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Determination of Pesticide Residues of Organochlorine in Some Local and Imported Foods in Qatar

Noora Mahmood Al-Shamary

Qatar University, QA

Email: noora.m@qu.edu.qa

Fresh fruit and vegetables are an important part of a healthy diet as they are a significant source of vitamins and minerals. However, fresh fruits and vegetables can also be a source of toxic substances such as pesticides. Pesticides are a several and diverse group of chemical compounds, which applied to crops at various stages of cultivation and during production and post-harvest treatment of agricultural commodities. Recently, the government of Qatar has attempted to encourage agricultural production. Despite a noticeable increase in agricultural production in Qatar, however, this increase does not fulfill the need of residents in Qatar. Therefore, Qatar imports large amounts of food products from other countries which are large agricultural producers. The lack of information regarding pesticide residues in food in Qatar implies the necessity to determinate their concentrations in food items. Organochlorine pesticides (also known as chlorinated hydrocarbons) are primarily insecticides with relatively low mammalian toxicity, fat soluble and normally persistent in the environment. Many chlorinated hydrocarbons have the ability to accumulate inside the body due to their lipophilic nature. The study was aimed to examine the occurrence of organochlorine pesticides residues in some local and imported vegetables and fruits in Qatar, as a prelude to assess the risks related to their consumption. In order to achieve this aim, the following specific objectives were carried out: (i) determine the amount of 10 organochlorine pesticides (Heptachlor, aldrin, dieldrin, Endrin, a-chlordane, g-chlordane, endosulfane I, methoxychlor, α-BHC and β-BHC) in seven mostly consumed vegetables and fruits in Qatar using Gas chromatography-electron capture detector (GC-ECD), (ii) screen the residues of pesticides in these vegetables and fruits using scan mode of Gas chromatography- mass spectrometry (GC-MS), and (iii) perform statistical analyses to data obtained. A total of 127 samples of most 7 consumed fresh vegetables and fruit from local and import production were analyzed. The samples included: 26 samples of fruits and 101 samples of vegetables. Samples were extracted within 24 hours and stored at 4°C until the analysis. The simple random sampling and stratified random sampling were used as sampling procedures for collecting the vegetables and fruits.

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For all vegetables and fruits except strawberries, simple random sampling was used. For strawberries sampling, the stratified random sampling procedure was used. The EPA Method 8081 was chosen as a reference method for the preparation and extraction method with some modification. Additionally, the Dionex Application Note 332 “Accelerated solvent extraction of pesticide residues in food products”, 2012, was used as extraction method for vegetables and fruits. The fresh fruits and vegetables samples were collected from farms and market a day before extraction. Each sample was divided in to two groups: washed sample with water and unwashed sample. No sample digestion was needed prior to extraction. A clean-up procedure is usually carried out to remove co-extracted compounds that may cause interference in the chromatographic determination or be detrimental to the analytical instrumentation. Following extraction, 5 g of anhydrous sodium sulfate were added to the collection vial to adsorb co-extracted water. The vial was shaken for 15 s and the water-free extract was decanted into a clean vial. Two solid phase extraction techniques were used (Florisil and Silica Gel). The EPA method 3620C- Florisil Cleanup and Method 3630C- Silica Gel Cleanup were used as reference methods for cleaning the samples. All samples were cleaned up using 2 g Florisil Clean-Prep Cartridge. However, some interferants that are not removed by Florisil Cartridge would be removed by a second cleanup technique which was Silica Gel cleanup. For the Quality Control and Quality Assurance measures (QC/QA), the Limit of detection and limit of quantitation were calculated for all analytes. The LOD and LOQ were calculated using 10 samples of the lowest concentration of spike (10 ppb). The evaluation of the recoveries of studied pesticides were done by adding known concentration of an internal standard (Decachlorobiphenyl) to about 10% of total number of samples. The range from 93.6% to 106.6% was the percent recoveries in spiked samples. Residues of 10 OCPs (Heptachlor, aldrin, dieldrin, Endrin, a-chlordane, g-chlordane, endosulfane I, methoxychlor, α-BHC and β-BHC) were identified using GC/ECD. The GC/ECD analyses were performed on an Agilent 6890 N equipped with a splitless injector and a 7683 autoinjector. The analysis by GC/MS was carried out on an Agilent 7890A MSD 5973 equipped with a split/splitless Inlet and a 7683B autoinjector. Separations were conducted using an HP-1 30 m 0.25 mm 0.25 µm column for GC/ECD and Rxi-5SILMS 30 m 0.25 mm 0.25 µm column for the GC/MS. The GC/MS data were acquired and processed with a wiley7n.1 and NIST98 Libraries. The most frequently detected OCPs in the samples were heptachlor, g-chlordane and a-chlordane. Two statistical analysis tests were used to determine significance (pair-difference t-test and analysis of variance (ANOVA)). In most of the comparisons between the washed and unwashed samples, no-significant differences were observed ($P > 0.05$). Here, there is a dire require for controlling program for residues of pesticides in food products. Since florisil and silica were used for cleanup, heavy matrix interferences were observed in most of the samples, consisting primarily of fatty acids. Thus the detection and identification of trace levels of pesticides in this complex profile can be very time-consuming and laborious. The level of pesticide residues contamination may be considered a potential public health problem, since pesticides are characterized by various degrees of toxicity to non-target species, including human beings. The results underscore the need for regular monitoring of large samples to determine the pesticide residues, especially for the imported samples. This research can be implemented as a regular monitoring for the presence of OCPs in fruits and vegetables. Also the results of this research can be used as reference for future work. Future studies should consider the processing factors other than washing with tap water in order to account for the reduction or removal of pesticides such as: washing (with acetic acid, sodium chloride and soap) and peeling. Also as a recommendation, we need to look to other types of food that are sold in Qatar and may contain pesticides residues, such as grains and dates. Future studies also should look to the presence of other type of pesticides such as organophosphorous and carbamates compounds.