch., 1855 (Coleoptera: Curculionidae), *Rhyzopertha dominica* (Fabr., 1792) (Coleoptera: Bostrichidae), *Tribolium castaneum* (Herbst, 1797) (Coleoptera: Tenebrionidae), *Cryptolestes ferrugineus* (Stephens, 1831) (Coleoptera: Cucujidae), and *Oryzaephilus surinamensis* (L., 1758) (Coleoptera: Silvanidae) has been reported (Lorini and Beckel 2002). It is possible that an inadequate technology of application may have favored a loss of effectiveness of insecticides used for stored grain protection.

In a spraying system, the nozzle is the most important component, since it is responsible for the flow, generation, and distribution of droplets that will carry the insecticide to the target to be controlled. Knowing the transversal volumetric distribution of the nozzle is highly important in a spray analysis

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and has been the object of study of several researchers (Roth *et al.* 1985; Debouche *et al.* 2000; Cunha and Teixeira 2001; Womac and Bui 2002). In the evaluation of volumetric distribution of spray nozzles, the International Organization for Standardization has defined clean water as the standard spraying liquid (ISO 5682/1-1996). On the other hand; the physical characteristics of the sprayed liquid can affect the volumetric distribution pattern of the nozzle (Krueger and Reichard 1985), thus compromising field experiments. The objective of this work was to study the effectiveness of the evaluation method employed for hydraulic nozzles used in experiments on the protection of stored grains, as described in the ISO 5682/1-1996 (E) standard.

Material and Methods

Application technology. A twin jet model TJ-60 8002EVS hydraulic nozzle (Spraying Systems Co.) was used for the study. A channeled table (patternator) was used to carry out the spray nozzle transversal volumetric distribution analysis experiments, standardized according to standard ISO 5682/1-1996 (E). The testing table (3.5 m long, 3.0 m wide) has channels spaced at 0.025 m, positioned at a 5% slope. In front of the table, a set of graduated cylinders (250 mL) collects the liquid from each channel. Clean water was used to evaluate the nozzle. The following parameters were evaluated: actual flow and transversal volumetric distribution, at a pressure of 200 kPa and a nozzle height of 0.5 m. The weighting method was used to obtain actual flow, and the volume collected during one minute in a plastic container was weighted in a precision balance. In order to determine transversal volumetric distribution and effective swath width, the nozzle was mounted on the boom and positioned at a 90º angle in relation to the assay table. The collection time was set until one of the graduated cylinders reached a volume of 230 mL. This collection time was used for the three replications.

After the effective swath width was determined, we studied the droplets spectrum. A mobile application system was built containing the nozzle, a manometer, a CO_2 tank, and a tank for the liquid to be applied. Three water-sensitive papers (0.076 m long, 0.026 m wide) were distributed on the extreme and central portions of the previously-defined effective swath width. The same height and working pressure adopted for the assay table were used, at a moving speed of 5 km h^{-1}. After spraying, the water-sensitive papers were collected and analyzed using a computerized image analysis system (Chaim *et al.* 2002), Gotas, version 1.0 (Embrapa Meio Ambiente, São Paulo, Brazil).

Corn and wheat cultivars Sol-da-Manhã and BRS 208 were used, respectively, both developed by Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA - Brazilian Agricultural Research Corporation). In order to determine the mass of grains per unit area, the corn and wheat were spread as a fine layer onto a plastic tarp, covering a 1 m^2 area, and were then weighed. Mean values of 5.0 and 4.0 kg m^{-2} were thus obtained for corn and wheat, respectively. A plastic tarp was placed between the rails and the grains were spread out on the tarp. The swath width where the grains were spread was established based on the transversal volumetric distribution of the nozzle, study performed previously. In order to verify the intended application rate, three glass slides (0.1 m length, 0.05 m width) were placed on the grains for later deposition quantification by means of gas chromatography. Fenitrothion and esfenvalerate were applied so as to produce theoretical concentrations of 10 and 0.5 mg/kg, respectively, on both types of grain. The commercial product Sumigranplus® (500 g of the a.i. fenitrothion + 25 g of the a.i. esfenvalerate/liter) was used.

During application, the mobile system was moved along the material to be treated; the operational specifications of the nozzle were the same as these in the laboratory tests. The moving speed of the cart was calculated for an application volume of 5 L/t. This spray volume facilitates the treatment and it does not increase the moisture of stored corn and wheat grains in laboratory conditions (Vásquez-Castro *et al.* 2006). Under these conditions, the insecticidal emulsion contained 0.4% of the commercial product. Three replications were made, generating six experimental plots, and two insecticides were analyzed, totaling twelve subplots. The same procedure was adopted for the control treatment, but in this case the spray consisted of water only. The temperature and relative humidity during spray were 26ºC and 71%, respectively. The physical properties of the mix were determined after spraying. Surface tension was determined by the burette method, according to the NBR 13241 standard for surface tension determination in agrochemicals (Associação Brasileira de Normas Técnicas 1994). Viscosity was determined with a Brookfield, model LVDV-III Ultra viscometer at 26ºC.

Deposition Analysis

Grain. Half an hour after the spray, grains was collected and processed with dry ice. To achieve this, a model TRF70 forage chopper was used. The dry ice was mixed with the grain at a 1:1 ratio prior to grinding, in order to maintain a temperature value that would minimize insecticide degradation during the operation. The analytical method was adapted from Ohlin Ohlin (1998). Homogenized samples (10 g) were placed in Schott bottles (100 mL) for residue extraction. Ethyl acetate (50 mL) and sodium sulfate (10 g) were added and later homogenized in a stirring table for 1 hour at 360 cycles min^{-1}. After this operation, the extracts were centrifuged for 5 min at 2,600 rpm for better separation of the liquid phase from suspension materials. Aliquots of the supernatant (10 mL) were transferred to 12-mL test tubes, corresponding to 2 g of the original sample, and were then added of dodecane (50 µL). The extracts were evaporated in a Turbo-Vap evaporator, in a water bath (30°C) aided by moving air previously dried through a blue silica gel desiccant filter. The insecticide residues were then resuspended in a cyclohexane+ethyl acetate mixture (1+1 by volume) (5 mL), homogenized in a vortex mixer/ultrasound and filtered through a Millipore, FG, 0.2 µm pore membrane filter mounted on a plastic hypodermic syringe (5 mL).

The extracts were cleaned by gel permeation chromatography (GPC) and eluted with a cyclohexane+ethyl acetate mixture (1+1 by volume). After this operation, the extracts were evaporated in a Turbo-Vap evaporator previously added of dodecane (50 µL) and were later resuspended in the cyclohexane+ethyl acetate mixture (1+1 by volume) (20.0 and 1.95 mL) for the fenitrothion and esfenvalerate residues, respectively.

The samples were analyzed by gas chromatography, with a Thermo Electron Corporation, model Finnigan Trace gas chromatograph, equipped with an electron capture detector (ECD, Ni^{63}) and a Restek Corp. RTX-5MS chromatography capillary column (30 m-long, 0.25 mm diameter, and 0.25 µm film thickness), with injections made in the splitless mode. The

chromatograph was operated under the following conditions: column temperature = 100°C (initial); then at 280°C (25°C/min ramp), remaining at this temperature for 10 min; injector temperature = 230°C; detector temperature = 320°C; purge time = 1 min; gas flow (mL/min): H_2 (skidding) = 1.2; N_2 (make up) = 45; and purge flow = 65. Under these conditions, retention time was 6 min and 20 sec for fenitrothion and 10 min and 25 sec for esfenvalerate, approximately. Residue amounts were calculated using the ChromQuest version 4.0 software, by comparing the chromatographic peak heights for the samples against the chromatographic peak heights for the corresponding analytical standards. The analytical method used for corn and wheat grains was validated by means of matrix fortification at the levels of 0.05; 0.5; and 10.0 mg/kg for fenitrothion, and 0.05; 0.1; and 1.0 mg/kg for esfenvalerate, with three replications for each level (nine fortified samples for each matrix). Recoveries between 70-120% were considered acceptable.

Glass slide. Three glass slides were placed into flasks (600 mL). Ethyl acetate (500 mL) was added and the insecticides were later extracted by ultrasound for 15 min. Aliquots (2 mL) were transferred to test tubes (12 mL) and were then added of dodecane (50 µL). The extracts were evaporated in a Turbo-Vap evaporator, in a water bath (30°C) aided by moving air previously dried through a blue silica gel desiccant filter. Later, the insecticide residues were resuspended with the cyclohexane+ethyl acetate mixture (1+1 by volume) (2 mL) and homogenized in a vortex mixer/ultrasound, and then diluted at an extract (1 mL) + cyclohexane+ethyl acetate mixture rate of 1+1 by volume (9 mL). The insecticide depositions on the glass slides were analyzed by gas chromatography, in the same manner as for depositions on the grains.

Statistical analysis. The data were submitted to analysis of variance, using a mathematical model for a completely randomized design in a split-plot arrangement, and the F test was used to evaluate the significance of factors grain type, insecticide, and interactions in the models (Steel and Torrie 1960, Pimentel-Gomes 1987).

Results and Discussion

Application technology. The actual flow of the nozzle with water was 0.66 L/min, 1.5% higher than the nominal flow of 0.65 L/min, according to the information provided by the manufacturer. The variation between actual and nominal flow was within the acceptable limit, since according to the World Health Organization (WHO 1976), the acceptable flow variation limit of a spraying nozzle is ± 4% in relation to the nominal flow indicated by the manufacturer. At our working conditions, a total swath width of 0.875 m was obtained, with a coefficient of variation (c.v.) of 41% (Fig. 1). The c.v. was higher than the 7% limit established by the international standard (European Committee for Standardization 1997). Although the flow was in accordance with the international standard, the transversal volumetric distribution showed great variation (Fig. 2), probably due to the presence of irregularities on the tip orifice of the spray. The problem presented above will cause irregular insecticide deposition and consequently the grains will receive under - or overdoses depending on their placement within the total deposition swath, thus compromising insecticide effectiveness and residue studies.

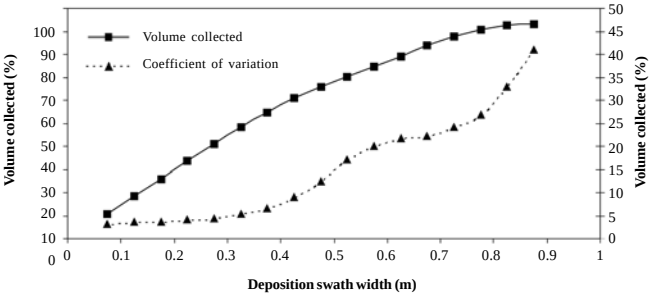

Figure 1. Transversal volumetric distribution of a TJ-60 8002EVS nozzle using clean water during spray.

In order to obtain an insecticidal mix distribution as uniform as possible, and considering that in Brazil a c.v. of up to 10% is acceptable, we calculated effective swath width and c.v. values of 0.425 m and 9%, respectively. Under these conditions, 65% of the sprayed water volume was collected within the effective swath width (Fig. 1). Therefore, the spraying equipment was calibrated to apply a total volume of 7.6 L/t, since 35% of this volume would remain outside the treatment area. Consequently, the grains would receive an effective application volume of 5 L/t as intended. The nozzle flow in the spraying equipment at a pressure of 200 kPa was 0.66 L min^{-1}; this value was very similar to the flow obtained with water in the laboratory test. In this situation, the cart moving speeds were 2.4 and 3.1 km/h for the corn and wheat sprays, respectively. The droplets spectrum for the nozzle under evaluation, working at pressure, height, and moving speed values of 200 kPa, 0.5 m, and 5 km/h, respectively, is presented in Table 1.

Because of the difficulty in determining the droplets spectrum, researchers and nozzle manufacturers frequently refer to the mean volumetric diameter to characterize sprays (Bouse 1994). According to the brochure of the manufacturer, the TJ-60 8002EVS nozzle produces fine droplets at all recommended working pressures; however, in the present study we obtained droplets of sizes between medium and large. The droplet size categories used in this experiment were the same as in the international ASAE (X-572) and BCPC standards. The differences in droplet diameter and consequently in droplet size category were possibly caused by the measurement technique used, since the international standards specify a laser system to evaluate the droplets spectrum. In this work, we

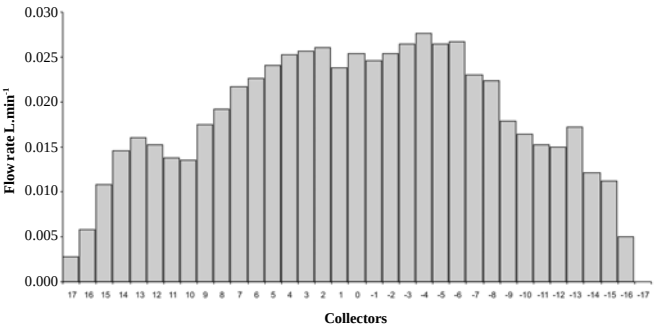

Figure 2. Transversal volumetric distribution pattern of a TJ-60 8002EVS nozzle using clean water during spray.

Table 1. Droplet analysis of TJ-60 8002EVS nozzle using clean water during spray.

Parameters	Position of water-sensitive paper on effective swath width		
	Left	Center	Right
Volume (L ha^{-1})	154 ± 17.9	87 ± 13.9	128 ± 18.6
Density (n^0 cm^{-2})	126 ± 12.5	122 ± 5.6	123 ± 12.7
Uniformity	1.8 ± 0.2	1.8 ± 0.0	1.8 ± 0.1
VMD (μm)	378 ± 14.6	320 ± 14.8	363 ± 15.5
NMD (μm)	214 ± 10.6	179 ± 8.5	201± 3.0
Coverage (%)	30 ± 2.8	19 ± 2.6	25 ± 3.3

VMD: Volumetric mean diameter. NMD: Numeric mean diameter.

used water-sensitive paper to obtain droplet stains to make diameter measurements at a later time using specific software. Several methods can be used to measure droplets spectrum. In this respect, computing methods have been used quite often (Wolf *et al*. 2000; Wolf 2003, 2005). On the other hand, all computing methods used for droplet measurement take into consideration a spreading factor; in the case of the software used in our study, it was validated (Chaim *et al*. 2002). At the center of the effective swath width, the droplets were smaller when compared with the droplets at the extreme points of the swath. During the droplet formation process, the hydraulic energy of the liquid is transformed into droplet kinetic energy (Amberg and Buttler 1969). One explanation for the results is that larger droplets have greater mass and therefore acquire higher kinetic energy. Consequently, large droplets have a greater capacity to overcome air resistance to horizontal movement, and may travel longer distances when compared with smaller droplets. In the same way, the volume and coverage values at the center of the effective swath width were lower than at the edge. This was probably due to the vortex effect generated by the cart moving at a speed of 5 km h^{-1}; very small droplets would then be dispersed outside the treatment area by the wind turbulence.

Deposition analysis. The insecticide recovery percentages in the fortified corn and wheat grains were acceptable (70-120%), thus validating the analytical method. None of the two insecticides were recovered from the control indicating that the grains were free from contamination by those compounds. The analysis of variance only detected a significant effect ($P \leq 0.05$) for insecticide (Table 2). This result demonstrates than neither grain type nor the interaction between grain type and insecticide has an influence on pesticide deposition. Consequently, deposition only depended on insecticide. The fenitrothion deposits were significantly higher than expected than those for esfenvalerate, both on grains and on glass slides (Fig. 3). In spite of the fact that the physico-chemical properties of these insecticides would determine greater esfenvalerate stability, more fenitrothion was recovered. The greater recovery of fenitrothion was due to the higher sensitivity of the chromatograph detector to this molecule. On the other hand, the depositions of both insecticides were always higher on the glass slides when compared with depositions on the grains. Probably, some spray droplets reached the plastic tarp through the empty spaces between the grains, resulting in lower depositions than those intended. Nevertheless, the analytical procedure for grains is much more complex than for the glass slides, and some degree of insecticide loss occurred in the agronomic matrix.

Deposition values on the glass slides of only 59 and 55% of the intended fenitrothion and esfenvalerate dosages were obtained, respectively. Despite our detailed study on the application method, insecticide depositions were lower than planned, and approximately 40% of the insecticidal spray did not reach the area that should have been treated. Surface tension and viscosity in the insecticidal mix reached values of 35.47 mN/m and 1.82 mPa.s, respectively. The mix surface tension value corresponded to 49% of the water surface tension value (71.97 mN/m). Conversely, mix viscosity was 82% higher than water viscosity (1.0 mPa.s). Clean water was used during spray in the hydraulic nozzle evaluation, as prescribed by the international standard, and the results obtained in this test were useful to calibrate the application system.

Table 2. F test probability descriptive levels for the analysis of variance of insecticide deposition on corn and wheat grains and on corresponding glass slides.

Cause of variation	Degrees of freedom	Insecticide deposition	
		Grain	Glass slides
		Pr > F	
Grain type	1	0.9730	0.3283
Insecticide	1	0.0025	0.0041
Grain type × Insecticide	1	0.5979	0.2580
Mean (%)	-	40.17	57.14
Coefficient of Variation (%)	-	2.51	2.26

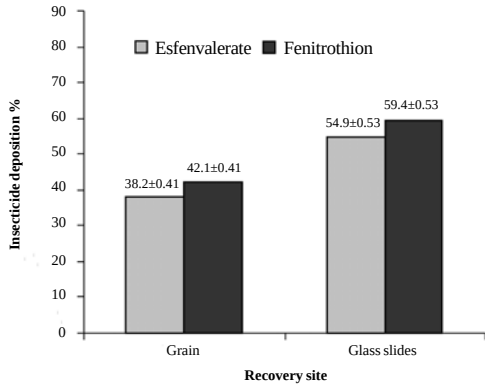

Figure 3. Mean ± SEM insecticide deposition on glass slides and grain, by active ingredient.

The extrapolation of the data generated with the application of water for the application of insecticidal mix was the major reason for obtaining deposition values lower than expected, because the physical characteristics of the mix might have changed the volumetric distribution pattern of the nozzle. In this regard, differences in the volumetric distribution pattern of flat-fan nozzles have been observed when different types of mixes were used, including water, particularly at low pressure values (Butler Ellis and Tuck 1999). The influence of the spraying mix physical properties on the volumetric distribution pattern and droplet spectrum generated by agricultural nozzles is not yet completely understood, especially in stored grain protection studies, in which low spraying volumes are used and mixes are highly concentrated. The results herein reported demonstrate that using the standard evaluation method for hydraulic nozzles (ISO 5682/1-1996) employed in insecticide effectives and residue experiments on stored grains is not viable. Therefore, it is recommended that the insecticidal mix be used to evaluate the volumetric distribution pattern and droplets spectrum of hydraulic nozzles, with later calibration of the spraying system using this information.

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