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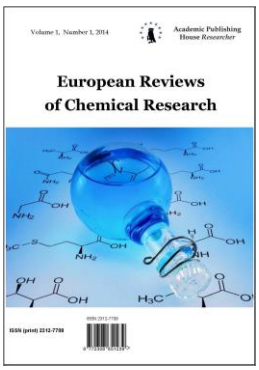

Impact Studies of Pest Control Products used in Closed Production Environments on Food Production Quality by SPME coupled to GC-MS

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Abstract

To determine concentration insecticides and rat poisons detected in the finished industrial samples, the solid phase micro extraction (SPME) is applied. The gas chromatography coupled to a mass spectrum (GC-MS) is elaborated. In order to get a best extraction, different process parameters are examined and optimized. The relevant finding are obtained using a time of unspecific binding properties of 30 minutes duration and mode of agitation for 30 min with agitation by magnetic stirrer and a temperature of 30° C. It has been demonstrated that insecticides, rat poisons and pesticides have a high chance of being found at a high concentration in finished industrial products.

Keywords: GC-MS; SPME; extraction; Performance; rat poisons; insecticides.

1. Introduction

Pesticides (rat poisons and insecticides) are compounds characterized by their diversity and their different physicochemical properties (Blasco et al., 2004). Their adverse side effects have been quickly identified. It turns out that the toxicity related to their molecule structures is, in principle, not limited due to the associated species which should be evinced. It has been shown that they are particularly toxic not only to humans (Bonansea et al., 2013; Jones et al., 2012) but also to the environment. These molecules tend to accumulate in different biotic and abiotic matrices, including water, air, soil, aquatic organism, blood and food (Dong et al., 2003; Hlotz et al., 2004). It is noted that pesticides are among the oldest organic synthetic ones being used in the agriculture activities in the world since 1940 because of their strong impact in the fight against pests and diseases (Jelen et al., 2012; Hays et al., 2003). However, pesticides are very toxic and persistent in the environment which tends to accumulate in living organisms. Following to low degradation and high solubility in organic materials, they easily enter the food chain as contaminants reaching humans through the consumption of drinking water and agricultural food products (Tanabe et al., 1993; Kirrluk et al., 1995). Although most of them have been banned from use, however, they are still detected in ecosystems (Kosikowska et al., 2010; Svjetlana et al., 2010; Raposo et al., 2007; Rianawati et al., 2009; Mesquita et al., 2011).

Recently, the analysis of pesticide residues has received increasing attention in many places including in north African countries. In particular, it has been remarked that the pesticides

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monitoring in industrials products is very important and requires high efficiency, unique selectivity, and high sensitivity techniques (Vidal et al., 2009; Shahsavand et al., 2018).

In agribusiness industries, the use of pesticides (rat poisons and insecticides) in the treatment of production and storage areas eliminate the pests that may be developed and impaired the quality of the finished products or the production process. The latter is framed by the law and controlled by the ONSSA (National Office of Sanitary Safety of Food Products) within the framework of an effective normative use of such chemical products without an impact on industrial food products ready for consumption.

In this present work, we first verify the impact of the use of these products (rat poisons, insecticides) used to treat pests in closed industrial areas (storage areas, production areas). Then, we check the presence or the absence of traces and concentrations of these products in the finished products. For this purpose, we adopt the method of the micro-extraction on the solid phase SPME, in the presence of the chromatography gas coupled to a mass spectrum (GC-MS). This coupling SPME/GC-MS provides a great sensitivity and precision even at very low concentration.

2. Materials and methods

In this section, we give the essential on the used methods including materials.

2.1 SPME solid phase micro-extraction method

For the detection and the quantification of molecules of the industriel products by the solid phase microextraction method (SPME), we immersed the SPME fiber in the studied samples. This step was carried out at room temperature for 15 minutes. It can be illustrated in (Figure 1).

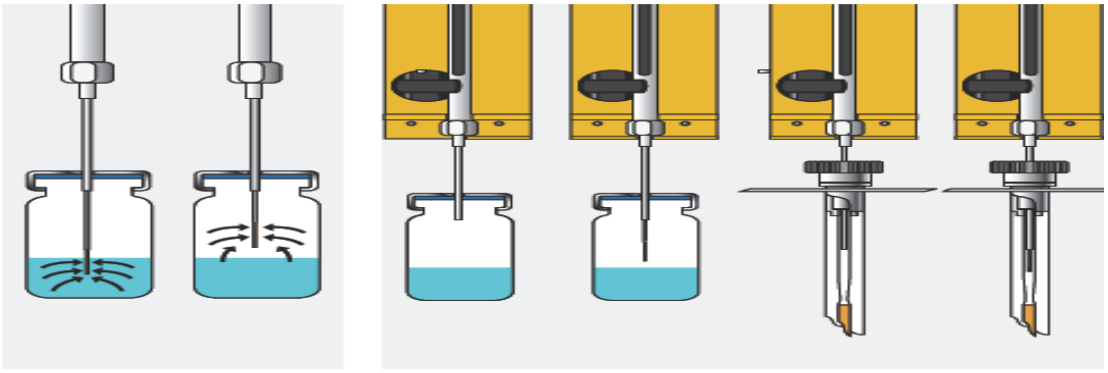


Fig. 1. Adsorption and desorption process by SPME

The used fiber consists of a Carboxen/PDMS/DVB 50/30 µm phase. The chemical compounds are first concentrated on the fiber (adsorption phase), then they are thermally desorbed in the GC-ECD injector. The analysis conditions are the same as the ones mentioned above (Anandhakumar et al., 2013).

2.2 Optimizing SPME

To increase the sensitivity and the effectiveness of the SPME method, we considered a optimization of various parameters such as the temperature, the exposure time, the duration and the mode of agitation, the effect of the pH and adding salt.

2.3 Calculation method

The concentration is determined according to the method of qualification of the peaks taking into account the volume of final extract and the volume of sample analyzed for each identified molecule. For the SPME method, it is given by

$$Ce = \frac{Ae \times Ci}{Ast}$$

Where C_e is the concentration of a compound in the sample. Ae indicates the air of a compound in the sample. Ast is the air of a compound in the standard. Ci represents initial concentration of a compound in the standard and V denotes the volume of the sample to be dosed

in ml. Ve is the volume of extract in ml. The percent recovery (PR%) or yield of each compound was calculated using peak air. Concretely, this has been done using the following equation

$$PR = \frac{Ce}{Ci} \times 100$$

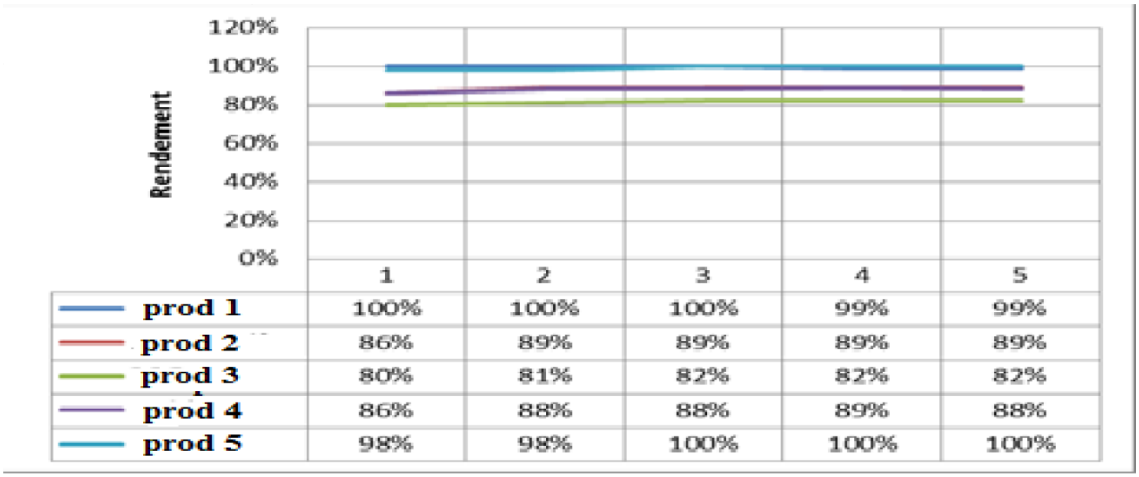
Where C_e now represents the concentration of the sample and where Ci denotes the standard initial concentration.

3. Results and discussions

In this section, we present the obtained results and the corresponding discussions. Before giving the main results of this work, we first cheek the stability and the validity of the studied method. It is recalled that the validation of an analytical method consists of the determination relevant parameters as the fidelity which itself represents a set of dispersion characteristics including repeatability, intermediate fidelity and reproducibility.

As a criterion for estimating the stability, we decide to elaborate a repeatability examination. It refers to test the same performed size under conditions as stable as possible and at short intervals. This measurement of the variation of the results should do in the same laboratory characterizes. The precision is obtained when the method is repeated by the same investigator under the same conditions (reagents, equipment, adjustment and laboratory) in a short time interval. This makes it possible to evaluate the accuracy of the method under the normal operating conditions.

To study the stability of SPME, a series of five repetitions analysis was performed on the chemical compost concentration (5µg/L) under optimized conditions. The obtained results are shown (Figure 2).



	1	2	3	4	5
prod 1	100%	100%	100%	99%	99%
prod 2	86%	89%	89%	89%	89%
prod 3	80%	81%	82%	82%	82%
prod 4	86%	88%	88%	89%	88%
prod 5	98%	98%	100%	100%	100%

Fig. 2. Variation stability of SPME a series of five repetitions analysis was performed on the chemical compost concentration (5µg/L)

From Figure 2, it can be seen that for each products there is no variation with the identical yield values for all tests.

Having discussed the stability of five products, we present now the results and give the corresponding discussions. The inductrial studied areas are given (Figure 3).

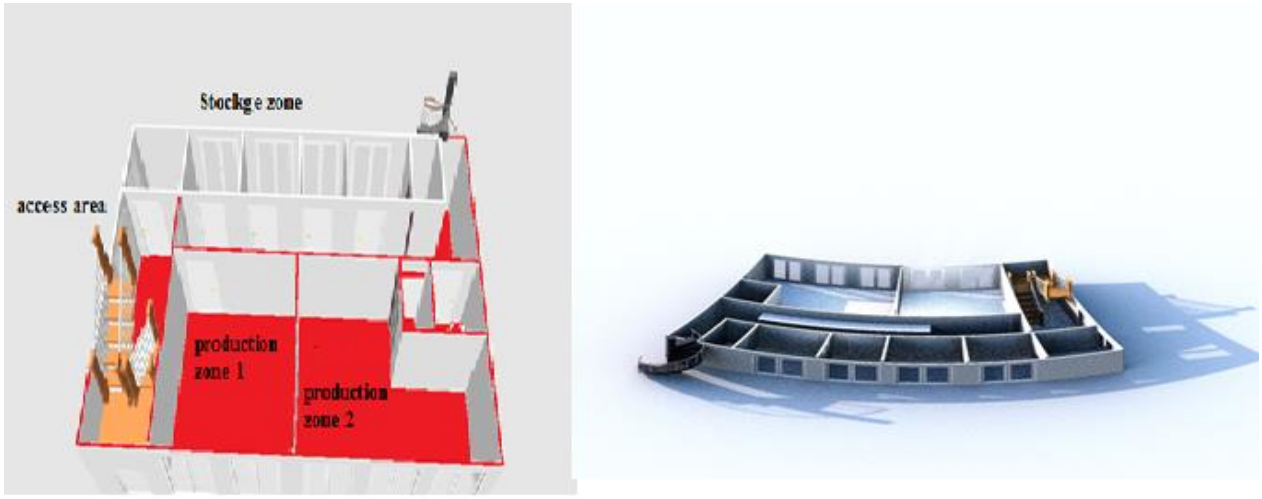


Fig. 3. Overall scheme of the building of products and storage treated by chemicals

Production areas (ZONE 1 and ZONE 2) were treated by spraying a liquid insecticide at the end of production at 9 pm. The treated products are illustrated in (Figure 4).

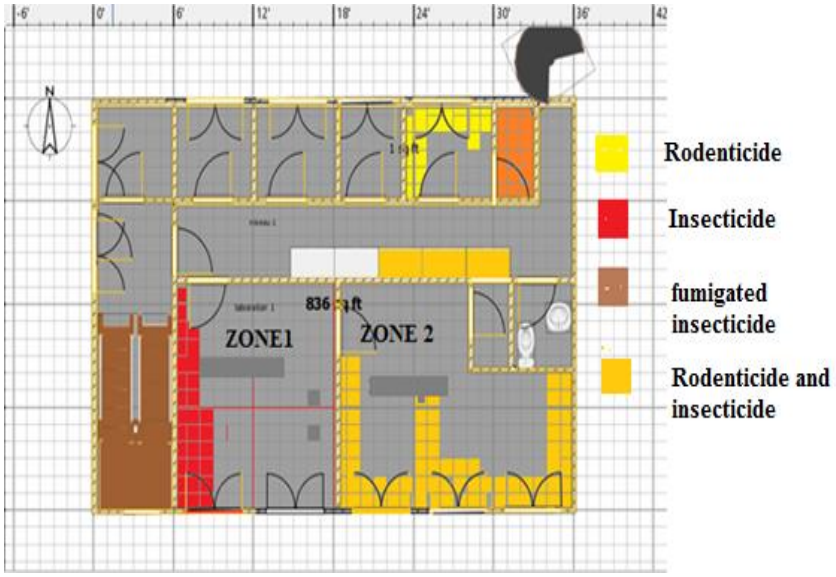


Fig. 4. Distribution of treatment areas by type of product used

Several varieties of insecticide insecticides were used in this study. In particular, we started by profiling the active ingredient of each industrial formulation (rat poison/insecticide). To be able to subsequently identify them, an analysis study should predormed. Indeed, samples are taken randomly in all the products before and after the surface treatment to see the impact of the treatment in the quality of the finished products.

After treatment, the SPME fiber was implanted at the outlet of the oven on the cooking belt to take advantage of the optimal adsorption condition already established in our preliminary study of implementation of the technique.

The following results represent the analytical profiles of the different peaks found by SPME/GC analysis: MS

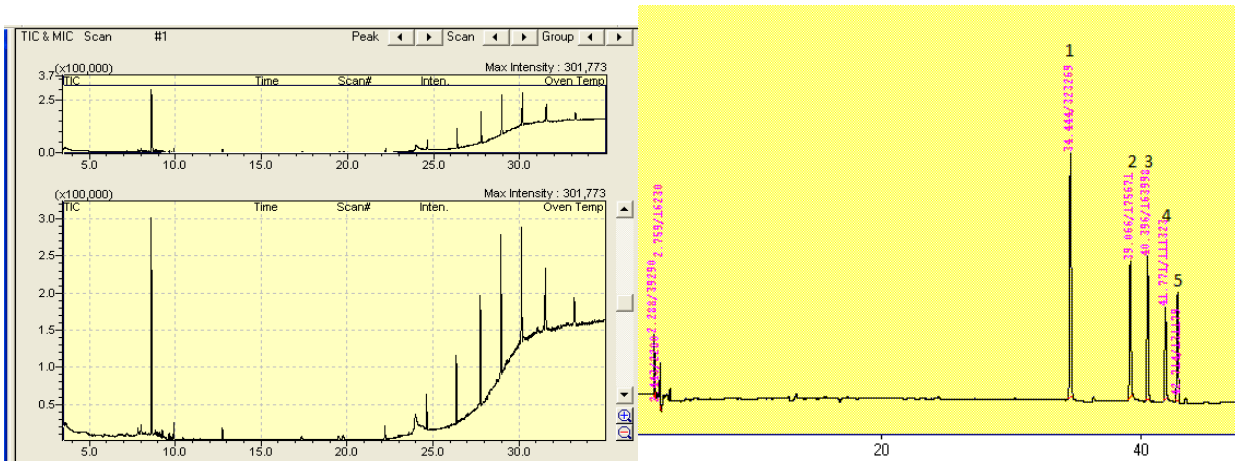


Fig. 5. Profile of the treatment product obtained by SPME coupled with GC-MS and GC-ECD

This figure represents the profile of the different chemical molecules that are present in the industrial formulation for insect treatment analyzed by SPME coupled to a GC-ECD and GC-MS.

In what follows we discuss the product analysis after 6 hours spent from the moment of surface treatment with insecticide raticides. The Figure 6 represents the results obtained after analysis of the food products produced in zone 1 after 6 hours of the surface treatment.

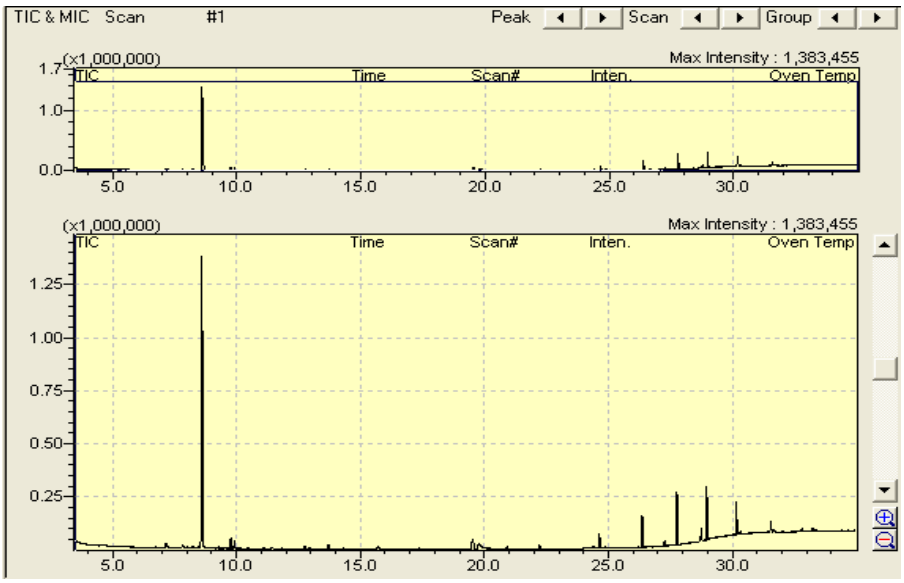


Fig. 6. Analysis results of the finished product by SPME coupled to GC-MS after 6 hours of treatment

After 6 hours of the surface treatment by pesticides and rat poisons by spraying, it has been noticed the presence of a low concentration of certain chemical products. This implies that the 6 hour delay is not sufficient to eliminate such chemical production after a spray.

The results obtained by SPME coupled to GC-MS after analysis of industrial production in a firm surface after 12 hours of surface treatment by spraying are illustrated in (Figure 7).

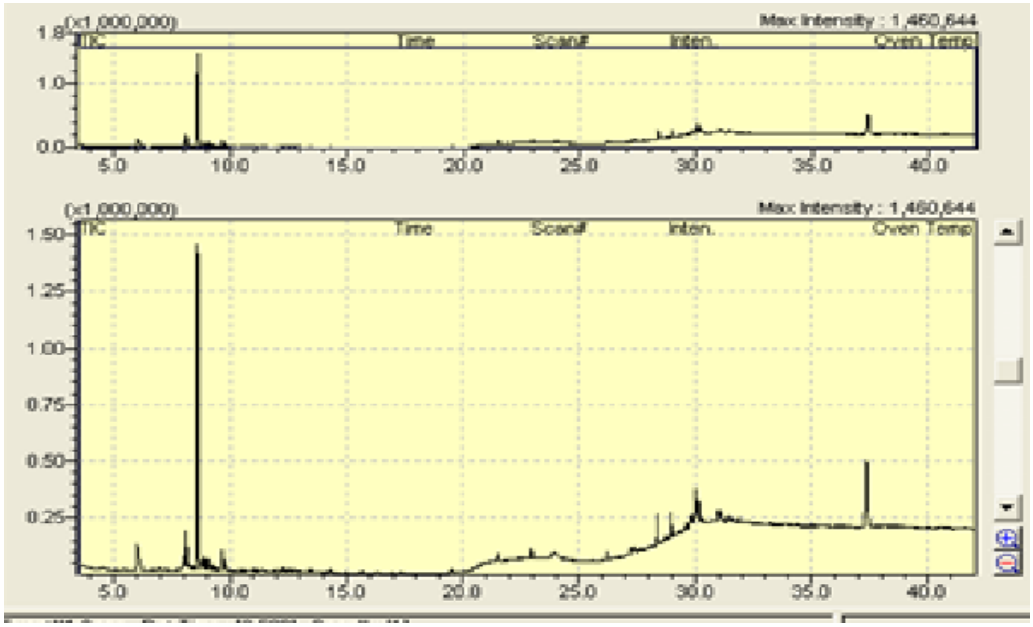


Fig. 7. Analysis results of the finished product by SPME coupled to GC-MS after 12 hours of treatment

After 12 hours of surface treatment, we found two molecules having very low concentration. It has been shown that the resumption of production after 12 hours of surface treatment can present risks on the quality of the finished product. The results obtained by SPME coupled to GC-MS after analysis of industrial production in a firm surface after 18 hours of surface treatment by spraying are given in (Figure 8).

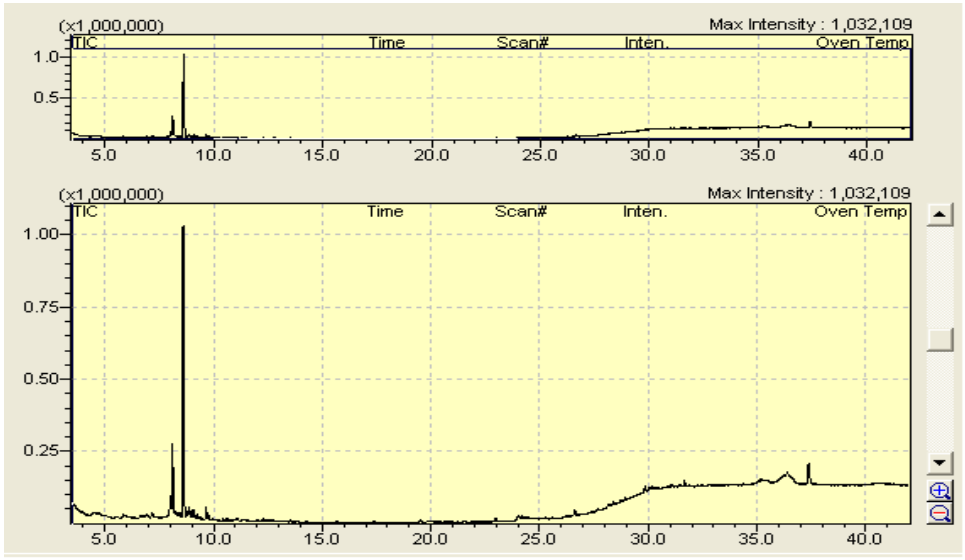


Fig. 8. Analysis results of the finished product by SPME coupled to GC-MS after 18 hours of treatment

It is noted that after 18 hours of surface treatment the presence of surface treatment products in the finished industrial products is very low or ignored. This allows us to think that 18h remains a minimum period of time of product to avoid the presence of the no desirable chemical element in the industrial production.

The results obtained by SPME coupled to GC-MS after analysis of industrial production in a firm surface after 24 hours of surface treatment by spraying are illustrated in (Figure 9).

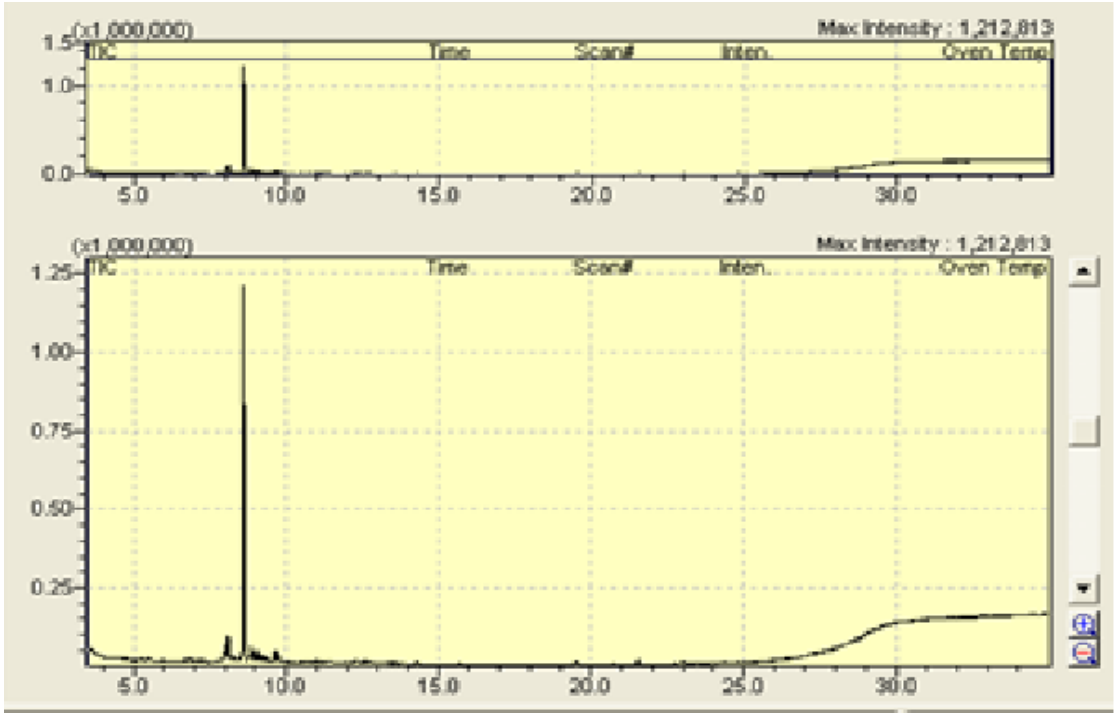


Fig. 9. Analysis results of the finished product by SPME coupled to GC-MS after 24 hours of treatment

After 24 hours of surface treatment, it is noted the total absence in the analysis.

We have worked on several pest control products that are used for the treatment of closed production surfaces and the impact of the use of these products on the quality of production on everything in the field agribusiness. Several industrial formulation profiles of insecticides and rat poisons were developed using the SPME technique coupled to GC-MS with repeatability and reproducibility to establish and verify the validity of the analytical method adopted. The present study respects the ISO quality norms. It has been observed that the use of these products may have a negative impact on the quality of the agri-food production of the units treated by chemical spraying on everything in the first 6 hours after treatment.

It has also been remarked that the use of SPME coupled to GC-MS in routine quality analyzes is very interesting, economical, efficient, stable in repeatability and reproducibility. It allows for accurate identification of all elements present at different concentrations even at the sample trace states.

4. Conclusion

In this work, we have studied that the impact of the use of these products (rat poisons, insecticides) used to treat pests in closed industrial areas (storage areas, production areas). In particular, we checked the presence or the absence of traces and concentrations of these products in the finished products by using the method of the micro-extraction on the solid phase SPME, in the presence of the chromatography gas coupled to a mass spectrum (GC-MS).

After different analysis are carried out by SPME coupled to the GC-MS on sample product in a closed environment treated by products of rat extermination/disinsection taken at different times per palliate of 6 hours according to the iso quality standard adopted by the agri-food production. It has been shown that the interval of 18 h of stopped production after each chemical treatment is necessary to avoid the presence of any treatment product in the production. Moreover, it consequently avoid the alteration of the finished product. In this work, it has been observed that the coupling SPME/GC-MS provides a great sensitivity and precision even at very low concentration.

5. Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of this paper. Also, they declare that this paper or part of it has not been published elsewhere.

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