

농식품 분야

PF-01

Determination of Total Arsenic, Inorganic Arsenic and Cadmium in Powdered Rice FAPAS 07187 using HPLC-ICP/MS and ICP/MS (HPLC-ICP/MS 와 ICP/MS를 이용한 FAPAS 07187 쌀의 총비소, 무기비소, 카드뮴 측정)

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Metal(loid)s such as environmental contaminants affect food safety. Metal(loid)s exist trace extremely in agriculture products. Therefore it is important to analyze accurately about trace these metals.

High resolution techniques and quality controls are needed to analyze these materials. In order to achieve quality results, standardized methods and techniques are required. So ISO(International Organization for Standardization) of laboratory proficiency testing is determination of laboratory testing performance by means of inter-laboratory comparison. The values have been derived as a consensus of a number of laboratories using a variety of methods. Proficiency tests were applied to analyze the metal(loid)s [inorganic arsenic (As), total As and cadmium (Cd)] in powdered rice FAPAS 07187 (Food Analysis Performance Assessment Scheme; February-April 2013). The rice powder sample was extracted with 1% HNO₃ for inorganic As for microwave-assisted extraction using water at 95°C for 90 min and their analysis by HPLC/ICPMS. Also, HNO₃ for total As and Cd using microwave-assisted extraction using at 200°C for 50 mins and analysis by ICP/MS. Validation check was performed for accuracy using CRM (NMIJ CRM 7503-a) for inorganic As, and CRM (IRMM-Rice CRM) for total arsenic and cadmium. The result were satisfactory. FAPAS 07187 the z-scores of inorganic As, total As and Cd in powdered rice were -1.8, -0.9 and 0.2 (|z| < 2, respectively).

Key words : Total arsenic, Inorganic Arsenic, Cadmium, FAPAS

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Validation of Analytical Method to Determine the Contents of Alternaria Toxins in Cereals

(곡류 중 알테나리아독소 시험법 밸리데이션)

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Alternaria toxins have been found to be natural food contaminants in grains, sunflowers seeds, and some visibly decayed fruits in many countries. Their natural occurrence in cereals has been reported in different countries. Several studies have reported the relevance of this genus in crop. However, there are no studies on the method validation of alternaria toxin in cereals in our country. A simple and sensitive analytical method based on HPLC with PDA and mobile phases using 0.1% phosphoric acid and acetonitrile was developed for simultaneous determination of alternaria toxins. A method validation for the determination of alternaria toxins in cereals was validated. In short, the method is as follows : A test portion of a sample is extracted with a mixture of methanol/water(90/10, v/v). This raw extract is then diluted, filtered, and applied to an SPE column. After washing and elution with acetonitrile, the elute is evaporated to dryness. After toxins in the dry residue in mobile phase are injected into a high performance liquid chromatography, and detected and quantified by PDA. For alternaria toxins in the cereals, Recovery test, calibration curves(Linearity), LOD and LOQ were successfully confirmed and Reproducibility relative standard deviations(RSD_R) and Repeatability relative standard deviations(RSD_r) for cereals samples were below 15% for the spiked cereals at 100 ug/kg. Since all these parameters lie well within the acceptable range set forth in EU mycotoxin method validation legislation. This method is The proposal method is sensitive, repeatable and rapid enough to apply to official routine inspection of agricultural products including cereals.

Key words : Mycotoxin, Alternaria toxins, Cereal, Validation

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PF-03

BioConcentration Factor (BCF) of Heavy Metals from Agricultural Soil to Agricultural Products

(농작물 재배지 토양 내 비소, 카드뮴 및 납의 농산물로의 생물농축계수 산출)

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The transfer of heavy metals from soil to plants under conditions of soil pollution is a process of significant importance, because it is considered as the main pathway of human exposure to heavy metal through foodchain. BioConcentration Factor (BCF) is an indicator of heavy metal mobility in soils and a factor that quantifies bioavailability of heavy metals to agricultural products. BCF is defined as ratio of heavy metal concentration in vegetable (dry weight) to that in soil (dry weight). This study was carried out to measure the transfer ability of Arsenic(As), Cadmium(Cd), and Lead(Pb) from soil to agricultural products. We investigated heavy metals (As, Cd and Pb) concentrations in 8 agricultural products (sesame leaf, spinach, chinese cabbage, lettuce, chinese chive, ginseng, deodeok, balloonflower) as well as in cultivated soils. BCF for heavy metals from soil to vegetables for three elements including As, Cd, and Pb was calculated based on total soil concentration. The result showed that BCF values was calculated average 0.006~0.026 (As), 0.015~1.395 (Cd), 0.015~0.076 (Pb). The mean BCF value was the highest as ginseng 0.026 in As, balloonflower 1.395 in Cd, chinese chive 0.076 in Pb. It were dependent on the vegetable type and species. Root vegetables are showed higher than leafy vegetables in As and Cd. We found that BCF are useful in predicting the uptake of heavy metals. Further study of uptake and accumulation mechanism of toxic metals by agricultural products will be required to assess the human health risk associated with soil contamination.

Table 1. BioConcentration Factor (BCF) of As, Cd, and Pb from total in soil to eight kinds of agricultural products

Toxic element	Sesame leaf	Spinach	Chinese cabbage	Lettuce	Chinese chive	Ginseng	Deodeok	Balloonflower
As	0.020	0.007	0.009	0.022	0.017	0.026	0.010	0.006
	(0.009 ~ 0.038)*	(0.001 ~ 0.029)	(0.000 ~ 0.069)	(0.000 ~ 0.145)	(0.000 ~ 0.068)	(0.000 ~ 0.215)	(0.000 ~ 0.042)	(0.000 ~ 0.035)
Cd	0.015	0.526	0.068	0.739	0.133	1.175	1.250	1.395
	(0.000 ~ 0.090)	(0.095 ~ 2.813)	(0.000 ~ 0.228)	(0.070 ~ 2.001)	(0.000 ~ 0.786)	(0.000 ~ 19.146)	(0.149 ~ 16.503)	(0.000 ~ 5.738)
Pb	0.023	0.015	0.023	0.020	0.076	0.022	0.022	0.021
	(0.000 ~ 0.039)	(0.004 ~ 0.041)	(0.000 ~ 0.084)	(0.005 ~ 0.070)	(0.008 ~ 0.535)	(0.000 ~ 0.049)	(0.004 ~ 0.115)	(0.004 ~ 0.066)

* Mean (Min-Max)

Key words : BioConcentration Factor (BCF), Agricultural products, Heavy metals, Monitoring, Soils

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Isolation of Volatiles from *Nigella sativa* Seeds Using Microwave-Assisted Extraction: Effect of Whole Extracts on Canine and Murine CYP1A (마이크로파 추출법을 이용한 *Nigella sativa*중의 휘발물질 분리: 추출물이개와 쥐의 CYP1A에 미치는 영향)

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The volatile components of *Nigella sativa* seeds were isolated using microwave-assisted extraction (MAE) and identified using gas chromatography. Further investigations were carried out to demonstrate the effects of whole extracts on canine (dog) and murine (rat) cytochrome P450 1A (CYP1A). The optimal extraction conditions of MAE were as follows: 25 mL of water, medium level of microwave oven power and 10 min of extraction time. A total of 32 compounds were identified under the conditions using GC-FID and GC-MS. Thymoquinone (38.23%), p-cymene (28.61%), 4-isopropyl-9-methoxy-1-methyl-1-cyclohexene (5.74%), longifolene (5.33%), α-thujene (3.88) and carvacol (2.31%) were the main compounds emitted from *N. sativa* seeds. Various extracts including pure compounds, essential oil, nonpolar partition, relatively high-polar/nonpolar partition, and polar partition extracts effectively inhibited the reaction of ethoxyresorufin O-de-ethylation, which is specified for CYP1A activity both in dog and rat. This in vitro data should be heeded as a signal of possible in vivo interactions. The use of human liver preparations would considerably strengthen the practical impact of the data generated from this study.

Key words : *Nigella sativa*, Volatile compounds, Microwave-assisted extraction, Ethoxyresorufin O-de-ethylation

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PF - 05

Risk Assessment and Survey of Mercury in Vegetables Collected from the Korean Market

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This study was carried out to survey the levels of mercury in root vegetables and assess dietary exposure / risk to the Korean population health. Various samples including *Panax ginseng*, *C. A. mayer*, *Codonopsis lanceolata*, and *Platycodon granditloun* were collected from markets across Korea. The concentrations of mercury were measured using automatic mercury analyzer. The analytical and sampling methods in this study were full validated. The mercury content in root vegetables was determined below 100 $\mu\text{g/kg}$ (maximum residue of mercury for salt established in Korea). For risk assessment, probable daily intake was calculated and compared with PTWI (tolerable weekly intake) established by JECFA. The level of overall exposure to mercury for Korean through root vegetables was below 1% of the recommended JECFA levels, indicating of little possibility of risk. Whereas, MFDS was conducted to assess the dietary exposure to mercury from food intake, based on several reports regarding mercury published by MFDS in the 2000s. The mean and 95th percentile for exposure to dietary mercury were 4.29 and 12.48 $\mu\text{g/day}$, corresponding to 13.6% and 39.7% of PTWI, respectively.

Key words : Mercury, *Panax ginseng*, *C. space A mayer*, *Codonopsis lanceolata*, *Platycodon granditloun*

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카드뮴 및 청국장 첨가 식이가 랫드의 카드뮴 분포에 미치는 영향 (Effect of Additional Cadmium and *Cheonggukjang* Diets on Cadmium Distribution in Rat)

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본 연구는 카드뮴의 축적을 완화하는 식품 소재를 구명하고자 기본사료에 청국장 분말을 0, 0.25, 0.50, 1.00 및 1.50% 되게 혼합시킨 식이와 Cd(NO₃)₂로 조제한 100 ppm 용액을 음용수로 랫드에 자유롭게 섭취케 하여 장기 조직의 카드뮴 함량을 조사하였다. 실험군은 기본사료를 섭취하는 정상군(TN), 기본사료에 카드뮴을 섭취케 한 대조군(TC), Cd+0.25% 청국장군(T1), Cd+0.50% 청국장군(T2), Cd+1.00% 청국장군(T3) 및 Cd+1.50% 청국장군(T4) 으로 구성하여, 1주간 기본사료로 적응시킨 후, 8주사육기간 동안 매일 정한 시간에 식이량 및 카드뮴 섭취량 조사, 5일 마다 체중을 측정하였고, 9주 후 랫드를 해부하여 간, 신장, 허파, 비장, 심장, 고환, 뇌 및 근육 조직을 적출 · HNO₃ : HClO₄(v/v, 2:1) 혼산 용액으로 습식 분해하여 ICP Emission Spectro Analyzer로 카드뮴 함량을 분석하였다.

랫드의 식이 섭취량은 TC군에 비해 청국장 첨가군(T1, T2, T3 및 T4군)이 평균 3.5% 높았다. 평균 체중 증가량은 청국장 첨가군이 TC군에 비해 약 2.79% 높았다(p<0.05). 식이효율은 청국장 섭취군이 평균 5.02로 TC군보다 높았으나, 유의성은 없었다. 음용수 섭취량은 카드뮴이 함유된 TC군 및 청국장 첨가군이 19.44-21.66 mL/day로 32.44 mL/day를 나타낸 TN군 보다 낮은 경향이였다. 랫드의 조직에 축적된 Cd 함량은 간>신장>비장>심장>허파>고환>뇌>대퇴부 근육>대퇴골>경골>전혈 순이었으며, 특히 간(33.2%), 신장(31.2%), 비장(6.1%)에서 약 70%의 축적률을 보였다. 청국장 첨가군은 TC군에 비해 카드뮴 함량이 낮았고, 특히, 청국장 1% 이상의 첨가군이 비교적 낮은 함량을 나타내어 카드뮴의 축적을 완화하는 효과가 있는 것으로 판단되었다(p<0.05).

주제어 : Cadmium, *Cheonggukjang*, Rat

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PF - 07

콜레스테롤 노출 랫드의 조직 변화에 끼치는 쌀된장의 식이 효과 (Effect of Rice-*Doenjang*-Added Diets on Histological Changes in Rats by Cholesterol Exposure)

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고지혈증 유발 완화 기능성을 조사하기 위하여 고지혈증 유도 기본식이에 1.5% cholesterol+0.05% choline chloride와 쌀된장(Rice-*Doenjang*: RD) 및 분말쌀된장(Powder Rice-*Doenjang*: PRD)을 각각 0, 2, 4 및 6% 되도록 조제하여 4주 동안 사료와 음용수를 자유롭게 섭취시키고 매일 오전 10시에 섭취량을 측정, 체중은 5일마다 정해진 시간에 측정하여 체중 증가율 및 식이 효율을 구하였다. 실험식이 4주 후, 간, 신장 및 비장조직을 적출하여 haematoxylin-eosin으로 염색한 후 검경하여 랫드의 조직 변화를 조사하였다.

최종 체중은 사료로 구성된 {정상군}과 {대조군, RD 및 PRD} 사이에 유의성이 인정되었으나 {대조군}, {RD}, {PRD} 사이에 유의성은 없었다($p < 0.05$). 식이 효율은 0.258-0.394 범위로 정상군이 가장 높고, PRD 6% 군이 가장 낮았다. 간, 비장 및 신장 조직을 haematoxylin-eosin으로 염색하여 검경한 결과, 간 조직의 경우만 변성 지방구가 확인되었고, 비장과 신장은 정상이었다. 간에 생성된 변성지방구는 쌀된장과 분말쌀된장의 섭취량 증가로 감소되는 경향이였다.

이상의 결과로 기본식이에 2-4% 분말쌀된장의 첨가가 기능성 지질화합물을 증가시키는 효과적인 수준으로 판단되었다.

주제어 : Rice-*Doenjang*, Histological changes, Kidney, Spleen, Liver, Degeneration of lipid droplet

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Antioxidant and Oxidative DNA Damage Protection Effect of Methanol Extract of Red Tea Stem

(홍차줄기 메탄올 추출물의 항산화 및 산화적 DNA 손상에 대한 보호효과)

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In aerobic metabolism, free radicals like superoxide radical ($O_2^{\cdot -}$) hydroxyl radicals ($\cdot OH$) and singlet oxygen (1O_2) are constantly generated as byproduct. Besides endogenous metabolism process, exogenous sources such as ionizing radiations, UV light, pesticides are also involved in production of highly reactive free radicals. Most of the tea produced in the world can be classified as non-fermented/aerated green tea, semi-fermented (oolong) tea and fermented black tea. Black tea, also known as Red tea, is more oxidized than green, oolong, and white teas and has a characteristic color and taste, mainly due to multimeric polyphenols. Despite the upsurge of interest in the therapeutic potential of plants as sources of natural antioxidants few studies has been carried-out using black tea. This study was undertaken to determine free radical scavenging capacity and oxidative DNA damage protecting activity of methanol extract of Red Tea stem. The extract was subjected to assess their antioxidant potential using various *in vitro* systems such as DPPH \cdot , ABTS \cdot^+ , super oxide and nitric oxide free radical with IC_{50} values of 68.88 ± 1.1 , 12.08 ± 0.65 , 404.38 ± 1.6 , 93.6 ± 2.7 , respectively. Red Tea stem extract also showed ferric reducing ability (FRAP) with 2606.85 mmol Fe (II)/g of extract. Furthermore, Methanol extract of Red Tea stem showed concentration dependent significant DNA damage protecting activity in concentration dependent manner against H_2O_2 + UV induced photolysis on pUC 19 plasmid DNA. Results of this study showed that the methanolic extract of *Red Tea* stem has strong antioxidant potential along oxidative DNA damage protecting capacity that would be the significant sources of natural antioxidants, which might be helpful in preventing the progress of various oxidative stress generated diseases. Further study is necessary for isolation and characterization of the active antioxidants, which may serve as a potential source of natural antioxidant.

Key words : Red Tea, Oxidative DNA Damage, Antioxidant, Superoxide, Nitric Oxide

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