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(12E, 14E, 18Z)- 13, 15, 17, 19-tetramethyltritriaconta-12, 14, 16, 18- tetraene isolatied and identified in *Chrysophyllum albidum* acetone leaf extract

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Abstract
Chrysophyllum albidum leaves were collected from their natural habitat of plain sandy soil of coastal plain sands in Calabar Municipality Cross River State, Nigeria. The sample were air-dried for two weeks and then milled into fine powder using a milling machine. The method of cold maceration was used in the extraction. The spectroscopic measurements were done on the isolates from the acetone extract of the leaf of *C. albidum.* The structural elucidation by spectroscopic methods (IR, ^{1}H NMR, ^{13}C and NMR) of *C. albidum* yielded a new compounds characterized as (12*E*, 14*E*, 18*Z*)-13, 15, 17, 19- tetramethyltritriaconta-12, 14,16,18- tetraene.

Keywords: *Chrysophyllum albidum*, acetone, extraction, spectroscopic, spectral analysis

Introduction

In developing countries, medicinal plants are used in traditional medicine (Gawai *et al.*, 2013) and most conventional medicines, food supplements, folk medicines and pharmaceutical intermediate are derived from medicinal plants (Ogbonnia *et al*., 2013) [8]. Ushie *at al.* (2014) [13] explained that the is presence of some phytochemicals which are medicinally active constituents in the *C. albidum* leaf extracts and these phytochemical compounds identified have been documented by many researchers to be bioactive and have been confirmed by previous workers to have medicinal as well as physiological activity and therefore could be said to be responsible for the efficacy of the leaves of the plants studied in treatment of different ailments. They are consumed as food or snack and thus serve as a delicacy and alternative source of food. *C. albidum* fruit taken as source of vitamins and sperm booster; generates income (Chima *et al.*, 2012) [4]. The fleshy pulp of *C. albidum* fruits contains: high amount of ascorbic acid (higher than those of orange and guava) vitamins, irons, flavours to diets. tannins, flavanoids terpenoids, proteins, carbohydrates and resins (Adisa *et al*., 2003). Also, oil extracted from the seed is used for soap making. The seeds are also used for local games or discarded. The plant is a crop of commercial value in Nigeria (Oboh, 2009) [7]. The fleshy fruit pulp is suitable for jams and is eaten especially as snack by many locals (Amusa *et al*., 2000).

Spectroscopy is the study of quantized interaction of energy (typically electromagnetic energy) with matter. In organic chemistry, we typically deal with molecular spectroscopy i.e the spectroscopy of atoms that are bond together in molecules (Field *et al.*, 2008) [5]. Preparative thin-layer chromatography has long been a popular method of analysis and isolation of organic natural and synthetic compounds. PTLC does not, however, require expensive equipment; separations can be effected rapidly and the amount of material isolated generally falls into the 1mg to1g range, which is certainly sufficient for structure elucidation purposes. (Satyajit *et al*., 2006) [11]. Ultra violet spectroscopy is very useful to measure the number of conjugated double bonds and also aromatic conjugation within the various molecules. It distinguishes between conjugated and non-conjugated double bonds and non-conjugated systems; α, β- unsaturated carbonyl compounds from β, γ-analogues; homoannular and heteroannular conjugated dienes etc (Sharma, 2008) [12].

Infrared spectroscopy is particularly valuable for determining the kinds of functional group present in an organic molecule (Brown & Forte 2001) [3] and thus, generally useful in elucidation of structure of mainly organic molecules (Ogugbuaja, 2000) [9]. Nuclear magnetic resonance spectroscopy (NMR), has become the preeminent technique for determining the structure of organic compounds. Sharma (2008) [12] explained that Nuclear magnetic resonance spectroscopy is an important tool in the hands of an organic chemist for getting information from the spectrum of an unknown compound. It provides a complete insight into the environment and the arrangement of atoms within a molecule. Nuclear magnetic resonance spectroscopy induces changes in the magnetic properties of certain atomic nuclei, notably that of hydrogen (hydrogen atoms in different environment can be detected, counted and analyzed for structure from determination). The aim of the study was to elucidate the structure of the bioactive components from the acetone leaf extract of *Chrysophyllum albidum* using the spectroscopic analysis of the isolated pure active components i.e interpretation and structural illustration by spectral analysis (UV, IR, ^{1}HNMR &^{13}CNMR) (Ushie *et al*., 2018) [14]

Materials and Methods Sampling and Extraction

Chrysophyllum albidum leaves were collected from their natural habitat of plain sandy soil of coastal plain sands in Calabar Municipality (04º 15ºN; 08º 25ºE), Nigeria. The sample were air-dried for two weeks and then milled into fine powder using a milling machine.

The method of cold maceration was used in the extraction using acetone (Pavia 1976) [10]. The extracts of the leaves was prepared by soaking 100 g of each in 250 ml hexane for four days with frequent agitation until soluble matter is dissolved. The resulting mixture was filtered by gravity filtration and the filtrate was concentrated by evaporation using rotatory evaporator and weighed. The extract was in a refrigerator under argon condition until required for testing.

Spectroscopic Measurements

The spectroscopic measurements were done on the isolates from the acetone extract of the leaf of *chrysophyllum albidum.* The spectroscopic measurements were infrared spectroscopy (IR) and nuclear magnetic resonance NMR measurements. Electronic absorption spectra were recorded on Perkin Elmer Lambda 900 UV-V is NIR spectrometer and were recorded in 10nm path cuvette. An infrared spectrophotometer model Brunker IFS 66 V/S was used to record infra-red measurements. NMR spectra were obtained with a Brunker AVANCE 400 (400 MH_z) Fourier transform NMR spectrometer with chemical shifts reported in parts per million (ppm) with respect to TMS.

Results

FTIR Spectra for Purified Acetone Fraction

The IR spectrum displayed C-H stretch in aliphatic hydrocarbon at 2928cm^{-1}. C=C double bond in aliphatic hydrocarbon at 1600. IR Analysis of isolated/purified component of purified acetone fraction was carried out and the result is summarized in Table 1 below.

^{1}HNMR and ^{13}CNMR Spectra Interpretation for Acetone Fraction (AF)

The ^{1}HNMR revealed the signal at δ 0.87 is due to methyl groups. The signal at δ 1.99 due to methyl groups attached to unsaturated carbon atoms. The signal at δ 1.25 is due to long chain methylene protons. The signal at δ 2.05 is due to the methylene group attached to C=C group. The signal at δ 5.11 is due to the presence of unsaturated protons. The result is presented in Table 20. The ^{13}CNMR spectral data shows the signal at δ 14.27 suggests the presence of methyl group. The signals at 16.15, 16.19 and 17.83 are due to the presence of the methyl groups attached to unsaturated carbon atoms. The remaining signals at δ 22.85, 25.84, 26.83, 28.43, 29.11, 29.32, 29.52, 29.67, 29.81, 32.08, 33.98, 39. 89 are due to long chain mehylene groups. The result is presented in Table 20.

Table 1: The IR Spectral Data and Interpretation of Isolated Acetone Component

Frequency range,cm^{-1}	Functional group	Interpretation
2928	C-H stretch	Aliphatic hydrocarbon
1600	C=C double bond	Aliphatic hydrocarbon

Table 2: ^{1}H NMR and ^{13}C NMR data for Purified Acetone Fraction

C- Positions	Carbon type or group	Carbon Signal (δ)	Proton Signal (δ)
C-1	CH_3	14.27	0.87s
C-2	CH_2	22.85	2.00m
C-3	CH_2	25.84	2.05m
C-4	CH_2	26.83	2.06m
C-5	CH_2	28.43	2.08m
C-6	CH_2	29.11	2.00m
C-7	CH_2	29.32	2.05m
C-8	CH_2	29.52	2.06m
C-9	CH_2	29.67	2.08m
C-10	CH_2	29.81	2.00m
C-11	CH_2	32.08	2.05m
C-12	CH	139.44	5.11s
C-13	C	114.22	-
C-14	CH	135.26	5.11s
C-15	C	124.44	-
C-16	CH	135.06	5.11s
C-17	C	124.47	-
C-18	CH	131.141	5.11s
C-19	CH_2	39.89	2.00m
C-20	CH_2	33.98	2.05m

C-21	CH2	39.89	2.06m
C-22	CH2	22.85	2.08m
C-23	CH2	25.84	2.00m
C-24	CH2	26.83	2.05m
C-25	CH2	28.43	2.06m
C-26	CH2	29.11	2.08m
C-27	CH2	29.32	2.00m
C-28	CH2	29.52	2.05m
C-29	CH2	29.81	2.06m
C-30	CH2	32.08	2.08m
C-31	CH2	33.98	2.06m
C-32	CH2	22.85	2.08m
C-33	CH3	14.27	0.87s
Me	CH3	16.15	1.99s
Me	CH3	16.19	1.99s
Me	CH3	17.83	1.99s

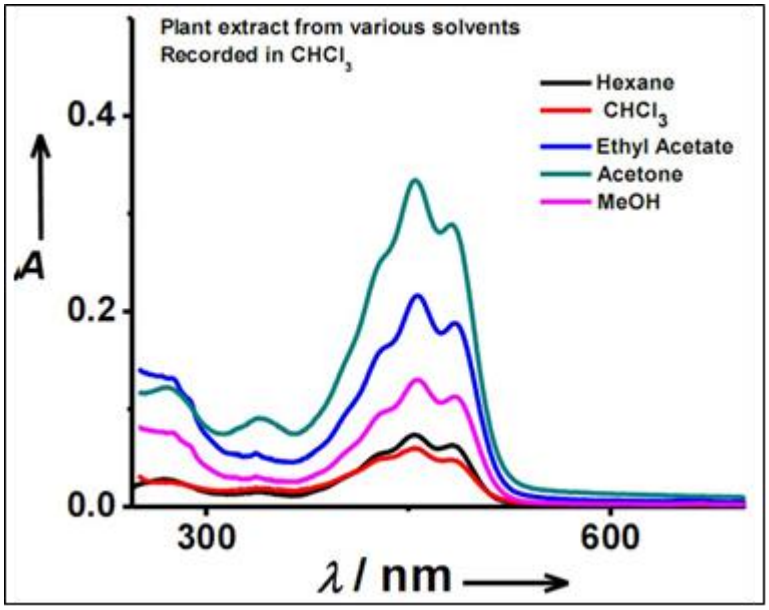

Fig 1: UV for various Fraction

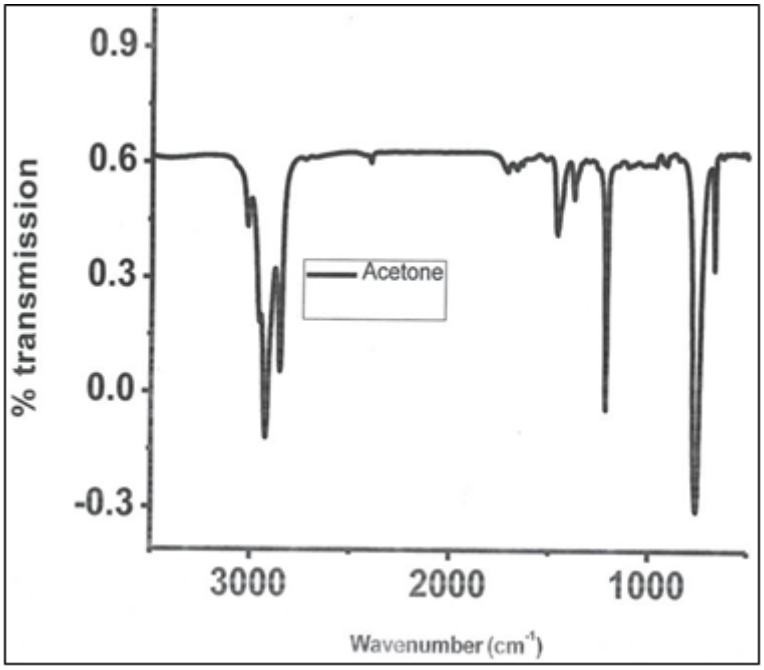

Fig 2: IR for Acetone Extract

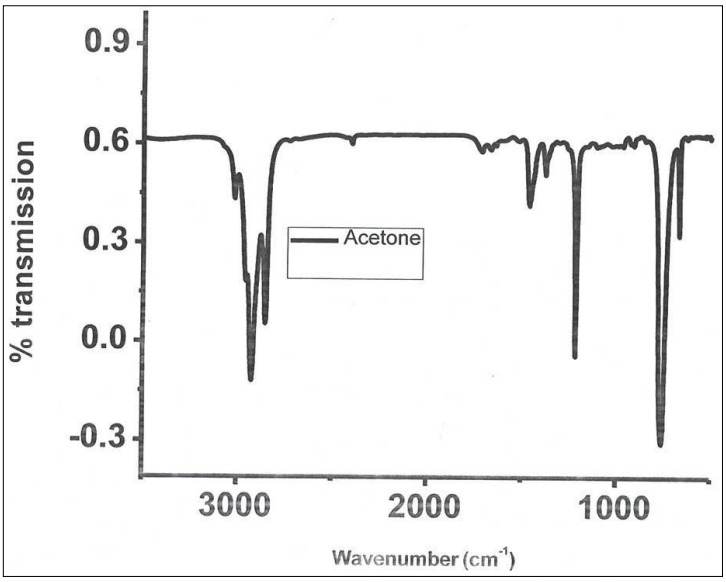

Fig 3

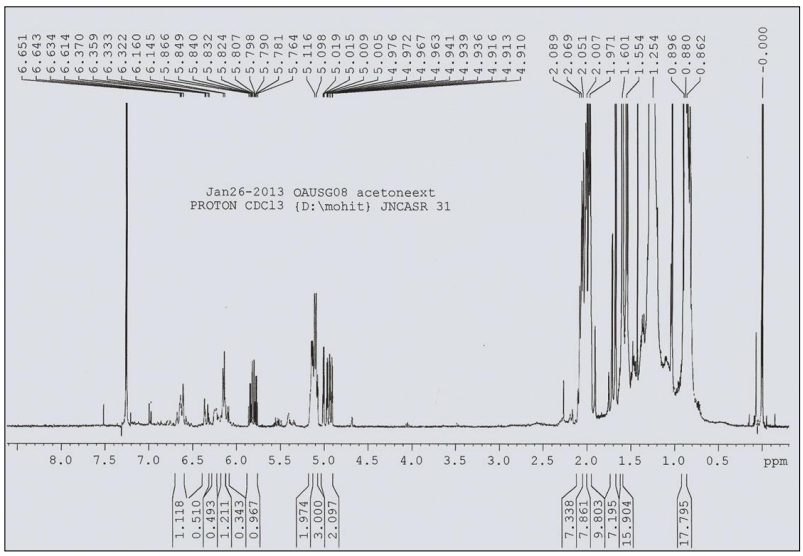

Fig 4: ^{13}HNMR for Acetone Fraction

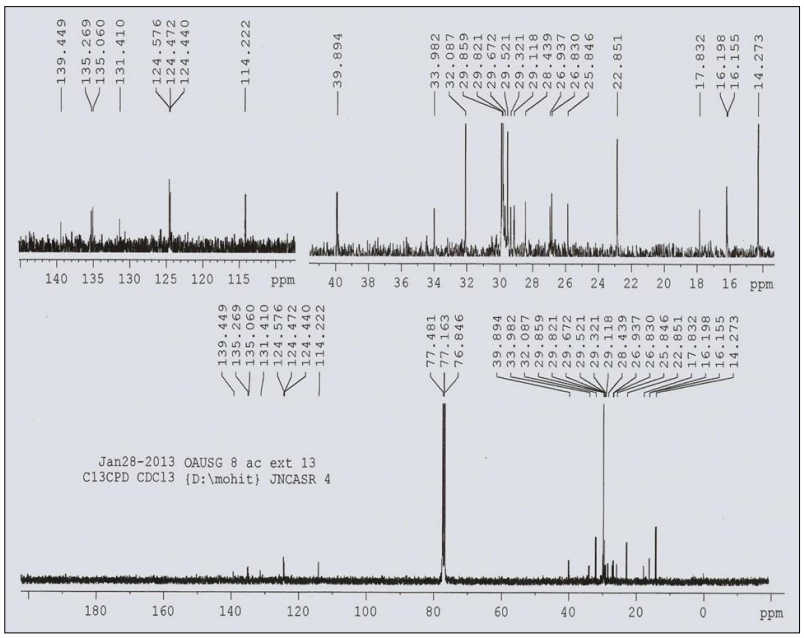

Fig 5: ^{13}CNMR for Acetone Fraction

Discussions

TLC and column chromatography were used for purification that was performed on the acetone extract. The IR spectra showed the presence of C-H group by exhibiting absorption bands at 2928 (stretching frequency) and C=C group by exhibiting a band at 1600 cm^{-1}. This suggests the absence of any other functional groups in the compound. The UV-Visible spectrum shows maximum absorptions at 450 and 470 nm suggesting a high degree of conjugation in the molecule.

In the ^{1}HNMR the signal at δ 0.87 is due to methyl groups, at 1.99 due to methyl groups attached to unsaturated carbon atoms, the broad singlet at δ 1.25 due to long chain methylene protons. The multiplet at δ 2.05 is due to the methylene group attached to C=C group and the signal at δ 5.20 is due to the presence of unsaturated protons. The above assignments are confirmed by the ^{13}C-NMR spectral data. The signal at δ 14.27 suggests the presence of methyl group. The signals at 16.15, 16.19 and 17.83 are due to the presence of the methyl groups attached to unsaturated carbon atoms. The signal at δ 114.22, 124.44, 124.47, 124.57, 131.41, 135.06, 135.26 and 139.44 are due to the presence of four double bonds. Among them four are trisubstituted and four carbons are tetra substituted. The remaining signals at δ 22.85, 25.84, 26.83, 28.43, 29.11, 29.32, 29.52, 29.67, 29.81, 32.08, 33.98, 39. 89 are due to long chain mehylene groups. Based on the above data and the explanation in the result, the compound 3 may be assigned as a long chain hydrocarbon (12*E*, 14*E*, 18*Z*)- 13, 15, 17, 19-tetramethyltritriaconta-12, 14,16,18- tetraene.

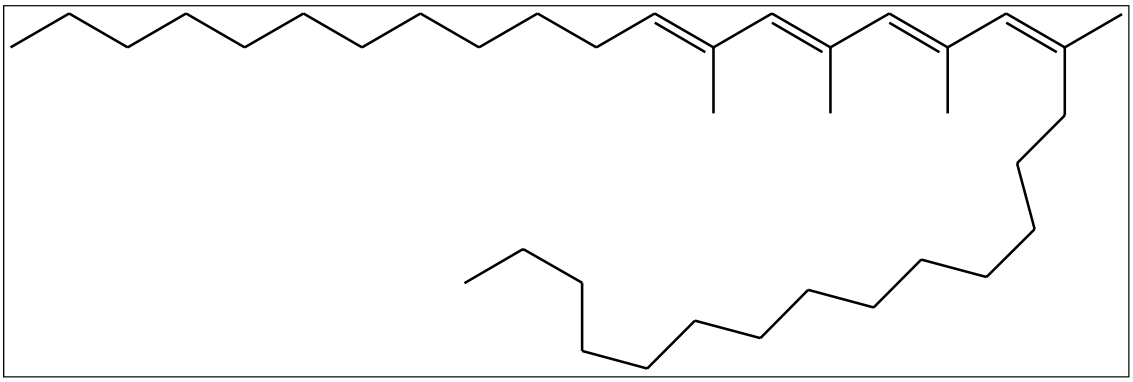

Fig 6: (12E, 14E, 16E, 18Z)-13, 15, 17, 19-tetramethyltritriaconta-12, 14, 16, 18-tetraene

Conclusion

It can be concluded that the structural elucidation by spectroscopic methods (IR, ^{1}H NMR, ^{13}C and NMR) of *C. albidum* yielded a new compounds characterized as (12*E*, 14*E*, 18*Z*)- 13, 15, 17, 19- tetramethyltritriaconta-12, 14,16,18- tetraene.

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