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Article

Liquid chromatography-electrochemical detection for the determination of ethoxyquin and its dimer in pear skin and salmon samples

August 2017 · Talanta 177

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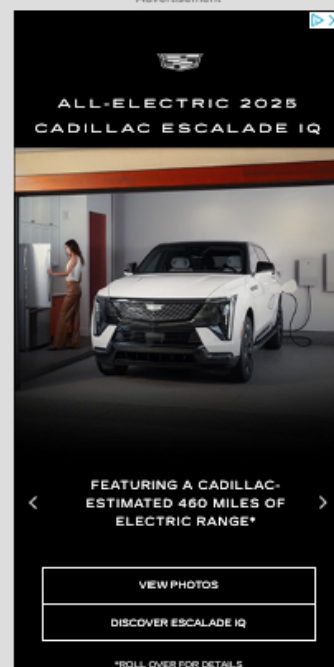


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Abstract

Ethoxyquin (EQ) is widely used as a synthetic antioxidant in animal feed, an anticaking agent in apples and pears and as a color preservative in some spices. Since the presence of EQ in food products could cause negative health effects it is necessary to develop reliable analytical methods to evaluate the risk of human exposure. In this work, a sensitive, selective and accurate method based on solid-liquid extraction followed by clean-up with solid sorbent and liquid chromatography-electrochemical detection analysis with boron doped diamond electrode (LC-EC) for the determination of ethoxyquin and its dimer (EQDM) in pear skin and salmon samples, was developed. The method was validated according to the European Commission guidelines. The main variables of extraction were accurately optimized. The amounts of solid sorbents for clean-up procedure were optimized by using experimental design. A Box-Behnken design to obtain the optimum conditions was applied. For validation, a matrix-matched calibration was established and a recovery assay with spiked samples was carried out. The limits of detection (LODs) found were 0.05 and 0.1 mg kg⁻¹ for EQ and its dimer, respectively. The precision (as relative standard deviation, RSD) was lower than 15% with recoveries of compounds close to 100% in spiked samples.

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... Currently, the methods for the determination of EQ mainly include thin layer chromatography [11], high performance liquid chromatography (HPLC) [12][13][14][15], gas chromatography (GC) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) [16]. The thin layer chromatography is cumbersome, time-consuming and not sensitive. ...

... HPLC and GC coupled with fluorescence detector, electrochemical detector or mass spectrometry have been widely used for the accurate determination of EQ. For example, Aoki et al. [14] developed a high performance liquid chromatography-fluorescence detection for EQ in animal-origin food with limit of detection (LOD) of 10 ng/g, while Rodríguez-Gómez et al. [15] developed a liquid chromatography-electrochemical detection for EQ in aquatic products with LOD of 5 ng/g. Moreover, several LC-MS/MS methods have been developed for the determination of EQ and its metabolites in aquatic animal tissues [17,18]. ...

... The limit of detection (LOD), limit of quantitation (LOQ), linearity, accuracy and precision were evaluated, respectively. The limit of detection (LOD) and limit of quantitation (LOQ) were calculated as the concentrations corresponding to three times and ten times peak areas (signal) as compared to chromatographic peak areas from blank sample (noise) [15,20]. The linearity was assessed by the calibration curves of EQ, EQDM and EQI, which were constructed by plotting the peak areas of the three target analytes against the corresponding concentrations of the three analytes and fitted by linear mode. ...

Analysis of ethoxyquin and its oxidation products in swine tissues by gas chromatography-tandem mass spectrometry for evaluating the feed-to-animal tissue transfer of ethoxyquin and its...

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... The adverse effects of EQ have been reported in animals and humans exposed to EQ, resulting in the establishment of the maximum limit of EQ in fish meal (i.e., 150 mg EQ/kg feed in the EU and Japan; 300 mg EQ/kg feed in Korea) to prevent inappropriate exposure to EQ (Bohne et al., 2008). However, high residues of EQ Merel et al., 2019;Nácher-Mestre, Ibanez, Serrano, Pérez-Sánchez, & Hernández, 2013;Portolés, Garlito, Nacher-Mestre, Berntssen, & Perez-Sanchez, 2017) and EQ metabolites, such as ethoxyquin dimer (EQDM) and quinine imine (2,6-dihydro-2,2,4-trimethyl-6-quinolone) (Bohne et al., 2008; EU Reference Laboratory for Single Residue Methods (EURL-SRM, 2016;Lundebye, Hove, Mage, Bohne, & Hamre, 2010;Negreira et al., 2017;Rodríguez-Gómez, Vandeput, Zafra-Gomez, & Kauffmann, 2018)) have been detected in fish meal. In particular, EQDM, the main metabolite of EQ showed high residue concentration in aquatic animals (Bohne et al., 2008;EURL-SRM, 2016;Merel et al., 2019;Negreira et al., 2017). ...

... The interday accuracy of EQ and EQDM was 75-114%, while the inter-day precision was 1.33-11.0%. Therefore, the validation results were favorable to analyze EQ and EQDM residues in various aquatic animal samples including eel, flatfish, loach, rockfish, salmon, and shrimp in comparison with those of some previous studies (EURL-SRM, 2016;Kranawetvogl & Elsinghorst, 2019;Rodríguez-Gómez et al., 2018). ...

... In all aquatic animal samples, EQ and EQDM were frequently detected and their residue concentrations ranged from 0.14 to 24.2 and 0.1-315 µg/kg, respectively (Table 2). In previous studies, analytical methods have been developed to determine EQ and EQDM in aquatic animals and fish meal (Bohne et al., 2008;Kranawetvogl & Elsinghorst, 2019;Nácher-Mestre et al., 2013;Negreira et al., 2017;Regueiro, Negreira, & Berntssen, 2016;Turnipseed et al., 2017), and the residue concentrations of EQ and EQDM have been investigated in aquatic animals (Lundebye et al., 2010;Nácher-Mestre et al., 2014;Portolés et al., 2017;Rodríguez-Gómez et al., 2018). One study identified no residue in salmon (Rodríguez-Gómez et al., 2018), but the number of samples (n = 6) was too small to be representative. ...

Residues and dietary exposure to ethoxyquin and ethoxyquin dimer in farmed aquatic animals in South Korea

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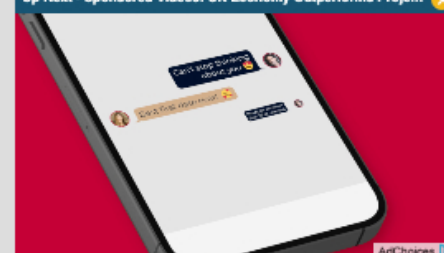
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... In this study, an electronic detection mechanism, known as single-electron effect, is proposed to detect and identify molecules dissolved in a liquid. To date, various methods for the identification of constituents of liquids, such as liquid chromatography [1,2] and microfluidics [3][4][5][6], have been used. Normally in these methods molecules are separated and then detected using ultraviolet, photoluminescence, fluorescence, electrochemical, refractive index, and electrical conductivity tests and procedures. ...

Ultra sensitive bio-detection using single-electron effect

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... Nonetheless, precision and trueness data indicated that the above-mentioned methodology was highly accurate. [179] The electrooxidation of natural sulfur-containing cofactor acid was compared on BDD electrode and GCE. In contrast to the GCE, the BDD electrode offered greatly improved performance in the cleaning step of the fouling, only rinsing with distilled water and immersing in the blank solution was required to achieve the same background current as before exposure to the α -lipoic acid. ...

A Review on Recent Advances in the Applications of Boron-Doped Diamond Electrochemical Sensors in Food Analysis

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... The quinoline-based, synthetic antioxidant ethoxyquin (6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline; CAS Number 91-53-2; EQ) possesses a broad range of applications because of its antioxidant capacity and its low production costs (Błaszczuk et al. 2013; Rodríguez-Gómez et al. 2018). It is globally used as an additive to retard oxidation in animal feed, as a preservative in dried forage crops and spices and was used as an anti-scalding agent in pears and apples in the past (Błaszczuk et al. 2013; Negreira et al. 2017). ...

Development of a human biomonitoring method for assessing the exposure to ethoxyquin in the general population

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... An accurate method for the determination of ethoxyquin and its dimer (EQDM) in pear skin and salmon samples was presented by Rodríguez-Gomez et al. (2018), using solid-liquid extraction followed by cleanup with solid sorbent and liquid chromatography-electrochemical detection analysis with boron-doped diamond electrode (LC-EC). The LOD for EQ and its dimer was obtained as 0.05 and 0.1 mg/kg, respectively. ...

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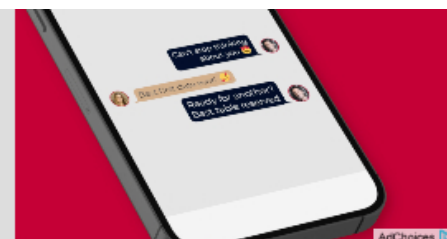
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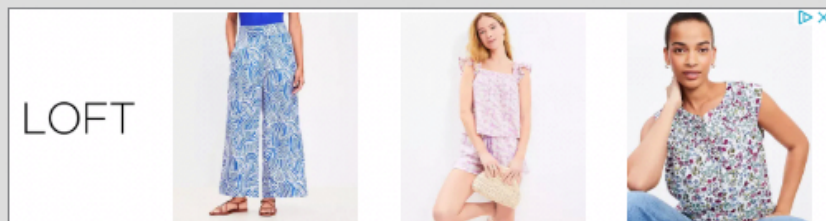
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Miyako Yamamoto

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Natural antioxidant extracts as food preservatives

October 2017 · Acta Scientiarum Polonorum, Technologia Alimentaria

Norma Francenia Santos Sánchez · Raul Salas-Coronado · Rogelio Valadez-Blanco · [...] · Paula Guadarrama

The food industry is becoming more specialized and processing methods are continuously being developed to meet consumer needs. Consumers demand products that are safe and preferably free of synthetic additives. These additives are associated with health effects, in most cases without reasonable justification. Consequently, consumers are looking for clearly labelled products that guarantee the ... [\[Show full abstract\]](#)

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Exploring solutions for healthy, safe, and sustainable fatty acids (EPA and DHA) consumption in The...

March 2019 · Sustainability Science

Anne Hollander · R. de Jonge · Sander Biesbroek · [...] · Michiel Zijp

Advisory bodies such as the World Health Organization and the Dutch Health Council (DHC) recommend including fatty fish in one's diet, based on the health benefits of their content of n-3 polyunsaturated fatty acids (eicosapentaenoic acid—EPA and docosahexaenoic acid—DHA) being, i.e., the reduction of the risk of fatal cardio vascular disease and stroke. These dietary advices on these fatty ... [\[Show full abstract\]](#)

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Mycotoxins in grains and feed: Contamination and toxic effect in animals

January 2015 · Biotechnology in Animal Husbandry

Jelka Pleadin

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