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Identification of Bioactive Compounds of *Detarium microcarpum* (Guill and Perr) Stem-Bark Using Molecular Networking

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ABSTRACT
The stem-bark of *Detarium microcarpum* (Guill. and Perr.) has long featured in traditional medicine across sub-Saharan Africa for managing oxidative stress-related ailments, yet its metabolome has never been characterised in detail. Here we report a comprehensive ultra-high-performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS) metabolite profile of the ethanolic stem-bark extract, generated on a Q-Exactive Focus Orbitrap LC-MS/MS system operating in positive and negative electrospray ionisation (ESI) modes. Sixteen compounds were identified using the Global Natural Products Social Molecular Networking (GNPS) platform together with literature comparison, spanning chemical classes that include sesquiterpenoids, amino acids, hydroxybenzoic acid derivatives, terpene lactones, fatty acids, and peptides. Notable among them are gallic acid methyl ester, gossonorol, sinularianin C, anaferine, lathyrine and leupeptin several with documented antioxidant, anti-inflammatory, and hepatoprotective activity. Together, these findings offer a chemical rationale for the ethnopharmacological use of *D. microcarpum* stem-bark and point to its promise as a source of bioactive leads for drug discovery.

Keywords: *Detarium microcarpum*; Metabolite Profiling, UHPLC-MS/MS, Molecular Networking, GNPS, Phytochemicals, Bioactive Compounds

INTRODUCTION

Detarium microcarpum (Guill. and Perr.) belongs to the Fabaceae family and occurs as a perennial tree throughout the savannah and Sahel belts of West Africa. It is referred to locally as "tauhra" (Hausa) and as sweet detar in English, reaches heights of roughly 15 metres, and holds both ecological importance and a long-standing place in traditional medicinal practice (Mohammed *et al.*, 2016; Aviara, 2015). Different parts of the plant bark, leaves, fruit and root are employed by local communities against diarrhoea, meningitis, tuberculosis and various conditions linked to oxidative stress, cancer-related ailments among them (Meda *et al.*, 2017; Yaro *et al.*, 2017).

Metabolomics, the profiling of small-molecule metabolites in a biological matrix, gives a broad picture of a sample's chemical make-up. When paired with mass spectrometry, it goes further, allowing structures to be assigned even to compounds present only at trace levels (Verpoorte *et al.*, 2007). One technique in particular, ultra-high-performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS), has become a leading approach for dereplicating and identifying secondary metabolites in plants, an outcome aided considerably by its integration with platforms such as Global Natural Products Social Molecular Networking (GNPS) (Pantami *et al.*, 2021; Bustaman *et al.*, 2020).

Although pharmacological interest in *D. microcarpum* is growing, a detailed picture of the stem-bark's chemical composition has yet to emerge. Conventional screening in earlier studies pointed to phenolics, flavonoids, and tannins (Hamza *et al.*, 2014; Mahmoud, 2022), yet none applied the high-resolution mass-spectrometric approaches needed to capture the metabolome more fully. This study sets out to close that gap, using UHPLC-MS/MS alongside GNPS-based molecular networking to systematically map the metabolite constituents of *D. microcarpum* stem-bark ethanolic extract and to consider the pharmacological significance of what was found.

MATERIALS AND METHODS

Plant Collection and Extraction

Fresh stem-bark of *Detarium microcarpum* was collected from *kwaya kusar* Local Government Area, Borno State, Nigeria, and authenticated at the Herbarium Unit, Department of Biology federal University of agriculture Zuru. The bark was air-dried at ambient temperature and pulverised to a fine powder; 200 g of powder was macerated in 1,000 mL of aqueous ethanol for 14 days. The filtrate was concentrated under reduced pressure using a rotary evaporator and stored in a desiccator until analysis. Percentage yield was 12.65% (w/w).

Sample Preparation for UHPLC-MS/MS

Two milligrams of the dried ethanolic extract were dissolved in 1 mL of liquid chromatography-mass spectrometry (LC-MS)-grade methanol, vortexed for 10 minutes, centrifuged at 10,000 revolutions per minute (rpm) for 10 minutes, and filtered through a 0.22 µm nylon membrane filter into a glass vial prior to injection.

UHPLC-MS/MS Instrumentation and Conditions

Chromatographic separation was performed on a C18 reversed-phase Hypersil GOLD aQ column (100 × 2.1 mm, 1.9 µm; Thermo, Waltham, MA, USA) maintained at 30°C, using a Dionex Ultimate 3000 UHPLC system fitted with a DAD-3000 diode-array detector (Thermo Fisher Scientific, Waltham, MA, USA). Gradient elution used Solvent A (0.1% formic acid and 10 mM ammonium formate in 70% methanol/30% acetonitrile) and Solvent B (0.1% formic acid and 10 mM ammonium formate in water), ramped from 20% A over 0–5 min to 80% A over 5–30 min at a flow rate of 0.2 mL/min. Injection volume was 10 µL, and ultraviolet (UV) detection was recorded at 210, 310, 410, and 510 nm.

Mass spectrometric detection was performed on a Q-Exactive Focus Orbitrap LC-MS/MS system (Thermo Fisher Scientific) with ESI operated in positive/negative switching mode, scanning m/z 100–1,500. Spray voltage was 4.2 kV; sheath gas flow rate, 40 (arbitrary units); auxiliary gas flow rate, 10 (arbitrary units); and capillary temperature, 350°C. Data were acquired and processed in Thermo Xcalibur 2.2 SP1.48.

GNPS Molecular Networking

Raw MS data files were converted to mzXML format using MSConvert and uploaded to the GNPS platform (http://gnps.ucsd.edu) via FileZilla (Pantami *et al.*, 2021). The MS/MS molecular network was generated using the GNPS Data Analysis Workflow with a precursor-ion mass tolerance of 0.02 Da and a fragment-ion mass tolerance of 0.02 Da; fragment ions below 10 counts were excluded. Network parameters required a minimum of 6 matched peaks and a cosine score ≥ 0.7. The resulting network was visualised and annotated in Cytoscape 3.7.1 (Institute of Systems Biology, Seattle, WA, USA) (Bustaman *et al.*, 2020).

RESULTS AND DISCUSSION

Results

UHPLC-MS/MS Metabolite Identification

Chromatographic separation in positive ESI mode resolved 16 distinct peaks, each characterised by m/z ratio, molecular formula, and fragmentation pattern. Compounds were identified by matching experimental molecular masses ($\Delta <$ 0.015 Da) and MS/MS spectra against the GNPS spectral libraries, the Metabolomics Workbench, and published literature. The identified metabolites are summarised in Table 1.

The resolved peaks spanned a mass range of m/z 176.0983 (peak 3, argininic acid) to m/z 441.3267 (peak 14, leupeptin), consistent with the low-molecular-weight profile typical of plant secondary metabolites. Amino acids and related nitrogenous compounds formed the largest group (peaks 3, 4, 14 and 15), followed by terpenoid-type metabolites (peaks 6, 12 and 13) and phenolic compounds (peaks 2 and 7); the remaining metabolites a fatty-acid ester, a keto acid, a benzamide, an amino alcohol, a ketone, and a halocarbon were each represented by a single peak. This pattern points to a metabolome in which nitrogenous and terpenoid constituents predominate, alongside a smaller phenolic contingent, broadly consistent with earlier phytochemical screening of D. microcarpum that flagged phenolics and terpenoid-type compounds as major constituents, while extending this picture with several nitrogen-containing metabolites not previously reported for the species

Table 1: Metabolites Identified in *Detarium microcarpum* Stem-Bark Ethanolic Extract by UHPLC-MS/MS

Peak No	M/Z	Chemical Formula	Deltal	COMMON Name	Synthetic name	Parent name
1	226.9537	$C_5H_7ID_2$	0.0014	Ethyl 3-iodo-2E-ocrylate	Ethyl 3-iodo-2E-propenoate	Fatty acid
2	219.1744	$C_{15}H_{22}O$	0.0000	Anaephene A	3[CE)-non-5-enyl]phenol	1-hydroxy-4-usubstituted benzenoid
3	176.0983	$C_6H_{13}N_3O_3$	0.0047	Argininic acid	(25)-5-carbamimido-2-hyroxpentanolic acid	Monosaccharides
4	183.0872	$C_7H_{10}N_4O_2$	0.0004	Lathyrine	(25)-2-amino-3(2-aminopyrimidin-4-yl)propanoic acid	Amino acid
5	231.0836	$C_{10}H_{14}O_6$	0.0027	4,7-Dioxosebacic acid	4,7-dioxodecanedioic acid	Keto acid derivatives
6	219.1684	$C_{15}H_{22}O$	0.0059	Gossonorol	6-methyl-2-(4-methylphenyl)-5-en-2-ol	Sesquiterprenoids
7	185.0437	$C_6H_8O_5$	0.0007	Gallic acid methy ester	Gallic acid methy ester	Hydroxybenzoic acid
8	291.0804	$C_{14}H_{14}N_2O_3S$	0.0006	N-Benyl-4-sulfamoyl-benzamide	N-Benyl-4-sulfamoyl-benzamide	Benzamides
9	274.3739	$C_{16}H_{35}NO_2$	0.0001	N-lauryldiethanolamine	2[dodecyl(2-hydroxyethyl)amino]ethanol	1,2aminoalcohols
10	227.1597	$C_{13}H_{22}O_3$	0.0045	6,6-dimethylundecane-2,5,10-trione	6,6dimethylundecane-2,5,10-trione	Ketone
11	225.1958	$C_{13}H_{24}N_2O$	0.0003	(-)-anaferine	1,3-di-(2R)-pipendin-2-ylacetone	Aliphatic tetramonocyclic compo
12	279.1595	$C_{16}H_{22}O_4$	0.0004	Sinularianin C	Nill	Terpene lactones
13	425.2011	$C_{22}H_{23}CIN_2S$	0.013	10-epi-kalihinol x	(1R,2R,4As,5S,8S,8as)-B[(2R,5S)-5-chloro-2,6,6-trimethyloxan-2-yl]-1-	Bifiorane and serrulatane diterpenoids

14	441.3267	$C_{21}H_{40}N_6O_4$	0.0083	Leupeptin	isocyano-5-isothiocyanato-2,5-dimethyl-1,3,4,4a,6,7,8,8a-octahydronaphthalen-2-ol (2s)-N[(2s)-1-[[5(diaminomethylideneamino)-oxopentan-2-yl]amino]-4methyl-1-oxopentan-2-yl]-4-methyl-2(propanoylamino)pentanamide	Peptide
15	206.1199	$C_9H_{19}NO_2S$	0.0010	Tetrahomethionine	2-amino-8-(methylsulfanyl)octanonic acid	Alpha amino acid
16	176.9707	$C_4H_7OCl_3$	0.0072	Chlorobutanol	1,1,1-trichloro-2-methylpropan-2-ol	

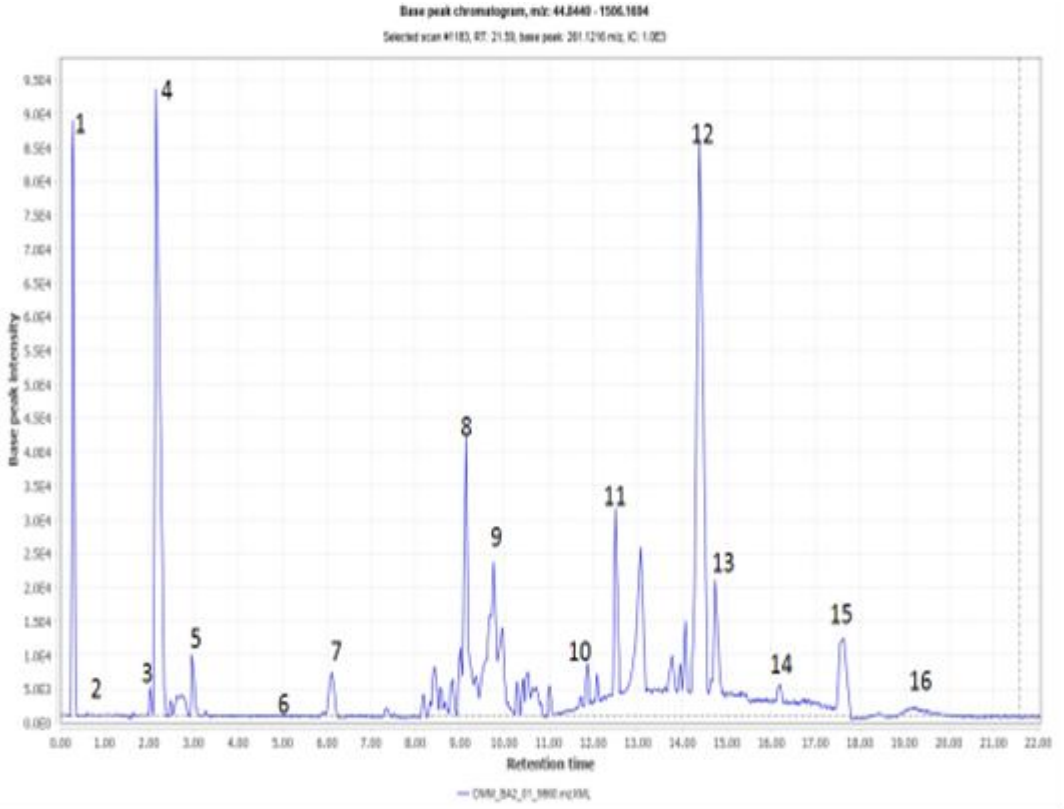
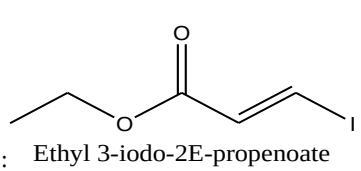
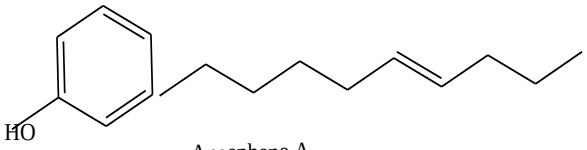


Figure 1: Total ion Charge (TIC) Chromatogram of LCMS-MZmine of *Detariummicrocapum* Stem Bark in Positive Mode. The Number Above Each Peak Represent Peak Number Corresponding to the Peak Number in Table 1

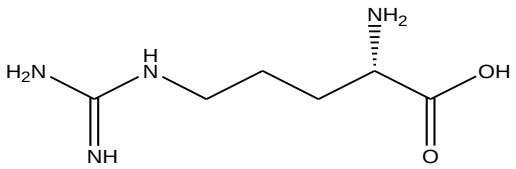
Structure of the Identified Compound



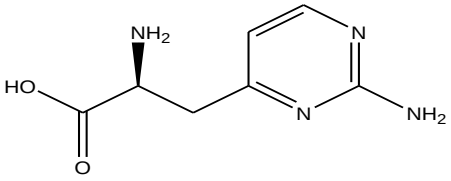
1: Ethyl 3-iodo-2E-propenoate



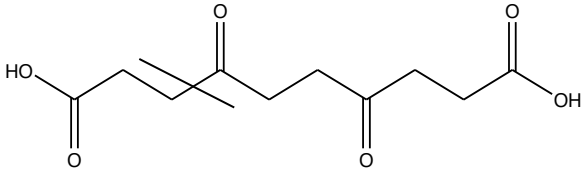
2: Anaephene A



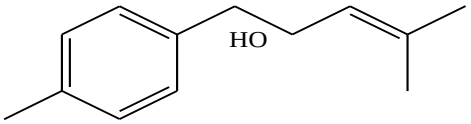
3: Argininic acid



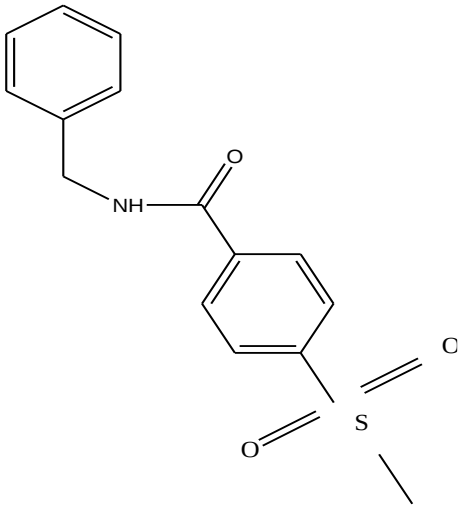
4: (2s)-2-amino-3(2-aminopyrimidin-4-yl)propanoic acid

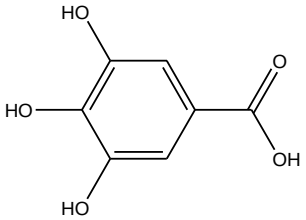


5: 4,7-Dioxosebacic acid



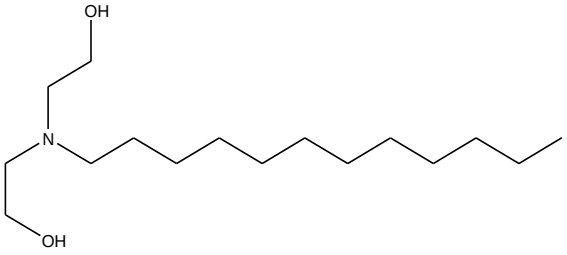
6: Gossonorol



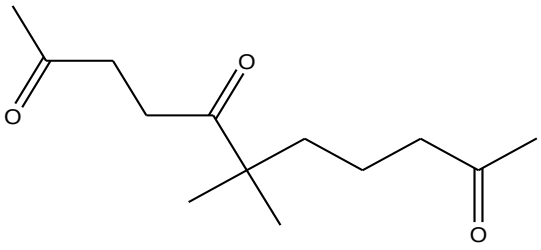


7: Gallic acid

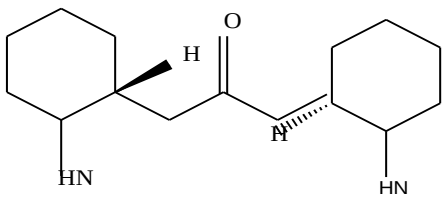
8: N-Benzyl-4-sulfamoyl-Benzamide



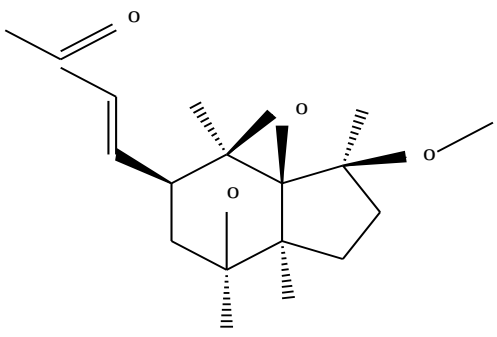
9: N-lauryldiethanolamine



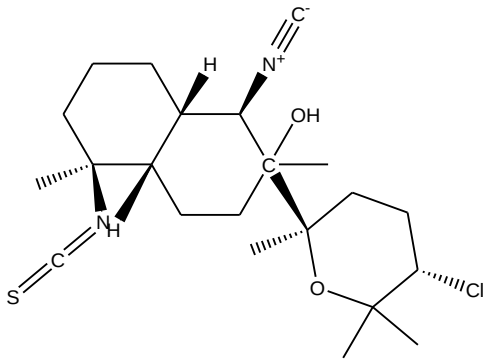
10: 6,6-dimethylundecane-2,5,10-trione



11: Anaferine

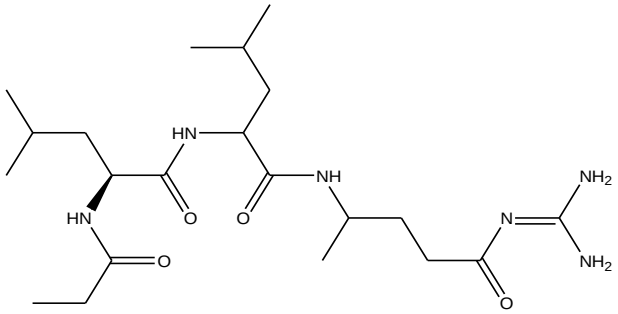


12: sinularian C



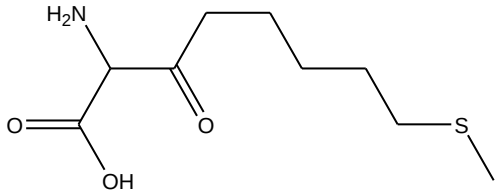
(1R,2R,4As,5S,8S,8as)-B[(2R,5S)-5-chloro-2,6,6-trimethyloxan-2-yl]-1-isocyano-5-isothiocyanato-2,5-dimethyl-1,3,4,4a,6,7,8,8a-octahydronaphthalen-2-ol

13.

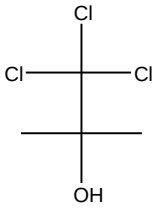


(2s)-N[(2s)-1-[[5(diaminomethylideneamino)-oxopentan-2-yl]amino]-4methyl-1-oxopentan-2-yl]-4-methyl-2(propanoylamino)pentanamide

14.



15: 2-amino-8-(methylsulfanyl)octanonic acid



16: 1,1,1-trichloro-2-methylpropan-2-ol

Discussion

Analysis by UHPLC-MS/MS of the *D. microcarpum* stem-bark ethanolic extract turned up 16 identifiable metabolites across eight chemical classes: sesquiterpenoids, amino acids and derivatives, hydroxybenzoic acid esters, terpene lactones, fatty acid derivatives, keto acids, peptides, and halogenated aliphatics. Such variety is in line with prior accounts of the phytochemical richness found across the genus *Detarium* (Mahmoud, 2022; Meda *et al.*, 2017), while adding finer resolution to what is known of this species' metabolome.

Among the terpenoid-related metabolites, gossonorol (peak 6; m/z 219.1684, C15H22O), a sesquiterpenoid alcohol, has previously been isolated from *Salvia* species and produced via enantioselective synthesis (González-López *et al.*, 2012); as far as we are aware, this marks its first report in *D. microcarpum*. Sinularianin C (peak 12; m/z 279.1595, C16H22O4), a terpene lactone, was first characterised in the soft coral *Sinularia* spp., displaying anti-inflammatory effects via inhibition of nuclear factor kappa B (NF-κB) (Bin Yan *et al.*, 2013); finding it in a terrestrial plant is noteworthy enough to call for confirmation of its structure by nuclear magnetic resonance (NMR) spectroscopy. 10-epi-Kalihinol X (peak 13; m/z 425.2011, C22H23ClN2S), normally a marine chlorinated diterpenoid, may instead represent co-elution with an isobaric compound, or a structurally related diterpenoid native to the plant, a question that will need further work to resolve.

Among the phenolic derivatives, gallic acid methyl ester (methyl gallate; peak 7; m/z 185.0437, C6H8O5), a well-studied phenolic ester, carries documented antioxidant, anti-inflammatory, and antimicrobial properties (Tzi *et al.*, 2018; Habibou *et al.*, 2022) and could plausibly account for the radical-scavenging capacity reported previously for *D. microcarpum* extracts (Meda *et al.*, 2017; Mahmoud, 2022).

Two further compounds, anaephene A (peak 2; m/z 219.1744, C15H22O), a hydroxy-substituted benzenoid, and anaferine (peak 11; m/z 225.1958, C13H24N2O), a piperidine alkaloid, have each been associated with antioxidant effects in earlier work (Alhusain, 2022).

The amino acid and peptide fraction of the extract comprises argininic acid (peak 3), lathyrine (peak 4), N-lauryldiethanolamine (peak 9), leupeptin (peak 14), and tetrahomethionine (peak 15). Lathyrine is a naturally occurring pyrimidine amino acid documented across several leguminous species, and it may well contribute to the extract's biological effects. Leupeptin, a protease-inhibitor peptide found in both microbes and plants, hints at a possible role for *D. microcarpum* in mitigating protease-driven tissue damage. N-Benzyl-4-sulfamoylbenzamide (peak 8), a benzamide derivative, has not yet had its pharmacological relevance explored in a plant context.

The remaining metabolites were more heterogeneous. 4,7-dioxosebacic acid (peak 5; C10H14O6) belongs to the keto acid derivatives, and 6,6-dimethylundecane-2,5,10-trione (peak 10; C13H22O3) has been reported before in the sponge *Hyrtios erectus* (Wirongrong *et al.,* 2018). Chlorobutanol (peak 16; C4H7OCl3) is a halogenated preservative and more probably reflects laboratory contamination than a true plant metabolite, so it should be read with caution. Whether ethyl 3-iodo-2E-acrylate (peak 1) genuinely occurs naturally in *D. microcarpum* is likewise a question that independent verification will need to settle.

Taken together, these findings offer a molecular rationale for several of the ethnomedicinal uses documented for *D. microcarpum* stem-bark. The co-occurrence of phenolic antioxidants (gallic acid methyl ester, anaephene A) with anti-inflammatory and protease-modulating constituents (sinularianin C, leupeptin) is consistent with the anti-inflammatory and analgesic activity reported for the stem-bark extract (Yaro et al., 2017) and with earlier evidence of apoptosis-inducing, cytoprotective activity in cancer cell models (Mohammed et al., 2016). At the same time, several identifications sinularianin C, 10-epi-Kalihinol X, and the trace compound ethyl 3-iodo-2E-acrylate remain tentative, having been assigned by spectral matching alone; their marine or otherwise atypical provenance means they should be treated as putative until confirmed by isolation and NMR. Chlorobutanol, a common laboratory and cosmetic preservative, most likely reflects incidental contamination rather than a genuine plant constituent. Future work combining bioassay-guided fractionation with NMR-based structure elucidation will be needed to confirm these tentative assignments and to establish direct causal links between individual metabolites and the antioxidant, anti-inflammatory, and hepatoprotective activities reported for this species (Meda et al., 2017; Mahmoud, 2022).

CONCLUSION

This study presents the first detailed UHPLC-MS/MS metabolite profile of *Detarium microcarpum* stem-bark ethanolic extract, using GNPS-based molecular networking to identify 16 compounds across diverse chemical classes. Key bioactive metabolites including gallic acid methyl ester, gossonorol, sinularianin C, and anaferine offer a plausible molecular rationale for the plant's reported antioxidant and hepatoprotective activity. These findings advance the chemical characterisation of *D. microcarpum* and lay the groundwork for future isolation, NMR-based structure elucidation, and pharmacological investigation of individual compounds. Untargeted metabolomics combined with complementary NMR spectroscopy is recommended to resolve tentative identifications and capture a fuller picture of this species' phytochemical diversity.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

REFERENCES

Alhusain, A. J. (2022). Promising antioxidant and antimicrobial potencies of chemical profiled extracts from Withania aristata (Aiton) Pauguy against clinically pathogenic microorganisms. Molecules, 27(11), 3644.

Aviara, N. A. (2015). Moisture-dependent physical properties of Detarium microcarpum seeds. Journal of Bioscience and Bioengineering, 40(3), 212–223.

Bin Yan, S., Liao, X., Lin, J., Guo, J., Zhou, X., Yang, X., and Liu, Y. (2013). New sinularianin sesquiterpenes from soft coral Sinularia spp. Marine Drugs, 11, 4741–4750.

Bustaman, M. S. A., Hamza, A. P., Awanis, A., Khozirah, S., Chong, C. M., Fardah, A., Norio, N., Maulidiani, M., Sanjoy, B., Fadzirul, S., and Intan, S. I. (2020). Complementary analytical platform of NMR spectroscopy and LC-MS analysis in the metabolite profiling of Isochrysis galbana. Marine Drugs, 18(5), 261.

González-López, S., Yus, M., and Ramón, D. J. (2012). Enantioselective synthesis of (+)-gossonorol and related systems using organozinc reagents. Tetrahedron: Asymmetry, 23(9–10), 611–615.

Habibou, H. H., Mahamane, I., Alio, A. S., Nodjitoloum, M., Moctar, C., Hama, G. R., Bakasso, S., and Ilagouma, A. T. (2022). Ethnobotanical pharmacology and phytochemistry of widely used medicinal plants in Niger: A review. Journal of Medicinal Plant Studies, 10(4), 46–60.

Hamza, H. G., Saleh, A., Mohammed, Z. K., and Ngadda, H. (2014). Effect of aqueous extract of Detarium microcarpum (Guill. and Sperr.) on mycotoxin-induced tissue damage in albino rats. Journal of Pharmacognosy and Biosciences, 4, 92–99.

Mahmoud, D. A. (2022). Biological activity and chemical composition of Detarium microcarpum Guill. and Perr.: A systematic review. Advances in Pharmacological Sciences, 2022, 1–24.

Meda, N., Fraisse, D., Gnoula, C., Vivier, M., and Felgines, C. (2017). Characterisation of antioxidants from Detarium microcarpum Guill. leaves using HPLC-DAD coupled with pre-column DPPH assay. European Food Research and Technology, 243(9), 1659–1666.

Mohammed, Z. K., Templeton, D., Hamza, H. G., Gidado, A., and Hussaini, I. M. (2016). Detarium microcarpum stem-bark extracts induce apoptosis in human breast adenocarcinoma MDA-MB-231 cells via cJNK activation and mitochondrial cytochrome C release. Journal of Pharmaceutical and Biomedical Sciences, 6(7), 439–444.

Pantami, A. H., Khozirah, S., Muhammad, S. A. B., and Intan, S. I. (2021). Metabolite profiling of different solvent extracts of the microalgae Chlorella vulgaris via 1H NMR-based metabolomics. Current Metabolomics and Systems Biology, 8, 61–74.

Tzi, B., Haskell-Ramsay, C., Cherng, W. L., Liu, F., Tsang, R., Tse, T. F., and Chan, H. (2018). Methyl gallate as antioxidant and anti-inflammatory agent: A review. Phytomedicine, 45, 162–167.

Verpoorte, R., Choi, Y. H., and Kim, H. K. (2007). NMR-based metabolomics at work in phytochemistry. Phytochemistry Reviews, 6(1), 3–14.

Wirongrong, K., Chulaborn, M. S. W., Pittaya, T. W., Somsak, R., and Hunsa, P. (2018). Sesterterpenes and phenolic alkenes from the Thai sponge Hyrtios erectus. Tetrahedron, 74, 316–323.

Yaro, A. H., Yusif, B. B., Mu'azu, A. B., Matinja, A. I., and Chutiyami, M. (2017). Anti-inflammatory and analgesic effect of Detarium microcarpum (Guill. and Perrs.) stem-bark methanol extract in rats and mice. International Research Journal of Pharmacy and Medical Sciences, 1(1), 7–10.