A SURVEY OF PESTICIDE RESIDUES IN CUT FLOWERS FROM VARIOUS COUNTRIES

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SUMMARY

As in any intensive culture, flowers require the use of a wide range of pesticides to control diseases and pests which can damage production and marketability. In order to evaluate the average levels of contamination of the cut flowers and to assess the risk for professionals exposed to pesticide residues when handling cut flowers, a survey was carried out with a group of florists from the Belgian largest cities. Fifty samples of roses (5 stems per bouquet) were collected: 45 bouquets were sampled in the 7 largest cities of Belgium (Antwerp, Brussels, Charleroi, Ghent, Leuven, Liege and Namur) and 5 were sampled from 5 supermarkets. Analysis of residual pesticide deposit is made by combining two multi-residue methods (GC-MS-MS and LC-MS-MS) in a laboratory accredited for pesticide residues. For all the samples analysed, a total of 97 active substances were detected, i.e. an average of 14 active substances per bouquet and a total average pesticide load of 26,03 mg/kg per flower sample. Most active substances (a.s.) reached high levels of residues, with concentrations between 10 and 50 mg/kg. Samples from Belgium and The Netherlands have a lower average number of a.s./sample, but the amount of residues is about the same in all samples (20-30 mg/kg) whatever the country of origin, except for the sample from Germany who is the worst case (22 a.s. with a total amount of 92 mg/kg). Most of the detected active substances are fungicides (dodemorph, spiroxamine, cyprodinil, fluopyram, pyrimethanil, benomyl (carbendazim), propamocarb, boscalid and iprodione) which are present on more than 20 of the 50 samples. All of them have a dermal acute toxicity. Consequently, florists who handle a large number of flowers are exposed daily with a potential effect on their health.

Key words: pesticide residues, cut flowers, roses, exposure assessment, florists.

INTRODUCTION

Floriculture is a most essential and vast part of horticulture. The total acreage allocated to cut flower production worldwide is now over 200,000 hectares, with roses, carnations, and chrysanthemums the dominant varieties (ITC, 2001). They are used on all religious festival occasions and given as birthday presents, wedding gifts or while meeting sick people and even at funerals and especially during the peak seasons (coinciding with Valentine's Day, Christmas, and other international holidays) (Korovkin, 2003; Palma *et al*., 2010).
Cut flower production in the world gained importance and has become a very popular commercial activity during last decade of the outgoing millennium, especially after the Second World War (Amar, 1995; Kendirli and Cakmak, 2007) with the globalisation of markets and the gradual appearance of strong players such as Colombia, Kenya, Ecuador and Zimbabwe (ITC, 2001).
Today the European Union market is a high potential importer of floriculture products. The growth trend in import is clear from the fact that the total imports which were 881 million dollars in 1990 improved to 1.684 million dollars in 2004. Roses accounted for a greater share of EU, whereas roses shot up to 61.94% (1,043 million dollars) 2004 from 33.6% (296 million dollars) in 1990. It is also evident that the roses are the preferred species in the EU from the

fact that over the period under review it experienced a compound growth rate of 9.41% which far exceeds the growth of other species of flowers imported into the EU (Ravinath, 2007).
As any intensive production, pesticide use is a significant strategy to fight against many pests (mainly mites and insects) and various diseases, so that ornamental producers can stay competitive in both national and international markets (Bethke and Cloyd, 2009).
While flowers are susceptible to pests and diseases, they are sprayed several times during their growth considering that no MRL are set for flowers. Therefore, residue deposits can be high and, as a consequence, a health hazard may exist to exposed individuals who handle those flowers contaminated by pesticides during cropping, cutting, sorting or bundling (Brouwer *et al*., 1992). The most common general signs and symptoms mentioned after exposure are weakness, fatigue with muscle pain, when other symptoms are often centred on the eye (eye itchiness and blurring of vision), ear, nose and throat or neuralgic (Lu, 2005). Mention may also be made about potential problems of reproduction (Restrepo *et al*., 1990; Weidner *et al*., 1998, Bell *et al*., 2001; Garry *et al*., 2002 & 2003; Beard *et al*., 2003; Hanke *et al*., 2004,), allergic reactions (Sato *et al*., 1998), increase in certain types of cancer (Dich *et al*., 1997; Infante-Rivard *et al*., 1999; Richter *et al*., 1999; Hardell *et al*., 2002; De Rose *et al*., 2003; Alavanja *et al*., 2003 & 2004; Bassil *et al*., 2007), neurological disorders (Baldi *et al*., 2003a & 2003b; Elbaz *et al*., 2004, Alavanja *et al*., 2004) such as Parkinson disease.
In order to assess the risk for professionals exposed to pesticide residues on flowers produced in Belgium or imported from various countries, a study has been carried out with a group of florist representatives of the Belgian largest cities on a voluntary basis.

MATERIALS AND METHODS

Fifty samples of roses (at least 5 stems/bouquet) were collected within 3 consecutive days. 45 bouquets were sampled from florists located in the 7 largest cities of Belgium (Antwerp, Brussels, Charleroi, Ghent, Leuven, Liege and Namur) and 5 were sampled from 5 supermarkets to evaluate the average residue levels of roses which are necessary to estimate the potential exposure of florists preparing bouquets.
During sampling, the bouquets were labelled and the countries of origin identified (asking the florists). After two centimetres of stem have been cut obliquely with a sterilised sharp knife to maintain water absorption, the bouquets were stored in a cool room in vases filled with tap water. All collected samples were transported within 2 days by road from Gembloux to the laboratory in Ghent.
The residual pesticide deposits on the bouquets were analysed in a laboratory holding a BELAC accreditation to ISO/CEI 17025 (PRIMORIS, Technologiepark 2/3, B-9052 Zwijnaarde – Ghent). PRIMORIS is a private, accredited and officially recognised service laboratory for herbal products.
The residues were extracted after the 5 flower stems had been totally crushed, taking a homogenous 10 g sub-sample. The extract was analysed using a combination of two multi-residue methods validated by the laboratory for pesticides residue analysis in foodstuffs. According to the active substances to be determined, the pesticide residue concentration was determined by GC-MS-MS or LC-MS-MS (gas chromatography or liquid chromatography with mass spectrometry). Gas chromatography was used to analyse relatively small, thermally stable, volatile, non-polar molecules. Liquid chromatography was used to analyse larger, thermolabile, non-volatile, polar molecules.

The combination of both methods allows the analysis of approximately 500 active substances in a single run (a screening of almost all pesticides usually sprayed on flowers). For most of the active substances, the quantification limit was ≤ 0.01 mg/kg.

RESULTS AND DISCUSSION

PESTICIDE RESIDUES CONCENTRATIONS ON ROSES

All flower samples appeared to be contaminated by pesticide residues whatever their origin. Most active substances (a.s.) reached high levels of residues, with concentrations between 10 and 50 mg/kg, about thousand times above the maximum limit value set for foodstuffs for most of those a.s. (EU MRL values) (Table 1).

Table 1. Pesticide residue concentrations in 50 samples of roses

Total Pesticide Residues Concentration (mg/kg, all a.s. together)	Samples with Pesticide Residues	
	Number of samples	%
0,01-0,99	2	4
1,00-4,99	4	8
5,00-9,99	7	14
10,0-50,00	33	66
>50,00	4	8
Total	50	100

FREQUENCY OF ACTIVE SUBSTANCE RESIDUES ON ROSES

Nine fungicides (dodemorph, spiroxamine, cyprodinil, fluopyram, pyrimethanil, benomyl (carbendazim), propamocarb, boscalid and iprodione) and one insecticide (imidacloprid) are the most frequently detected active substances. They are present on more than 20 of the 50 samples when most of the other active substances (29) are detected only once (Table 2).

97 different active substances were identified on the rose samples. 53 a.s. (roughly a half of all a.s.) with a frequency below 10%, and 29 a.s. (about 30%) were only detected in a single bouquet.

Table 2. Active substances found in the samples, number of bouquets contamined by each a.s. detected and frequency of detection (in %, with LOQ < 0.01 mg/kg)

Active substances detected in the samples	Number of samples where a.s. are detected (out of 50)	Frequency of detection (in %)
Dodemorph	37	74
Spiroxamine	34	68
Cyprodinil	31	62
Fluopyram,pyrimethanil	23	46
Benomyl (carbendazime), propamocarb	22	44
Imidacloprid	21	42
Boscalid, iprodione	20	40
Fludioxonil	19	38
Flonicamide, procymidone	18	36
Dimethomorph	17	34
Acephate, fluopicolide	15	30
Methamidophos	14	28

Active substances detected in the samples	Number of samples where a.s. are detected (out of 50)	Frequency of detection (in %)
Ethirimol , fenhexamide	13	26
Acetamiprid, clofentezine , lufenuron	12	24
Famoxadone	11	22
Pirimicarb	10	20
Bupirimate, kresoxim-methyl, methoxyfenozid, spinosad	9	18
Thiametoxam	8	16
Fipronil, pyraclostrobine, thiacloprid	7	14
Ametoctradin, azoxystrobin, cyhalothrin, cypermethrin, novaluron, pymetrozin	6	12
Fenamidone, iprovalicarb, mandipropamid, metalaxyl (metalaxyl-m), metrafenone, spinetoram	5	10
Difenoconazole, prochloraz, tebuconazole	4	8
Buprofezin, chlorantraniliprole, chlorothalonil, cyfluthrine, étoxazole, flubendiamide, hexythiazox, indoxacarb , methomyl (thiodicarb), oxycarboxine, triflumizole	3	6
Bifénazate, chlorfénapyr, diazinon, dimethoate, dinotefuran, fenpropidin, furalaxyl, mépanipyrim, picoxystrobin, trifloxystrobine	2	4
6-benzyladénine, acrinatrin, bénalaxyl , bifenthrine, bitertanol , carboxin , chloridazon, cyflufenamid, deltaméthrine, dicofol, fenarimol , fensulfothion-oxon, fenamiphos, fenvalerate , flufénoxuron. Forchlorfenuron, fosthiazate, isocarbofos. Méthiocarbe, myclobutanil, oxamyl, pyridabène , pyridalyl, quinalphos , spirotetramat ,tétradifon , thiabendazole, thiophanate-méthyl, triforine	1	2

PESTICIDE RESIDUES ON ROSES BY COUNTRY OF ORIGIN

Only 20 (40%) of all samples collected originated from EU countries (8 from Belgium). This table (Table 3) reflects the importance of flower exchanges in the world and how many flowers, mainly roses, are coming from third countries (Latin America and Africa) and sold into the EU market.

Table 3. Country of origin of rose samples, number of samples/country analysed, average number of active substances/sample, average of total amount of pesticide residues/sample (mg/kg) and number of active substances detected in samples

Country of origin (declared by florists)	Number of samples/country	Average number of active substances /sample (max-min)	Average of total amount of pesticide residues/sample (mg/kg)	Number of active substances detected in samples
Belgium	8	10.1	27.7	38
Colombia	2	19.0	31.8	24
Ecuador	9	14.8	18.8	60
Ethiopia	3	12.3	22.9	29
Germany	1	22.0	92.0	22
Israel	2	16.0	29.6	27
Netherlands	11	10.5	20.6	54
Kenya	9	15.6	26.5	48
(Sold in) Supermarkets	5	15.8	28.2	36
Total	50	13.6 (3-28)	26.03	97

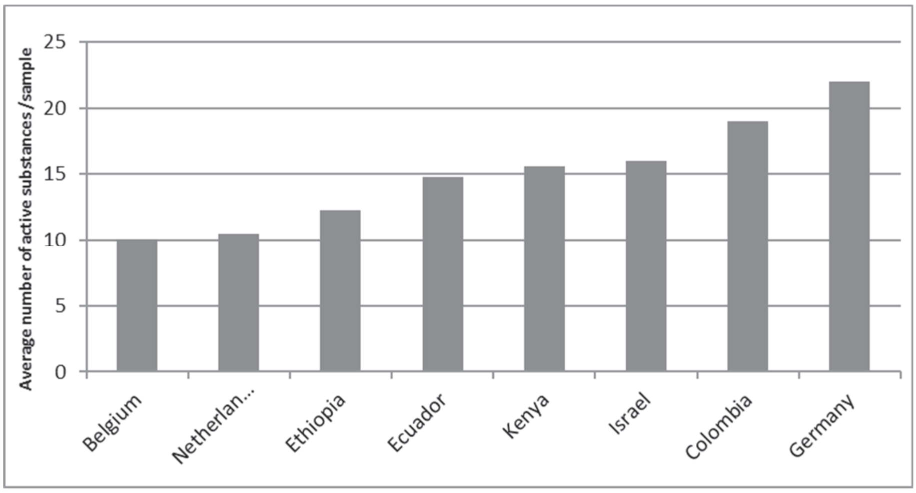

Figure 1 . Classification of countries based on the average number of pesticides detected/sample of roses

Samples from Belgium and The Netherlands have a lower average number of a.s./sample, but the amount of residues is about the same in all samples (20-30 mg/kg) except for the sample from Germany which is the worst case (22 a.s. with a total amount of 92 mg/kg) while some countries use a huge number of different pesticides on flowers (60 for Ecuador, 54 for The Netherlands). Anyway, those results indicate an intensive use of pesticides to prevent pests and diseases on flowers.

Risk assessment for florists

The risk is generated by combination of the "hazard" (mode of action; acute and chronic toxicity of a.s.) and "exposure" (concentration levels on flowers; routes of exposure: oral or dermal). The main route of exposure to plant protection products is the oral route, yet most exposures to operators, workers, bystanders and residents will be via dermal and / or inhalation routes. Therefore, it is necessary to assess the exposure for the operator (i.e. applicators, cropworkers, harvesters) for the different likely routes of exposure.

Classification of active substances according to their biological activity

Forty-seven % of detected active substances are insecticides and 46% of fungicides. Three active substances are growth regulators and one substance is an herbicide. Of the 97 detected active substances, most of the pesticides belong to the following chemical groups: organophosphates (9 a.s); pyrethroids (7 a.s); neonicotinoids (6 a.s); carbamates, triazole and strobilurins (5 a.s. each). Pesticides from those groups are known for their toxicological properties (action on the nervous system after exposure; acute toxicity).

Classification of active substances according to acute toxicity

Table 4. Classification of the active substances detected on oral acute toxicity values (***oral route of exposure***): number of a.s. for each Category (ILO classification) and number of samples where at least one a.s. belong to this Category

Categories	LD_{50} (mg/kg body weight)	Hazard wording	Number of active substances	Number of samples
1	[0-5]	Fatal if swallowed	1	1
2	]5-50]	Fatal if swallowed	7	17
3	]50-300]	Toxic if swallowed	12	36
4	]300-2000]	Harmful if swallowed	19	47
5	]2000-5000]	May be harmful if swallowed	58	50

For oral exposure, only a few a.s. (8 a.s.) belong to the most toxic groups but they are not rare: 18 samples from 50 (almost 40%) are contamined with such very toxic pesticide residues (the active substance belonging to Category 1 is the oxon-fensulfothion). 72% of samples contain active substances belonging to the Category 3 ''*Toxic if Swallowed*'' and 92% to the Category 4 ''*Harmful If Swallowed*''. All rose samples contain one or several active substances belonging to the less toxic Category 5.

Even if the oral contact is not the usual route of exposure, the risk still exists to have an exposure resulting from a “hand to mouth” contact. This accidental exposure can result from a lack of hygiene (florists do not wash their hands frequently and do not wear gloves systematically as observed in our survey of 25 professionals).

Table 5. Classification of the active substances detected on dermal acute toxicity values (***cutaneous route of exposure***): number of a.s. for each Category (ILO classification) and number of samples where at least one a.s. belong to this Category

Categories	LD_{50} (mg/kg body weight)	Hazard wording	Number of active substances	Number of samples
1	[0-50]	Fatal in contact with skin	2	2
2	]50-200]	Fatal in contact with skin	1	1
3	]200-1000]	Toxic in contact with skin	3	16
4	]1000-2000]	Harmful in contact with skin	5	38
5	]2000-5000]	May be harmful in contact with skin	86	50

Two active substances oxon-fensulfothion and isocarbofos belong to Category 1 *''Fatal in contact with skin*''. 76% of samples contain active substances belonging to the Category 3 *''Harmful in contact with skin*''. All rose samples contain one or several active substances belonging to the less toxic Category 5 *''May be harmful in contact with skin*''.

For dermal exposure, which is supposed to be the main route of exposure during handling, the risk can be considered as moderate when 94% of a.s. belong to the less toxic Categories, but all substances found on roses have more or less harmful effects in contact with skin.

Classification of active substances according to the EU Pesticides Database

As the florists handle the flowers every day in the course of their work, they are exposed to plant protection products like other “operators”. The “Acceptable Operator Exposure Level” (AOEL) is the reference value to consider for professionals exposed to pesticides. AOEL is defined in Regulation (EC) 1107/2009 as *"... the maximum amount of active substance to which the operator may be exposed without any adverse health effects*." AOEL values relate to the internal (absorbed) dose available for systemic distribution from any route of absorption and are expressed as internal levels (mg/kg bw/day). When the operator exposure remains below this limit, the risk for them is considered as “acceptable” (*Regulation (EC) 1107/2009*) (Table 6).

The active substances can also be classified on their hazard category according to the CLP regulation (for "Classification, Labelling and Packaging”) (*Regulation (EC) 1272/2008*) is a European Union regulation from 2008, which aligns the European Union system of classification, labelling and packaging of chemical substances and mixtures to the Globally Harmonised System (GHS). It is expected to facilitate global trade and the harmonised communication of hazard information of chemicals and to promote regulatory efficiency (Table 7).

AOEL values (mg/kg bw/d)	Number
[0.001-0.01 [	19
[0.01-0.1 [	43
[0.1-1[	18
>1	1
No AOEL*	16

Table 6. Number of active substances detected on the bouquets classified according to the AOEL values *(Source: EU Pesticides Database 2016, European Commission/DG HEALTH, Regulation (EC) 1107/2009)*

*Active substances which have no AOEL values; not assessed at European level.

Table 7. Number of active substances detected on the cut roses classified in each hazard category according to the CLP regulation *(Source: Regulation (EC) 1272/2008)*

Class	Category	Code (Hazard)	Number of a.s. in the category
Acute toxicity	Category 1	H310: Fatal in contact with skin	2
	Category 2	H300: Fatal if swallowed	10
		H330: Fatal if inhaled	6
	Category 3	H301: Toxic if swallowed	7
		H311: Toxic in contact with skin	2
		H331: Toxic if inhaled	10
	Category 4	H302: Harmful if swallowed	21
		H312: Harmful in contact with skin	7
		H332: Harmful if inhaled	3
Carcinogenicity	Category 2	H351:Suspected of causing cancer	13
Serious eye damage/ eye irritation	Category 1	H318: Causes serious eye damage	2
	Category 2	H319: Causes serious eye irritation	3
Germ cell mutagenicity	Category 1,1A or 1B	H340: May cause genetic defects	1
	Category 2	H341: Suspected of causing genetic defects	1
Reproductive toxicity	Category 1,1A or 1B	H360: May damage fertility or the unborn child.	3
	Category 2	H361: Suspected of damaging fertility or the unborn child.	11

	Additional category for effects on or via lactation	H362: May cause harm to breast-fed children	2
Sensiti-sation of the respiratory tract or the skin	Respiratory sensitisers category 1,1A or 1B	H334: May cause allergy or asthma symptoms or breathing difficulties if inhaled	1
	Skin sensitisers category 1,1A or 1B	H317: May cause an allergic skin reaction	21
Skin corrosion / irritation	Category 1,1A or 1B	H314: Causes severe skin burns and eye damage	1
	Category 2	H315: Causes skin irritation	6
Specific target organ toxicity (single exposure)	Category 3	H335: May cause respiratory irritation	4
Specific target organ toxicity (repeated exposure)	Category 1	H372: Causes damage to organs through prolonged or repeated exposure	2
	Category 2	H373: May cause damage to organs through prolonged or repeated exposure	7

CONCLUSION

The first conclusion after analysis of the residual deposits on roses is the high level of contamination of all samples whatever their origin: 97 active substances detected, i.e. an average of almost 14 active substances/sample and a total average pesticide load of 26.03 mg/kg per flower sample, while the accumulated total of all the residues was as much as 97.03 mg/kg for a single bouquet of 5 Belgian roses. This reflects the intensive use of pesticides on cut flowers in general.

Dodemorph (a fungicide) was not only the most frequently detected substance but also the active substance for which the highest maximum concentration (41.9 mg/kg) was measured on the rose samples analysed.

As the flowers are susceptible to pests and disease, they are regularly treated till the harvest time without any restriction on the pesticide use because there are no maximum residue limits (MRLs) for flowers, unlike other cultures. The high levels of pesticide residues in cut flowers are linked to high rates of pesticides but also to repeated sprayings during the growing season.

On the other hand, analyses of roses from Belgian or Dutch origins revealed an abnormal presence of active substances which are not authorised for use in the EU. 19 of the active substances detected on the 50 rose samples analysed are not authorised in the EU. The unauthorised active substances are more frequently detected in the Belgian samples. However, those results should be put into perspective as it was not possible to have a firm guarantee of the origin of the samples, as they were taken from the premises of the retailers rather than from the producers.

Even if pesticides are generally less toxic by cutaneous compared to the oral route, people who handle a large number of flowers every day are susceptible to be affected by various diseases after contact with skin and percutaneous absorption. All active substances identified on roses could affect the skin of florists if they do not wear protective equipment. Some active substances (acephate, methiocarb, monocrotophos, methomyl, deltamethrin etc.) on flowers have a direct effect on the nervous system and could cause accidental poisoning by transfer from the hands to the mouth. To better assess the risk, dislogeable residues and potential transfers are still to be investigate at the lab.

To reduce the exposure of florists to pesticide residues, solutions could be recommended: a better management of the pesticide used (IPM at the field or even organic flower production, a potential niche market); a stronger quality control of imported cut flowers. Finally, it could be interesting to set up a Maximum Residue Limit for flowers to decrease the risk for professionals and all other people in contact with flowers.

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