



Quality Assessment of Antidiabetic Herbal Products Marketed Within Zaria Metropolis, Kaduna State, Nigeria

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Abstract

The increasing prevalence of diabetes mellitus in Nigeria has led to increased reliance on herbal medicines as affordable alternative treatment to conventional medicine. Nevertheless, the quality of these herbal products remains uncertain due to inadequate regulation, contamination, and possible adulteration with synthetic drugs. This study assessed the quality of sixteen (16) antidiabetic herbal products marketed within Zaria metropolis, Nigeria. Physicochemical evaluations including moisture content, total ash, water soluble ash, acid insoluble ash, extractive values, pH values and weight variations, were carried out in line with the World Health Organization (WHO) and British Pharmacopeia (BP) guidelines. Phytochemical constituents of the herbal product were determined using the procedure reported by Trease and Evans. Thin layer chromatography (TLC) was employed to screen the samples for the presence of metformin as an undeclared adulterant, while Pb, Cd, Ni, Fe, Mn, and Zn were determined using Atomic Absorption Spectrophotometry (AAS) after wet digestion of HNO₃ and HClO₄ in a ratio of 3:1. None of the analyzed samples pass weight uniformity test, the moisture content of the samples ranged from 6.88 – 9.55% with two samples being more than the National Agency for Food and Drugs Administration and Control (NAFDAC) limit. None of the samples had water extractable and ethanol values above the minimum limit. The range for total ash values (7.30- 13.85) of all the samples were within the European pharmacopoeia (EP) limit. The water-soluble ash and acid-insoluble ash values were within the ranges of 1.35 – 7.10 % and 6.83 - 13.18 % respectively. Alkaloids, flavonoids, saponins carbohydrates and cardiac glycoside were present in all the samples. No evidence of synthetic adulteration with metformin was detected. Analysis of the level of contamination of the herbal preparation with the toxic heavy metals indicated that 25% of the samples contained cadmium above WHO permissible limits (0.3µg/g) specified for herbal medicine and 25% of the samples contained lead (Pb) above WHO permissible limits (10µg/g) specified for herbal medicines.

Keywords: Metformin, heavy metals, physiochemical, phytochemicals, diabetes, herbal preparations.

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Introduction

The World Health Organization (WHO) defines herbal medicines as remedies containing crude herbs, herbal materials, herbal preparations and finished herbal products, which contain parts of plants as active ingredients (WHO, 2022). Herbal medicinal products have demonstrated therapeutic potential in various health conditions such as malaria, bacterial infections, diabetes mellitus and hypertension among others (Balogun *et al.*, 2016; Okello and kang, 2019; Oguntibeju, 2019; Samaha *et al.*, 2019). According to WHO, about 80% of the population in developing countries rely on plant-based medicine for primary health care (WHO, 2013).

Diabetes mellitus is a chronic metabolic disease characterized by hyperglycemia, insulin resistance, significant decrease in insulin production, as well as an imbalance in the metabolism of carbohydrates, proteins and fats (Nagja *et al.*, 2017). It is estimated that over 800 million adults worldwide are living with this disease, mainly due to a transition to a more sedentary lifestyle, unhealthy dietary practice and lower level of physical exercise (WHO, 2024). About 3.0 million adults in Nigeria are living with diabetes, with 79% undiagnosed, and the number is projected to increase to 6.6 million by 2050 (IDF 2025). The international diabetes federation (IDF) further reported that diabetes was responsible for 216,000 deaths across Africa in 2024. This rising prevalence has led many Nigerians to seek alternative treatments, with herbal medicines being widely used due to their low cost and cultural acceptance (World Health Organization 2013). Orthodox drugs for the treatment of diabetes such as metformin, sulfonylureas, and insulin remain the mainstay treatment; however, their high cost and side effects have encouraged the widespread use of herbal medicine.

Many people believe that herbal drugs are comparatively safer and devoid of side effects because they are natural in origin (Ekor, 2014). However, concerns about the quality, safety, and efficacy of these products persist due to inadequate regulation, contamination, adulteration, and lack of standardization (Steinhoff, 2019), with documented cases of adulteration with synthetic drugs such as metformin (Chowdhury, 2018; Nahid *et al.*, 2021) as well as toxic metals contamination (Samali *et al.*, 2017; Usman *et al.*, 2021; Tarfa *et al.*, 2022). This study therefore aimed to assess the quality of some selected antidiabetic herbal products marketed within Zaria metropolis, Nigeria.

Materials and Methods

Sixteen (16) antidiabetic herbal products (P1-P16) were purchased within Zaria metropolis.

Manufacturers' address, manufacturing and expiry dates, batch and National Agency for Food and Drug Administration and Control (NAFDAC) listing numbers of the samples were all recorded. Moisture content, pH, extractable and ash values of the herbal samples were determined in triplicate (WHO 2011).

Determination of pH

The pH of the samples was determined in their liquid form using a digital pH meter. The instrument was calibrated twice using standard buffer solutions of pH 7 and 4 before the pH readings of the samples were taken.

Moisture content (MC)

A quantity (1 g) of the solid samples was placed in an oven at 105°C for one hour and later cooled in a desiccator to prevent absorption of atmospheric moisture. Thereafter, the process was repeated until two consecutive weights do not differ by more than 0.005. Moisture content was calculated using the formula:

$$MC (\%) = \frac{\text{Weight of sample} - \text{Weight of oven dried sample}}{\text{Weight of sample}} \times 100$$

Extractable substances

Water-extractable and ethanol-extractable substances were determined by shaking a portion of each sample (4 g) in 100 mL distilled water or ethanol (96 %) respectively for 4 hours, then allowed to stand for 24 hours. The samples were then filtered, and the filtrates were evaporated to dryness and weighed (W). The water-extractable substances (% WES) and ethanol-extractable substances (% EES) were calculated using the formula:

$$\begin{aligned} \% \text{ WES} &= \frac{W}{\text{initial weight}} \times 100 \\ \% \text{ EES} &= \frac{W}{\text{initial weight}} \times 100 \end{aligned}$$

Total ash value

Quantities (2 g) of each solid sample were weighed into previously ignited and tarred crucibles. Each sample was spread in an even layer and ignited by gradually increasing the heat until it turns white, indicating the absence of carbon. The residue was cooled in a desiccator and weighed.

The total ash value was calculated using the formula

$$\frac{\text{Final weight}}{\text{Initial weight}} \times 100$$

Water soluble ash

The total ash obtained was boiled with distilled water (25 mL) for 5 minutes and then filtered at low temperature. Water soluble ash was obtained from the difference between the weight of the insoluble matter and that of total ash. The percentage of water-soluble ash was calculated with reference to the air-dried drugs.

Acid Insoluble Ash

The total ash obtained was boiled with dilute hydrochloric acid (25 mL) for 5 minutes and filtered. The residue was washed with hot acidulated water, ignited, cooled and weighed. The percentage of acid-insoluble ash was calculated with reference to the air-dried drugs.

Weight Variation Studies

Weight variation was conducted on samples that were formulated as tea bags, as 20 units from each herbal sample were selected randomly, weighed individually and the mean weight determined. The percentage deviation of each unit from the mean was then calculated.

$$\% \text{ Mean deviation} = \frac{(X-Z)}{Z} \times 100$$

Elemental analysis of the antidiabetic herbal samples

Sample Preparation

A di-acid digestion was performed following the Association of Official Analytical Chemists (AOAC) (1990) procedure. Quantities (1 g) of the herbal samples were placed in 250 mL crucibles and 15 mL of the digested mixture (HNO_3 and HClO_4 in a ratio of 3:1). The mixture was boiled gently for 15 minutes until dense white fumes appeared. The solution was cooled and filtered and then made up to 50 mL with distilled water.

Sample Analysis

The digested samples were analysed for the presence magnesium, iron, zinc, nickel, lead and cadmium using Atomic Absorption Spectro photometer (AAS) (Model 6800 Shimadzu Japan). (ABU Multiuser lab). Concentrations ($\mu\text{g/g}$) of the metals were obtained from the linear equations derived from their calibration curves. Concentrations ($\mu\text{g/g}$) of the analytes were determine using the formula;

$$(\mu\text{g/g}) = \frac{(C) * (V) * (Df)}{W}$$

Where, C = concentration of metal in the sample solution in ppm, V = volume of the undiluted sample solution in ml, Df = dilution factor, W = sample weight in grams

Phytochemical Screening of the Antidiabetic Herbal Samples

A quantity (4 g) of each herbal sample in solid form was macerated using methanol (30 mL) for three days. The mixture was filtered, and the phytochemical constituents present were determined according to procedures reported by Trease and Evans (2009).

Detection of metformin as an adulterant in the antidiabetic herbal samples

Sample preparation

Stock solution (1000 $\mu\text{g/mL}$) of metformin was prepared by weighing 10 mg of metformin standard powder into 10 mL volumetric flasks containing 2 mL methanol and made up to mark with the same solvent. Two grams of the antidiabetic herbal sample was weighed and transferred into a beaker (25 mL) containing 10 mL methanol, the mixture was then shaken, filtered and the resulting filtrates were used for thin layer chromatographic (TLC) analysis.

TLC Method Development

Thin layer chromatography (TLC) method was developed using various solvent systems, including ethanol, ethyl acetate, acetic acid, and methanol after assessing the systems, methanol and acetic acid in the ratio of (6:4 v/v) was selected. the mobile phase was transferred into the developing chamber, covered and allowed to saturate for fifteen (15) minutes.

Co-TLC

Solutions of the standard drug and filtrate of the test samples were spotted on the TLC plate with the aid of capillary tube, with each placed at least 1 cm apart and 2 cm from the lower end of the plate (baseline). The spotted TLC plate was allowed to dry, placed in the TLC chamber and allowed to develop to the marked level. After development, the plate was removed, allowed to dry and were subsequently viewed under UV light at 254 nm, as no visible spots were detectable under daylight.

Statistical analysis

Results were expressed as mean $\pm$ SEM standard error of the mean using Microsoft excel 2010 for statistical analysis.

Results

Label Information on the Antidiabetic Herbal Products

Evaluation of label information on the antidiabetic herbal products revealed variations in product labeling and regulatory compliance. Majority of the products 15 (93.75%) were manufactured in Kano State, while 1, P7 (6.25%) was manufactured in Kaduna State Nigeria. Manufacturing dates ranged from 2022-2025 whereas expiry dates extended from 2025-2030. Some products lacked complete product identification details. The results showed that 75% of the samples lacked batch numbers and NAFDAC listing numbers,

indicating incomplete label information among the herbal antidiabetic products (Table 1).

pH Value of the Antidiabetic Herbal Products Formulated as Liquid

The pH value ranged from 3.2-5.5. Sample P7 recorded the highest pH value (5.5 ± 0.00), while sample P16 showed the lowest value (3.2 ± 0.00). Out of the twelve liquid samples analyzed, three had pH values below the lower acceptable limit of 4.0, while 75.0% fell within the acceptable pH range of 4.0–7.0, in line with WHO guidelines (World Health Organization, 2007). Most of the analyzed herbal products exhibited acidic to slightly acidic pH characteristics (Table 2).

Table 1: Packaging Information on the Herbal Products

Code	Manufacturers' Address	Manufacturing Date	Expiry Date	Batch Number	NAFDAC Number
P1	Kano, Nigeria	2025	2030	002	8132091
P2	Kano, Nigeria	2022	2025	0018	NIL
P3	Kano, Nigeria	2022	2026	2444595	NIL
P4	Kano, Nigeria	2024	2028	NIL	NIL
P5	Kano, Nigeria	2025	2029	NIL	1542911
P6	Kano, Nigeria	2025	2030	NIL	NIL
P7	Kaduna, Nigeria	2024	2027	NIL	132805
P8	Kano, Nigeria	2024	2027	NIL	NIL
P9	Kano, Nigeria	2023	2026	NIL	NIL
P10	Kano, Nigeria	2025	2029	NIL	NIL
P11	Kano, Nigeria	2025	2030	003	NIL
P12	Kano, Nigeria	2024	2027	NIL	2444595
P13	Kano, Nigeria	2025	2029	NIL	NIL
P14	Kano, Nigeria	2024	2028	NIL	NIL
P15	Kano, Nigeria	2024	2028	NIL	NIL
P16	Kano, Nigeria	2022	2030	NIL	NIL

Table 2. pH profiles of the herbal products

S\No	Code	Mean pH
1	P1	4.9± 0.00
2	P4	4.1 ± 0.00
3	P5	4.3 ± 0.00
4	P6	3.8 ± 0.02*
5	P7	5.5 ± 0.00
6	P10	4.3 ± 0.00
7	P11	4.4 ± 0.00
8	P12	4.4 ± 0.00
9	P13	5.3 ± 0.03
10	P14	4.0 ± 0.00
11	P15	3.4± 0.01*
12	P16	3.2 ± 0.00*

* Below minimum limit of 4.0 (WHO, 2007).

Moisture content and Extractive Values of the Antidiabetic Herbal Samples

The moisture content of the 4 solid samples ranged from 6.88 – 9.55% with two samples (P2 and P3) above the National Agency for Food and Drugs Administration and Control (NAFDAC) limit, while sample P8 and P9 were below the NAFDAC limit of 8% (Table 3). The water-soluble extractive values of the 4 solid samples ranged from 2.25-6.17%, with sample P3 having the lowest value and sample P9 having the highest value (Table 3). The ethanol-soluble extractive values ranged from 5.33-8.00%, with sample P3 having the lowest value and sample P8 having the highest value (Table 3). Both the water-soluble extractive and ethanol-soluble extractive values in 4 of the samples were below the 15% minimum limit specified by the European Pharmacopoeia (EP, 2007).

Total Ash Values Water Soluble Ash and Acid Insoluble Ash Values of the Antidiabetic Herbal Samples

The total ash content of the 4 solid antidiabetic herbal products ranged from 7.30- 13.85%. Sample P3 recorded the highest total ash content, whereas sample P9 showed the lowest value (Table 4). The total ash content was within the maximum permissible limit of 14% established by the European Pharmacopoeia (EP, 2023) for all the tested products (Table 4). The water-

soluble ash values ranged from 1.35-3.46 (Table 4). Sample P2 recorded the highest water-soluble ash value while sample P9 recorded the lowest value. The acid-insoluble ash values ranged from 6.83-13.18 (Table 4.7), the highest acid-insoluble value was observed in sample P3, while the lowest value was recorded in sample P9.

Weight Uniformity of two Antidiabetic Herbal Products Formulated in tea bags

Weight uniformity test was performed only on the two antidiabetic herbal products formulated as sachet (Table 5). The two products failed the weight uniformity test, with the weights of more than two dosage units in each product deviating from the mean weight by more than the prescribed 7.5% (BP, 2009).

Metals Concentration (µg/g) in the Antidiabetic Herbal Samples

The concentrations of selected metals (Mn, Pb, Ni, Fe, Zn, and Cd) in the antidiabetic herbal preparations revealed varying levels across samples.

Lead (Pb) was detected in all the samples, ranging from 1.75 (P1) to 102.5 µg/g (P8) (Table 6). This far exceeds the WHO permissible limit of 10 µg/g in four samples. Cadmium (Cd) concentrations ranged from 0.08-4.5 µg/g, in four samples P2, P3, P8 and P9, surpassing the WHO limit of 0.3 µg/g. For the other samples the values were within the recommended limit. Iron (Fe) levels ranged from 2.12-541 µg/g with

sample P6 having the lowest value and sample P8 having the highest value (Table 6). Zinc (Zn) was in the concentrations between 0.25 µg/g and 49.25 µg/g, all within the WHO limit of 50 µg/g. The highest value was in sample P2, while sample P11 recorded the lowest. Manganese (Mn) concentrations ranged from below detection limit (BLD) for sample P9 to 96 µg/g

for sample P8 (Table 6). Nickel (Ni) was present in concentration range of 0.17–17.75 µg/g. Although there is no established WHO limit for Ni, Mn and Fe in medicinal plants, variations in their concentrations were observed among the samples.

Table 3. Moisture contents and extractive values of the 4 solid herbal samples

Code	mean moisture (%) ± SEM	mean water extractives (%) ± SEM	mean alcohol extractives (%) ± SEM
P2	8.12 ± 0.14*	5.08±1.16**	6.75±0.29**
P3	9.55 ± 0.09*	2.25±1.38**	5.33±0.08**
P8	6.88 ± 0.03	2.58±0.08**	8.00±0.90**
P9	7.37 ± 0.30	6.17±0.36**	5.91±0.08**

*Above NAFDAC limit (8 %), ** Below European Pharmacopeia minimum limit (15 %)

Table 4. Ash values of the 4 solid samples

Code	mean ash (%)	mean water soluble ash (%)	mean acid insoluble ash (%)
P2	8.72 ± 0.02*	7.10±0.17	7.45±0.20
P3	13.85 ± 0.08*	3.17±0.17	13.18±0.18
P8	10.12 ± 0.11*	3.46±0.25	8.80±0.30
P9	7.30 ± 0.40*	1.35±0.06	6.83±0.03

*Below European Pharmacopeia limit (14 %)

Table 5: Weight Uniformity of two Antidiabetic Herbal Products

S\No.	Samples	Mean weight (g)	Mean deviation range (%)	Number of Sample that deviated from the mean by >7.5%
1	P3	5.85 ± 0.68	2.16 - 17.87	5
2	P9	4.45 ± 1.44	5.20 - 74.78	7

British Pharmacopeia acceptance limit of not more than 2 samples should differ from the mean by 7.5 % (BP, 2009)

Table 6. Concentration ($\mu\text{g/g}$) of metals in the Antidiabetic Herbal Samples

Code	Mn	Pb	Ni	Fe	Zn	Cd
P1	1.8	1.75	0.38	8.975	0.54	0.09
P2	76.5	78*	17.75	132.25	49.25	3.25*
P3	20.25	94.75*	17.25	375.5	23.5	4.25*
P4	0.61	1.88	0.15	6.56	0.81	0.08
P5	1.83	1.77	0.28	14.75	0.71	0.08
P6	0.38	1.88	0.17	2.12	0.35	0.08
P7	4.26	2.28	0.45	17.995	1.29	0.09
P8	96	102.5*	17.75	541	18.00	4.25*
P9	BLD	99.25*	13.50	164	13.25	4.5*
P10	1.32	2.05	0.29	3.86	0.33	0.08
P11	1.71	2.07	0.20	3.46	0.25	0.10
P12	5.86	2.52	0.46	22.86	0.84	0.12
P13	1.14	2.31	0.29	5.44	0.50	0.09
P14	1.01	2.53	0.42	12.43	0.44	0.13
P15	6.84	2.75	0.32	3.47	0.51	0.1
P16	1.01	2.20	0.26	4.52	0.65	0.1

*Above FAO/WHO recommended limit.

Phytochemical Constituents of the Antidiabetic Herbal Samples

The results showed that most of the herbal products contained multiple phytochemical classes with differences in composition across formulations. Alkaloids, flavonoids, saponins carbohydrates and

cardiac glycoside was present in all the samples. Steroids were absent in sample P13 and P15 but detected in 14 samples. Tannins were absent only in sample P1, while terpenoids were present in 15 samples (Table 7).

Table 7. Phytochemical Constituents of the Antidiabetic Herbal Samples

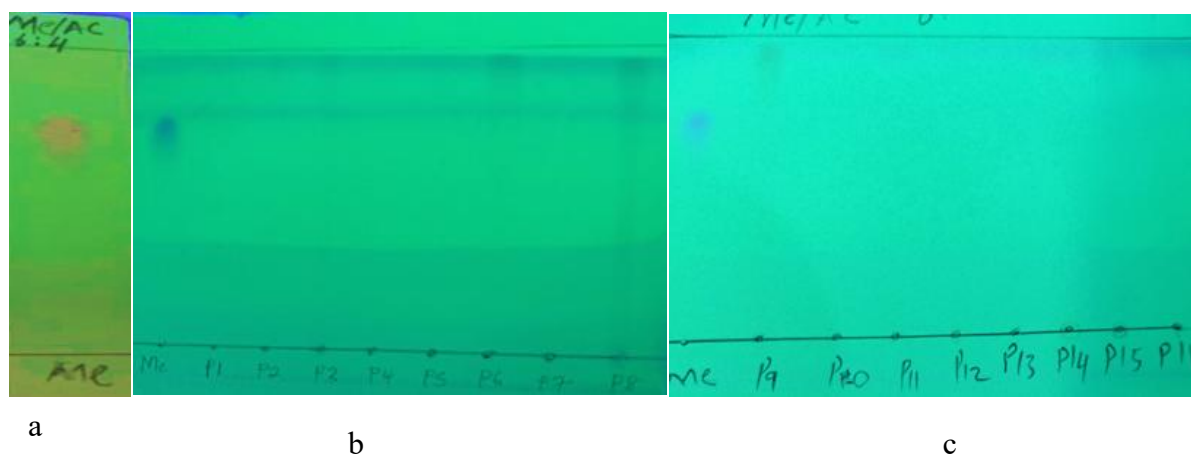
Codes	Carbohydrates	Steroids	Flavonoids	Tannins	Terpenes	Alkaloids	Saponins	Cardiac glycosides
P1	+	+	+	-	+	+	+	+
P2	+	+	+	+	+	+	+	+
P3	+	+	+	+	+	+	+	+
P4	+	+	+	+	+	+	+	+
P5	+	+	+	+	+	+	+	+
P6	+	+	+	+	+	+	+	+
P7	+	+	+	+	+	+	+	+
P8	+	+	+	+	+	+	+	+
P9	+	+	+	+	+	+	+	+
P10	+	+	+	+	+	+	+	+
P11	+	+	+	+	-	+	+	+
P12	+	+	+	+	+	+	+	+
P13	+	-	+	+	+	+	+	+
P14	+	+	+	+	+	+	+	+
P15	+	-	+	+	+	+	+	+
P16	+	+	+	+	+	+	+	+

+ = present, - = absent

Thin Layer Chromatography Screening of Selected Antidiabetic Herbal Products

Thin layer chromatography (TLC) analysis of the antidiabetic herbal products using metformin as the reference standard showed differences in

chromatographic profiles among the samples. None of the samples produced spots corresponding to the retention factor (R_f) of the metformin standard, indicating that metformin was not detected under the experimental conditions employed (Figure 7).

**Figure 1:** Co-TLC Plates of Metformin alone (a), Metformin with Samples P1, P2, P3, P4, P5, P6, P7 and P8 (b) and Metformin with Samples P9, P10, P11, P12, P13, P14, P15 and P16 (c)

Discussion

All the antidiabetic products are indigenous, indicating that the products are locally produced using traditional knowledge practices. This observation is consistent with several studies conducted in Nigeria where herbal preparations are predominantly indigenous (Adefolaju, 2014; Saidu *et al.*, 2024). 75% in liquid form, 12.5% in solid and 12.5% packaged in sachets. The predominance of liquid dosage form suggests that liquid preparation remains preferred form of traditional herbal medicine due to easier extraction of phytochemicals faster absorption and flexible dosing (Gbolade, 2009; Bone and Mills, 2013; Frimpong *et al.*, 2024).

The pH of a formulation is an important physicochemical parameter because it affects the stability, safety, and microbial susceptibility of the product. Lower pH can affect palatability and potentially irritate the gastrointestinal tract with fewer bacteria infestation, while higher pH levels encourage microbial growth (Lamikanra, 1999). Aigberua and Izah (2019), observed pH values between 3.00 and 5.09 in commercial liquid herbal products in Nigeria. Similarly, Usman *et al.* (2021) reported a pH range of 5.6 - 9.8 in twenty antihyperglycemic products sold in Kaduna metropolis, with one sample exceeding the pharmacopeial limit.

The high moisture content observed in some samples may be attributed to inadequate drying, poor packaging, or improper storage conditions. Usman *et al.* (2021) reported that 64% of herbal antihyperglycemic preparations assessed had moisture contents above the permissible 8% threshold. In another investigation involving 29 herbal formulations, Saidu *et al.* (2024) reported that 12% of the samples they analyzed had moisture content above the permissible limit. Elevated moisture content levels, as observed in some of the samples in this study, can lead to microbial contamination and chemical instability of the products; which could lead to reduced product potency. As a result, individuals who use such herbal medicines may be exposed to microbial infection or may be consuming products with reduced efficacy or possible toxicity.

The higher ethanol extractive values across the samples suggest that the phytochemicals present in the samples are moderately polar and could be quantitatively extracted using a moderately polar solvent. Low extractive values may indicate adulteration or errors during processing, drying, storage, or formulation,

which could result in reduced therapeutic activity due to incomplete extraction of active constituents (Tripathi *et al.*, 2013). Similar finding was reported with water extractive values between 1.83–18.51% and ethanol extractive values between 0.14–1.11% in marketed herbal medicines (Okoli *et al.*, 2015). Usman *et al.* (2021) also observed low extractive values in most marketed antidiabetic herbal products, while Kassim *et al.* (2022) reported that 60% and 90% of herbal anti-asthmatic products met the 15% minimum limit for water- and ethanol-soluble extractives, respectively.

Total ash reflects the presence of inorganic matter such as phosphates, carbonates, and silicates in herbal preparations. The total ash values were within the 14% maximum limit recommended by the European Pharmacopoeia (EP, 2023). This indicates minimal contamination by extraneous inorganic matter. Saidu *et al.* (2024) also observed total ash within the acceptable range in all samples, while Kassim *et al.* (2022) reported that 30% of herbal anti-asthmatic products exceeded the 14% total ash limit. Water-soluble ash values reflect variations in mineral content or possible adulteration (Brain and Turner, 1975). The high Acid-insoluble ash values across the samples may likely be due to siliceous materials in the formulations. Kassim *et al.* (2022) reported acid-insoluble ash values of 4.25–14.25% in herbal anti-asthmatic products.

Uniformity of weight is an essential quality control parameter in assessing the consistency of medicinal products. The deviations in uniformity of weight of the samples may suggest irregular dosing, lack of adherence to good manufacturing practices (GMP) during processing and packaging of the products (BP, 2009). This could compromise the therapeutic effectiveness of the products. Saidu *et al.* (2024) reported that 88% of herbal aphrodisiac products failed weight uniformity tests.

Lead is toxic with no physiological function, and chronic exposure can cause oxidative stress, kidney damage, neurological problems, and worsen cardiovascular complications in diabetic patients (Jabeen *et al.*, 2010; Flora *et al.*, 2012). Samaila *et al.*, (2017) observed 33% of the herbal sample sold in Kano State contain Pb above WHO permissible limits. Cd is highly toxic, accumulates in the liver and kidneys, and can cause renal dysfunction, skeletal damage, cancer, and impaired glucose metabolism (Godt *et al.*, 2006; Khan *et al.*, 2008). Samali *et al.* (2017) found 100% of samples exceeded Cd limits, while Fawehinmi *et al.* (2024) reported Cd in 75% of

commercial herbal preparations sold in Lagos, with concentration ranging from 0.01–1.23 ppm.

Excess Fe can induce oxidative stress and worsen insulin resistance in type 2 diabetes (Fernández-Real and Manco, 2014). High Fe concentrations in some samples could pose additional risks. Fe levels have previously been reported to range from 163.04–350.95 µg/g (Samali *et al.*, 2017) and 0.39–40.17 µg/g (Tarfa *et al.*, 2022).in commonly used herbal medicinal plants in local markets in Abuja, Nigeria

Zn is important for insulin synthesis, storage, secretion, and antioxidant defence (Chausmer, 1998; Jayawardena *et al.*, 2012). Adequate Zn may improve glycemic control. Previous studies reported Zn ranging from 0.36–35.4 µg/g (Adie and Adekunle, 2017) and 1.05–6.91 µg/g (Tarfa *et al.*, 2022). local medicinal plants and extracts sold in Ibadan and herbal medicinal plants in Abuja, Nigeria.

Mn is required for carbohydrate metabolism and antioxidant defence. Low levels can impair glucose tolerance, while high levels may cause neurotoxicity (Aschner and Aschner, 2005; Zidenberg-Cherr and Keen, 1991). Tarfa *et al.* (2022) reported Mn to be within the range of 1.45–36.21 µg/g commonly used herbal medicinal plants in local markets in Abuja, Nigeria

Excess Ni can cause dermatitis, respiratory issues, and is potentially carcinogenic (Duda-Chodak and Blaszczyk, 2008). A study conducted shows Ni within the range of 0.79–15.63 µg/g and 9.94–15.63 µg/g, respectively (Samali *et al.*, 2017; Tarfa *et al.*, 2022). Although there is no established WHO limit for Ni.

The widespread occurrence of phytochemicals in the antidiabetic herbal products suggest that the formulations contain a wide range of bioactive constituents. These results agree with previous reports showing alkaloids, flavonoids, saponins, terpenoids, and glycosides as key compounds in herbal plants used to lower blood glucose and improve insulin sensitivity (Alhassan *et al.*, 2017; Clement *et al.*, 2023).

The results indicated that none of the samples produced spots corresponding to the standard metformin reference, suggesting that the products were free from undeclared metformin adulteration. However, some of the TLC plates showed spots that may be attributed to other possible adulterants that were not evaluated in this study since only metformin was used as the reference standard. Adulteration of herbal antidiabetic products with drugs such as metformin, glibenclamide, or phenformin is widely reported and poses serious safety risks including hypoglycemia, drug–herb interactions, and

unmonitored drug intake (Ernst, 2002; Posadzki *et al.*, 2013).

Conclusion

The antidiabetic herbal products showed no evidence of metformin adulteration, but their overall quality was unsatisfactory, as none of the samples passed the quality test conducted.

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