

Phytochemical Profiling of Dichloromethane Grain Extract of *Digitaria exilis* by Liquid Chromatography-Mass Spectrometry (LC-MS) and Its Pharmacological Relevance

Moses Dele Adams

Clinical Biochemistry, Phytopharmacology and Biochemical Toxicology Research Laboratory (CBPBT-RL), Department of Biochemistry, Baze University, Abuja 900108, Nigeria.

moses.adams@bazeuniversity.edu.ng

ABSTRACT: Many medicinal plants are used traditionally, but their active principles are unknown. Depending on the extraction solvent, medicinal plants contain myriads of phytochemicals and bioactive compounds, which are of pharmacognostic importance. Studying the pharmacological relevance of plant extracts is essential to discover new, effective, and safe therapeutic agents (drugs) for treating diseases, as they contain useful phytoconstituents and bioactive compounds. This study therefore focused on the phytochemical profiling of dichloromethane grain extract of *Digitaria exilis* (DGEDE) by liquid chromatography coupled with mass spectrometry (LC-MS) and the pharmacological relevance of the identified compound. The extraction, phytochemistry and LC-MS of DGEDE was carried out using standard protocol. Phytochemical screening of DGEDE revealed the presence of alkaloids (2.57 ± 0.03), tannins (1.89 ± 0.02), phlobatannin (1.47 ± 0.01), flavonoids (2.92 ± 0.03), saponins (2.25 ± 0.01), phenols (2.17 ± 0.02), cardiac glycosides (0.39 ± 0.01), terpenoids (0.78 ± 0.01), steroids (0.63 ± 0.01) and anthraquinone (1.21 ± 0.01). LC-MS analysis of DGEDE, via positive mode, revealed thirty-three compounds, with various base peak (m/z) and retention time (minute), including buthylsopropylamine (114.233, 0.131); 2-butyl amino ethanol (116.123, 1.661); ethylmethylsilane (74.054, 10.954); N-(Octadecanoyl)-sphing-4-enine (565.844, 12.748); N-hexadecyl-5-methyl-2-pyrrolidone (323.384, 17.676) and ethanolamine (61.132, 18.764) among others. The compounds present in DGEDE were identified based on their mass-to-charge (m/z) ratio and fragmentation pattern. The identified compound with specific peaks (compounds) is responsible for certain therapeutic effects. The pharmacological importance of the dichloromethane (DCM) extract of *D. exilis* grain include purgative property, antidiabetic action, antioxidant potential, hepatoprotective benefit and lipid/obesity management ability. The study provides information to bridge the traditional knowledge about medicinal plants with modern drug discovery and evidence-based medicine.

KEYWORDS: Dichloromethane, *Digitaria exilis*, Liquid chromatography-mass spectrometry, Pharmacological relevance, Phytochemical.

I. INTRODUCTION

The use of herbal extract from medicinal plants to treat metabolic or other clinical conditions is grossly limited as they are complex and contain hundreds of compounds without knowledge of the actual therapeutic compound or if two or more compounds work together to elicit their pharmacological effect (Balkrishna et al., 2024; Latif and Nawaz, 2025). Phytochemistry and pharmacognosy are interdisciplinary sciences that study medicinal natural product derived from plants. Phytochemistry is a sub-field of chemistry and botany that focuses on the chemical compounds produced by plants, specifically secondary metabolites, their biosynthesis, and their therapeutic applications. phytochemistry specializes in the extraction, isolation, and analysis of secondary metabolites and allied bioactive compounds (e.g., alkaloids, flavonoids) for drug discovery, safety, and quality control (Kaushik et al., 2021). Phytochemistry focusses on the study of phytochemicals-chemical compounds by plants including secondary metabolites like alkaloids, phenolics, flavonoids, saponins, tannins, phlobatannins, anthraquinones, cardiac glycosides, terpenoids, and cardenolides. It connects chemistry and botany to analyze plant structure, biosynthesis and biological functions, mostly playing significant role in drug discovery and advancing traditional medicine. Phytochemicals are bioactive, non-nutritive natural compounds produced by plants to offer self-defense potential against metabolic diseases, pest and environmental stressors. Pharmacognosy is the study of crude medications that are obtained from medicinal plants, and other allied sources (Popovic and Rijkers, 2025). Pharmacognosy is the specialized, applied science that studies medicinal drugs derived from natural sources, including plants, microbes, and animals. It encompasses the physical, chemical, biochemical, and biological properties of these substances, covering their history, cultivation, extraction, and identification to discover new therapeutic agents. It merges traditional medicine with modern drug discovery. It also focusses on identification, cultivation, and evaluation of crude drugs (Shrivastav et al., 2025).

Plant-derived drugs offer protection, management and treatment against metabolic or other clinical conditions including inflammation, insulin resistance, atherosclerosis, redox imbalance, constipation, ulcer, neurodegenerative disorders among others. A plant implicated in this regard is *Digitaria exilis*. *Digitaria exilis* is classified to the family *Poaceae* and it is called *Hungry Rice* in English. *D. exilis* grain is popularly called by Nigerian names like *Acha* (Hausa), *Suru* (Yoruba) and *Achara* (Igbo). It is an annual and erect herbaceous plant reaching a height of 20 -70 cm (Ballogou et al., 2013). The ears of the plant have three-five narrow part ears that are up to 13cm long. The spikelets are composed of fertile and sterile flowers which eventually give rise to a fonio plant. The grain is a caryopsis that is flanked by husks and glumes. The grain has a small size of 1.5mm. The grain naturally exists in colours like white, black, yellow and purple (Adams et al., 2023). It is mostly grown in Nigerian States like Borno, Jigawa, Taraba, Kaduna, Plateau, Yobe, Nasarawa, Niger, Adamawa, Bauchi, Kano and Kebbi, among others, where the grains are used to prepare a native delicacy locally called 'Gwate' (porridge in Hausa), others are cake, soup, flour meal and local beverage drink ('Kunu' in Hausa) (Adams and Yakubu, 2020).

DCM (methylene chloride) is a volatile, colourless, non-flammable liquid with a sweet, chloroform-like odour liquid mostly used as organic extraction solvent. It is widely used in pharmaceuticals and as an industrial organic solvent for liquid-liquid extraction, chromatography, degreasing, and purification (Ayafor et al., 2024). Dichloromethane is produced by the chlorination of methane or methyl chloride at high temperatures, followed by distillation to isolate it from other chlorinated products. The use of DCM as the organic extracting solvent in this study is due to its ability to pull numerous constituents of the plant sample during the extraction process (Lin and Zhang, 2023). LCMS is a great analytical device that combines the physical separation potential of liquid chromatography (LC) with the high-

sensitive mass analyzing power of mass spectrometry (MS) (Pitt, 2009). It separates the complex mixtures of a sample and identifies the components on the basis of their molecular mass-to-charge (m/z) ratio. LC separates compounds present in a test sample based on their affinity for the stationary/mobile phase, while MS provides definitive, structural, and quantitative analysis (Prasanthi et al., 2024). In LC, the common ionization interface methods to convert the liquid-phase sample to gas-phase ions include Electrospray Ionization (ESI) and Atmospheric Pressure Chemical Ionization (APCI). The Tandem MS (MS/MS) is employed to further break down specific ions to enhance the sensitivity and molecular identification of the sample. LC-MS possess high sensitivity and specificity as it detects trace components in complex mixtures. It has broad applicability as it analyzes non-volatile, polar and thermally labile compounds in a sample that GC-MS cannot. It also has a high speed that allows fast scanning for high-throughput for multiplexed analyses (Alanazi, 2025).

This study intends to advance the existing knowledge on *Digitaria exilis* grains by leveraging on the enormous benefits of using a vital organic extracting solvent (DCM) as well as the unique advantages of LC-MS over GC-MS and other analytical techniques to detect useful phytochemicals that could serve as drug leads in the treatment of diseases.

II. RELATED WORKS

A several studies relative to *Digitaria exilis* (DE), DCM and LC-MS have been reported in the open scientific literature to give insight into the pharmacological benefits of phytochemicals derived from natural products. Ezim and Agwu (2024) used DCM to examine the dermal wound healing components of a plant extract. A study by Adams (2025a) examined the hepatoprotective property of DE in acetaminophen-induced rats. Arika et al (2019) examined the effect of DCM leaf extract of *Gnidia glauca* in high fat diet-induced obese rats. Hamma and Mohammed (2025) provided base line information on which DE and *Digitaria iburua* can be of great importance in food, health and nutritional security of the citizens. Adams (2025c) evaluated the contraceptive potential of DE in experimental models. Ounissi et al (2024) used LC-MS/MS technique to study the antioxidant property of a plant extract. Da et al (2017) and Taya et al (2025) used DCM to study the anti-inflammatory and cancer chemopreventive property of plant extract respectively. Adams et al (2023) studied the purgative activity of DE in rifaximin-induced constipated rat. Omoregie and Uboh (2024) assessed the protective effect of DCM fraction against induced hepato-renal dysfunction in Swiss rats. Studies by Adams and Yakubu (2020) as well as Adams (2025b) demonstrated the action of DE against diabetes mellitus. Aadesariya et al (2017) used DCM and LC-MS as techniques for extraction, isolation and identification of useful phyto compounds from natural products. Despite these studies, reports on the LC-MS of phytochemicals derived from DGEDE, with a bid to explore its possible pharmacological advantage(s) of these phytoconstituents, is missing in the documented reports, creating a research vacuum that needs to be filled. Therefore, this study examined the pharmacological relevance of phytochemicals obtained from dichloromethane grain extract of *Digitaria exilis* using liquid chromatography-mass spectrometry (LC-MS).

III. METHODOLOGY

A. Plant Collection and its Authentication

Digitaria exilis grains was collected from herb sellers at Gwagwalada, Abuja was identified and confirmed at the Plant Biology Unit, University of Ilorin, where a Plant Identification Number (I.U.V. No. 007) was assigned at the Herbarium.

B. Preparation of *Digitaria exilis* Dichloromethane Grain Extract

The grains of *D. exilis* was spread open to dry at room temperature before grinding with an electric blender (Kenwood, Model KT-750, England, UK). A known amount of the powdered grain (1000 g) was extracted in 2.5 L of dichloromethane for 72 h at 25 °C. The mixture was filtered with Whatman No. 1 filter paper (Maidstone, UK) and the resulting filtrate was concentrated in a Rotary Evaporator (Model: BH-45A, Shanghai Ya Rong Biochemistry Instrument Factory, China). The filtrate was then dried in a steam bath (Model: NL-450S, NEWLIFE® Medical Instrument, England) to yield 7.36 g extract (DEGE). The resulting dichloromethane grain extract was used for phytochemical screening and LC-MS analysis.

C. Qualitative and Quantitative Phytochemical Screening of *Digitaria exilis* Dichloromethane Grain Extract

The qualitative and quantitative phytochemical screening of *Digitaria exilis* dichloromethane grain extract (DEGE) was carried out by adopting the procedure previously described by Trease and Evans (1983).

Procedure for Liquid Chromatography-Mass Spectrometry (LC-MS)

Principle: Liquid chromatography depends on easy the samples distribute themselves between the mobile phase and stationary phase on the basis of properties like size, polarity and charge. Components of the sample with higher affinity for the mobile phase exit and elute the column faster than those with lower affinity for the mobile phase. The distribution and separation can be on the basis of partition, adsorption, ion exchange or size-exclusion mechanism. Procedure: The method described by Porvaznik (2023) was used to carry out liquid chromatography. The stationary phase (silica gel) of the Agilent Liquid Chromatography Equipment (Agilent 55 MB), Cambridge, MA, USA coupled to Agilent 6520 Accurate-Mass Q-TOF mass spectrometer with dual ESI source) with the following column (Agilent Zorbax Eclipse XDB-C18, Narrow-Bore 2.1x150mm, 3.5micron (P/N: 930990-902)) conditions, was packed into a column, and the system was equilibrated with the mobile phase (dichloromethane: methanol). An auto-injector was used to introduce 0.05 µl of the sample into the mobile phase stream. The pump maintains a constant flow of the mobile phase, carrying the sample through the column, where differential migration occurs. As components exit the column, they pass through a detector, basically a mass spectrometer, that generates a signal, typically producing a chromatogram. The resulting chromatogram showed peaks corresponding to the base concentration (base peak) and retention time (RT) of each separated compound.

D. Statistical Analysis of Data Obtained

The protocols detailed in one-way ANOVA and Duncan multiple Range Test were used to statistically analyze the results obtained from the study (n=3). Statistical Product and Service Solution, SPSS version 23 was used to determine significant difference at $p < 0.05$.

IV. RESULTS AND DISCUSSION

Qualitative phytochemical screening of DGEGE revealed the presence of flavonoids, phlobatannins, saponins, alkaloids, cardiac glycosides, anthraquinones, steroids, phenols, terpenoids and tannins. Cardenolides was absent in the plant extract (Table 1).

Table 1: Qualitative Phytochemicals in *Digitaria exilis* dichloromethane grain extract

Phytochemical	Observation
Alkaloids	+
Tannins	+
Phlobatannins	+
Flavonoids	+
Saponins	+
Phenols	+
Steroids	+
Terpenoids	+
Anthraquinones	+
Cardenolides	-
Cardiac glycosides	+

Key: + = present; - = absent

The quantitative phytochemical screening of DGEGE revealed the quantity of ten phytochemicals, in mg/ml, namely: alkaloids (2.57±0.03), tannins (1.89±0.02), phlobatannin (1.47±0.01), flavonoids (2.92±0.03), saponins (2.25±0.01), phenols (2.17±0.02); cardiac glycosides (0.39±0.01), terpenoids (0.78±0.01), steroids (0.63±0.01) and anthraquinone (1.21±0.01) (Table 2).

Table 2: Quantitative phytochemicals in *Digitaria exilis* dichloromethane grain extract

Phytochemical	Quantity (mg/ml)
Alkaloids	2.57±0.03
Tannins	1.89±0.02
Phlobatannins	1.47±0.01
Flavonoids	2.92±0.03
Saponins	2.25±0.01
Phenols	2.17±0.02
Steroids	0.63±0.01
Terpenoids	0.78±0.01
Anthraquinones	1.21±0.01
Cardenolides	-
Cardiac glycosides	0.39±0.01

Data are average of three determinations ± SEM

n = 3; SEM = Standard Error of Mean

LC-MS analysis of *Digitaria exilis* dichloromethane grain extract detected thirty-three compound (Table 3). The LC-MS analysis was accompanied by a chromatogram that showed the peaks, peak area and retention time among others (Figure 1).

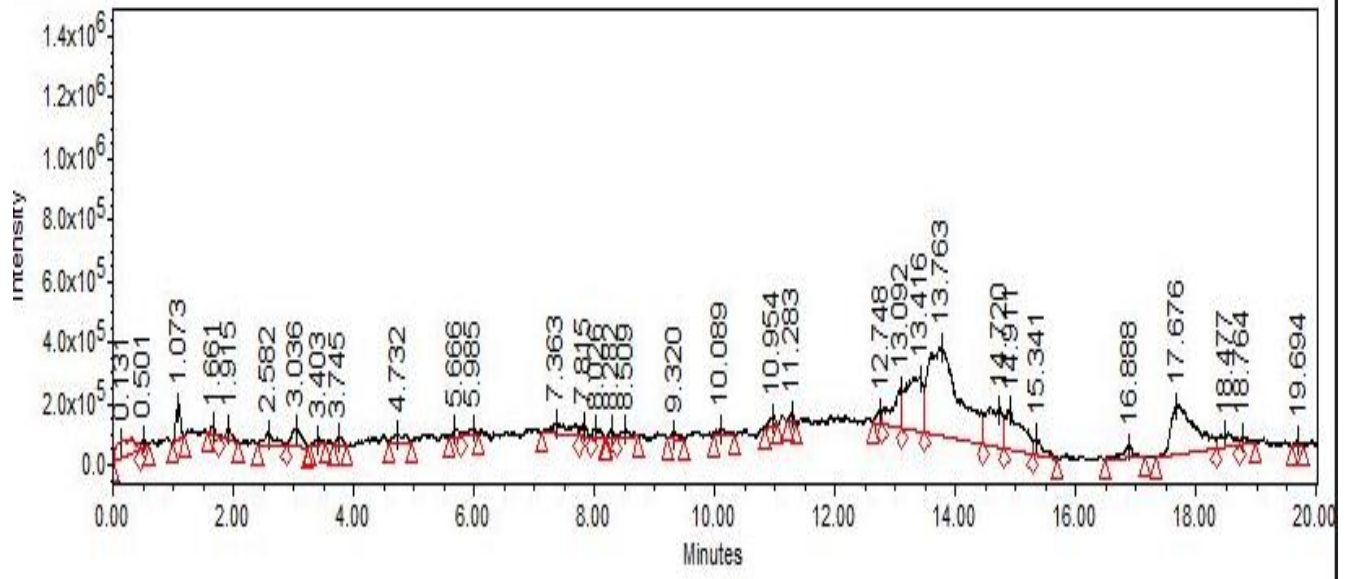


Figure 1: LC-MS chromatogram of *Digitaria exilis* dichloromethane grain extract

Table 3: Compounds identified in *Digitaria exilis* dichloromethane grain extract through liquid chromatography-mass spectrometry (LC-MS)

Peak	IUPAC Name of Compound	Base Peak (m/z)	RT (Minute)	Molecular Mass (g/mol)	Molecular Formular
1.	Buthylisopropylamine	114.233	0.131	87.16	C ₅ H ₁₃ N
2.	4-pentenenitrile	81.212	0.501	81.12	C ₅ H ₇ N
3.	Amino methylphosphoric acid	109.990	1.073	111.04	CH ₆ NO ₃ P
4.	2-Butyl amino ethanol	116.123	1.661	117.19	C ₆ H ₁₅ NO
5.	Heliotrine (Triclocarban)	312.662	1.915	315.58	C ₁₃ H ₉ Cl ₃ N ₂ O
6.	4-bromo-1-butene	132.883	2.582	135.00	C ₄ H ₇ Br
7.	Semicarbaxide	74.047	3.036	75.07	CH ₅ N ₃ O
8.	2-Aminoheptane	114.190	3.403	115.22	C ₇ H ₁₇ N
9.	1-hydroxyethane	205.036	3.745	46.07	C ₂ H ₆ O
10.	12-HHTrE (Imipramine)	280.201	4.732	280.41	C ₁₉ H ₂₄ N ₂
11.	Desoxypeganine	171.263	5.666	172.23	C ₁₁ H ₁₂ N ₂
12.	Stepharine	297.366	5.985	297.35	C ₁₈ H ₁₉ NO ₃
13.	Ergovaline	533.650	7.363	533.62	C ₂₉ H ₃₅ N ₅ O ₅
14.	Man4GlcNAc2-11 (G09724ZC)	1072.056	7.815	1073.00	C ₄₀ H ₆₈ N ₂ O ₃₁
15.	Permethylated N-acetyl galactosaminyl- (beta 1,3)-galactosyl- (alpha 1,4)- galactosyl-(beta 1,4)-glucitol	919.422	8.026	919.50	C ₄₁ H ₇₇ NO ₂₁
16.	Indole-3-acetyl-L-valine	274.319	8.282	274.32	C ₁₅ H ₁₈ N ₂ O ₃
17.	3-beta-acetoxy-5-alpha-lanost-8-en-7-alpha-yl nitrite ester	515.539	8.509	515.77	C ₃₂ H ₅₃ NO ₄
18.	Lanosoprazole_BTP_M3	267.055	9.320	385.36	C ₁₆ H ₁₄ F ₃ N ₃ O ₃ S
19.	Malforminic C	529.231	10.089	529.7	C ₂₃ H ₃₉ N ₅ O ₅ S ₂
20.	Ethylmethylsilane	74.054	10.954	74.20	C ₃ H ₁₀ Si
21.	Songorine	357.513	11.283	357.49	C ₂₂ H ₃₁ NO ₃
22.	N-(Octadecanoyl)-sphing-4-enine	565.844	12.748	565.95	C ₃₆ H ₇₁ NO ₃
23.	2-methyl-4-(methylthio)-2-morpholinepropiofenone	279.136	13.092	279.40	C ₁₅ H ₂₁ NO ₂ S
24.	Cer[NP]t41:0	653.774	13.416	654.10	C ₄₁ H ₈₃ NO ₄
25.	Ceramide (d18:1/18:0)	565.554	13.763	565.95	C ₃₆ H ₇₁ NO ₃
26.	Cer[NS]d44:1	677.639	14.720	677.67	C ₄₄ H ₈₇ NO ₃
27.	Monensin	672.640	14.911	670.87	C ₃₆ H ₆₂ O ₁₁
28.	Resorcinol bis (diphenylphosphate)	573.008	15.341	574.45	C ₃₀ H ₂₄ O ₈ P ₂
29.	4-bromobenzoylmethyl (12Z,15Z)-9,10-epoxyoctadecadienoate	489.768	16.888	477.47	C ₂₆ H ₃₇ BrO ₃
30.	N-hexadecyl-5-methyl-2-pyrrolidone	323.384	17.676	323.56	C ₂₁ H ₄₁ NO
31.	N,N-dimethyloctanamide	171.413	18.477	115.17	C ₆ H ₁₃ NO
32.	Ethanolamine	61.132	18.764	61.08	C ₂ H ₇ NO
33.	1,3-propanediamine,1,3-diaminopropane	74.318	19.694	74.13	C ₃ H ₁₀ N ₂

RT = Retention Time; IUPAC = International Union of Pure and Applied Chemistry

The use of LC-MS for phytochemical profiling of medicinal plants can provide valuable insight into bioactive compounds and other phytocompounds of pharmacological importance. The pharmacological relevance of these bio-compounds and other phytochemicals have the capacity to prevent, treat or manage metabolic or other clinical condition. Phytochemicals are naturally occurring chemicals present in or extracted from plants. Some phytochemicals are nutrients for the plant, while others are metabolites produced to enhance plant survivability and reproduction. Phytochemicals are chemicals produced by plants through primary or secondary metabolism. They generally have biological activity in the plant host and play a role in plant growth or defense against competitors, pathogens, or predators. Phytochemicals are generally regarded as research compounds rather than essential nutrients because of their possible health effects (Kaushik et al., 2021; Kumar et al., 2023). Polyphenols like flavonoids improve immune function and modulate immune cells. It reduces inflammation and acts as an antioxidant. Flavonoids protect against infections and microorganism-causing diseases: this is achieved by maintaining a balanced immune system. Flavonoids also function as antimicrobial agents where they reduce the ability of viruses and bacteria to grow and proliferate (Olaniyan et al., 2023; Trivadila et al., 2025).

Phenols and other phenolic compounds act in chemoprevention to hinder cancer development in various parts of the biological system (Ashong, 2024). Cancer chemoprevention by phenols occurs through prevention of DNA damage. Phenols mediate in repair of mutated cancerous genes, slowing cancer growth, development and proliferation. Phenols participate in triggering abnormal cancer cells to die more quickly (Rudzinska et al., 2023). Terpenoids protect the brain, enhance neural plasticity, boost cognitive function, and improve sleep quality by modulating the gut-brain axis, reducing oxidative stress, and reducing neuroinflammation. Terpenoids also support brain plasticity by boosting the ability of the brain to adapt and modify in response to new information, sensory stimulation, damage and other impactful experiences (Tkaczenko et al., 2025). It plays a role in cognition which includes memory, attention and learning ability. Terpenoids prevent/delay the progression of neurodegenerative disorders like Alzheimer and Parkinson's disease. Sleep mode is also regulated by terpenoids via reduction of insomnia (Adhikary et al., 2025).

Tannins and phlobatannins support heart health through the prevention of cardiovascular diseases by decrease in inflammation, improved cholesterol absorption, prevention of oxidative stress and reduction of blood pressure (Arshad et al., 2025). They also offer potent antioxidant and anti-inflammatory benefits via improvement in endothelial function, reduction in LDL oxidation, management of blood pressure, and reduction in atherosclerosis risk (Alum, 2025). Other phytochemicals such as alkaloids, anthraquinones and saponins comprise a broad range of organic compounds found in plants. They play an important role in the medicinal properties of plants, contributing to antimicrobial, anti-inflammatory, antioxidant and anticancer effects. These compounds serve as the foundation for natural remedies and are integral to modern pharmacology (Trant, 2024).

Liquid Chromatography-Mass Spectrometry (LC-MS) is an indispensable tool for identifying and quantifying phytochemicals in plant extracts. LC-MS enables the separation and characterization of diverse, non-volatile, or thermally unstable bioactive compounds such as phenolic acids, flavonoids, and terpenoids, which are subsequently linked to pharmacological activities like antioxidant, anticancer, antidiabetic, and antimicrobial roles (Abilkassymova et al., 2024). Liquid chromatography (LC) separates mixture components based on their differential affinity between a stationary phase (solid column packing) and a liquid mobile phase. As the sample moves through the column, components with higher affinity for the stationary phase move slower, while those with higher affinity for the mobile phase move faster, resulting in separation. Buthylsopropylamine, a compound identified in DGEDE via

LC-MS, is a branched secondary amine that acts as a, crucial building block in the synthesis of various pharmaceutical compounds, agrochemicals, and specialty chemicals. Its primary pharmacological relevance stems from its use as an intermediate in producing active pharmaceutical ingredients (APIs), while its derivative, N-nitroso-ethylisopropylamine (NEIPA), is significant as a potent mutagenic impurity requiring strict regulatory monitoring (Xia et al., 2025). Aminomethylphosphonic acid (AMPA), identified via LC-MS in this study, is primarily known as the main, highly persistent environmental degradation product of the herbicide glyphosate, but it possesses distinct pharmacological, toxicological, and biochemical activities. While historically considered to have low toxicity similar to glyphosate, recent research indicates that AMPA has specific, often more potent, effects on cellular, enzymatic, and molecular pathways in both plants and animals (Parajuli et al., 2016; Chavez-Reyes et al., 2024).

2-Aminoheptane, also known as tuaminoheptane (or 2-heptylamine), is a pharmacological agent primarily recognized as a sympathomimetic stimulant and a potent vasoconstrictor. Sympathomimetic stimulants are drugs or chemical agents that mimic the effects of the body's natural sympathetic nervous system "fight or flight" response, increasing alertness, heart rate, blood pressure, and metabolic activity. They function by acting directly on adrenergic receptors or indirectly increasing neurotransmitters like norepinephrine, dopamine, and epinephrine (Costa et al., 2022).

Lansoprazole, identified via LC-MS in this study, is a proton pump inhibitor that reduces gastric acid secretion. It has proved effective in combination regimens for the eradication of *Helicobacter pylori* and as monotherapy to heal and relieve symptoms of gastric or duodenal ulcers and gastro-oesophageal reflux. After initial healing, it may be used to prevent recurrence of oesophageal erosions or peptic ulcers in patients in whom *H. pylori* is not the major cause of ulceration and to reduce basal acid output in patients with Zollinger-Ellison syndrome. Lansoprazole is a proton pump inhibitor that reduces gastric acid secretion in a dose-dependent manner via inhibition of H^+/K^+ -adenosine triphosphatase in gastric parietal cells. It also exhibits antibacterial activity against *Helicobacter pylori* *in vitro* (Bown, 2002).

N-(Octadecanoyl)-sphing-4-enine, more commonly known as C18-ceramide (Cer d18:1/18:0), is a key sphingolipid component with significant pharmacological and physiological relevance, particularly as a mediator of cell stress and signalling. As a ceramide, it functions as a potent bioactive molecule that induces apoptosis, inhibits proliferation, and regulates cell differentiation (Li et al., 2026). In humans, mediators of cell stress and signalling occur through highly conserved molecular pathways that detect intracellular or environmental perturbations and trigger adaptive, protective, or pro-apoptotic responses. When cells face stress such as nutrient deprivation, protein misfolding, or reactive oxygen species (ROS) they activate specific surveillance systems (sensors) that initiate intracellular signalling cascades (mediators) to restore homeostasis (Fulda et al., 2010). Songorine, a diterpenoid alkaloid which was identified via LC-MS in the present study possesses a diverse range of pharmacological activities demonstrated in pre-clinical models. It is particularly noted for its neuroactive properties, including anxiolytic and central nervous system (CNS) stimulatory effects, as well as peripheral actions such as anti-arrhythmic, anti-inflammatory, and wound-healing properties (Khan et al., 2018). In man, neuroactive properties occur through the synthesis, release, and action of specialized chemical agents including neurotransmitters, neuromodulators, neurohormones, and neuroactive steroids that influence neuronal network function. These properties are produced both by neurons and glial cells in the central nervous system (CNS) and by peripheral endocrine glands (Melcangi, 2024).

4-bromobenzyl methyl (12Z,15Z)-9,10-epoxy octadecadienoate, detected in via LC-MS in this study, represents a synthetic, halogenated derivative of an epoxy-fatty acid methyl ester,

combining structural features that suggest potential application in drug discovery and therapeutic research, particularly in anti-inflammatory and anti-proliferative areas. It is a compound with similar structure with epoxy-fatty acid methyl esters and derivatives of linolenic acid. It is known to act as anti-inflammatory agents by suppressing inflammation, such as 12-O-tetradecanoyl-phorbol-13-acetate (TPA)-induced inflammation. It also exhibits anticancer and cytotoxicity property. This occur in presence of an epoxide group in combination with a modified methyl ester tail often contributes to cytotoxic activity against tumor cell lines, often acting through topoisomerase inhibition, inducing apoptosis, or causing cell cycle arrest at the G2/M phase (Dong et al., 2022). N,N-dimethyloctanamide (also known as N,N-dimethylcaprylamide or DMOA), identified via LC-MS in this study, is a fatty acid amide with pharmacological relevance primarily as a transdermal penetration enhancer and as a specialized cosolvent in the formulation and synthesis of pharmaceutical compounds. It acts as an amphiphilic agent that helps increase the solubility of drugs, thereby improving their transport across biological barriers like the skin. Transdermal penetration enhancement in humans occurs primarily by overcoming the barrier function of the stratum corneum (SC), the outermost, nearly impermeable layer of the skin. This process, essential for delivering therapeutic drugs into the systemic circulation, is achieved through chemical, physical, and pharmaceutical techniques that temporarily and reversibly increase skin permeability (Ramadon et al., 2022).

Ethanolamine (MEA), identified via LC-MS in this study, is a vital organic compound with significant pharmacological relevance, primarily acting as a fundamental building block for biological membranes, a neurotransmission modulator, and an active agent in medical procedures. It is essential for life, found in all human cells as a component of phospholipids, particularly phosphatidylethanolamine (PE). In humans, biological membranes act as sophisticated modulators of neurotransmission by controlling the physical environment, location, and functional state of signaling proteins (receptors, transporters, and channels) embedded within them. Rather than being passive barriers, these lipid bilayers specifically in the synaptic cleft—regulate neuronal communication through direct lipid-protein interactions and indirect modulation of membrane physical properties (Barrantes, 2024).

1,3-Propanediamine (also known as 1,3-diaminopropane or trimethylenediamine, CAS 109-76-2) is a versatile aliphatic diamine with significant pharmacological relevance, primarily acting as a building block in the synthesis of diverse pharmaceutical agents. Its structural properties; two primary amine groups separated by a three-carbon chain allow it to serve as an intermediate in the production of drugs, particularly those targeting neurological disorders, and as a component in biological research. It acts as a building block in the synthesis of pharmaceutical agents by serving as a fundamental, often modular, structural unit that is chemically combined with other building blocks to create complex, bioactive molecules. These foundational components ranging from simple amino acids to complex heterocyclic compounds contain specific functional groups that allow them to be linked together through reactions like condensation, substitution, or coupling, enabling the construction of diverse therapeutic agents with precise, predefined structures (Kluczyk et al., 2002). Desoxypeganine (also known as Deoxypeganine or Deoxyvasicine) is a natural alkaloid, identified by LC-MS in this study, possesses significant pharmacological relevance, primarily due to its actions on the central nervous system (CNS) as a reversible acetylcholinesterase (AChE) and selective monoamine oxidase A (MAO-A) inhibitor. It is known for treating cognitive, addiction, and psychiatric disorders, showing a favourable safety profile compared to other inhibitors like galantamine (Tuliaganov et al., 1986). Dual inhibitors targeting both Acetylcholinesterase (AChE) and selective monoamine oxidase A (MAO-A) act as multi-target-directed ligands

(MTDLs) designed to treat complex neurodegenerative and psychiatric conditions by simultaneously boosting neurotransmitter levels, reducing neuroinflammation, and combating oxidative stress. They are primarily investigated for Alzheimer's disease (AD) and related neurocognitive disorders, offering both symptomatic relief and potential neuroprotective, disease-modifying benefits (Asim et al., 2025).

V. CONCLUSION

The study demonstrated the suitability of LC-MS for the identification of non-volatile, polar and thermally labile phytochemicals inherent in samples that cannot be effectively analyzed by GC-MS and other related techniques. These phytochemicals exhibit potential as lead compounds for drug discovery and development. The findings further suggest that phytochemicals identified in dichloromethane grain extract of *Digitaria exilis* via LC-MS analysis exhibit significant pharmacological potential for the management and/or treatment of various metabolic or clinical conditions. More so, the results suggest that dichloromethane is an effective solvent for the extraction of bioactive compounds.

REFERENCES

- [1] Aadesariya, M.K., Ram, V.R., & Dave, P.N. (2017). Extraction, Isolation and Identification of Useful Phyto Constituents from Dichloromethane Leave Extract of *Abutilon Pannosum* and *Grewia Tenax* Using Q-TOF LC/MS. *International Journal of Advanced Research in Chemical Science*, 4(10), 1-14. DOI: <http://dx.doi.org/10.20431/2349-0403.0410001>.
- [2] Abilkassymova, A., Aldana-Mejía, J.A., Katragunta, K., Kozykeyeva, R., Omarbekova, A., Avula, B., Turgumbayeva, A., Datkhayev, U.M., Khan, I.A., & Ross, S.A. (2024). Phytochemical Screening Using LC-MS to Study Antioxidant and Toxicity Potential of Methanolic Extracts of *Atraphaxis pyrifolia* Bunge. *Molecules*, 29(18), 4478. doi: 10.3390/molecules29184478.
- [3] Adams, M.D., Manu, H.A., & Enyioma-Alozie, S. (2025a). Molecular Docking, Contraceptive Property and Histopathological Changes in Experimental Models by *Digitaria exilis* Grain Extract via Interference with Steroidogenesis at Ovarian Level. *Tropical Journal of Natural Product Research*, 9(7), 3349 – 3359. <https://doi.org/10.26538/tjnpr/v9i7.63>.
- [4] Adams, M.D. (2025b). *In Silico* Evaluation of Phytochemicals from *Digitaria exilis* Aqueous Grain Extract as Potential Inhibitors of α -Amylase in Management of Type-II Diabetes Mellitus. *Dutse Journal of Pure and Applied Sciences*, 11(4b), 148-165. <https://dx.doi.org/10.4314/dujopas.v11i4b.14>.
- [5] Adams, M.D. (2025c). *Digitaria exilis* Methanol Grain Extract Halts Acetaminophen Hepatotoxicity Onslaught Via Upregulation of Antioxidant Enzyme and Improved Hepatic Function in Wistar Rats. *Arid Zone Journal of Basic and Applied Research*, 4(6), 113-129. <doi.org/10.55639/607.02010086>.
- [6] Adams, M.D., Muftaudeen T.K., & Saliu, O.A. (2023). Polyphenol-rich Extract of *Digitaria exilis* (Kippist) Grain Lowers Gastrointestinal Dysmotility and Enhanced Colonic Peristalsis in Rifaximin-induced Constipated Rat. *Nigerian Journal of Biochemistry and Molecular Biology*, 38(3), 131-138, DOI: <https://dx.doi.org/10.4314/njbmb.v38i3>.
- [7] Adams, M.D., & Yakubu, M.T. (2020). Aqueous extract of *Digitaria exilis* grains ameliorate diabetes in streptozotocin-induced diabetic male Wistar rats. *Journal of Ethnopharmacology*, 249 (2020), 112383. <https://doi.org/10.1016/j.jep.2019.112383>.

- [8] Adhikary, K., Ganguly, K., Sarkar, R., Abubakar, M., Banerjee, P., & Karak, P. (2025) Phytonutrients and their neuroprotective role in brain disorders. *Frontier in Molecular Bioscience*, 12, 1607330. doi: 10.3389/fmolb.2025.1607330.
- [9] Alanazi, S. (2025). Recent Advances in Liquid Chromatography-Mass Spectrometry (LC-MS) Applications in Biological and Applied Sciences. *Anal Sci Adv*, 6(1), e70024. doi: 10.1002/ansa.70024.
- [10] Alum, E.U. (2025). Role of phytochemicals in cardiovascular disease management: Insights into mechanisms, efficacy, and clinical application. *Phytomedicine Plus*, 5(1), 100695. DOI: 10.1016/j.phyplu.2024.100695.
- [11] Arika, W.M., Kibiti, C.M., Njagi, J.M., & Ngugi, M.P. (2019). Anti-obesity effects of dichloromethane leaf extract of *Gnidia glauca* in high fat diet-induced obese rats. *Heliyon*, 5(11), e02800. doi: 10.1016/j.heliyon.2019.e02800.
- [12] Arshad, M.T., Ali, M.K.M., Maqsood, S., Ikram, A., Hossain, M.S., Aljameel, A.I., Al-Farga, A., & Gnedeka, K.T. (2025). Dietary Phytochemicals in Cardiovascular Disease Prevention and Management: A Comprehensive Review. *Food Science and Nutrition*, 13(9), e70872. doi: 10.1002/fsn3.70872.
- [13] Ashong, A. (2024). Examining the role of phytochemicals in cancer prevention and treatment. *Clinical Nutrition and Hospital Dietetics*, 44(3),01-02. DOI:10.12873/0211-6057.44.03.230.
- [14] Asim, A., Jastrzebski, M.K., & Kaczor, A.A. (2025). Dual Inhibitors of Acetylcholinesterase and Monoamine Oxidase-B for the Treatment of Alzheimer's Disease. *Molecules*, 30(14), 2975. <https://doi.org/10.3390/molecules30142975>.
- [15] Ayafor, C., Burton, T., George, N., Morose, G., & Wong, H.W. (2024). Safer Solvents for Active Pharmaceutical Ingredient Purification Using Column Chromatography. *ACS Environ Au*. 2024 Jun 24;4(5):236-247. doi: 10.1021/acsenvironau.4c00015.
- [16] Balkrishna, A., Sharma, N., Srivastava, D., Kukreti, A., Srivastava, S., & Arya, V. (2024). Exploring the Safety, Efficacy, and Bioactivity of Herbal Medicines: Bridging Traditional Wisdom and Modern Science in Healthcare. *Future Integrative Medicine*, 3(1), 35-49. doi: 10.14218/FIM.2023.00086.
- [17] Ballogou, V.Y., Soumanou, M.M., Toukourou, F., & Hounhouigan, J.D. (2013). Structure and nutritional composition of Fonio (*Digitaria exilis*) grains: a review. *International Research Journal of Biological Sciences*, 2(1), 73–79.
- [18] Bown, R.L. (2002). An overview of the pharmacology, efficacy, safety and cost-effectiveness of lansoprazole. *International Journal of Clinical Practice*, 56(2), 132-139.
- [19] Barrantes, F.J. (2024). Modulation of a rapid neurotransmitter receptor-ion channel by membrane lipids. *Frontiers in Cell Developmental Biology*, 11, 1328875. doi: 10.3389/fcell.2023.1328875.
- [20] Chavez-Reyes, J., Sarachaga-Terrazas, F., Colis-Arenas, O.A., Lopez-Lariz, C.H., Villalón, C.M., & Marichal-Cancino, B.A. (2024). Aminomethylphosphonic Acid (AMPA), a Glyphosate Metabolite, Decreases Plasma Cholinesterase Activity in Rats. *Journal of Xenobiotics*, 14(2), 604-612. <https://doi.org/10.3390/jox14020035>.
- [21] Costa, V.M., Grando, L. G. R., Milandri, E., Nardi, J., Teixeira, P., Mladenka, P., & Remiao, F., (2022). Natural Sympathomimetic Drugs: From Pharmacology to Toxicology. *Biomolecules*, 12(12), 1793. <https://doi.org/10.3390/biom12121793>.
- [22] Da, F.L., Keugni, A.B., Belemtougri, G.R., Fotio, T.L.A., & Dimo, T. (2017). Acute and subacute anti-inflammatory activities of dichloromethane extract of *Cassia alata* (Linn.) leaves in Wistar rats. *African Journal of Traditional, Complementary and Alternative Medicines*, 15(1), 174–182. <https://doi.org/10.21010/ajtcam.vi15.1.18>.

- [23] Dong, H., Wang, L., Guo, M., Stagos, D., Giakountis, A., Trachana, V., Lin, X., Liu, Y., & Liu, M. (2022). Antioxidant and Anticancer Activities of Synthesized Methylated and Acetylated Derivatives of Natural Bromophenols. *Antioxidants*, 11(4), 786. <https://doi.org/10.3390/antiox11040786>.
- [24] Ezim, O.E., & Agwu, J.A. (2024). Amino Acid Profile, Total Protein and Histological Changes in Female Wistar Rats After Administration of Dichloromethane Extract of *Heterotis rotundifolia* Leaves on Dermal Wound. *The International Journal of Research and Scientific Innovation*, 11(2), 215-223. DOI: 10.51244/IJRSI.2024.1102017.
- [25] Fulda, S., Gorman, A.M., Hori, O., & Samali, A. (2010). Cellular stress responses: cell survival and cell death. *International Journal Cell and Biology*, 2010, 214074. doi: 10.1155/2010/214074.
- [26] Hamma, I.I. & Mohammed, A.A. (2025). Study on the physiological characteristics, proximate and quantitative phytochemical analysis of White fonio (*Digitaria exilis*) and Black fonio (*Digitaria iburua*) in Funakaye LGA, Gombe State. *Journal of Advanced Science and Optimization Research*, 7(9), 116-145. DOI: <https://doi.org/10.70382/sjasor.v7i9.021>.
- [27] Kaushik, B., Sharma, J., Yadav, K., Kumar, P., & Shourie, A. (2021). Phytochemical properties and pharmacological role of plants: secondary metabolites. *IOSCI Biosciences and Biotechnology Research Asia*, 18(1), 23-35. DOI: <http://dx.doi.org/10.13005/bbra/2894>.
- [28] Khan, H., Nabavi, S.M., Sureda, A., Mehterov, N., Gulei, D., Berindan-Neagoe, I., Taniguchi, H., & Atanasov, A.G. (2018). Therapeutic potential of songorine, a diterpenoid alkaloid of the genus *Aconitum*. *European Journal of Medicine and Chemistry*, 153, 29-33. doi: 10.1016/j.ejmech.2017.10.065.
- [29] Kluczyk, A., Popek, T., Kiyota, T., de-Macedo, P., Stefanowicz, P., Lazar, C., & Konishi, Y. (2002). Drug evolution: p-aminobenzoic acid as a building block. *Current Medical Chemistry*, 9(21), 1871-92. doi: 10.2174/0929867023368872.
- [30] Kumar, A.P.N., Kumar, M., Jose, A., Tomer, V., Oz, E., Proestos, C., Zeng, M., Elobeid, T., K.S., & Oz, F. (2023). Major Phytochemicals: Recent Advances in Health Benefits and Extraction Method. *Molecules*, 28(2), 887. DOI: 10.3390/molecules28020887.
- [31] Latif, R., & Nawaz, T. (2025). Medicinal plants and human health: a comprehensive review of bioactive compounds, therapeutic effects, and applications. *Phytochemistry Reviews*, 2025; 1-44. <https://doi.org/10.1007/s11101-025-10194-7>.
- [32] Lin, L. & Zhang, Y. (2023). Chemical constituents and antidiabetic activity of Dichloromethane extract from *Ficus carica* leaves. *Diabetes, Metabolic Syndrome and Obesity: Targets and therapy*, 16, 979-991. <https://doi.org/10.2147/DMSO.S405150>.
- [33] Li, T., He, H., Zhang, E., Hu, F., Wang, Z., Xu, J., Zeng, M., & Peng, B. (2026). The physiological and pathological effects of sphingolipid metabolism and signaling in the central nervous system. *Brain Pathology*, 36(1), e70033. DOI: 10.1111/bpa.70033.
- [34] Melcangi, R.C. (2024). Role of Neuroactive Steroids in Health and Disease. *Biomolecules*, 14(8), 941. <https://doi.org/10.3390/biom14080941>.
- [35] Olaniyan, M.F., Muhibi, M.A., & Olaniyan, T.B. (2023). Immune-Boosting Phytochemicals in Nigeria. *Matrix Science Pharma*, 7(1), 1-6. DOI: 10.4103/mtsp.mtsp_5_23.
- [36] Omoregie, F.O., & Uboh, E.F. (2024). Effects of dichloromethane fraction of ethanol extract of *Phyllanthus amarus* leaf on 1, 2-dimethylhydrazine induced hepato-renal

- dysfunction in swiss albino mice. *FUDMA Journal of Sciences*, 8(6), 280-287. <https://doi.org/10.33003/fjs-2024-0806-2939>.
- [37] Ounissi, I., Slimani, S., Bounaas, J., Bensouici, C., Aouzal, B., & Bouacha, A. (2024). Phytochemical profile by LC-MS/MS, total phenolic content and Antioxidant Properties of *Erodium guttatum* from Algeria. *Global NEST Journal*, 26(10), 1-10. <https://doi.org/10.30955/gnj.06464>.
- [38] Parajuli, K.R., Zhang, Q., Liu, S., & You, Z. (2016). Aminomethylphosphonic acid inhibits growth and metastasis of human prostate cancer in an orthotopic xenograft mouse model. *Oncotarget*, 7(9), 10616-26. doi: 10.18632/oncotarget.7055.
- [39] Pitt, J.J. (2009). Principles and applications of liquid chromatography-mass spectrometry in clinical biochemistry. *Clin Biochem Rev*, 30(1):19-34.
- [40] Popovic, L., & Rijkers, G.T. (2025). *Phytochemicals: Principles and Practice*. Biology (Basel), 15(1), 18. doi: 10.3390/biology15010018.
- [41] Porvaznik, M. (2023). Detection and quantitation through Liquid chromatography. *Journal of Chromatographic Separation Techniques*, 14, 504-512.
- [42] Prasanthi, V.A., Latha, P.V.M., Umadevi, P., & Sivalalitha, P. (2024). An analytical overview of liquid chromatography-mass spectroscopy (LC-MS) instrumentation and applications. *International Journal of Creative Research Thoughts*, 12(7), c952-c958.
- [43] Ramadan, D., McCrudden, M.T.C., Courtenay, A.J., & Donnelly, R.F. (2022). Enhancement strategies for transdermal drug delivery systems: current trends and applications. *Drug Delivery and Translational Research*, 12(4), 758-791. doi: 10.1007/s13346-021-00909-6.
- [44] Rudzinska, A., Juchaniuk, P., Oberda, J., Wisniewska, J., Wojdan, W., Szklener, K., & Mańdziuk, S. (2023). Phytochemicals in Cancer Treatment and Cancer Prevention-Review on Epidemiological Data and Clinical Trials. *Nutrients*, 15(8), 1896. DOI: 10.3390/nu15081896.
- [45] Shrivastav, G., Dubey, A., Kumari, M., & Shukla, T. (2025). Advances in pharmacognosy for modern drug discovery and development. *Journal of Biosciences and Public Health*, 1(3), 45-63. DOI: <https://doi.org/10.5455/JBPH.2025.14>.
- [46] Taya, S., Singai, C., Wongppprnchai, R., & Khantamat, O. (2025). Phytochemical Profiling and *In Vitro* Mechanistic Evaluation of Dichloromethane Extract from *Phyllanthus emblica* Fruit for Cancer Chemoprevention. *Chiang Mai Journal of Science*, 52(6), 1-15. DOI:10.12982/CMJS.2025.102.
- [47] Tkaczenko, H., Buyun, L., Kołodziejska, R., Kamiński, P., & Kurhaluk, N. (2025). Neuroactive Phytochemicals as Multi-Target Modulators of Mental Health and Cognitive Function: An Integrative Review. *International Journal of Molecular Science*, 26, 8907. <https://doi.org/10.3390/ijms26188907>.
- [48] Trant, E. (2024). The Significance of Phytochemicals in Drug Development and Disease Prevention. *Der Pharmacia Lettre*, 16(4), 15-16.
- [49] Trease, G.E. and Evans, W.C. (1983). *A Textbook of Pharmacognosy*, Twelfth ed. Baillie
- [50] Tindall Ltd., London, pp. 343–383.
- [51] Trivadila, T., Iswantini, D., Rahminiwati, M., Rafi, M., Salsabila, A.P., Sianipar, R.N.R., Indariani S., & Murni, A. (2025). Herbal Immunostimulants and Their Phytochemicals: Exploring *Morinda citrifolia*, *Echinacea purpurea*, and *Phyllanthus niruri*. *Plants (Basel)*, 14(6), 897. DOI: 10.3390/plants14060897.
- [52] Tuliaganov, N., Sadritdinov, F.S., & Suleïmanova, G.A. (1986). Pharmacological characteristics of desoxyepanine hydrochloride. *Farmakol Toksikol*, 49(3), 37-40.

- [53] Xia, L., Yan, J., Chen, Y., Mitchell, K., McGovern, T.J., Atrakchi, A.H., & Heflich, R.H., Chen, T. (2025) N-nitroso-ethylisopropylamine mutagenicity in rat liver using the cII transgenic mutation assay and duplex sequencing analysis of genomic DNA. *Chemical and Biological Interaction*, 418, 111603. doi: 10.1016/j.cbi.2025.111603.