



# QUALITY CONTROL STUDIES OF SIX HERBAL PRODUCTS MARKETED WITHIN

# ZARIA METROPOLIS

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## Abstract

Several reports have shown toxicity of herbal products due to adulteration and/or toxic components. The aim of this research is to conduct quality control studies on six different herbal products marketed in Zaria metropolis. Samples were purchased and then coded. Moisture content and ash values were determined using World Health Organisation, 2011 method while elemental analysis (Pb, As, Cd, Zn and Fe) was conducted using MP-AES after nitric acid-hydrochloric acid (1:3) digestion. The moisture contents (2.37-5.46 %) were within the acceptable limit of 10 %. Samples were pure with no likelihood of adulteration as indicated by the low values of total ash (1.8-11 %), water soluble ash (0.2-5 %) and acid insoluble ash (0.5-2 %). Samples were also found to contain Zn (1-13 ppm) within the permissible limit while As was not detected by the method. The concentrations of Pb (2.53-13.27 ppm) and Cd (0.23-2.37 ppm) were within the permissible limit with the exception of HP3 and HP3-HP6 respectively. Samples were rich in iron content (1848-8108 ppm). Two out of the six herbal products were found to be safe for consumption.

Key Words: Herbal Products, Moisture Contents, Ash Values and Elemental Analysis

# Introduction

Herbal drugs are plants or plant parts that have undergone simple processes including harvesting, drying, and storage (EMEA, 1998). In some instances, they contain natural organic and inorganic active ingredients that are not of plant origin (WHO, 1996). Calixto and Barz (2000) reported that herbal drugs have been used time immemorial in the treatment of diseases. About 80 % of the world's population, especially in the developing countries, uses herbal medicine for their primary healthcare (Bodeker *et al.*, 2005). Proliferation of herbal products in the markets has led to their adulteration and toxicity. Contamination of herbal medicines with Pb, Cd, Al, and Hg has been reported to cause serious health problems in humans (Yap *et al.*, 2010). The safety and quality of these products have become a major concern to health authorities, pharmaceutical industries and the public (WHO, 2007). The aim of this research is to carry out quality control studies on six different herbal products marketed in Zaria metropolis.







#### Methodology

#### **Sample Collection**

Six herbal products marketed in Zaria metropolis were purchased and coded HM1-HM6. Their moisture contents, ash values and heavy metal contents were determined according to World Health Organisation, 2011 method:

### **Moisture Content**

One gram of each sample was weighed into empty labeled crucibles and the resultant weights were recorded as W1. The samples were heated in an oven at 105 °C for one hour, cooled and reweighed. The procedure was repeated until when the difference between two successive weights was not more than 0.005. The final weight was labeled W2 and % moisture content was calculated using;

% Moisture content = 
$$\frac{W1 - W2}{W1} \times 100$$

## Ash Value

Total ash

Two grams of each sample was weighed in previously ignited and tared crucibles. Samples were evenly spread and ignited by gradually increasing the temperature up to 400 °C until they became white, indicating the absence of carbon. The crucibles containing the samples were cooled in desiccators, weighed and total ash value was calculated as;

$$Total \ ash \ = \ \frac{Initial \ Weight - Final \ Weight}{Initial \ Weight}$$

#### Water soluble ash

The total ash obtained was boiled in 25 mL of distilled water for 5 minutes and then filtered. The insoluble matter was collected on an ashless filter paper, washed with hot water and ignited to constant weight at low temperature. Water soluble ash was obtained from the difference between weight of the insoluble matter and that of the total ash. The percentage of water soluble ash was calculated with reference to air dried drug.

### Acid insoluble ash

This was carried out by boiling the total ash obtained in 25 mL of 0.1N hydrochloric acid for 5 minutes and then filtered. The insoluble matter was collected on an ashless filter–paper, washed with 0.1N hydrochloric acid, ignited, cooled and weighed. The percentage of acid insoluble ash was calculated with reference to air dried drugs.

#### **Elemental Analysis**

The heavy metal content of the selected products was determined as follows:

Sample preparation: Samples were prepared using a wet digestion method: 0.5 g of each sample was weighed into digestion tubes and 25 mL of the digestion mixture was added. The mixture was heated slowly until it became clear. This was then filtered and readings were taken against a blank.

#### Sample analysis

The digested samples were analysed for the presence of lead, cadmium, mercury and





arsenic using micro plasma atomic emission spectrophotometer (MP-AES).

# **Statistical Analysis**

Results were expressed as mean  $\pm$  standard deviation (S.D). Data were analysed using

one-way analysis of variance (ANOVA) followed by Tukey HSD post hoc test for multiple comparison at 95 % confidence interval using IBM SPSS statistics 20.

### Result

#### **Moisture Content and Total Ash**

The moisture content, total ash, water soluble and acid insoluble ash values are presented in table 1,

S/No.	Codes	% MC	% TA	% WSA	% AIA
1	HP1	2.92	11.0	5.00	1.50
2	HP2	2.37	5.40	2.00	1.50
3	HP3	2.59	2.90	0.50	0.50
4	HP4	5.13	1.80	0.20	0.90
5	HP5	5.16	11.00	4.00	2.00
6	HP6	5.46	4.80	1.50	1.00

**Table 1:** Total, Water Soluble and Acid Insoluble Ash values

HP= herbal product





# **Elemental Analysis**

The calibration curve data and heavy metal concentrations of the heavy metals are presented in table 2:

Parameters	Heavy Metals							
	As	Cd	Pb	Zn	Fe			
Wave length (nm)	193.7	228.8	405.7	213.9	372			
Range	3-9	0.3-0.5	5-10	0.3-0.5	5-10			
Correlation coefficient (r)	0.999	1.000	1.000	1.000	1.000			

#### Table 2: Calibration Standards Curve Data

#### Table 3: Heavy Metal Concentration

Codes		Concentration (µg/g)					
	Pb	Cd	Zn	Fe	As		
HP1	5.667	0.32	1.50	4405.7	nd		
HP2	8.533	0.23	4.67	8108.9	nd		
HP3	13.27	0.67	1.00	4411.3	nd		
HP4	2.53	2.37	1.60	3434.3	nd		
HP5	4.20	0.93	13.00	3669.0	nd		
HP6	4.90	1.20	10.13	1848.7	nd		

nd= not detected





#### Discussion

The moisture contents were found to be within the WHO acceptable limit of 10 % (WHO, 2007). Hence, products can be stored without fear of microbial growth, insects' fungi or contamination and deterioration following hydrolysis (WHO, 2007). It was observed that HP6 had the highest moisture content while the HP2 had the least. The significant difference (p < 0.05) in moisture contents between samples could be due to several factors especially processing. Ash value is an important qualitative parameter as it determines the authenticity and purity of herbs (Brain and Turner, 1975). The samples had low total ash values (table 1) thereby indicating their purity and excluding fear of adulteration. This was further confirmed by the low values of water soluble and acid insoluble ash of the samples (table 1). Acid insoluble ash measures the amount of silica especially sand and siliceous earth while water soluble ash value indicates the mineral components in herbs (Brain and Turner, 1975; WHO, 2007).

The WHO permissible limits (PL) for Zn, Pb and Cd in herbs are 50, 10 and 0.3 mg/kg respectively (WHO, 2007). Samples were found to contain lead within PL except sample HP3 (table 3). Lead, when bioaccumulated causes brain and kidney damage, hearing and visual impairment and reproductive defects (Salawu *et al.*, 2009). It was also observed that the concentration of cadmium in the sample exceeded the PL except samples HP1 and HP2 (table 3).

Cadmium is said to be slowly excreted by the kidney and therefore bioaccumulates resulting to irreversible renal impairment (Maobe *et* al., 2012). In cadmium intoxication, kidney is the major target; other systems affected include immune and vascular systems (Maobe et al., 2012). The zinc concentrations in all samples were below the permissible limit (table 3). Intake of zinc beyond permissible limits lowers the immune system, blood lipoprotein levels, and copper level among others (Fosmire, 1990). Samples were found to be rich in iron (table 3). Iron is one of the essential trace metals with several functions which include oxygen supply, energy production, and immunity (Abou-Arab and Abou Donia, 2000). However, overdose of iron can lead to dizziness, nausea and vomiting, diarrhea, joints pain, shock, and liver damage (Abou-Arab and Abou Donia, 2000). Arsenic was not detected in the samples which could be due to their concentration not within the calibration range.

Heavy metal contamination of herbal products has been reported in Africa, India, Europe, United State (Olujohungbe *et al.*, 1994; Stewart *et al.*, 1999; Ernst, 2002; Denholm, 2010).

## Conclusion

Out of the six herbal products studied, only two passed all the determined quality control parameters.

## Reference

Abou-Arab, A. A. K. and Abou Donia, M. A. (200). Heavy metals in Egyptian

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spices and medicinal plants and the effect of processing on their levels. *Journal of Agricultural and Food Chemistry*. 48(6): 2300–2304.

- Bodeker, C. Bodeker, G. Ong, C. K. Grundy, C. K. Burford, G. and Shein, K. (2005). WHO Global Atlas of Traditional, Complementary and Alternative Medicine. *World Health Organization, Geneva* British Journal of General Practice 49 (447): 823–828.
- Brain, K.B. and Turner, T.D. (1975). Quantitative Extractive Values. *The Practical Evaluation of Phytopharmaceuticals.* John Wright and sins LTD., Dorset Press, Dorchester, London. pp. 81-89.
- Calixto, J. B. and Barz, J. (2000). Efficacy, safety, quality control, marketing and regulatory guidelines for herbal medicines (phytotherapeutic agents). *Med Biol Res.* 33: 179-189.
- Denholm, J. (2010). Complementary Medicine and Heavy Metal Toxicity in Australia. *TOXICOLOGY*. 1(9):WMC00535.
- EMEA (1998). Quality of Herbal Medicinal Products. Guidelines. European Agency for the Evaluation of Medicinal Products (EMEA), London. *Family Physician* 73 (1): 72–78.
- Ernst, E. (2002). Toxic heavy metals and undeclared drugs in Asian herbal medicines. *Trends in Pharmacological Sciences*. 23(3):136-9.

- Fosmire, G. J. (1990). Zinc toxicity. American Journal of Clinical Nutrition. 51(2): 225–227.
- Maobe, M. A. G. Gatebe, E. Gitu, L. and Rotich, H. (2012). Profile of Heavy metals in selected medicinal plants used for the treatment of diabetes, malaria and pneumonia in Kisii Region southwest Kenya. Olujohungbe, A. Fields, P. Sandford, A. and Hoffbrand, A. (1994). Heavy metal intoxication from homeopathic and herbal remedies. *Postgraduate Medical Journal*. 70 (828):764-9.
- Salawu, E.O. Adeleke, A. A. Oyewo, O.O. Ashamu, E. A. Ishola, O.O. Afolabi, A.O. and Adesanya, T.A. (2009). Prevention of renal toxicity lead exposure by from oral of administration Lycopersicon esculentum. Journal of Toxicological and Environmental Health Sciences. 1 (2): 022-027.
- Stewart, M. Moar, J. Steenkamp, P. and Kokot, M. (1999). Findings in fatal cases of poisoning attributed to traditional remedies in South Africa. *Forensic science international*. 101(3):177-83.
- WHO (1996). Quality Assurance of Pharmaceuticals: A Compendum of Guidelines and Related Materials, Good Manufacturing Practices and Inspection. World Health Organization, Geneva. 2.

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- WHO (2007). WHO guidelines for assessing quality of herbal *Global Journal of Pharmacology*. 6 (3): 245–251.
- medicines with reference to contaminants and residues. WHO Press, World Health Organization, 20 Avenue Appia, 1211 Geneva 27, Switzerland. Page 1-105.
- WHO. (2011). Quality Control Methods for Herbal Materials. WHO Press, World Health Organization, 20 Avenue Appia, 1211 Geneva 27, Switzerland.
- Yap, C.K. Fitri, M. Mazyhar, Y. and Tan, S.G. (2010). Effects of Metal contaminated soils on the accumulation of heavy metals in different parts of *Centella asiatica:* A laboratory Study. *Sains Malaysiana*; 39:347-52.