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Cycloartanes from the Jamaican ball moss (*Tillandsia recurvata L.*)

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ABSTRACT

Given the high occurrence of cancer worldwide and the major source of the discovery of new lead molecules being medicinal plants, this research undertook investigations of the Jamaican ball moss extract *Tillandsia recurvata, L,* since previous research showed that extracts from this plant displayed promising anti-cancer and anti-inflammatory properties. LC/MS was used to identify the various components in the most bioactive ball moss extracts. Several molecular weight matches cycloartane compounds were observed in differing extracts and the features are consistent with steroid-like structures. The spectral data obtained for these compounds compare well with those reported in literature. These results will prove useful in drug design as anti-cancer and anti-inflammatory agents.

KEYWORDS

Jamaican ball moss;
Tillandsia recurvate;
LS-MS;
Cycloartane.

INTRODUCTION

With approximately 30% of its terrestrial plants being endemic[1], Jamaica is known for its rich biodiversity and its abundant usage of medicinal plants as ethno medicines. Medicinal plants continue to play a role in drug discovery and development because of vast structural diversity of molecules found in the plant kingdom, some of which become new drugs or leads for the development of new drugs[2] *Tillandsia recurvata* L. (Bromeliaceae) which is commonly called the Jamaican Ball Moss or the Old Man's beard is one of the several important plants found in Jamaica. Previous work done in our lab showed that the Jamaican ball moss exhibits both anti-cancer and anti-inflammatory activities. Mechanistic studies further showed its anti-cancer activity was expressed through the induction of apoptosis in the five cancer cell lines (namely; PC-3 prostate cancer; Kaposi Sarcoma, B-cell lymphoma, breast cancer, and B-16 melanoma[3]; investigated.

While there is no official report that the Jamaican Ball Moss is used in Jamaican ethno-medicinal practices, several countries report its use in their ethnomedicine. The major reported use is in Brazil where the plant is used against rheumatism, ulcers and hemorrhoids[4]. Previous phytochemical studies showed the presence of; five hydroperoxyclycloartanes, a dicinnamate, a flavanone and a caffeic acid ester from the whole plant extracts[5,6] while the methanol extract was shown to possess bioactivity against some histo-

genic cancer cell lines[3].

This research undertook investigations to identify the possible phytochemicals responsible for the observed anti-cancer and anti-inflammatory activities in ball moss and to further isolate these phytochemicals. Since natural compounds are ideal screening agents because once taken in the appropriate concentration, their associations with toxicity should be less when compared with synthetic ones. We believe the identification of these natural isolates will prove useful in drug design aimed at treating selected cancers will fewer side effects as well as being anti-inflammatory agents.

Identification of the various components is based on LC/MS ions as they correlated to expected literature data[5,7-9]. Several molecular weight matches cycloartane compounds were observed in differing extracts (TABLE 1). ^{1}H NMR analysis was performed and the features are consistent with steroid-like structures.

TABLE 1 : Cycloartanes identified by LC-MS in ball moss (*Tillandsia recurvate L.*)

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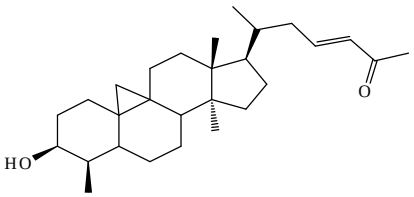
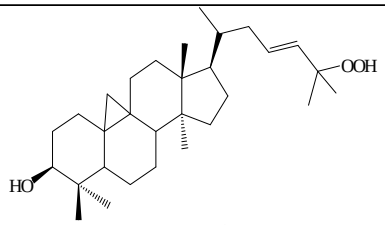
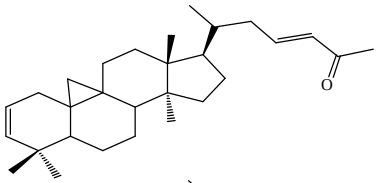
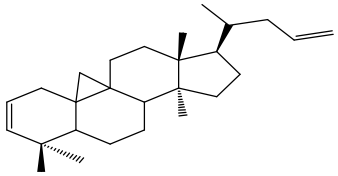
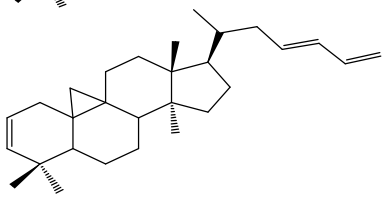
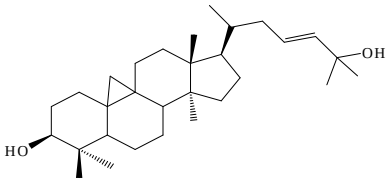
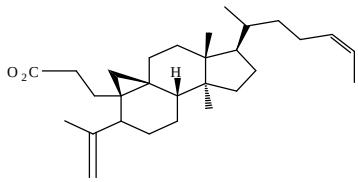
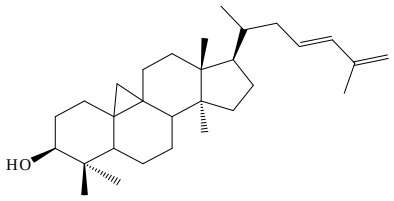
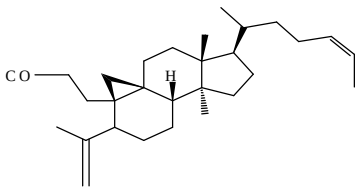
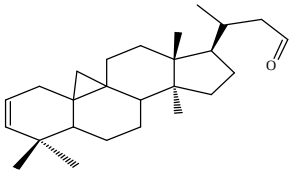
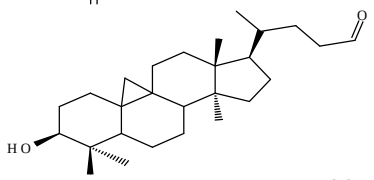
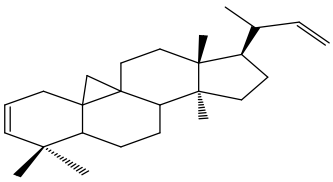
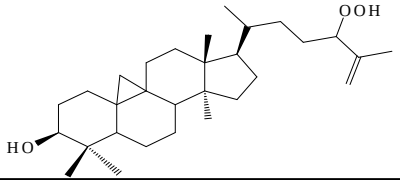
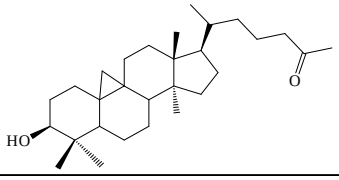
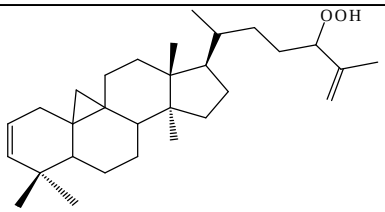
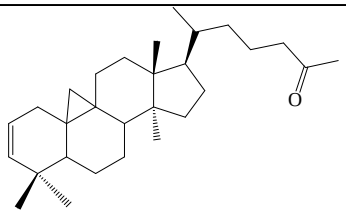
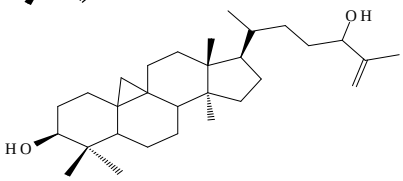
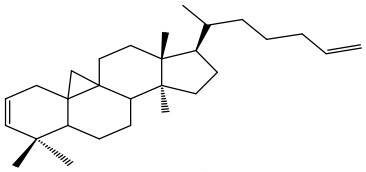
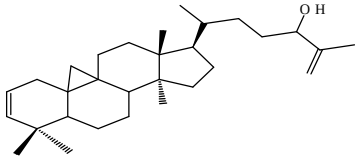
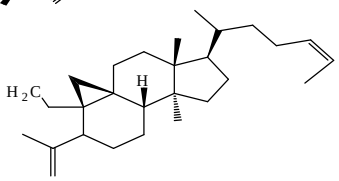
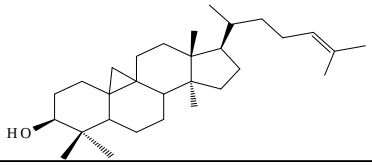
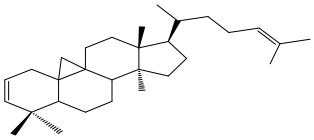
	426.35		458.38
	408.34		366.33
	392.34		442.38
	424.33		424.37
	408.34		368.31
	400.33		352.31
	458.38		428.37

Note

Possible Structure	Exact Mass	Possible Structure	Exact Mass
	440.37		410.35
	442.38		394.36
	424.37		380.34
	426.39		408.38

More and more research groups are identifying the presence of cycloartanes in not only the Tillandsia genus[7] but other plants as well[10], however, bioactivity screens on these compounds are yet still insufficient as a number of research groups are still in the screening phase[11]. Notwithstanding, selected extracts from the Jamaican ball boss showed promising bioactivity (killing 99.1 % of B-16 cells) and several results show consistency in the bioactive fractions containing the cycloartanes which leads us to believe that these compounds could be responsible for the observed bioactivy. These results will prove useful for future work in the arena of anti-cancer treatments and anti-inflammatory agents as well.

LC/MS showed signals consistent for various cycloartanes being present in the Jamaican ball moss extracts from which the promising anti-cancer and anti-inflammatory properties were observed.

EXPERIMENTAL

Samples were sonicated (10 ml 1:1 acetonitrile and water with 0.05% formic acid) for 15 minutes after which they were analyzed using an Agilent 1100 series MSD electrospray (source in positive and negative ion modes). Further analysis was conducted using a decreased fragmentor power of 20. LCMS with UV detection was accomplished using an Agilent diode array detector. HPLC separation was achieved using a Phenomenex Luna C18 column, 5µm (2 x 50mm) at a flow rate of 0.3ml/min.

PLANT MATERIAL

The whole *T. recurvata* plant was collected from trees and electricity poles in Kingston, Jamaica and a voucher specimen (IJ 3411) was deposited at the Institute of Jamaica Herbarium.

EXTRACTION AND FRACTIONATION

The plant material (284g) was air dried under shade, pulverized into a powder and extracted at room temperature for 48 hours in the dark with 95% ethanol followed by CH_2Cl_2. Following the addition of water to the ethanol extract, a further extraction was carried out in hexane. The hexane extract was combined with the CH_2Cl_2 extract and evaporated to dryness resulting in a gummy dark green residue (8.55g). 3.32g of the residue was fractionated by dry column flash chromatography on Si gel using hexane/ CH_2Cl_2/ethyl acetate and mixtures of increasing polarity yielding 9 fractions

(F1- F9). The fractions were subjected to HPLC (column Phenomenex Luna C18, 5µm, 2×50mm; eluent, acetonitrile with 0.05% MeOH). Cycloartanes were identified in all 9 fractions.

Sonication of the samples was carried as previously mentioned yielding an insoluble solid that was filtered prior to LC-MS.

COMPETING INTEREST

The authors declare no competing interest at this time

AUTHOR'S CONTRIBUTION

Conceived and designed the experiments: HICL and JB, Acquisition of data: CW and NT, Analyzed and interpreted the data: NT and SB. Wrote the paper: SB and NT, Critical and intellectual revision of the article contents: SB and NT. All authors read and approved the final manuscript.

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