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RESEARCH PAPER

Rapid Analysis of Trace Salbutamol and Clenbuterol in Pork Samples by Mass Spectrometry

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Abstract: Salbutamol and clenbuterol as β-agonists are illegally added in pig feed, resulting in pork contaminated and even leading excessive excitability of athletes. Therefore, it has a great significance for establishing a new rapid detection method of salbutamol and clenbuterol in pork. In this study, internal extraction electrospray ionization mass spectrometry (iEESI-MS) technique was used for directly qualitative and quantitative analysis of salbutamol and clenbuterol in pork tissues without any sample pretreatment. The results showed that this method had a high sensitivity to salbutamol and clenbuterol analysis with detection limits of (LOD) 6.2 and 9.8 ng kg^{-1}, respectively, while the analysis time for detecting single sample and single index was less than 30 s. In a range of 0.01–1000 μg kg^{-1}, the logarithm of signal intensity (Y) and the logarithm of concentration (X) had a good linear relationship. This method had many advantages such as rapid analysis, low sample consumption and high sensitivity, which was ideal for the rapid detection of trace salbutamol and clenbuterol.

Key Words: Internal extraction electrospray ionization; Salbutamol; Clenbuterol; β-Agonists; Organizational analysis

1 Introduction

"Lean meat essence" refers to a class of β-agonist compounds that have similar structure, the representatives are salbutamol and clenbuterol which generally used for treating shock, asthma and other diseases on clinical[1]. Because of the ability of inhibiting animal fat's synthesis, promoting lean growth and redistribution the livestock ratio between fat and lean, β-agonist compounds were often illegally added in livestock feed to improve the efficiency of livestock production. According to the Ministry of Agriculture Bulletin No. 235, salbutamol and clenbuterol are forbidden in food (meat). Conventionally, the level of β-agonist in Pork was in the range of 0.1–100 μg kg^{-1}. Human consumption of meat products that containing lean meat essence would cause high blood pressure, increase heart rate and other reactions, even affect the results of sports, long-term absorb also bring cardiovascular system serious harm and life-threatening[2]. Therefore, it is significant for food safety to understand and master the dangers of lean meat essence, establish a fast and accurate detection method for β-agonist in succulent food[3–6].

Up to now, lots of methods have been developed for detection of β-agonist in animal tissues, including colloidal gold immune chromatography (GICA)[7,8], enzyme-linked immunosorbent assay (ELSA)[9,10] and surface plasma resonance biochip[11,12], gas chromatography-mass spectrometry (GC-MS)[13,14], liquid chromatography-mass spectrometry (LC-MS)[15–17], and high performance liquid chromatography (HPLC-MS)[18,19]. GICA method are always used for large scale sample screening, while it's just suitable for preliminary determination as the test results are easy affected by environment or human factors. In spite of its high

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sensitivity, ELISA is not an ideal method because of its false positive results with poor reproducibility and specificity. HPLC-MS and GC-MS method have high sensitivity and accuracy, but they have a time-consuming and laborious sample pretreatment process, which are difficult to meet the requirements of large scale samples detection.

The internal extraction electrospray ionization mass spectrometry (iEESI-MS) technique could directly import the extraction agent inside tissue sample to achieve small molecule component from internal tissue samples for direct mass spectrometric analysis[20–23]. Herein, based on iEESI-MS technique, we developed an iEESI-LTQ-MS detection platform to directly analyze salbutamol and clenbuterol in succulent food. The results indicated that iEESI-MS was a rapid and accurate qualitative and quantitative method for salbutamol and clenbuterol detection. Furthermore, we successfully applied this detection method for real pork samples analysis.

2 Experimental

2.1 Equipment, reagents and materials

The iEESI-MS experiments were carried out by a homemade internal extractive electrospray ion source coupled with a linear trap quadruple (LTQ) mass spectrometer (Thermo Scientific, San Jose, USA). The fused silica capillary (0.10 mm i.d., 0.15 mm o.d., Agilent Technologies Co., Ltd., USA) was used to inject extractive solution into tissue samples. Methanol (HPLC grade) was purchased from ROE Scientific Inc. (Newark, U.S.A). The deionized water used for the experiments was provided by ECUT chemistry facility at laboratory.

The standards of clenbuterol (batch number 0043-5479) and salbutamol (batch number 103578-453245) were purchased from Laboratorien Berlin-Adlershof GmbH Company.

The standard stock solution of clenbuterol and salbutamol (10 mg L^{-1}) was prepared by dissolving the standards in distilled water, followed by storing at –18 ºC.

Standard solution with a serials concentration of 0.01, 0.1, 1, 10, 100 and 1000 μg L^{-1} were prepared by diluting the standard stock solution.

The pork samples were provided by the Sports Science Institute of Jiangxi Province, China. These pork samples had been detected by national standard method and the results showed there were no salbutamol, clenbuterol and other β-agonists in the pork.

2.2 Experimental methods

The iEESI-MS schematic was shown in Fig.1. The capillary inserted into the tissue sample, the distance of capillary tip inside the sample apart from the sample tip was 2 mm, and the distance between the sample tip and the mass spectrometer inlet was 4–5 mm. The mixture of methanol and water (1:1, *V*/*V*) was employed as extraction agent, which was injected from the capillary directly into the interior of pork sample at a flow rate of 1 μL min^{-1}.

The analytes were extracted by the injected extractive solution and carried along the electric field gradient inside the bulk volume of the sample. Fine charged droplets containing analytes were generated on the apex of analyzed tissue sample. After desolvation, the ions of analytes were brought into mass spectrometer for interrogation. The temperature of MS capillary inlet was typically set at 150 °C with tube lens voltage at 100 V and capillary voltage at 10 V. The voltage used for spray ionization was 5 kV under the positive ion detection mode. Tandem mass spectrometer for structural confirmation was carried out using collision induced dissociation (CID) experiments. An isolation window of 1.5 Da and normalized collision energy of 16%–25% were chosen. Furthermore, other parameters were set as the default values of instrument, and no further optimization was performed.

2.3 Qualitative and quantitative analysis

First, the pork sample without clenbuterol and salbutamol identified by Standard addition method[24,25] and iEESI-MS methods was used as blank sample. Secondly, according to the standard addition method, blank pork samples were cut into same small strips (20 mm × 2 mm × 2 mm/1.55 mg), and then immersed in salbutamol and clenbuterol standard solution. After soak for 10 h, iEESI-MS were used for qualitative analysis via characteristic fragment of salbutamol and clenbuterol. Finally, according to the standard addition method and two characteristic fragment ion signal of target ion, a linear correlation curve between the logarithm of analyte characteristic fragment ion intensity (*Y*) and the logarithm ofspiked concentration (*X*) was drawn in the range of 0.01–1000 μg kg^{-1} for the complete quantitative analysis.

3 Results and discussion

3.1 Qualitative analysis of salbutamol and clenbuterol in pork

To realize qualitative analysis of salbutamol and clenbuterol in pork samples, the pork samples adsorbed salbutamol and clenbuterol were used for iEESI-MS analysis, and chemical fingerprint spectra was shown in Fig.2. Under the experimental

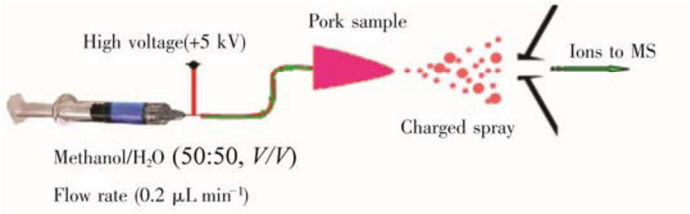

Fig.1 Schematic diagram of iEESI-MS

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