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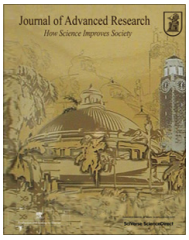

SHORT COMMUNICATION

Residue analysis of orthosulfamuron herbicide in fatty rice using liquid chromatography–tandem mass spectrometry

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ABSTRACT

In the present study, orthosulfamuron residues were extracted from fatty (unpolished) rice and rice straw using a modified QuEChERS method and analyzed using liquid chromatography–tandem mass spectrometry. The matrix-matched calibration was linear over the concentration ranges of 0.01–2.0 mg/kg with determination coefficient (R^2) $\geqslant$ 0.997. The recovery rates at two fortification levels (0.1 and 0.5 mg/kg) were satisfactory and ranged between 88.1% and 100.6%, with relative standard deviation (RSD) $<$8%. The limit of quantitation, 0.03 mg/kg, was lower than the maximum residue limit, 0.05 mg/kg, set by the Ministry of Food and Drug Safety in the Republic of Korea. The developed method was applied successfully to field samples harvested at 116 days and none of the samples were positive for the residue.

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Introduction

Rice is one of the most consumed grains in the world. As its consumption has been increased in accordance with population growth, the use of pesticides, including pre- and post-emergence herbicides, insecticides, and fungicides, raised consequently to improve its production during the various stages of cultivation [1]. In the Republic of Korea, the main pesticides employed are herbicides (before rice transplantation) and fungicides or insecticides, depending upon the conditions (rain or insect attack). After harvesting, several steps are needed to produce the final marketing products; including paddy, brown, and white rice [2]. As the nutritional components are mainly exit in the germ and bran layers, the nutritional quality of various rice forms is diverse. In rice, the high and low molecular weight components are either enhancing the response or interfering with compound identification and quantitation in chromatographic analysis [3]. Rice straw, which is separated from

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rice grains by a combine tractor, was used as a feed for livestock production in the Republic of Korea. So far, little is known on which proportion of the pesticide originally applied in the field could be found in various types of rice and rice straw, which necessitates residue analysis.

Orthosulfamuron, one of the sulfonylureas (SUs), is a selective and systemic early post-emergence herbicide, which is absorbed by foliage and root and translocated apoplastically and symplastically into the plants. It inhibits acetolactate synthase (ALS) enzyme, which catalyzes the first committed step in the branched-chain amino acids (valine, isoleucine, and leucine) biosynthetic pathway and hence stop cell division and plant growth [4]. ALS-inhibiting herbicides are important in all crops due to their efficiency at low rates, flexibility of use, favorable environmental profile, and low mammalian toxicity [5] compared to other alternative herbicides [6]. Orthosulfamuron controls the post-emergence of annual and perennial broad leaves weeds, sedges and barnyard grass, in dry and water-seeded and transplanted rice [4]. It is therefore, possible that the residues of this herbicide may contaminate and be accumulated in grains, including barely, wheat, rice, and soybeans [7]. Because of their low application rates and thermal instability, the determination of SU residues continues to present an analytical challenge, which promotes the development of sample pre-treatment and analytical detection [8,9].

High-performance liquid chromatography (HPLC) with an UV or diode array, mass spectrometry (MS) or tandem mass spectrometry (MS/MS) detection system was the most common approach for the determination of SUs in grains because of their polar characteristic, low volatility, and thermal instability [8,10–12]. In the literature survey, orthosulfamuron has been analyzed neither as a single nor among multiple residue analysis in grains. In this study, a simple liquid chromatography–tandem mass spectrometry (LC/MS/MS) method was established to detect the residues of orthosulfamuron in brown fatty (unpolished rice) and rice straw using the QuEChERS as an extraction method.

Experimental

Chemicals

Orthosulfamuron of purity 99.34% was kindly donated from KYUNG NONG CO. LTD. (Seoul, Republic of Korea). HPLC-grade acetonitrile (MeCN) was supplied by Burdick and Jackson (Ulsan, Republic of Korea). Sodium acetate (NaOAc, purity 98.0%) and anhydrous magnesium sulfate ($MgSO_4$, purity 99.5%) were provided by Junsei Chemical Co. Ltd. (Kyoto, Japan). Sodium chloride (NaCl, purity 99.5%) was obtained from Merck (Darmstadt, Germany). Primary secondary amine (PSA) and C_{18} were supplied by Agilent Technologies (Palo Alto, CA, USA). All other chemicals were of analytical and/or HPLC grade.

Matrix-matched calibration

Orthosulfamuron stock solution was prepared in MeCN at a concentration of 1000 μg/mL. A working solution of 10 μg/mL was prepared by diluting the stock solution with blank rice or rice straw extracts, which were confirmed previously to be free of the target analyte. A matrix-matched calibration was prepared by mixing the working standard solution with blank sample extracts to reach a concentration range of 0.01–2 mg/kg. Stock solution was stored at −26 °C in a dark amber bottle, whereas calibration standards were kept at 4 °C.

Field trails

Experimental field trials were carried out at Chonnam National University, Gwangju, Republic of Korea. The on-farm research product, tablet for direct application (DT) of 1.5% orthosulfamuron, was applied to two paddy field plots at two different doses on fifteen days after transplanting the rice seedlings. The first plot received the herbicide at the recommended dose of 500 g/10 a (a.i. [active ingredient] 0.0075 kg/10 a) (T1) and the second one was sprayed with double the recommended dose 1000 g/10 a (a.i. 0.015 kg/10 a) (T2), along with the untreated control (T3). Representative rice (800 g) and rice straw (500 g) samples were collected at harvest (116 days) from the treated and untreated plots. The collected rice and straw were dried to approximately 12% moisture content in a drying room. Subsequently, the dried grains were incompletely husked to make unpolished rice. Unpolished rice grains and straw samples were then ground using a mechanical grinder and used for residue analysis. The samples were stored at −20 °C until analyzed.

Sample preparation

Sample preparations for fatty rice and rice straw were based on the acetate-buffering QuEChERS method [13] following minor modifications. At no point the extraction conditions were optimized. Rather, the experimental variables including solvents, salting out agents, and cleanup procedure were predicated based on our experience.

Unpolished fatty rice

Ten grams of well-ground rice sample was placed into a 50-mL Teflon centrifuge tube. Ten milliliters of distilled water was added to the tube and then vortex-mixed for 1 min. Afterward, MeCN (20 mL), NaCl (2 g), $MgSO_4$ (4 g), and NaOAc (1.5 g) were added to the mixture and shaken by a vortex-mixer for 2 min. The extract was centrifuged for 5 min at 5000 rpm and 5 °C, and the supernatant was aspirated into a 1.5-mL microcentrifuge tube that contained 0.03 g of both PSA and C_{18}. Following shaking for 1 min, the tubes were centrifuged for 5 min at 5000 rpm. The purified extract was subjected to filtration using a polytetrafluoroethylene (PTFE) membrane filter (0.2 μm, ADVANTEC®, Toyo Roshi Kaisha, Ltd., Tokyo, Japan) and 1 mL of the extract was ready for analysis using LC/MS/MS.

Rice straw

Five grams of sample was placed into a 250-mL Erlenmeyer flask, to which MeCN (100 mL), distilled water (50 mL), NaCl (10 g,) $MgSO_4$ (10 g), and NaOAc (5 g) were added. After vigorous shaking for 1 min, the mixture was kept in a shaking incubator for 30 min. The mixture was vacuum filtered through a filter paper (Whatman No. 6, GE Healthcare Co.

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