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QUALITY ASSESSMENT OF HERBAL APHRODISIAC PRODUCTS; PHYSICOCHEMICAL AND PHYTOCHEMICAL ANALYSIS

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ABSTRACT
A huge population is using herbal products for the treatment of various ailments such as erectile dysfunction, hypertension, cancer, and asthma. Substantial amounts of these products have been reported to have poor quality. The aim of this study is to assess the quality of some herbal aphrodisiac products vis-à-vis their physicochemical properties and phytochemical constituents, a total of 22 samples were collected. Weight uniformity, moisture content, water and ethanol extractable substances, total ash values, water-soluble ash and acid-insoluble ash values were determined using standard methods. Phytochemical constituents of the products were also determined using standard methods. Only three samples passed the weight uniformity test as none of the dose units deviated by more than 7.5 % as stipulated by British pharmacopoeia. The moisture content of the samples ranged from 3.67 – 9.33 % with two samples being more than the National Agency for Food and Drugs Administration and Control (NAFDAC) limit. Only 13 % of the ethanol extractive values were found to be above the minimum limit while none of the water extractable values of the sample is above the minimum limit. The range for total ash values (3.67 - 9.33) 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 7.33 – 14.00 % and 2.83 - 13.17 % respectively. Alkaloids, flavonoids, carbohydrates and steroids were found in all the samples.
The analyzed samples contain alkaloids, flavonoids, steroids and carbohydrates which are vital in enhancing penile erection however, none of the samples passed all the quality assessments, hence are of poor quality.
Keywords: Aphrodisiacs, phytochemical, physicochemical, erectile dysfunction, Herbal preparations

INTRODUCTION

Herbal medicines are plant-derived materials or preparations with therapeutic or other health benefits, containing either raw or processed ingredients from one or more plants (WHO, 2005). Plant-based traditional medicine system continues to play an essential role in health care, with about 80% of the world's inhabitants relying on it for their primary health care (WHO, 2019).

Erectile dysfunction (ED), also called impotency, is defined as the inability to achieve or maintain penile erection sufficient for sexual intercourse (Pastuszak, 2014). ED is on the increase with prevalence rates at approximately 20% before the age of 30, 25% at the age of 30 to 39, 40% at the age of 40 to 49, 60% at the age of 50 to 59, 80% at the age of 60 to 69 years, and 90% in individuals above the age of 70 (WHO, 2019).

ED can lead to intimacy withdrawal, and such changes in sexual behaviour can cause confusion for the partner, worry about an affair, and a belief that the man is losing interest. These anxious thoughts can have a major impact on self-esteem and feelings of attractiveness (Li *et al.*, 2016). Report shows that an increase in unfulfilled sexual desires and sexual dysfunction has led to a rise in the prevalence and use of approved and unapproved aphrodisiacs/sexual enhancement drugs (Bhagavathula *et al.*, 2016). The reasons for the use of aphrodisiacs among men and women tend to be similar across the globe. These reasons include the desire to prolong sexual intercourse, increase sexual desire and satisfaction, and enhance aggression during sexual intercourse (Dumbili, 2016). Also, it includes addressing erectile dysfunction and premature ejaculation (Danquah *et al.*, 2011) and enhancing self-confidence and adherence to embedded sexual scripts of masculinity (Dumbili, 2016).

In Nigeria, Aphrodisiacs are hawked by the roadside and sold in stores and most of them are

not certified by NAFDAC. They lack manufacturing dates, expiry dates, batch numbers and any information about their chemical compositions (Iwuozor, 2019).

Contrary to the widespread perception that products sourced from nature are safe and provide the best outcome in treating ailments, research shows that some of the products contain some toxic constituents (Usman *et al.,* 2021; Kassim *et al*., 2022).

The aim of this study is to determine the physicochemical properties and phytochemical constituents of some herbal aphrodisiac products marketed within Dutse metropolis of Jigawa state Nigeria.

MATERIALS AND METHODS

Sampling of herbal aphrodisiac products

Twenty-two products were sampled and coded S1 - S22. Manufacturing and expiry dates, batch and NAFDAC registration numbers of the samples were recorded.

Physicochemical analysis of the herbal aphrodisiac samples

Physicochemical parameters namely; moisture content, extractable substances and ash values were determined in triplicate using the method described by WHO (2011).

Moisture Content (MC)

A quantity (1 g) of the solid samples were 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 constant weight was obtained. Moisture content was calculated using the formula:

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

Extractable substances

Water-extractable and ethanol-extractable substances were determined by shaking a portion of each sample (4 g) in 100 ml distilled water and ethanol (96 %) respectively for 4 hours, then allowing it to stand for 24 hours. The extracted 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:

$$\text{\% WES} = \frac{\text{W}}{\text{initial weight}} \text{ x } 100 \quad \text{and} \quad \text{\% EES} = \frac{\text{W}}{\text{initial weight}} \text{ x } 100$$

Total ash value

Quantities (2 g) of each solid sample were weighed accurately 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:

$$\text{Ash value} = \frac{\text{Final weight}}{\text{Initial weight}} \text{ x } 100$$

Water soluble ash

The total ash obtained was boiled with distilled water (25 ml) for 5 minutes and then filtered on an ashless filter paper. The residue was washed with hot water and ignited to constant weight 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

This was conducted on solid aphrodisiac samples, 20 units from each herbal sample were randomly sampled, weighed individually and the mean weight determined. The percentage deviation of each unit from the mean was then calculated.

Phytochemical screening of the herbal aphrodisiac samples

A quantity (4 g) of each herbal sample in solid form was macerated using methanol (30 ml) for 3 days. The extract was filtered off and the phytochemical constituents present were determined according to procedures reported by Trease and Evans (2009).

Statistical analysis

All measurements were done in triplicates and expressed as mean ± SEM. Data were further analysed using one-way analysis of variance (ANOVA) followed by Dunnett's post hoc test for multiple comparisons using IBM SPSS statistics 20.

RESULTS AND DISCUSSION

Proper packing and labeling of any product meant for human consumption is very important quality assurance measure. The label information on the samples in this study (Table 1) showed that 5 % of the samples do not have manufacturing and expiry dates respectively, while 68 % do not have batch numbers and 77 % of the samples lack NAFDAC listing numbers.

This indicates that the majority of the products are not certified by the regulatory agency (NAFDAC). Thus, the safety of these products cannot be guaranteed as the minimum requirement for NAFDAC listing of herbal products is a validated proof of safety.

Weight uniformity is an essential parameter in ensuring the quality of medicinal products. The weight uniformity result of this study shows 88 % of the samples failed the uniformity of weight test (Table 2) as their % means deviation differs by more than 7.5 % recommended by BP (BP, 2009). This indicates that there is no dosage consistency in packaging the products, and therefore, the therapeutic effect needed may not be achieved.

Moisture content is paramount in assessing the quality of herbal medicinal products. The moisture content of 12 % of the solid samples analyzed (Table 3) was found to be above the 8 % acceptable limit (NAFDAC SOP, 2000), this could be due to lack of proper drying of the herbal samples before packaging or lack of proper storage. Usman *et al.* (2021) reported moisture content higher than the 8 % permissible limit in 64 % of the herbal antihyperglycemic products analysed. Another study conducted on 29 different herbal remedies reported a moisture content range of 3.55 – 8.58 % (Abdu *et al.*, 2015). The high moisture content observed in some of the samples can lead to microbial activities and promote degradation through hydrolysis and oxidation reactions which often leads to loss of potency. Thus, individuals who use these herbal products may end up taking less potent or toxic products.

Extractive value plays a vital role in the evaluation of quality and purity of drugs of herbal medicines. In this study, the ethanol-soluble extractive values were found to be higher than the water-soluble extractive values in 82 % of the samples (Table 3) indicating that the phytochemicals in the samples are moderately polar.

Water extractive value in all the samples and ethanol extractive value in 77 % were found to be lower than the 15 % minimum limit stipulated by European Pharmacopeia (EP, 2023). Low extractive value in a preparation is an indication that there might be adulteration or incorrect processing during drying, storage or formulation, therefore, leading to little or no activity when taken as there is no maximum extraction of the active ingredients (Tripathi *et al.*, 2013). Kassim *et al.*, (2021) reported that 60 % and 90 % of the samples analyzed pass the 15 % minimum limit for water and ethanol extractables respectively in the herbal anti-asthmatic product they analyzed.

Ash values which comprise total ash, water-soluble ash and acid-insoluble ash, represent the inorganic residues such as phosphates, carbonates and silicates present in herbal drugs. These are important indices illustrating the quality as well as purity of herbal medicine.

The total ash value indicates the originality of the sample (if they are organic or not) the total ash values in all of the samples (Table 4) were within the 14 % maximum acceptable limit recommended by European Pharmacopoeia (EP, 2023). Thus indicating little or no residual extraneous matter in the herbal products. A study conducted on some herbal anti-asthmatic remedies reported that only 30 % of the analyzed samples have total ash values greater than 14 % maximum limit accepted by European Pharmacopoeia (Kassim *et al.*, 2022).

Water soluble ash values of the samples (Table 4) are significantly ($p < 0.05$) different from each other. Water soluble ash values in herbal products represent mineral substances such as sulphates and phosphate or adulteration (Brain and Turner, 1975). A study conducted on some herbal anti-asthmatic products reported water-soluble ash values within the range of 2.25 - 11.53 % (Kassim *et al.*, 2022).

The acid-insoluble ash values of the samples (Table 4) are significantly different ($p < 0.05$) from each other, the values observed in the samples may be due to silica materials in the formulations. A study conducted on some herbal anti-asthmatic products reported acid-insoluble ash values within the range of 4.25- 14.25 % (Kassim *et al.*, 2022).

Phytochemical screening of the herbal aphrodisiac samples revealed the presence of alkaloids, flavonoids, carbohydrates, and steroids, in all the samples. It also shows the presence of cardiac glycosides and saponins in 21 samples, terpenoids in 20 samples and tannins in 19 samples (Table 5).

Alkaloids, Flavonoids, Saponins, Tannins, steroids and carbohydrates are some of the phytochemicals reported in herbal plants used in the treatment of erectile dysfunction (Chen *et al.*, 2008; Nikaido *et al.*, 1989; Berhow *et al.*, 2000).

Table 1: Label information of the herbal aphrodisiac samples

Code	Country of Origin	Manufacturing Date	Expiry Date	Batch Number	NAFDAC Number
S1	Nigeria	April, 2023	Dec, 2026	___	1456804
S2	Nigeria	Jan, 2023	Dec, 2026	___	___
S3	Nigeria	Sep, 2021	Sep, 2024	___	___
S4	Nigeria	Jan, 2020	Dec, 2023	___	___
S5	Nigeria	Jan, 2020	Dec, 2023	___	___
S6	Nigeria	Jan, 2020	Dec, 2025	___	___
S7	Nigeria	Jan, 2020	Dec, 2025	BN:2577573	___
S8	___	Jan, 2022	Dec, 2026	___	___
S9	Nigeria	Nov, 2021	Nov, 2024	___	___
S10	Nigeria	Jan, 2022	Dec, 2026	___	___
S11	Nigeria	Aug, 2019	Aug, 2024	___	___
S12	___	June, 2020	Jun, 2024	___	___
S13	Nigeria	Jan, 2020	Dec, 2025	2577573	___
S14	Nigeria	___	___	0001	A7-5231L
S15	Nigeria	Feb, 2022	Feb, 2024	___	___
S16	Nigeria	Jan, 2022	Dec, 2026	___	___
S17	Nigeria	Jan, 2020	Dec, 2023	___	___
S18	____	Jan, 2021	Dec, 2024	___	___
S19	Nigeria	Jan, 2021	Dec, 2023	MS-00001	A7-4719L
S20	Nigeria	Jan, 2023	Jan, 2025	MS003	A7-4720L
S21	Nigeria	Jan, 2021	Oct, 2024	3292352	___
S22	Nigeria	Jan, 2023	Dec, 2023	JHML018	A7-2077L

Table 2: Weight uniformity of the herbal aphrodisiac samples

Code	Mean weight (g) ± SEM	Mean deviation (%) range	Deviated samples
S1	6.55 ± 0.22	0.15 - 30.74*	15
S2	5.47 ± 0.30	6.63 - 41.71*	17
S3	1.93 ± 0.01	0.00 - 3.98	0
S4	3.16 ± 0.09	0.32 - 22.74*	6
S5	3.84 ± 0.23	4.35 – 50.00*	17
S6	4.99 ± 0.17	0.40 - 26.65*	12
S7	4.41 ± 0.19	0.45 - 43.18*	14
S8	3.92 ± 0.28	0.51 - 39.04*	18
S9	3.95 ± 0.23	1.40 – 58.00*	14
S10	4.19 ± 0.08	1.87 - 15.86*	6
S11	4.95 ± 0.19	0.80 - 34.52*	16
S12	5.81 ± 0.16	3.33 - 21.80*	13
S13	5.75 ± 0.22	0.17 - 41.63*	11
S14	7.09 ± 0.05	0.28 - 6.22*	0
S15	9.63 ± 0.06	0.41 - 5.71	0
S16	5.41 ± 0.22	0.37 - 39.42*	10
S17	3.13 ± 0.13	2.49 - 39.11*	16

* Significantly ($p < 0.05$) higher than British pharmacopoeia acceptance limit: No more than two of the powders or granules should differ from the average weight by 7.5 % (BP, 2009)

Table 3: Moisture content and extractive values of the herbal aphrodisiac samples

Code	Mean Moisture content (%) ± SEM (n=3)	Mean Ethanol extractable (%) ± SEM	Mean Water extractable (%) ± SEM
S1	6.0 ± 0.58	15.33 ± 0.33	9.67 ± 0.17**
S2	7.67 ± 0.33	12.17 ± 0.44**	9.83 ± 0.17**
S3	9 ± 0.58*	9.17 ± 0.60**	8.00 ± 0.29**
S4	8 ± 0.00	14.17 ± 0.17**	8.33 ± 0.17**
S5	6 ± 0.58	15.50 ± 0.29	10.17 ± 0.44**
S6	6.33 ± 0.33	12.33 ± 0.33**	7.50 ± 0.29**
S7	9.33 ± 0.33*	11.00 ± 0.00**	9.33 ± 0.17**
S8	4.33 ± 0.67	12.67 ± 0.33**	9.50 ± 0.00**
S9	5.67 ± 0.67	10.83 ± 0.60**	14.00 ± 0.29**
S10	5.0 ± 0.58	13.00 ± 0.00**	10.17 ± 0.73**
S11	5.67 ± 0.33	16.83 ± 0.17	10.33 ± 0.73**
S12	7.67 ± 0.33	18.17 ± 0.44	10.00 ± 0.76**
S13	7 ± 0.58	8.17 ± 0.17**	7.33 ± 0.44**
S14	5.0 ± 0.58	11.33 ± 0.44**	12.83 ± 0.60**
S15	6.33 ± 0.33	8.50 ± 0.29**	10.00 ± 0.50**
S16	3.67 ± 0.33	10.33 ± 0.33**	9.00 ± 0.50**
S17	8 ± 0.58	8.33 ± 0.17**	7.67 ± 0.17**

NAFDAC limit: 8 % (NAFDAC SOP, 2000)

*Significantly ($p < 0.05$) higher than the NAFDAC limit

** Significantly ($p < 0.05$) lower than 15 % European Pharmacopoeia minimum limit (EP, 2023)

Table 4: Total ash, water-soluble ash and acid-insoluble ash values of the herbal aphrodisiac samples

Code	Mean Total ash (%) ± SEM (n=3)	Mean Water soluble ash (%) ± SEM (n=3)	Mean Acid insoluble ash (%) ± SEM (n=3)
S1	6.0 ± 0.58	9.67 ± 0.17	6.00 ± 00
S2	7.67 ± 0.33	9.83 ± 0.17	5.00 ± 00
S3	9 ± 0.58	8.00 ± 0.29	2.83 ± 0.17
S4	8 ± 0.00	8.33 ± 0.17	4.17 ± 0.17
S5	6 ± 0.58	10.17 ± 0.44	5.67 ± 0.17
S6	6.33 ± 0.33	7.50 ± 0.29	6.83 ± 0.17
S7	9.33 ± 0.33	9.33 ± 0.17	6.00 ± 0.29
S8	4.33 ± 0.67	9.50 ± 0.00	13.17 ± 0.17
S9	5.67 ± 0.67	14.00 ± 0.29	6.50 ± 0.29
S10	5.0 ± 0.58	10.17 ± 0.73	6.67 ± 0.33
S11	5.67 ± 0.33	10.33 ± 0.73	7.0 ± 0.00
S12	7.67 ± 0.33	10.00 ± 0.76	6.00 ± 0.29
S13	7 ± 0.58	7.33 ± 0.44	7.00 ± 0.29
S14	5.0 ± 0.58	12.83 ± 0.60	3.00 ± 0.50
S15	6.33 ± 0.33	10.00 ± 0.50	5.50 ± 0.29
S16	3.67 ± 0.33	9.00 ± 0.50	4.67 ± 0.17
S17	8 ± 0.58	7.67 ± 0.17	7.50 ± 0.50

European Pharmacopoeia limit (EP, 2023) 14% for total ash

Table 5: Phytochemical screening of methanol extract of the herbal aphrodisiac samples

Code	Alk	Fla	CaGly	Carb	Saponins	Steroids	Terpenoids	Tannins
S1	+	+	+	+	+	+	+	+
S2	+	+	+	+	+	+	+	+
S3	+	+	+	+	+	+	+	+
S4	+	+	+	+	+	+	+	+
S5	+	+	+	+	+	+	+	+
S6	+	+	+	+	+	+	+	+
S7	+	+	+	+	+	+	+	+
S8	+	+	+	+	+	+	+	+
S9	+	+	+	+	+	+	+	+
S10	+	+	+	+	+	+	+	+
S11	+	+	+	+	+	+	+	+
S12	+	+	+	+	+	+	+	+
S13	+	+	+	+	+	+	+	+
S14	+	+	+	+	+	+	+	+
S15	+	+	+	+	+	+	+	+
S16	+	+	+	+	+	+	+	+
S17	+	+	+	+	+	+	+	+
S18	+	+	-	+	-	+	+	+
S19	+	+	+	+	+	+	-	-
S20	+	+	+	+	+	+	+	-
S21	+	+	+	+	+	+	+	+
S22	+	+	+	+	+	+	-	-

Present = + Absent = -

Alk = Alkaloids, Fla = Flavonoids, Cargly = Cardiac Glycosides, Carb = Carbohydrates

CONCLUSION

The analyzed samples contain alkaloids, flavonoids, steroids and carbohydrates which are vital in enhancing penile erection, however, none of the samples is of good quality.

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