UNIVERSITI PUTRA MALAYSIA

A TRACEABILITY SYSTEM FOR SULFONAMIDE RESIDUES IN CHICKEN MEAT-BALLS

ISMAIL FITRY BIN MOHAMMAD RASHEDI

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

A TRACEABILITY SYSTEM FOR SULFONAMIDE RESIDUES IN CHICKEN MEAT-BALLS

By

ISMAIL FITRY BIN MOHAMMAD RASHEDI

December 2008

Chairman : Jinap Binti Selamat, PhD

Faculty : Food Science and Technology

Studies on the effect of different temperature and time for boiling and deep-frying, and power and time for micro-waving on sulfonamides (SAs) residues i.e. sulfadiazine (SDZ), sulfamethazine (SMZ), sulfamethoxazole (SMX), and sulfaquinoxaline (SQX) in chicken meat-balls were carried out. The purpose was to use the data collected to develop a traceability system model for SAs residues. Blank chicken meat was fortified with the mixed SAs standard and chicken meat-balls were produced. The cooking methods were conducted consecutively from boiling, deep-frying and micro-waving.

Several methods were evaluated for the analysis of the SAs residues in the chicken meat-balls, which resulted in acceptable range of recoveries,

from 82.0 to 98.9 % and RSDs from 0.7 to 7.6 %. The chromatogram of both the raw chicken meat and the chicken meat-balls showed no interfering peaks from other compounds present in the SAs analysis.

Boiling of chicken meat-balls showed that temperature needs to be at 100 °C to obtain significant reduction ($p<0.05$) compared to 80 and 90 °C. A significant reduction ($p<0.05$) was observed at boiling for 6 min. In deep-frying study, significant reduction ($p<0.05$) of SAs concentrations was observed against the control for temperature factor. Significant reductions ($p<0.05$) of SAs concentrations among all the treatments and against the control were observed for the time factor. In micro-waving, the power and time factor showed various effects on reducing SAs residues in chicken meat-balls. Pearson correlation coefficient showed that time had greater effect compared to temperature on the reduction of SAs concentration upon boiling and deep-frying. Power had greater effect on micro-waving process compared to time. The SAs concentration was reduced at the same time the internal temperature increased during boiling and deep-frying. There were negative correlations for deep-frying and micro-waving between the SAs concentration and weight changes parameters. Analyses on the Reducing Half Life (RHL) showed that microwaving processes had the shortest RHL of SAs followed by deep-frying and boiling processes. The RHL for the three cooking methods were from 0.9 to 83.9 min.

The traceability system model was developed by using the Visual Basic 6.0 software with the percentage of reduction and linear regression were applied as the main method for detection of SAs residues. The first detection method for the traceability system model was based on the actual percentage reductions data of SAs residues. The percentage of reductions for every SAs at different cooking method were recorded earlier from the chemical analysis and calculated to be keyed in into the system. The second detection method to track and trace the SAs residue was by using the linear regressions developed from the result of effect of cooking methods. The equations of the linear regressions were determined from the data collected and inserted into the traceability system to be used for prediction of the SAs amount. The traceability system can be used to monitor the MRLs of the SAs before and after the processing, which will make the screening and monitoring work easier for the regulatory agencies, industry workers and consumers. The system could be used to gain the confidence on the safety of the chicken meat-balls from SAs residue and also could be applied for other type of veterinary drugs and pesticides residues.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi syarat untuk Ijazah Master Sains

TRACEABILITY RESIDU SULFONAMIDE DI DALAM BEBOLA DAGING AYAM

Oleh

ISMAIL FITRY BIN MOHAMMAD RASHEDI

December 2008

Pengerusi : Jinap Binti Selamat, PhD

Fakulti : Sains dan Teknologi Makanan

Kajian terhadap kesan suhu dan masa yang berbeza untuk merebus dan menggoreng, dan kuasa dan masa untuk penggunaan ketuhar gelombang terhadap residu sulfonamides (SAs) iaitu sulfadiazine (SDZ), sulfamethazine (SMZ), sulfamethoxazole (SMX), and sulfaquinoxaline (SQX) di dalam bebola daging ayam telah dijalankan. Tujuannya ialah untuk menggunakan data yang diperolehi untuk membangunkan sebuah model sistem ’traceability’ untuk residu SAs. Daging ayam tanpa residu telah ditambah dengan campuran ’standard’ SAs dan bebola daging ayam telah dihasilkan. Kaedah-kaedah memasak dijalankan berturutan dari merebus, menggoreng dan menggunakan ketuhar gelombang.

Beberapa kaedah telah diubahsuai dan digunakan untuk menganalisa residu SAs di dalam bebola daging ayam, di mana keputusan pemerolehan semula berada di dalam kadar yang dibenarkan iaitu dari 82.0 kepada 98.9 % dan RSDs dari 0.7 kepada 7.6 %. Kromatogram untuk kedua-dua daging ayam mentah dan bebola daging ayam menunjukkan tiada gangguan oleh bahan lain terhadap analisa SAs.

Merebus bebola daging ayam menunjukkan suhu diperlukan pada tahap maksimum (100 °C) untuk memperolehi pengurangan yang bererti ($p<0.05$) berbanding suhu 80 dan 90 °C. Pengurangan yang bererti ($p<0.05$) boleh dilihat pada masa ujikaji 6 min. Untuk proses menggoreng, pengurangan bererti ($p<0.05$) kepekatan SAs terhadap sampel kawalan telah diperolehi untuk faktor suhu. Sementara itu, pengurangan bererti ($p<0.05$) kepekatan SAs di antara semua ujikaji dan terhadap sampel kawalan telah diperolehi untuk faktor masa. Di dalam penggunaan ketuhar gelombang, faktor kuasa dan masa menunjukkan kesan yang pelbagai terhadap pengurangan SAs di dalam bebola daging ayam. Korelasi Pearson menunjukkan faktor masa mempunyai kesan yang lebih terhadap pengurangan SAs untuk proses merebus dan menggoreng. Faktor kuasa telah memberi kesan yang lebih terhadap proses ketuhar gelombang berbanding faktor masa. Kepekatan SAs mengurang pada masa yang sama suhu dalaman meningkat untuk proses merebus dan menggoreng. Terdapat perhubungan yang negatif untuk proses menggoreng dan ketuhar gelombang bagi parameter di antara kepekatan SAs dan perbezaan berat. Analisa terhadap kemusnahan separuh menunjukkan proses ketuhar gelombang mempunyai masa kemusnahan separuh yang paling singkat diikuti oleh proses menggoreng dan merebus. Kemusnahan separuh untuk ketiga-tiga cara masakan ini ialah di antara 0.9 ke 83.9 min.

Model sistem ’traceability’ telah dibangunkan menggunakan perisisan Visual Basic 6.0 dengan peratusan pengurangan dan regrasi lurus diaplikasikan sebagai kaedah utama untuk mengesan residu SAs. Cara pengesanan yang pertama untuk model sistem ’traceability’ adalah berdasarkan data sebenar peratusan pengurangan residu SAs. Peratusan pengurangan untuk setiap SAs pada kaedah memasak yang berbeza telah dicatatkan terlebih dahulu dari analisa kimia dan dimasukkan ke dalam sistem. Cara pengesanan kedua untuk menjejak residu SAs ialah dengan menggunakan regrasi lurus yang dihasilkan dari keputusan kajian kesan memasak. Persamaan regrasi lurus diperolehi dari data yang dikumpulkan dan dimasukkan ke dalam sistem ’traceability’ untuk meramal jumlah residu SAs. Sistem ’traceability’ ini boleh digunakan untuk memantau Had Residu Tertinggi (MRL) untuk SAs sebelum dan selepas pemprosesan, di mana ia akan memudahkan kerja saringan dan pantauan oleh pihak berkuasa, pekerja industri dan pengguna. Sistem ini boleh digunakan untuk mendapatkan kepercayaan terhadap keselamatan bebola daging ayam dari residu SAs dan juga boleh diaplikasikan terhadap residu ubat-ubatan veterinar dan pestisid yang lain.

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I certify that a Thesis Examination Committee has met on 1 December 2008 to conduct the final examination of Ismail Fitry bin Mohammad Rashedi on his thesis entitled "A Traceability System for Sulfonamide Residues in Chicken Meat-Balls" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Member of the Thesis Examination Committee were as follows:

SHARIFAH KHARIDAH SYED MUHAMAD, PhD
Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

SUHAILA MOHAMED, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Internal Examiner)

PAUZI ZAKARIA, PhD
Associate Professor
Faculty of Environmental Studies
Universiti Putra Malaysia
(Internal Examiner)

AYUB MOHD YATIM, PhD
Associate Professor
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
(External Examiner)

BUJANG KIM HUAT, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 19 February 2009

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science.

The members of the Supervisory Committee were as follows:

JINAP SELAMAT, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

JAMILAH BAKAR, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

SALEHA ABDUL AZIZ, PhD
Professor
Faculty of Veterinary Medicine
Universiti Putra Malaysia
(Member)

HASANAH GHAZALI, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 10 April 2009

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that is has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

ISMAIL FITRY BIN MOHAMMAD RASHEDI

Date:

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LIST OF ABBREVIATIONS

SAs	Sulfonamides
SDZ	Sulfadiazine
SMZ	Sulfamethazine
SMX	Sulfamethoxazole
SQX	Sulfaquinoxaline
HPLC	High Performance Liquid Chromatography
DAD	Diode Array Detector
ANOVA	Analysis of Variance
LOD	Limit of Detection
LOQ	Limit of Quantification
r^2	correlation of determination
RHL	Reducing Half Life
GUI	Graphical User Image

Chapter I

Introduction

Sulfonamides (SAs) are used as antimicrobial agent to treat certain diseases and as growth promoter in animal husbandry. The over dosage and slaughter of animal before withdrawal period can lead to the presence of SAs residue, which are known to cause health problem in human. The Maximum Residue Limit (MRL) for SAs in Malaysia is set at 100 µg/kg of foodstuffs such as meat (Food Regulation, 1985); however violation still occurs and raised food safety issue among consumers and industries.

The growing concern on safety level of the meat due to SAs residues lead the monitoring and screening to be carried out more frequently by the regulatory officials. However, the problem with manpower, equipment and time consuming for testing procedure have restricted the monitoring process. The demand for traceability to be incorporated in meat and meat-based products has increased the responsibilities of these officials. The main problem remains on how SAs residue can be tracked and traced in meat and meat-based products.

Traceability system has been widely implemented in European countries and United States as tool to maintain the quality and control the safety of food. According to ISO 8402:1994 (1994), traceability is the ability to trace the history, application or location of an entity, by means of recorded information. Different approaches on the definition of the traceability have been reported based on the substance involved. The analytical methodology and computer technology implemented to perform the traceability system also varies according to the type of food and conditions.

Animal feed and its ingredients is the subject of traceability since it goes through the food chain from animal to food product (Schwägele, 2005). SAs added into the feed are considered as one of the ingredients. Chemical analysis on the detection of SAs residues in raw meat sample to control the MRL is not sufficient since the residues can still be carried over in the processed and cooked products made from such meat. Thus, SAs should be traceable from raw meat to processed and cooked product along the entire food chain.

Cooking may be one of the ways to ensure foods are safe from the risk of SAs residues. It has been reported that some cooking procedures can reduce the SAs residues in meat (Papapagiotou *et al*., 2005) with the different cooking methods produce different concentration of SAs residues

(Furusawa & Hanabusa, 2002). However, no attempt has been made to use data from the cooking methods to develop traceability system.

A traceability system model focusing on SAs residues can be developed from raw chicken meat-balls to end product (cooked chicken meat-balls). This model will include tracking forward the location and concentration of the SAs residues at different stages of meat processing, i.e. after precooked in the factory and after cooking and reheating at home. The model can also be used to trace backward from stages of reheating, cooking, precooked and raw meat.

The objectives of this research were:

1) To evaluate several methods to be used for simultaneously detection of sulfonamide residues in raw chicken meat, processed chicken meat-ball dough and cooked chicken meat-ball.

2) To determine the effect of different time (min) and temperature (°C) of boiling and deep-frying, and different time (s) and power (watt) of micro-waving on sulfadiazine, sulfamethazine, sulmethoxazole and sulfaquinoxaline residues in chicken meat-balls.

3) To develop a traceability system model for SAs residue in raw chicken meat, chicken meat-balls dough, boiled, deep-fried and micro-waved chicken meat-balls based on the percent of concentration reduction and linear regression reduction of SAs residue by using Visual Basic 6.0 software.