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Data Article

Dataset of metabolites extracted from African walnut (*Tetracarpidium conophorum*) using two different solvents

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ABSTRACT

A variety of walnut known as *Tetracarpidium conophorum* is widely cultivated in several parts of Africa for its edible nuts. These nuts have been reported for their huge antioxidant, anti-obesity, and anti-depressant potentials, but remain underutilized due to their poor storage and preservation. This is why the nuts are mostly cooked and consumed as snacks whenever in season. This data article reports the untargeted metabolite profile of boiled and dried African walnut extracted using two different mixtures of solvents. The raw nuts obtained from a local market in Osun State, Nigeria, were processed by cooking for 20 min, deshelled, diced, dried at 60 ± 2 °C for 6 h, and stored until further analysis. The dried walnut samples were extracted with acetonitrile/methanol/water (40:40:20 v/v/v) and methanol/water (80:20 v/v) as solvents, before being analysed by gas chromatography high-resolution time of flight mass spectrometry (GC-HRTOF-MS) system. Data obtained from the analysis were further classified into different compounds, including alcohols, esters, hydrocarbons, phytosterols, vitamins, and many more. Their retention time, observed ion mass-to-charge ratio, molecular formula, and average peak areas were also reported. These data thus serve as a source of metabolites comparison for other walnuts, may be useful for the identification of functional compounds available in this ne-

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glected food crop, and encourage its utilization in developing functional foods.

Specifications Table

Subject	Food Science: Food Chemistry
Specific subject area	Processing; Food composition and analysis; Metabolomics
Type of data	Table Figure Spectra
How the data were acquired	Raw walnuts were boiled under pressure for 20 min, sliced and dried at 60 ± 2 °C for 6 h. The dried cooked nuts were further grounded using laboratory mortar & pestle, and then extracted using two different combinations of organic solvents, acetonitrile/methanol/water (40:40:20 v/v/v), and methanol/water (80:20 v/v). The extracts were analyzed using the GC-HRTOF-MS system (LECO Pegasus, St Joseph, USA). This system featured 50,0 0 0 FWMH resolution (full peak with at one-half maximum), mass accuracies/errors of <1 ppm with acquisition rates up to 200 spectra/s, and was equipped with an Agilent 7890A gas chromatograph (Agilent Technologies, Inc., Wilmington, DE, USA). This GC-HRTOF-MS operates at high resolution and is equipped with a Gerstel MPS multipurpose autosampler (Gerstel Inc., Mülheim an der Ruhr, Germany) and a Rxi ®-5ms column (30 m × 0.25 mm ID × 0.25 µm) (Restek, Bellefonte, United States).
Data format	Raw data Analyzed data Filtered data Spectra of commonly identified compounds
Description of data collection	The already processed walnut (1 g) in its ground form was weighed and metabolites were extracted using the 10 mL mixture of different solvents (acetonitrile/methanol/water (40:40:20 v/v/v), and methanol/water (80:20 v/v) in each case. Thereafter, each sample was vacuum concentrated and reconstituted in chromatography-grade methanol (1 mL), then filtered with a 0.22 µm syringe into amber vials. Each sample (1 µL) was auto-injected into the GC-HRTOF-MS machine in triplicates and analyzed. The identities of the metabolite obtained were determined using NIST, Mainlib and Feihn metabolomics databases.
Data source location	Raw African walnuts were sourced from a local market in Modakeke, Osun State Nigeria (N 7°22' 54.848" E 4°16'3.737") on the 27th June, 2021 and processed within 24 h after collection. Thereafter, the extraction and analyses were carried out at the University of Johannesburg, Doornfontein, Johannesburg, Gauteng, South Africa (S 26°11' 32.6" E 28°03' 28.9").
Data accessibility	Raw & processed dataset, and mass spectra of the metabolites have been deposited in the Mendeley repository. It is accessible using the details below: Repository name: Mendeley data. DOI: 10.17632/s9vrhj8tsk.1 Direct URL to data: https://data.mendeley.com/datasets/s9vrhj8tsk

Value of the Data

- The data contributed to the identification of metabolites in African walnuts and provided information on the versatility of different solvent mixtures in metabolite extraction.
- The information provided herein will assist in understanding the usefulness of African walnuts and promote their cultivation to prevent crop extinction.
- The data could be useful for a comparative analysis of the metabolite composition in raw and processed, domestic or foreign walnuts, and the developed products.
- The data would be useful for food processors and researchers aiming to develop novel functional foods from African walnuts.

- The data would be useful in identifying constituents that may be responsible to sensory, functional, and nutritional effects and concentration in developed food products.
- The data would be useful resource for nutritionists, agronomists, food, and data scientists.
- The data indicates that untargeted GC-HRTOF-MS analysis could facilitate the identification of compounds that may be responsible for the nut's health-promoting effects.

Objective

The African walnut (*Tetracarpidium conophorum*) plant has been extensively investigated for its high nutrients, anti-oxidants, anti-diabetic, anti-inflammatory, and other therapeutic benefits. The nut has also been explored in a few food products such as functional cookies [1], however, the nut has remained poorly utilized. This study identified metabolite present in ready-to-eat African walnuts that may enhance the potential exploitation of the nut as food ingredients in the development of functional food products that could solve many health issues.

1. Data Description

The dataset deposited in the repository contain two files (excel sheets and word document). The excel sheet 1 contains the raw data collected from the GC-HRTOF-MS analysis, it described the retention time (min), sample code, observed mass per charge number of ions, formula, area, name and synonym of the compounds extracted. Sheet 2 contains the data that was processed using the DataPrep solutions software and the class of the identified compounds, while sheet 3 represent the common compound that occurred at least two times in three injection in both samples analysed. The sample labelled W1 represent the walnut sample extracted with the mixture of acetonitrile/methanol/water (40:20:20 v/v/v), and W2 represent walnut sample extracted with the mixture of methanol/water (80:20 v/v). In addition, the word document deposited in the repository enclose the spectra of each compound identified in both samples. The spectrum of each compound typically shows a number of signals and the true peak at the highest mass per charge ion ratio. This will provide the scientist community with the structural information and the whole molecule identification.

The metabolite data obtained from extracted walnut samples are presented below. Table 1 represents metabolites obtained from walnut extracted using the mixture of acetonitrile/methanol/water (40:20:20 v/v/v), and methanol/water (80:20 v/v) mixed solvent. The data in each table shows information regarding the name of each extractable compound identified, their retention time, observed ion mass-to-charge ratio, molecular formula and average peak area. These data were generated from GC-HRTOF-MS analysis and the spectra obtained were compared with NIST, Mainlib and Feihn metabolite databases. The raw and analyzed data along with the spectra of the identified compounds are available in a supplementary file deposited in the repository [2]. Figure 1 summarizes the percentage distribution of the compounds found from at least 2 out of 3 injections of each extracted sample from the extraction solvent.

2. Experimental Design, Materials and Methods

2.1. Walnut collection and processing

Matured raw walnuts (*Tetracarpidium conophorum*) were sourced from a local market in Nigeria (N 7°22' 54.848" E 4°16'3.737") on the 27th June, 2021. They were physically cleaned and washed under running water to remove extraneous materials. The nuts were cooked (Pressure Pot, Master Chef, 12L) for about 20 minutes after pressure has been built within the system. The nuts were allowed to cool, de-shelled, and shredded into smaller sizes using a hand grater. The

Table 1
Metabolites identified in the walnut sample that was extracted using the two different solvents mixture.

Retention Time (Min)	Observed Ion *m/z*	Name	Molecular Formula	Average Area	
				W1	W2
Acyclic alkanes					
14.976	268.9873	Eicosane	$C_{2n}H_{42}$	ND	135601
Alcohols/Phenols					
2.987	32.0259	Methyl Alcohol	CH_4O	2517321	ND
12.317	220.1821	Butylated Hydroxytoluene	$C_{15}H_{24}O$	ND	84268
Aldehydes					
7.576	120.0569	Benzaldehyde, 2-methyl-	C_8H_8O	698094	ND
15.962	234.1612	3,5-di-tert-Butyl-4-hydroxybenzaldehyde	$C_{15}H_{22}O_2$	ND	12759
Amides					
21.457, 21.742	142.1226, 156.1383	3-Cyclopentylpropionamide, N,N-dimethyl-	$C_{1n}H_{19}NO$	180548	300932
Amines					
22.582, 22.580	144.1019, 144.1019	Bis(2-(Dimethylamino)ethyl) ether	$C_8H_{2n}N_2O$	421813	382242
Esters					
21.490	225.4713	2-Propenoic acid, 3-(4-methoxyphenyl)-, 2-ethylhexyl ester	$C_{18}H_{26}O_3$	ND	19523
17.921, 17.918	292.2026, 292.2033	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, methyl ester	$C_{18}H_{28}O_3$	27667	32997
30.801	530.4694	Benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-, octadecyl ester	$C_{35}H_{62}O_3$	ND	111822
22.581	219.0679	Carbonic acid, 2-dimethylaminoethyl 2-methoxyethyl ester	$C_8H_{17}NO_4$	ND	418022
21.606, 21.654	170.0830, 219.1150	Carbonic acid, 2-dimethylaminoethyl isobutyl ester	$C_9H_{19}NO_3$	316407	172290
25.181	297.2416	Decanedioic acid, bis(2-ethylhexyl) ester	$C_{26}H_{5n}O_4$	ND	196860
23.307	279.1594	Dicyclohexyl phthalate	$C_{2n}H_{26}O_4$	ND	67658
3.082	88.0519	Ethyl Acetate	$C_4H_8O_2$	ND	8575634
18.124	223.5891	Dibutyl phthalate	$C_{16}H_{22}O_4$	80025	ND
24.783, 24.780	279.1576, 279.1592	Mono(2-ethylhexyl) phthalate	$C_{16}H_{22}O_4$	81688	48734
25.443	503.1072	Phthalic acid, 8-chlorooctyl decyl ester	$C_{26}H_{41}ClO_4$	191547	ND
11.734	149.1072	Succinic acid, 3-methylbut-2-en-1-yl 3-methoxyphenyl ester	$C_{16}H_{2n}O_5$	111337	ND
24.557	328.2897	Octadecanoic acid, 2,3-dihydroxypropyl ester	$C_{21}H_{42}O_4$	ND	646767
25.622	226.9907	Phthalic acid, 4-methylhept-3-yl pentyl ester	$C_{21}H_{32}O_4$	ND	53421
Ethers					
8.715, 12.656	131.1513, 131.1238	1,1,1,2,3,3,3-Heptafluoro-2-methoxypropane	$C_4H_3F_7O$	7819	7624

(*continued on next page*)

Table 1 (*continued*)

Retention Time (Min)	Observed Ion *m/z*	Name	Molecular Formula	Average Area	
				W1	W2
Fatty acid ethyl esters (FAEEs)					
22.989, 22.990	300.2605, 311.2588	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester	$C_{19}H_{38}O_4$	540587	751444
Fatty Acid Methyl Esters (FAMEs)					
19.473, 19.516	292.2395, 292.2398	9,12,15-Octadecatrienoic acid, (Z,Z,Z)-, Methyl 8,11,14-heptadecatrienoate	$C_{18}H_{3n}O_2$	1476801	1179328
19.398, 19.398	294.2553, 294.2552	9,12-Octadecadienoic acid, methyl ester	$C_{19}H_{34}O_2$	1064918	1214274
21.261, 21.260	293.2822, 293.2820	9-Octadecenoic acid (Z)-, methyl ester	$C_{19}H_{36}O_2$	69858	151120
17.665, 17.663	270.2551, 270.2553	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	2729756	2953203
19.674, 19.673	298.2860, 298.2868	Methyl stearate	$C_{19}H_{38}O_2$	1685892	2403367
23.094, 20.541	219.2037, 223.6918	Tridecanoic acid, methyl ester	$C_{14}H_{28}O_2$	197152	112146
23.035	199.1692	Undecanoic acid, methyl ester	$C_{12}H_{24}O_2$	118716	ND
19.451, 19.448	296.2707, 296.2708	trans-13-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	1003628	812770
Hydrocarbons					
13.420	131.1726	Butane, 1,1,1,2,3,3,4,4,4-nonafluoro-2-(trifluoromethyl)-	C_5F_{12}	6718	ND
15.032, 8.761	175.0624, 155.1432	Hexadecane	$C_{16}H_{34}$	84165	41015
11.656	139.0982	Pentadecane	$C_{15}H_{32}$	ND	79208
Indoles					
8.677, 8.680	117.0573, 117.0573	Indole	C_8H_7N	59297	46098
Ketones					
17.716, 17.710	276.1710, 267.0356	7,9-Di-tert-butyl-1-oxaspiro(4,5)deca-6,9-diene-2,8-dione	$C_{17}H_{24}O_3$	48189	94470
15.033, 15.032	188.1192, 219.1732	Methanone, (1-hydroxycyclohexyl)phenyl-	$C_{13}H_{16}O_2$	219837	287750
9.877	269.0488	7-Chloro-1,3,4,10-tetrahydro-10-hydroxy-1-[[2-[1-pyrrolidinyl]ethyl]imino]-3-[3-(trifluoromethyl)phenyl]-9(2H)-acridinone	$C_{26}H_{25}ClF_3N_3O_2$	ND	977127
Miscellaneous compounds					
19.981	292.2392	1,2-Benzenediol, O-(2-furoyl)-O'-(pentafluoropropionyl)-	$C_{14}H_7F_5O_5$	ND	160588
20.243	225.0656	1,8,11-Heptadecatriene, (Z,Z)-	$C_{17}H_{3n}$	ND	273401
20.422	219.1343	1-Acetoxynonadecane	$C_{21}H_{42}O_2$	ND	75610
17.249	131.1923	1H-1,3-Benzimidazole-1-ethanol, a-(4-morpholinylmethyl)-	$C_{14}H_{19}N_3O_2$	ND	30635
24.268	131.0521	1H-Indole, 4-methyl-	C_9H_9N	ND	50238
13.318, 11.381	69.1009, 69.0252	2-Propynenitrile, 3-fluoro-	C_3FN	56110	10723
20.441	322.2496	3,4-Dimethoxybenzoic anhydride	$C_{18}H_{18}O_7$	ND	68270
11.937	503.1066	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-3,5,5-tris(trimethylsiloxy)tetrasiloxane	$C_{18}H_{52}O_7Si_7$	ND	284860

(*continued on next page*)

Table 1 (*continued*)

Retention Time (Min)	Observed Ion *m/z*	Name	Molecular Formula	Average Area	
				W1	W2
13.434, 13.433	157.0884, 157.0882	3-Methyl-4-phenyl-1H-pyrrole	$C_{11}H_{11}N$	62006	50364
20.423	265.1814	Heneicosyl acetate	$C_{23}H_{46}O_2$	98265	ND
6.115	144.0419	Acetic acid, trifluoro-, ethyl ester	$C_4H_5F_3O_2$	ND	43042
23.999	269.0457	Anthranilic acid, 2TMS derivative	$C_{13}H_{23}NO_2Si_2$	ND	9488
6.517	357.0670	Cyclopentasiloxane, decamethyl-	$C_{1n}H_{3n}O_5Si_5$	ND	126744
19.172	219.1294	Dimethylmalonic acid, di(2-formylphenyl) ester	$C_{19}H_{16}O_6$	ND	3508
22.124	504.1076	Heptasiloxane, hexadecamethyl-	$C_{16}H_{48}O_6Si_7$	ND	176174
21.455	170.1539	Octanamide, N,N-dimethyl-	$C_{1n}H_{21}NO$	ND	319657
14.843	210.0891	Methyl 3-(4-hydroxy-3-methoxyphenyl)propanoate	$C_{11}H_{14}O_4$	29002	ND
5.246	141.0699	Methyl 3-O-benzyl-alpha-d-glucofuranoside 5,6-carbonate	$C_{15}H_{18}O_7$	156833	ND
19.564, 19.562	503.1055, 505.1041	Octasiloxane, 1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15-hexadecamethyl-	$C_{16}H_{5n}O_7Si_8$	118565	166258
13.576, 18.274	219.1379, 219.0369	Phosphine, tris(trifluoromethyl)-	C_3F_9P	4708	6508
6.721, 6.719	139.0991, 139.0991	Quinoline, decahydro-	$C_9H_{17}N$	200840	170716
13.535, 18.732	131.1894, 131.0855	Tris(trifluoromethyl) bromomethane	C_4BrF_9	6445	5364
O-glycosyl					
14.108, 14.093	217.1589, 137.0398	Ethyl a-d-glucopyranoside	$C_8H_{16}O_6$	352422	146265
Phenols/Alkylphenols					
12.252, 12.250	206.1660, 206.1658	2,4-Di-tert-butylphenol	$C_{14}H_{22}O$	537673	454311
22.354, 22.352	340.2395	Phenol, 2,2′-methylenebis[6-(1,1-dimethylethyl)-4-methyl-	$C_{23}H_{32}O_2$	1244321	1333882
Phenylpropanes					
27.948	278.0431	4-tert-Octylphenol, TMS derivative	$C_{17}H_{3n}OSi$	1089432	ND
Phytosterols/Sterols					
29.015, 29.010	412.3703, 412.3677	Stigmasta-5,24(28)-dien-3-ol, (3ß,24Z)-	$C_{29}H_{48}O$	274827	248085
28.506, 28.504	412.3700, 412.3699	Stigmasterol	$C_{29}H_{48}O$	452029	599436
Pyrazine/Pyridines					
4.866	123.0679	4(H)-Pyridine, N-acetyl-	C_7H_9NO	593145	ND
19.154	116.0705	1,4-Di(methyl-d3)benzene-d4	C_8D_{1n}	ND	72152
Sesquiterpenoids					
12.023	204.1864	(1R,5R)-2-Methyl-5-((R)-6-methylhept-5-en-2-yl)bicyclo[3.1.0]hex-2-ene	$C_{15}H_{24}$	ND	52683
10.838	199.9870	Bicyclo[7.2.0]undec-4-ene, 4,11,11-trimethyl-8-methylene-,[1R-(1R*,4Z,9S*)]-	$C_{15}H_{24}$	ND	28580

(*continued on next page*)

Table 1 (*continued*)

Retention Time (Min)	Observed Ion *m/z*	Name	Molecular Formula	Average Area	
				W1	W2
Silane-related compounds/Cyclics					
21.020	432.0861	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsiloxy)tetrasiloxane	$C_{13}H_{4n}O_5Si_6$	ND	184425
18.940, 14.547	504.1078, 415.0368	Cyclooctasiloxane, hexadecamethyl-	$C_{16}H_{48}O_8Si_8$	132164	214475
28.287	221.0456	Cyclotrisiloxane, hexamethyl-	$C_6H_{18}O_3Si_3$	3083849	ND
8.916, 8.915	432.0872, 432.0848	Cyclohexasiloxane, dodecamethyl-	$C_{12}H_{36}O_6Si_6$	315246	401864
Trialkylheterosilanes					
11.938	503.1089	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-3,5,5-tris(trimethylsiloxy)tetrasiloxane	$C_{18}H_{52}O_7Si_7$	198812	ND
Vitamins					
26.254, 26.253	402.3488, 402.3486	d-Tocopherol	$C_{27}H_{46}O_2$	1045910	686038

ND: Not detected; *m/z*: mass-to-charge ratio.
W1: Walnut sample extracted with acetonitrile/methanol/water (40:20:20 v/v/v); W2: Walnut sample extracted with methanol/water (80:20 v/v).

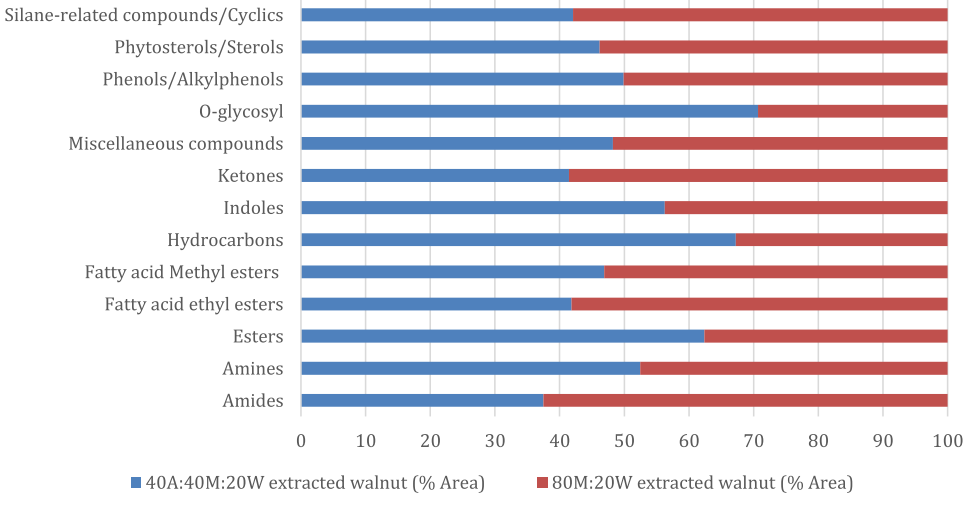

Fig. 1. Percentage distribution of compounds common to both extracted walnut samples.

grated walnuts were dried in a food dehydrator (Bosch BS-6605, Germany) set at a temperature of 60 ± 2 °C for 6 h. The dried walnut was allowed to stand at room temperature before milling (Perten 3600, Sweden) to a coarse powder.

2.2. Metabolites extraction from the samples and analysis using GC-HRTOF-MS

The cooked walnut powder sample was extracted using two different mixtures of extraction solvents, acetonitrile/methanol/water (40:40:20 v/v/v) and methanol/water (80:20 v/v), following

the method previously described by Oyedeji et al. [3]. One (1) gram of each of the walnut samples was weighed separately into 50 mL centrifuge tubes, 10 mL of each extraction solvent was added, and vortexed (Vortex-Gernie K-550-GE, Bohemia USA) vigorously to ensure even mixing. The tube containing the mixture was sonicated (Ultrasonic AU-200 Argo Lab, Italia Italy) for an hour, and then centrifuged (Eppendorf 5702R, Merck, Modderfontein South Africa) for 5 min at 4°C and 3500 rpm. The supernatants from each centrifuge tube were decanted into new tubes, and allowed to dry in a vacuum concentrator (Eppendorf Plus, Merck, Modderfontein South Africa). These recovered dried extracts were reconstituted with 1 mL chromatography-grade methanol (99.9% pure), and vortexed to ensure there is even dissolution of the extracts in each tube. The extracts was filtered into dark amber vials using PTFE-L 0.22 µm. Using the Pegasus GC-HRTOF-MS system (LECO Corporation, St. Joseph, MI, USA) with a resolution of 50,0 0 0 FWMH (full peak with at one-half maximum), mass accuracies/errors of < 1 ppm and acquisition rates of up to 200 spectra/s, the samples were analysed. This analytical system was equipped with a multipurpose sampler (Gerstel Inc., Mülheim an der Ruhr Germany) and Rxi ®-5 ms column (30 m $\times$ 0.25 mm ID $\times$ 0.25 µm) (Restek, Bellefonte, USA). An aliquot of each sample was injected without spit and pumped with helium as the carrier gas at a constant flow rate of 1 mL/min. Inlet and transfer line temperatures were set at 250 and 225 $^{\circ}C$ respectively and the ion source temperature was at 250 $^{\circ}C$. The oven temperature cycle used was: 70 $^{\circ}C$, 0.5 min for initial temperature; then an increase form 10 $^{\circ}C$/min to 150 $^{\circ}C$ for 2 min; then ramped up to 330 °C at 10 °C/min and held for 3 min to allow the column to 'bake-out'. The solvent blanks were also tested in parallel to monitor for potential impurities and contamination. When processing the raw data with DataPrep solutions, parameters such as a signal-to-noise ratio of 50, a similarity match of over 70 % and at least twofold occurrence of metabolites from the triplicate data were strictly considered. The properties of the metabolites were identified by matching the spectra to NIST, Mainlib, and Feihn reference library databases. Data obtained from samples extracted with acetonitrile/methanol/water (40:40:20 v/v/v) and methanol/water (80:20 v/v) are presented in Table 1, and commonly detected compounds are summarised in Fig. 1. The raw and processed data, are presented in the supplementary file along with the raw spectra of some identified compounds.

Ethics Statements

This work does not involve chemicals, procedures or equipment that have any unusual hazards inherent in their use, and it does not involve human subjects, animal experiments, or any data collected from social media platforms.

CRediT Author Statement

Beatrice Mofoluwaso Oladimeji: Conceptualization, Sample preparation, Formal data analysis, Methodology, Visualization, Validation, Writing- original draft; **Oluwafemi Ayodeji Adebo:** Conceptualization, Funding acquisition, Data curation, Methodology, Resources, Software, Visualization, Supervision, Writing –review & editing.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Supplementary data for manuscript on metabolites extracted from African walnut (Tetraca rpidium conophorum) using two different solvents (Original data) (Mendeley Data)

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