Type of the Paper (Article)

THIAMETHOXAM IN SOIL AND WATER IN PAPAYA CULTIVATION (*Carica papaya Linnaeus*) ASSOCIATED WITH WATERMELON CULTIVATION (*Citrullus lanatus*) IN MEXICO

Megchún-García Juan Valente[1], Castañeda-Chavez María del Refugio[2]*, Rodríguez-Lagunes Daniel Arturo[1], Murguía-González Joaquín[1], Lango-Reynoso Fabiola[2] and Leyva-Ovalle Otto Raúl[1]

[1] Facultad de Ciencias Biologicas y Agropecuarias, Universidad Veracruzana [1]; juanmg_3@hotmail.com
[2] Tecnológico Nacional de México/Instituto Tecnológico de Boca del Río [2]; Email: mariacastaneda@itboca.edu.mx
* Correspondence: mariacastaneda@itboca.edu.mx

Abstract: Thiamethoxam is a neonicotinoid, with systemic and contact action, used in Mexico for the care of different traditional fruit crops, mainly in the cultivation of papaya. International organizations such as the EPA (Environmental Protection Agency) and the EFSA (European Food Safety Authority) establish maximum permissible limits of 0.4 mg/kg and 0.05 mg/kg of thiamethoxam in papaya. The aim of this study was to determine the presence and concentration of thiamethoxam in soil and water in papaya crops associated with watermelon from the central area of Veracruz, Mexico. A diagnosis of thiamethoxam management in different soil types of the region was made; through an experimental plot where soil and water samples were taken in the different stages of papaya cultivation associated with watermelon, using HPLC-UV equipment for its determination. The design was random blocks with six repetitions and the software used for data analysis was the Statistica 2007 program. Thiamethoxam was concentrated in amounts of ≥0.40 mg/L in 79% of the samples in water and ≥0.55 mg/kg in 75% of the samples in soil. The highest values of thiamethoxam in soil were in the stage of watermelon culture with 0.4 mg/kg and in the soil preparation of the papaya crop with concentrations of 0.8 mg/kg. Whereas irrigation water from the watermelon cultivation and the soil preparation for the papaya showed concentrations of 0.5 and 0.7 mg/L respectively. The presence of thiamethoxam was identified in 100% of the samples analyzed in the papaya soil preparation stage. The values of the concentrations in soil and irrigation water exceeded the maximum limits established by international standards.

Keywords: Neonicotinoids, pesticides, modified ecosystems

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1. Introduction

Agroecology is fundamental for the study of sustainable agroecosystems, uses ecological principles and concepts as tools to understand the processes of land degradation, such as erosion, desertification, salinization, compaction and decrease of fertility (9). It is essential for the Earth's health to preserve the physical and chemical characteristics of soils. Given the eminent growth of the human population and urbanization, land is considered a limited and economically scarce resource; therefore, the demand for safe food, goods and services is increasing, and it is the responsibility of the human being to preserve soil as a natural source for the development of human life quality (29).

Agricultural activities such as conventional tillage, the massive use of pesticides and all anthropogenic activities contribute to the phenomenon of groundwater and surface water pollution. This phenomenon limits and impedes the enjoyment of the benefits of quality water, which can result in impacts to aquatic organisms and destruction of soil macrofauna. The consumption of agricultural food contaminated with neonicotinoid pesticides, causes damage to public health by provoking reproductive inhibition or failure, suppression of the immune system and teratogenic effects (23).

The use of pesticides in agriculture began its activity in 1940, since then more than 70,000 chemical substances have been generated for crop management. The sales volume of pesticides is around 2, 800 million kilograms of active ingredients a year and more than 50 thousand commercial formulations (15). Thiamethoxam is recommended for 116 agricultural species and is present in 64 countries worldwide (5). Thiamethoxam was recorded in 2004 and is used to control whitefly (*Bemisia tabaci*) in tomatoes, chili peppers and fruit trees such as papaya (12). It is also used for whitefly control in the USA., The European Union, Brazil and India, currently and due to its excessive use, insect pests have become resistant to applications of neonicotinoid insecticides, such is the case of Imidacloprid. For this reason, it has been considered necessary the use of new chemical molecules such as thiamethoxam, this molecule acts in a systemic and contact form, being used for the control of aphids, thrips, mites and soil pests (19). In addition, there have been positive results on the control of some pests such as *Mahanarva fimbiolata* in perennial crops (8,14). In Venezuela it is also recommended for the control of *Togosodes orizicolus* Muir in rice cultivation, with doses of 75 and 100g ha^{-1} (34).

Thiamethoxam is also used for the treatment of seeds, recommending doses of 200 to 300 ml per 100 kg, improving its vigor and germination rate (28). The contamination of surface and underground water can be generated through runoff processes, accumulating in bodies of water large quantities of pesticides such as thiamethoxam. There are studies of thiamethoxam discharges to natural currents or to the ground that is generating focalized contamination (23). This pesticide has high mobility in the C horizon of soils; in Argentina, there is a potential risk of leaching pesticides in groundwater by the increase of its net recharge to a value of 2.3 mm day^{-1}, since the presence and residuality of thiamethoxam in soil depends on the frequency of its use in vegetables such as tomato, parsley, cauliflower, bulb onion, broccoli (3).

The management of tropical agroecosystems with the use of neonicotinoid insecticides can generate a risk of ecological damage such as the death of aquatic organisms, birds and mammals, and damage to public health. The cultivation of the papaya fruit tree, which due to its organoleptic characteristics has acceptance in the national and international market, presents numerous phytosanitary problems, such as the presence of virosis, phytoplasmas, mites and aphids, for which the producers use pesticides as neonicotinoids massively for its control. The instability of the market allows the papaya crop to be associated with vegetables such as watermelon, with the purpose of obtaining additional income during the year. This is why the objective of this study was to determine the presence and residuality of the thiamethoxam insecticide in soil and water of the papaya (*Carica* *papaya L.*) and watermelon (*Citrullus lanatus*) crop as a relay crop in the municipality of Cotaxtla, Veracruz, located in the center of the country in the Gulf of Mexico, where 45% of the total producers use thiamethoxam in the management of the papaya crop associated with watermelon (1, 19, 25, 26). The training and transfer of new technologies friendly to the environment are necessary to achieve the implementation of good practices and soil and water conservation, among them are terraces, green manures, family gardens, intercropping systems, use of living walls and level curves, efficient and rational management of pesticides (6, 21).

2. Materials and Methods

The study was developed in the municipality of Cotaxtla, Veracruz. In an experimental plot of papaya Maradol-watermelon (Figure 1), located at the coordinates of 18°53'51'' north latitude and 96°22'37 '' west latitude.

Figure 1. Location of the papaya producing area in the town of Cotaxtla, Veracruz, México

In the study area, papaya producers use thiamethoxam during the papaya crop cycle, with a total volume of 500 ml ha^{-1} for the control of whitefly, thrips and aphids. The soils of the agroecosystem with papaya are characterized by the fact that 45% is predestined for the cultivation of papaya and watermelon at different sowing dates. This pesticide has been used for the cultivation of papaya in this area for the last 5 years.

Method

To know the types of soils in the area where the insecticide was applied, a diagnostic study was carried out based on the local knowledge of the farmer called "classification of peasant lands" (2). An experimental plot of papaya cultivation associated with watermelon with a population density of 2,778 plants/ha was established in the study area. Samples were taken from the experimental plot during four phenological stages in the year to know the presence and concentrations of thiamethoxam. The phenological stages in the papaya-watermelon crop, correspond to the following study treatments, which are mentioned below as T1: Relay cultivation (watermelon); T2: Preparation of the land previous papaya transplant; T3: After the papaya transplant; T4: Papaya production.

The soil sampling technique was as established by the Official Mexican Standard NOM-AA-105-1988 and the International Standard ISO/TC 190 (ISO 11074: 2015/DAM 1:2017-Soil Quality). The sampling consists of using the zigzag method, with strains of 20 by 20 and 25 cm deep, collecting a sample of 2 kg for the determination. For the sampling in irrigation water, the Mexican Standard NMX-AA-003-1980, the Official Mexican Standard NOM-AA-104-1988, and the International Standard ISO 5667-10-1992 (31) were used. The total of samples that were raised by phenological stage of the crop were 6 composite samples obtained through the aforementioned method. The samples of soil and irrigation water were taken after: 100 days planting the relay crop (watermelon), 200 days in the stage of preparation of land for the transplant of papaya, 300 days in the stage of transplant of the papaya and 450 days in the papaya production stage. The soil and water samples were from April, 2016 to March, 2017. The samples collected were transferred for analysis to the laboratory of the Technological Instituto Tecnológico de Boca del Río, to the Investigación de Recursos Agropecuarios Laboratory (LIRA).

The HPLC-UV is integrated of a quaternary pump, autosampler of 200 vials, with syringe of 250 μL, a fluorescence and visible UV detector, contains four solvents and a separation column of 10 cm by 4.6 of diameter of 5 μ and loop of 20 μL sample with a capture system. The instrumental conditions of the chromatographic column is C18 RP 5 μm, with a temperature of 25° C, a mobile phase of 45/55 (AC/AG), with a flow of 1mL min^{-1} and λ = 254 nm, the working range was 10 to 100 μg of thiamethoxam L^{-1}, with a linearity of R≥0.9 and a recovery of ≥90%.

For the thiamethoxam determination in soil the following procedure was carried out, samples were macerated, then 10 ml of Acetonitrile (CH_3CN) was added, it was allowed to sit for 5 minutes, it was shaken vigorously for 1 minute using a Vortex mixer. Afterwards, 3 ml of HPLC water, 6 ml of hexane (C_6H_{14}), 4 g of Magnesium Sulphate ($MgSO_4$), 1 g of N-acetylcysteine (NAC), and 500 mg of sodium citrate ($Na_3C_6H_5O_7$) were added. The sample was subjected to manual shaking for 20 minutes, and vortexed for 1 minute, then centrifuged for 10 minutes. And later, 6 ml of excess was taken and deposited in tube II with 90 mg of magnesium sulfate, 150 mg of Primary and Secondary Amina (PSA), 150 mg of Octadecyl Carbon Chain (C18). The sample was vortexed then for 1 minute and centrifuged for 10 minutes. Next, 3 ml of the excess was taken and dried at 40° C with N_2, afterwards 200 μl of methanol (CH_3OH) was added, measuring 100 μl + 900 μl of mobile phase and then filtered with 0.22 μl disks, finally it was recovered in vials.

The statistical design for soil and irrigation water was randomized complete blocks with 6 repetitions. The statistical software was with the program statistica 2007, with parametric statistical analysis (ANOVA) and non-parametric Kruskal-Wallis, and graphs categorization with pie charts.

3. Results

The values of concentrations of thiamethoxam in the soil of the experimental plot that were found at a depth of 30 cm. in the area of the macrofauna and microflora were ≥0.55 mg/kg, these values of thiamethoxam are higher than those found in the irrigation water of ≤0.45 mg/L. Thiamethoxam was present in 79% of the analyzed samples of irrigation water during the phase of papaya and watermelon cultivation. Whereas in soil, 75% of the total samples analyzed showed average concentrations of thiamethoxam of 0.57 mg/kg, this response is due to the use and management of thiamethoxam when applied directly to the soil and in the irrigation water system.

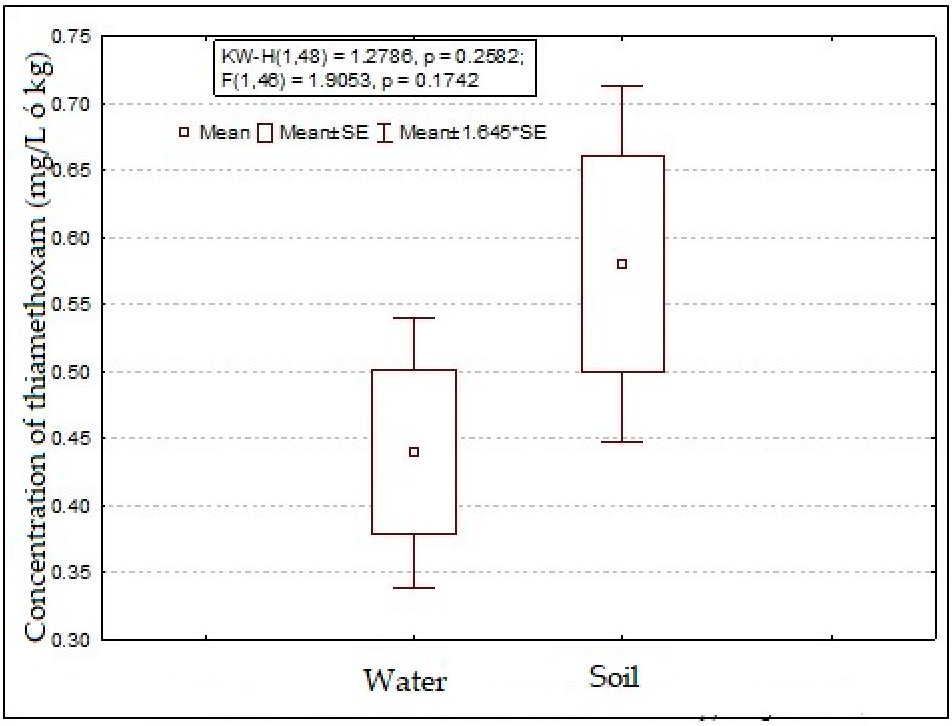

Figure 2. Concentration of thiamethoxam in irrigation water and soil in the papaya crop associated with watermelon in the area of Cotaxtla, Veracruz.

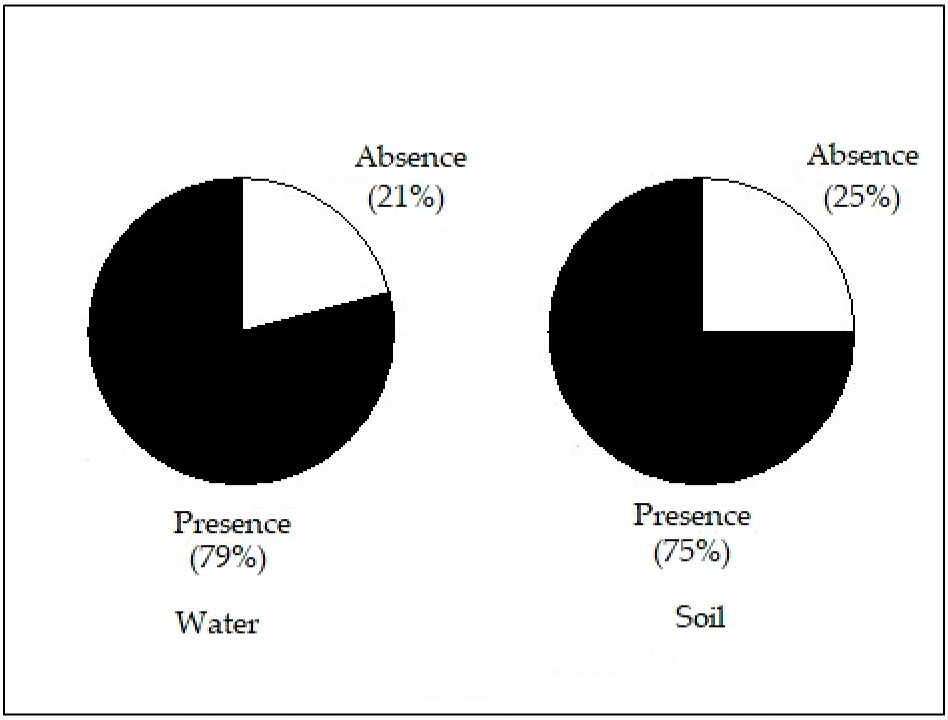

Figure 3. Percentage of absence and presence of thiamethoxam in water and soil of the papaya crop associated with watermelon in the area of Cotaxtla, Veracruz.

The analyzed soil showed that, among the different phenological stages of the papaya crop, there was a higher concentration of thiamethoxam in the stage of papaya transplant and soil preparation, with values higher than 0.8 mg/kg of thiamethoxam (Figure, 4). The lowest concentrations were present in the papaya fruiting stage, which is when the papaya crop demands more nutrients for its development and fruit production, the concentration values of the insecticide were less than 0.2 mg/kg. Values higher than 0.7 mg/L of thiamethoxam were found in the samples of irrigation water while in the stage of preparation of land, which is when the soil of the papaya crop is without any vegetal cover, this allows thiamethoxam to be retained by clay particles. Because of the above, it is important to mention that when comparing the relay crop that corresponds to watermelon with the papaya crop in fruit production, the greater presence of thiamethoxam in irrigation water and soil corresponded to the relay crop (watermelon) with values higher than 0.5 mg/L in irrigation water and 0.4 mg/kg in soil (Figure 4 and 5). In the soil preparation stage, thiamethoxam concentrations are retained by soil particles and some particles of thiamethoxam are mobilized into the shallow waters when there are no established crops that extract the thiamethoxam particles through the roots or stomata of the plants.

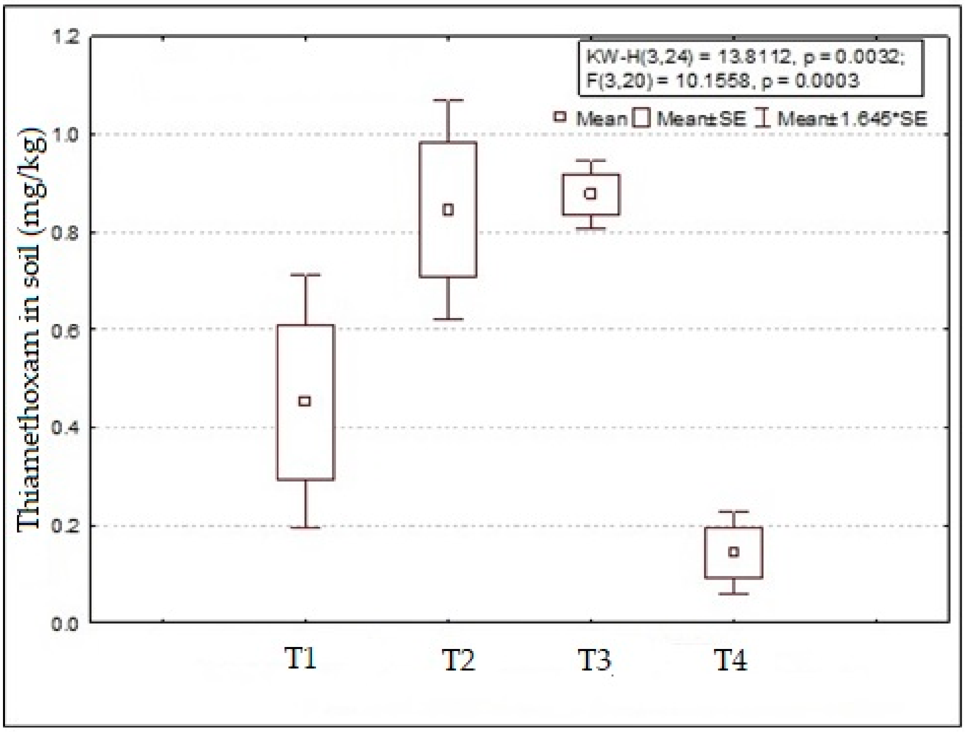

Figure 4. Concentration of thiamethoxam in soil in different stages of the papaya crop associated with watermelon (relay crop).

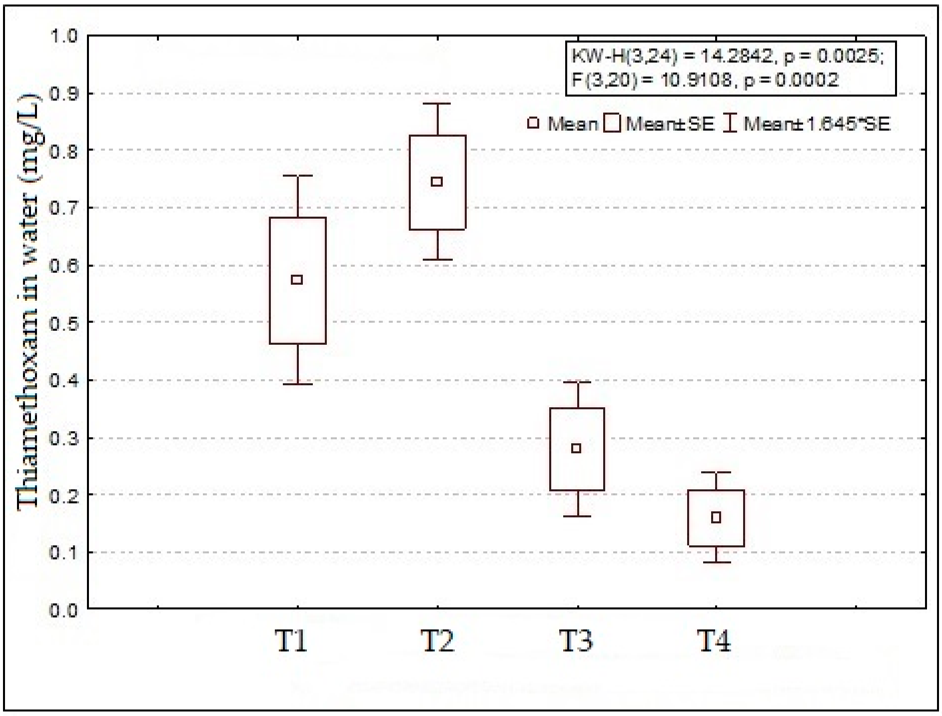

Figure 5. Concentration of thiamethoxam in irrigation water in different stages of the papaya and watermelon crop (relay crop).

In samples from different types of soil and irrigation water showed that the presence of thiamethoxam in watermelon cultivation was 100% in the irrigation water. In the opposite case at the beginning of the papaya crop, in the stage of seedling production, it was found that 100% of the samples had thiamethoxam in soil and irrigation water. Also, the crop in production showed thiamethoxam particles in 33% of the samples analyzed in soil and 50% in irrigation water, this is explained, because the rest of thiamethoxam particles were mobilized towards the plant and papaya fruits via sympathetic and apoplastic (Figure 6). In addition, this agrees with the results of the diagnosis made to papaya producers in the municipality of Cotaxtla, Veracruz, they apply the highest volume of thiamethoxam to papaya cultivation mainly in free soils, while in sandy soils there is no application. Therefore, significant differences were found with the volume of thiamethoxam used in clay soils when compared with the volume of thiamethoxam used in sandy and loamy soils (Figure 7).

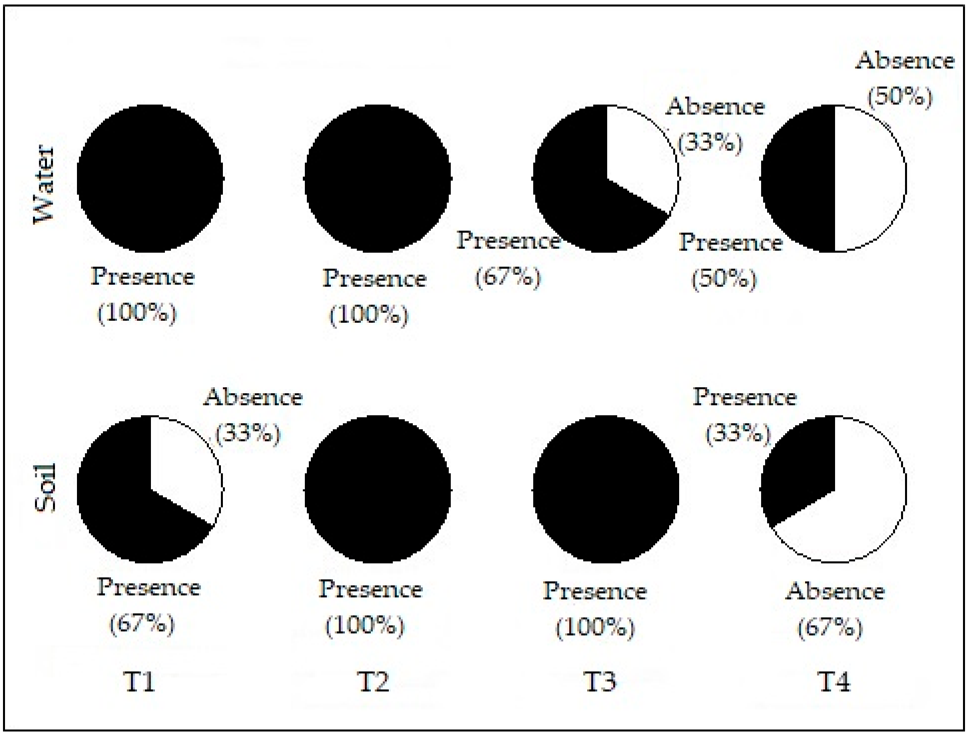

Figure 6. Absence and presence percentage of thiamethoxam in irrigation water and soil in the papaya crop associated with watermelon (relay crop) in an experimental plot.

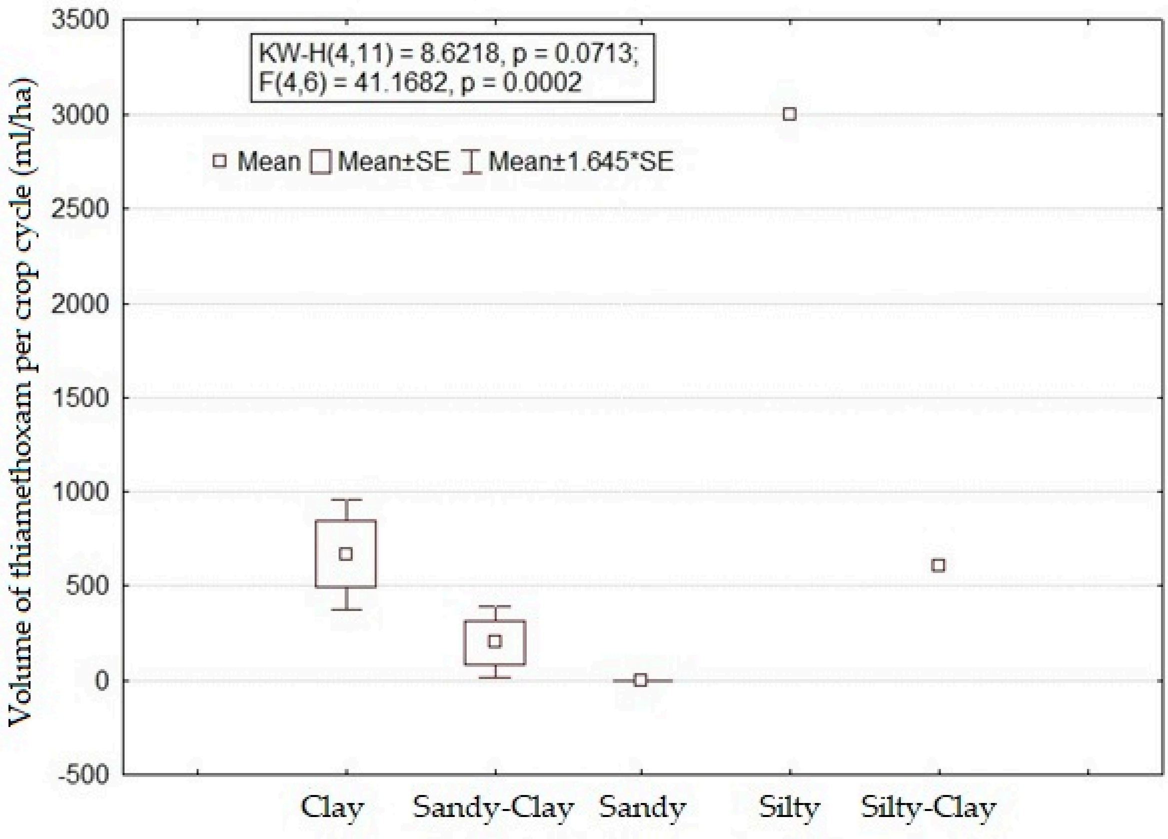

Figure 7. Volume of thiamethoxam per crop cycle managed in different types of soil in the municipality of Cotaxtla, Veracruz.

4. Discussion

In the main papaya producing areas in Mexico, 27% of the producers have orchards under the monoculture scheme and 45% of them associate the papaya orchards with different vegetable species, this allows having a greater diversity of pests and diseases. Under the risk of causing economic and ecological damage, producers use thiamethoxam in doses of 3 L ha^{-1} for the control of agricultural pests and a minimum dose of 300 L ha^{-1} in the cycle of cultivation of papaya. It is estimated that the amount of thiamethoxam used during the papaya crop cycle in the main papaya producing area in Cotaxtla, Veracruz, is 220.5 kg/year (22), comparing these results with the valley of Culiacán Sinaloa, Mexico, the total volume of thiamethoxam is 692.26 kg/year and for the group of neonicotinoids the amount applied is 1682.25 kg/year of active ingredient (16).

One of the characteristics of thiamethoxam is its mobility capacity, that is why it is found in wells and deep wells that are used for domestic and agricultural use, such case is similar in Argentina, where there is presence of thiamethoxam in the C horizon soil (Mineral layers that have been affected by pedogenetic processes in a minimal way) with high leaching capacity to groundwater (3). In addition, the presence of thiamethoxam in water and soil (Figure 6) reinforce the theory of chemical contaminants migration, where the highest concentration of pesticides are present at shallower depths of soil, mainly in shallow waters (20). On the other hand in Venezuela, in soils of loamy sandy texture, with guava plantations, it was observed good aeration, medium permeability and little retention of water and nutrients, this allowed thiamethoxam absorption isotherms with values of 2.95 ± 0.83 mg/kg; this phenomenon depends on the characteristics of the soils, such as the clay content, because thiamethoxam has the capacity to protonate, due to its size and polarity. The percolation of thiamethoxam to groundwater depends on the texture of the soil, in soils with high sand content the thiamethoxam tends to percolate, considering that thiamethoxam in soil degrades slowly in aerobic conditions, but in concentrations of 4100 mg l^{-1} at temperatures of 20° C is highly soluble in water, according to the above there is a risk that thiamethoxam particles may infiltrate into groundwater (10, 31). Studies show that concentrations of 10 μg/L in water causes death of aquatic species, and in turn, 10 μg/kg of thiamethoxam in the topsoil damages the macrofauna and microflora of the soil. In addition, if we consider that soil degradation (LD_{50}) is moderately persistent with 39 days in the field. It is important to mention that some studies show that they can persist in soil for up to 3 years (31,32). Thiamethoxam has been found with drinking water values of 0.06, 0.2 and 1.0 mg/l, this is the result of applications made in crops; furthermore, thiamethoxam is considered an effective contaminant when applied to the foliage of plants. Another feature of thiamethoxam, is its ability to evaporate in less than 30 seconds when it reaches a drop diameter of 337 μm, an important factor is the relative humidity, when there are values of 60%, this favors the evaporation of thiamethoxam to the environment (7, 27). Thiamethoxam is absorbed by the plant through the roots, stems and leaves, observing that the largest thiamethoxam particles are concentrated in the stage of fruit production. When the management of thiamethoxam is towards the canopy of the trees, these particles can enter through stomata and lenticels and translocate to the different organs of the plants (11). The Environmental Protection Agency (EPA) recommends that studies be conducted to address the safety of food (33). For the cultivation of papaya, the EPA (2012) established the tolerance levels with concentrations of 0.4 mg/kg of thiamethoxam, and 0.05 mg/kg for the European Union (EFSA, 2012) (18).

Concentrations of thiamethoxam found in Veracruz in irrigation water and soil of the agroecosystem with papaya, exceed the established by the international standards of the United States and the European Union (Figure 2). In the phase of cultivation in soil, the concentrations of thiamethoxam, in the stage of production of papaya fruit, exceed the established by international standards. Similar case in Ecuador, with concentrations of thiamethoxam in irrigation water, in turn contaminating surface waters, where the following values were found: 0.32 μg/L in Río Chico, 0.17 μg/L in Río Portoviejo, 0.48 μg/L in Río the Ceibal; while, in drinking water, concentrations of 0.27 μg/L were found (4). Therefore, it is necessary to have a participatory technical knowledge with producers to define the quality of the soils where the papaya crop is established, mainly to define the texture, pH and content of organic matter, this would allow to establish strategies to improve the production of the fruit trees and annual crops, using sustainable technologies (13). Currently, an alternative could be the use of nanoparticles, since they can replace the use of chemical substances in crops. It is noteworthy that companies like Bayer and Syngenta are conducting research in this area of nanotechnology, aimed at finding a lower dose of active ingredient, lower residuality and with it, a lower load of contaminants in the environment. With these characteristics it is possible to generate fertilizers, growth regulators and more powerful and efficient pesticides for the control of pests and weeds, which respond to extreme weather conditions, probably leading to the disadvantage of generating contamination to soils and bodies of water (17). Organic agriculture such as the use of insecticides of vegetable origin could be an alternative for the control of agricultural pests, it is also important to consider the use of low doses of chemicals, or insecticides of biological origin, as an alternative for the control of specific pests (22, 24). Such is the case for Mexico where the optimum dose of thiamethoxam of 0.3 kg/ha was evaluated for papaya cultivation, spraying every 15 days for the control of pests such as *Diabrotica* sp., *Laspeyre siapomonella, Frankliniella occidentallis, Myzus persicae, Bemisia tabaci, Nezara viridula, Anthocoris nemoralis, Planococcus* sp., *Anastrepha ludens* Loew and *Toxotrypana curvicauda* (18).

5. Conclusions

Concentrations of thiamethoxam that exceed the maximum permissible limits of the international standards of the EPA and EFSA were found in the agroecosystem with papaya. These were ≥0.40 mg/L in 79% of samples in irrigation water and 0.55 mg/kg in 75% in the soil. The highest values of thiamethoxam in soil were in the relay crop stage (watermelon) and soil preparation for the papaya transplant, with values higher than 0.4 and 0.8 mg/kg and in irrigation water of 0.5 and 0.7 mg/l respectively. Another highlight is that 100% of the samples analyzed in the stage of preparation of land for the planting of papaya presented this pesticide. It is therefore necessary to establish planning criteria on the management and use of thiamethoxam in papaya agroecosystems with the purpose of reducing pollution to the ecosystem.

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