A SYSTEM TO EVALUATE THE PERFORMANCE OF HYDRAULIC NOZZLES USED IN STORED GRAIN PROTECTION TRIALS

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ABSTRACT: We developed a system to evaluate the performance of hydraulic nozzles used in stored corn and wheat grain protection experiments. An insecticidal mix was used as test fluid to determine the transversal volumetric distribution and droplets spectrum of a model TJ-60 8002EVS hydraulic nozzle. A mobile application system was built to apply a rate equivalent to 5 L t^{-1} and obtain theoretical concentrations of 10 and 0.5 mg kg^{-1} of fenitrothion and esfenvalerate, respectively. The corn and wheat grains were spread out as a fine layer. Three glass slides (0.1 × 0.05 m) were placed on the top surface of the grains to ensure that the intended application rate was achieved. After treatment, the deposits on both matrices were analyzed by gas chromatography. The fenitrothion deposit was higher than esfenvalerate, and the deposit on wheat was higher than on corn grains ($P < 0.05$). The deposits on the glass slides reached values of 100 and 93% of the intended theoretical fenitrothion and esfenvalerate concentrations, respectively. Deposits on the grains were lower than on the glass slides, with values of 64 and 52% of the intended theoretical fenitrothion and esfenvalerate concentrations, respectively. The results obtained demonstrate a high effectiveness of the method for evaluation of the performance of hydraulic nozzles when an insecticidal mix is used as test fluid. The factors that influenced insecticide deposition on glass slides and on grains are discussed in the present work.
Key words: application technology, gas chromatography, insecticide deposits, corn, wheat

SISTEMA PARA AVALIAR O DESEMPENHO DE BICOS HIDRÁULICOS UTILIZADOS EM EXPERIMENTOS DE PROTEÇÃO DE GRÃOS ARMAZENADOS

RESUMO: A tecnologia de aplicação na proteção de grãos armazenados é de fundamental importância para melhorar a uniformidade de distribuição dos agrotóxicos na massa de grãos. Foi desenvolvido um sistema para avaliar o desempenho de bico hidráulico utilizado em experimentos de proteção de grãos de milho e trigo armazenados. Para determinar a distribuição volumétrica transversal e o espectro de gotas do bico hidráulico modelo TJ-60 8002EVS, utilizou-se calda inseticida como líquido teste. Um sistema móvel de aplicação foi construído para aplicar uma taxa equivalente a 5 L t^{-1} e obter uma concentração teórica de 10 e 0,5 mg kg^{-1} de fenitrotion e esfenvalerato, respectivamente. Os grãos foram espalhados em fina camada para o tratamento. Lâminas de vidro foram colocadas sobre a massa de grãos para certificar a aplicação da dose pretendida. Os depósitos em ambas matrizes foram analisados por técnica de cromatografia gasosa. O depósito de fenitrotion foi superior ao do esfenvalerato e o depósito nos grãos de trigo foi superior a do milho. Nas lâminas de vidro os depósitos atingiram valores de 100 e 93% da concentração teórica pretendida de fenitrotion e esfenvalerato, respectivamente. Nos grãos os depósitos foram inferiores aos das lâminas de vidro, com valores de 64 e 52% da concentração teórica pretendida de fenitrotion e esfenvalerato, respectivamente. Foi comprovada a eficiência do método de avaliação no desempenho de bicos quando utilizada a calda inseticida como líquido teste. Os fatores que influenciaram o depósito dos inseticidas nas lâminas de vidro e nos grãos são discutidos no trabalho.
Palavras-chave: tecnologia de aplicação, cromatografia gasosa, depósito de inseticidas, milho, trigo

INTRODUCTION

Knowing the best application technique for grain protection is of fundamental importance for studying the effectiveness of the pesticide treatment. The international literature contains scientific papers that fail to define the application method used, making it difficult to compare results obtained from different researches. 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. In a hydraulic nozzle performance study, the following parameters are evaluated: flow, volumetric distribution, and droplets spectrum. In this respect, the International Organization for Standardization has established that clean water should be used as test fluid (ISO, 1981). On the other hand, the physical properties of the sprayed fluid influence the spray characteristics, droplets spectrum, and deposition pattern of pesticides (Sundaram et al., 1987a; 1987b; Sundaram & Retnakaram, 1987; Matuo et al., 1989). Calibration of the spray system using data obtained in hydraulic nozzle performance studies, as described in ISO standard 5682/1-1981 (E), might be responsible for overdose or underdose applications of insecticides. The literature shows the need of research on the evaluation of application techniques on stored grains. Thus, the objective of this work was to develop such methodology to evaluate the performance of hydraulic nozzles used in this purpose.

MATERIAL AND METHODS

Application technology

A twin jet, model TJ-60 8002EVS hydraulic nozzle was used. A channeled table (patternator) was used to carry out the spray nozzle transversal volumetric distribution analysis experiments, based on the ISO 5682/1-1981 (E) standard. The testing table (3.5 m long, 3.0 m wide) has channels spaced at 0.025 m, with a 5% slope. On the front part of the table, a set of graduated cylinders (250 mL) collects the fluid from each channel. An insecticidal mix (0.4% Sumigranplusâ EC) was used as test fluid. 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 weighing method was used to obtain actual flow, and the volume collected during one minute in a plastic container was weighed 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 90° angle in relation to the assay table.

Collection time was set until one of the graduated cylinders reached a volume of 230 mL. This collection time was used for the three replicates. After the effective swath width was determined, we studied the droplets spectrum. For that effect, 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, Gotas, version 1.0 (Embrapa Meio Ambiente, São Paulo, Brazil).

Corn and wheat cultivars Sol-da-Manhã and BRS 208 were used. To determine the mass of grains per unit area, the corn and wheat were spread as a fine layer onto a plastic tarp, covering 1 m^2 area, and were then weighed. 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 uniformly spread out on the tarp. The swath width where the grains were spread was established based on the nozzle's transversal volumetric distribution study performed previously. In order to check on the intended application rate, three glass slides (0.1 m length, 0.05 m width) were placed on the grains for later deposition quantification using gas chromatography. Insecticide losses were evaluated by collecting and analyzing seven plastic tarp samples (0.1 m length, 0.1 m width). Fenitrothion and esfenvalerate were applied so as to produce theoretical concentrations of 10 and 0.5 mg kg^{-1}, respectively. A commercial product containing 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 (Figure 1); the nozzle's operational specifications were the same as in the laboratory tests. The system's moving speed was calculated for an application volume equivalent to 5 L t^{-1}; under these conditions the insecticidal emulsion contained 0.4% of the commercial product. Three replicates 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 only of water. The temperature and relative humidity during spray were 26.2°C and 76%, respectively.

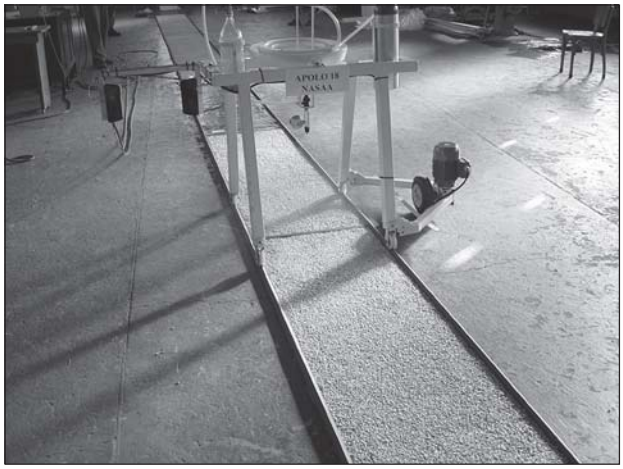

Figure 1 - Grain treatment.

Deposition analysis

Grain

Half an hour after the spray, the grains were collected and processed together 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 subsamples obtained from the homogenized material were stored under freezing conditions at -20ºC.

The analytical method for pesticide analyses was adapted from Ohlin (1998). Ten g of homogenized samples were placed in 100 mL Schott bottles for residue extraction. Fifty mL ethyl acetate and 10 g sodium sulfate 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. Ten mL aliquots of the supernatant were transferred to 12-mL test tubes, corresponding to 2 g of the original sample, and were then added of 50 mL dodecane. The extracts were evaporated in a Turbo-Vap evaporator, in water bath at 30°C aided by moving air previously dried through a blue silica gel desiccant filter. Later the insecticide residues were resuspended in 5 mL of a cyclohexane / ethyl acetate mixture (1:1, v/v), homogenized in 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, v/v). After this operation, the extracts were evaporated in a Turbo-Vap evaporator previously added of 50 µL dodecane and were later resuspended in 20.0 and 1.95 mL of the cyclohexane / ethyl acetate mixture (1:1, v/v) for the fenitrothion and esfenvalerate residues, respectively. The samples were analyzed by gas-phase chromatography, with a Thermo Electron Corporation, model Finnigan Trace Ultra 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 µm 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 (start); then at 280°C (25°C min^{-1} ramp), remaining at this temperature for a period of ten minutes; injector temperature = 230°C; detector temperature = 320°C; purge time = 1 minute; gas flow (mL min^{-1}): H_2 (carrier) = 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^{-1} for fenitrothion and 0.05, 0.1, and 1.0 mg kg^{-1} for esfenvalerate, with three replicates for each level (nine fortified samples for each matrix). Recoveries between 70 - 120% were considered acceptable.

Glass slide

Three glass slides were placed into 600 mL flasks. Five hundred mL ethyl acetate were added and the insecticides were later extracted by ultrasound for 15 min. Two-mL aliquots were transferred to 12-mL test tubes and were then added of 50 µL dodecane. The extracts were evaporated in a Turbo-Vap evaporator, in water bath at 30°C aided by moving air previously dried through a blue silica gel desiccant filter. Later, the insecticide residues were resuspended with 2 mL of the cyclohexane / ethyl acetate mixture (1:1, v/v) (1:1 mL) and homogenized in a vortex mixer/ultrasound, and then diluted at a rate of 1 mL of extract + 9 mL of the cyclohexane / ethyl acetate mixture (1:1, v/v), followed by injection in the chromatograph system.

Plastic tarp

Seven 100 cm^2 samples were cut into small pieces and placed in 100 mL Schott bottles. Fifty mL ethyl acetate were added and the insecticides were later extracted by ultrasound for 15 min. Upon completion, 5 mL aliquots of the solution were filtered through a Millipore, FG, 0.2 µm pore membrane filter mounted on a plastic hypodermic syringe (5 mL) and then diluted at proportions of 0.1 mL of the extract +

19.9 mL ethyl acetate for fenitrothion analysis, and 0.1 mL of the extract + 0.9 mL ethyl acetate for esfenvalerate, followed by chromatographic analysis.

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 species, insecticide, and interactions) in the model (Pimentel-Gomes, 2000).

RESULTS AND DISCUSSION

Application technology

The nozzle's actual flow was 0.672 L min^{-1}, 3.4% higher than the nominal flow of 0.650 L min^{-1} as defined by the manufacturer. The variation between actual and nominal flow was within the acceptable limit since according to the WHO (1976) the acceptable flow variation limit of a spraying nozzle is ± 4% in relation to the nominal flow indicated by the manufacturer. At the present working conditions a total swath width of 0.95 m was obtained, with a coefficient of variation (cv) of 34% (Figure 2). The cv was higher than the 7% limit established by the international standards (ECS, 1997). Even though the flow value was in accordance with the international standard, the transversal volumetric distribution varied (Figure 3), probably due to the presence of irregularities on the spray tip's orifice. The model studied is an "even-spray nozzle" or continuous deposition type, and is only used in swath applications. This problem could cause irregular insecticide deposition; consequently the grains would receive under- or overdoses depending on their placement in the total swath width, thus compromising insecticide effectiveness and residue studies.

Le Patourel (1992), Jermannaud & Pochon (1994), and Acda et al. (1994) have demonstrated that great insecticide deposition variation occurs in stored grains. On the other hand, deposition variations could favor the evolution of insect resistance to insecticides. In order to obtain an insecticidal mix distribution as uniform as possible, and considering that the spray system had a swath width application capacity of up to 0.6 m, we determined effective swath width and cv values of 0.6 m and 5.14%, respectively. Under these conditions, 71.59% of the sprayed application volume were collected within the effective swath width (Figure 2). Therefore, the spraying equipment was calibrated to apply a total volume of 7 L t^{-1}, since 28.41% of this volume would remain outside the treatment site. Consequently, the grains would receive an effective application volume of 5 L t^{-1} as intended. The nozzle flow in the spraying equipment at a pressure of 200 kPa was 0.660 L min^{-1}; this value was similar to the flow obtained in the laboratory test. In this situation, the sprayer moving speeds were 1.88 and 2.36 km h^{-1} for the corn and wheat treatments, respectively.

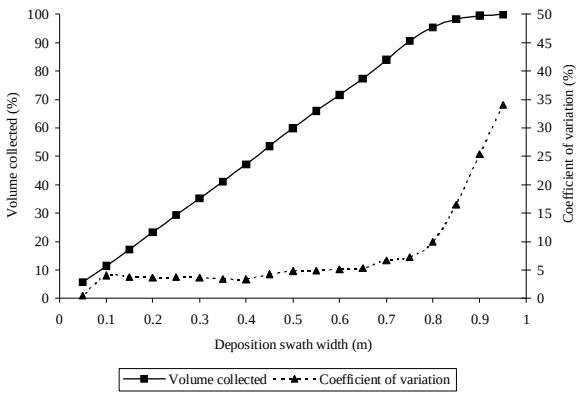

Figure 2 - Transversal volumetric distribution of a TJ-60 8002EVS nozzle using an insecticidal mix during spray.

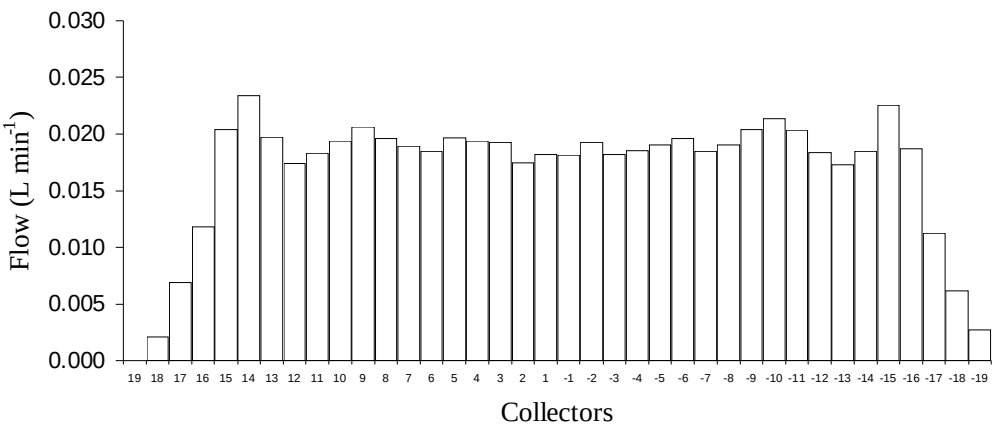

Figure 3 - Transversal volumetric distribution pattern of a TJ-60 8002EVS nozzle using an insecticidal mix during spray.

The droplets spectrum for the nozzle under evaluation, working at pressure and moving speed values of 200 kPa and 5 km h^{-1} is presented in Table 1. The droplets spectrum was uniform across the entire effective swath width. Pesticide sprays are generally classified based on droplet size, with particular reference to volumetric mean diameter (Matthews, 2000). According to the manufacturer's brochure, the TJ-60 8002EVS nozzle yields fine droplets under all recommended work pressures; however, large droplets were obtained in the present study. The droplet size categories used in this experiment were the same as in the international ASAE (X-572) and BCPC standards. The differences in actual 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 used water-sensitive paper to obtain droplet marks and to make diameter measurements at a later time using specific software. On the other hand, the viscosity and surface tension of the insecticidal mix may have increased droplet size. Emulsions cause a rapid fluid sheet disintegration with the formation of large droplets (Butler Ellis et al., 1997).

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 was recovered from the control, indicating that the grains were free from contamination by those compounds. The F test in the analysis of variance detected a significant insecticidal deposition effect ($P < 0.05$), both on grains and on glass slides. In addition, there was an effect ($P < 0.05$) of grain species on insecticide deposition. The lack of a significant interaction ($P > 0.05$) between grain species and insecticide indicates that deposition on grain species is independent from insecticide and vice versa.

The fenitrothion deposits were higher than those for esfenvalerate, both on grains and on glass slides (Figure 4). In spite of the fact that the vapor pressure of these insecticides would determine greater esfenvalerate stability, more fenitrothion was recovered. The environmental conditions during spray were adequate for this operation, and processing of the corn and wheat samples included the use of dry ice. Consequently, all steps that preceded the analytical stage prevented losses of both insecticides; therefore, the greater recovery of fenitrothion was due to the higher sensitivity of the chromatograph detector to this molecule. The depositions of both insecticides were always higher on the glass slides when compared with depositions on the grains. Depositions on the pieces of plastic tarp corresponded on average to 8.9 ± 2 and 6.5 ± 0.4% of the theoretical insecticide dose in corn and wheat, respectively. Some droplets reached the plastic tarp through the empty spaces between the grains, therefore resulting in lower depositions than those intended. The sum between grain and plastic tarp depositions should be near the glass slide deposition values, but was considerably lower. One explanation for these results is that 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. Greater effectiveness of the artificial target in collecting pesticides in agricultural nozzle performance studies is therefore demonstrated. Insecti-

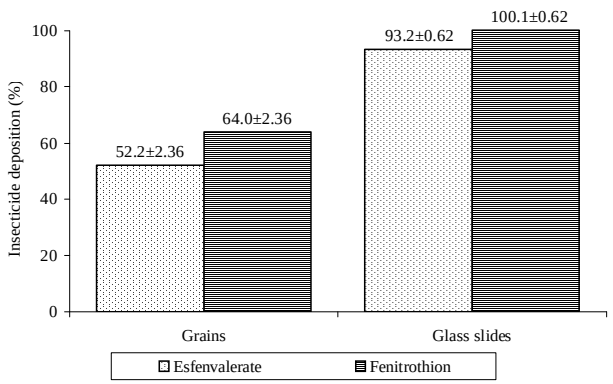

Figure 4 - Means and standard errors of fenitrothion and esfenvalerate depositions on grains and corresponding glass slides.

Table 1 - Droplet analysis for a TJ-60 8002 EVS nozzle using an insecticidal mix during spray. VMD: Volumetric mean diameter, NMD: Numeric mean diameter.

Parameters	Position of water-sensitive paper on effective swath width		
	Left	Center	Right
Volume (L ha^{-1})	121.841 ± 6.217	130.279 ± 7.694	142.770 ± 15.542
Density (n^o cm^{-2})	128 ± 15	125 ± 1	120 ± 11
Uniformity	1.817 ± 0.019	1.883 ± 0.100	1.855 ± 0.084
VMD (µm)	362 ± 9	370 ± 15	384 ± 33
NMD (µm)	194 ± 4	197 ± 2	207 ± 8
Coating (%)	24.708 ± 1.574	25.706 ± 0.790	27.428 ± 1.717

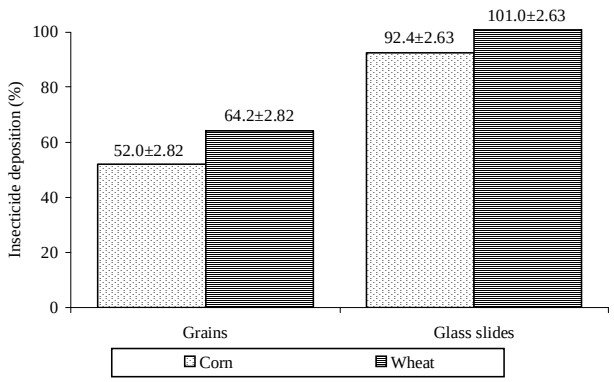

Figure 5 - Means and standard errors of insecticide depositions on corn and wheat grains and on corresponding glass slides.

cide deposition on wheat was higher than on corn, both for grains and glass slides (Figure 5). However, the difference in the case of glass slides was not significant ($P > 0.05$), probably due to its grain morphology, wheat provided a higher specific contact surface area for droplets. Finally, the results herein reported demonstrate the high efficiency of this new method to evaluate the performance of hydraulic nozzles when an insecticidal mix is used as test fluid.

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