Determination of veterinary drug residues in bovine raw milk by GC/MS using QUECHERS extraction

C.F. A. A. Campos[(1)], M. A. Andrezzi[(1)], I. P. Emanuelli[(1)], F. C. B. Cavalieri[(1)], N. U. Yamagushi[(1)], J. E. Gonçalves[(1)]

[(1)] *Program of Master in Clean Technologies and Cesumar Institute of Science, Technology and Innovation - ICETI. University Center of Maringa, Ave. Guedner, 1610, 87.050-900, Maringa, Brazil, e-mail: jose.goncalves@unicesumar.edu.br*

1. Introduction

Milk is a food rich in proteins, fats, carbohydrates, minerals and vitamins. However, some factors can influence their quality, such as: presence of disease in the herd, lack of hygiene during milking, inadequate cleaning and hygiene of milking equipment and utensils, poor water quality, inadequate packaging and transportation. If milk is not produced with good milk production practices, it can be a source of contaminants, such as veterinary drugs. Due to human food safety, organizations around the world have set maximum residue limits (MRLs) for different veterinary drugs in milk. In Brazil, veterinary drugs have been monitored by the National Plan for the Control of Residues and Contaminants, which defines MRLs based on international regulations for food of animal origin. Thus, the objective of this work was to analyse samples of milk produced in the northwest region of Paraná, in order to detect the presence of residues of veterinary drugs, by means of gas chromatography coupled to mass spectrometry.

2. Experimental

The QuEChERS method was used to extract residues of veterinary drugs (cypermethrin, chlorpyrifos, amitraz, dichlorvos, eprinomectin and fluazuron) and sample preparation, where 50 samples of raw cow's milk were collected at the school farm of the University Center of Maringá (Unicesumar) and used for extraction and optimization of the methodology.

3. Results and Discussion

The proposed method proved to be quite effective in the simultaneous extraction of the studied veterinary drugs, since average recoveries were higher than 90%, excellent accuracy (<20%) and low detection and quantification limits (Table 1).

Table 1:

Veterinary Drug	**cyp**	**chlorpy**	**amitraz**	**dichlorvos**	**epri**	**fluazuron**
Detection limit ($\mu g\ L^{-1}$)	**6.8**	**18.7**	**19.5**	**5.9**	**8.4**	**9.7**
Quantification limit ($\mu g\ L^{-1}$)	**8.7**	**27.0**	**27.7**	**7.7**	**10.5**	**11.4**

cyp = cypermethrin; chlorpy = chlorpyrifos; epri = eprinomectin

4. Conclusions

The QuEChERS methodology, combined with gas chromatography coupled to mass spectrometry, provides a qualitative and quantitative extraction and analysis of the studied veterinary drugs with high efficiency and low detection, being a simple and easily applied method for the development of routine analyzes without control quality of food.

5. References

[1] CODEX ALIMENTARIUS Commission. Joint FAO/WHO Food Standards Programme, 2002.
[2] WANG, Y.; LIU, Z.; REN, J.; GUO, B. *Foodborne Pathogens and Disease*, **12**(8), (2015) p. 693.