Residues of Thiamethoxam and Acetamaprid, Two Neonicotinoid Insecticides, in/on Okra Fruits (*Abelmoschus esculentus* L)

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Synthetic pesticides are important component of modern agriculture. About 13-14% of total pesticide used in India, are consumed in vegetable crops only. The produce is harvested at short intervals and consumed fresh in many cases. The survey of market samples show high level of pesticides (Agnihotri, 1999) in vegetables. Therefore target specific and extremely low dose pesticides are being introduced specially for vegetables. Okra (*Abelmoschus esculentus* L) is an annual vegetable crop, which is attacked by a number of insect pests like shoot/fruit borer, jassids and aphids resulting in the reduction of yield to an extent of 69% and quality of fruits (Dewan et al. 1967). To protect the crop from such pests various pesticides from different class are applied (Rawat and Sahu, 1973; Patel et al. 2001). Neonicotinoids (Yamamoto, 1996) represents a novel and distinct chemical class of insecticides with remarkable chemical and biological properties with low application rates (Yamamoto and Casida, 1999). The effectiveness of imidacloprid, a chloroneonicotinoid insecticide, has been reported on okra, brinjal and chilli fruits against various insect pests (Mote et al. 1994; Jarande and Dethe, 1994). Acetamaprid ((E)-N1-{[6-chloro-3-pyridyl)methyl]}-N2-cyano-N3-methyacetamidin; I, Figure 1) has been introduced by Nippon Soda with common structural features of first generation neonicotinoids (Maenfisch et al., 1999). Thiamethoxam ([E]-3-(2-chloro-1,3-thiazol-5-ylmethyl)-5-methyl-1,3,5-oxadiazinan-4-ylidene (nitro) amine; 2, Figure 1) is a novel neonicotinoid belonging to sub class of thiacotinyl compounds and it represents the first example of second generation neonicotinoids with a unique structure and outstanding insecticidal activity (Maenfisch et al., 1999a) introduced by Novartis. Both are systemic insecticides for soil and foliar applications and control a variety of pests such as aphids, whiteflies, thrips, beetles, leaf hopper, bugs and borers in fruiting, corn, tuberous vegetables, cotton and fruits (Roberts and Hutson, 1999b). GC method has been used for the determination of acetamaprid and its degradation product in soil by Tokieda et al, 1999c. HPLC method has also been developed for the detection of thiamethoxam from water, soil and vegetables (Singh et al, 2004; Karmakar et al, 2005). In this study we report the dissipation and residues of these two neonicotinoids viz., thiamethoxam and acetamaprid by HPLC in okra fruits following foliar applications at different stages of crop growth.

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Figure 1. Chemical structures of acetamiprid (1) and thiamethoxam (2)

MATERIALS AND METHODS

Technical grade thiamethoxam was obtained from M/s Syngenta India Ltd. Acetamiprid was extracted from formulation and purified by column chromatography over silica gel. Finally it was crystallized from benzene before use. All the solvents were analytical grade and glass distilled. Anhydrous sodium sulphate (AR grade) was used as a drying agent for different samples. HPLC grade acetonitrile and water were used for HPLC analysis. Formulation Actara \(^R\) (25% WG) and Pride \(^R\) (20% SP) for thiamethoxam and acetamiprid respectively were purchased from market.

Field experiment was conducted at IARI, New Delhi, during August to November, 2003. Okra (variety Pusa kranti) was grown in plots of 5 x 8 m using RBD replicated 4 times with 50 cm spacing between the rows and 30 cm between plants. Two sets of experiments were conducted. In the first experiment foliar application of thiamethoxam and acetamiprid at recommended doses of 140g and 75g a.i. ha\(^{-1}\) respectively was given in separate replicated plots at the time of flowering. Okra fruits were harvested and analysed for the insecticide residues from these plots. In the second experiment for the waiting period studies two sprays of each pesticide were given in separate replicated plots. First spray was given at the time of flowering and a second spray of both the insecticides (thiamethoxam and acetamiprid) at recommended doses was given at the time of fruiting (in the month of November) in separate respective plots. From this experiment fruit samples were collected at 0, 1, 3, 5 and 7 days for respective insecticide analysis.

The okra sample (50g) was blended in a blender with acetone (100 ml) and contents were filtered through Buchner funnel using suction. The extraction was done twice more with the same solvent (50 ml each time) and filtered in the same way. The combined filtrate was then concentrated by evaporating the solvent on a rotary vacuum evaporator at 35-40 °C to 2-3ml. The extract concentrate was diluted with 150ml of 10% NaCl (aq.) solutions and partitioned with hexane (25ml), which was discarded. The aqueous phase was extracted thrice with dichloromethane (50+50+30 ml). The organic phase was dried over anhydrous