



PRELIMINARY DETERMINATION OF PHYTOCHEMICAL COMPOUNDS IN *SENNA SIAMEA* LEAVES POWDERS FOR INSECT PEST CONTROL

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ABSTRACT

Laboratory evaluation of phytochemical compounds and insecticidal/repellent effect of *Senna siamea* L. leaves was conducted. Ethanol, methanol and distilled water were used as extraction solvents for the conduct of untargeted Gas Chromatography and Mass Spectrometry (GC-MS) analysis of the leave extracts. To mention but few, the ethanol extracts revealed the presence of 2-[2-(2-Ethoxyethoxy) ethoxy]ethyl acetate (C₉H₁₈O₅); 3-Methylmannoside (C₇H₁₄O₆); 7H-Chromen-7-one, 8-hydroxy-2,4-dimethyl (C₁₁H₁₀O₃); 3,5-Dimethylanisole C₉H₁₂O as compound with the highest percentage area. Methanol extracts showed Thiophene, tetrahydro-2-methyl- (C₅H₁₀S); 4,6-Di-O-methyl-.alpha.-d-galactose (C₈H₁₆O₆); 2-Furancarboxaldehyde, 5-[(5-methyl-2-furanyl)methyl]- (C₁₁H₁₀O₃); 5,9-Undecadien-2-one, 6,10-dimethyl- (C₁₃H₂₂O) as compound with the highest percentage area. Distilled water extraction revealed Phosphoric acid, dimethyl (4-methoxy-3-t-butylphenyl) ester (C₁₃H₂₁O₅P); Vitamin E (C₂₉H₅₀O₂); Naphtalene-1,3-dicarbonitrile, 5,6,7,8-tetrahydro-2-hydroxy-4-phenyl (C₁₈H₁₄N₂O); Lanosterol (C₃₀H₅₀O) as compound with the highest percentage area. Phytochemicals have been used for many years to control insect pest damage in agricultural crops. Pyrethre, Nicotine, Rotenone and tobacco have long been used as control agents against insects in some regions of sub-Saharan Africa. Unlike synthetic chemical insecticides that kill both pests and non-target organisms, botanicals pesticides are relatively target specific. They are also biodegradable, environmentally friendly, and can also be used in insecticide resistance management programs. Hence, could serve as good alternatives to chemical insecticides.

Keywords: Determination, Leaves powder, Pest control, Pesticides, Phytochemical.

INTRODUCTION

The genus *Senna*, (*Senna siamea* (Lam.) H.S. Irwin & Barneby) belongs to the family Fabaceae; subfamily Caesalpinioideae, consists of about 350 tropical and warm temperate species of trees, shrubs and herbs. In West Africa, the genus contains about 22 indigenous species apart from those introduced (Hutchinson and Daziel, 1958). Burkill, (1995) reported about 19 species in West African floristic region with the whole 19 species in Nigeria (Soladoye and Lewis, 2003) and at least 8 species in South Western Nigeria especially in Oyo and Ogun States. The genus was divided into six sections: Psilorhegma, Chamaefistula, *Senna*, Peiranisia, Paradyction and Astroites (Irwin and Barneby, 1982; Randell and Barlow, 1998). The synonym *Cassia siamea* is, however, still widely used today and this should be noted where searching for information on the species. More than 500 different active as well as non-active phytoconstituents have been reported from the genus. The primary chemical constituents



of *Cassia* include cinnamaldehyde, gum, tannis, mannitol, coumarins, and essential oils (aldehydes, eugenol and pinene); it also contains sugars, resins, and mucilage, among other constituents (Singh and Khan, 1990). The leaves and seeds of *Siamese senna* contain anthraquinones, alkaloids, phylobatannins, saponins, tannins (though not condensed tannins), oxalates and phytate (Alli Smith, 2009; Ingweye, *et al.*, 2010). Kendeson, *et al.*, (2018) found that the leaves of *S. siamea* contain alkaloids, saponins, tannins, steroids, terpenoid, flavonoids, volatile oils, glycosides however, anthraquinones and phylobatannins were not detected in their studies.

The insecticide activity of *Cassia siamea* extracts and pure compounds has been reported recently (Kamara, *et al.*, 2011; Mamadou, *et al.*, 2014). Fresh leaves from *S. siamea* is used for repelling or killing insects such as termites, bed bugs and mosquitoes (Jimoh, *et al.*, 2013). In India, *Cassia* species is used as a natural pesticide in organic farms (Shivjeet, *et al.*, 2013). Many plants from the cassia genus; *C. didymobotrya*, has been reported to exhibit larvicidal activity against *C. quinquefasciatus* in Ethiopia (Nagappan, 2012), *Cassia fistula* against *Culex tritaeniorhynchus* in India (Govindarajan, *et al.*, 2011), *Cassia nigricans* against mosquito and white flies in West Africa (Georges, *et al.*, 2008). Control of storage pests have been largely depending and conducted using conventional synthetic insecticides in the study area, but resistance to organophosphates and other class of the synthetic insecticides has been observed by local farmers. The study was aimed to determine the phytochemical compounds in *Senna siamea* leaves powders found in Gombe State and their insecticidal potentials on storage pests.

MATERIALS AND METHODS

Experimental Site

The study was carried out at the Research Laboratory of Biological Sciences Department, the Federal University of Kashere (FUK), Gombe State, located at a GPS coordinate (DMS) 10° 16' 44.9040" N and 11° 10' 23.0160" E; Latitude 10.27914; Longitude 11.17306 and Altitude 454m, Gombe, Nigeria.

Collection of Plant Materials

Fresh plant materials of *Senna siamea* leaves were locally collected in Gombe and suburb subsequently, identified at the herbarium of Biological Sciences Department (specimen voucher number 090), Faculty of Science, Federal University of Kashere (FUK), Gombe State, Nigeria.

Preparation of Plant Materials

All the plant materials collected from *Senna siamea* (leaves) were shade dried to a crispy dry condition and thereafter was grounded using pestle and mortar, blended with an electric blender and then sieved through a mesh size of 600µm to obtain fine powders.

Preparation of Crude Extracts of the Plant Materials

A weight of 1.8 kg was obtained for each of the powdered plant parts. A 600g portions of the powdered leaves and stem bark were soaked separately in 1.8 litres of 70% aqueous ethanol, 1.8 litres of 70% aqueous methanol and 1.8 litres of distilled water for 24 hours. After 24hours, the extract was sieved with a muslin cloth and this was stored in a refrigerator when not in use.

Phytochemical Screening of the Crude Extract

The processed crude extracts were aseptically carried using Laboratory sample bottles (to avoid contamination on transit) to the National Research Institute for Chemical Technology (NARICT) Basawa, Zaria, kaduna State where laboratory tests were conducted on the crude



extracts of the powdered specimens using standard procedures (Untargeted Gas Chromatography-Mass Spectrometry (GC-MS).

GC-MS Experimental Conditions

The analysis was performed using Agilent Gas chromatography couple to the mass spectrometer system (model GC Agilent S/N 7890A and 5975A). HP 5ms 5% phenyl Methyl) siloxane Capillary Colum (30M x 250m) was used under the following conditions: Oven temperature 70°C for 1 min, then increase to 280°C by 10°C/min for 10 min and Injector temperature of 260°C. Helium gas was used as the carrier gas with flow rate of 1.9m/min, the volume of the injected sample was 1uL of diluted extract in ethanol, methanol and distilled water. Split injection techniques were used during sample injection with ionization energy 70ev in the electron ionization (EI) mode, ion source temperature 230°C scan mass range of M/Z 50-500.

Identification of Constituents

The constituents of the plant material were identified base on the result obtained from the library search and mass spectra of most of the compound with data generated under identical experimental conditions by applying a search algorithm considering the retention index as well as mass spectra similar with those of authentic compounds available in NIST, 2011 and NIST, 2014 Library.

RESULTS AND DISCUSSION

The result presented in (Table 1) was limited to only first four compounds with the highest percentage area (peaks) and their chromatogram together with chemical structures (Figure 1-4) out of the numerous compounds screened in the various extracts. The compounds names, Molecular Formula, Molecular Weight, Retention Time (Min), and Area (%) are presented in Table 1. The results showed that ethanol extracts of *S.siamea* Leaves Powder revealed the presence of 2-[2-(2-Ethoxyethoxy) ethoxy]ethyl acetate (C₉H₁₈O₅) (45.32%); 3-Methylmannoside (C₇H₁₄O₆) (22.79%); 7H-Chromen-7-one, 8-hydroxy-2,4-dimethyl (C₁₁H₁₀O₃) (7.81%) and 3,5-Dimethylanisole (C₉H₁₂O) (4.92%) as the first four compounds with highest percentage area and their chromatograms together with chemical structures (figure 1a – 4a). In a related study, Hafez *et al.* (2019) in their review, reported anthraquinones, anthracenes, Phenolic compounds and their derivatives and some miscellaneous compounds have been isolated from different *Cassia* species, Onoarigo, *et al.* (2017) reported that polyphenols were highly present in the foliage while Flavonoids were observed with slight coloration in the foliage. Phlobatannins were beyond detectable limits in both parts of the plant for this species. Tannins were observed as moderately present while alkaloids were slightly present in the plant as indicative of the slight and deep coloration of the test samples. The methanol extracts of *S.siamea* Leaves Powder result presented in Table 1, revealed the presence of Thiophene, tetrahydro-2-methyl- (C₅H₁₀S) (41.66%); 4,6-Di-O-methyl-.alpha.-d-galactose (C₈H₁₆O₆) (24.29%); 2-Furancarboxaldehyde, 5-[(5-methyl-2-furanyl)methyl]- (C₁₁H₁₀O₃) (8.24%) and 5,9-Undecadien-2-one, 6,10-dimethyl- (C₁₃H₂₂O) (7.59%) as the first four compounds with the highest percentage area and their chromatograms together with chemical structures (figure 1b – 4b). In a related study, GC-MS chromatogram of the methanolic extract of the leaves of *C. angustifolia* analysis carried out by Shahina, *et al.*, (2016) showed the presence of 45 different phytocomponents. They found that squalene, 6,6-Dideutero-Nonen-1-Ol-3, pentadecanoic acid, 1 butanol, 3 methyl acetate, and phytol are the major components.

Besides these compounds, vitamin E (3.85 %), gamma-tocopherol (0.58%), and several other antioxidants are also present.

Table 1: GC-MS Analytical Report of Ethanol, Methanol and Distilled Water Extracts of *Senna siamea* Leaves

Extraction solvent	Peak	Compounds	Molecular Formula	Molecular Weight	Retention Time (Min)	Area (%)
Ethanol	1	2-[2-(2-Ethoxyethoxy)ethoxy]ethyl acetate	C ₉ H ₁₈ O ₅	206	14.931	45.32
	2	3-Methylmannoside	C ₇ H ₁₄ O ₆	194	15.395	22.79
	3	7H-Chromen-7-one, 8-hydroxy-2,4-dimethyl	C ₁₁ H ₁₀ O ₃	190	18.141	7.81
	4	3,5-Dimethylanisole	C ₉ H ₁₂ O	136	10.909	4.92
Methanol	1	Thiophene, tetrahydro-2-methyl-	C ₅ H ₁₀ S	102	14.960	41.66
	2	4,6-Di-O-methyl-.alpha.-d-galactose	C ₈ H ₁₆ O ₆	208	15.383	24.29
	3	2-Furancarboxaldehyde, 5-[(5-methyl-2-furanyl)methyl]-	C ₁₁ H ₁₀ O ₃	190	18.153	8.24
	4	5,9-Undecadien-2-one, 6,10-dimethyl-	C ₁₃ H ₂₂ O	194	20.436	7.59
Distilled water	1	Phosphoric acid, dimethyl(4-methoxy-3-t-butylphenyl) ester	C ₁₃ H ₂₁ O ₅ P	288	17.369	39.55
	2	Vitamin E	C ₂₉ H ₅₀ O ₂	430	20.413	22.95
	3	Naphtalene-1,3-dicarbonitrile, 5,6,7,8-tetrahydro-2-hydroxy-4-phenyl	C ₁₈ H ₁₄ N ₂ O	274	18.170	14.82
	4	Lanosterol	C ₃₀ H ₅₀ O	426	19.097	5.67

Figure 1 (1a – 4a): GC-MS Chromatogram Together with Chemical Structures of Ethanolic Extract of *S. siamea* Leaves

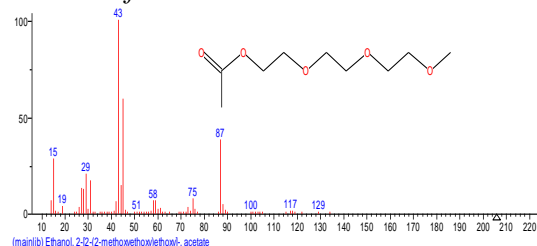


Figure 1a.

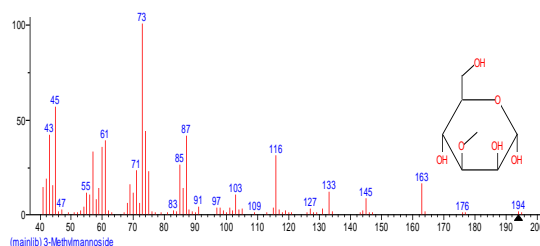


Figure 2a.

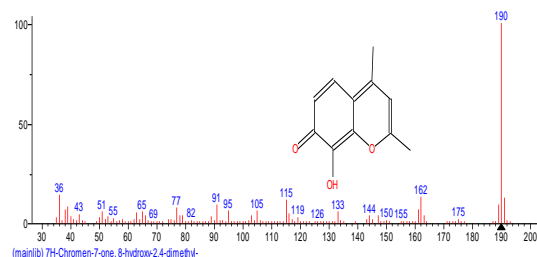


Figure 3a.

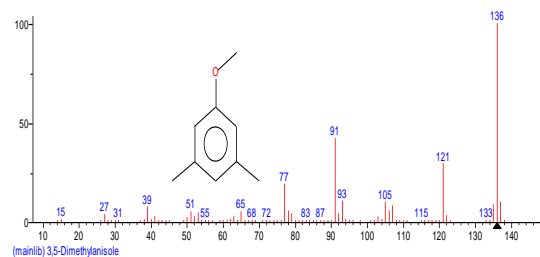


Figure 4a.

Figure 2 (1b – 4b): GC-MS Chromatogram Together with Chemical Structures of Methanolic Extract of *S. siamea* leaves

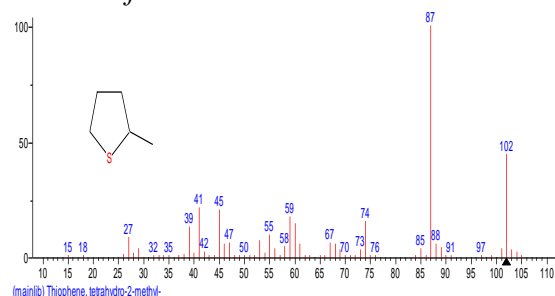


Figure 1b.

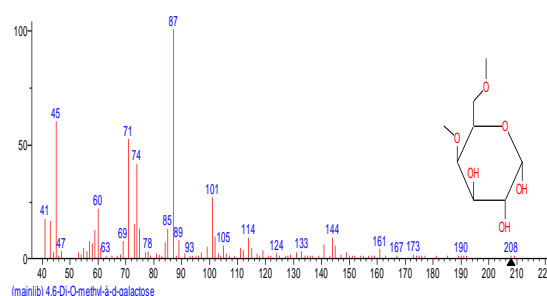


Figure 2b.

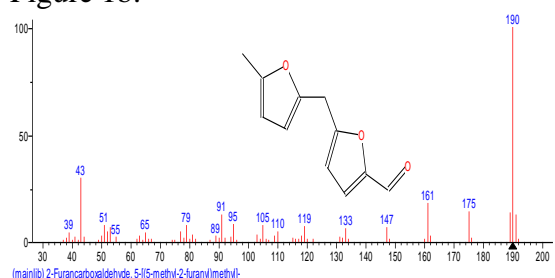


Figure 3b.

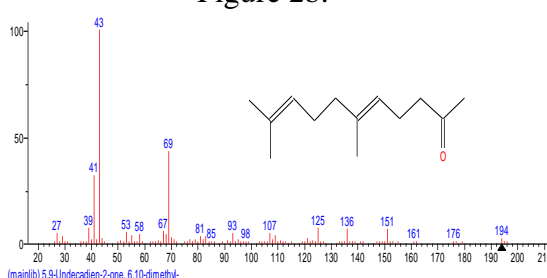


Figure 4b.

Figure 3 (1c – 4c): GC-MS Chromatogram Together with Chemical Structures of Distilled Water Extract of *S. siamea* Leaves

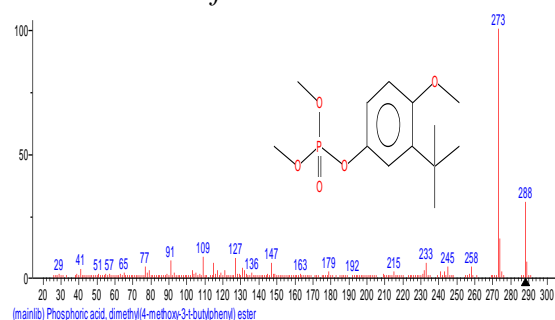


Figure 1c.

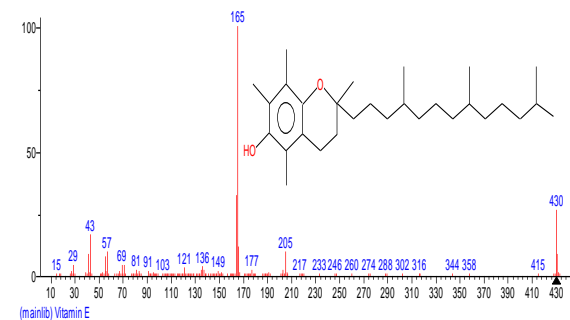


Figure 2c.

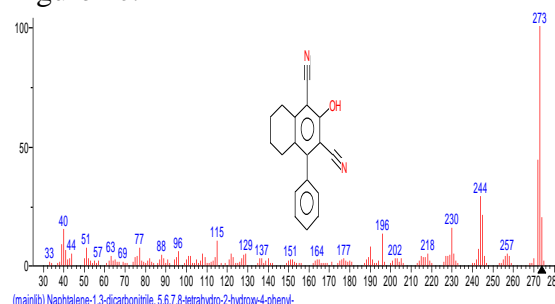


Figure 3c.

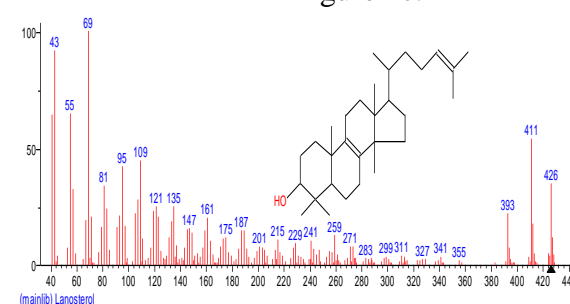


Figure 4c.

Result of the distilled water extracts of *S. siamea* Leaves Powder presented in Table 1, revealed Phosphoric acid, dimethyl(4-methoxy-3-t-butylphenyl) ester ($C_{13}H_{21}O_5P$) (39.55%); Vitamin E ($C_{29}H_{50}O_2$) (22.95%); Naphtalene-1,3-dicarbonitrile, 5,6,7,8-tetrahydro-2-hydroxy-4-phenyl ($C_{18}H_{14}N_2O$) (14.82%) and Lanosterol ($C_{30}H_{50}O$) (5.67%)



as the first four compounds with the highest percentage area and their chromatograms together with chemical structures (figure 1c – 4c). The use of an alternative solvent, such as water, has increased due to environmental, health and safety awareness; moreover, the cost and economics are also a concern (Wang and Weller, 2006). Duke, (2016) reported some of the antioxidants found to be present in *senna* leaf extract are tetradecanoic acid (synonym: Myristic acid), hexadecanoic acid, heptadecanoic acid, squalene (synonym: 2,6,10,14,18,22-tetracosahexaene), vitamin E (synonym: D-alpha-tocopherol), gamma-tocopherol (synonym: 7,8-dimethyltocol), campesterol (synonym: Ergost-5-en-3.beta.-ol), and β -stigmasterol (synonym: Stigmast-5-En-3-Ol, [3.Beta.]). The insecticide activity of *Cassia siamea* extracts and pure compounds has been reported recently (Kamara *et al.*, 2011; Mamadou *et al.*, 2014). Fresh leaves from *S. siamea* are used for repelling or killing insects such as termites, bed bugs and mosquitoes (Jimoh *et al.*, 2013). In a study conducted by Tapondjou, *et al.*, (2002), it was reported that the powder prepared from dry leaves of *C. ambrosioides* at a dosage of 0.4% killed more than 60% of the bruchids, *C. chinensis*, *C. maculatus*, and *Acanthoscelides obtectus* within 2 days, while a dosage of 6.4% induced total mortality of *Sitophilus granarius*, *Sitophilus zeamais* and *Prostephanus truncatus* within the same exposure period. In many parts of the world, locally available plants are currently in wide use to protect stored products against damage caused by insect infestation (Tripathi, *et al.*, 2009; Khater, 2012).

CONCLUSION AND RECOMMENDATIONS

It could be concluded that findings from this study revealed the presence of numerous compounds in the various extracts from *S. siamea* leaves but only the first four compounds with the highest percentage area (peaks) and their chromatogram together with chemical structures were reported. That Fresh leaves from *S. siamea* contained phytochemicals that are bio insecticidal which have been used for repelling or killing insects such as Coleoptera: (bruchids) in the fields and storage, termites, bed bugs and mosquitoes. *Cassia* species is also used as a natural pesticide in organic farms. It could be recommended that, large-scale cultivation of plants with pesticidal potential such as *Cassia* species should be encouraged and the plants used up should be re-planted periodically. Not only will these ensure sustainable availability of raw materials for pesticide formulations, but the plants will provide cleaner air, reduce desertification, and the by-products will provide food and medicine for humans and animals.

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