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Full Length Research Article

QUANTITATIVE DETERMINATION OF HEAVY METALS AND FTIR-BASED PHYTOCHEMICAL PROFILING OF *CASSIA SINGUEANA* LEAVES EXTRACT AS A HERBAL MEDICINE

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ABSTRACT
Cassia singueana leaves have several applications in traditional medicine and are commercially sold as herbal crude drugs in Northern Nigeria. This research aims to evaluate the heavy metal contamination and functional group profile of various extracts of *Cassia singueana* for its identification and quality control. The *Cassia singueana* sample was digested, and heavy metals were analysed using AAS. Furthermore, the sample was extracted by maceration in ethanol, water, and petroleum ether (60-80 ^{o}C) for FTIR analysis. Heavy metals, including Mn, Pb, Cr, Cd, Fe, and Cu, were all within the WHO limit, except for Mn. The FTIR analysis of the aqueous, methanol, and petroleum ether extracts of cassia singueana revealed the presence of functional groups such as alcohols, alkanes, carboxylic acids and alkenes across the extracts. This research provides referential data for standardization of *Cassia singueana* crude drug which in turn helps the herbal formulation industries to abide by good manufacturing practices thereby improving the value chain and assisting regulatory agencies in pharmacovigilance of the crude drug.

Keywords: *Standardization*, *Cassia singueana*, Herbal medicine, Heavy metals, Crude drug, pharmacovigilance

INTRODUCTION

Cassia singueana Del. belongs to the class Leguminosae and family Caesalpinioideae. It is a woody annual herb or under shrubs between 1.2 and 1.5m high with small yellow flowers. It is widely spread in India and tropical Africa including northern Nigeria, especially in cultivated or old clearings by the road side and open grassy areas (Ior *et al.*, 2015). *C. singueana* leaf is well-known for being valuable in the management of different ulcer cases by the Fulani and Hausa herbal medicine practitioners of Northern Nigeria (Ode and Asuzu, 2011). It is also used for the treatment of acute malaria (Ior *et al*., 2015). It was indicated for the treatment of stomach ache (Dambatta and Aliyu, 2011). In Ethiopia, the inner bark is chewed fresh to ease stomach contraction and smoke from its wood and bark is used for purposes of smoke baths to containers of milk and milk products (Alsiede *et al*., 2015). In some parts of Ethiopia, the leaves as well as the bark of the plant are traditionally used for the treatment of a form of skin cancer locally called *'Minshiro Nekersa'* (Abate, 1989; Mebrahtom, 2012). Qualitative phytochemical screening revealed the presence of alkaloids, anthraquinones, cardiac glycosides, flavonoids, proteins, saponins and tannins in aqueous, ethanol and petroleum ether extracts of *Cassia singueana* leaves (Uba *et al*., 2021).

The quality assessment of herbal formulations is of paramount importance in order to justify their acceptability in modern system of medicine (Rasheed *et al*., 2012).The availability and quality of the raw materials are frequently problematic, the active principles are diverse and may be unknown, and quality of different batches of preparation may be difficult to control and ascertain. In most countries, herbal products are launched into the market without proper scientific evaluation, and without any mandatory safety and toxicological studies (Kunle *et al*., 2012). Drug adulteration also comes into play because of commerce; in this practice, the genuine substances are replaced by other similar materials (Poonam, 2016). Whenever a drug is scarcely available or costly adulteration is done. Similarly, confusion in vernacular names, lack of knowledge about authentic source, similarity in morphology, lack of authentic plant, similarity in color, careless collection, etc., are the reasons for adulteration (Poonam, 2016).
Therefore, the aim of this research is to evaluate the heavy metal contamination and functional groups present in various extract of *cassia singueana*. The outcome of the present study will be helpful in identification, authentication and quality control of the plant material and the development of a monograph for the correct identification of the plant.

MATERIAL AND METHODS

Collection and Processing of Plant Material

Three sample of **C***assia singueana* leaves were collected in the wild from Dange-Shuni (12^{0}45'53.64"N, 5^{0}25'34.716"E), Sabon Birni (13^{0}24'11.178"N, 6^{0}16'23.682"E) and Rabah (13^{0}11'26.58"N, 5^{0}38'36.018) local governments of Sokoto State. The fresh leaves were identified by a plant taxonomist (Mal. Umar Shehu Gallah) in the Department of Pharmacognosy and Ethnopharmacy, Pharmaceutical Sciences, Usmanu Danfodiyo University, Sokoto. The voucher number, PCG/UDUS/LEGU/0001 was issued by matching with an existing voucher specimen. The three samples were thoroughly washed and garbled before drying in a hot air oven at 45^{0}C. After drying, the samples were garbled again and coarsely powdered using a sterilized electric blender. Representative samples were quantitatively taken from each sample and homogenized using mortar and pestle. The homogenized sample was stored in an air tight plastic container for further use.

Sample Digestion

All glassware was cleaned by soaking in dilute HNO_3, rinsed with de-ionized water and air dried before use. One gram each of the sample was taken into separate digestion tubes. About 20.00 cm^3

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of 69.5 % concentrated nitric acid was added and heated in a digestion block until about one third of each of the content was left. Another 10 cm^3 of the concentrated HNO_3 and 2.00 cm3 of 60 % $HClO_4$ acids were added and the heating process was continued until clear solutions were obtained. The digests were each diluted with about 20 cm^3 de-ionized water and boiled for another 15 minutes. The contents were allowed to cool and further transferred into 50 cm^3 volumetric flasks. These were made to the mark with deionized water. The solutions were then filtered using Whatman No. 42 filter paper into separate screw capped polyethylene bottles (Uba *et al.,* 2016_a). This experiment was done in triplicate and each bottle was labeled.

Atomic Absorption Spectrophotometric Analysis

The heavy metal analysis of lead (Pb), manganese (Mn), copper (Cu), chromium (Cr), iron (Fe) and cadmium (Cd) levels in *C. singueana* leaf powder was determined using atomic absorption spectroscopy (AAS).

Standard stock solutions (1000 pm) of the ions of the elements were prepared, which was used to prepare working solutions by appropriate dilution. The spectrophotometer (AAnalyst 400 by Perkin Elmer, USA) was being set up in accordance with the manufacturer's instructions for each element to be analysed. The blank and the samples were aspirated into the flame and their concentrations in ppm recorded. The concentration of each analyte (X) in the sample was calculated using the equation below.

$$X = \frac{Y(\text{ppm}) \times V\ (L)}{W\ (Kg)} \quad (1)$$

where; X = actual concentration of analyte in mg/Kg, Y = sample ppm, V = volume of aliquot in litres, and W = weight of sample in Kg.

Extraction of Plant Material

The powdered plant (10 g) each was extracted in 100 ml of distilled water, ethanol, and petroleum ether (60-80 ^oC) with occasional shaking on an oscillator (150rpm) for 24hrs at room temperature. The extracts were then filtered through Whatmann No. 2 filter paper and evaporated on a water bath. The extracts were then dried in a hot air oven at 45^0C (Kamble and Gaikwad, 2016; Devi, *et al.,* 2018).

FTIR spectroscopic analysis

The aqueous, ethanol (80%) and petroleum ether extracts of *C. singueana* leaf were mixed with KBr salt, using a mortar and pestle, and compressed into a thin pellet. Infrared spectra were recorded on an FTIR Spectrometer (Carry 630, Agilent Technologies), between 4,000–650 cm^{-1}(Kumar, *et al*., 2014).

RESULTS AND DISCUSSION

RESULTS

Table 1: Concentration of heavy metals in *C. singueana* sample with limits of each metal

Metals	concentration (mg/Kg)	WHO Limits (mg/Kg)
Lead (Pb)	ND	10
Manganese (Mn)	9.61 ± 3.72	2
Cadmium (Cd)	ND	0.3
Iron (Fe)	19.08± 4.91	20
Copper (Cu)	1.51 ± 0.39	20/150/3
Chromium (Cr)	ND	0.05

Mean ± Standard deviation; ND = Not Detected

Table 2: FTIR peak values and functional groups of *C. singueana* leaf extracts

Bond	Frequency (cm^{-1}) ET	AQ	PE
O-H stretch	3242.8	3246.5	3414.2
C-H stretch	2922.2	2929.7	2918.5
C-H stretch	2853	—	2851.4
C=O stretch	—	—	1736.9
C=C stretch	1602.8	—	—
C-O stretch	1036.2	1069.7	1088.4
NO_2	—	1397.8	1379.7
N-H bend	—	1558.0	—
C-X stretch	808.8	—	719.4
C-H bend	—	—	894.6/976.6

ET= ethanol extract; AQ= aqueous extract; PE= petroleum ether extract

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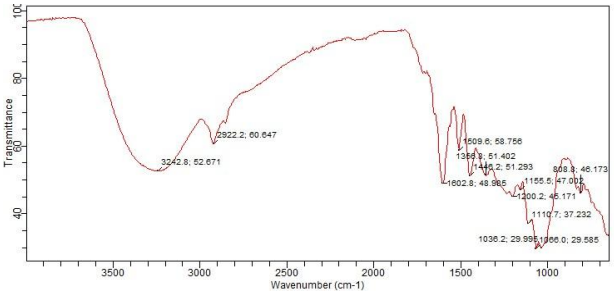

Figure 1: FTIR spectrum of ethanol extract of *C. singueana*

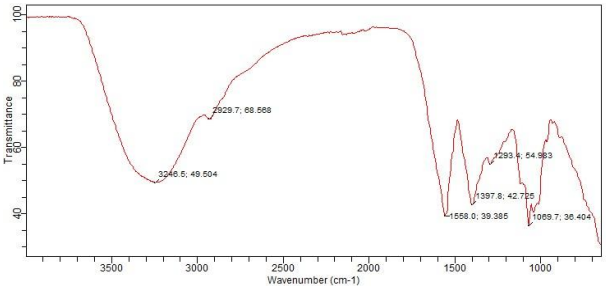

Figure 2: FTIR spectrum of aqueous extract of *C. singueana*

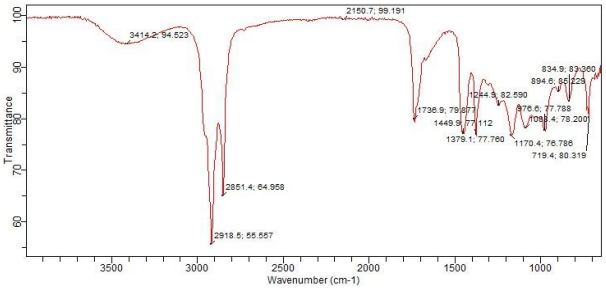

Figure 3: FTIR spectrum of petroleum ether extract of *C. singueana*

DISCUSSION

Heavy Metal Analysis

Contamination of herbal drugs with chemically toxic substances in herbal drugs can be attributed to environmental pollution, soil composition and fertilisers (WHO, 2005). It was reported also that the accumulation of heavy metals in herbal plants depends on climate factors, plant species, air and soil pollution (Maghrabi, 2014). In this study, the concentrations of Mn, Pb, Cd, Fe, Cu and Cr (Table 2) was investigated in *Cassia singueana* samples collected from the wild using atomic absorption spectroscopy (AAS).

In this study, Lead (Pb) was not detected in *C. singueana*. Similarly, in a study carried out in Gombe by Usman *et al.* (2018), lead was not detected in *Cassia singueana* powdered leaves. However, in another study, the concentration of lead in some commercial samples obtained from Kara, Marina and Old markets of Sokoto metropolis was found to be 1.133±0.001 mg/Kg, 0.600±0.001 mg/Kg and 0.576±0.001 mg/Kg respectively which is significantly lower than the WHO/FAO maximum allowable limit (10 mg/Kg) for herbal raw materials (Uba and Baburo 2016$_a$; Uba and Baburo 2016$_b$; Uba *et al.,* 2016). The contamination of these commercial samples by lead could be attributed to environmental pollution due to human activities such as fuel combustion and vehicular emissions due to heavy traffic on the roads close to the markets (Ogundele *et al*., 2015). Lead is considered as one of the most toxic elements, causing both acute and chronic poisoning with adverse effect on various body systems such as kidney, liver, renal, digestive, brain damage and disorder of the central nervous system (Umar *et al.,* 2016).

The concentration of Mn in *Cassia singueana* leaves was found to be 9.61 ± 3.72 mg/Kg which is significantly above the maximum allowable limit for edible plants set by WHO at 2 mg/Kg. The maximum allowable limit for edible plants was used because WHO permissible limit for manganese in medicinal herbs has not yet been set (Jabeen *et al*., 2010; Onyedikachi *et al*., 2018). In similar studies of *C. singueana*, the concentration of Mn in market samples obtained from Marina market and Old market of Sokoto metropolis were found to be considerably higher (14.977±0.001 and 18.136±0.001 mg/Kg) than that of the present sample (Uba and Baburo 2016$_a$; Uba and Baburo 2016$_b$). In a study carried out by Uba *et al.* (2016), it was found that the concentration of Mn in commercial sample of *C. singueana* is 4.600±0.001 mg/Kg which is lower than that of the current study but still above the maximum allowable limit for edible plants. Manganese (Mn) is an essential metal but at high concentrations, Mn is harmful to the environment (Louhi *et al.,* 2012).

In this study, cadmium (Cd) was not detected in *C. singueana* leaves. However, the concentration of Cd was found to be way above the WHO limit (0.3 mg/Kg) for herbal raw materials in samples of *Cassia singueana* leaves collected in Gombe state, Nigeria (Usman *et al.,* 2018). In another study of heavy metals conducted on commercial *C. singueana* leaf powder, the concentration of cadmium was found to be 0.103±0.005 mg/Kg 0.034±0.001 mg/Kg and 0.038±0.001 mg/Kg which are below the WHO limit for herbal raw materials (Uba and Baburo 2016$_a$; Uba and Baburo 2016$_b$; Uba *et al.,* 2016). Cadmium is a cumulative toxin of which acute and chronic exposure has a negative impact to human health. Acute oral ingestion results in severe gastroenteritis while chronic exposure primarily affects the bones, kidneys and possibly the lungs (Olsson *et al.,* 2002; Uddin *et al.,* 2012).

The limit for iron in medicinal plants has not been established therefore 20 mg/Kg for edible plants have been used (Jabeen *et al*., 2010; Onyedikachi *et al*., 2018). In the current study, the concentration of Iron (19.08 ± 4.91 mg/Kg) in *Cassia singueana* leaves was found to be slightly below the limit for edible plants. Market samples in some major crude Drug outlets have shown significantly lower concentration of Fe in *Cassia singueana* (Uba and Baburo 2016$_a$; Uba and Baburo 2016$_b$; Uba *et al.,* 2016). The high concentration of iron could be attributed to low soil pH as some plants secrete acid from the roots. These plants can take up too much iron which could lead to toxicity (Ancuceanu *et al*., 2015). Iron overdose is associated with symptoms of dizziness, nausea and vomiting, diarrhea, joints pain, shock, and liver damage. Iron toxicity has an adverse effect on various metabolic functions and cardiovascular system (Dghaim *et al.,* 2015).

The regulatory limit for copper (Cu) in raw herbal materials based on WHO regulations has not been established yet but China and Singapore set the permissible limit of 20 mg/Kg and 150 mg/Kg respectively (Umar *et al.,* 2016). Also, the maximum allowable limit

for Cu in edible plants has been set at 3 mg/Kg (Jabeen *et al*., 2010; Onyedikachi *et al*., 2018). In the present study, the concentration of copper was found to be within the stipulated limits (1.51± 0.39 mg/Kg). In a similar study conducted on *Cassia singueana* sample collected in Bauchi, the concentration of Copper was found to be in agreement with that of the current study (Usman *et al.,* 2018). In similar studies of commercial samples sold in some major crude drugs outlets in Sokoto metropolis, the level of copper was also found to be in agreement with the current study (Uba and Baburo 2016_a; Uba and Baburo 2016_b; Uba *et al.,* 2016). Copper is an essential element for all living organisms but is toxic at high levels. It regulates various biological processes inside the body like oxidation reduction reactions, energy production, connective tissues formation, iron metabolism, synthesis of neurotransmitters, melanin production and so on (Saeed *et al*., 2010; Louhi *et al.,* 2012; Dghaim *et al.,* 2015).

The regulatory limit for chromium (Cr) based on WHO regulations has not been established yet but Canada set the permissible limit of 2 mg/Kg in raw herbal materials (Umar *et al.,* 2016). In the present study, chromium was not detected in the *Cassia singueana* sample. Similarly, in a study of heavy metals in *Cassia singueana* crude drug chromium was not detected (Usman *et al.,* 2018). However, in a study of commercial samples of herbal crude drugs conducted by Uba *et al.* (2016), the concentration of chromium in *Cassia singueana* was found to be significantly above the stipulated limit. Despite the importance of chromium in glucose metabolism, it is very harmful to living organisms when present in high concentration especially in its hexavalent form (Saeed *et al*., 2010; Louhi *et al.,* 2012).

FTIR Fingerprint Analysis

The results of functional group analysis (Table 2; Fig. 1-3) demonstrate the existence of various characteristic functional groups in *C. singueana* leaves extract. The size of the peaks in the spectrum is a direct indication of the amount of compound present (Kumar *et al.,* 2014).

The spectrum of the ethanol extract of *Cassia singueana* is shown in figure1. In the ethanol extract, six major peaks ranging from O-H stretching (3242.8 cm^{-1}), C-H stretching (2922.2 and 2853 cm^{-1}) and C=C stretching (1602.8, 1509.6 and 1446.2 cm^{-1}) were observed. The peaks showed stretching vibrational absorption of alcohol, alkane and aromatic ring stretching respectively (Kamble and Gaikwad, 2016; Nithyadevi and Sivakumar, 2015). The peaks at 1200.2 cm^{-1}, 1155.5 cm^{-1}, 1110.7 cm^{-1}, 1036.2 cm^{-1}, 1066.0 cm^{-1}, and 808.8 cm^{-1} indicates the possibility of organic sulfates/sulfonates, primary or secondary alcohol bends, phenol C-O stretch, organic siloxane or silicone (Si-O-Si/Si-O-C), tertiary alcohol C-O stretch, ethers/esters (C-O), amines (N-H), aliphatic phosphates and alkyl halide (Rajiv *et al.,* 2017; Segneanu *et al.,* 2012; Starlin *et al.,* 2012; Coates, 2000). The FTIR spectrum of the ethanol extract of *Cassia singueana* showed the presence of functional groups which have medicinal properties responsible for its biological activities.

In the aqueous extract (Fig.2), the peaks at 3246.5 cm^{-1} (O-H stretch), 2929.7 cm^{-1} (C-H stretch), represent the presence of alcohol and alkane respectively, while the peak at 1558.0 cm^{-1} indicates the presence of a nitro group (aromatic) (Kamble and Gaikwad, 2016). The peaks at 1397.8, 1293.4 and 1069.7 cm^{-1} indicate the presence of nitro compounds, aliphatic amines (C-N), ethers and esters (C-O) (Rajiv *et al.,* 2017; Coates, 2000). The presence of these functional groups gives *Cassia singueana* its biological activities, especially the presence of strong OH groups which renders antioxidant properties of the plant.

In the FTIR spectrum of petroleum ether extract (Fig. 3), the peaks observed at 3414.2, 2918.5, 2851.4 and 1736.9 cm^{-1} indicate the presence of alcohol (O-H), alkane (C-H) and carbonyl compound (C=O) respectively (Rajiv *et al.,* 2017; Kamble and Gaikwad, 2016). The peaks at 1449.9 and 1379.1 cm^{-1} indicates methyl C-H bend (Kamble and Gaikwad, 2016). The peak at 1244.9, could be associated with the presence of ether (C-O stretch), C-H stretch and O-H deformation of carboxyl groups and to the N-H of amide (Rajiv *et al.,* 2017; Szymczycha-Madeja *et al.,*2013; Coates, 2000). The peaks at 1170.4 and 1088.4 cm^{-1} could be assigned to ethers, esters (C-O) and aliphatic amines (C-N), (Rajiv *et al.,* 2017; Kamble and Gaikwad, 2016; Coates, 2000). The peaks at 976.6, 894.6, 834.9 and 719.4 cm^{-1} indicate the presence of alkene C-H bend, primary/secondary amines and alkyl halide (Kamble and Gaikwad, 2016; Murugan and Mohan, 2014; Coates, 2000). The functional groups present in the extract shows the presence of bioactive molecules responsible for the antimicrobial activities of the drug as well as possible free radical scavenging activity.

Among the functional groups observed in the extracts, OH group was found to be present uniformly in all the leave extracts of *Cassia singueana*. Because OH group has the ability of forming hydrogen bonds, presence of OH group in all the extracts explains the potential of the crude drug towards inhibitory activities against microorganisms; as has been demonstrated in studies carried out by Mølgaard *et al.* (2001), Adeyanju *et al.* (2011), Mebrahtom *et al.* (2014) and Alsiede *et al.* (2015). Also, for future *C. singueana* extraction using ethanol, water and petroleum ether, the FTIR spectra in the current study can be used for comparison by intuitive evaluation method which is to compare the similarities and/or differences in the shapes and sizes of the FTIR fingerprints which can be used to ensure that the functional groups in the new extracts are present in a reproducible manner.

Conclusion

The heavy metal concentration, FTIR fingerprint analysis and fluorescence analysis of *C. singueana* crude drug was carried out. The concentrations of selected heavy metals determined in *Cassia singueana* are within limit except for Manganese. Therefore, prolong intake could be of risk to health. However, there is the need for frequent monitoring (especially for Mn) of metals in the crude drug. The FTIR analysis of the aqueous, methanol and petroleum ether extracts of *Cassia singueana* revealed the presence of hydroxyl and alkane functional groups. While the carbonyl and alkene functional groups were peculiar to Pet. Ether and ethanol extracts respectively, among the functional groups observed in the extracts, OH group was found to be present uniformly in all the leave extracts of *Cassia singueana*. The spectra could be used for future reference for intuitive evaluation of presence and relative amounts of functional groups in *C. singueana* samples. The outcome of the present study will be useful in identification, authentication and quality control of the plant material and the development of a monograph for the correct identification of the plant.

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