OPEN ACCESS
Scholars International Journal of Biochemistry
Abbreviated Key Title: Sch Int J Biochem
ISSN 2616-8650 (Print) |ISSN 2617-3476 (Online)
Scholars Middle East Publishers, Dubai, United Arab Emirates
Journal homepage: https://saudijournals.com

Original Research Article

GC-MS and FTIR Analysis of Aqueous Extract of *Citrillus lanatus* (Water Melon) Rind

Bichi S. A[1*], Abdulmumin Y[1], AbdulmuminT.M[1], Muhammad I.A[1], Murtala M[1], DalhatuM.M[1], Amina L. A[1], Sarki S.I[1], Danjaji H.I[2] and Mustapha R.K[3]

[1]Department of Biochemistry, Kano University of Science and Technology, Wudil Kano, Nigeria
[2]Department of science Laboratory Technology, Kano University of Science and Technology, Wudil Kano, Nigeria
[3]Department of Chemistry, Yusuf Maitama Sule University, Kano, Nigeria

DOI: 10.36348/sijb.2022.v05i04.001 | **Received:** 12.03.2022 | **Accepted:** 18.04.2022 | **Published:** 27.04.2022

***Corresponding author:** Bichi S. A
Department of Biochemistry, Kano University of Science and Technology, Wudil Kano, Nigeria

Abstract

Over the years, the *Citrulluslanatus* rind has been considered as waste and disposed ignoring the fact that it can be utilized as a value-added product and has many health benefits. The watermelon rind constitutes nearly a third of the watermelon weight. The evaluation of *Citrulluslanatus* (watermelon) rind as a possible raw material for a pharmaceutical and as a value added product of the fruit rind and this will encourage cultivation, utilization and possible application of water melon rind in the treatment of various diseases. The aim of this research work is to determine the functional group using FTIR and chemical components of aqueous extract of watermelon rind using Gas Chromatography–Mass Spectrophotometry (GC-MS)**.** Dried powder of *Citrulluslanatus* (water melon) rind extracts was used for FTIR analysis. 10 mg of the dried extract powder was encapsulated in 100 mg of KBr pellet, was prepared to obtain a translucent sample discs. The powdered of *Citrulluslanatus* was loaded in FTIR analysis (Shimadzu, IR Affinity 1, Japan), having a Scan range from 400 to 4000 cm-1. While The phyto-constitutes of *Citrulluslanatus* (water melon) aqueous rind extract were determined by Gas Chromatography (Agilent 6890 series) coupled with HP-5MS column mass spectrometer at column temperature of 30°C and was heated to 300°C at 10°C for 5min. The helium was used as carrier gas at a flow of 1.0ml/min. The identification of the constituents of water melon rind aqueous extract was performed by matching their mass spectra and retention indices with those obtained from authentic samples and/or NSIT/Wiley spectra libraries, using different types of search (PBM/NIST/AMDIS) and available literature data. The FTIR analysis *Citrilluslanatus* aqueous rind extract shows different absorption band which indicated the characteristic peak of different functional groups such as OH, C=O C-O, H-N, C-H etc which are associated with phenols, ketones, flavonoids, aldehydes, amides, alkenes etc. while the Gas Chromatography-Mass spectrometry analysis of aqueous extract of watermelon rind reveals the presence of different fatty-acid, methyl ester and volatile organic compounds this includes Hexadecanoic acid,2-hydroxy-1-(hydroxymethyl)ethyl ester, Eicosane,2methyl-, 1-Hexadecanesulfonic acid,3,5-dichloro-2,6-dimethyl-4-pyridyl ester, 9,12-Octadecadienoic acid(Z,Z)-2,3-dihydroxypropyl ester, Estra-1,3,5(10)-trien-17β-ol etc**.** The finding showed that *Citrilluslanatus aqueous rind extract* have thirty-one (31) bioactive constituents of pharmacological important identified by GC-MS and FTIR analysis. These compounds from *Citrilluslanatus* rind can be used in pharmaceutical industry for development of novel lead drugs for management of diseases.

Keywords: *Citrulluslanatus*, Rind, Phytoconstituents, GC-MS, FTIR.

INTRODUCTION

The plant kingdom has proven to be the most useful in the treatment of diseases and have provides an important source of material to pharmaceuticals industries worldwide. The most important of these bioactive constituents of plants are steroids, terpenoids, carotenoids, flavanoids, alkaloids, tannins and glycosides. Plants in all facet of life have served a valuable starting material for drug development (Ajibesin, 2011). Antibiotics or antimicrobial substances like saponins, glycosides, flavonoids and alkaloids etc are found to be distributed in plants, yet

these compounds were not well established due to the lack of knowledge and techniques.

Watermelon is used amazingly for it nutritional and medicinal values because of its high water content which contained sugar and energy booster and hydrate body during dehydration, especially during the hot seasons (Ayoola *et al.,* 2011). Watermelon's Seeds, the dried seeds (dark flat) of the fruit are used as snacks when salted and roasted in China, Israel, etc. In Africa, the seeds are made into coarse flour or oil may be extracted from them and use for domestic consumption, and the juice can be extracted from watermelon to produced wine. The imperfect fruit of the Watermelon are used as Livestock feed, while the immature fruits can be prepared and used as summer squash (Stray, 1998).

Watermelon was said to possess high level of antioxidants which decreases the risk of kidney stone and bone loss due to old age, and it is a powerful diuretic diet, has the availability of amino acids and Beta -Carotene which protect heart disease. Also rich in lycopene, which is a pigment that gives the red color that naturally, occurred in *Citrulluslanatus* which prevent ailments of prostate and oral cancer. It is a good source of vitamins such as A, B, C, and Thiamine.

The seeds of the watermelon are said to contained considerable amount of minerals such as calcium, iron, manganese, phosphorus, potassium, sodium, zinc, copper and magnesium which assist in the growth and development of the healthy body that take part in metabolic activities of all living organisms (Worthington-Roberts, 2008).

In some Asian and African countries, up to 80% of the population relies on traditional medicine for their primary health care needs. When adopted outside of its traditional culture, traditional medicine is often called complementary and alternative medicine.

The World Health Organization notes, that "inappropriate use of traditional medicines or practices can have negative or dangerous effects" and that "further research is needed to ascertain the efficacy and safety" of several of the practices and medicinal plants used by traditional medicine systems. Practices known as traditional medicines include Ayurveda, Siddha medicine, Unani, ancient Iranian medicine, Irani, Islamic medicine, traditional Vietnamese medicine, traditional Chinese medicine, traditional Korean medicine, traditional African medicine, and many other forms of healing practices(WHO, 2002).

2. METHODS

Collection and Identification

Citrulluslanatus (water melon) was collected from Wudil farm and was identified at Biology Department, Kano University of Science and Technology; the collected fruits were rinsed several times with distilled water before subjecting them to analysis. Fresh samples of the fruits were sliced with cleaned knife to separate the rind (exocarp) from the pulp (mesocarp). The seeds were carefully removed from the pulp. The rind was chopped into tiny cubes while the pulp was shredded and the rind was bench dried for twenty days and pulverized into coarse powder and kept in polythene bags at room temperature for extraction.

Extraction of the water melon rind extract

Exactly 5 grams of the crushed watermelon rind was carefully weighed and transferred into a clean 50ml conical flask and diluted with 10ml of distilled water. The 50ml conical flask was then placed as a shaking water-bath and heated at a temperature of 60ºc for ten minute to increase the value of the water soluble polyphenols in the watermelon rind extract. After the ten minute, the conical flask was removed and cool. The cold watermelon rind was then filtered using Whatman number 1 filter paper. A light-green colored filtrate was obtained as the watermelon rind aqueous extract. The aqueous extract was allowed to dry at a room temperature and it was dissolved in methanol and taken for GC-MS and FTIR analysis.

Fourier Transform Infrared (FTIR) Spectroscopy Analysis of Water Melon Rind Aqueous Extract.

Dried powder of *Citrulluslanatus* (water melon) aqueous rind extracts was used for FTIR analysis. 10 mg of the dried extract powder was encapsulated in 100 mg of KBr pellet, was prepared to obtain a translucent sample discs. The powdered of *Citrulluslanatus* was loaded in FTIR machine (Shimadzu, IR Affinity 1, Japan), having a Scan range from 400 to 4000 cm-1.

Gas Chromatography – Mass Spectroscopy (GC-MS) Analysis of *Citrulluslanatus* (Water Melon) Rind Aqueous Extract.

The phyto-constitutes of *Citrulluslanatus* (water melon) rind extract were determined by Gas Chromatography (Agilent 6890 series) coupled with HP-5MS column mass spectrometer at column temperature of 30°C and was heated to 300°C at 10°C for 5min. The helium was used as carrier gas at a flow of 1.0ml/min. The identification of the constituents of water melon rind aqueous extract was performed by matching their mass spectra and retention indices with those obtained from authentic samples and/or NSIT/Wiley spectra libraries, using different types of search (PBM/NIST/AMDIS) and available literature data (Kulkarni *et al*., 2015).

3. RESULTS AND DISCUSSION

FT-IR analysis of aqueous extracts of *Citrilluslanatus*(watermelon) rind.

Citrilluslanatus aqueous rind extract shows characteristics absorption band at 3622, 3946, 3819,

3674, 3651 and 3525cm^{-1} which indicated the presence of OH band stretch and this reveals phenols while the peak band at 3867,3905, and 3838cm^{-1} indicated the presence of C-H band stretch indicated for aldehyde. Also the band observed at 780,817 and 672cm^{-1} were for alkyl halides. The characteristics absorption band exhibited at 2113, 2206, 2530 and 1950cm^{-1} were for functional group C=O stretch which indicated the presence of aromatic ketone. Also, the band observes at 3353, 3320, 3264 and 3294cm^{-1} was for Amine due to N-H stretch and 1596cm^{-1} was for primary amine due to the N-H bend. The absorption band at 2151, 2050, 2020 and 2091cm^{-1} showed the presence of C-N stretch which indicates flavonoids and also band observed at1242 and 1030cm^{-1} were for Ethers due to C-O bend. The absorption at 719 and 2921cm^{-1} indicated the presence of C-H bends which showed Alkenes and also peak at 1393cm^{-1} showed the presence of sulphonamide due to S =O stretch (Table 1/ Figure1).

Table-1.0: FT-IR analysis of aqueous rind extracts of *Citrilluslanatus* (watermelon)

Absorption spectrum, frequency (cm^{-1})			
S/N	**Frequency (cm-1)**	**Components Peaks**	**Functional Group**
1	3946	OH stretch	Phenols
2	3819	OH Stretch	Phenols
3	3905	C-H stretch	Alkenes
4	3674	OH Stretch	Amide
5	3867	C-H Stretch	Aldehyde
6	3838	C-H Stretch	Aldehyde
7	3651	OH Stretch	Phenols
8	3525	OH stretch	Phenols
9	3622	OH stretch	Phenols
10	3353	N-H stretch	Amine
11	3320	N-H stretch	Amine
12	3264	N-H stretch	Amine
13	3294	N-H stretch	Amine
14	2921	C-H stretch	Alkenes
15	2530	C=O stretch	Ketones
16	2206	C=O stretch	Ketone
17	2113	C=O Stretch	Ketones
18	2091	C-N stretch	Flavonoid
19	2151	C-N Stretch	Flavonoid
20	2050	C-N stretch	Flavonoid
21	2020	C-N stretch	Flavonoid
22	1950	C=O stretch	Ketones
23	1990	C=O stretch	Ketones
24	719	C-H Bend	Alkenes
25	1596	N-H bend	Primary amine
26	1398	S=O stretch	Sulphonamide
27	1242	C-Cl bend	Ether
28	780	C-Cl stretch	Alkyl halide
29	817	C-Cl bend	Alkyl halide
30	672	C-Cl or C-Br stretch	Alkyl halide
31	1030	C-O bend	Ether

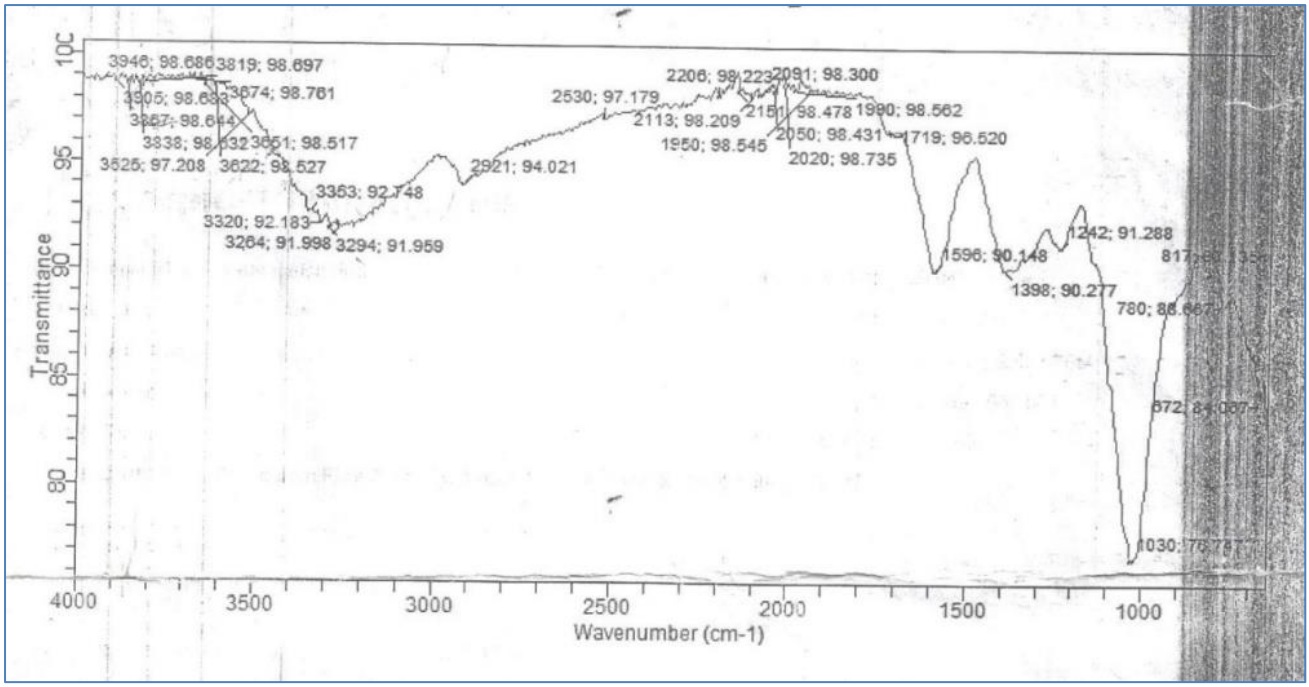
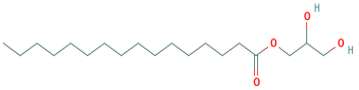
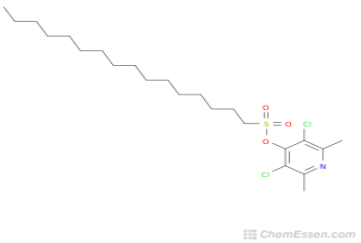


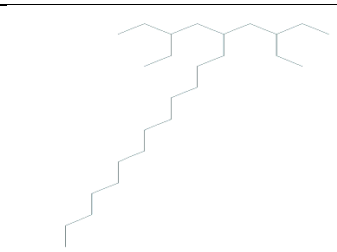
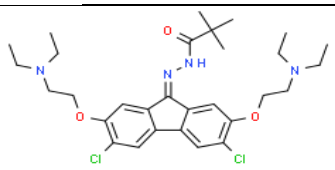
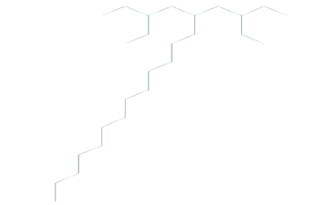
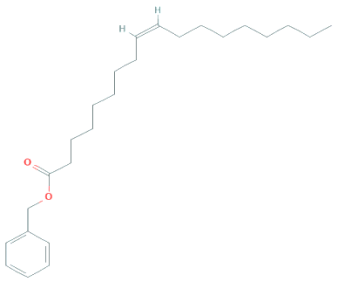
Fig-1: FT-IR spectrum of aqueous rind extracts of *Citrilluslanatus* (watermelon)

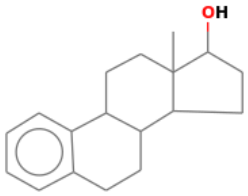
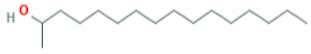
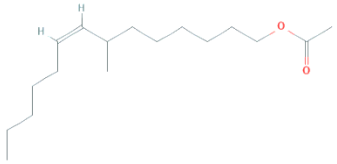
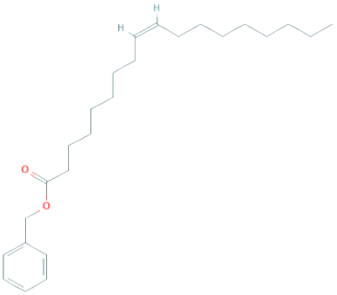
GC-MS analysis of aqueous Rind extract of *Citrilluslanatus*

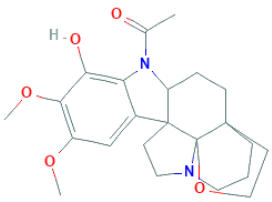
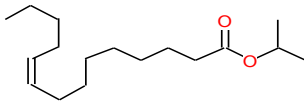
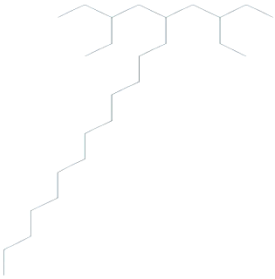
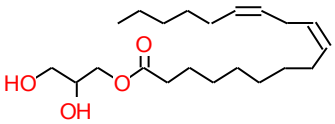
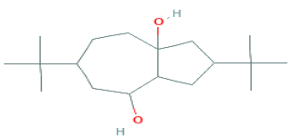
The aqueous rind extracts of *Citrilluslanatus* revealed several peaks which represents different compounds as shown in the chromatogram by Gas Chromatography-Mass Spectrometry analysis in (figure 2). The peaks in the chromatogram were integrated and compared with the database of spectrum of known components stored in the Gas chromatography-mass spectrometry library. The Gas Chromatography-Mass spectrometry analysis of aqueous extract of watermelon rind reveals the presence of different fatty-acid, methyl ester and volatile organic compounds (table 2). Xzz

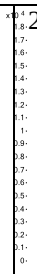
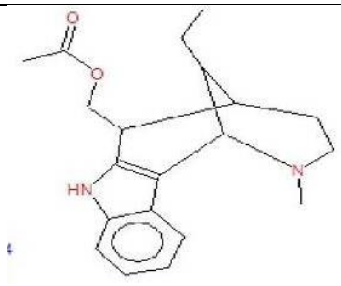
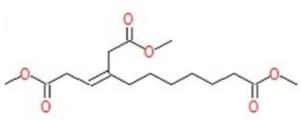
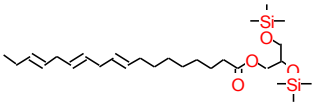
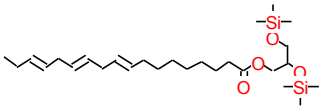
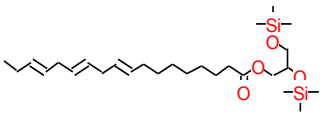
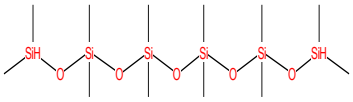
Table-2.0: GC-MS analysis of aqueous Rind extract of *Citrilluslanatus*

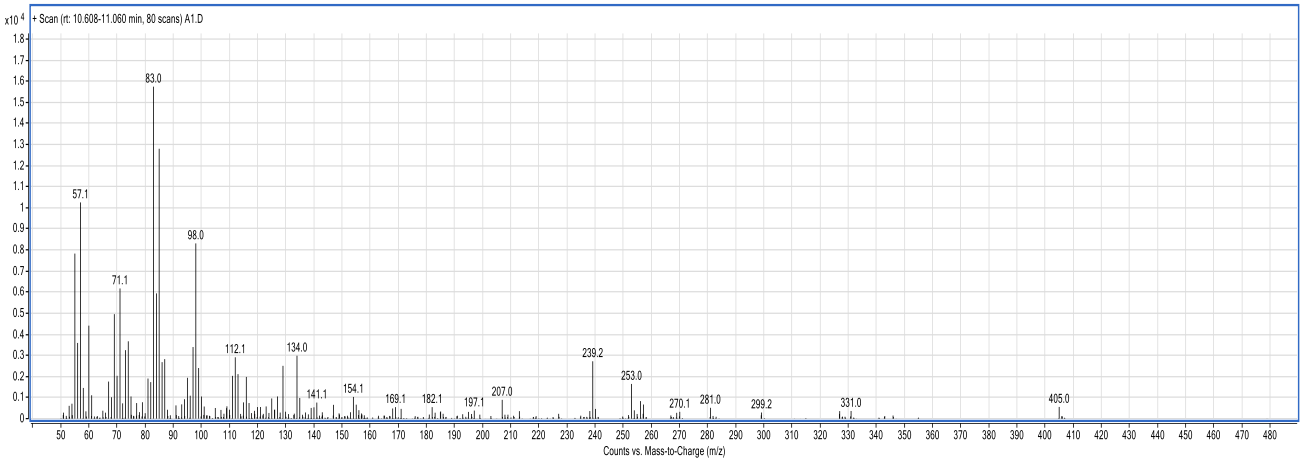
Peak	Retention time	IUPAC Name	Molecular/ Structural Formular	Nature and pharmacological Uses
1.	10.9	Hexadecanoic acid,2-hydroxy-1-(hydroxymethyl) ethyl ester	 $C_{19}H_{38}O_4$	Palmtic acid ester : ,antioxidant, lubricant, pesticides, flavor, nematicide (Imis, 2016)
2.	12.948	Eicosane,2methyl-	$C_{21}H_{44}$	Acyclic alkene; present in petroleum product, generally flavoring agent used in food including condiments and seasonings, fragment and odour agents. (Kennedy *etal*.,2004)
3.	13.183	1-Hexadecanesulfonic acid,3,5-dichloro-2,6-dimethyl-4-pyridyl ester	 $C_{23}H_{39}CL_2NO_3S$	Anti-microbial, antioxidant. (Hamad 2015)

4.	13.446	Octadecane,3ethyl-5-(2-ethylbuthyl)-	 $C_{26}H_{54}$	A s a volatile component and have anti-microbial effect (Thilina Gunathilaka,2019)
5.	14.098	9-(2'2'-Dimethylpropanoilhydrazono)-3,6-dichloro-2,7-bis[2-(diethylamino)-ethoxy]fluorine.		Anti-bacterial, anti-pyrotic analgesic effect and improve immunity. (Nat prod Rep.2004 dec)
6.	14.825	Hepatacosane	$C_{27}H_{56}$	As a volatile oil component and plant metabolites. Anti-microbial and cytotoxic properties. (Martin. 2015)
7.	15.065	Tetracosane	 $C_{24}H_{50}$	An alkene hydrocarbon; suitable solvent used in the synthesis Zns nano-particles. May be employed as the wax component to study the reduction in pour point and hydrocarbon solvent containing wax crystals on addition of polymer additive.
8.	15.615	Octadecane,3-ethyl-5-(2-ethybutyl)-	 $C_{26}H_{54}$	A cylic alkene; A s a volatile component and have anti-microbial effect. (Thilina Gunathilaka, 2019).
9.	16.341	9-Octadecenoic acid(Z)-, phenylmethyl ester	 $C_{25}H_{40}O_2$	Anti-microbial activities. and, antibacterial activites (jeneciu *et al.*, 2012)
10.	16.999	4-Methyl-docosane	 $C_{23}H_{48}$	Perfuming agent, anti-microbial

11.	17.686	Tetradecane,2,6,10-ttimethyl	 $C_{17}H_{36}$	Anti-microbial activities and cytotoxic properties
12.	18.355	Heptacosane	$C_{27}H_{56}$	Anti-microbial and cytotoxic properties. As a volatile oil component and plant metabolites (Martin 2015)
13.	19.042	Estra-1,3,5(10)-trien-17β-ol	 $C_{18}H_{24}O$	Anti microbial activities (P.R 2012)
14.	19.895	2-Hexadecanol	 $C_{16}H_{34}OH$	Palmitic acid; Anti-microbial, anti-inflamatatory, emulsifier and thickening agent (The Good scent company,2018)
15.	22.338	7-methyl-Z-tetradecen-1-olacetate	 $C_{17}H_{32}O_2$	Fatty acid; component of flavouring agent and perfumes.
16.	22.967	9-Octadecenoic acid(Z)-phenylmethyl ester	 $C_{25}H_4O_2$	Antibacterial activities.(jeneciu *et al.*, 2012)
17.	23.648	Ethanol 2-(octadecyloxy)	 $C_{20}H_{42}O_2$	Ant toxicity, anti-microbial (OH DH, *et al.*,1993)

18.	24.095	Apidopermidin-17-ol,1-acetatyl-19,21-epoxy-15,16-dimethyl-	 $C_{23}H_{30}N_2O_5$	Anti-microbial activities. (Adelani-Akande*etal*., 2015)
19.	25.256	Heptacosane	$C_{27}H_{56}$	As a volatile oil component and plant metabolites. Anti-microbial and cytotoxic properties (Martins,2015)
20.	25.846	2-hexadecanol	OH $C_{16}H_{34}0$	Palmitic acid; Antimicrobial,anti-inflamatatory,emulifier and thickening agent,(The Good scent company,2018)
21.	26.664	i-propyl 9-tetradecenoate	 $C_{17}H_{32}O$	Perfuming agent,binding agents, polar emollient used in cometics, antimicrobial,antioxidants,stimul ants (kadhim *etal*;2016)
22.	26.978	Octadecane,3-ethyl-5-(2-ethylbutyl)	 $C_{26}H_{54}$	A s a volatile component and have anti-microbial effect
23.	27.19	9,12-Octadecadienoic acid(Z,Z)-2,3-dihydroxypropyl ester	 $C_{21}H_{38}O_4$	Volatile organic compounds, has anti-microbial activities
24.	27.751	2,6-di-t-butyloctahydroazulene-3a,8-diol	 $C_{18}H_{30}O_2$	As a component in insect repellents

25.	 28.043	Davcarpidan-1-methanol,acetate (ester)	 $C_{20}H_{26}N_2O_2$	Fatty acid; occurs naturally in plants and animals derivatives and its used in cosmetic products, thickeners and hardeners.
26.	28.523	4-methoxycarbonylmethylundec-3-enedioic acid,dimethyl ester	 $C_{16}H_{26}O_6$	Anti-fungal, antioxidants (Saenjum*etal.*, 2012)
27.	29.216	9,12,15-Octadecadienoic acid)-2,3bisi(trimethylsilyl)-oxypropyl ester(Z,Z,Z)		Antibacterial activities, anti-inflammatory, cancer prevention, anti-acne (Hadi Hameed, 2015)
28.	29.565	9,12,15-octadecatrienoic,acid2,3-bisi(trimethylsily)oxylpropyl ester,(Z,Z,Z)		Antibacterial activities, anti-inflammatory, cancer prevention, anti-acne (Hadi Hameed, 2015)
29.	29.839	9,12,15-octadecatrienoic,acid2,3-bisi(trimethylsily)oxylpropyl ester,(Z,Z,Z)		Antibacterial activities, anti-inflammatory, cancer prevention, anti-acne (Hadi Hameed, 2015)
30.	30.555	Hexasiloxane 1,1,3,3,5,5,7,7,9,9,11,11-dodecamethyl		Volatile organic compounds, has antimicrobial activities (Adekunle, 2009)



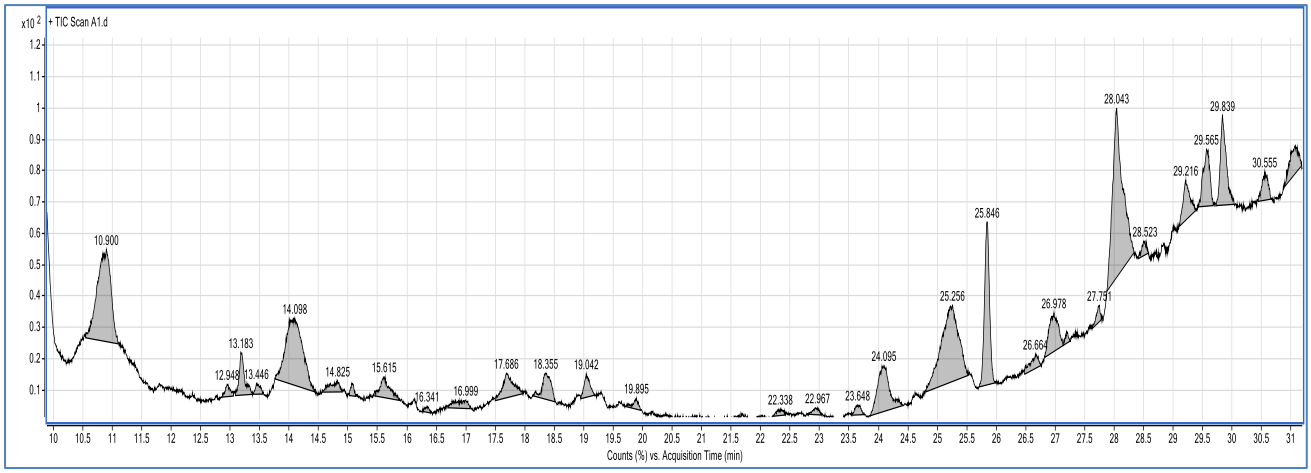


Fig-2: GC-MS Chromatogram of aqueous Rind extract of *Citrilluslanatus*

4. CONCLUSION

The finding showed that *Citrilluslanatus aqueous rind extract* have thirty-one (31) bioactive constituents of pharmacological important identified by GC-MS and FTIR analysis. These compounds from *Citrilluslanatus* rind can beused in pharmaceutical industry for design and develop of novellead drugs for the treatment of diseases.

5. ACKNOWLEDGEMENT

The authors wish to acknowledged the support of Tertiary Education Trust fund (TETFUND) Nigeria and Kano University of Science and Technology Wudil, Kano, Nigeria for sponsoring the Research under Institutional Based Research Grant TETFUND/DESS/UNI/WUDIL/IBR/2018/VOL.I).

6. ETHICAL APPROVAL

As per international standard written ethical permission has been collected and preserved by the author(s).

7. COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

- Adekunle, A.S. (2009). Preliminary assessment of antimicrobial properties of aqueous extract of plants against infectious diseases, Biol Med. 20-24
- Adelani-Akande, T. A., Ajiba L. C., Dahunsi S. O., Oluyori, A. P. (2015). Antibacterial activity of watermelon (Citrulluslanatus) seed against selected microorganisms; African Journal of Biotechnology, *14*(14); 1224-1229
- Kulkarni, A., Govindappa, M., Ramachandra, Y.L., Koka, P. (2015). GC-MS analysis ofmethanol extract of Cassia fistula and its in vitro anticancer activity on humanprostate cancer cell line. *Indo. Am. J. Pharm. Res*. 5, 937–944.
- Kumar, P.P., Kumaravel, S., Lalitha, C. (2010). Screening of antioxidant activity, total phenolics and GCMS study of Vitexnegundo, *Afr.J. Biochem*, 4; 191-195.
- Lakshmipathy, R., & Sarada, N. C. (2015). "A fixed bed column study for the removal of Pb2+ ions by watermelon rind," Environmental Science: *Water Resource Technology*, 244;250
- Leong, L.P., Shui, G. (2002). An investigation of antioxidant capacity of fruits in Singapore markets. Food Chemistry, *76*, 69; 75.
- Mandel, H., Levy, N., Izkovitch, S., & Korman, S. H. (2005). Elevated plasma citrulline and arginine due to consumption of Citrullus vulgaris (watermelon). Berichte der deutschenchemischen Gesellschaft, *28*, 467; 472
- Musmade, A.M., Desai, U.T., Cucumber., & Melon. (1998). *"Handbook of Vegetable Science and Technology:* Production, Composition, Storage, and Processing", Marcel Dekker, Inc., NY, 245-272
- Njoroge, G.N., Gemmill, B., Bussmann, R., Newton, L.E., Ngumi, V.W. (2007). Pollination ecology of Citrulluslanatus at Yatta, Kenya. International Journal of Tropical Insect Science. ICIPE, 24, 73-77.
- Oluyori, A.., Arun, K.S., Olatunji, G.A., Preeti, R., Sanjeev, M., Dipak, D., Ashish, A., Sama, A., & Puli S.R. (2016). Sweet Potato Peels and Cancer Prevention. *Nutrition and Cancer –An International Journal 68*(8); 1330-1337
- Oluyori, A.P., & Olatunji, G. (2016). Phytochemical Analysis, Antimicrobial and Antioxidant Activity of Peels' extracts from Ipomoea batatas Lam; *Journal of International Research in medical and Pharmaceutical Sciences*; 6(4); 157-164.
- Rimando, A.M., & Perkins P. (2005). Determination of citrulline in water melon rind. *J. Chm. A.* 1078(1-2): 196 200.
- Parmar, H.S., Kar, A. (2009). Protective role of Mangiferaindica, Cucumis melon and Citrullus

vulgaris peel extracts in chemically induced hypothyroidism, *Chemico Biological Interactions*,177, 254-258

- Parmar, H.S., Kar, A. (2009). Protective role of Mangiferaindica, Cucumismelo and Citrullus vulgaris peel extracts in chemically induced hypothyroidism, ChemicoBiological Interactions ,177, 254-255
- Quek, S. Y., Chok, N. K., & Swedlund, P. (2007). "The physicochemical properties of spray-dried watermelon powder,"Chemical Engineering Process 46 : 386 – 392. .
- Liu, C., Ngo, H. H., & Guo, W. (2012). "Watermelon rind: agro-waste or superior biosorbent?" *Applied Biochemistry and Biotechnology*, 167; 699–715.
- Schaefer, H., Renner, C., Susanne, S. (2001). Phylogenetic Relationships in the Order Cucurbitales and a New Classification of the Gourd Family (Cucurbitaceae), Taxon, 60, 122–138.
- Shimadzu, I.R., Affinity, l., Japan, A. P. Desbois., & V. J. Smith. (2010). "Antibacterial free fatty acids:activities, mechanisms of action and biotechnological poten-tial," Applied Microbiology and Biotechnology, *85*(6); 1629–1642,
- Thamaraiselvi, P.L., & Jayanthi, P. (2012). Preliminary studies on phytochemicals and antimicrobial activity of solvent extracts of Eichhorniacrassipes (*Mart.*) Solms. Asian *Journal of Plant Science and Research*: *2*(2);115-122.