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Analytical Methods

A chemiluminescence method for screening of fluoroquinolones in milk samples based on a multi-pumping flow system

Christina Vakh[a,*], Aleksei Pochivalov[a], Stanislawa Koronkiewicz[b], Slawomir Kalinowski[b], Viktor Postnov[a], Andrey Bulatov[a]

[a] Institute of Chemistry, Saint-Petersburg University St. Petersburg State University, SPbSU, SPbU, 7/9 Universitetskayanab., St. Petersburg 199034 Russia
[b] University of Warmia and Mazury, Department of Chemistry, 10-957 Olsztyn, Poland

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Fleroxacin (PubChem CID: 3357)
Lomefloxacin (PubChem CID: 3948)
Norfloxacin (PubChem CID: 4539)
Ofloxacin (PubChem CID: 4583)
Ammonium cerium (IV) nitrate (PubChem CID: 9869224)
Terbium chloride hexahydrate (PubChem CID: 203091)
Sodium sulfite (PubChem CID: 24437)
Ammonium hydroxide (PubChem CID: 14923)
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Methanol (PubChem CID: 887)

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ABSTRACT

An automated and miniaturized chemiluminescence method for screening of fluoroquinolones in milk samples was proposed. The method was based on magnetic dispersive micro-solid phase extraction of analytes followed by the chemiluminescence determination of the total fluoroquinolones content using a multi-pumping flow system. In the developed method, Zr-Fe-C magnetic nanoparticles were used as an efficient sorbent for separation of fluoroquinolones from sample matrix. The chemiluminescence intensity obtained for Ce^{4+}-SO_3^{2-}-Tb^{3+} chemiluminescence system was greatly increased in the presence of the analytes. Under the optimal conditions, the detector response for fluoroquinolones was linear in the concentration ranges from $5{\cdot}10^{-9}$ to $1{\cdot}10^{-6}$ mol L^{-1} with respect to fleroxacin. The limit of detection, calculated from a blank test based on 3σ, was $2{\cdot}10^{-9}$ mol L^{-1} with respect to fleroxacin. The presented method demonstrated to be a good tool for available and cost-effective point-of-need screening fluoroquinolones in milk samples.

1. Introduction

Fluoroquinolones (FQs) are among widely used antibacterial agents for the treatment of bacterial infections in animals. FQs possess several useful characteristics that make them suitable for veterinary uses such as rapid and broad-spectrum antimicrobial activity (Kalunke, Grasso, D'Ovidio, Dragone, & Frazzoli, 2018). However, residues of antibiotics in food of animal origin are a matter of concern for public health. The presence of FQs in food can result in allergic or toxic reactions and antimicrobial resistance leading to potential hazard to human health (Jiang et al., 2015). To ensure the safety of food, efficient analytical techniques are required.

Variety of efficient analytical techniques based on the high-performance liquid chromatography (HPLC) with ultraviolet (UV) detection (Xu, Li, Li, Chen, & Xiao, 2018), fluorescence detection (FLD) (Xu et al., 2016; Pochivalov, Timofeeva, Vakh, & Bulatov, 2017; Timofeeva, Timofeev, Moskvin, & Bulatov, 2017) and mass-spectrometry detection (Tang, Yang, Tan, Luo, & Agric, 2009), as well as capillary electrophoresis with UV detection (Tian et al., 2014) have been developed for the FQ determination in different food samples (Table 1). In general, these techniques can be recognized as confirmatory techniques (Naik et al., 2017) due to these techniques being used for quantitative determination of FQs.

Recently, screening techniques have been of interest to semi-quantitative/quantitative determination of class of analytes at the level of interest in a given matrix with rapid analysis and low cost (Kalunke

* Corresponding author.
E-mail addresses: kristina-fulmes@mail.ru, k.vakh@spbu.ru (C. Vakh).

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Table 1
Figure of merits for the proposed method and those of other methods reported for the determination of FQs in milk samples.

Type of method	Method	Analytes	Sample preparation	Limit of detection	Linear range	RSD, %	Sample amount	Refs.
Confirmatory	HPLC-FLD	Ciprofloxacin enrofloxacin difloxacin sarafloxacin ofloxacin pefloxacin lomefloxacin	Magnetic graphene dispersive micro-solid phase extraction	0.05–0.3 $\mu g\ L^{-1}$	0.2–2000 $\mu g\ L^{-1}$	1.6–3.4	2 g	Xu et al. (2016)
Confirmatory	LC-ESI-MS/MS	Norfloxacin, ciprofloxacin, ofloxacin, enrofloxacin, rufloxacin	Solid-phase extraction	0.8922–0.9884 $\mu g\ kg^{-1}$	0.5–200 $\mu g\ kg^{-1}$	2.27–10.14	1 g	Pochivalov et al. (2017)
Confirmatory	HPLC-UV	Ciprofloxacin rufloxacin danofloxacin enrofloxacin	Hexafluoroisopropanol-induced salt-free catanionic surfactant coacervate extraction	0.3–1.4 $\mu g\ L^{-1}$	5–200 $\mu g\ L^{-1}$	–	5 mL	Xu et al. (2018)
Confirmatory	FLD	Norfloxacin, ofloxacin ciprofloxacin gatifloxacin	Salting-out assisted liquid–liquid ultrasonic extraction	9–16 $\mu g\ L^{-1}$	0.015–2.25 $mg\ L^{-1}$	0.54–2.48	10 mL	Xia, Yang, and Liu (2012)
Confirmatory	LC-UV	Ofloxacin ciprofloxacin lomeflosacin	Magnetic molecularlyimprinted polymers	1.8–3.2 $\mu g\ kg^{-1}$	5–500 $\mu g\ kg^{-1}$	9.5–12.5	10 g	Zheng et al. (2014)
Screening	Optical β-galactosidase-based screening assay	Ciprofloxacin	–	100 $\mu g\ kg^{-1}$	100–2000 $\mu g\ kg^{-1}$	–	–	Kalunke et al. (2018)
Screening	Bio-optical	Ciprofloxacin enrofloxacin	–	100 $\mu g\ kg^{-1}$	100–4000 $\mu g\ kg^{-1}$	–	–	Appicciafuoco et al. (2015)
Screening	MPFS-CL	Norfloxacin fleroxacin ofloxacin lomefloxacin	Magnetic dispersive solid phase microextraction	30 $\mu g\ kg^{-1}$ with respect to fleroxacin	100–15000 $\mu g\ kg^{-1}$ with respect to fleroxacin	5–8	1 g	This work

HPLC-FLD, HPLC-UV – high performance liquid chromatography with fluorescence and ultraviolet detection, respectively; CE-UV – capillary electrophoresis with ultraviolet detection; LC-ESI-MS/MS – liquid chromatography with electrospray ionization tandem mass spectrometric detection; MPFS-CL – multipumping flow system with chemiluminescence detection; RSD –relative standard deviation; LC-UV – liquid chromatography with ultraviolet detection; FLD – fluorescence detection.

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