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RESEARCH ARTICLE

Essential products derived from thermal methanolic processing of *Gmelina arborea* leaves

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ABSTRACT - Industrialization has brought the need for an increase in raw material sources. On the other hand, globalization is seeking for waste utilization and recycling. In the quest to search for industrial raw materials from waste products, research was carried out on the thermal methanolysis (methanolic process) of leaves of Gmelina arborea using barium chloride as a catalyst. The methanolic process was conducted at 60°C reaction temperature and varying reaction times. The products were analyzed using gas chromatography and mass spectroscopy (GC-MS). The GC-MS analysis revealed the presence of benzofuran-2-carboxaldehyde, 2,5-dimethyl 3-(H) furanone (DMHF), and 9-octadecenamide. These compounds are essential metabolites in the food, cosmetic, and pharmaceutical industries. The yields of the metabolites were 195.24 mg/g (4.94%), 79.96 mg/g (1.95%), and 364.76 mg/g (8.89%). There is a decrease in the yield of each of the three products with reaction time. The production of the metabolites would further add value to the Gbmelina arborea plantation.

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1.0 INTRODUCTION

Metabolites (bioactive molecules) are essential raw materials for pharmaceuticals, food, and cosmetic industries. They are contemporary synthesized using other industrial chemicals. Thus, their production is expensive, nonrenewable, and may be menacing. Aligning to the global campaign for sustainability and renewability of raw materials for industrialization, there is a need for a renewable and sustainable approach to the production of these bioactive molecules. Such alternative methods will address the high cost of raw materials, utilize renewable materials and provide green waste management. Three bioactive molecules are considered for production using leaves of Gmelina arborea.

1.1 Benzofuran-2-carboxaldehyde

Benzofuran-2-carboxaldehyde ($C_9H_6O_2$) also known as 2-benzofuran carboxaldehyde, benzo[B]furan-2-carboxaldehyde, 1-benzofuran-2-carbaldehyde is a member of benzofuran [1]. It has 9 carbon, 6 hydrogens, and 2 oxygen atoms in its molecule. It is a crystalline solid with bitter almond odours and a CAS number 4265-16-1. Benzofuran compounds are found useful as antibacterial, antifungal, anti-inflammatory, analgesic, antidepressant, anticonvulsant, antitumor, imaging, Anti-HIV, antidiabetic, antitubercular, antioxidant, and miscellaneous activity [2]. Miao et al. [1] described benzofurans as having anti-tumour, antibacterial, anti-oxidative, and anti-viral activities. Also, Khodarahmi et al. [3] described benzofuran compounds as anti-inflammatory, antimicrobial, antifungal, antihyperglycemic, analgesic, antiparasitic, and antitumor. Benzofuran-2-carboxaldehyde and other benzofuran derivatives are used to treat cancers[4, 5, 6]. Sun et al. [7] reported using Benzofuran-2-carboxaldehyde for cancer treatment and inhibition of MCF7 and HepG2 tumour proliferation. MCF-7 is a human breast cancer cell line with estrogen, progesterone, and glucocorticoid receptors [8] derived from the Michigan Cancer Foundation [9]. HepG2 is a human liver cell cancer line derived in 1975 from the tissue of a 15-year-old Argentine [10]. Human hepatoma is most commonly used in drug metabolism and hepatotoxicity studies [11, 12].

Benzonfuran-2-carboxaldehyde has coal tar as a natural source and can be obtained from the dehydrogenation of ethyl phenol [5]. However, ethyl phenol is in high demand for the production of phenolic resins [13] which is a very useful product. The extraction of benzofuran from coal tar takes a long time to complete (up to 24 hours) and requires a very high temperature (above 100oC) [14]. The production route is no doubt expensive. It can be naturally obtained from Asteraceae [15, 16, 17]. Asteraceae is a family of lettuce, sunflower seeds, artichokes, and sweetening agents [18, 19, 20].

1.2 2,5-dimethyl-3(2H)-furanone

The phytochemical compound, 2,5-dimethyl-3(2H)-furanone ($C_6H_7O_3$) also known as 4-hydroxy-3(2H)-furanones are considered exceptional aroma compounds [21, 22]. It has a strong caramel-like odour and is used for flavoring jams, jellies, beverages, ice creams, alcoholic drinks, and sweets [23, 22, 24]. Xiao et al. [25] describe it as a potent aroma compound highly used in the food industry due to its attractive sensory properties. It is used extensively in jelly sweets

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and gelation products such as ice cream Chan [26]. It has high demand due to its extensive use as a food flavouring agent [27]. It is found in many fruits such as brambles, mangoes, strawberries, and pineapples [28]. It is also found in tomatoes, and buckwheat [29]. 2,5-dimethyl-3(2H)-furanone was first obtained from pineapple and identified in 1965 [30, 25]. It can be produced from strawberries [31, 32].

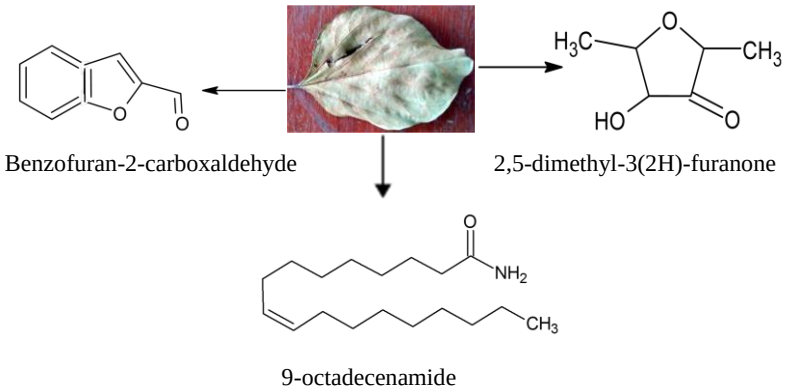
The contemporary method of synthesizing DMHF is via a Maillard reaction between an organic acid (amino acid) and simple sugar (pentose) [33]. The process involves heating pentose sugar with glycine or L-alanine at 90 oC for one hour [34]. The Maillard reaction gives rise to harmful products, including a process contaminant, acrylamide (ACR) [35], heterocyclic amines, and advanced glycation end products (AGEs) [36]. This ACR is considered carcinogenic to humans [37].

1.3 9-Octadecenamide

Oleamide or oleic acid amide are other names of 9-octadecenamide ($C_{18}H_{35}NO$) which belongs to the fatty amides class of organic compounds [38, 39]. It is a waxy solid, odourless, and insoluble but dispersible in water [40]. It is a primary amide discovered in the cerebral spinal fluid of sleep-deprived cats [41]. It was first detected in human plasm (serum) in 1989 [42]. Another useful phytochemical compound is 9-octadecenamide, which is a form stabilizer [43]. A report by Naumoska et al. [44] claimed that the introduction of 9-octadecenamide into the human body is metabolites in vivo into oleic acid. Mueller & Driscoll [42] reported that 9-octadecenamide has effects on thermoregulation and acts as an analgesic. It is well-known for being used as a lubricant and slip additive [45, 46]. It is an antioxidant used as a drug to induce drowsiness or sleep [47]. It has been extracted from the plant, Allium cepa (onion) using ethanol as a solvent [48]. It is also found in other plants such as Allium sativum (garlic), Triticum Vulgare (wheat), and Nigella sativa (black cumin) [49, 50]. The compound, 9-octadecemide is a natural product found in Glycine max (soybean) [51].

The contemporary method of producing 9-octadecenamide is via amidation reaction. The process involves the conversion of carboxylic acid, first, into ammonium salt, which then produces an amide on heating [52]. However, the aminolysis stage produces a mixture of primary, secondary, and tertiary amines and quaternary salt [53]. Separation of this complex mixture is complicated.

The major sources of these three metabolites are vegetables which are sources of food. Their application in the extraction of these metabolites would cause food crises. These vegetables should be left for whole food consumption while another renewable, sustainable, and environmentally benign source that would not compete with food be used for the production of the metabolites. In this study, a novel production method, utilizing a renewable raw material (Gmelina arborea leaves), for the production of three bioactive molecules (benzofuran carboxaldehyde, 2,5-dimethyl-3(2H)-furanone, and 9-Octadecenamide) was investigated. The leaves of Gmelina arborea are sustainable renewable and environmentally harmless. The production of the three metabolites is summarized in the Reaction Scheme 1.



Scheme 1. Flow process diagram of methanolic process of bioactive molecules

2.0 METHODS AND MATERIAL

The materials used in this study include a 1000 mL Pyrex conical flask, a 1000 mL Pyrex beaker, a 1000 mL separating funnel, a glass funnel, a ceramic mortar, and pestle, a GallenKamp hot plate magnetic stirrer, a mercury in glass thermometer, and distilled water. The analytical reagents used were methanol and barium chloride.

The dry leaves of Gmelina arborea were collected at Kaduna Polytechnic, Kaduna, Nigeria. It was cleaned, dried, and pulverized with ceramic mortar and pestle. The pulverized material was sieved with 250 and 300 µm mesh sizes following Ibrahim and Ali [54]. The methanolysis reaction was carried out as per Ibrahim and Ali [55]. A solution of 0.5g of a barium chloride catalyst in 500 mL distilled water was prepared in a 1000 mL conical flask. This 0.5 g barium chloride is equivalent to 1.0% (w/w) of 50 g of the feed (Gmelina arboria leaves). A mass of 50 g of the leaves was added to the catalyst solution and stirred. The solution was placed on a hot plate and heated to 60 ^{o}C for 10 minutes at the stirring rate of 250 rpm. The product was filtered into a 1000 mL beaker, first with cotton wool, and then with filter paper. The filtrate

was left in the beaker to settle for 5 hours and then decanted. The upper liquid content was decanted into a conical flask and weighed. The experiment was repeated for 20, 30, 40, 50, and 60 minutes, in turn. Samples of each product were collected and analyzed with GC-MS to determine the chemical components and their compositions. The procedure is as shown in the process flow diagram depicted in Figure 1.

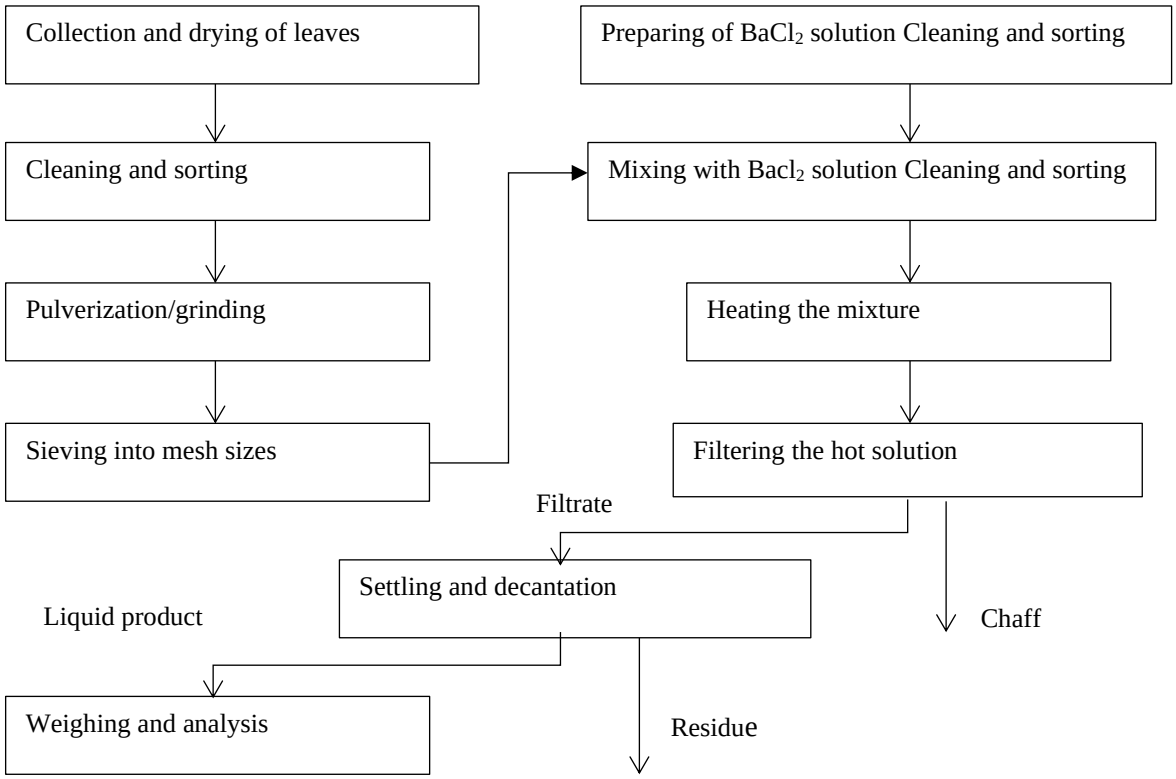


Figure 1. Flow process diagram of methanolic process of bioactive molecules

3.0 RESULTS AND DISCUSSION

The GC-MS analysis revealed the compositions of the three useful metabolites: DMHF, benzofuran-2-carboxaldehyde, and 9-octadecenamide as presented in Table 1 The variation of the yield of the three metabolites with reaction time is depicted in Figure 1. Generally, there is a decrease in the yield of each of the three products with reaction time. This decreased behaviour could be attributable to a chain reaction. As residence time increases, the desired product may undergo further reaction producing an entirely different product. This speculation was equally inferred by Ali and Ibrahim [56] in research on the production of bio-disinfectant from Gmelina arborea leaf. The latter researchers inferred that a longer reaction time is unfavorable to the production of the 2,3-hydroxyl propanal due to chain reaction.

Table 1. Yields of three essential molecules from Gmelina arborea leaf

Reaction time (min)	10	20	30	40	50	60
Benzo-2-carboxaldehyde						
Yield (%)	4.09	4.94	4.57	4.87	4.17	4.16
2,5-Dimethyl-3(2H)-furanone						
Yield (%)	1.95	1.57	1.44	2.77	1.79	1.89
9-Octadecenamide						
Yield (%)	8.89	7.88	7.70	8.58	8.71	9.12

The three essential molecules have their highest yields within the first 20 minutes as depicted in Figure 2. The highest yield obtained by 9-octadecenamide was 364.76 mg/g in the 10-minute reaction time. Benzofuran-2-carboxaldehyde had its highest yield of 195.24 mg/g in the 20-minute reaction time. The highest yield for the third product, DMHF, was 79.96 mg/g in the 10-minute reaction time. This production process favours the production of 9-octadecenamide over the other for all conditions of reaction times as depicted by the results in Figure 2. The yields of DMHF do not show much significant difference with reaction times.

Katritzky et al. [57] reported a 67% yield of benzofuran-2-carboxaldehyde from the lithiation of DMF and $POCl_3$ by n-Butyllithium (n-BuLi). The production of 2,5-dimethyl-3(2H)-furanone (DMHF) carried out by Hecquet et al. [58] had a residence time of 11 days. The latter used D-fructose 1,6-bisphosphate, and sodium salt as the reactants and obtained the highest yield of 110 ppm. This yield is too insignificant for commercial production. Similarly, Koutsompogeras et

al. [31] produced DMHF using strawberries and 1,2-propanediol. The investigator obtained the highest yield of 48% after 4 days of reaction. Strawberry is a highly nutritional fruit and is used in the production of vitamin C due to its ascorbic acid content [59]. It is also used in the production of ice cream, jam, fruit leather, and mixed drinks [60]. Its use in the manufacture of bioactive metabolite may be competing and very expensive.

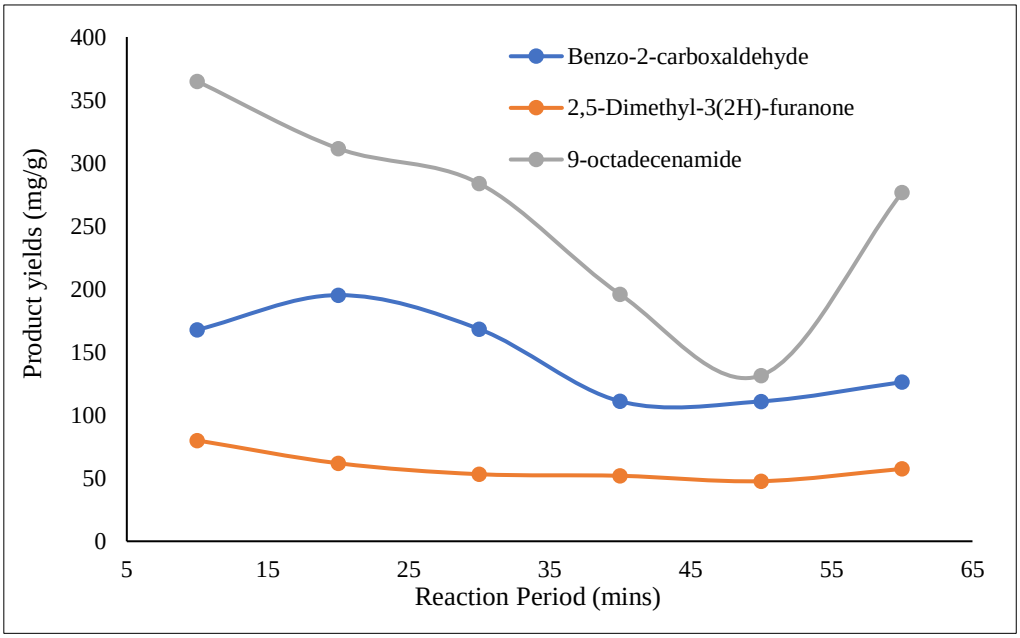


Figure 2. Yields of the metabolites with reaction time

Despite numerous applications of oleamide, Mueller & Driscoll [42] claim that the biochemical mechanism of its synthesis has not yet been defined. Abel-Anyebe et al. [43] carried out two separate aminolysis reactions at 50 minutes reaction time. In the first experiment, they used peanut oil while castor oil was used in their second experiment. They obtained 83% fatty amides of 9-octadecenamide (oleamide) and 79% fatty amides of hexadecanamide (palmitamide) from the first and second reactions, respectively. All the aforementioned processes have a long residence time and therefore do not favour commercialization. In contrast, the proposed method of production using leaves of Gmelina arborea has a short residence time. The process is renewable, and sustainable, and converts waste-to-wealth.

4.0 CONCLUSION

In this investigation, research was carried out on the thermal methanolysis of Gmelina arborea leaves using barium chloride as a catalyst. The GC-MS result of the product revealed the presence of benzofuran-2-carboxaldehyde, 2,5-dimethyl 3-(H) furanone (DMHF), and 9-octadecenamide. The yields of these three metabolites were 195.24 mg/g (4.94%), 79.96 mg/g (1.95%), and 364.76 mg/g (8.89%), respectively. There is a decrease in the yield of each of the three products with reaction time. Thus, three bioactive molecules, 9-decenamide, benzofuran-2-carboxaldehyde, and DMHF can be synthesized from Gmelina arborea leaves via methanolysis.

CONFLICT OF INTERESTS

We declare no conflict of interest.

AUTHORS CONTRIBUTION

H. Ibrahim (Conceptualization; Formal analysis; Visualisation; Supervision)

Abubakar M. Ali (Methodology; Data curation; Writing - original draft; Resources)

M.D. Jibrin (Methodology; Data curation; Writing - original draft; Resources)

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