OPTIMIZATION OF OPEN-VESSEL WET DIGESTION AND ASHING METHODS FOR PLANT MATERIALS FOR DETERMINATION OF Cd, Pb AND Cr USING ICP-AES

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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TO MICHAEL

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

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Chairman: **Mohd Shamsul Hairi Bin Mohd Salleh, Ph.D.**

Faculty: **Science and Environmental Studies**

This study describes the optimization of experimental conditions of two decomposition procedures for plant material namely, open-vessel wet digestion and ashing. Subsequent determination of cadmium, lead and chromium using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) was performed. The effectiveness of the optimized methods was tested by conducting recovery studies, precision tests and sensitivity tests. Finally, the two methods were compared on the basis of blank values, analysis time, sample weight, accuracy, precision and detection limit.

For open-vessel wet digestion method, nitric acid was found to be the most effective oxidant for decomposition while hydrogen peroxide was found to be the best combination reagent. Improvements in recoveries were achieved by optimizing the quantities of nitric acid and hydrogen peroxide; a 2:1 ratio was found to be optimum. However, increasing the amount of sample decreases the percent recovery of the three metals, due to organic matrix effect. The optimized method in

general, is applicable to sample weight less than 2 g. Low temperature heating is required at the initial stage of decomposition, to avoid vigorous reaction leading to losses. For the remaining hours of digestion, a higher temperature could be used, provided that no spattering/bumping occurs.

For the ashing method, sulphuric acid was found to be a suitable chemical modifier for cadmium, with 550 ºC as optimum ashing temperature and seven hours as optimum ashing time. For lead, sulphuric acid was also found to be a suitable modifier, with 600 ºC as optimum ashing temperature and four to six hours as optimum ashing time. The optimum volume of sulphuric acid, required for 1 g of the plant material studied, water hyacinth (*Eichhornia crassipes*), was 2.0 to 2.5 mL. No modifier was found necessary for the complete recovery of chromium, for ashing at 600 ºC for seven to eight hours. However, the effect of sample matrix on recovery is evident at high sample weights. The optimized method is applicable to sample weights less than 5 g. Charring is the most critical step of ashing; slow-continual or step-wise increase of temperature, during charring, is very important.

Lower blank values were achieved for ashing than for wet digestion. Ashing is more accurate than wet digestion. Large sample weights can be used for ashing. However, wet digestion has the advantage of short analysis time. Precision and detection limit are comparable for both methods.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

UJIAN KEADAAN OPTIMUM PENCERNAAN BASAH BEKAS TERBUKA DAN PENGABUAN UNTUK PENGURAIAN BAHAN TUMBUHAN PENENTUAN LOGAM KADMIUM, PLUMBUM DAN KROMIUM MENGGUNAKAN ICP-AES

Oleh

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Kajian ini dijalankan untuk menentukan keadaan yang optimum bagi dua kaedah eksperimen iaitu pencernaan basah turus terbuka dan pengabuan. Penentuan kehadiran logam seperti kadmium, plumbum dan kromium dilalenkan dengan meggunakan Spektrometer Pancaran Atom-Plasma Gandingan Aruhan (ICP-AES). Keberkesanan kaedah yang telah dioptimumkan telah diuji melalui penemuan semula, ujian kejituan dan ujian kepekaan. Akhir sekali dua kaedah ini telah dibandingkan melalui nilai 'blank', masa analisis, berat sampel, ketepatan, kejituan dan had pengesanan.

Untuk kaedah pencernaan basah turus terbuka, didapati asid nitrik adalah bahan pengoksidaan terbaik untuk proses penguraian manakala hidrogen peroksida adalah reagen gabungan terbaik. Keadaan yang optimum untuk kuantiti asid nitrik dan hidrogen peroksida adalah pad nisbah 2:1. Walau bagaimanapun penembahan berat sample mengurangkan peratus penemuan semula bagi ketiga-tiga logam, disebabkan oleh kesan matriks. Kaedah optimum secara umumnya boleh digunakan untuk berat sample buang dari 2 gram. Pemanasan pada suhu rendah diperlukan

pada peringkat awal penguraian, ini adalah untuk mengelakkan tindak balas cergas yang akan menyebabkan kehilangan sampel. Untuk pemanasan seterusnya suhu yang lebih tinggi boleh digunakan selagi tiada pemercikan berlaku.

Untuk keadah pengabuan pula, didapati asid sulfurik adalah bahan pengubahsuai yang sesuai bagi kadmium, dengan suhu pada 550 °C sebagai suhu pengabuan yang optimum dan 7 jam sebagai masa optimum untuk pengabuan. Untuk plumbum didapati asid sulfurik adalah bahan pengubahsuai yang sesuai dengan suhu pada 600 °C sebagai suhu pengabuan yang optimum dan 6 jam sebagai masa optimum untuk pengabuan. Didapati sebanyak 2 hingga 2.5 mL asid sulfurik adalah optimum bagi 1 gram bahan tumbuhan yang digunakan dalam kajian ini iaitu keladi bunting (*Eichhornia crassipes*). Didapati tiada pengubahsui yang diperlukan untuk melengkapkan penemuan semula logam kromium pada suhu 600 °C untuk 7 hingga 8 jam. Walau bagaimanapun, kesan yang jelas terhadap matriks sangat ketara bagi sampel pada berat sampel yang lebih tinggi. Keadah optimum ini hanya sesuai untuk sampel yang kurang daripada 5 gram. Pembentukan arang adalah langkah yang sangat kritikal bagi kaedah pengabuan; kenaikan suhu secara berperingkat atau berterusan secara pelahan adalah sangat penting.

Nilai 'blank' yang lebih rendah diperolehi bagi kaedah pengabuan berbanding kaedah pencernaan basah. Keadah pengabuan adalah lebih tepat daripada kaedah pencernaan basah. Dalam kaedah pengabuan, berat sampel yang lebih besar boleh digunakan. Walau bagaimanapun, pencernaan basah mempunyai kelebihan dari segi masa analisis yang singkat ujian analisis. Kejituan dan had pengesanan adalah hampir sama untuk kedua-dua kaedah.

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I certify that the Examination Committee met on **May 7, 2004** to conduct the final examination of **Eleanor B. Gonzales** on her **Master of Science** Thesis entitled "**Optimization of Open-Vessel Wet Digestion and Ashing Methods for Plant Materials for Determination of Cd, Pb and Cr Using ICP-AES** " in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the examination Committee are as follows:

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