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EVALUATION OF THE QUALITY OF SOME SELECTED HERBAL PRODUCTS IN PORT HARCOURT METROPOLITAN

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ABSTRACT

The herbal medicine industry is currently facing significant challenges that undermine its potential to provide safe, consistent, and efficacious herbal products to consumers. These challenges encompass various aspects of the herbal product lifecycle and pose substantial risks to both consumers and the industry itself. The study was aimed to evaluate the quality of some selected herbal products in Rivers State which include Dibandu tea, Cancer control tea, Ijele hibiscus tea and Ginseng root. The physico-chemical parameters were determined using standard analytical methods and the heavy metals were determined using Atomic Absorption Spectrophotometry (AAS) after wet digestion. They were evaluated for Weight uniformity, physico-chemical properties, phyto-chemical constituent, presence of heavy metals and microbial contaminations following standard procedures. The result of the physiochemical parameters which include moisture content, water soluble ash, acid insoluble ash, water extractive value, alcohol extractive values, choloroform extractive values, foaming index for the four products. The phytochemical screening results showed a varied composition in each of the products. Weight uniformity and variations showed a deviation from the average weight within the range of 0.034-0.066. All the herbal products contain heavy metals which shows that they were present in quantities higher than the WHO stipulated limit. Microbial contamination count shows the presence of bacteria and fungi colonies in this product with some of them exceeding the WHO recommended limits. Based on the analysis of the four herbal products, the findings have revealed with respect to microbial load, heavy metals and weight uniformity are compromised while other parameters are within the standard.

Keywords: Herbal products, quality evaluation, physiochemical parameters, phytochemical constituents, heavy metals, microbial contamination.

INTRODUCTION

An herb is a plant or part of a plant valued for its medicinal, aromatic, or savoury qualities. Herbs can be viewed as biosynthetic chemical laboratories, producing a number of chemical compounds. Herbal remedies or medicines consist of portions of plants or unpurified plant extracts containing several constituents, which often work together synergistically. In recent years, consumption of herbal drugs has grown enormously as indicated by the marked increase in global expenditure on these products from $20 billion in 1997 to $83 billion in 2008 (Nirmal*et al.,* 2013). Consumers become more educated and enthusiastic about the safety, efficacy, and quality of herbal medicine because of potential negative outcomes, which cannot be ignored. Various, Ayurvedic medicines purchased online contained 20% detectable levels of lead, mercury, and arsenic (Saper *et al.,* 2009). Standardization and quality control of herbal drugs include, pharmacognostic evaluation (colour, odour, taste, texture, size, shape, microscopical characters, and histological parameters), chemical and physicochemical parameters (limit tests, chemical tests, foreign matter, total ash, acid-insoluble ash, swelling and foaming index, assay, successive extractive values, moisture content, viscosity, pH), etc. Other parameters such as disintegration time, friability, hardness, flow capacity, flocculation, sedimentation, alcohol content, etc. However, chromatographic and spectroscopic analysis includes TLC, HPLC, HPTLC, GC, UV, IR, FT-IR, LC-MS, GC-MS, etc. (Agrawal and Paridhavi, 2007) as well as determination of contaminants include aflatoxins, pesticides, heavy metals and microbiological load (WHO, 2011)

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There is a widespread yet false perception that herbal products are safe. However, relatively few herbal drugs have been evaluated scientifically to prove their safety, potential benefits and effectiveness (Medicines Control Agency, 2002., Kataria *et al.,* 2011). The herbal raw material is prone to a lot of variation due to several factors, such as identity of the plants and seasonal variation, the ecotypic, genotypic and chemotypic variations, drying and storage conditions (Patil and Shettigar, 2010). In Nigeria, herbal products are launched into the market without proper scientific evaluation, mandatory safety and toxicological studies. There is no effective machinery to regulate manufacturing practices and quality standards. Consumers can buy herbal products without a prescription and might not recognize the potential hazards in an inferior product (Bandaranayake *et al.,* 2006., Mosihuzzanman and Choudhary, 2008., Kunle *et al.,* 2012). The aim of this study is to evaluate the quality of some selected medicinal herbs used for birth detoxification, blood pressure management and cancer (tumor).

MATERIALS AND METHODS

Source of Herbal Products

The herbal products were selected randomly from major shopping malls in Port Harcourt, Rivers State due to their availability and abundance. The products were taken to the laboratory for investigation.

Phytochemical Screening

The Herbal Powder was screened for the presence or absence of secondary metabolites using standard methods

Loss of drying (Gravimetric method)

Using the analytical weighing balance, 2g of the powdered crude drug was accurately weighed into a previously tarred flat-bottom glass dish and the weight, W1 was noted of the glass dish and the weighed crude drug. The glass dish containing the crude drug was placed in an oven at 100-105^{0}C for 1 hour. It was allowed to cool in a desiccators and weighed. This was later replaced in the oven at the same temperature for another hour. It was allowed to cool and weighed. The drying, cooling and weighing was repeated until there was no further loss in weight (or of a difference of not more than 0.005g) and this was noted as final weight W2

The moisture content of the crude drug was calculated using the formula:

Moisture content = $\frac{100\ (W1-W2)}{W1-Wd}$

Where Wd= weight of the empty dish.

The parameters determined for proximate analysis include ash value, moisture content, extractive value, total solid content and crude fiber content of the drug

Determination of Ash values

The Ash remaining following ignition of herbal material is determined by three different methods which measure total ash, acid-insoluble ash and water-soluble ash.

Extractive Values

A 10 g of each sample was weighed and transferred to a stoppered flask containing 100 ml of water. The mixture was placed on a magnetic stirrer for 4 hours. The mixture was filtered and 50ml of the filtrate was transferred into a tarred flat -bottomed crucible and placed in a water bath, evaporated to dryness and weighed, the water-soluble extractive value was calculated from the weight of the residue as a percentage of the powdered sample. This protocol was repeated using absolute Ethanol and chloroform respectively (WHO, 2011).

Determination of Foaming index

A 1g of the product was transferred to 500 ml conical flask containing 100 ml of boiling water maintain at moderate boiling at 80- 90 C for about 30 min. It was filtered into a volumetric flask and added sufficient water through the filter to make the volume up to 100 ml (V1). Cleaned ten (10) stopper test tubes were taken and marked with 1 to 10. Successive portions of 1 ml, 2 ml up to 10 ml drug was put into the separate tubes and their remaining volumes were adjusted with the liquid up to 10 ml in each test tube. After closing the tubes with stoppers, they were shaken for 15 seconds and allowed to stand for 15 minutes, then their heights were measured. If the height of the foam in each tube is less than 1cm, the foaming index is less than 100 (not significant). Here, if the foam is more than 1cm height after the dilution of plant material in the sixth tube, then corresponding number of the test tube is the index sought. If the height of the foam in every tube is more than 1 cm, the foaming index is more than 1000. In this case, 10 ml of the first decoction of the plant material needs to be measured and transferred to a volumetric flask of 100 ml capacity (V2) and volume is to be maintained up to 100 ml and follow the same procedure. Foaming index is calculated by using the following formula

Foaming index = 1000/a in case of V1

Foaming index = 1000 × 10/a in case of V2

Where, a = Volume (ml) of decoction used for preparing the dilution in the tube where exactly 1 cm or more foam is observed.

Determination of Heavy Metals

The digestion of samples was carried out according to the standard procedure described by Okalebo *et al.,* (2002). One gram of the samples was digested with 5 mL of 16 M HNO_3 in the covered beakers to near dryness, and another 5 mL essential portion of 16 M HNO_3 was further added until the sample solutions became clear. 5 ml of 12 M HCl was then added to ensure complete digestion and then cooled to room temperature. The digested solutions were diluted to 100 mL with deionized water. The samples were analyzed using atomic absorption spectrophotometer (AAS) for the concentrations of lead, chromium and cadmium.

Spread Plate Method for bacteria and fungi count

Pipette out 0.1 ml from the appropriate desired dilution series onto the center of the surface of an agar plate. Dip the L-shaped glass spreader into alcohol. Flame the glass spreader (hockey stick) over a Bunsen burner. Spread the sample evenly over the surface of agar using the sterile glass spreader, carefully rotating the Petri dish underneath at the same time. Incubate the plate at 37°C for 24 hours. Calculate the CFU value of the sample. Once you count the colonies, multiply by the appropriate dilution factor to determine the number of CFU/mL in the original sample.

RESULTS AND DISCUSSION

Table 1: Result of phytochemical screening of the herbal preparation

Tests	Ginseng Root	Dibandu tea	Twinings cancer	Ijele Hibiscus
Flavonoids	+	+	+	+
Saponin	+	+	+	+
Tannin test	-	-	-	-
Phlobatannins	-	-	+	+
Alkaloids	+	-	+	-

Cardiac Glycoside	+	-	-	+
Triterpenoids	+	-	-	+
Anthraquinone	+	-	-	-

Key: + = present; - = absent

Table 2: Results of Moisture content and ash values

Sample	Moisture content %	Total ash (%)	Water soluble ash (%)	Acid insoluble ash (%)
Dibandu Tea	19	9	5	1
Ijele hibiscus	9.5	11	9	2
Cancer control Tea	16	8	3.5	3.5
Ginseng root	8.5	5	1	1.5
Sample	Moisture content %	Total ash (%)	Water soluble ash (%)	Acid insoluble ash (%)
Dibandu Tea	19	9	5	1
Ijele hibiscus	9.5	11	9	2
Cancer control Tea	16	8	3.5	3.5
Ginseng root	8.5	5	1	1.5

Table 3: Extractive Values of Herbal Products

Sample	Water extractive values (%)	Ethanol extractive values (%)	Chloroform extractive values (%)
Dibandu Tea	11	7	8
Ijele hibiscus	15	10	10
Cancer control Tea	8	5	2
Ginseng root	11	7	8

Table 4: Foaming Index of the Herbal Products

Product sample	Foaming index
Twinings cancer	111.11
Ijele Hibiscus	125.00
Dibandu	<100
Ginseng Root	125

Table 5: Weight Uniformity and Variations of the Herbal Products

SAMPLE	AVERAGE WEIGHT
Dibandu Tea	1.433±0.034
Ijele Hibiscus tea	3.492±0.066
Cancer Control Tea	2.078±0.066
Ginseng root	-

Values are Mean±SD of replicates of Herbal products

Table 6: Heavy Metal Analysis of the Herbal Products

S/N	Sample	Cr (mg/kg)	Cd (mg/kg)	Pb (mg/kg)
1	Dibandu Tea	7.30	NIL	20.20
2	Twining Cancer Tea	16.00	1.10	NIL
3	Ijele Hibiscus Tea	NIL	NIL	11.00
4	Ginseng Root	6.20	NIL	2.65
WHO permissible standard for herbal plants		20.00	0.3	10.0

Key: Cr = Chromium; Cd = Cadmium; Pb = Lead; mg/Kg = milligram per kilogram

Table 7: Total Bacteria Count and Fungi Count of Organism Isolated from the Herbal Products.

S/N	Sample	Plate 1	Plate 2	Average	Cfu/ml	Plate 1	Cfu/ml
1	Dibandu tea	180	178	179	$1.79x10^8$	_	_
2	Twining Cancer Tea	50	1	25.5	$2.55x10^8$	6	$6.0x10^7$
3	Ijele Hibiscus Tea	TNTC	TNTC	TNTC	TNTC	_	_
4	Ginseng Root	110	110	110	$1.10x10^9$	1	$1.0x10^7$
Permissible limit (WHO, 2005)					$\leq 1.0x10^5$		$1.0x10^7$
		Total Bacteria count			**Total Fungi Count**		

Key; TNTC=too numerous to count; cfu/ml = colony forming unit per milliliter

The increasing demand in the consumption of herbal products as result of perceived safety, high cost and adverse effects of orthodox medicines, has contributed immensely to making commercial production of herbal medicines a fast-growing industry (Yadav *et al.,* 2011). Furthermore, the recent surge in commercial advertisements, promotion, and tradomedical fairs on herb-based products with various sometimes spurious claims globally and particularly in Nigeria calls for appropriate post market quality evaluation and control. Hence, this report on the Phytoconstituents, physicochemical, Microbial loads and Heavy Metal assessment of four Nigeria brands of commercially available herbal products indicated for the management of Immune system diabetes mellitus, high blood pressure, and cancer control in Choba, Rivers State. The four herbal products were within their indicated shelf life as at the time of the study.

Phytochemical screening was carried out for the four herbal products to ascertain the secondary metabolites present in the selected products. The result revealed the presence of flavonoids in the four herbal products. Research has shown that flavonoids can prevent cancer, have the ability to induce cell cycle arrest, antiproliferation and apoptosis (Harborne, 1998). Flavonoids have a broad spectrum of biological activities that deregulates tumor progression (Middleton *et al.,* 2000). These plant components have beneficial health effects and can be used as a possible therapeutic agent against tumor. Similarly, Alkaloids was found in ginseng and Twining bitter.

The moisture content reported in this study showed that Dibandu Tea recorded the highest 19% while Ginseng root recorded the least

8.5%. The moisture contents of the tea will support the growth of microorganisms since the recommended limit is 10-14% (Pulok *et al.*, 2015). Moisture content is one of the major factors responsible for the deterioration of the drugs and formulations, with low moisture content always desirable for stability of drug compounds including herbs and herbal products. Thus, all these herbal product brands are expected to be stable to deterioration due to microbial contamination growth. Water-soluble extractive value plays an important role in evaluation and quality control of crude drugs. Water-soluble, alcohol-soluble and Chloroform extractive for the different samples appear in the range of 8 to 15%w/w, 5 to 10%w/w and 2 to 10%w/w respectively (Table 3) complied with the WHO limit of not less than 8.0 %w/w for water extractive and 10.0 %w/w alcohol extractives. Generally, alcohol-soluble extractive values are lesser than their water-soluble extractive values, indicating less alcohol-soluble phytochemicals constituents, while water-soluble extractive values are generally higher than alcohol and chloroform soluble extractives. The alcohol soluble extractive value indicates the presence of polar constituents like phenols, alkaloids, steroids, glycosides, and flavonoids as earlier report in Table 1 and also corroborated by Junejo *et al.,* (2014).

The total Ash value was aimed at identifying possible contamination, substitution, adulteration or carelessness in preparing the drug or drug combinations for marketing (Anuj *et al.,* 2014; Posadzki *et al.,* 2013). Total cash value for these polyherbal formulations investigated at 5 to 11%w/w were within the WHO specification (< 14 %w/w) A high ash value is indicative of contamination, substitution or adulteration by minerals which are absent in the four selected herbal products. Water-soluble ash is the part of the total ash content, which is soluble in water and is an indicator water-soluble salts in the drug or incorrect preparation. As obtained with the total ash, the water-soluble ash values for all the brands varied from 1 to 9%w/w, and were within the acceptable limit set by WHO (< 10 %w/w), Furthermore, acid-insoluble ash which measures the amount of silicate present (sand and siliceous earth) is an indicative of contamination, substitution, adulteration, or carelessness in preparation of drug combinations for marketing (Bele and Khale, 2011). The acid-insoluble ash values of the different formulations which ranged from 1 to 3.5 %w/w compiled with WHO limit (< 4 %w/w).

Based on uniformity of weight and variations, these results buttressed that Dibandu Tea weighed an average of 1.433±0.034 g, Ijele hibiscus tea 3.492±0.066 g, Cancer control Tea 2.078±0.066g and Ginseng root had not significant weight.

heavy metal investigation of the four brands showed that highest concentration of Lead (Pb) was found to be in the herbal samples of Dibandu tea and Ijele Hibiscus tea while Cadmium (Cd) was found in Twining Cancer Tea only. Dibandu tea had the highest value of Lead with no record of Cadmium, Cancer control tea is the only plant with Cadmium content (1.10mg/Kg) and highest content of Chromium. The heavy metals investigated in this study were within the WHO permissible limit. Herbal plants could be contaminated during manufacturing and agronomic processes including growing, harvesting, transportation, processing and storage, due to pesticide formulations, chemical fertilizers and irrigation with poor-quality water (Luo *et al.,* 2021). For example, Cd and Pb may enter the soil due to fertilizer impurities, non-ferrous smelters, lead and zinc mines, sewage slug application and combustion of fossil fuels (Khan *et al.*, 2008). Additionally, fumigants containing heavy metals may also be applied for preventing rats and mildew (Fujita *et al.*, 2016)

In this study, the microbial load of herbal products revealed that Ijele Hibiscus tea had the highest bacterial load, too numerous to count with no fungal report while cancer control tea had the highest fungal count of $6.0x10^7$. The relatively high contamination of locally produced herbal preparation in this study is similar to that reported in several other developing countries such as Iran (Enayatifard *et al.,* 2010), Togo (de Souza *et al.,* 2011) and Nigeria (Nura *et al.,* 2021). In Nigeria, Abba *et al.,* (2009) reported that about 47% of herbal remedies sampled from the Kaduna metropolis were contaminated with mainly bacteria; *Salmonella typhi, Shigella, Staphylococcus aureus* and *Escherichia coli*.

CONCLUSION

From the result of the product evaluated in this research, there quantities with respect to microbial load, heavy metals and weight uniformity are compromised while other parameters are within the standard.

Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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