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Determination of antimicrobial residues in milk samples

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INTRODUCTION:

In farming activities, antimicrobials are widely used as a therapeutic measure in the treatment of bacterial infections, disease prophylaxis and as a growth promoter (food additives) (Marshall & Levy, 2011). The presence of antimicrobial residues in milk may represent risks for human health, namely allergic reactions on hypersensitive individuals, or even cause problems indirectly by development of resistant strains of bacteria (Tillotson, Doern, & Blondeau, 2006).

OBJECTIVES:

This study aims the development of new clean-up and pre-concentration methods for milk samples, through a solid-phase extraction (SPE), in order to perform a better identification and quantification of antimicrobials by High-Performance Liquid Chromatography (HPLC).

MATERIALS AND METHODS:

The extraction method of the antibiotic residues involves the deproteinization of 27 spiked milk samples and their centrifugation, followed by a solid-phase extraction (SPE). Antimicrobial residues were, then, identified and quantified by HPLC-DAD.

RESULTS AND DISCUSSION:

The recovery percentage for ciprofloxacin was $94.54 \pm 6.90\%$. For sulfamethoxazole, the recovery percentage was $93.18\% \pm 5.60\%$. It was possible to identify the presence of sulfamethoxazole in one of the samples. The limits of detection (LD) were in the range of 2.43ng/mL and 162ng/mL and the limits of quantification (LQ) were ranging between 7,36 ng/mL and 492 ng/mL, which means that the developed new methods have LD and LQ below the maximum residue limits (MRLs) ruling in the European Union for five antimicrobials tested, in milk (Commission Regulation (EU) No 37/2010, OJ L15,20.1.2010, p11).

Table 1 - Recovery percentage of antimicrobial residues, using the SPE-HPLC-DAD methods

| Antimicrobial | Recovery (%) |
|------------------|--------------|
| Ciprofloxacin | 94.54 |
| Sulfamethoxazole | 93.18 |

CONCLUSION:

The combination of the proposed methods of clean-up and pre-concentration by SPE and multi-residue HPLC-DAD allows, therefore, the detection and quantification of antimicrobial residues in milk, making this an important and useful alternative in quality control process for the food industry and other relevant areas.

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