INTERGOVERNMENTAL GROUP ON TEA

INTERSESSIONAL MEETING

Rome, Italy

5-6 May 2014

Working Group on MAXIMUM RESIDUE LEVELS IN BREW[1]

[1]Submitted by China and India in their capacity as Co-chair of this Working Group.

Introduction

The pesticide residue problem remains important in the consumption and trade of tea in the world. The pesticide maximum residue levels (MRLs) in tea issued by regulatory authorities, especially from the tea importing countries, become more stringent in recent years. However, the concentration of pesticide in tea brew which is directly consumed should be considered as realistic value that establishment of MRLs in tea should be based on. Thus, it is critical to conduct investigations on pesticide residues in tea brew for establishment of MRLs in tea. Since 2008, the Working Group on MRLs in Tea Brew in IGG on Tea has been paying attention to the transfer of pesticide residue from dry tea to tea brew and deliberating on how to establish the MRLs in tea more scientifically and rationally. One clear indication is that the water soluble pesticides have been a matter of concerns. A number of steps have been taken in the past years.

Activities of the Working Group

The activities conducted as the following aspects:

1. The verification of the status of water-soluble pesticides in the safety assessment in Tea According to the analysis results on nearly 20,000 tea samples from different categories (green, black, oolong and dark tea) from 18 tea producing provinces in China and different tea products (instant tea and Ready-To-Drink tea) by using the UPLC-MS/MS, a high detection rate on imidacloprid, acetamiprid and bifenthrin was discovered. The actual intake amounts of imidacloprid and acetamiprid via tea brew, due to the high water solubility of the neonicotinoid pesticides, could be much higher than the non-polar pesticide bifenthrin. During the past 3 decades, certain amounts of literatures on transferring rate of pesticide residues to tea brew were published, including the results of imidacloprid, acetamiprid, dimethoate, fenpropathrin, deltamethrin, cypermethrin, L-cyhalothrin, hexaconazole, propiconazole and bifenthrin (shown in Table 1). It is quite obvious that the amounts of pesticide residues in dry tea transferred to tea brew were related to water solubility and octanol/water participation coefficient. More attention should be paid therefore, to water soluble pesticides, like imidacloprid, acetamiprid etc., in tea and tea brew rather than non-polar pesticides.

Table 1 Physical-chemical properties and transferring rate of pesticides from dry tea to tea brew

Pesticide	Water solubility (mg/L, 20℃)	Log Kow (pH7, 20℃)	Harry's law constant (Pa m^3 mol^{-1}, 25℃)	Transferring rate (%)	Author
Bifenthrin	0.001	6.6	7.74×10^{-5}	4.2-4.6	Chen ZM et al (1986)
				4.58	Barooah et al (2011)
				1.5-14	Tewary et al (2005)
Imidacloprid	610	0.57	1.7×10^{-10}	29.2-42.0	Gupta et al (2006)
				38.6-43.9	Gupta et al (2006)
				3.67-10.69	Sanyal et al (2006)
				84.9	Wang et al (2013)
				63.1(green tea) 62.2(black tea)	Hou et al (2013)
				28	Chen ZM (2010)
Acetamiprid	2950	0.8	5.30×10^{-8}	47.1-53.3	Gupta et al (2009)
				68-85	Chen ZM (2010)
				78.3(green tea) 80.6(black tea)	Hou et al (2013)
Dimethoate	39800	0.704	1.42×10^{-6}	97.5-98.3	Chen ZM et al (1991)
Hexaconazol	17	3.9	3.33×10^{-4}	1.14	Barooah et al (2011)
Propiconazol	100	3.72	9.2×10^{-5}	13.15	Barooah et al (2011)
Fenpropathrin	0.0141	6	1.8×10^{-4}	0.19	Barooah et al (2011)
Cypermethrin	0.004	6.6	4.2×10^{-7}	0.51	Barooah et al (2011)
				1.4-2.1	Chen ZM et al (1986)
				1.5-14	Tewary et al(2005)
L-cyhalothrin	0.005	7	2×10^{-2}	1.11	Barooah et al (2011)
				1.94	Zhang et al (2013)
Fenpyroximate	0.023	5.01	1.3×10^{-6}	3.1	Barooah et al (2014)
Thiacloprid	185	0.73	1.1×10^{-10}	49.7	Wang et al (2013)
Thiamethoxam	4100	-0.13	4.6×10^{-15}	93.3	Zhang et al (2013)
Indoxacarb	0.02	4.65	6.0 × 10-5	6.2	Zhang et al (2013)

In 2013, several papers reported the neurotoxicity of neonicotinoid pesticides. EFSA (European Food Safety Authority) concluded in January that three neonicotinoids – thiamethoxam, clothianidin and imidacloprid – posed an unacceptable risk to bees. These three pesticides will be banned from use for

two years on flowering crops such as corn, oilseed rape and sunflowers, upon which bees feed. In December of 2013, the EFSA **Panel on Plant Protection Products and their Residues (PPR)** reported a paper entitled "Scientific Opinion on the Developmental Neurotoxicity Potential of Acetamiprid and Imidacloprid" (EFSA Journal, 2013, 11(12) 3471-3522). In this paper, it reported that the evidence of effects on offspring from developmental neurotoxicity studies for imidacloprid in rats, carried out within the European Union (EU) and Environmental Protection Agency (EPA) assessment frameworks, reported decreased pup body weights, reduced motor activity level and changes in dimensions of brain structures. The developmental neurotoxicity study for acetamiprid, carried out within the US-EPA assessment framework, showed decreased pup body weights, reduced pup pre-weaning survival and decreased maximum auditory startle response. The panel therefore concluded that both compounds may affect neuronal development and function. It was considered that current ARfDs may not be protective enough for the possible developmental neurotoxocity of acetamiprid and imidacloprid. The developmental neurotoxicity potential of acetamiprid and imidacloprid has been considered by the EU and USA EPA. It is worthy to point out that these two pesticides should be especially drawn attention to during application in tea industry due to their high water solubility (42-60% extracted in tea brew) during the tea brewing process. Acetamiprid and imidacloprid are applied as major pest control agrochemicals in tea producing countries to protect the plantations from severe attacks of sucking pests responsible for huge crop loss.

2. The substitution action of water-soluble pesticides in China

According to the information mentioned above, we have been conducting an action to select some pesticides with low water solubility to substitute the imidacloprid, acetamiprid, and dimethoate since 2012. The results of bioassay and residue dynamic experiment under field conditions were conducted in 12 tea producing provinces in China indicated that we have successfully selected chlorfenapyr, indoxacarb and tolfenpyrad as substitutions of imidacloprid, acetamiprid and dimethoate. Around 24,000 hactares of tea garden were applied with chlorfenapyr, indoxacarb and tolfenpyrad to control the most serious tea pests in Chinese tea industry including the tea leafhopper (*Empoasca vitis*), tea geometrid (*Ecrotropis obliqua*) and tea thrip (*Dendrothrips minowai*), satisfied results were obtained.

3. The registration and MRL situation of substituting pesticides

The supervised field trials as par Good Agricultural Practice (GAP) for chlorfenapyr, indoxacarb and tolfenpyrad have been conducted and completed in China during 2011-2013. The field trials for chlorfenapyr, indoxacarb and tolfenpyrad were conducted in four locations in 2010-2012, 2011-2012 and 2012-2013 respectively. The results are summarized in Table 2.

Table 2 Results of the field trials for three pesticides in tea plants in China

Pesticides	HL_{50} on fresh tea leaves(day)	Processing factor (%)	Brew factor (%)	Water solubility (mg/L)	Recommended MRL(mg/kg)	PHI (day)
Chlorfenapyr	5.6	54	0.15	0.12	20	7
Indoxacarb	2.3	34.5	6.2	0.02	3(China), 5(JMPR)	10
Tolfenpyrad	3.6	41.6	0.2	0.087	20	5

Chlorfenapyr and indoxacarb were registered in China in 2012 and 2013 respectively. We have completed the bioassay and field trials of tolfenpyrad in 2013 and will apply the registration in 2014.

The MRL of chlorfenapyr is issued at 50 and 40 mg/kg in EU and Japan respectively, and chlorfenapyr was almost unextractable from dry tea into tea brew due to its very low water solubility (almost insoluble in water). Chlorfenapyr poses relatively low risk to consumers from the viewpoint of extremely low brewing factor.

Indoxacarb is a pesticide manufactured by DuPont. The MRL of indoxacarb in tea was set at 3 mg/kg in China in 2013. In the same year we applied for establishing MRL of indoxacarb in tea in Codex, and submitted the information of eight experiments conducted in China. The MRL of indoxacarb was approved to be set at 5 mg/kg in JMPR meeting in October, 2013 and will be discussed further as Codex MRL in tea in JMPR meeting in May, 2014. The default MRL in tea issued in EU was 0.05 mg/kg, and the uniform MRL in tea issued in Japan was 0.01 mg/kg.

Tolfenpyrad is introduced by Japan, and the MRL in tea was set at 20 mg/kg in Japan. We just finished the bioassay and field trials in 2013 and will apply for the registration in tea in China in this year. The recommended MRL in tea will be at 20 mg/kg.

Action Plan

1. Establishment of the methodology in determining the pesticide residue in tea brew, especially the residues of some water-soluble pesticides

   The methodology for determination of the water-soluble pesticides in tea brew is somewhat different from the methodology in determining the residue of non-polar pesticides in dry leaves. Establish different methods for the determination of hydrophilic and lipophilic pesticide residues in black tea, green tea and tea brew. Before establishing the methodology, a ring test covering the tea producing and tea consuming countries will be organized.

2. Determination of the concentration of various pesticides in tea infusion

   Select 6 pesticides used in tea industry according to their water solubility [two pesticides with low water solubility (<1mg/L), two with median water solubility (1-<100 mg/L) and others with high water solubility (>100 mg/L)], determine the concentration of pesticides in tea infusion, and compare the difference between the residue level with different water solubility in tea.

   Based on the comparison above, the Working Group recommend the major tea producing countries to arrange a plan to analyze the residue levels of various pesticides in representative tea, tea product samples (instant tea and Ready-To-Drink tea) and tea infusion.

3. Risk assessment

   According to the results, conduct the risk assessment on the major pesticides in tea brew with following equation:

$$IESTI = \frac{LP \times HR\text{-}P \times ER}{BW\ (kg)}$$

   LP: 97.5% point, the highest amount of tea used for drinking per day (Green tea, Black tea, Oolong tea 13 g/d; Dark tea 26 g/d);
   HR-P: Highest residue in tea under recommended harvest interval after manufacture (mg/kg);
   ER: Maximum extracting rate of pesticide during tea brewing (%);
   BW: Average body weight of adult (60 kg in China)

4. Establishment of a procedure to set the pesticide MRLs in dry tea based on the brewing factors of pesticide.

In 2010, a policy document entitled "Assessment of MRLs for pesticide residues in tea" was prepared by the Working Group on MRLs in Tea Brew, and submitted to the 43rd CCPR Meeting. In paragraph 160 of the CCPR 43rd Report, the conclusion was recorded as follows:

"160. The Committee emphasized the need for processing studies to refine the dietary risk assessment of tea and supported the comments of the FAO JMPR Secretariat."

In 2011, another Document entitled *Assessment of MRLs for Pesticide in Tea* was prepared by the Working group on MRLs in Tea Brew and submitted to the 44th Session of the CCPR (April, 2012,Shanghai, China) via China Delegation and India Delegation. It was adopted as agenda item 12b and CRD 10.29 and disseminated to the delegates of 44th Session of the CCPR. The recommendation of taking into account the pesticide residue in tea infusion should be considered in the establishment of MRLs in tea was adopted by the CCPR Committee. It was concluded in the Paragraph 178 of the 44th CCPR Report.

"178. The Committee supported the current procedure of JMPR in the establishment of MRLs for pesticides in tea and encouraged countries to submit relevant data/information on brewing factors and standard methods to JMPR for consideration in estimation of MRLs for pesticides in tea."

As discussed and agreed at the 43rd and 44th Session of CCPR, the MRLs for pesticides in tea would be set on the dried tea but the residues in the tea brew should be considered in the establishment of MRLs on dried tea. It is therefore necessary to set up a recommended procedure on the setting of MRLs in dry tea based on the brewing factors of pesticides in tea brew. This recommended procedure would be first discussed at the next session of the IGG on Tea and then submitted to the CCPR and JMPR for consideration.

A recommended document on the establishment of indoxacarb MRL in tea attached with information of 8 experiments conducted in China was submitted to JMPR by China Delegation in September, 2013. It was recommended to set at 3 mg/kg based on the brewing rate of 6.2% of indoxacarb pesticide in tea brew, and this recommendation was approved to set at 5 mg/kg.

A recommended document on the establishment of fenpyroximate MRL in tea based on supervised field experiments conducted in India was also submitted for JMPR review in 2013. It was recommended to set MRL at 5 mg/kg based on the brew factor on 0.031 and transferring rate of 3.1 % of fenpyroximate residues in tea brew.

Two other recommended documents on the establishment of imidacloprid and propiconazole MRLs in tea based on supervised field experiments conducted in India were also submitted for JMPR review in December 2012. MRLs were recommended after risk assessment based on brewing factors of 0.7 and 0.13, respectively for the two pesticides.

In this IGG Working Group Meeting, a draft of the procedure on how to set up a MRL of pesticide in dry tea based on the brewing factor of pesticide in brewing process will be discussed for submission to JMPR, CCPR.

5. Re-identify the pesticides used in tea-producing countries according to the efficacy on the major tea insect pests, disease, and the current residue levels in market tea samples and the current MRL of pesticides in major tea importing countries, then make a revision on the pesticide list in tea production and classify pesticides into recommended, replaceable and withdrawn categories. It is recommended to pay more attention to those pesticides with high water solubility, such as imidacloprid, acetamiprid, triazophos, dimethoate, dichlovos and methomyl. It is recommended to send a pesticide list including the recommended, replaceable and withdrawn categories from tea producing counties and will be discussed in the IGG meeting 2015.

6. Collaboration on establishment of database and prediction model of transferring rates vs. pesticide chemical physical properties.

   It is recommended that the investigations of transferring rate of pesticide residues during infusion processing collaborated with countries and laboratories should be accelerated when the determination methodologies on pesticides in tea and tea brew are established. Collect data on tea brew as well as leaf to build the database of transferring rates vs. pesticide chemical physical properties. Identify the relationship between physical chemical properties and transfer rate of pesticide rate from dry tea to tea brew, ascertain the pesticide character affect the transfer rate most, and build a model for predicting the transferring rate of new introduced pesticides applied in tea in future.

Ring test: As per the decision taken at the last intersessional meeting held in Washington, DC, a ring test is being organized for both black and green tea. The field treated black tea samples have already been prepared by India and the green tea samples are being prepared in China. For the black tea ring test, three pesticides- acetamiprid, fenpyroximate and flubendiamide have been used and the method based on LC-MS/MS selected. For the green tea ring test, three pesticides- acetamiprid, tolfenpyrad and carbendazim have been recommended and the method based on the LC-MS/MS, UPLC-MS/MS method. Background work carried out since the last meeting both in India and China has indicated the difficulty encountered in the analysis of water soluble pesticides by conventional methods could be addressed only by either LC-MS/MS or UPLC-MS/MS based methods. Hence the ring test as planned earlier got delayed and now to be carried out in April-May 2014 using advance test methods. Details will be discussed in the intersession al meeting in Rome.