Introduction

The goal of a pesticide spray treatment is to locate adequately the correct amount of active ingredient on the vegetation in the safest and cheapest way (Hislop, 1987). Many researchers have worked at assessing the real or potential effectiveness of spray applications of plant protection products by studying the deposition patterns of treatments on the surface of vegetation (Salyani et al., 1988; Ebert & Hall, 1999). Similarly, there is a huge amount of research comparing deposition patterns of different machines, application techniques or operating conditions in order to assess the optimal way of distributing pesticides in the field (Cross et al., 2003; Farooq & Salyani, 2004).

Spray deposition is often described in terms of coverage, which is the percent of surface covered by impinging droplets (hereafter called impacts), number of impacts per square area and average size of impacts. Coverage is the most accurate parameter that can be estimated (Salyani & Fox, 1999). Moreover, when impacts overlap, it is the only feature that is worth analyzing (Holownicki et al., 2002).

Information on the distribution of the spray on vegetation has usually been obtained by image analysis of droplet distributions on leaves or on artificial targets (Jiang & Derksen, 1995; Holownicki et al., 1996; Cross et al., 1997).

In the case of applications of treatments to citrus trees, natural leaves are not completely flat, which causes errors in the measurements using optical devices, and droplets may slip during handling. In addition, it is difficult to generate a good contrast between the droplets and the surface of leaves because of their dark-green colour. For these reasons, researchers have used small portions of white polyvinyl chloride (PVC) sheets as artificial targets to simulate the behaviour of citrus leaves (Vidal et al., 2003; Chueca et al., 2010; Garcerá et al., 2011) since previous work showed that these collectors did not show statistically significant differences in coverage percentage, number of impacts per square centimeter and mean area of the impacts.
when compared to citrus leaves (Mercader et al., 1995). Despite this important advantage, PVC collectors are very difficult to handle in the field, because droplets formed on their surface easily slip, as happens on leaves, which changes the apparent distribution of impacts, and their use is often restricted to the laboratory.

Alternatively, water sensitive papers (WSP) are widely used in field experiments as artificial targets because they are relatively easy and cheap to use in order to visually estimate spray distribution and penetration (Fox et al., 2003). They provide useful information about the penetration of the spray into the canopy and have been used to evaluate different types of sprayers, nozzles, or operating conditions (Marçal & Cunha, 2008; Salyani et al., 2013). However, they retain the spray very differently compared to plant surfaces (Holownicki et al., 2002), absorbing and expanding the aqueous portion of the spray, so they cannot be used for an accurate determination of spray pattern deposition.

In summary, WSP are very useful and convenient in field experiments but have different properties than citrus leaves, while PVC collectors adequately represent citrus leaves but are difficult to handle in the field. Field tests with WSP provide important information about the distribution of the pesticides in the canopy, while laboratory tests with PVC generate essential data to understand the efficacy of plant protection products on pests. In both cases, it is important to assess the quantity of product deposited on the target for assessing the relationship between the amount of pesticide and its effect on the population of the pests (Garcerá et al., 2011, 2012). Also these considerations highlight the importance of relating coverage observed on PVC targets to that observed on WSP in order to relate results in laboratory and to those collected in field experiments.

On the other hand, it is well known that physico-chemical properties of pesticide solutions affect the distribution pattern (Butler-Ellis et al., 2001). For these reasons, this paper is aimed at generating mathematical models of these relationships using different commercial pesticides that are commonly employed in citrus cultivated in Spain. First, it shows a method to estimate coverage on both artificial collectors from deposition data for different pesticide solutions. Secondly, it shows the relationship between the coverage observed on both collectors, which can be used to relate field and laboratory observations. Finally, in the discussion section, it describes how these models could be applied to link laboratory data on biological efficacy of treatments (obtained with PVC collectors) with the expected efficacy of field treatments once deposition patterns in the field have been observed with the help of WSP.

Material and methods

Collectors

Experiments were carried out to determine the relationship between the coverage obtained on two different artificial targets when using different solutions of pesticides in water. Trials consisted of spraying a series of volumes (0.25, 0.4, 0.5, 0.6, 0.8, 1, 1.5, 2, 2.5, 3 and 4 mL) of different solutions on PVC collectors and water-sensitive papers (WSP). The selection of the range of volumes to be tested was based on preliminary tests, since these volumes cover a wide range of coverage levels achieved in field treatments. The upper tested volume was 4 mL because higher volumes sprayed on PVC targets makes droplets coalesce in such a way that they run off from the target surface, as it is often observed on other surfaces (Ebert & Downer, 2006).

Collectors consisted of 4 × 4 cm pieces of white PVC sheets and WSP measuring 7.6 × 2.6 cm (Teejet, Spraying Systems Co., IL, USA). Droplets impacting WSP change the color of the surface from yellow to dark-blue, providing sufficient contrast for subsequent image analysis.

When spraying the PVC targets, 2% of chelated iron (Sequestrene 138 Fe G-100, Syngenta Agro S.A., Madrid, Spain) was added as a dye to produce the necessary drop/background contrast. Previous assays demonstrated that this product did not affect the spray pattern (data not shown).

Sprayed solutions

Plant protection products tested in this study were the most frequently applied solutions against one of the most important pests of citrus cultivated in Spain, which is California red scale (Aonidiella aurantii Maskell). All the products were dissolved at the concentration recommended in the label, which is assumed to be the same that the one employed in field applica-
tions. It should be remarked that modifications of these concentrations or the use of adjuvants not included in the commercial formulation may affect the deposition characteristics of the spray, but in this work experiments have been conducted with solutions that are comparable to those commonly employed by farmers. The following solutions were tested:

- Deionized water, as a reference.
- Two organophosphate insecticides, both used at maximum label concentration:
  - Dursban® 75 WG (a.i.: chlorpyrifos 75% [WG] w/w) (Dow AgroSciences Ibérica, Madrid, Spain) at 1.25 g L⁻¹.
  - Reldan® E (a.i.: chlorpyrifos-methyl 22.4% [EC] w/v) (Dow AgroSciences Ibérica, Madrid, Spain) at 4 mL L⁻¹.
- An insect growth regulator, used at maximum label concentration: Atominal® 10 EC [a.i.: pyriproxyfen 100 g L⁻¹ (EC) w/v] (Sumimoto Chemical Co. Ltd., Tokyo, Japan) at 0.75 mL L⁻¹.
- Two petroleum-derived paraffinic spray oils, both used at a concentration of 15 mL L⁻¹:
  - Laincoil®, an nC21 oil with a content of 83% w/v; unsulfonated residue 92%; density (20°C) 0.82-0.86 g mL⁻¹; viscosity (40°C): 14.38 cSt (Lainco, Barcelona, Spain).
  - Sunspray Ultrafine®, an nC21 with a content of 85% w/v; unsulfonated residue 92%; density (20°C) 0.85-0.86 g/mL; viscosity (40°C): 12.60 cSt (Sun Oil Co., Antwerp, Belgium).
- Additional mixtures also used in the currents:
  - Dursban® 75 WG at 1.25 g L⁻¹ plus Atominal® 10 EC at 0.75 mL L⁻¹.
  - Dursban® 75 WG at 1.25 g L⁻¹ plus Laincoil® at 10 mL L⁻¹.

All eight products/product combinations were tested in 11 different volumes on the two types of collectors with three replications for a total of 528 applications. Applications were performed in a random order and the spraying device was always carefully cleaned before each application.

**Spraying system**

Applications were made with a pneumatic Potter Spray Tower fitted with its finest nozzle (orifice diameter: 0.762 mm) (Burkard Scientific, Uxbridge, UK) (Potter, 1952). A diagram of the spray tower can be seen in Fig. 1. This device has been reported to be widely used in bioassays, but it has to be taken into account that it generates droplet patterns whose distribution in sizes may differ from that produced in field applications. This is one of the reasons for this work to consider other parameters, more related to the amount of product that is deposited, namely the spray coverage and the amount of deposited solution.

A tube on top of the Potter Spray Tower contains the volume to be applied at each application. An air lead is connected to the nozzle to generate an airflow that induces the spray. Air pressure was fixed at 0.1 MPa. The spray cloud falls down through the tower and reaches a Petri dish situated at the bottom, on top of the spray table.

Potter (1952) acknowledged that differences in temperature and humidity affect the deposit on a target situated on the spray table. Moreover, previous experiments showed that part of the applied volume did not reach the Petri dish because they stuck on the walls of the tower or because the finest droplets evaporated. In order to estimate the actual amount of solution deposited per unit area (µg cm⁻²) on the spray table for each spray volume, the tower was calibrated every day before the applications. For this purpose, a set of volumes (0.5, 1, 2, 3 and 4 mL) of deionized water were sprayed over Petri dishes with a known surface area (63 cm²) in a random order and with five replicates. Petri dishes were weighed before and immediately after the application using an analytical balance (XR 205 SM-DR, Precisa Instruments Ltd., Dietikon, Switzerland). The average increase of weight produced by the deposition of water was divided by the surface of the Petri dish and a correcting function was calculated by linear regression. This function was applied to all the
tests performed on that day, implicitly assuming that the weight of the pesticides in the solutions was negligible and that the products did not affect the amount of deposited solution. Table 1 shows the average and standard deviation of the deposited water for all the tests.

### Data acquisition and analysis

Collectors were photographed immediately after application and the images analyzed using the methodology described by Chueca et al. (2010) to estimate coverage on each collector, expressed as percentage of area occupied by the impacts against the total area (%). Finally, the average coverage on each collector of each combination of volume and sprayed solution was calculated.

Plots of solution deposition versus coverage data were drawn for each type of collector before proposing the models. After observing these plots, a set of rules were imposed to the functions to fit the experimental data. These were the rules for the WSP functions: a) they should be sigmoidal or exponential, and asymptotic to 100% coverage on WSP; b) they must intersect (0,0); c) a deposit of 3,000 µg solution cm\(^{-2}\) or higher should imply at least 90% coverage on PVC. These two figures were chosen arbitrarily from the observed data and had similar purpose than before.

A series of basic models described by one or two parameters were proposed to adapt to the data (Fig. 2). Basic models were adjusted to observed data using Excel Solver (Frontline Systems Inc., Microsoft® Office Excel 2007). This tool finds an optimal value for a parametric function, called objective function, by iteratively modifying the values of its parameters. In this study, the objective function was the sum of the quadratic differences between real coverage values on the artificial collector and the calculated values of the estimation functions. The objective was to minimize this sum by modifying the values of the parameters of the selected model.

Once the models were adjusted for each tested solution, their goodness-of-fit was assessed by calculating the coefficient of determination (\(R^2\)) between the observed and the estimated coverage values. For each type of collector, the two models with the highest \(R^2\) value for the majority of solutions were selected as candidates. However, \(R^2\) values are very sensitive to outliers, and considering high \(R^2\) alone may lead to the selection of over-fitted functions that cannot be generalized. Furthermore, the root mean squared error of prediction (RMSEP) was calculated to select the models with lowest estimation error. Moreover, the distribution of the residuals (differences between the observed and the estimated values) was also investigated to select only models with residuals that were close to the normal distribution. Normality of residuals was assessed using the Shapiro-Wilk’s normality test (Shapiro & Wilk, 1965). We assumed \(p\)-values higher than 0.1 to imply a normal distribution of the residuals of the estimation functions, with a confidence level of 90% or higher. Random distribution of residuals was tested by plotting them against the deposit.

Differences of coverage behavior between solutions were analyzed by plotting predicted functions and by studying the differences between their parameters.

### Table 1. Water deposition (mean ± SE) on Petri dishes when spraying water with a Potter Tower (pressure 0.1 Mpa using the fine nozzle)

| Volume Sprayed (mL) | Deposited water (µg cm\(^{-2}\)) |
|---------------------|----------------------------------|
| 0.250               | 128.18 ± 10.84                   |
| 0.400               | 263.44 ± 16.02                   |
| 0.500               | 352.37 ± 17.57                   |
| 0.600               | 440.19 ± 22.51                   |
| 0.800               | 656.83 ± 32.45                   |
| 1.000               | 821.80 ± 37.88                   |
| 1.500               | 1,363.37 ± 66.91                 |
| 2.000               | 1,782.61 ± 78.97                 |
| 2.500               | 2,382.55 ± 114.89                |
| 3.000               | 2,821.87 ± 128.16                |
| 4.000               | 3,817.05 ± 164.18                |

Similarly, another set of rules was imposed to the functions to model the relationships between coverage and deposition on PVC: a) they should be sigmoidal or exponential, and asymptotic to 60% coverage on PVC (which was observed to be close to the saturation of the PVC collectors in the above mentioned plots); b) they must intersect (0,0); c) a deposit of 5,000 µg solution cm\(^{-2}\) or higher should imply at least 50% coverage on PVC. These two figures were chosen arbitrarily from the observed data and had similar purpose than before.
Highest values of $R^2$ were obtained with basic models 1 and 2 for both types of collectors (Fig. 2 and Table 2). Moreover, these models also showed the lowest values of RMSEP (Table 3), so they were preselected.

All $p$-values for the Shapiro-Wilk’s test for normality of residuals for basic model 1 were above 0.1 for both collectors and all tested solutions (Table 4). However, $p$-values of basic model 2 were lower than those for model 1 in most cases and below 0.1 in some cases. Random distribution of residuals for this model was checked.
As a result, basic model 1 was selected for both collectors since it had higher goodness-of-fit and residuals could be considered normally distributed for all solutions. Eq. [1] is proposed for the best estimation of coverage on WSP from the spray deposit:

$$\text{Table 2. Goodness-of-fit (R}^2\text{) of the different basic models tested for each pesticide solution and collector}$$

| Solution                | WSP Function | Function | Function | Function | PVC Function | Function | Function | Function | Function |
|-------------------------|--------------|----------|----------|----------|--------------|----------|----------|----------|----------|
| Water                   | 0.887^1      | 0.871^2  | 0.773    | 0.802    | 0.915^1      | 0.900^2  | 0.893    | 0.889    |
| Dursban® 75 WG          | 0.892^1      | 0.881^2  | 0.780    | 0.825    | 0.948^1      | 0.931^2  | 0.927    | 0.895    |
| Reldan® E               | 0.844^1      | 0.831^2  | 0.715    | 0.741    | 0.960^1      | 0.951^2  | 0.938    | 0.931    |
| Atominal® 10 EC         | 0.878^1      | 0.853^2  | 0.734    | 0.758    | 0.950^2      | 0.952^1  | 0.942    | 0.932    |
| Laincoil®               | 0.920^1      | 0.907^2  | 0.764    | 0.789    | 0.890^1      | 0.890^2  | 0.883    | 0.871    |
| Sunspray Ultrafine®     | 0.885^1      | 0.855^2  | 0.803    | 0.815    | 0.949^1      | 0.937^2  | 0.933    | 0.927    |
| Dursban® 75 WG + Atominal® 10 EC | 0.883^1 | 0.872^2  | 0.759    | 0.783    | 0.939^2      | 0.925    | 0.930    | 0.946^1  |
| Dursban® 75 + Laincoil® | 0.890^1      | 0.883^2  | 0.825    | 0.836    | 0.904^1      | 0.898    | 0.884    | 0.902^2  |

1 The highest R^2 observed for each solution on each collector. 2 The second highest R^2 observed for each solution on each collector.

| Table 3. Root mean squared error of prediction (RMSEP) of the different basic models tested for each pesticide solution and collector |
|---------------------------------------------------------------|---------------------------------------------------------------|
| Solution                | WSP Function | Function | Function | Function | PVC Function | Function | Function | Function | Function |
|-------------------------|--------------|----------|----------|----------|--------------|----------|----------|----------|----------|
| Water                   | 10.804^1     | 11.557^2 | 20.360   | 15.707   | 4.759^1      | 5.411^2  | 5.872    | 5.498    |
| Dursban® 75 WG          | 10.454^1     | 10.938^2 | 20.906   | 14.265   | 3.566^1      | 4.363^2  | 4.655    | 6.024    |
| Reldan® E               | 12.840^1     | 13.533^2 | 23.191   | 18.796   | 3.540^1      | 3.978^2  | 5.367    | 4.704    |
| Atominal® 10 EC         | 10.777^1     | 12.163^2 | 22.089   | 17.723   | 3.563^1      | 3.586^2  | 4.177    | 4.804    |
| Laincoil®               | 9.251^1      | 10.154^2 | 21.569   | 16.894   | 5.645^1      | 5.792^2  | 6.064    | 6.715    |
| Sunspray Ultrafine®     | 11.896^1     | 12.192^2 | 14.513   | 13.656   | 4.482        | 4.026^2  | 3.914^1  | 4.382    |
| Dursban® 75 WG + Atominal® 10 EC | 10.744^1 | 11.312^2 | 21.981   | 17.070   | 3.905^1      | 4.930    | 4.241^2  | 6.500    |
| Dursban® 75 + Laincoil® | 9.871^1      | 10.395^2 | 19.252   | 14.187   | 5.002^1      | 5.411^2  | 5.744    | 6.877    |

1 The lowest RMSEP observed for each solution on each collector. 2 The second lowest RMSEP observed for each solution on each collector.

As a result, basic model 1 was selected for both collectors since it had higher goodness-of-fit and residuals could be considered normally distributed for all solutions. Eq. [1] is proposed for the best estimation of coverage on WSP from the spray deposit:

| Table 4. Shapiro-Wilk’s test over the residues of basic models 1 and 2 for each solution and collector (p-values) |
|---------------------------------------------------------------|---------------------------------------------------------------|
| Solution                | WSP Function | Function | Function | Function | PVC Function | Function | Function | Function |
|-------------------------|--------------|----------|----------|----------|--------------|----------|----------|----------|
| Water                   | 0.437***     | 0.003    |          |          | 0.718***     | 0.087    |          |          |
| Dursban® 75 WG          | 0.260*       | 0.836*** |          |          | 0.679***     | 0.208*   |          |          |
| Reldan® E               | 0.171***     | 0.006    |          |          | 0.722*       | 0.975*** |          |          |
| Atominal® 10 EC         | 0.123***     | 0.015    |          |          | 0.562***     | 0.477*   |          |          |
| Laincoil®               | 0.972***     | 0.131*   |          |          | 0.825***     | 0.213*   |          |          |
| Sunspray Ultrafine®     | 0.537***     | 0.305*   |          |          | 0.358*       | 0.477*** |          |          |
| Dursban® 75 WG + Atominal® 10 EC | 0.633*** | 0.461*   |          |          | 0.575***     | 0.212*   |          |          |
| Dursban® 75 + Laincoil® | 0.660***     | 0.571*   |          |          | 0.865***     | 0.411*   |          |          |

* p-value > 0.1 (residuals are considered as normally distributed). ** Highest p-value for each solution and collector.
In the case of PVC, the relationship is defined by Eq. [2]:

\[
\text{Coverage PVC} (\%) = 60 \times \left(1 - e^{-b \times \text{deposit}}\right) \quad [2]
\]

Table 5. Values of “a” in Equation [1] relating deposit and coverage on WSP for each solution

| Solution                          | a    |
|----------------------------------|------|
| Atominal® 10 EC                  | 0.081|
| Reldan® E                        | 0.082|
| Laincoil®                        | 0.083|
| Dursban® 75 WG + Atominal® 10 EC | 0.085|
| Dursban® 75 WG + Laincoil®       | 0.088|
| Dursban® 75 WG                   | 0.091|
| Water                            | 0.091|
| Sunspray Ultrafine®              | 0.110|

Optimal values for parameters a were calculated for each solution (Table 5). This parameter is related to the rate increase of the data to reach the asymptotic horizontal line, and affects the slope of the initial part of the curve. The higher a, the higher the initial slope and the lower the volume of the corresponding solution necessary to saturate WSP, that is, to reach 100% coverage.

The value of the parameter a for solutions with Dursban 75® was close to that of water, while those for Atominal®, Reldan® and Laincoil® were lower, which means that these solutions needed a slightly higher level of deposition to increase PVC coverage. On the other hand, for Sunspray Ultrafine® solutions parameter a had values much higher than water, which implies a lower deposition requirement to increase coverage and to saturate the collector.

In the case of using PVC collectors, results were slightly different (Table 6). As with WSP, parameter b for solutions with Dursban 75® were close to water. However, those for Atominal®, Reldan® and Laincoil® were higher, while parameter b for Sunspray Ultrafine® were much higher than the rest here, too, implying that it requires less deposit to increase coverage and to saturate the collector.

Differences and similarities among solutions for the entire range of deposition are graphically shown by plotting the estimation functions (Fig. 3). It is important to note that the two petroleum-derived spray oils showed big differences in coverage, probably due to the use of different adjuvants in their formulation. Furthermore, from Eqs. [1] and [2], a function relating coverage on WSP and coverage on PVC can be obtained (Eq. [3]):

\[
\frac{\text{Coverage WSP} (\%)}{\text{Coverage PVC} (\%)} = 100 \left[1 - \left(1 - \frac{\text{Coverage PVC}}{60}\right)^{\gamma}\right] \quad [3]
\]

**Discussion**

This work opens the possibility to estimate the deposition of a plant protection product in field applications from coverage values measured on WSP using the inverse of the function shown in Eq. [1]. Nansen et al. (2010) also proposed a method to use WSP to estimate deposition of products, but their system was based on image analysis to measure the intensity of the blue colour generated on WSP, reporting that the major drawback of their method was its dependence on the lighting system and on the digitizer. In our opinion, it is also important to remember the limitation of WSP collectors for both methods in displaying an asymptotic relationship between coverage and deposition.

Previous work showed mathematical models to relate the deposition of plant protections products with the mortality inflicted on different developmental stages of California red scale (Aonidiella aurantii Mask) (Garcé et al., 2011, 2012). The current study makes it feasible to estimate coverage on non-saturated WSP by using Eq. [1], thus allowing the determination of optimal coverage in field applications to obtain maximum efficacy without a disproportionate use of pesticide.

For instance, it has been demonstrated that 1.01 µL cm⁻² of Dursban® 75 WG or Reldan® E solutions were sufficient to control 90% of Aonidiella aurantii in citrus and that higher depositions did not increased the...
efficacy of the treatments (Garcé r et al., 2011). From Eq. [1], it can be predicted that optimal efficacy can be obtained if coverage on WSP is of around 26% with Dursban® 75 WG or 43% with Reldan® E. This provides a method for establishing simple recommendations on the level of coverage to be achieved after an application for technicians and farmers in the citrus industry.

In addition, these models can also be used to estimate the expected efficacy of field applications from coverage obtained on WSP situated at different positions of the canopy, by calculating the expected control of the pest using the efficacy models (Garcé r et al., 2014).

The present work can be further exploited for estimating the required coverage on WSP for treatments against other pests of citrus one. The relationship between the deposit of a pesticide and the level of control of the pest has to be established. This can be done in the laboratory using PVC sheets. In such trials, the relationship of coverage on PVC and deposit can be established using the same method that led to Eq. [2]. Finally, using the approach that was used to establish Eq. [3], the equivalent coverage on WSP can be assessed. Nevertheless, it is important to remember that further studies are required in order to apply the proposed method to other pests as it ultimately depends upon the effect of the products on the pests.

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