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Food safety in Vietnam:

Norway-Vietnam cooperation on pesticide residue analysis in vegetables

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Bioforsk in Norway and Northern Pesticide Control Centre (NPCC) in Vietnam have cooperated on implementing a validated multi-residue method for pesticide residue analysis at NPCC. We have also performed a pilot study on pesticide residues in two vegetable crops in three provinces in northern Vietnam.

INTRODUCTION

The interest for Southeast-Asian vegetables is increasing in Norway and EU, and creates new market opportunities for vegetable and fruit producers and processors in Vietnam. However, Vietnamese vegetables are today predominantly exported to markets where importers do not demand pesticide residue testing of the produce (e.g. Russia and China). In Vietnam, vegetable farmers apply pesticides intensively, and often at higher doses than recommended (Pham 2010). Pesticide use in Vietnam increased from 15.000 to 76.000 tons from 1991 to 2005. More than 7000 incidents of food poisoning from pesticide residues were reported in 2002 (Nguyen 2003). Little is known about the extent and amounts of pesticide residues in fruit and vegetables produced in Vietnam.

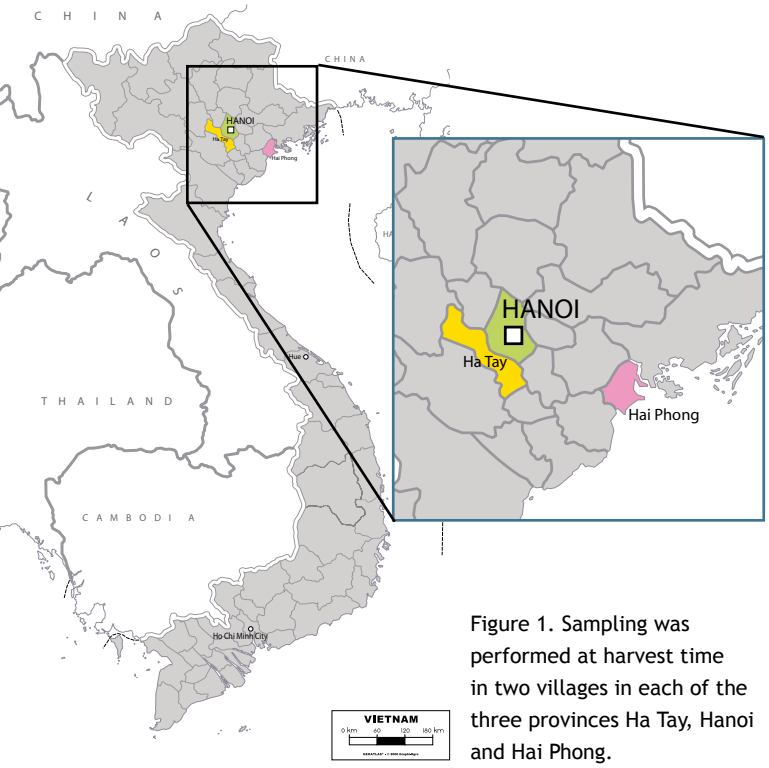

Figure 1. Sampling was performed at harvest time in two villages in each of the three provinces Ha Tay, Hanoi and Hai Phong.

In Vietnam, pesticide residue analysis is performed by two laboratories under the Ministry of Agriculture, namely the Northern Pesticide Control Centre (NPCC) in Hanoi, and the Southern Pesticide Control Centre (SPCC) in Ho Chi Minh City. NPCC has been accredited according to ISO 17025 for the testing of pesticide residues in crops since 2005. NPCC performs pesticide residue testing of vegetables and fruits from markets and various vendors in Hanoi and surrounding provinces. This testing is not performed regularly, and a national monitoring programme for pesticide residues in crops needs to be established in Vietnam.

In 2005 Bioforsk started cooperation with NPCC with the objective of implementing multi-pesticide residue methods at NPCC. The pesticide laboratory at Bioforsk is accredited according to the ISO 17025 standard and performs all monitoring of pesticide residues in domestic and imported fruit and vegetables in Norway, in cooperation with the Norwegian Food Safety Authority. NPCC wanted to implement analytical methods that could be used to analyse a wide range of pesticides in fruit and vegetables during one run; i.e. multi-residue methods.

Implementation of analytical multi-residue method for pesticide residue analysis

A Bioforsk method for the analysis of pesticides in vegetables was used as a template for the preparation of a method protocol for the analysis of 27 pesticides in fruit and vegetables at NPCC. The pesticides selected included organochlorines, organophosphates and pyrethroids that are used in Vietnam. The NPCC method protocol is not completely identical with the Bioforsk method, as Bioforsk use GC-MS as instrument for the analysis, whereas NPCC use GC-ECD/FPD; i.e. the analytical detectors are different. There are also some modifications with regard to samples preparation due to different equipment.

During the project period, mutual workshops both in Norway and Vietnam were executed in order to discuss storage of samples, sample preparation, use of internal standards, preparation of pesticide standards, GC-MS analysis, LC-MS/MS analysis, validation plans and validation reports. Hands-on training on GC and GC-MS was performed at NPCC and on LC-MS/MS at Bioforsk.

The new multi-residue method was validated by the staff at NPCC according to a validation plan. The purpose of the validation was to ascertain that the method met predetermined criteria for various important parameters such as recovery, quantification limits and repeatability. The validation at NPCC was documented in a validation report and showed that the staff could use the method appropriately and that the method was fit for purpose. All documents were prepared both in Vietnamese and English.

Figure 2. Sampling of long beans and leaf mustard and interviews of farmers by NPCC personnel.

Analysis of vegetables from three provinces in the north of Vietnam

As an extended part of the validation, an inter-laboratory comparison with analysis of vegetable samples from three provinces; Ha Tay, Hanoi and Hai Phong was performed. The study involved two crops: long beans (*Vigna sesquipedalis*) and leaf mustard (*Brassica juncea*). The crops were sampled at harvest time by personnel from NPCC and 24 samples of each crop were sampled. The samples were analysed for pesticide residues at NPCC using the new multi-residue method. The homogenized vegetable samples were then shipped on dry ice to Bioforsk for pesticide residue analysis with the Bioforsk GC-MS multi-residue method. The results from the analyses are summarized in Table 1.

Figure 3. Vegetable sample preparation at NPCC with the new method: homogenisation, extraction, centrifugation and analysis.

Table 1. Pesticide residues (mg/kg) detected in vegetable samples from three provinces (Prov.) in northern Vietnam, analysed with GC-ECD/FPD at NPCC and with GC-MS at Bioforsk. ND = Not detected. Red results= pesticides that are not included in the other laboratory's method and therefore not possible to detect there. Blue results= pesticides that should have been found at both laboratories, even though the analytical instruments were different.
The NPCC results have been colour coded:

Red box = results exceeding both Norwegian and Vietnamese MRLs
Yellow box = results exceeding Norwegian MRLs only
Orange box = results exceeding Vietnamese MRLs only

Table 1a. Results from long bean samples

Prov.	No.	NPCC result	mg/kg	Bioforsk result	mg/kg
Hai Phong	1	ND		ND	
	2	ND		ND	
	3	ND		ND	
	4	ND		ND	
	5	fipronil	0.02	ND	
	6	fipronil	0.02	ND	
	7	fipronil	0.02		
		cypermethrin	0.05	cypermethrin	0.05
	8	fipronil	0.02		
		cypermethrin	0.12	cypermethrin	0.07
Hanoi	21	cypermethrin	0.21	cypermethrin	0.11
				trichlorfon	0.17
	22	cypermethrin	0.25	cypermethrin	0.15
				trichlorfon	0.12
	23	cypermethrin	0.37	cypermethrin	0.33
		trichlorfon	0.06	trichlorfon	0.14
	24	cypermethrin	0.44	cypermethrin	0.28
				trichlorfon	0.20
	25	endosulfan alfa	0.03	ND	
		endosulfan beta	0.04		
	26	endosulfan alfa	0.03	ND	
		endosulfan beta	0.04		
	27	trichlorfon	0.14	trichlorfon	0.65
	28	cypermethrin	0.11	cypermethrin	0.10
		trichlorfon	0.17	trichlorfon	1.00
Ha Tay	41	ND		ND	
	42	ND		ND	
	43	chloramfenapyr	0.04	ND	
	44	ND		ND	
	45	ND		ND	
	46	ND		ND	
	47	permethrin	0.06	permethrin	0.12
		cypermethrin	0.14	cypermethrin	0.17
		acephate	0.15		
	48	permethrin	0.06	permethrin	0.11
		cypermethrin	0.20	cypermethrin	0.15
		acephate	0.15	chlorpyrifos	0.05

Table 1b. Results from leaf mustard samples

Prov.	No.	NPCC result	mg/kg	Bioforsk result	mg/kg
Hai Phong	9	cypermethrin	0.59	cypermethrin	1.30
		profenofos	4.11	profenofos	16.00
		hexaconazol	0.08		
	10	cypermethrin	1.11	cypermethrin	1.70
		profenofos	4.15	profenofos	18.20
	11	lambdacyhalothrin	0.03	lambdacyhalothrin	0.06
		profenofos	0.26	profenofos	0.30
	12	lambdacyhalothrin	0.05	lambdacyhalothrin	0.06
		profenofos	0.43	profenofos	0.26
	13	cypermethrin	0.98	cypermethrin	1.50
		profenofos	6.27	profenofos	18.50
	14	cypermethrin	0.63	cypermethrin	1.70
		profenofos	6.05	profenofos	12.40
	15	profenofos	10.42	profenofos	16.00
	16	profenofos	14.22	profenofos	22.40
				alfacypermethrin	0.26
Hanoi	29	cypermethrin	0.18	cypermethrin	0.30
		chlorothalonil	0.06		
	30	cypermethrin	0.33	cypermethrin	0.39
		chlorothalonil	0.07		
	31	chlorothalonil	0.06	ND	
	32	chlorothalonil	0.07	ND	
	33	ND		ND	
	34	chlorothalonil	0.03	ND	
	35	cypermethrin	0.09	cypermethrin	0.12
		chlorothalonil	0.03	chlorpyrifos	0.33
				quinalfos	0.07
	36	chlorothalonil	0.06	cypermethrin	0.06
				chlorpyrifos	0.26
				quinalfos	0.05
Ha Tay	49	permethrin	0.08	permethrin	0.11
	50	permethrin	0.10	permethrin	0.16
	51	permethrin	2.23	permethrin	3.40
	52	permethrin	1.14	permethrin	1.40
	53	lambdacyhalothrin	0.26	lambdacyhalothrin	0.23
		cypermethrin	0.87	cypermethrin	0.58
		chlorothalonil	0.12	metalaxyl	1.10
				permethrin	0.08
	54	cypermethrin	0.06	cypermethrin	0.05
		chlorothalonil	0.06	permethrin	0.08
		permethrin	0.06	metalaxyl	0.04
	55	lambdacyhalothrin	0.06	lambdacyhalothrin	0.05
	56	lambdacyhalothrin	0.06	lambdacyhalothrin	0.06

Table 1 reveals good correlation between the Bioforsk results and the NPCC results with an 83% match of pesticides detected. Some pesticides (shown in red) can only be found at one of the laboratories because the monitoring programme was different. Some pesticides are found by only one of the laboratories due to lower limits of quantification with the instrument used at that laboratory. Chlorothalonil was not detected during the Bioforsk analysis, probably due to degradation of the compound before delivery of the homogenised samples at Bioforsk. Chlorothalonil is known to be rapidly degraded in homogenised samples. This is probably also the case for fipronil, which was found only in the NPCC samples. Some pesticides (in blue) should have been found in the samples at both laboratories. 8 pesticide residues were not found by NPCC, and 4 pesticide residues were not detected by Bioforsk. 6 out of the 8 residues not found by NPCC were due to the pesticides trichlorfon and chlorpyrifos. Trichlorfon is problematic to detect with the method at NPCC, due to a high limit of quantification (0.20 mg/kg). However, of the "blue" findings, only the hexaconazole residue (sample no.9) represented a concentration level exceeding Norwegian MRL.

The correlation in pesticide amounts found at Bioforsk and NPCC was good, but a few of the pesticides were reported at lower concentrations at NPCC than at Bioforsk. This can be observed for profenofos and trichlorfon and could be due to improper calibration. The deviations in the results were discussed and possible solutions suggested. But overall, we were very satisfied with the results from this inter-laboratory study. Due to the uncertainty of the methods it is not possible to get exactly the same results when samples are analysed at different laboratories, with different personnel and different analytical instruments. In EU, a default uncertainty figure of 50% in general covers the inter-laboratory variability between European laboratories and is recommended to be used by regulatory authorities in cases of MRL-exceedances (SANCO 2009). If an uncertainty figure of 50% is applied to the NPCC and Bioforsk pesticide residue amounts in Table 1, all but two results (profenofos in samples no. 9 and 10) are within the same range.

A comment on MRL-exceedances in the vegetable samples

The analysis results from NPCC showed that 15 of 24 long bean samples contained pesticides, whereas 23 of 24 leaf mustard samples contained pesticides. Norwegian maximum residue levels (MRLs) were violated in 17% of the long bean samples and in 88% of the leaf mustard samples. The Vietnamese MRLs are in general higher than the MRLs used in Norway and EU. As a consequence, Vietnamese MRLs were violated in 17% of the long bean samples and in only 29% of the leaf mustard samples.

Due to the high content of pesticide residues, most of the leaf mustard samples would have been rejected from the Norwegian market, if they had been exported to Norway. In general, the Norwegian monitoring programme has revealed that there is a reason for concern regarding pesticide residues in Vietnamese commodities. In 2008, five fruit and vegetable samples from Vietnam were analysed by Bioforsk, and all of them contained pesticide residues. Four out of the five samples had levels exceeding MRL. The pesticides detected at highest level were cypermethrin, esfenvalerat, hexaconazol and clorothalonil (Mattilsynet 2009). Very few samples from Vietnam were analysed in 2009 and 2010. An extended study on pesticide residues from commodities from Asia will be performed by the Norwegian Food Safety Authority and Bioforsk in 2012.

We suspect that many of the violations of MRLs seen in our study are related to insufficient pre-harvest intervals, particularly in leaf mustard, a crop that is grown intensively (harvested 30-40 days after germination). Current regulations in Vietnam set the pre-harvest interval to 7 days for most pesticides, whereas in Norway, the interval is 14 days. Leafy vegetables are particularly prone to violations in pesticide residue levels, as the entire plant is sprayed and consumed. In Vietnam, leafy vegetables are used daily in soups ("pho").

Application of the multi-residue method at NPCC

The multi-residue method is now in regular use at NPCC. NPCC applied the method in a national study (Min. ag. 2007) on pesticide residues in vegetables (long bean, leaf mustard and water spinach), fruit (orange and grape), nuts and tea from 5 different provinces in Vietnam. The study revealed that 14% of the leaf mustard samples had pesticide levels exceeding the (Vietnamese) MRLs, whereas 8% of the long beans, 7% of the water spinach samples and 32% of the grape samples exceeded the MRLs. Of the 5 provinces, vegetables from Hai Phong and Hanoi contained the highest pesticide amounts.

CONCLUSION

Bioforsk and Northern Pesticide Control Centre (NPCC) in Hanoi, have cooperated on implementing a validated multi-residue method for analysis of 27 pesticides in vegetables at NPCC. The method can also be applied to the analysis of fruit. An inter-laboratory study between Bioforsk and NPCC showed a good match between the analysis results at Bioforsk and at NPCC. Analysis of leaf mustard from 3 provinces in northern Vietnam revealed a high content of pesticide residues in the samples. Vietnamese MRLs were violated in 29% of the leaf mustard samples, whereas the Norwegian MRLs were violated in 88% of the leaf mustard samples. A combination of higher MRLs and shorter PHIs in Vietnam than in Norway, increase the risk of high levels of pesticide residues in the Vietnamese produce and increase the risk of being rejected from the European market. Analytical competence, good instrumentation and properly validated analytical methods are important both in Norway and Vietnam in order to be able to monitor pesticide residues in domestic and imported fruit and vegetables and ensure food safety.

Acknowledgement

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