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alcaloïdes d'une plante saharienne de la famille Apiaceae**

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Abstract

The objective of this study was the valorization of alkaloid extracts of *Deverra scoparia* Coss. & Dur. (Apiaceae), a plant widely used in traditional recipes, because it produces bioactive molecules that have therapeutic potential.

The study of extracts and fractions of volatile, basic, acidic, and free alkaloids was followed by the quantification of total alkaloid content by a spectroscopy method, the identification of volatile alkaloids by GC-MS analysis, and the identification of fixed alkaloids by HPLC analysis. Evaluation of potential pharmacology by different tests.

Quantifications of alkaloid fractions and extracts obtained show that AVD has the highest alkaloid content 62895.83 mg AE/kg. GC-MS analysis of volatile alkaloids showed that the fractions AVD, AVE, AWAED, B2, B3, and B4 contain volatile alkaloids, amino acids, and biogenic amines such as 2-(tetrahydrofuran-2-yl) piperidine, triethanolamine (Sterolamide), 1-decyl-1H-imidazole-2-methanol, and [(2-fluorophenyl)methyl] 1H-Purin-6-amine, and the presence of fixed alkaloids such as atropine, berberine chloride that have been identified and quantified. On the other hand, 15 compounds belonging to the class of phenolic acids and flavonoids were identified, and quantities included, including epicatechin, apigenin-7-glucoside, chlorogenic acid, vanillic acid, p-coumaric acid, quercetin, by HPLC.

The separation and purification of the studied fractions, of which 3 isolated products were identified using GC/MS analysis, identified nonacosan-10-one as a pure compound isolated from the B1-1 fraction, with apigenin-7-glucoside and naringenin in MAAE-FCR and C108-99M, respectively, by HPLC.

The study of the biological activities of tested fractions and extracts, *Deverra scoparia* Coss. & Dur., has been shown to have significant antioxidant potential that has been evaluated by the DPPH, phenolthroline, and β -carotene tests. The antidiabetic activity showed that free alkaloids and volatile alkaloid fractions have higher potential inhibitors of the enzyme α -amylase ($IC_{50}=260.81 \pm 1.87$, 1630.69 ± 1.41 $\mu\text{g/ml}$ respectively) than acarbose ($IC_{50}=3650.93 \pm 10.70$ $\mu\text{g/ml}$) the positive control. On the other hand, the majority of the fractions tested have a very important inhibitory power of the enzyme butyrylcholinesterase with IC_{50} value ranging from 1.81 ± 0.87 to 116.39 ± 0.55 $\mu\text{g/ml}$. The test for determining the ability of fractions to protect the skin from sunlight showed a remarkable protective factor. The basic alkaloid extract fractions revealed consistent insecticide potential. All fractions and extracts tested show that the potency of a fraction correlates with its composition.

These results justify the important use of *Deverra scoparia* Coss. & Dur. in traditional medicine.

Key words: *Deverra scoparia*, alkaloids, GC-MS, HPLC, antioxidant, antidiabetic, anti-Alzheimer, SPF, bio-insecticides.

Résumé

L'objectif du présent travail est la valorisation des extraits alcaloïdiques de *Deverra scoparia* Coss. & Dur. (Apiaceae), une plante d'une vaste utilisation dans les recettes traditionnelles, à cause de produit des molécules bioactives qui ont le potentiel thérapeutique.

L'étude des extraits/fractions d'alcaloïdes volatiles, basiques, acidiques et les alcaloïdes libres a été suivie par la quantification du contenu alcaloïde total par une méthode spectroscopique, l'identification des alcaloïdes volatiles par GC-MS, l'identification des alcaloïdes fixes par LCHP et l'évaluation de la potentielle pharmacologie est réalisée par différents tests.

Les quantifications des fractions/ d'extraits alcaloïdes obtenues montrent que l'AVD présente le taux alcaloïdique le plus élevé 62895.83 mg AE/kg. La GC-MS analyse des alcaloïdes volatiles a montré que les fractions AVD, AVE, AWAED, B2, B3 et B4 contiennent des alcaloïdes volatiles, des acides aminés et des amines biogéniques tels que 2-(tetrahydrofuran-2-yl) piperidine, Triethanolamine (Sterolamide), 1-decyl-1H-imidazole-2-methanol et [(2-fluorophenyl) methyl] 1H-Purin-6-amine, et la présence des alcaloïdes fixes tels qu'atropine, berberine chloride qui ont été identifiés et quantifiés. D'autre part, 15 composés appartenant à la classe des acides phénoliques et des flavonoïdes ont été identifiés et quantités dont Epicatechin, Apigenin-7-glucosid, Chlorogenic acid, Vanillic acid, p-Coumaric acid, Quercetin par LCHP.

La séparation et la purification des fractions étudiées dont 3 produits isolés sont identifiés à l'aide de la GC/MS, le nonacosan-10-one a été identifié comme un composé pur isolé de la fraction B1-1, avec l'apigenin-7-glucosid et le naringenin dans MAAE-FCR et C108-99M respectivement par LCHP.

L'étude des activités biologiques des fractions/ extraites testées, de *Deverra scoparia* Coss. & Dur., s'est révélée que la plante a un potentiel antioxydant important qui a été évalué par les tests DPPH, Phenanthroline et le test de β -carotène. L'activité antidiabétique a montré que les fractions des alcaloïdes libres et volatiles ont des potentiels inhibiteurs de l'enzyme α -amylase plus élevés ($IC_{50}=260.81 \pm 1.87$, 1630.69 ± 1.41 μ g/ml) que l'acarbose ($IC_{50}=3650,93 \pm 10,70$ μ g/ml) le contrôle positif. En revanche, la majorité des fractions testées possèdent un pouvoir inhibiteur très important de l'enzyme butyrylcholinestérase avec un IC_{50} variait de 1.81 ± 0.87 à 116.39 ± 0.55 μ g/ml. Le test de détermination de la capacité des fractions à protéger la peau contre la lumière du soleil a montré un facteur de protection remarquable. Les fractions d'extrait d'alcaloïdes basiques ont révélé un potentiel insecticide consistant. Toutes les fractions/extraits testées présentent que le pouvoir d'une fraction est en corrélation avec sa composition.

Ces résultats justifient l'utilisation importante de *Deverra scoparia* Coss. & Dur. En médecine traditionnelle.

Mots clés : *Deverra scoparia*, alcaloïdes, GC-MS, LCHP, antioxydant, antidiabétique, anti-Alzheimer, SPF, bio-insecticides.

الملخص:

تهدف هذه الأطروحة إلى دراسة وتثمين المستخلصات القلويدية لنبات *Deverra scoparia* (Apiaceae) Coss&Dur. وهو نبات يستخدم على نطاق واسع في الوصفات التقليدية، لأنه ينتج جزيئات نشطة بيولوجيا لها إمكانات علاجية.

أعقبت دراسة المستخلصات والكسور من القلويدات المتطايرة والقاعدية والحمضية والحرّة تقدير كمية المحتوى القلوي الكلي بطريقة التحليل الطيفي، وتحديد القلويدات المتطايرة عن طريق تحليل GC-MS، وتحديد القلويدات الثابتة عن طريق تحليل HPLC وكذا تقييم الفعالية البيولوجية للمستخلصات والكسور عن طريق بعض الاختبارات المختلفة مخبريا.

يظهر التقدير الكمي للمحتوى القلويدي في الكسور والمستخلصات التي تم الحصول عليها أن AVD يحتوي على أعلى محتوى قلويدي 62895.83 mg EA/kg. أظهر تحليل GC-MS للقلويدات المتطايرة أن الأجزاء AVD، AVE، AWAED، B2، B3 و B4 تحتوي على قلويدات متطايرة وأحماض أمينية وأمينات حيوية مثل 22--2 tetrahydrofuran-2-yl)piperidine, Triethanolamine (Sterolamide), 1-decyl-1H-imidazole-2-methanol و 1H-Purin-6-amine [(2-fluorophenyl)methyl]. كما أظهرت نتائج التحليل بواسطة HPLC وجود قلويدات ثابتة مثل atropine و berberine chloride التي تم تحديدها وتحديد كميتها إلى جانب مركبين آخرين. ومن ناحية أخرى تم تحديد 15 مركباً تنتمي إلى صنف الأحماض الفينولية والفلافونويدات، نذكر منها

Epicatechin, Apigenin-7-glucosid, Chlorogenic acid, Vanillic acid, p-Coumaric acid, Quercetin

أدى فصل وتنقية الأجزاء المدروسة، الى تحديد 3 مركبات معزولة، حيث تم التعرف على nanoncosan-10-one كمركب نقي معزول من الكسر B1-1 باستخدام تحليل GC/MS، إضافة إلى apigenin-7-glucosid و naringenin في FCR-MAAE و C108-99M، على التوالي بواسطة HPLC.

دراسة النشاط البيولوجي للجزيئات والمستخلصات المختبرة. *Deverra scoparia* Coss. وقد ثبت أن لها إمكانات كبيرة مضادة للأوكسدة والتي تم تقييمها بواسطة اختبارات DPPH، phenonthroline و β -carotene. أظهر النشاط المضاد لمرض السكر أن الأجزاء القلويدية للقلويدات الحرّة و المتطايرة لها مثبطات محتملة أعلى لإنزيم الأميليز ($IC_{50}=260.81 \pm 1.87$ ، $IC_{50}=1630.69 \pm 1.41$ $\mu g/ml$ على التوالي) مقارنة بالأكاربوز ($IC_{50}=3650.93 \pm 10.70$ ، $IC_{50}=116.39 \pm 0.55$ $\mu g/ml$)، كميّار القياس الإيجابي. من ناحية أخرى، فإن غالبية الأجزاء التي تم اختبارها لديها قوة مثبطة مهمة جداً لإنزيم بيوتيريل كولينستراز والتي تتراوح قيم IC_{50} لها ما بين 1.81 ± 0.87 و 116.39 ± 0.55 $\mu g/ml$. أظهر اختبار تحديد قدرة الأجزاء على حماية الجلد من أشعة الشمس وجود عامل وقائي ملحوظ. كشفت أجزاء المستخلص القلوي الأساسي عن إمكانات ثابتة للمبيدات

الحشوية. تظهر جميع الكسور والمستخلصات التي تم اختبارها أن فعالية الكسر ترتبط بتكوينه. هذه النتائج تبرر أهمية استخدام نبات *Deverra scoparia* Coss & Dur. في الطب التقليدي.

الكلمات المفتاحية: *Deverra scoparia*، قلويدات، GC-MS، HPLC، مضاد للأوكسدة، مضاد لمرض السكر، مضاد للزهايمر، معادل الحماية من أشعة الشمس، مبيدات حشرية حيوية.

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Abbreviations list

AC	tartaric acid
BuOH	Butanol
BHA	butylhydroxyanisole
BHT	butylhydroxytoluene
CC	Column chromatography
CN	acetonitrile
DCM	dichloromethane
DTNB	5,5'- dithiobis-[2 nitrobenzoate]
LD₅₀	dose required to kill 50%
LD₉₉	dose required to kill 99%
PE	Petroleum ether
n-Hex	n-hexane
Et₂O	ethyl ether
H₂SO₄	sulfuric acid
HCl	hydrochloric acid
MeOH	Methanol
EtOH	Ethanol
NH₄OH	Ammonium hydroxide
IC₅₀	Half-maximal inhibitory concentration
V/V	Volume/ volume
TA	Total alkaloids
MS	Mass sample

TLC	Thin layer chromatography
PTLC	Preparative thin layer chromatography
HPLC	High performance liquid chromatography
GC/MS	Gas chromatography- mass spectrometry.
CA	Nonpolar basic alkaloid
AVD	Alkaloids volatile dichloromethane
AVE	Alkaloids volatile ethyl ether
AWAEE	Acidic water alkaloid extract ethyl ether
AWAED	Acidic water alkaloid extract dichloromethane
FAEDE	Free alkaloid extract dichloromethane
FAEA	Free alkaloid extract ethyl acetate
MAAE	Methanol alkaloid acidic extraction
FAEB	Free alkaloid extract butanol
EA	Ethanol extract basic alkaloid

Introduction

Introduction

Several essential elements of a natural product's biological activity are present, whether used for medicinal or agrochemical purposes. One has to do with the traditional dose-response connection[1]. To ascertain a chemical's toxic level, thorough experimentation is required, as Paracelsus acknowledged in 1541. He identified the differences between a chemical's toxic and therapeutic qualities and believed that, aside from dose, they might be identical. He declared, "No substance (chemical) is not a poison. All substances are poisons." The dosage is the only distinction between a poison and a treatment. Most medications have a limited therapeutic range and become toxic at larger dosages[2].

The study of the chemistry of plants is still highly relevant despite their age. This is mostly because the kingdom of plants is a significant source of a vast array of bioactive compounds[3].

Numerous compounds with various uses in food, medicine, industry, cosmetics, dermatopharmacy, and agriculture is present in this plant material. These substances include flavonoids, phenolic acids, tannins, terpenes, coumarins, and alkaloids [4].

Alkaloids are heterocyclic organic compounds containing nitrogen found mainly in plants, fungi, and some animals. They are naturally basic and form a heterogeneous group in terms of structure as well as chemical or biological properties. Alkaloids often have a defensive role[5]. These groups of compounds represent the largest single class of plant secondary metabolites. They have a remarkable range of frequently dramatic pharmacological activity and are often toxic to human. Many alkaloids are used in therapeutics and as pharmacological tools. A wide range of biological effects has been reported for alkaloids, including emetic, anti-cholinergic, antitumor, diuretic, sympathomimetic, antiviral, antihypertensive, hypo-analgesic, anti-inflammatory, anti-diabetic, anti-Alzheimer and insecticides activities [6]. However, because alkaloids and other natural chemicals are often complicated, it's important to show how their structure and activity are correlated by employing a variety of experimental techniques to study their pharmacological actions[7].

The Algerian flora, with its different species belonging to several botanical families of which 15% are endemic, remains very much explored on the phytochemical level as well as on the pharmacological level[8]. This has given traditional pharmacopeia invaluable wealth. Endemism, traditional use, and the

few studies concerning the species to be studied led us to focus our study on them [9].

In the course of our continuing search for bioactive natural plant products, this study is destined to evaluate the alkaloid constitution and some other secondary metabolites of the plant *Deverra scoparia* Coss & Dur (Apiaceae) growing in Algeria from Tamanrasset, as well as to estimate their biological activity.

Our study is divided into two parts: theoretical and practical study. The first part includes two chapters: chapter one is destined to the alkaloid compounds. In second is the family of Apeaciae, including the studied plant, and anterior studies concerning the genus *Deverra* and *Deverra scoparia* species. The second part is a practical divided into material and methods which describes different experiments and results obtained and their discussions, concerning the plant material, the different methods of extracting alkaloids; Volatile, basic, acidic, and free alkaloids, phytochemical screening, spectrophotometric determination of alkaloids in the different extracts, fractionation of obtained extracts, the purification and identification of volatile, fixed alkaloids as determined by the TLC, GC-MS and HPLC, and phenolic compounds also, and the in vitro evaluation of the pharmacological properties of the fractions were discussed. Four biological activities were estimated:

- The antioxidant potential of different fractions by three methods: DPPH, phenanthroline, and β -carotene.
- The inhibitory potential of enzymes: α -amylase (antidiabetic), and butyrylcholinesterase (ant Alzheimer), in addition to the photoprotective (SPF).
- Evaluation of the Bio-insecticide potential of fractions against the insect *Parlatoria blanchardi*.

Our work was complemented with a conclusion that included all collected results, punctuated with perspective.

Theoretical study

Generalities

I.Generalities

I.1. Generalities on Alkaloids

I.1.1. Definition

Alkaloids are nitrogen-containing compounds found widely across various plant groups. They are typically alkaline and often optically active. Alkaloids are traditionally described as plant-derived, pharmacologically active, basic compounds originating from amino acids and containing one or more heterocyclic nitrogen atoms. In general, most nitrogen-containing secondary metabolites are categorized as alkaloids, unless they can be more appropriately classified as amines or glucosinolates. The term "alkaloid" originates from the Arabic word "al-qali," an early form of soda ash, which also gave rise to the term "alkali." [10]. Alkaloids are normally grouped on the basis of the ring system present. Several common ring systems, including indolizidine- and quinolizidine-based systems [11] and quinoline-, quinazoline-, and acridone-based systems [12] were recently reviewed, including their biosynthesis [13]. Many of the alkaloids are directly derived from the aromatic amino acids, phenylalanine, tyrosine, and tryptophan Figure 1. On the chemical level, they constitute a very heterogeneous group, however possessing some common physico-chemical properties. Must possess a marked biological activity that has aroused, for a long time, a therapeutic interest. Alkaloids are one of the largest families of secondary metabolites, with nearly twelve thousand different structures [14].

Alkaloids, derived from plant extracts, have been used for poisons, narcotics, stimulants, and medicines for thousands of years. Many modern drugs, such as caffeine, quinine, and nicotine, are alkaloid-based. Stronger examples like cocaine, morphine, and strychnine exist. These compounds can be derived from amino acids, terpenes, or aromatics. Research on natural products, particularly alkaloids, began about 150 years ago. British and German chemists developed isolation techniques to obtain pure samples with significant biological effects, though their chemical structures were initially unknown [15]. Efforts to determine the structures of alkaloids started in the mid-19th century and continue today. Alkaloids have diverse and intricate three-dimensional chemical structures. Cocaine's structure was identified in 1898, quinine's in 1944 [16], and morphine was synthesized in 1956 [17]. Due to their complexity, alkaloids are typically extracted from plants rather than being synthesized.

Generalities

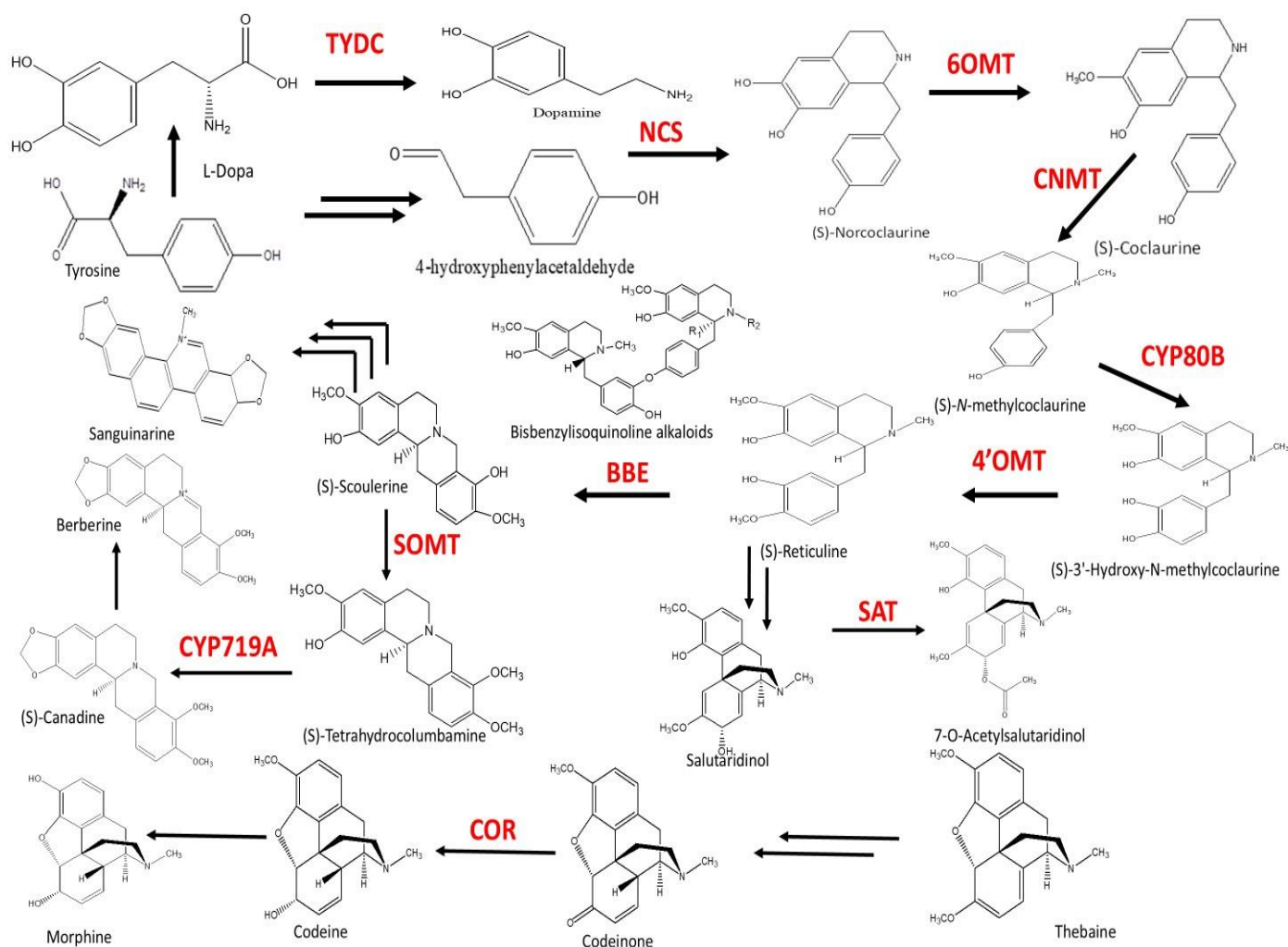


Figure 1: The biosynthesis of alkaloid component[12]

I.1.2.Natural state and distribution

Alkaloids are mainly present in the plant kingdom in angiosperms that contain 10 to 15% and are rare in bacteria (pyocyanin) (*Pseudomonas aeruginosa*) and fungi (psilocin). These compounds are also present in animals (flustramin, saxitoxin, samandarín, etc.). Alkaloids in plants are often located in peripheral tissues (external bases of stem and root barks, root integuments, etc.). They are stored in cellular vacuoles, and their synthesis is often done at specific sites: growing roots, specialized cells of laticifers, and chloroplasts, before being transported to their storage sites[18-20]. In plants, alkaloids exist in soluble form, salts, or in combination with tannins. The alkaloids are located in the peripheral tissues: seed coat, outer stem and root bark layers, epidermis, and sub-epidermal leaves layers. They are stored in cell vacuoles. Synthesis of alkaloids takes place at

Generalities

specific sites: growing roots, specialized cells of laticifers, and chloroplasts [21], [22].

I.1.3. Classification

The chemistry of alkaloids is one of bioorganic chemistry's most interesting and important topics. The term alkaloid" was initially applied to N-containing compounds of plant origin that had a distinctly basic nature which they had a volatile or fixed nature. They are classified using various signatures, such as natural sources or chemical nature. The most correct and common classification of alkaloids is their distribution according to their principal structure, the principal C-N skeleton. Alkaloid compounds can be classified from the position of the nitrogen atom such as position, structure, etc. [23-25]; Heterocyclic alkaloids/ Alkaloids with an oxo ring nitrogen atom and aliphatic amines/ putrescine, spermidine, and spermine alkaloids/ alkaloid peptides and terpene and steroid alkaloids. Presently, alkaloids are classified into three main categories, as shown in Figure 2. This chemical classification of alkaloids is universally adapted and mainly depends on the type of heterocyclic ring structure present in Figure 3. Alkaloids are further classified according to the amino acids (or their derivatives) from which they originate Figure 4. The most important classes are derived from the amino acids, ornithine and lysine, from the aromatic amino acids Figure 5, phenylalanine, and tyrosine; or from tryptophan and a moiety of mavelonoid origin. Several alkaloid-based compounds are also derived from anthranilic acid or nicotinic acid [26-28].

Generalities

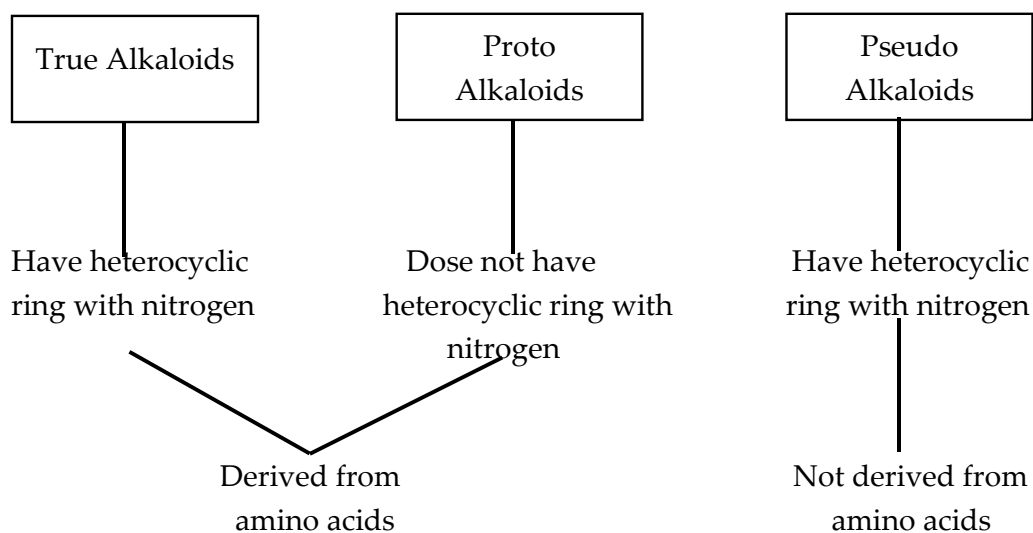


Figure 2: Classification of Alkaloids according to amino acid and heterocyclic ring [26]

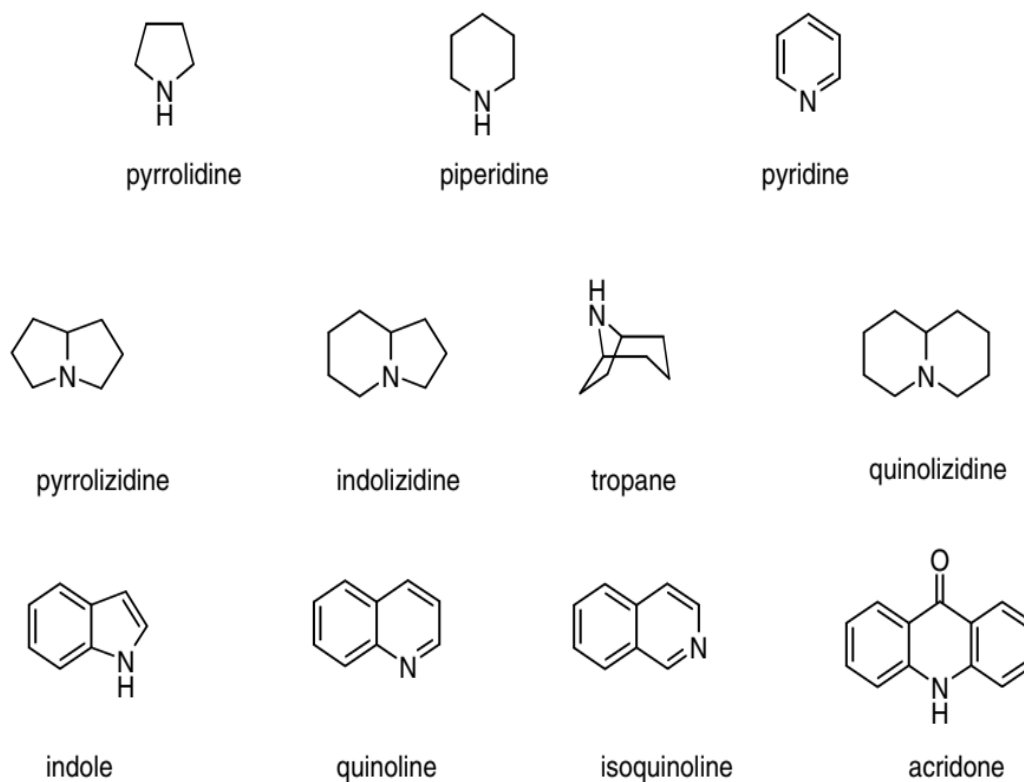


Figure 3: Heterocyclic ring structures found in many alkaloids [27]

Generalities

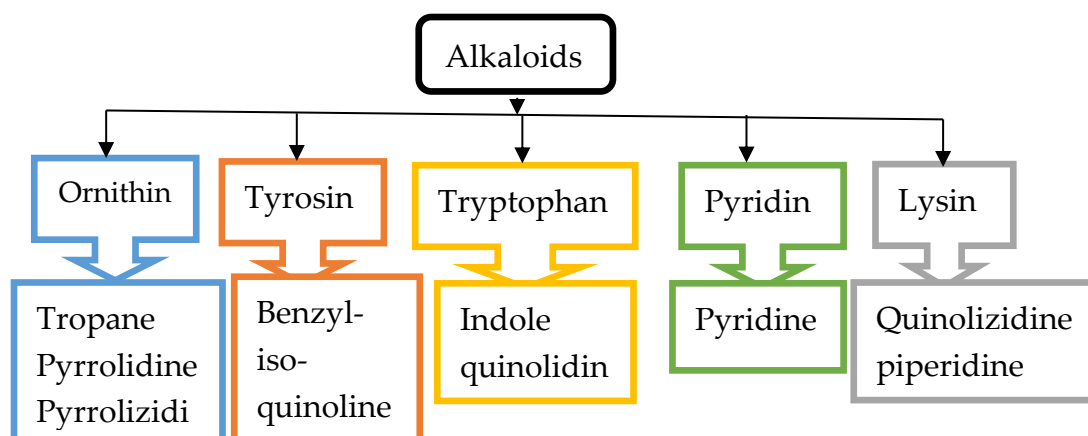


Figure 4: Classification of alkaloids on the basis of amino acid from which it has been derived

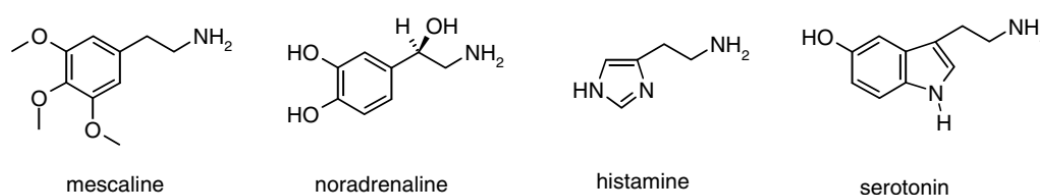


Figure 5: Some aromatic amine structure [28]

I.1.4. Physicochemical property

Alkaloids have molecular masses ranging from 100 to 900. The majority of alkaloids are solid a few exceptions to this rule are coniine and nicotine, which are liquid alkaloids. Except for berberine and betaine, which are yellow and red, respectively, emetine is amorphous and colorless. Because it contains a nitrogen atom, its basicity is dependent on the availability of free electrons, the type of hybridization, and aromaticity. It is also insoluble in water, except for liquid alkaloids like nicotine, which are soluble in it. In general the non-oxygenated alkaloids are liquid at ordinary temperature while the oxygenated alkaloids are crystallizable solids, rarely colored. The alkaloids deflect polarized light, and crystallized bases give sharp melting points, without decomposition (< 200) [29], [30]. Esters, quaternary compounds, salts, N-oxide, and other forms are among the various forms found in plants. They are unstable in the presence of heat or light, but they dissolve in an aqueous solution of sodium carbonate or ammonia when treated with alcohol, generating an ester [31], [32].

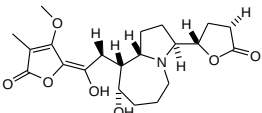
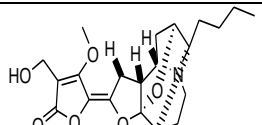
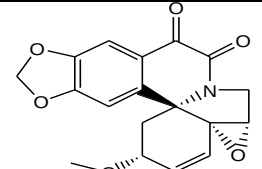
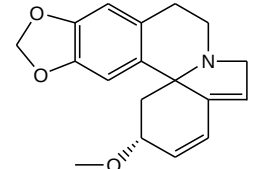
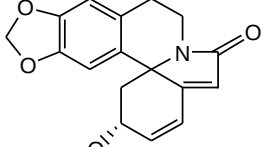
I.1.5. Pharmacological properties

The pharmacological properties of alkaloids notable indole alkaloids include reserpine, an antihypertensive alkaloid from Indian snakeroot (*Rauwolfia serpentina*), and vinblastine, one of the antitumor alkaloids, from the rosy

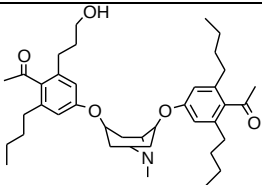
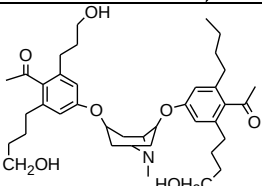
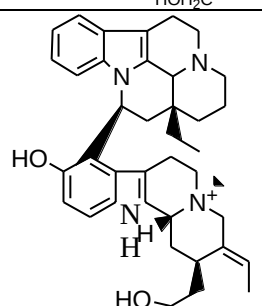
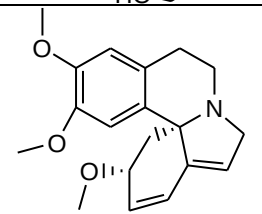
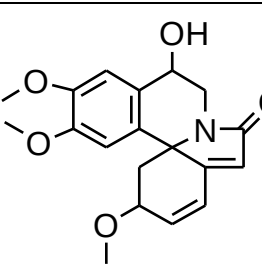
Generalities

periwinkle (*Catharanthus roseus*). In contrast, Two alkaloids, vincristine, and vinblastine (from *Catharanthus roseus*, formerly *Vinca rosea*), are widely used as chemotherapeutic agents in the treatment of many types of cancer[24]. Many alkaloids have a bitter taste, and a large number of them exhibit potent physiological effects on mammals. For example, morphine shows narcotic effects; reserpine is an antihypertensive agent; atropine is a smooth muscle relaxant; cocaine is a local anesthetic and a potent central nervous system stimulant; and strychnine is a nerve stimulant[25]. Alkaloids in plants serve as chemoprotective antiherbivore agents or as growth regulators, such as the well-known plant hormone, indole-3-acetic acid, IAA (an indole derivative synthesized from tryptophan[33] as well as include analgesics (morphine), antimalarials (quinine), oxytocic's (ergonovine), neuralgia (aconitine), cholergics, and laxatives (poldine), reflex excitability (lobeline), antiglaucoma agents (pilocarpine), bronchodilators (ephedrine), and so on, are categorized according to their pharmacological activity[7], [34-36].

Table 1: Some alkaloids with a biological property

Alkaloid	Structure	Origin plant	Biological properties	Ref.
13-dimethoxy-11(<i>S</i> *),12(<i>R</i> *)-dihydroprotostemonine		<i>Stemona japonica</i> (Stemonaceae)	Insecticidal	[37]
16-hydroxystemofoline				
10, 11-dioxo-6,7 α -erythraline epoxide		<i>Erythrina corallodendron</i>	Neurodegenerative diseases	[38]
erythralin				
8-oxo-erythraline				

Generalities

Hypocretine (2 compounds)		<i>Rivea hypocraeteriformis</i>	Antihemolytic	[39]
				
bisindole alkaloids (bisnicalaterines)		<i>Hunteria zeylanica</i>	Vasorelaxant activity	[40]
Erysotrine				
Hydroxyerysotramidine		<i>Erythrina speciosa</i>	/	[41]

I.2. Generalities on studying plant

I.2.1. Apiaceae family

Apiaceae is a family created by Antoine Laurent de Jussieu in 1789 under the name Umbellifera, then named Apiaceae by John Lindley in 1836. This family is described under the name of Umbellifera with the umbellar structure of the inflorescences. In classical systematics, the work of Drude shows that the Apiaceae are divided into three subfamilies according to the morphological characters of the fruit: the Apioideae (the largest subfamily), the Hydrocotyloideae and the Saniculoideae [42]. Apiaceae are a very homogeneous family, easy to recognize due to their typical umbel inflorescence. They are herbaceous, biennial, or perennial plants, sometimes shrubs [43]. The leaves are an alternate compound, rarely simple. Often the petals are widened at their base, sheathing the stem. The stem is often hollow. The flowers are united in simple or compound umbels with bracts called involucels at the base. This important family is less distributed and represented in the Sahara, especially in its central part [44-47]. On the other hand, the Saharan Apiaceae (Umbellifera) are very different from each other and their determination does not present great difficulties, except the distinction between the species of *Pituranthos*. Take care to collect samples bearing ripe fruit. The African Saharan contains 126 genus of the Apeacea family 50 genus were endemic [48].

I.2.2. Tradicional uses of Apiaceae

Certain plants of the Apiaceae family represent an economic interest, such as aromatic food plants such: *Anethum graveolens* L (dill), *Apium graveolens* L. (celery), *Carum carvi* L (caraway),... etc. Others are used as natural additives in the food industry, some species are edible, such as: *Daucus carota* (carrot), *Pastinaca sativa* (parsnip), *Foeniculum vulgare*, etc. Some species are used as condiments or spices, such as *Carum carvi* (cumin), *Anetum graveolens* (dill), *Pimpinella anisum* (anise) *Petroselinum sativum* (parsley). Others are used as flavorings for drinks, such as *Angelica archangelica* (angelica), *Laserpitium gallicum* and several species of *Heracleum*. Some genera, however, are highly toxic, such as *Conium* (the great hemlock, which is said to have been used in the suicide of Socrates), and *Cicuta* (the hemlock) [49], [50]. On the other hand, some species are used in traditional medicine such us the *Ammi majus* fruit is used to cure psoriasis and to color white areas on the skin that occur in cases of vitiligo. Its ability to photosensitize is caused by the existence of many furocoumarins that are derived from psoralen, bergapten being the most well-known [51]. The *Anethum graveolens* (dill) characteristics are akin to those of fennel and anise, namely antispasmodic, eupeptic, carminative, and diuretic effects on the

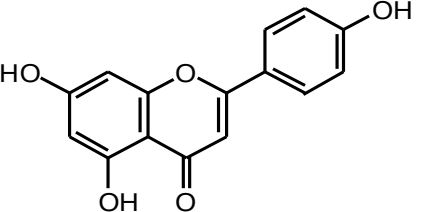
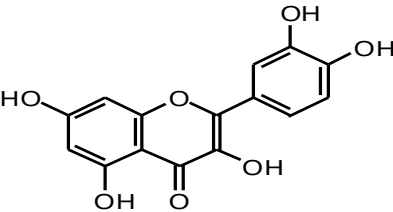
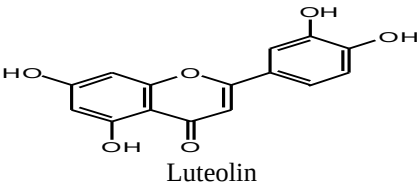
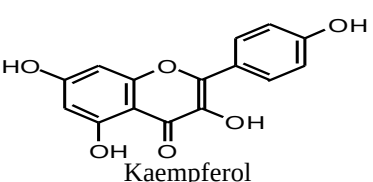
Generalities

digestive system [52]. The root of *Angelica archangelica* (angelica) contains angelicin, a furocoumarin with sedative properties. The essence of angelica is responsible for its stomachic, eupetic, and carminative qualities [51]. In addition to the *Apium graveolens* (marsh ache) was used as diuretic, carminative and tonic, *Cicuta virosa* (hemlock) used in homeopathy (epilepsy), *Conium maculatum* (grande cigue) was used as an antispasmodic and sedative agent at low dose, *Heracleum sphondylium* (great hogweed) used as hypotensive, stimulant and digestive agent [53]. furthermore, the group of Ouafae Benkhningue and his colleagues also found in a study that 10 species of the Apiaceae are used in the treatment of diabetes in the region of Al Haouz-Rhamna (central Morocco) with other different species (other families) [54-56]. On the other hand, a study on the search for and identification of some medicinal plants with a hypoglycemic nature from the traditional pharmacopeia of the communities of the M'zab valley (northern Sahara in eastern Algeria). The study of the chemical composition and the biological activity of a food plant is also used in industrial cosmetics it is the *Apium graveolens* (Celery) of the family Apiaceae [46].

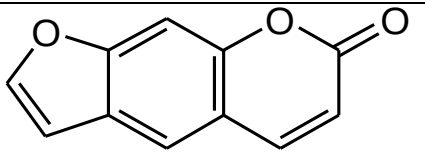
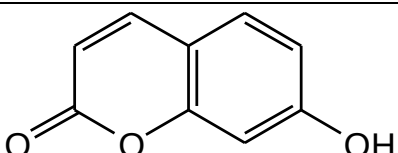
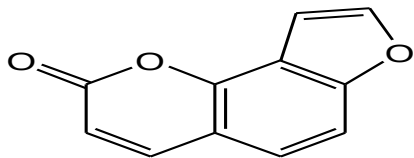
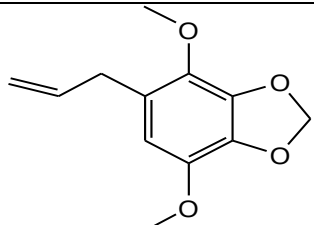
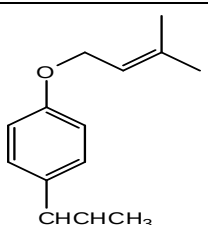
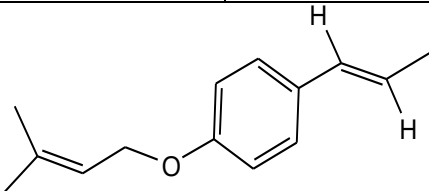
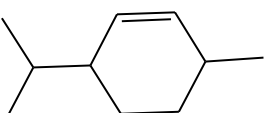
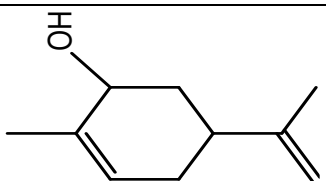
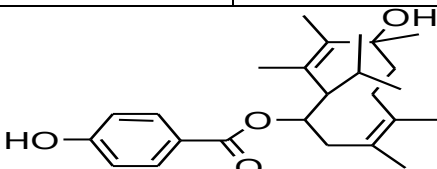
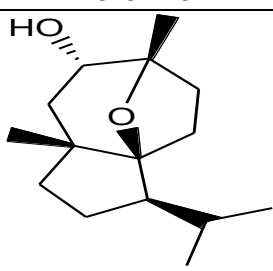
I.2.3. Chemistry of Apiaceae

Apiaceae are known for the great diversity in secondary metabolites and all the readings and the results of the work show that this family includes a wide variety of coumarins[57], [58], sesquiterpenes [59], [60], sesquiterpene lactones [61], acetyl compounds[62], essential oils[63], flavonoids [64]. These active ingredients are the main secondary metabolites of this family. In addition of the presence of chromones [65], chalcones [66], saponins [67], and alkaloids[68] has been reported in some species of this family[69].

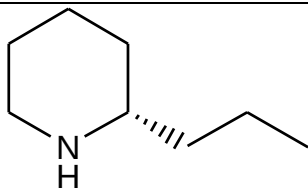
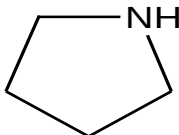
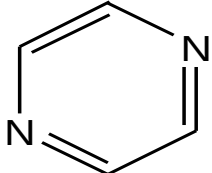
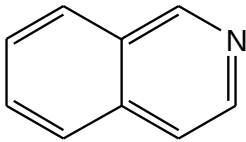
Table 2: Some class compounds of Apiaceae

Classes	Compounds (Structure)		Ref.
Flavone			[70]
Flavanol			
	Apigenin	Quercetin	
	Luteolin	Kaempferol	

Generalities

Coumarins	 Psoralen	 Umbelliferone	[71]
	 Angelicin		
phenylpropanoid	 Apiol	 Foeniculine	[72]
	 Foeniculin		
Terpene	 β -phellandrene	 Carveol	[73]
	 feroline		
	 Daucol		

Generalities

Alkaloid	 Coniine	 Pyrrolidine	[74] [68]
	 Pyrazine	 Isoquinoline	

I.2.4. *Deverra* genus

The genus *Deverra* (or *Pituranthos*) includes more than 20 species. Of which the Algerian floristic potential of this genus includes four endemic species: *Pituranthos rebondii*, *Pituranthos battendier*, *Pituranthos chloranthus*, and *Pituranthos scoparius* [8].

I.2.5. Traditional use of the genus

The genus *Deverra* is widely used in traditional medicine in many areas.

Certain species of *D. scoparia*, *D. chlorantha*, *D. triradiatus* and *D. tortuosus*, are used against diabetes, fever, urinary tract infections, stomach pains, intestinal parasites, or as a regulating agent of menstruation in women. They are used in the treatment of asthma, and hepatitis and to relieve pain associated with rheumatism. The sweet flowers are also popular. They are soaked in water to extract the sugar. The stems help salivate and reduce thirst; together with the leaves, they give off a pleasant aroma, so we cover the fresh meat with a bed of green stems of these plants and roast the upper part of the pancakes by burning them. dry stems. Nomades use this plant to flavor meat and flatbread [75-77].

I.2.6. *Deverra scoparia* species

Deverra scoparia (Coss. & Dur.); *Pituranthos scoparius* (Benth & Hook), It is an endemic spontaneous growing plant belonging to the Apiaceae family which is one of the main and most distinctive families of flowering plants, with yellowish stems, in tufts, branched at the top only simple and parallel to each other in their lower half bearing lateral umbels; peduncles often short; narrowly veined white

Generalities

petals [8]. The whole plant gives off a pleasant smell. This species is known in Arabic as "Guezzah" or "Tattayet" in Tamahaq. The Tuareg use it as an aroma for meat and bread on the other hand it is used for the treatment of diarrhea, back pain, eczema, asthma, and rheumatism and aids digestion. This toxic plant is avoided by sheep during flowering and whose toxicity has been linked to the presumed presence of alkaloids. In Algeria, it is observed in the regions of Hoggar (Tamanrasset), Bechar, Biskra, Souk-Ahras, Batna, Laghouat, and Ghardaïa[78-80].

I.2.7.Classification

Kingdom: Plant

Class: Magnoliopsid.

Order: Apiales.

Family: Apiaceae.

Genus: *Deverra*.

Species: *Deverra Scoparia* Coss. & Dur.

Table 3: Previous studies on *D. scoparia*

Study plant part	phytochemical investigation	Compounds	Biological investigation	Ref.
STEMS, SEEDS	Identification of 16 volatile compounds	α -Pinene/ β -Pinene/ α -Phellandrene/ <i>p</i> -Cymene/ Limonene/ Thymol/ Methyleugenol/ Germacrene D/ Myristine/ Spathulenol/ Dill apiol/ Bornylacetate/ <i>trans</i> -Verbeno/ Apiol.	Antibacterial	[81]
Aerial parts	Identification of 8 volatile active compounds	α -Pinene/ Germacrene D/ Myristicin/	/	[82]

Generalities

		Dill apiole/ Bornyl acetate/ Apiol/ Limonene/		
	Volatile components	α -Thujene/ α -Pinene/ Sabinene/ Myrcene/ D-3-Carene/ Ocimene/ Terpinene-4-ol/ Pulegone/ Eugenol/ β -Eudesmol.	Acaricidal (<i>Tetranychus urticae</i> (Acari: Tetranychidae)	[83]
	Identification of Volatile components	(Z)-Ligustilide/ β -Eudesmol/ τ -Muurolol/ Dill apiole/ 6-Methoxyelemicine/ Myristicine/ Germacrene D/ Terpinen-4-ol/ δ -Terpinene/ β -Phellandrene/ Limonene/ α -Phellandrene.	/	[84], [85]
Root parts	Identification of isocoumarins	(A) 3-n-propyl-5-methoxy-6-hydroxy-isocoumarin (B) 3-n-propyl-5,7-dimethoxy-6-hydroxy-isocoumarin	/	[71]
Aerial parts	Identification of 2 cinnamic acid/ 13 flavonoids	5-O-Caffeoylquinic acid/ 5-O-Feruloylquinic acid/ Apigenin 6,8 di-C-glucoside (Vicenin-2) /	/	[86]

Generalities

		Quercetin 3- <i>O</i> -gentobiosyl-pentosidea/ Quercetin 3- <i>O</i> -sophoroside/ Quercetin 3- <i>O</i> -gentobioside 0.3/ Isorhamnetin 3- <i>O</i> -gentobiosyl-pentosidea/ Isorhamnetin 3- <i>O</i> -sophoroside/ Quercetin 3- <i>O</i> -glucosyl-pentoside/ Isorhamnetin 3- <i>O</i> -gentobioside/ Quercetin 3- <i>O</i> -neohesperidoside.		
Aerial and root parts	Polar and non-polar extracts	/	Antibacterial	[87]
Aerial parts	Polar and non-polar extracts	/	- Antioxidant - Hemolytic potential	[88]
Aerial and root parts	Polar and non-polar extracts/ phytochemical screen	-	-Antiuro lithiatic	[89]
Aerial parts	Volatile compounds	-	- Antioxidant - Antimicrobial	[78]
	Polar extract	-	- antioxidant	[90][91][92]
	Phenolic compounds	Salicylic acid/ Rosmarinic acid/ Apigenin	- analgesic -anti-inflammatory	[79]
	Phenolic compounds	Flavanone 7- <i>O</i> -glucoside	- inhibition of porcine liver	[93]

Practical study

II. Material and Methods

II.1. Vegetal material

The Ariel part of *Deverra scoparia* was collected from the region of Tamenghrest "Oued TASSENA", North 22° 47' 672" , East 005° 35' 429" during the flowering period (September - November 2018, 2019 and 2021), identified with the herbarium (number PM/02), the study part was dried away from the light, in the open air, and the dark Figure 6.

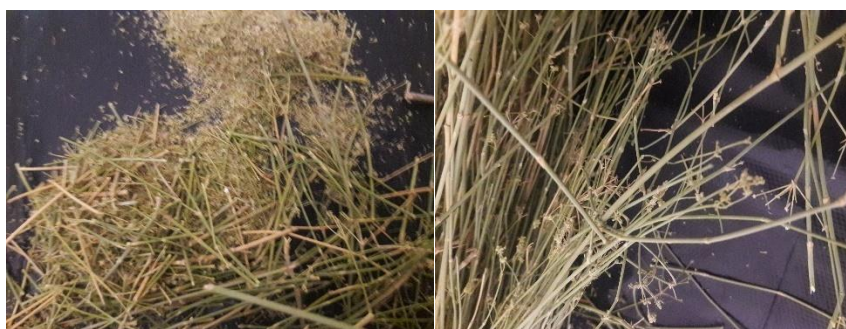


Figure 6: The dried ariel part of *D. scoparia*

II.2. Phytochemical study

II.2.1. Extraction of Alkaloid

II.2.1.1. Extraction of volatile Alkaloids (AV)

The air-dried material was subjected to hydro-distillation for 4h using a Clevenger material. The essential oil was collected and the hydrolat phase was acidified (0.5N HCl) and extracted with Et₂O (liquid-liquid extraction) 3 times. After concentration, a yellowish oily fraction **AVE** was obtained. The aquatic phase was treated with NH₄OH (0.5N) pH= 9 and exhausted with DCM the absence of the response of Dragendorff (orange precipitate) indicates the extraction was ended (negative Dragendorff response), concentrated, and dried over anhydrous MgSO₄ to obtained brownish extract **AVD** figure 7. The fractions were conserved at -4°C until analysis[94].

Material and Methods

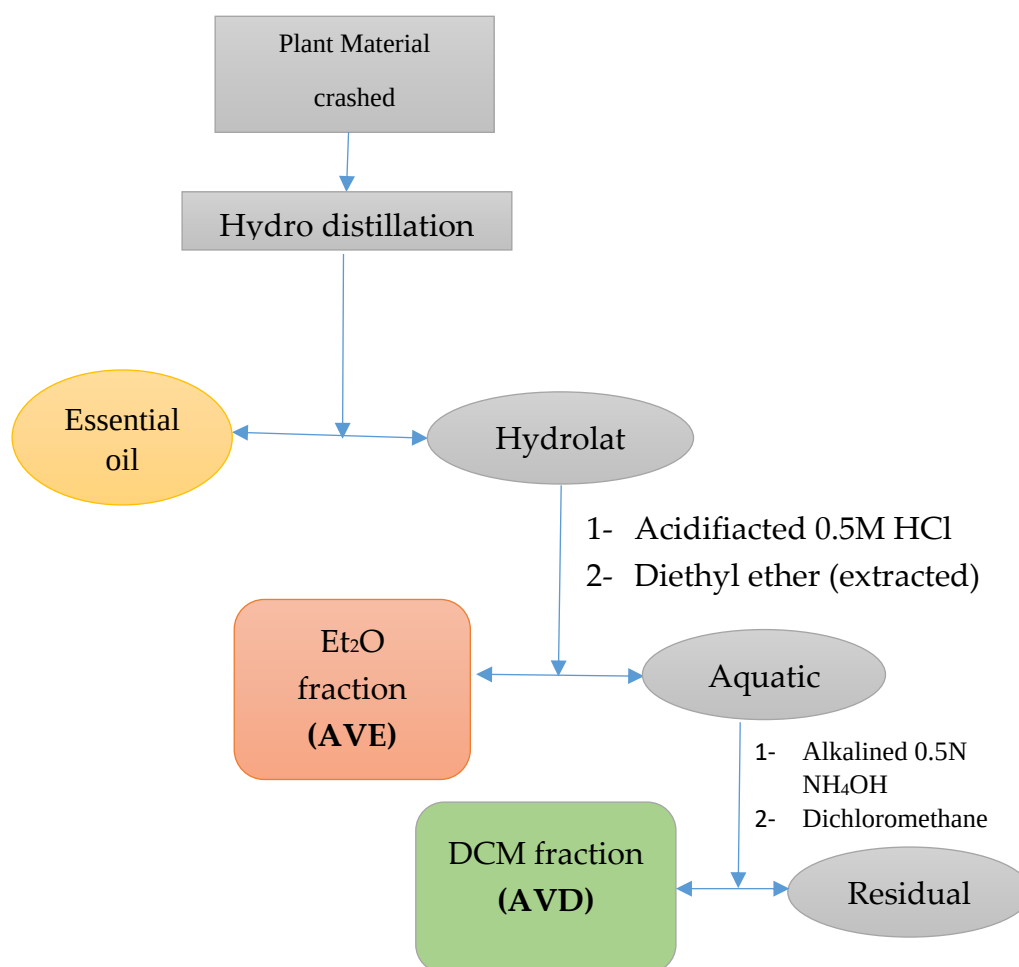


Figure 7: Graphic extraction protocol of volatile alkaloids

II.2.1.2. Extraction of fixed alkaloids

A. Extraction In an alkaline medium

A.1. Extraction with non-polar solvent

The dried plant material was powdered, 1 kg was (have been delipidated with petroleum ether PE (3L), and after 8h was filtrated. The residue was alkalized with NH₄OH (0.5M) for 24h. The marc was extracted by Soxhlet (5 cycles) with DCM solvent.

The organic extract was concentrated and washed with 150 ml of sulfuric acid (SA) (0.5 N) (1/3) (V/V) three times. The aqueous solution containing the salt alkaloids was treated with (NH₄OH) (0.5 N) until PH=9 was exhausted with

Material and Methods

ethylic ether. The organic phase was extracted with DCM to obtain a crude alkaloid CA[29] (Figure 8).

A.2. Extraction with polar solvent

The residual marc was extracted with ethanol by maceration for 24 hours (thrice). The extract was filtrated and concentrated to get a brown extract EA, figure 8.

B. In an acidic medium with the polar solvent

100 g of the plant material dried and powdered was pulverized with 0.2% tartaric acid and extracted with MeOH by reflux system for 8h [95] to obtain the methanolic acidic alkaloid extract MAAE (Figure 9).

C. Extraction with multiple solvents

C.1. Extraction with acidic water AW

A dried plant material was extracted with (900 ml, 0.1 M) phosphoric acid under agitation for 30 min. Extraction was repeated four times to obtain an acidic water alkaloid extract AWAE as solution. The AWAE solution was filtered and reunited to the same funnel. The AWAE solution was three times extracted with 1/3 V/V hexane in order to have non-polar compounds removed, then extracted with diethyl ether solvent to get AWAE fraction. The acid solution was brought to pH ~ 9 with ammonia hydroxide solution (25%, w/w) and it was partitioned with DCM 1/3 v/v, until negative to Dragendorff's reagent. The organic phase was dried with anhydrous Na₂SO₄ and concentrated to give the final alkaloid fraction AWAED[96] (Figure 10).

C.2. Free alkaloids extract FAE

The marc, considered as the remaining insoluble material resulting from the alkaloid extraction, was dried and the remaining solids were dissolved in a series of solvent systems. Hexane (300 ml) was added to the marc and the system remained under agitation for 30 min. After that, the system was decanted and filtered. This procedure was repeated three times under the same conditions. The combined hexane solutions were evaporated and originated fraction hexane FAEH. A second dissolution done similarly was performed with a mixture of dichloromethane (DCM) and ethanol (EtOH) (1:1, v/v), resulting in a fraction of DCM/EtOH FAEDE. The third dissolution was made with ethyl acetate, resulting in a fraction of FAEA. Finally, the remaining marc was partitioned with a 20% ethanol: H₂O solution and extracted with three portions of 250 ml each of butanol, originating the fraction butanol FAEB[96] (Figure 10).

Material and Methods

The yield of all obtained extracts was calculated with the following equation:

$$\text{Yield (\%)} = (M_1 / M_0) \times 100$$

M1: extract masse

M0: vegetal material

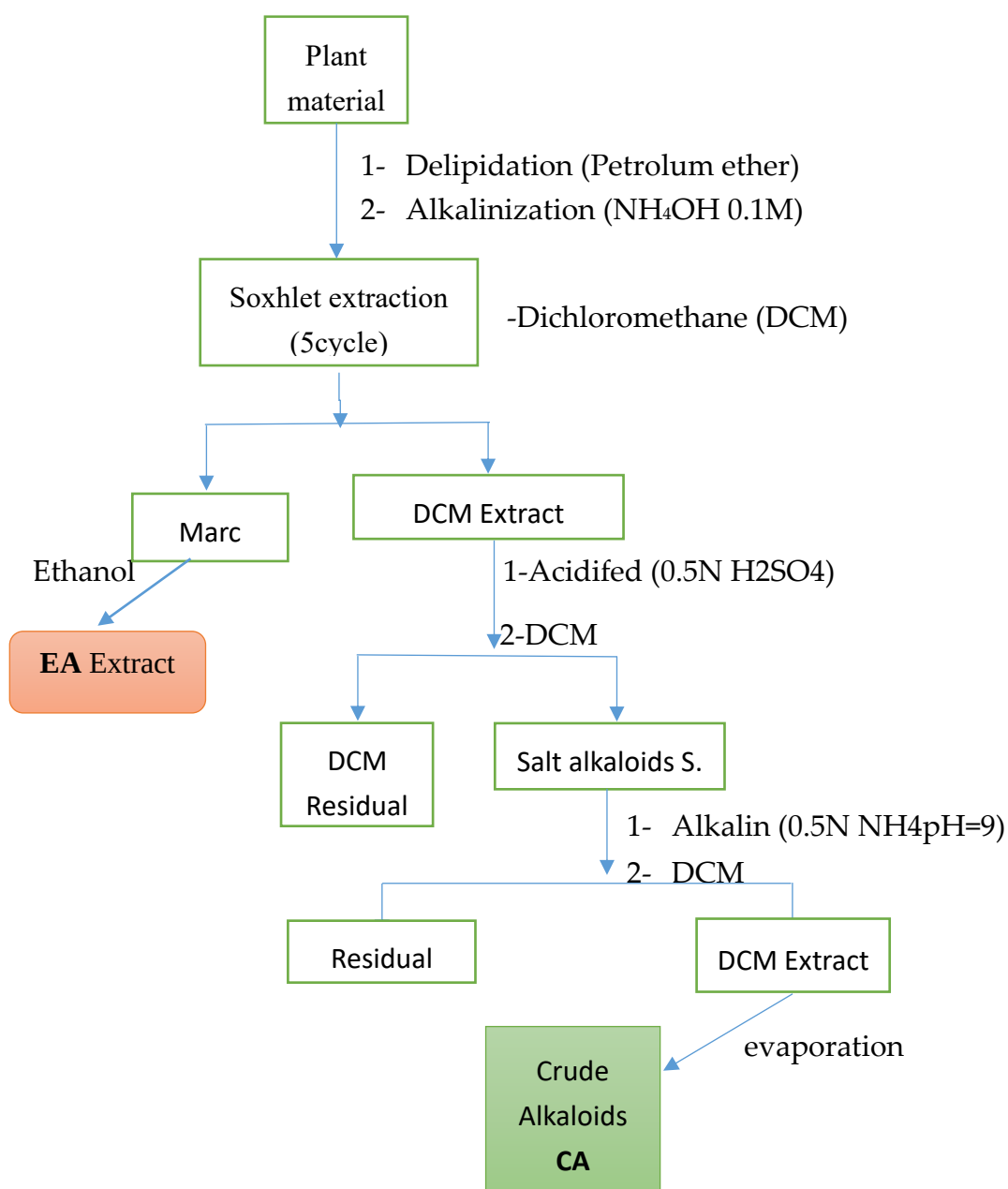


Figure 8: Graphic extraction protocol of basic alkaloids (polar, non-polar)

Material and Methods

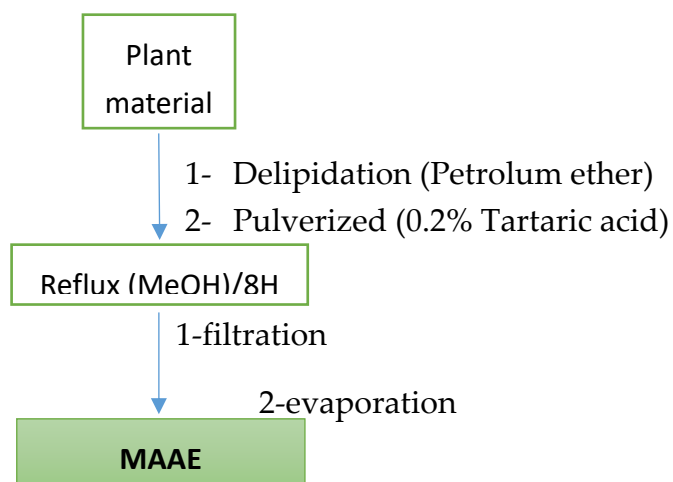


Figure 9: Graphic extraction protocol of acidic alkaloids

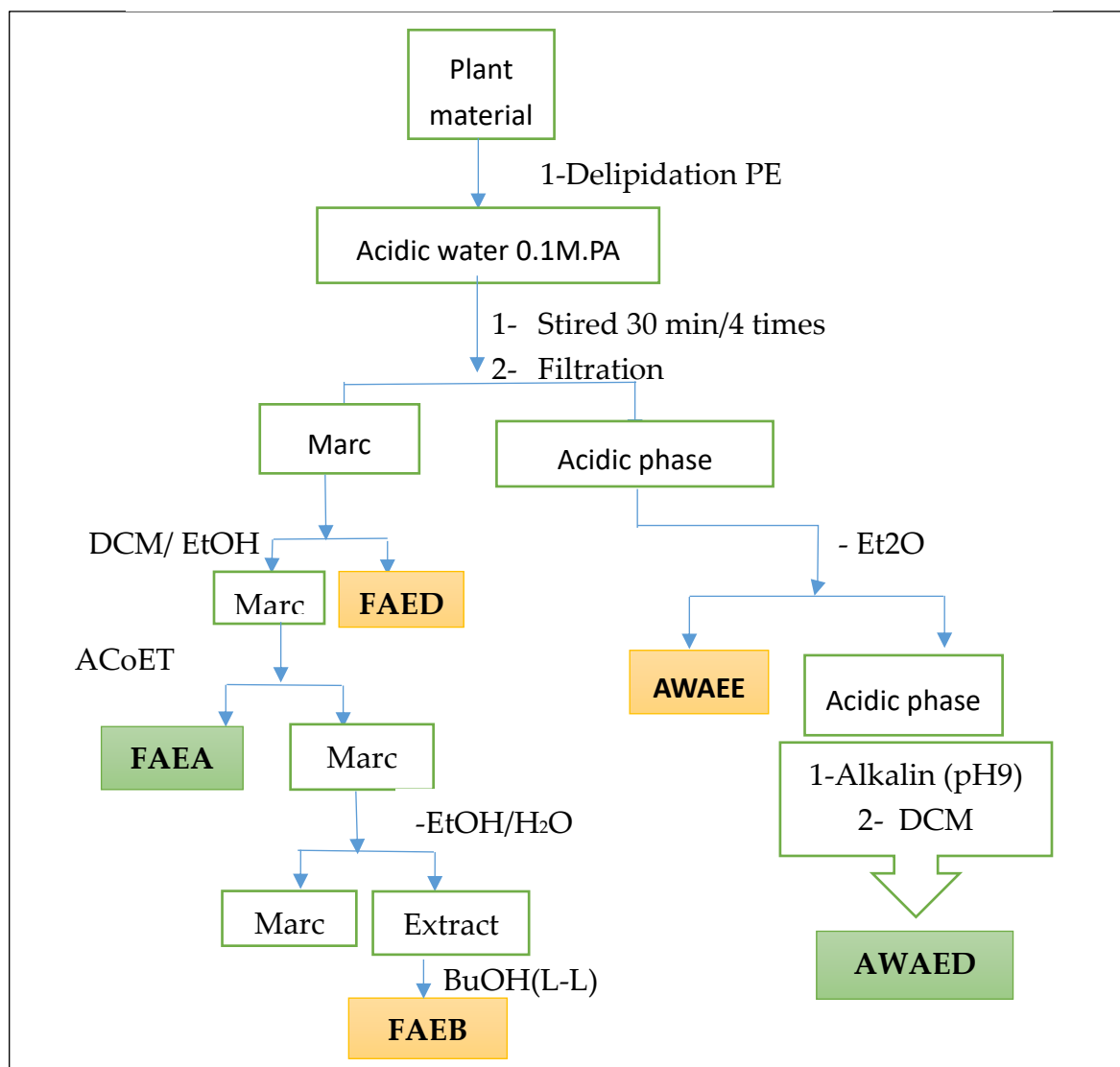


Figure 10: Graphic extraction protocol of free alkaloids

Material and Methods

II.2.2. Phytochemical screening

The CA extract of *D. scoparia* was subjected to a phytochemical screening to detect the presence of potential phytochemical constituents such as alkaloids, flavonoids, polyphenols, and tannins according to published procedures[97], [98]. These qualitative analyses were based on coloration and/or precipitation reactions (table 4).

Table 4: The phytochemical screening tests

Metabolite	Test	Detection
Polyphenol	Ellagic acid	Black, brown or red color
Flavonoid	Shinoda	Red color
Alkaloids	Dragendorff	A red-brown precipitate
	Mayer	A white-yellow precipitate
Tannins	FeCl ₃	Green or blackish black color

II.2.3. Quantification of total alkaloids (TA)

The level of alkaloids in all extracts was determined according to the method described by Shamsa et al., . To 1 ml of each extract are added 10 ml of phosphate buffer solution (pH 4.7) and bromocresol green solution with a 1:1 (V/V) ratio and 4 ml of chloroform, the mixture is stirred vigorously. After decantation, the chloroform phase is removed and the phase containing the extracts is collected in a 10 ml volumetric flask and the volume is adjusted with chloroform. The absorbance is measured at 470 nm against the blank prepared without extract. Atropine is used as the standard. The alkaloid concentration is expressed in mg of atropine equivalent/kg of extract (mg EA/kg)[99].

II.2.4. Fractionation, Separation, and Purification

To separate the alkaloid compounds from different extracts, several separation methods and techniques were applied.

II.2.4.1 Precipitation method

To obtain a pure compound or less charge alkaloid fraction we used the precipitation method based on the solubilization of their constituent in several solvents with croissants polarity; (n-hexane **n-Hex**, ethyl ether **Et₂O**, DCM, Toluene, Acetonitrile **CN**, EtOH, and MeOH)[100].

Material and Methods

II.2.4.2. Analytical thin layer chromatography (TLC)

The different extracts, fractions, and purified sub-fractions are analyzed on TLC plates silica gel 60 as a stationary phase. The type of plate is GF254 Merck, 0.1 mm on aluminum support.

We made some TLC analysis to select the corrected fractions with major alkaloid compounds, and low complex. for choosing the suitable mobile phase “Q” for column chromatography:

CHCl_3 / diethylamide (9:1) = **QA**

EtOAc/ MeOH/ NH_4OH (9:1:1) = **QB**

Ether/ EtOAc (8/2) = **QC**

CH_2Cl_2 / MeOH (1:1) = **QD**

EtOAc/ CH_2Cl_2 (1:1) = **QE**

n-hexane/ CH_2Cl_2 (20/1) = **QF**

The revelators used were:

Ninhydrin reagent (NIH): 30 mg of ninhydrin is dissolved in 10 ml n-butanol, followed by 0.3 ml of 98% acetic acid. After spraying (8~10 ml), the plate is heated for 5~10 min under observation, and evaluated in vise. NIH was used to detect the amino acids and biogenic amines[101].

Dragendorff reagents (DRG): used to detect alkaloids, heterocyclic nitrogen compounds, and quaternary amines[101].

The Dragendorff reagent was prepared by following the procedure of 0.85 g basic bismuth nitrate dissolved in 40 ml water and 10 ml glacial acetic acid, followed by the addition of 8 g potassium iodide dissolved in 20 ml water.

II.2.4.3. Chromatography column CC

The chromatography column is a separation method used to separate and purify a complex or less complex mixture according to the behavior of its constituents (compounds) when in contact with the stationary and mobile phase [102]. Several types of stationary and mobile phases can be used in glass columns, the length and diameter of the column were chosen according to the quantity of samples to be separated or purified. There grouping of the obtained fractions was done according to their TLC profile similarity.

Material and Methods

A. Fractionation of the basic alkaloids extract CA

The CA extract was mixed with a few grams of silica gel and then pulverized until a homogeneous powder was obtained. The obtained mixture was adsorbed on 100g of silica gel G60 by using column chromatography ($\phi=3\text{cm}$, $L=60\text{cm}$), and eluted with DCM/MeOH with gradient polarity (100:0- 75:25- 50:50- 25:75- 0:100), to obtain fractions; B, C, C', D, E, and F (Table5) (Figure 11). All fractions were analyzed with TLC.

Table 5: CA fractionation by CC

Eluent %	Fraction	Color
DCM 100	B	Dark Brown
DCM/MeOH 75/25	C	Dark green
	C'	Green
DCM/MeOH 50/50	D	Brown
DCM/MeOH 25/75	E	Brown
MeOH 100	F	Light brown

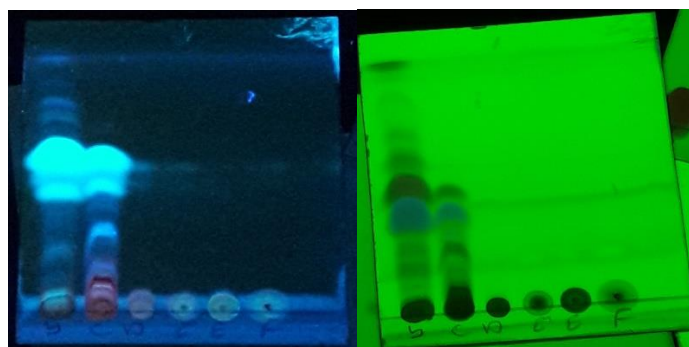


Figure 11: TLC profile of CA fractions 365/254 nm “QD”

B. Separation and purification of B fraction:

The **B** fraction (2144.8 mg) has been divided into two parts, the first part (1072.4 mg) was chromatographed on CC ($\phi=3.5\text{cm}$, $L=80\text{cm}$).

Stationary phase: silica gel 100g.

Mobile phase: n-hexane/ DCM (20/1, 15/1, 10/1, 5/1, V/V), DCM/ n-Hex/ MeOH (1/1/0,1)(Table 6).

The B1 fraction was dissolved in n-hexane three times and filtered, the precipitate was dissolved again in DCM to obtain a white precipitate washed with MeOH to get a white crystal “**B1-1**”.

Material and Methods

The regrouping was done two times **B12** and then **B6** according to the TLC profile results.

Table 6: The CC fractionation of B fraction.

Elution system	Fractions	GF-name / mass (mg)
n-Hex/ DCM %		
20/1	1 – 23	B1/ 132,9
15/1	24-37	B2 / 28,5
10/1	38-44	
5/1	45-50	
DCM/ n-Hex/ MeOH		
1/ 1/ 0,1	51-59	B3/ 78,4
1/ 1/ 0,15	60-67	
1/1/0,2	68-78	
1/1/0,25	79-83	
1/0,5/ 0,5	84-86	
1/0,5/ 0,6	87-99	
1/0,5/ 0,7	100-104	
1/0,5/ 0,8	105-110	
1/ 0,5/ 0,9	111-118	
1/ 0,5/ 1	119-126	
DCM/ MeOH		
1/1	128-139	B4/ 34,6
	140-148	B5
MeOH		
1	149- 160	B6

The second part of the **B** fraction was precipitated with n-hexane **B'**. The n-hexane layer was purified on CC ($\phi=1.5\text{cm}$, $L=20\text{cm}$).

Stationary phase: silica gel

Mobile phase: n-Hex/DCM (Table 7).

Material and Methods

The fractions FB 1-10 after drying were washed firstly with DCM and then with MeOH, the sub-fractions **SFB1-4 MeOH** indicate the presence of pure compound analyzed by TLC (Figure 13). But it hasn't been analyzed with other methods because of a few quantities.

Table 7: The CC result of the precipitate B' fraction

Elution system%	Fractions	Group-fractions name
n-Hex/ DCM		
20/1	1-6	FB1-10 / FB11-12
20/5	7-12	
20/10	13-16	FB13
20/20	17-19	FB14
DCM/ n-Hex/MeOH		
20/20/1	20-22	FB15
20/20/2	23-24	FB16
20/10/4	25-26	FB17
20/10/8	27-29	FB18
20/5/10	30-34	FB19
DCM/MeOH		
20/20	35-41	FB20
15/30	42-44	FB21
5/40	45-48	FB22
MeOH (1)	49/ 55	FB23
MeOH/ acidic water (1/0,5)	56-61	FB24

Material and Methods

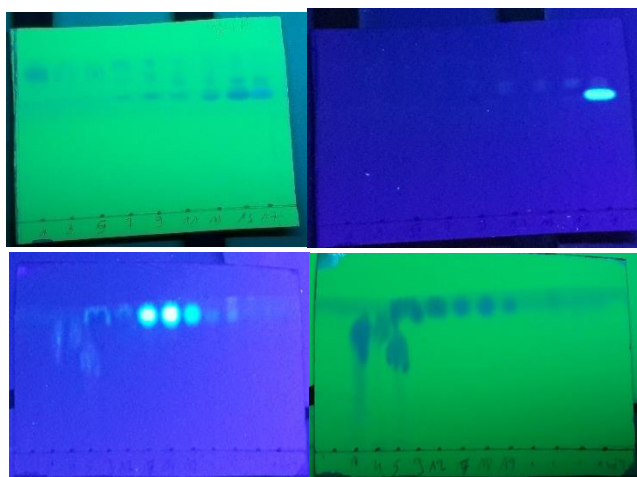


Figure 12: The TLC profile of FB fractions

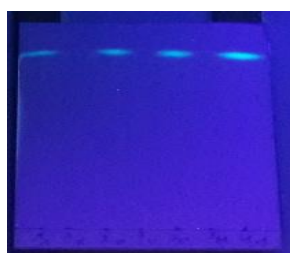


Figure 13: The TLC profile of FB1-4 M fractions.

The FB17-19 sub-fractions regrouped to **FB14** and then were separated on pasture pipette ($\phi=1\text{cm}$, $L=15\text{cm}$) with Et₂O and PE solvents (1:4; V/V) isocratic mode to obtain 18 sub-fractions.

The precipitate was purified with Sephadex® LH-20 gel as a stationary phase by CC ($\phi=1.5\text{cm}$, $L=20\text{cm}$). Mobile phase: DCM/MeOH (1:1; V/V).

C. Separation and purification of C fraction

The **C** fraction (2320.6 mg) was separated on CC ($\phi=3.5\text{cm}$, $L=80\text{cm}$).

Stationary phase: silica gel.

Mobile phase: n-Hex/DCM/acetic acid/ MeOH (Table 8).

The fractions **C1-10** were regrouping to rechromatographed.

The fractions **C17, 22, 30, 34, 75, 78, 80, 84, 99-108**, and **109** were precipitated and washed with a series of solvents (n-Hex, Et₂O, DCM, and MeOH) to isolate alkaloid compounds.

Material and Methods

Table 8: The C fractionation by CC.

Elution system (ml)	Fractions groups	Note
DCM/n-Hex/ Acetic acid		
150/150/30	C 1-45	FC1-10 reg, C17-22-30-34 precipitated
n-Hex/DCM/ AA/ MeOH		
140/150/30/10	C 46-53	Complex mixture
130/150/30/20	C 54-61	
130/150/15/25	C 62-67	
110/150/15/ 45	C 68-73	
100/150/15/55	C 74-82	C 75-78-80 precipitated
75/150/10/75	C 83-94	C 84 precipitated
50/150/10/100	C 95-108	C 99-108 precipitated
25/150/5/ 125	C 109-113	C 109 precipitated
DCM/MeOH/AA		
150/150/5	C 114-120	Complex mixture
200/50/5	C 121-129	

The fractions **C (3-10)** (751,9 mg) were regrouped **C1-10R**. The **C1-10R** was treated with 5 ml of 0.1N NH₄OH and separated on a chromatography column (ϕ = 2cm, L= 30cm).

- Stationary phase: silica gel
- Mobile phase: n-hexane/DCM/MeOH (Table 9).

The C1-10R fractionation was followed by the purification of SFC 2, which was washed with n-hexane and then DCM to get a fraction containing three separate compounds, and SFC 4 with several solvents. The SFC4 was washed to purify until it arrived at a fraction of SFC4' containing two compounds. These fractions were rechromatographed on PTLC to isolate one compound, SFC4-2, from both fractions.

Material and Methods

Table 9: The C3-10R fraction purification by CC.

Elution system %	Fractions	Group-fractions name
n-Hex		
1	1-2	SFC 1
n-Hex/DCM		
1 / 0,5	3-4	SFC 2
1/0,75	5-7	SFC 3
1/1	8	
0,75/1	9	SFC 4
0,5/1	10-11	SFC 5
0,25/1	12-13	SFC 6
DCM		
1	14-16	SFC 7
DCM/ MeOH		
1/0,5	17-25	SFC 8
1/0,75	26-34	SFC 9
1/1	35-45	SFC 10
0,75/1	46	SFC 11
0,5/1	47-49	SFC 12
0,25/1	50-51	SFC 13
MeOH		
1	52-60	SFC 14

The C' fraction was treated with NH₄OH and then purified on a silica gel by chromatography column ($\phi=3\text{cm}$, $L=60\text{cm}$) with n-hex/ DCM as mobile phase (Table 10).

Table 10: The C' fraction fractionation by CC

Elution system %	Fractions
n-Hex	
1	FC'1
n-Hex/ DCM	
1/ 25	FC'2
1/75	
1/1	FC'3
0,75/1	FC'4
0,65/1	FC'5
0,5/1	

Material and Methods

0,3/ 1	FC'6
0,25/1	FC'7,8,9
DCM	
1	
DCM/ MeOH	
1/0.25	FC'10, 11
1/0,5	FC'12,13,14
1/0,75	
1/ 1	
0,75/1	FC'15, 16
0,5/1	
0,25/1	
MeOH	
1	FC'17, 18

The FC' (7, 8, 9) were regrouping as FC'9' and precipitated with n-hexane, the precipitate separated on CC ($\phi=1.5\text{cm}$, $L=20\text{cm}$)

- Stationary phase: Sephadex ® LH-20
- Mobile phase: DCM/ MeOH (1/1, V/V) with isocratic mode.

The fractionation allowed us to obtain 17 fractions (Figure 14).

- The n-hexane layer was separated on CC ($\phi=1.5\text{cm}$, $L=20\text{cm}$).
- Stationary phase: Sephadex® LH-20
- Mobile phase: n-hexane/DCM/ MeOH (1/1, V/V) with isocratic mode to get 22 fractions.

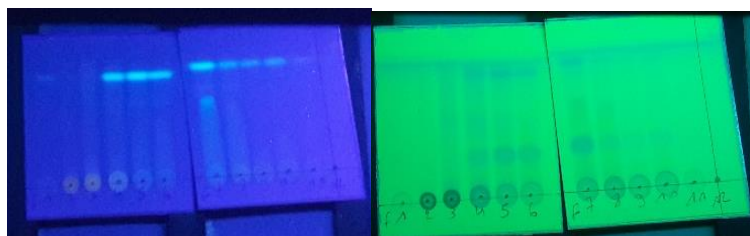


Figure 14: TLC profiels of FC'9' fractions 365/254 nm.

D. Separation and purification of D fraction

The D fraction was separated on a silica gel column ($\phi=1.5\text{cm}$, $L=20\text{cm}$) with ethyl ether **Et₂O**/ petroleum ether **PE** as a mobile phase (Table 11).

Material and Methods

Table 11: The D fraction fractionation by CC

Elution system (%)	Fractions	Group-fractions name
Et₂O/ PE		
1 /4	1-13	D1
n-Hex/Et₂O/ PE		
0,5/0,5/4	14-24	D2
1/0,5/4	25-33	D3
1,5/0,25/3	34-36	D4
1,5/0/3	37-39	
n-Hex/ PE		
2/3	40-47	D5
2,5/2,5	48-56	D6
4/1	57-67	D7
n-Hex/ PE/ DCM		
4/0,5/0,5	68-72	D8
n-Hex/DCM		
4/1	73-76	D9
3/2	77-79	
2,5/2,5	80-83	D10
2/3	84-91	D11
1/4	92	D12
n-Hex/DCM/MeOH		
0,5/ 4/0,5	93-94	
DCM/ MeOH		
4/1	95-104	
3,5/1,5	105-117	D13
2,5/2,5	118-123	D14
1,5/3,5	124-128	D15
0,5/4	129-139	D16
0/4	140-143	
MeOH/H₂O		
4/0,5	144-159	D17
4/1	160-162	D18
3/2+ Drop acid	163-170	D19

All purified fractions were chosen according to positive Dragendroff detection results and their least complication.

All fractions were collected in a tube and analyzed by TLC.

Material and Methods

The collected fractions were grouped according to their chromatographic profile similarity on thin layer plates; these were developed with different mobile phase systems, and visualized under a UV lamp at 254, 366 nm, and after Dragendorff reagent (visible, 366nm).

II.2.4.4. Preparative thin layer chromatography (PTLC)

The preparative plate was prepared with silica gel 60 HF 254 to separate the SFC 2 and 4, and the n-hexane/ DCM (1:1; V/V) was used as the mobile phase Figure 15.

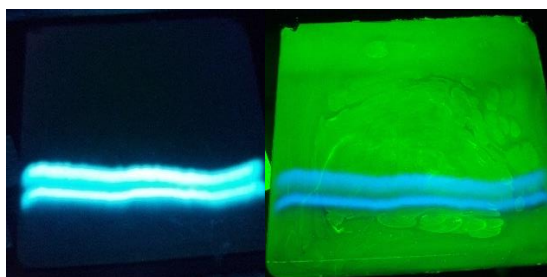


Figure 15: The PTLC profile of the SFC4 fraction

II.2.5. Identification of compounds

II.2.5.1. Gas chromatography-mass spectrum analysis GC-MS

To identify some volatile alkaloids and volatile compounds of **AVD**, **AVE**, **B1-1**, **B2**, **B3**, **B4**, and **AWAED**, GC/MS analyses were carried out. The method was performed on a Hewlett–Packard 6890N gas chromatograph equipped with fused silica capillary column DB-5MS (5% phenylmethylsiloxane, 30 m 0.25 mm, film thickness 0.25 μ m, Agilent Technologies, USA) and coupled with a 5975B mass selective detector from the same company. The injector and interface were operated at 250 and 290 C, respectively. The oven temperature was raised from 70 to 290 C at a heating rate of 5 C/min and then isothermally held for 20 min. As a carrier gas He at 1.0 mL/min was used. The samples, 1 μ L of the solutions in Et₂O (ca. 1 mg in 1 mL of Et₂O), were injected in a pulsed split mode (the flow was 1.5 mL/min for the first 0.5 min and then set to 1.0 mL/min throughout the remainder of the analysis; split ratio 40:1). Mass selective detector was operated at the ionization energy of 70 eV, in the 35–650 amu range and scanning speed of 0.34 s[60]. The Hewlett–Packard 6890N gas chromatograph equipped with fused silica capillary column DB-5MS (5% phenylmethylsiloxane, 30 m 0.25 mm, film thickness 0.25 μ m, Agilent Technologies, USA) and coupled with a 5975B mass selective detector machine from the same company was used.

Material and Methods

II.2.5.2. High-Performance Liquid Chromatography HPLC

In this work, the HPLC analysis was used to identify and quantify the chemotype of the obtained alkaloid extracts of *D. scoparia* based on some alkaloid and phenolic compounds in purified fractions and extracts according to the UV retention time of the compounds compared to the standards. The alkaloids' and phenolics' concentrations were expressed by mg/kg MS (mass sample). The HPLC instrument used was from the Shimadzu Nexera-i LC-2040C 3D Plus system coupled with a PDA detector.

A. Identification of Alkaloid compounds

The HPLC analysis of alkaloid compounds. The study was carried out on the reverse phase C₁₈ column (250*4.6 mm, 5 µm, (UP) LC column Phenomenex) at 30 °C. The mobile phase was used with gradient elution, the solvent 0.1% formic acid in water **A** and the methanol **B**: 5-15% **B** at 0.01-5min; 15-25% **B** at 5-15 min; 25-50% **B** at 15-25 min; 50-70% **B** at 25-35 min, [103].

B. Identification of phenolic compounds

For phenolic compounds, the HPLC analysis was carried out on the phenyl hexyl C₆ column (4.6*150 mm, 3 µm, GL Sciences InterSustain Made in Japan) at 30°C. The mobile phase was used with gradient elution, the solvent 0.1% formic acid in water **A** and the acetonitrile **B**: 0.01-7 min, 5-9.5% **B**; 7-20 min, 9.5-17% **B**; 20-35 min, 17-40% **B**; 35-40 min, 40-0% **B**. About with a flow rate of 1 ml.min⁻¹ while the injection volume was 10µl for both analyses, using Shimadzu Nexera-i LC-2040C 3D Plus system coupled with a PDA detector[104].

With a flow rate of 1 ml.min⁻¹ while the injection volume was 10µl for both analyses.

II.3. Biological tests

Biological tests are necessary to determine the active extract/fractions and guide the fractionation and separation process to isolate bioactive compounds. We select some tests; the Anti-oxidant assay « DPPH, BHT, and B-carotene » " anti-diabetic, sun protection factor SPF, and insecticidal test as biological tests related to human/plant disease in this work.

II.3.1. Anti-oxidant evaluation

II.3.1.1. DPPH test

The DPPH test is based on the theory that a hydrogen donor is an antioxidant. The violet-color radical DPPH•, which has a characteristic absorption band at 517 nm, accepts hydrogen released by the antioxidant to form

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DPPH. The antioxidant effect is proportional to the disappearance of the radical DPPH• and the variation of the solution from violet to yellow (Figure 16) [105].

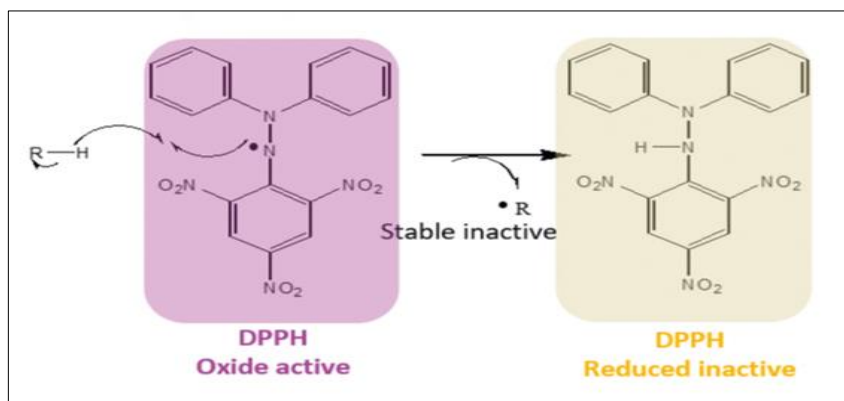


Figure 16: Antioxidative properties – DPPH [105].

The evaluation of the free radical scavenging capacity of the obtained extracts and fractions was carried out according to Blois's methods [5]. In the microplate (96-well) 160 μ l of DPPH (2,2-diphenyl-1-picrylhydrazyl) methanol solution (0.1M) was added to 40 μ l of various concentrations of each fraction; DV, and ETV. After 30 min incubation, the mixture in the darkness at ambient temperature was read at 517 nm by using the microplate reader (Perkin Elmer EnSpire, Singapore). The BHT and BHA were used as antioxidant standards and the IC₅₀ value was calculated according to the inhibition percentage determined at various fractions concentration (μ g/ml).

The formula calculated the inhibition percentage:

$$\% \text{ Inhibition} = (A_0 - A_F) / A_0 \times 100 \quad (1)$$

A₀: control absorbance

A_F: fraction absorbance.

II.3.1.2. Phenanthroline PHT

Phenanthroline activity was determined using the method of Szydłowska-Czerniaka et al., (2008)[106].

This method is based on the reduction of Fe³⁺ ion to Fe²⁺ by an antioxidant. The Fe²⁺ ion then reacted with ortho-phenanthroline into a red-orange complex. The phenanthroline method is more accurate and simpler with a good correlation with the FRAP method[107].

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(10 μ l extract and 50 μ l FeCl₃ (0.2%)) and 30 μ l phenanthroline (0.5%) and 110 μ l methanol) are mixed and incubated in the dark for 20 min at 30°C, and the optical density is read at 510 nm. BHT and BHA are used as standards.

II.3.1.3. B-carotene/ linoleic acid

The β -carotene/linoleic acid model system was applied to determine the inhibition potential of β -carotene for *D. scoparia* fractions/ extracts using the following method[108]. The β -carotene/linoleic acid emulsion was prepared by 0.5 mg of beta-carotene dissolved in 1 mL of chloroform. in a flask with 200 μ l tween 40 and 20 μ l linoleic acid, the previous mixture was added. The vacuum evaporator was used to remove chloroform, and 50 μ l of peroxide hydrogen was adjoined. The mixture absorbance was regulated between 0.8 and 0.9 at 470nm.

In the microplate (96 well) 160ul emulsion was added to 40ul of fractions / positive standard with a different concentration. the microplate was incubated for 120 min at 50°C, then the absorbance measured at t=0 and t=120 at 470 nm.

The percentage inhibition is expressed by the following equation:

$$I\% = 1 - [(AF_0 - AF_t) / (AC_0 - AC_t)] \times 100$$

I (%): percentage of inhibition

AF₀: β -carotene absorbance on fraction at t = 0.

AC₀: β -carotene absorbance on negative control at t = 0.

AF_t: β -carotene absorbance on fraction at 120 min.

AC_t: β -carotene absorbance on negative control at 120 min.

II.3.2.Enzymatic inhibition

II.3.2.1. Antidiabetic affects

To examine the inhibition potential of alpha-amylase enzyme we used the iodine/potassium iodide technique Zengin et al.,(2014)[109]. with a few adjustments. Using amylase solution in 1 U of sodium phosphate buffer SPB (6 Mm NaCl/ pH=6.9) and 25 μ L of the sample fraction at different concentrations, the reaction mixture was produced in a 96-well microplate. 50 μ l of 1% starch solution was added to start the reaction after the final solution was incubated for 10 minutes at 37 °C. Simultaneously, an enzyme solution-free control was made. After a 20-minute re-incubation period at 37°C, the reaction was halted by adding 25 μ L of 1 M HCl and 100 μ L of iodine-potassium iodide solution. At 630 nm, the absorbance was measured. The a-amylase inhibition percentage was evaluated by the following equation:

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$$I\% = 1 - [(Absc - Abse) - (Absf - Absb) / (Absc - Abse)].$$

Absf = Absorbance (fraction, Starch, Enzyme, IKI, HCl);

Absb = Absorbance (fraction, SPB, IKI);

Abse = Absorbance (fraction solvent, Enzyme, Starch, HCl, IKI);

Absc = Absorbance (fraction solvent, SPB, Starch, HCl, IKI).

II.3.2.2. Anti-Alzheimer (Anti-cholinesterase) effect

The method of Ellman et al., (1961)[110] is used to determine the inhibitory activity of acetylcholinesterase and butyrylcholinesterase.

Briefly, 150 μ l of sodium phosphate buffer (100 Mm, pH 8.0), 10 μ l of a sample solution dissolved in ethanol at different concentrations, and 20 μ l of BChE (6.85 10^{-3} U) were mixed and incubated for 15 min at 250 C, then 10 μ l of DTNB (0.5 mM) [5,5'-dithiobis (2-nitrobenzoic acid)] were added. The reaction was then initiated by the addition of 20 μ l of butyrylthiocholine chloride (0.2 mM); the galantamine was used as a reference compound. The hydrolysis of this substrate was followed by spectrophotometry by the formation of a yellow color of methyl anion 5-thio-2 nitrobenzoate, according to the result of the reaction of DTNB with thiocholine, released by enzymatic hydrolysis of butyrylthiocholine chloride at a wavelength of 412 nm, using a 96-well microplate reader[111].

The formula determines the inhibition percentage of BChE:

$$\text{The inhibition percentage of BChE (\%)} = [(SE - SS) / E] \times 100$$

-SE: the activity of the enzyme without extract

-SS: the activity of the enzyme with the extract

II.3.3. Sun Protection Factor SPF

The photoprotective effect characterized by the Sun protection factor (SPF) was assessed according to the method of Mansur, et al. (1986)[112][113]. The samples were diluted in absolute methanol to a concentration of 2 mg/mL (2000 ppm). As shown in the (Table 12), the absorbance was measured at different wavelengths, starting from 290 nm to 320 nm, every 5 nm using a multimode microplate reader (Perkin Elmer Enspire, Singapore). Each spectrophotometric measurement was performed in triplicate and the SPF value was calculated by the following formula [114]:

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$$SPF = CF \cdot \sum_{290}^{320} EE(\lambda) \cdot I(\lambda) \cdot Abs(\lambda),$$

Where *CF* is the correction factor (*CF* = 10), *EE* is the erythemal effect spectrum, *I* is the solar intensity spectrum, *Abs* is the absorbance of the sunscreen product, and ($\sum$) is a constant determined by Sayre, et al.,(2002)[26].

Table 12: The different wavelengths applicated

Wavelength λ (nm)	EE(λ)x I(λ)(Standards)
290	0,0150
295	0,0817
300	0,2874
305	0,3278
310	0,1864
315	0,0837
320	0,0180
Total	1

II.3.4. Bio-insecticidal test

Date palm scales (*Parlatoria blanchardi*) are challenging arthropod pests to manage due to their protective morphological traits against insecticides. They are immobile and remain in the crawler stage. Both adult and nymphal instars have a waxy coating that shields them from insecticides. Therefore, finding effective control methods for scale insects is crucial[115], [116]. Plant extracts contain secondary metabolites that act as repellents, feeding deterrents, and toxins, providing defense against herbivores, pests, and pathogens. Extracts and essential oils from plants, with their phytochemicals, show promise as alternative pest control methods with fewer drawbacks compared to traditional insecticides[117], [118].

Sufficient pieces of palm leaflets of Deglet Nour (*Phoenix dactelyfera* L.), (Arecaceae) equal to 2 cm from highly infested and non-infested leaves by *Parlatoria blanchardi* Targ (Hemiptera – Diaspididae) were prepared for the treatment; each piece was placed under the binocular magnifier to count the number of living individuals. After that, 3 pieces of leaflets were put in glass Petri dishes that contained soaked cotton with distilled water to keep them moist. Effectively, each sample was uniformly sprayed (1 ml/dish) on the entire surface of the leaflets and each treatment was replicated 3 times for five applied

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concentrations with Acetone as control. As for the incubation, it was at 30 °C and lasted 24 hours.

After 24 h, a second count was made to determine the mortality rate[119].

$$\text{Percentage of mortality} = (\text{number of dead white scale individuals after treatment} / \text{total number of white scale before treatment}) \times 100$$

To valid this test, the corrected mortality percentage must be calculated to value the observed mortality by using Abbott formula[120], [121], [122].

$$\text{Percent Corrected Mortality} = (\text{Percent Observed Mortality} - \text{Percent Control Mortality} / 100 - \text{Percent Control}) \times 100$$

A mathematical transformation of the values performed on the percentages of mortality (Probits); the dose-response curve is a straight line from which the LD₅₀ and LD₉₉ value is derived[123][123]

Material and Methods

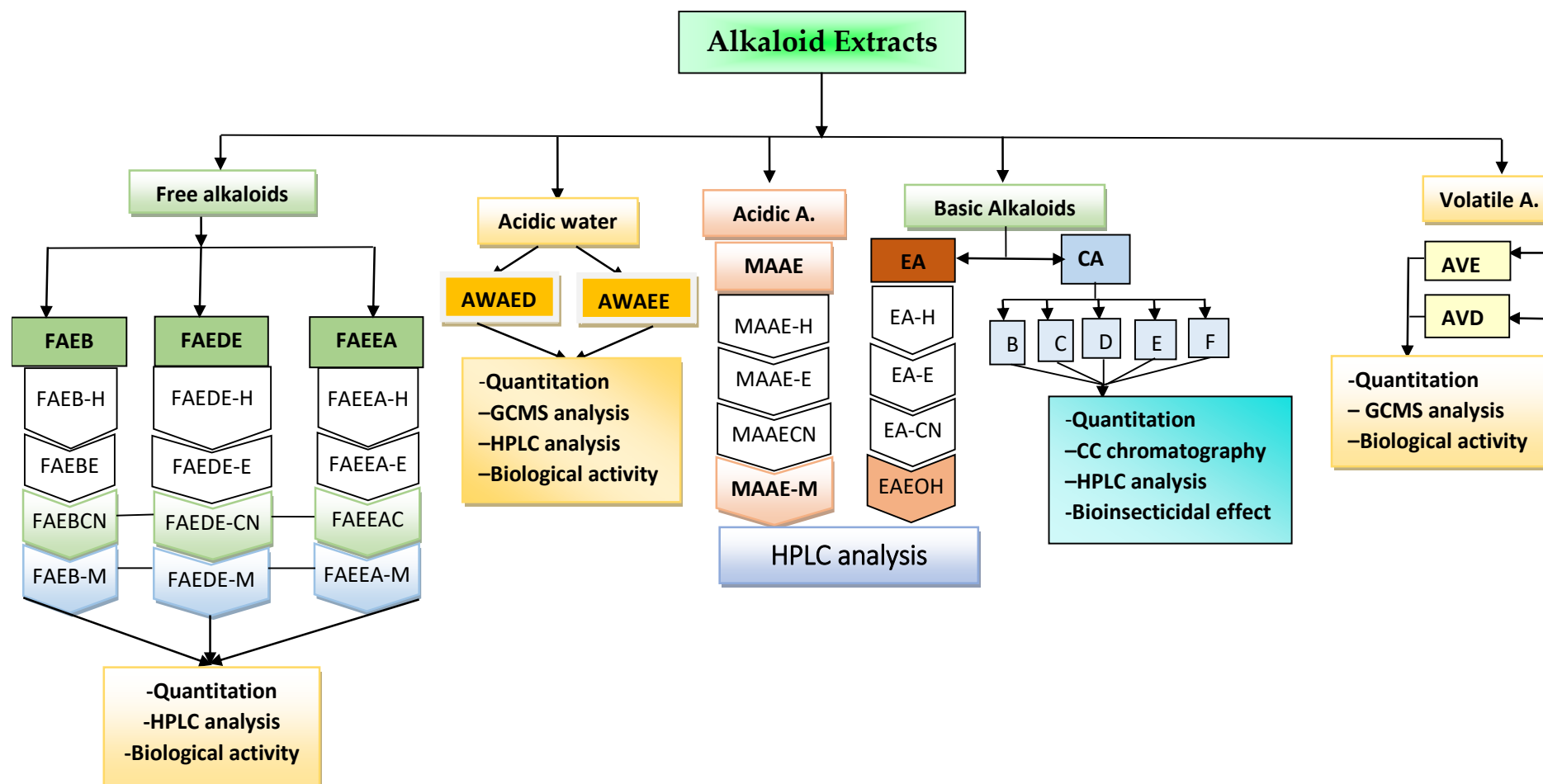


Figure 17: The experimental regrouping work diagram

III. Results and discussion

III.1. Extraction and quantification

The extraction of alkaloid compounds from the aerial parts of *Deverra scoparia* Coss. & Dur. was carried out in four methods. Firstly, the Clevenger was used to obtain the extract of volatile alkaloids extract. The extract was split into two DV and ETV containing nitrogen (amine) and volatile alkaloids. The second is the extraction of alkaloids in the basic medium by Soxhlet with a non-polar solvent to extract the basic alkaloids, followed by extraction with ethanol, as well as acid reflux extraction in methanol to extract the acidic alkaloids. Finally, the extraction by maceration with different polarity gradient solvents (for obtaining free alkaloids) followed by fractionation. This series of extractions and global fractionation made it possible to obtain eleven alkaloid extracts/fractions: **AV** (AVD, AVE), **CA**, **EA**, **MAAE**, **AWAEE**, **AWAED**, **FAEH**, **FAEDE**, **FAEA** and **FAEB** (Figure 17). These extractions were investigated for the first time on this plant in our knowledge in this study.

Applying several selective methods to extract alkaloids allowed us to obtain extraction yields varying from 0.0078 to 11.46 %, where the highest yield was for the basic alkaloid ethanolic extract **EA** followed by **FAEDE** extract, while the lowest was for the volatile alkaloid fraction **AVD** (Figure 18). The highest yield is at the level of the ethanolic extract noted as a polar solvent. On the other hand, this yield is lower at the level of volatile extracts as a non-polar solvent. These results agree with those obtained in the bibliography, which indicates that polar solvents have higher extraction yields compared to non-polar solvents[90].

Results and Discussion

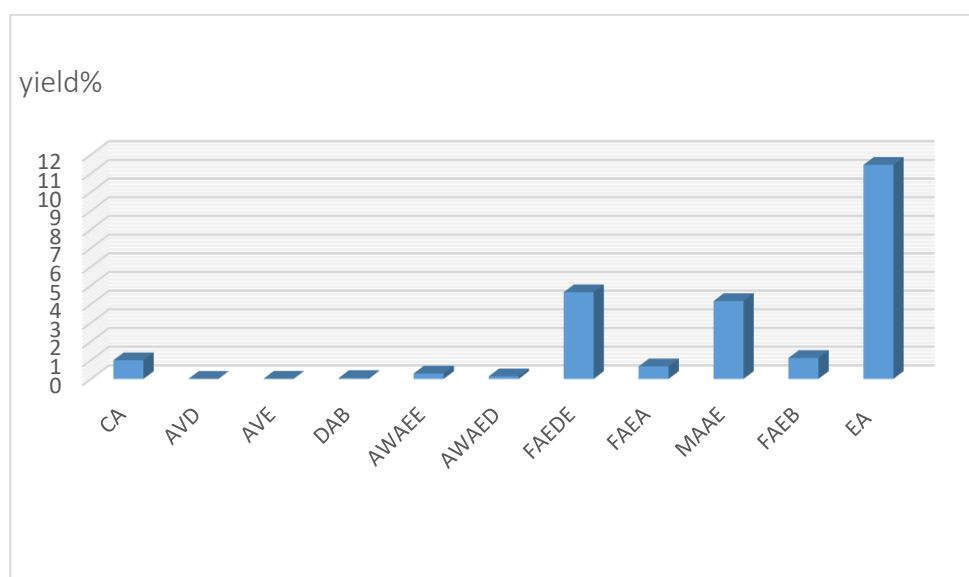


Figure 18: The extraction yield of alkaloid type in different solvent/ medium

AVD; alkaloid volatile DCM fraction/ **AVE;** alkaloid volatile Et₂O fraction/ **AWAEE;** acidic water alkaloid extract Et₂O fraction/ **AWAED;** acidic water alkaloid extract DCM fraction/ **CA;** crude alkaloid extract/ **EA;** ethanol alkaloid extract/ **FAEDE;** free alkaloid extract with DCM-EtOH/ **FAEA;** free alkaloid extract with ACoET/ **FAEB;** free alkaloid extract with BuOH and **MAAE;** acidic alkaloid extract with MeOH.

The choice of a solvent for the extraction of plant material must respect several criteria, including low toxicity, and ease of evaporation at low temperatures. The choice also depends on the target compounds to be extracted[124], [125].

In general, the variability of yields obtained for the different extracts/fractions can be related to several factors, namely, the stage of development of the plant, its geographical origin, the duration of storage of plant material, the method of drying, the extraction methods applied and the solvents used[126], [127].

The quantity of total alkaloids in the different extracts is evaluated by the Bromocresol method by using the atropine as a quantitative equivalence standard (Figure 19). The studied extractions/ fraction showed the presence of alkaloid content with a remarkable concentration difference. The volatile AV extract showed a better richness in alkaloids (62895.83 mg AE/kg MS) than the other extracts with an observed difference (Figure20). $AVD > FAEA > AVE > FAEDE > MAAE > CA > AWAEE > AWAED > FAEB$. The alkaloid content of *D. scoparia* plant extracts were determined for the first time in this study, as there is no previous study that determined the amount of alkaloids in this species. However, Niko Radulovic et al., recorded that *Conium maculatum* L., (Apiaceae) plant contains volatile alkaloid compounds[41].

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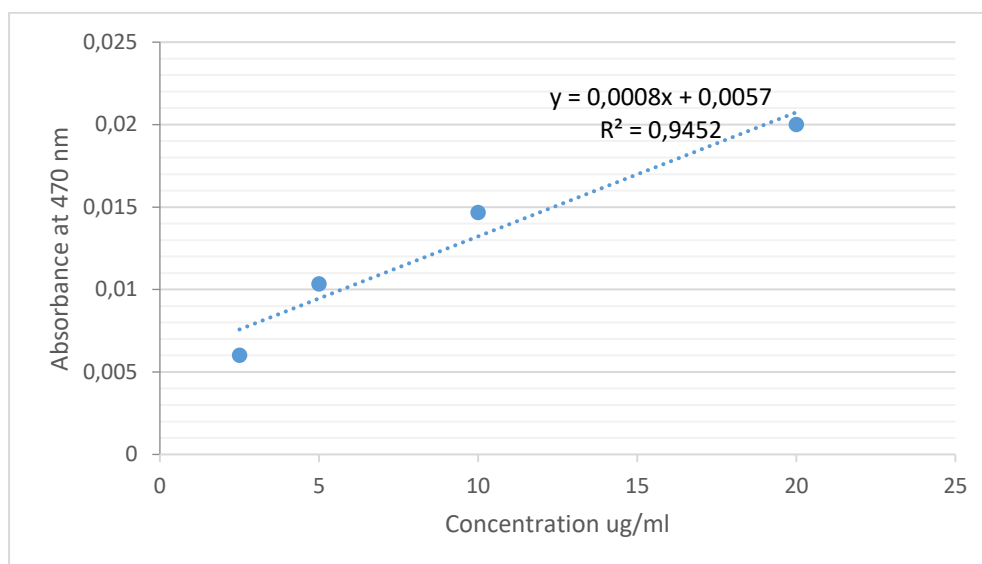


Figure 19: Calibration right of Atropine

Through the results of the extraction yield and the estimation of the alkaloid content of the extracts, we note that the extract with the lowest yield recorded the greatest value for the alkaloid content. This can be explained by the selectivity of the extraction, as well as the fact that most of the plant's alkaloid compounds are volatile. These results agree with those obtained in the bibliographies which indicate that the aerial parts of the plant accumulate the most alkaloids[128], [129], [130]. The quantitative study of alkaloids by Adele Murauer et al., [131] confirms the low content of tertiary alkaloids in the parts not chlorophylls, compared to the chlorophyll parts containing a quantity at least ten times greater.

These variations may be justified by the differential distribution of secondary metabolites, due to genotype factors that control the accumulation of these compounds in the plant[131]. The extraction method adopted, the specific reagents used, and the solubility of the extraction solvent can have an impact on the total alkaloid yield[132], [133], [134]. Moreover, this yield depends, in particular, on the variety, the harvest season, the geographical location, the different diseases that can affect the plant, the maturity of the plant, and the organs studied[135], [136], as well as the manner and duration of conservation[137], [138]. According to that, it is difficult to compare our results with those of the bibliography. Briefly, the extract yield varies according to the method and the conditions in which the extraction was carried out, the species, and the geographical origin of the plant, the conditions and duration of storage, and the harvest period.

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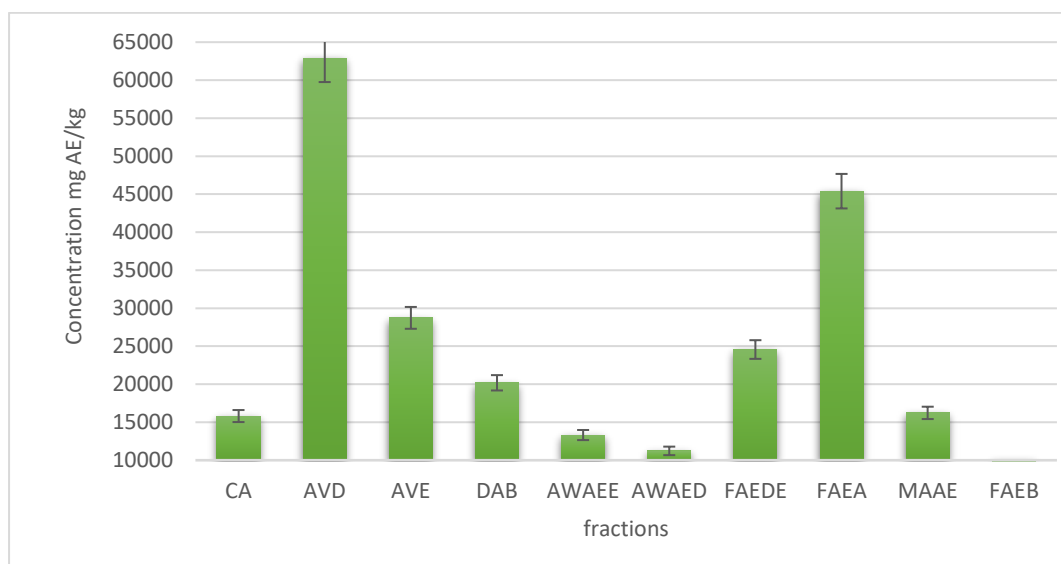


Figure 20: The total alkaloid content in the different extracts of *D. scoparia*

AVD; alkaloid volatile DCM fraction/ **AVE;** alkaloid volatile Et₂O fraction/ **AWAEE;** acidic water alkaloid extract Et₂O fraction/ **AWAED;** acidic water alkaloid extract DCM fraction/ **CA;** crude alkaloid extract/ **EA;** ethanol alkaloid extract/ **FAEDE;** free alkaloid extract with DCM-EtOH/ **FAEA;** free alkaloid extract with ACoET/ **FAEB;** free alkaloid extract with BuOH and **MAAE;** acidic alkaloid extract with MeOH.

III.2. Fractionation of basic alkaloid extract CA

A first fractionation of the basic alkaloid extract of *D. scoparia* was performed by adsorption chromatography on a silica gel column with pressure to give five fractions. This is followed by the fractionation of fractions; B, C, C' and D on an open gel silica column chromatography (Figure 21).

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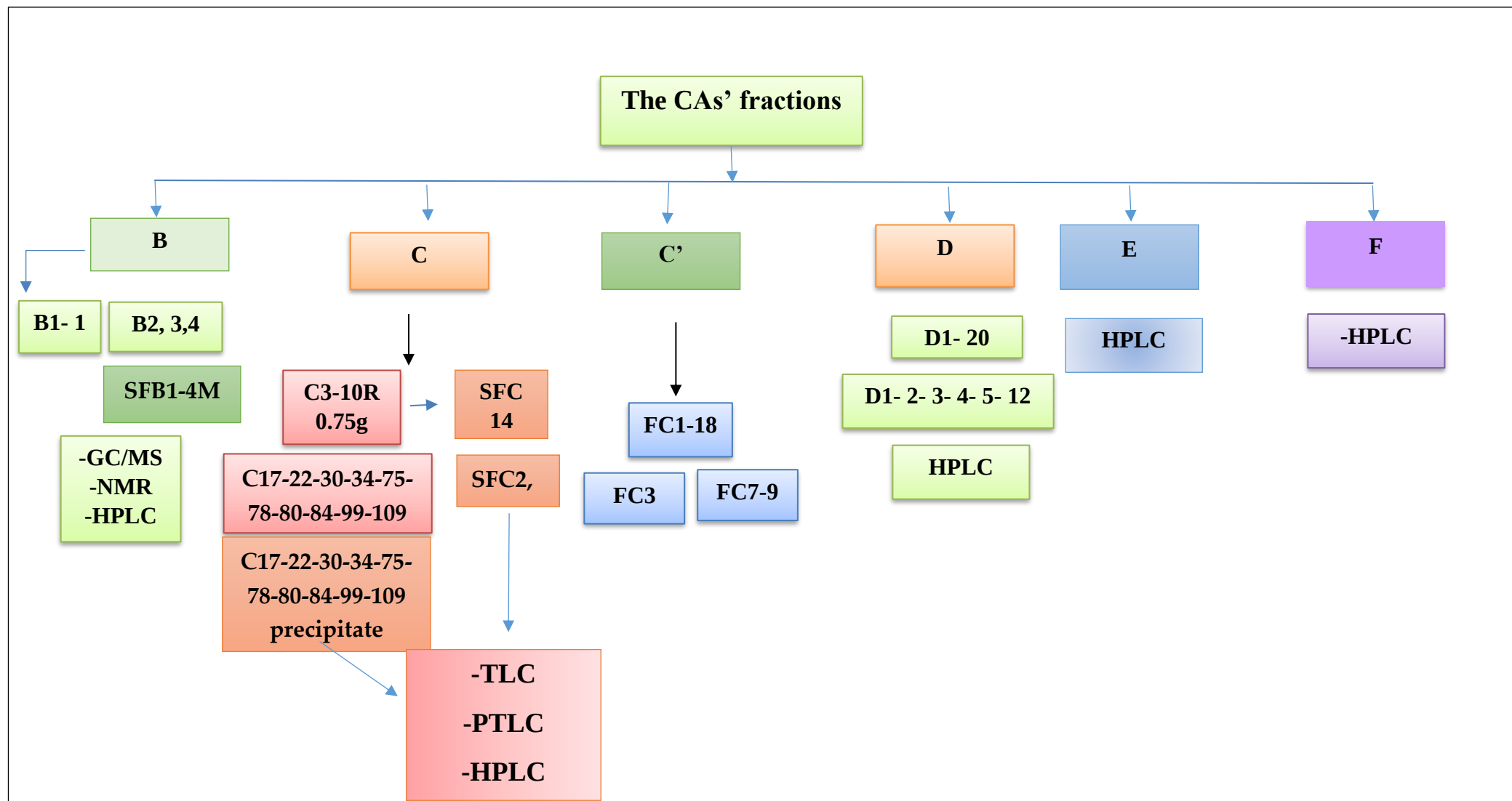


Figure 21: Fractions Diagram resume fractionation and analyses selected

Resultas and Discussion

III.2.1. Qualitative screening

The separation of the CA extract gives five fractions (B, C, D, E, and F). A preliminary phytochemical screening illustrates the presence of alkaloids and polyphenols in all fractions, the absence of flavonoids in B fraction, and the absence of tannins in all fractions (Table 13). The difference in the chemical composition of the studied plant, and of the same plants in another region explains that factors influence the presence, absence, and distribution of the different active principles such as the natural condition, the extraction methods, and their solubilization in different solvents.

Table 13: Phytochemical screening of CA fractions' results

	CA	B	C	D	E	F
<i>Flavonoids</i>	+	-	+	+	+	+
<i>Tannins</i>	-	-	-	-	-	-
<i>Polyphenols</i>	+	+	+	+	+	+
<i>Alkaloids</i>	+	+	+	+	+	+

+: presence, - Absence

III.2.2. Quantification of total alkaloid in CA's fractions

The quantification of the alkaloid content in five fractions obtained from basic alkaloid extract illustrates that fractions D, E, and F had a high concentration of alkaloid content, with the highest concentration in the E fraction. The CA extract, C, D, E, and F fractions have a remarkable concentration, while the B fraction registered the lowest concentration. (Figure 22).

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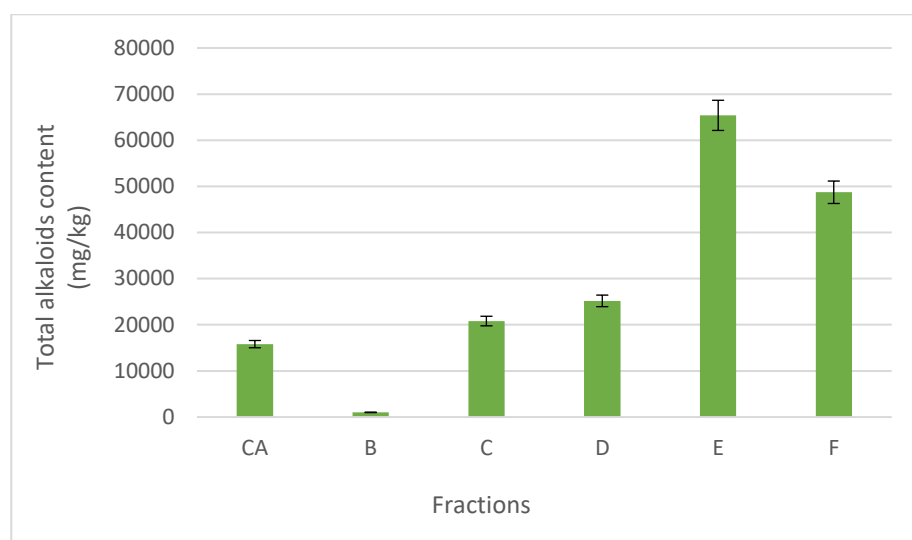


Figure 22: The total alkaloid content in CAs' fractions

III.3. Identification of alkaloid compounds

III.3.1. Volatile alkaloids/ nitrogen compounds

To identify the volatile compounds containing alkaloids and nitrogen compounds contained in the different fractions; volatile, basic, and acidic we applied several extraction and identification methods.

III.3.1.1 In fractions' volatile alkaloid extract (AVD, AVE)

The TLC profile of the AVD and AVE fractions revealed the presence of alkaloid by Dragendorff reagent response, an orange color was obtained in both fractions (Figure 23).

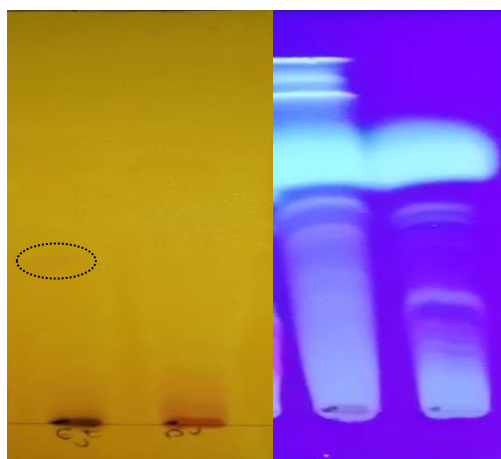


Figure 23: TLC profile of AVE and AVD fractions revealed with dragendroff reagent "QC"

Results and Discussion

On the other hand, the GC-MS analysis results show a variation in the chemical composition of AVD and AVE; Forty-two compounds were identified: 25 for AVD and 17 for AVE. The AVD fraction provided 5 major chemical families compared to 4 families in AVE. The oxygenated monoterpenes (11.77%, 17.88%), non-terpenes/ fatty acid (79.67%, 73.34%), and nitrogen compounds (NC) (2.02%, 0.6%) were similarly identified in AVD and AVE in order, additionally of the sesterterpenes and sesquiterpenes specifically in AVD (Figure26), while the monoterpenes were identified only in AVE (Figure26).

The AVD fraction showed the high amount of NC 2.02% with nine compounds such as dimethylamino-3-phenylpropanoate(**1D**), 1H-Indole, 3-methyl-2-propanoyl (**2D**), Bicyclo[2.2.1]heptane-2-acetamide, N-(1,3-dihydro-5,6-dimethoxy-3-oxo-4-is(**3D**), 2H-Pyran-2-acetamide, tetrahydro-.alpha.-hydroxy-2-methoxy-5,6-dimethyl-4- (**4D**), Deacetylvindoline (**5D**), 2-amino-6-ethyl-4,5,6,7-tetrahydro-benzo[b]thiophene-3-carboxylic acid ethyl ester (**6D**), 1-(3-methyl butyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1H-Pyrazole (**7D**), N-[2-[[[5-[(dimethylamino)methyl]-2-furanyl]methyl]thio]ethyl]-N-methyl-1,1,2-Ethenetriamine (**8D**), 2-(tetrahydrofuran-2-yl)piperidine (**9D**). Compared to AVE with 0.6% of NC with two compounds 1-Decyloxymethyl-3-methyl-1,3-dihydrobenzoimidazol-2-ylideneamine(**1E**), Triethanolamine (Sterolamide) (**2E**)(Figure24, 25, Table 14). These results confirmed the observed TLC analysis results with the positive Dragendorff response of the AVs' fractions related to the presence of nitrogen compounds.

It's important to notice that the chemotype of AV's fractions showed the presence of one similar compound Myristicin, which is the main compound in AVE (13.58%), while Apiol is the main compound in AVD (45.01%).

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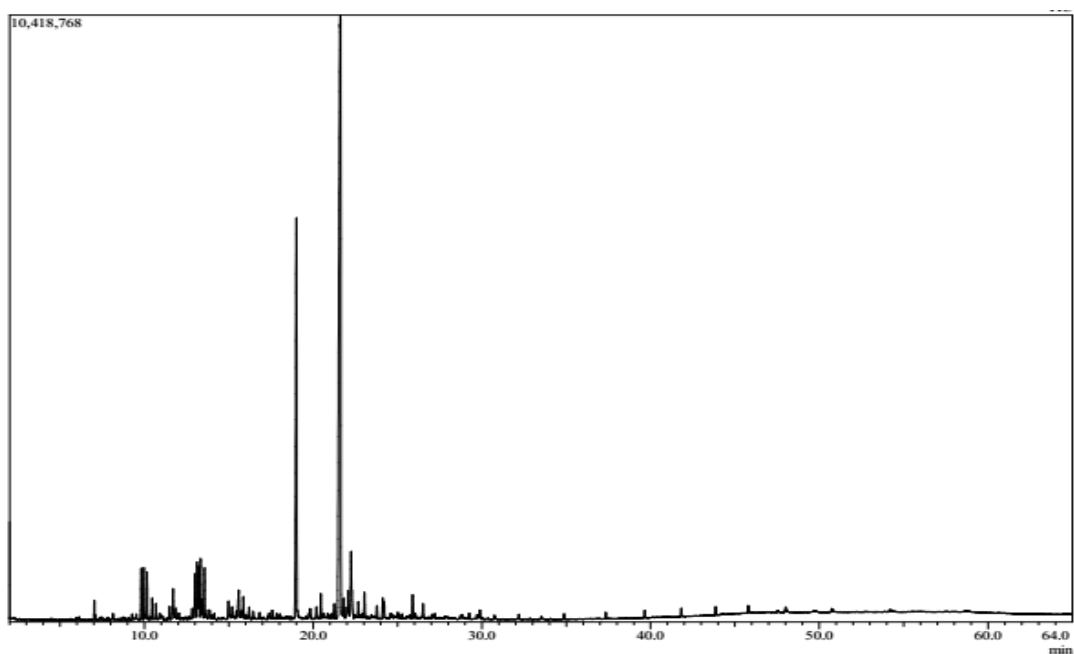


Figure 24: GC-MS chromatogram of AVD fraction

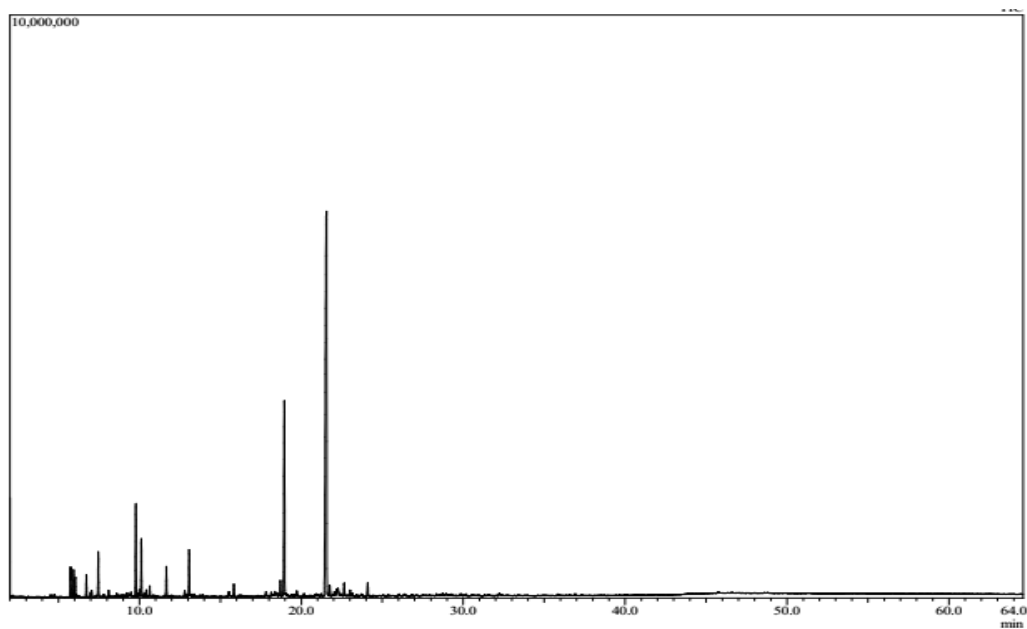


Figure 25: GC-MS Chromatogram of AVE fraction

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Table 14: GC-MS identified compounds results of AVD and AVE analyses

R.T	Name	(A%)	
		AVD	AVE
5.73	Isocineole	-	1.40
5.77	α -Terpinene	-	1.21
5.94	o-Cymol	-	1.26
6.04	D-Limonene	-	0.92
6.72	δ -Terpinene	-	1.03
7.46	p-Cymenene	-	2.29
9.77	L-4-terpineneol	-	5.03
9.80	L-terpinen-4-ol	1.12	-
9.97	p-cymenol	1.11	-
10.11	(+)-Fenchol	-	3.10
10.14	(1S)-1,3,3-trimethylnorbornan-2-ol	1.04	-
10.42	(+)-Sabinol	-	0.36
10.63	l-Verbenone	-	0.59
11.66	(+)-Carvotanacetone	-	1.68
11.69	p-Menth-4-en-3-one	0.97	-
12.99	(Z)-6-Methyl-2-(nonadec-10-en-1-yl)-2H-pyran-4(3H)-one	1.40	-
13.07	Carvacrol	-	2.53
13.11	o-Thymol	1.31	-
13.21	trans- β -Dihydroterpineol	1.35	-
13.55	2-hydroxy-1,8-cineol	2.61	-
15.09	2-(tetrahydrofuran-2-yl) piperidine (A)	0.30	-
15.53	Triethanolamine (Sterolamide)	-	0.34

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15.58	(+)-Pinanediol	1.09	-
15.83	1,2-dimethoxy-4-(2-propenyl) Benzene	-	0.67
18.70	Butylated Hydroxytoluene	-	0.97
18.95	Myristicin	14.35	13.58
21.54	Apiole	35.01	45.01
21.58	1,2,3b,6,7,8-hexahydro-Cyclopenta [1,3] cyclopropa[1,2] cyclohepten-3(3aH)-one	23.51	-
21.61	4,7-dimethoxy-5-(2-propenyl)-1,3-Benzodioxole	12.88	-
22.24	(+)-Rosifoliol	2.67	-
22.60	N-[2-[[[5-[(dimethylamino)methyl]-2-furanyl]methyl]thio]ethyl]-N-methyl-1,1,2-Ethenetriamine (B)	0.08	-
24.12	(E)-3-Butylidene-4,5-dihydroisobenzofuran-1(3H)-one	0.60	-
24.20	Alloaromadendrane-4 β ,10 α -diol	0.46	-
26.51	dimethylamino-3-phenylpropanoate (C)	0.56	-
27.15	3-methyl-2-propanoyl-1H-Indole (D)	0.12	-
30.35	N-(1,3-dihydro-5,6-dimethoxy-3-oxo-4-isobenzofuranyl) Bicyclo[2.2.1]heptane-2-acetamide (C)	0.08	-
36.92	1-Decyloxymethyl-3-methyl-1,3-dihydrobenzoimidazol-2-ylideneamine (C)	-	0.15
43.10	tetrahydro-.α.-hydroxy-2-methoxy-5,6-dimethyl-4-2H-Pyran-2-acetamide (C)	0.04	-
44.55	Deacetylvindoline (E)	0.07	-
46.86	2-amino-6-ethyl-4,5,6,7-tetrahydro-benzo[b]thiophene-3-carboxylic acid ethyl ester (C)	0.06	-

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47.35	1-(3-methylbutyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)- 1H-Pyrazole (F)	0.06	-
Total		98.84	37,11
A: True alkaloids, B: Amine, C: Aminoacid, D: Indole alkaloids, E: Monoterpene indole alkaloid, F: Amine-pyrazoles			

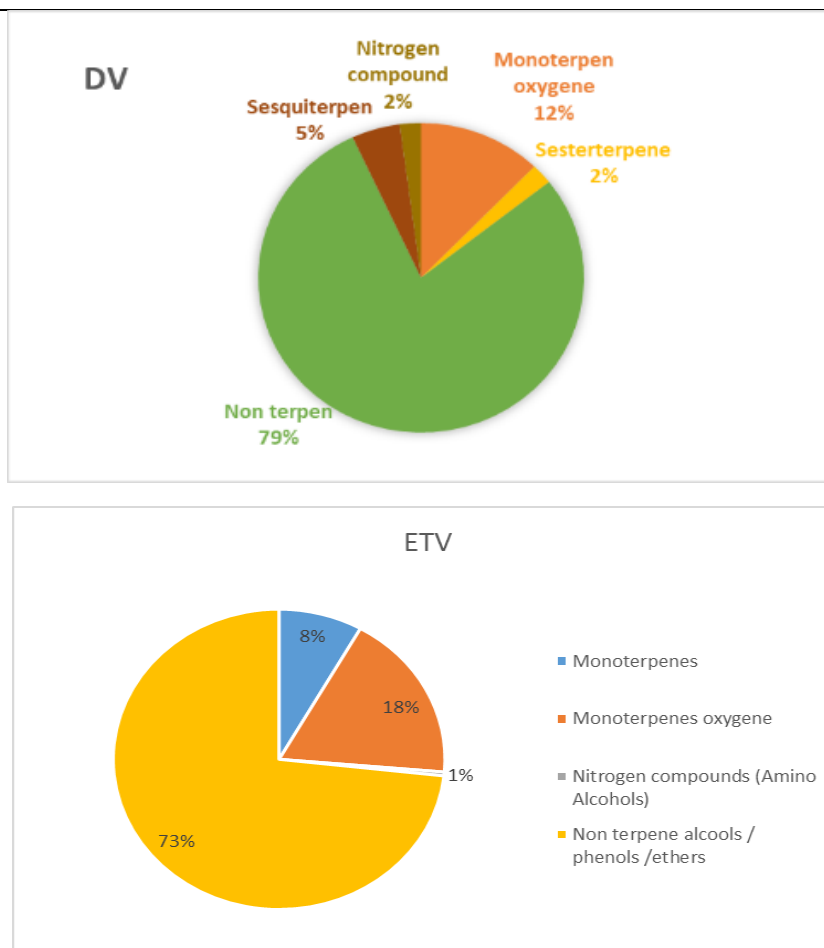


Figure 26: Volatile chemical families present in AVD and AVE fractions

III.3.1.2. Identification in fractions' basic alkaloid extract B2, B3 and B4

The TLC profile of **B** sub-fractions illustrates the presence of nitrogen compounds detected with dragendroff reagent for alkaloid detection, and Ninhydrin reagent for detecting amino acids and biogenic amines (Figure 27).

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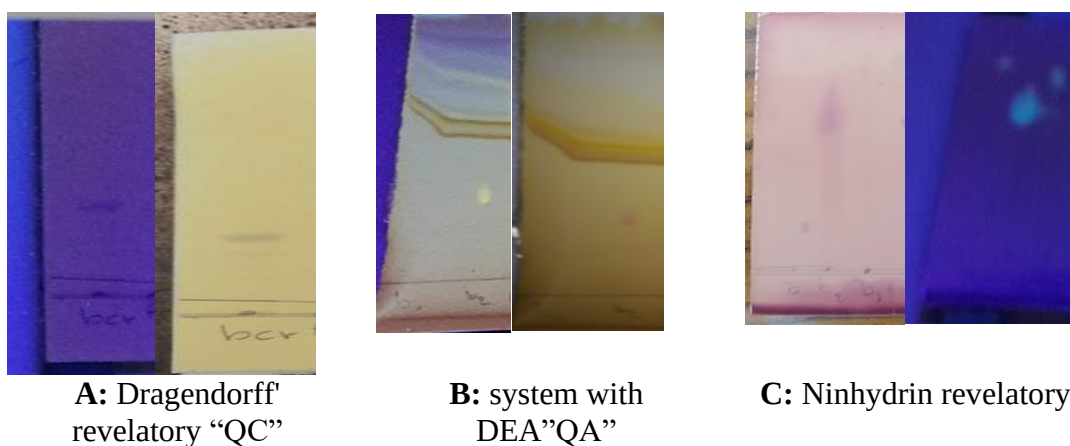


Figure 27: TLC profile of B2 with different detection methods

Identifying B2, B3, and B4 fractions by a GC-MS analysis (Figure 28- 30) gives 28 compounds with (B2; 5 compounds, B3; 13 compounds, and B4; 9 compounds) (Table 15).

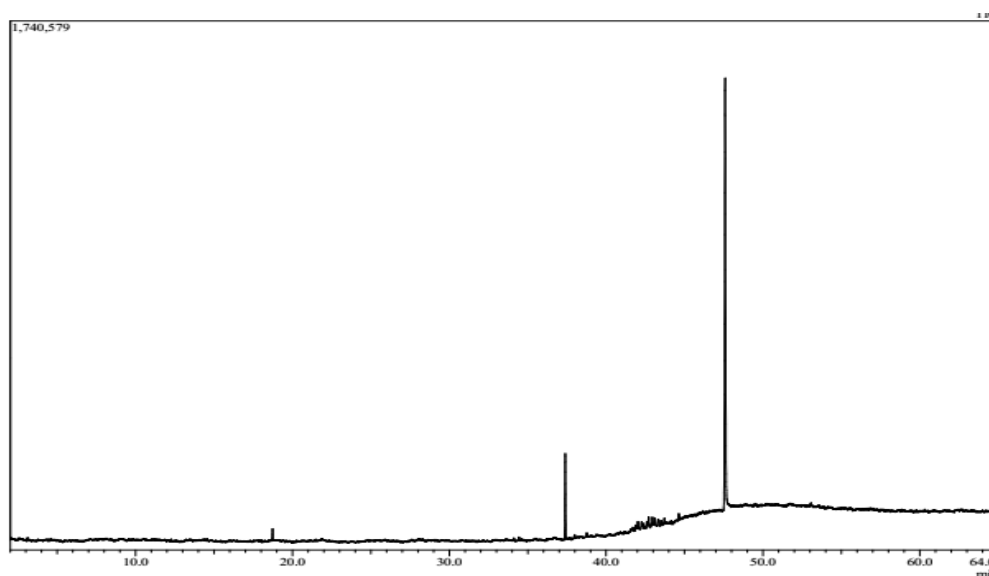


Figure 28: GC-MS Chromatogram of B2 fraction

Among the five identified compounds on the B2 fraction; two were nitrogen volatile compounds (N-phenyl Acetamide/ N, N-dimethylhexadecanamide) 0.24% (Figure 31, Table 15).

Ten nitrogen volatile compounds among thirteen compounds were identified in B3 fractions (4-(4-chloro-5-methyl-3-nitropyrazol-1-yl) Butyric acid/ 2-[(2,4-Dimethoxybenzylidene)hydrazino]-4-morpholino-6-(1-pyrrolidinyl)-1,3,5-triazine/ N-(3-acetylaminomethyl-5,5,6 trimethylbicyclo[2.2.1]hept-2-en-2-yl) Acetamide/ N2-[(2,4-dichloro phenoxy)acetyl]- 1-Lysine/ N-(1H-1,3-benzimidazol-2-ylmethyl)-4-iodo-1H-Pyrazole-1-acetamide/ N-[4-[1-(4 methyl

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phenyl)ethylidenehydrazinocarbonyl]phenyl] Hexanamide/ N-[[2-Oxo-1,2'-bi(tricyclo[3.3.1.1[3,7]]decan)-4'-yl]methyl]acetamide/ N₄,N₄-diethyl-2-methyl-1,4-Benzenediamine / 1-decyl-1H-imidazole-2-methanol/ 4-acetyl-5-(2-fluorophenyl)-1,5-dihydro-3-hydroxy-1-methyl-2H-Pyrrol-2-one.

The identified compounds were classified as amino acids such as N²-[(2,4-dichlorophenoxy)acetyl]- L-Lysine, and biogenic amine compounds (Figure 31, Table 15).

Among 9 identified compounds in B4 one nitrogen volatile compound was identified as a volatile alkaloid "[(2-fluorophenyl)methyl] **1H-Purin-6-amine**". (Figure 31, Table 15).

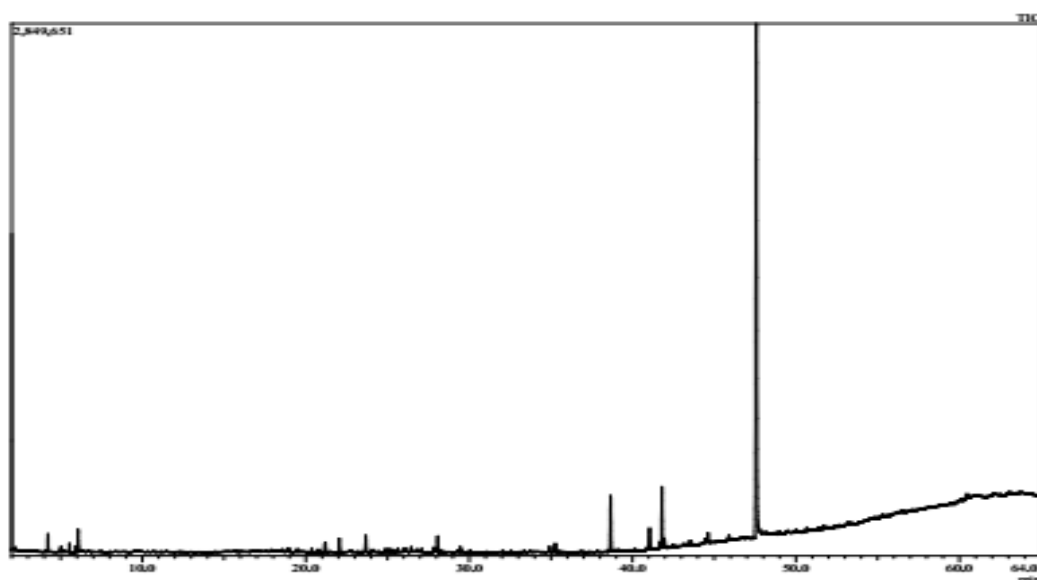


Figure 29: GC-MS Chromatogram of B3 fraction

Resultas and Discussion

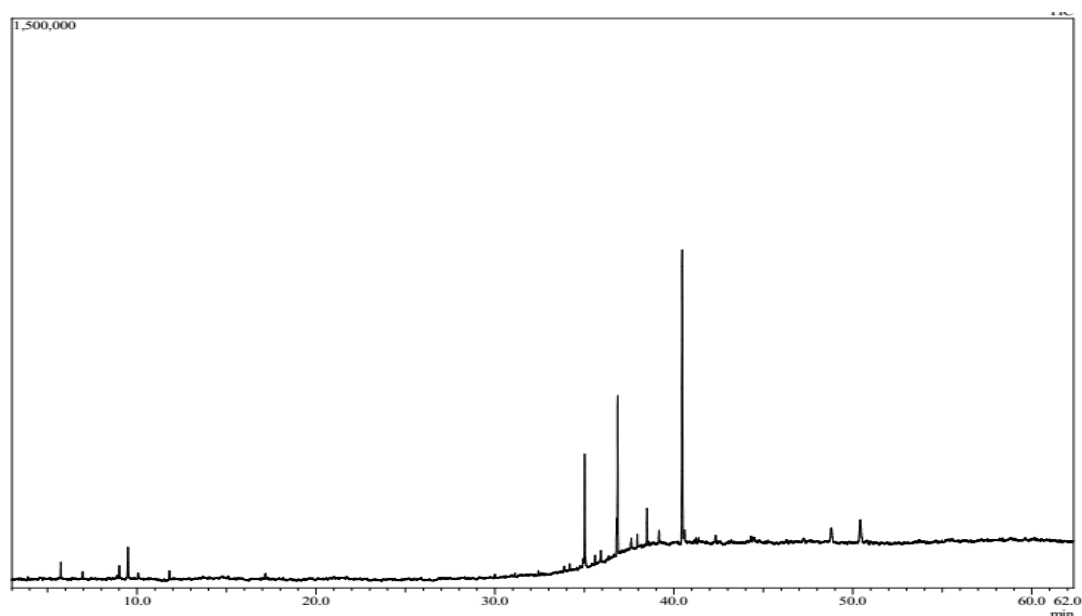


Figure 30: GC-MS chromatogram of B4 fraction

Table 15: GC-MS identified compounds results of B2, B3 and B4 analyses

R.T	Name	A%		
		B2	B3	B4
4.22	(-)- α -pinene		0.83	
6.06	D-Limonene		1.41	
6.69	N-phenyl Acetamid (Acetanilide) (G)	0.10		
8.38	4-(4-chloro-5-methyl-3-nitropyrazol-1-yl) Butyric acid (F)		0.18	
9.49	2,4-di-tert-butyl phenol			4.01
10.94	2-[(2,4-Dimethoxybenzylidene)hydrazino]-4-morpholino-6-(1-pyrrolidiny)-1,3,5-triazine (B)		0.17	
16.25	N-(3-acetylaminoethyl-5,5,6-trimethylbicyclo[2.2.1]hept-2-en-2-yl)Acetamide (C)		0.14	
18.17	N2-[(2,4-dichloro phenoxy)acetyl]-l-Lysine (C)		0.14	
18.73	Butylated Hydroxytoluene	2.09		

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22.68	N-(1H-1,3-benzimidazol-2-ylmethyl)-4-iodo-1H-Pyrazole-1-acetamide (F)		0.14	
24.44	N-[4-[1-(4-methyl phenyl)ethylidenehydrazinocarbonyl]phenyl] Hexanamide (G)		0.13	
28.06	Hexadecanoic acid, methyl ester		1.15	
34.57	N, N-dimethylhexadecanamide (G)	0.10		
34.94	Tetracosane			1.05
35.27	N-[[2-Oxo-1,2'-bi(tricyclo[3.3.1.1[3,7]]decan)-4'-yl]methyl]acetamide (G)		1.3	
36.79	Hexatriacontane			3.75
37.39	2,2'-methylenebis[6-(1,1-dimethyl ethyl)-4-methyl phenol]	11.23		
38.65	Octan-2-yl palmitate		3.16	10.16
41.04	Squalane		2.17	
41.80	Octadecanoic acid, octyl ester		4.02	
44.45	[(2-fluorophenyl)methyl] 1H-Purin-6-amine (H)			1.02
44.61	Tetrapentacontane		1.22	3.59
44.79	N4,N4-diethyl-2-methyl-1,4-Benzenediamine		0.13	
45.06	1-decyl-1H-imidazole-2-methanol (I)		0.17	
45.32	4-acetyl-5-(2-fluorophenyl)-1,5-dihydro-3-hydroxy-1-methyl-2H-Pyrrol-2-one (J)		0.14	
40.48-47.57	Nonacosan-10-one (Ginnone)	71.83	51.83	36.70
48.78	Phytyl stearate			3.81
50.41	Benzenepropanoic acid, 3,5-bis(1,1-dimethyl ethyl)-4-hydroxy octadecyl ester			5.97
Total		85.35	68.43	83.3

Resultas and Discussion

B: Amine, **C:** Aminoacid, **D:** Indole alkaloids, **E:** Monoterpene indole alkaloid, **F:** Amine-pyrazoles, **G:** Acetamide, **H:** Purine alkaloids, **I:** imidazole alkaloid, **J:** Pyrrole alkaloid.

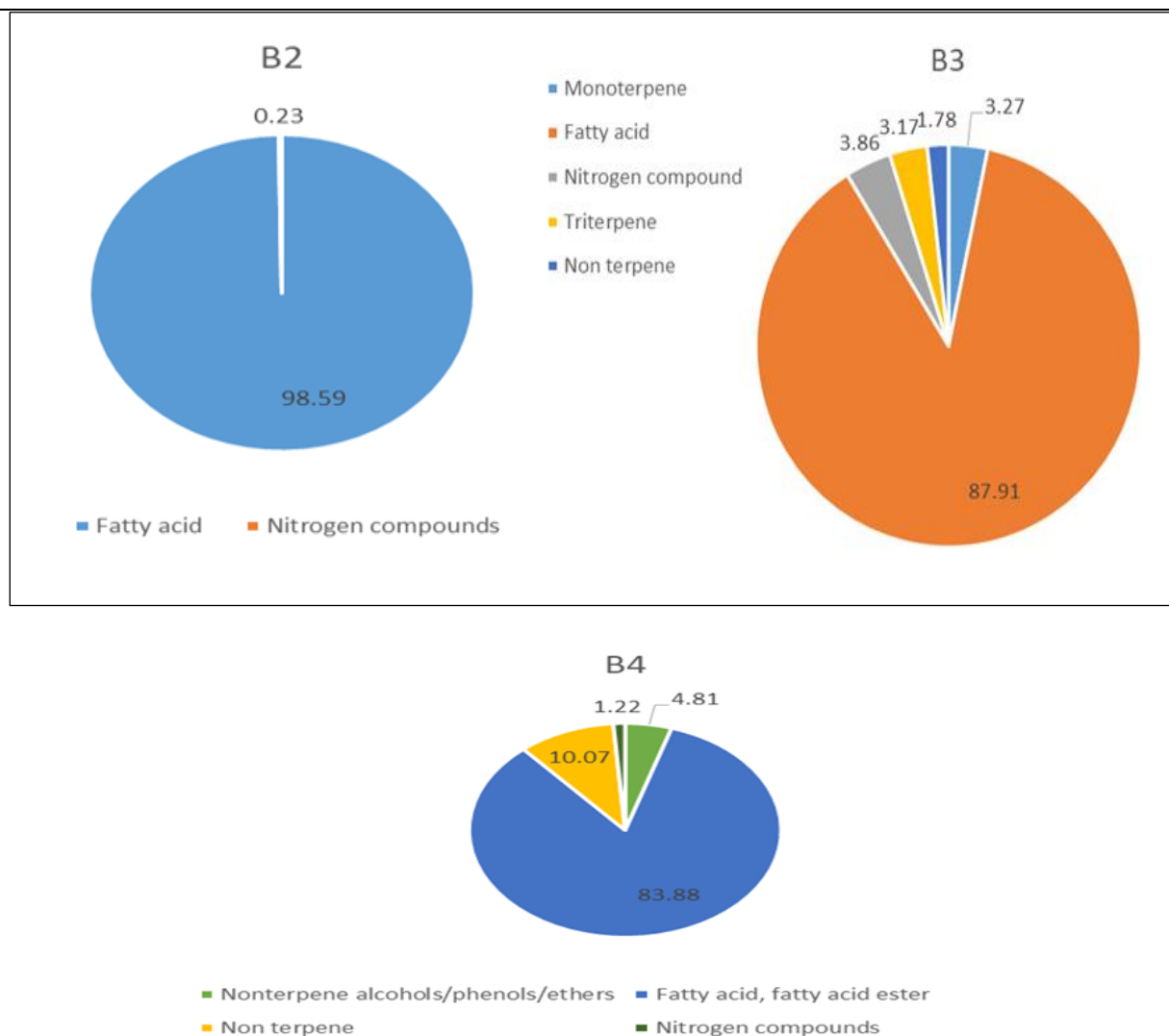


Figure 31: Chemical families classes identified in B2, B3 and B4 fractions

III.3.1.2. Identification in fractions' Acidic water alkaloid extract AWAED

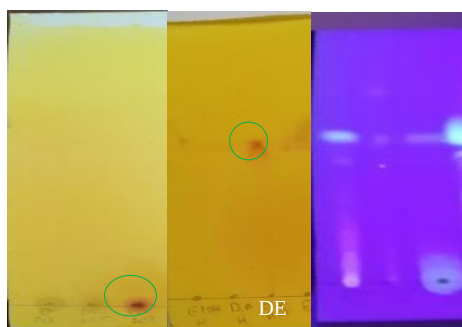


Figure 32: TLC profiles of AWAED-CN fraction with different elution "QC"

Resultas and Discussion

The TLC analysis of AWAED-CN fraction shows the presence of alkaloid compounds with intense response (Figure 32), this result was confirmed by the GCMS analysis of fraction, which is illustrated that fraction contain volatile alkaloids with an amine compound Sterolamide was identified as a main compound (Figure 33). Among 25 identified compounds, six compounds; sterolamide, 2-Isopropylpiperazine, m-nitrophenethyl acetate, 2-Hydroxy-2-(4-methoxy-phenyl)-N-methyl-acetamide, 7-[2,6-Dichlorobenzyl]-4-chloropyrrolo[2,3-d]pyrimidine, and 4,5,5-Trimethyl-2,4-diphenyl-2-oxazoline were identified as a volatile nitrogen compounds (Table 16).

It is important to mention that this is the first time for the species *D. scoparia* that Sterol amide has been identified and found as the majority component in this fraction with the five other volatile nitrogen compounds identified.

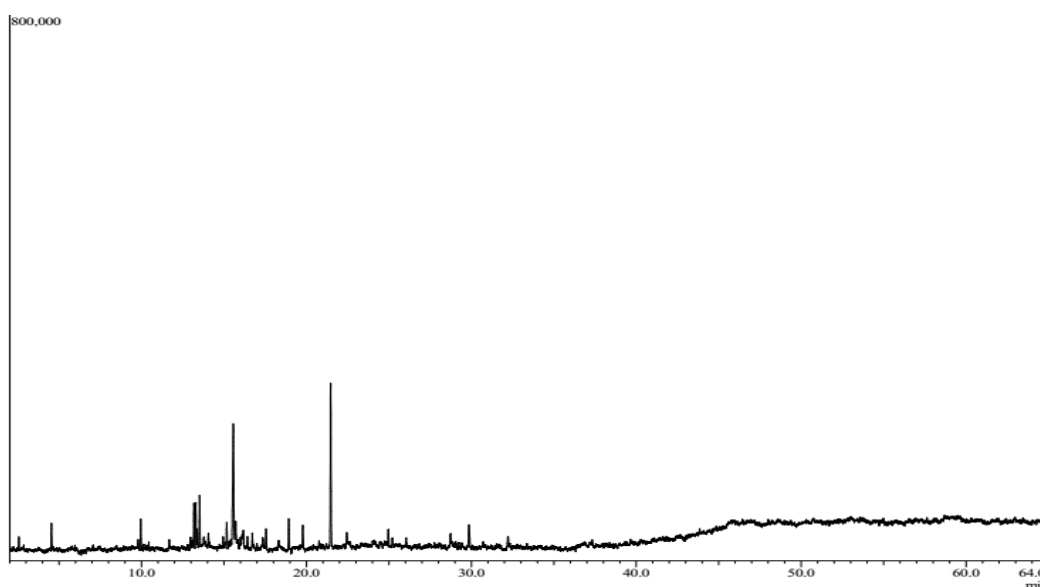


Figure 33: GCMS chromatogram of AWAED-CN fraction.

Table 16: The identified compounds of AWAED-CN by GC-MS analysis.

Resultas and Discussion

R. T	Name	AWAED-CN (A%)
2.557	(R)-2-Hexanol	0.75
4.529	Glycerin	1.75
6.589	2-Isopropylpiperazine (K)	0.46
9.950	4-(1-methylethyl) Benzenemethanol	1.68
13.176	Terpin	2.64
13.284	2-Hydroxycineol	2.76
13.380	2-methyl-5-(1-methylethyl)-1-Cyclopentene-1-methanol	1.16
15.165	Sobrerol	1.63
15.568	Triethanolamine (Sterolamide) (L)	12.77
15.710	2-Octen-1-ol, 3,7-dimethyl-, isobutyrate, (Z)-	2.04
15.850	m-nitrophenethyl acetate	0.4
16.110	Caryophyllane, 4,8- β -epoxy	1.18
16.170	1-Hydroxy-p-menth-3-one	1.01
17.549	2-(4-methylolcyclohex-3-en-1-yl) propan-2-ol	1.97
18.926	Myristicin	2.30
19.779	2-Hydroxy-2-(4-methoxy-phenyl)-N-methyl-acetamide (G)	1.77
21.468	Dill apiole	10.36
24.965	1,3,7,7-tetramethyl-Spirio-10-(2,11-dioxabicyclo[4.4.1]undeca-3,5-diene)-2'-(oxirane)	1.37
28.739	n-Hexadecanoic acid	1.01
29.853	palmitic acid	2.20
32.224	Oleic Acid	1.17

Resultas and Discussion

42.536	7-[2,6-Dichlorobenzyl]-4-chloropyrrolo[2,3-d]pyrimidine (M)	0.42
42.645	4,5,5-Trimethyl-2,4-diphenyl-2-oxazoline (B)	0.48
	Total	53.28
B: Amine, C: Aminoacid, G: Acetamide, L: Sterol amide, M: Pyrimidine		

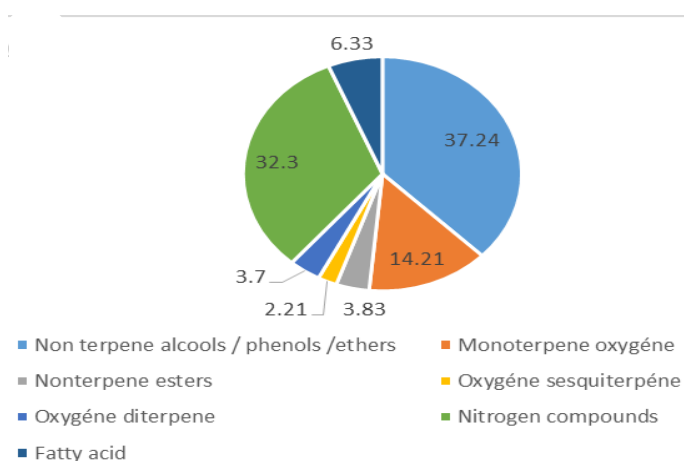


Figure 34: Chemical families classe of AWAED-CN fraction

Myristicin	Apiole
2,2'-methylenebis[6-(1,1-dimethyl ethyl)-4-methyl phenol]	Octan-2-yl palmitate

Resultas and Discussion

Figure 35: the main identified volatile compounds

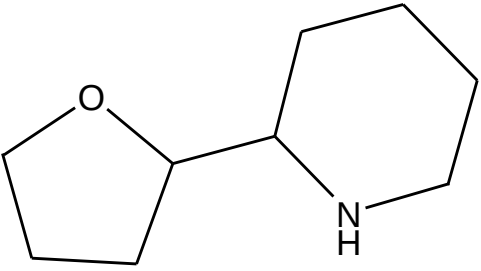
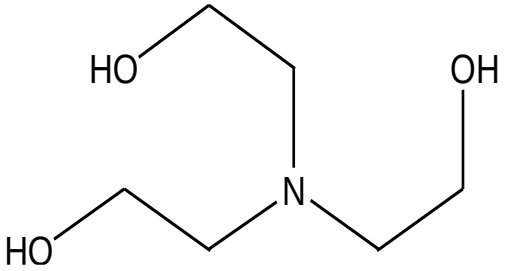
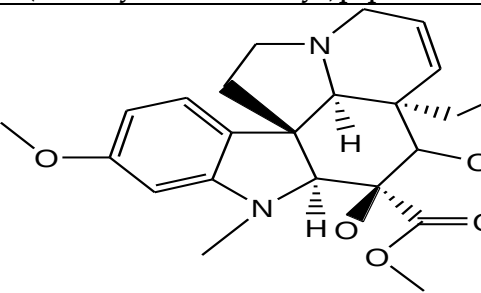
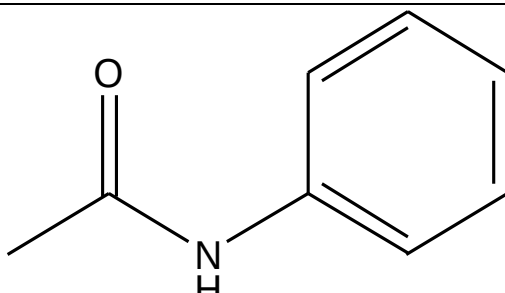
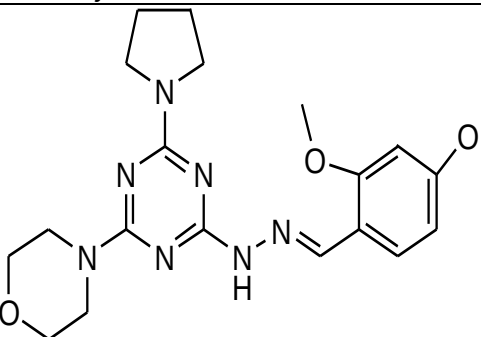
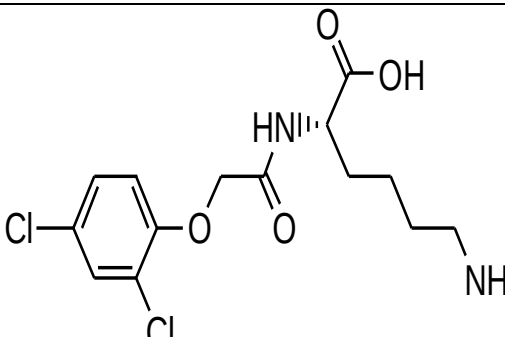
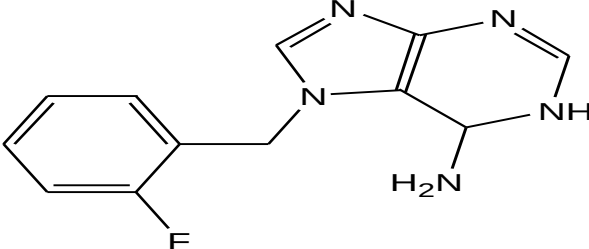
	
2-(tetrahydrofuran-2-yl)piperidine	Triethanolamine
	
Deacetylvindoline	Acetanilide
	
2-[(2,4-Dimethoxybenzylidene)hydrazino]-4-morpholino-6-(1-pyrrolidinyl)-1,3,5-triazine	N2-[(2,4-dichloro phenoxy)acetyl]-1-Lysine
	
[(2-fluorophenyl)methyl] 1H-Purin-6-amine	

Figure 36: Some volatile alkaloid identified structure

Resultas and Discussion

III.3.2. Fixed alkaloids

III.3.2.1. Identification In a Basic alkaloid extract

A. TLC analysis results of C fraction

The separation of the C fraction allows us to obtain 129 sub-fractions, the fractionation, and regrouping of the sub-fractions are monitored under their TLC profile and the positive dragendroff reagent, the TLC analysis showed that some of these fractions exhibited the presence of alkaloid compounds, among of these fractions the C34, C75, C78, C80, and C84 fractions were selected, then were precipitated with **n-Hex**, **Et₂O**, **DCM**, and **MeOH** respectively to isolate the pure alkaloid compounds Figure38.

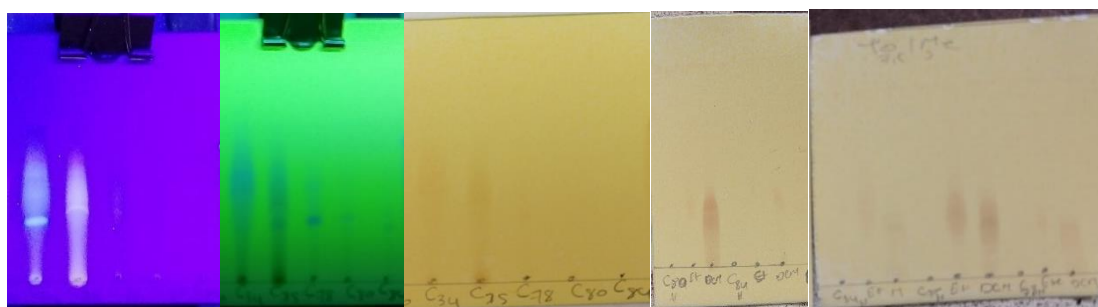


Figure 37: The TLC profile of C34, 75, 78,80, and 84 fractions developed and revealed with Dragendorff reagent "QC, QF".

The TLC profile of the five precipitate fractions revealed with Dragendorff reagent showed the presence of one or two spots in these fractions, identifying the presence of alkaloids. The C34 was given 2 isolated alkaloid compounds (C34-EtO "1" compound **1.6 mg** and C34-Me "1 compound" **1.2 mg**), the C75-EtO and DM were given 1 similar isolated alkaloid compound **2.3 mg**, the C78-DM was given 1 isolated alkaloid compound **trace**, the C80-DM was given 1 isolated alkaloid compound **1.4 mg**, and the C84-DM was given 1 isolated alkaloid compound **2.1 mg**.

The **C 3-10R** (figure 39) were regrouped and then refracted to give 60 sub-sub-fraction **SFC** regrouped to 14 fractions, analyzed with TLC, and revealed with Dragendorff (Figure 40-41).

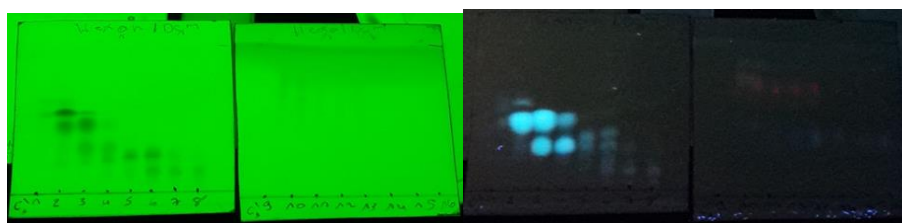


Figure 38: TLC profile of C3-10 (254, 365nm) "QF"

Resultas and Discussion

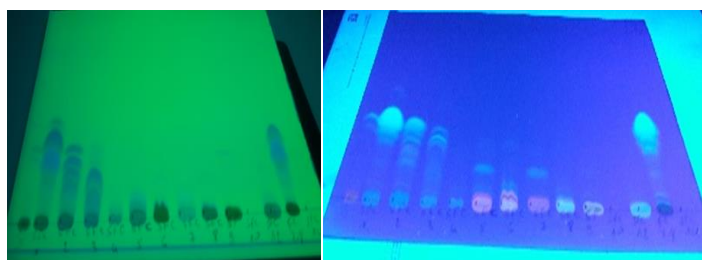


Figure 39: The regrouped sub-sub-fraction SFC1-14 without Dragendorff (254, 365 nm) "QC"

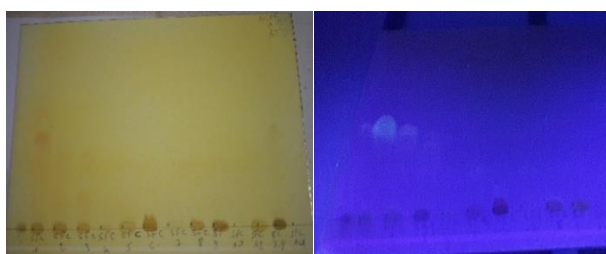


Figure 40: The regrouped sub-sub-fraction SFC1-14 revealed with Dragendorff (Vis, 365nm) "QC"

The **SFC1- 7** were washed with n-H then developed again and revealed with Drangendorff (Figure 42) where they have shown the presence of alkaloid compounds with deference Rf in these sub-fractions.

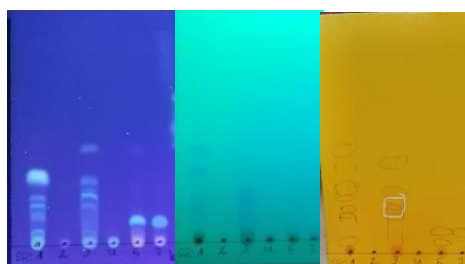


Figure 41: TLC profile of SFC1-7 revealed with Dragendorff (365, 254 nm, Vis) "QF"

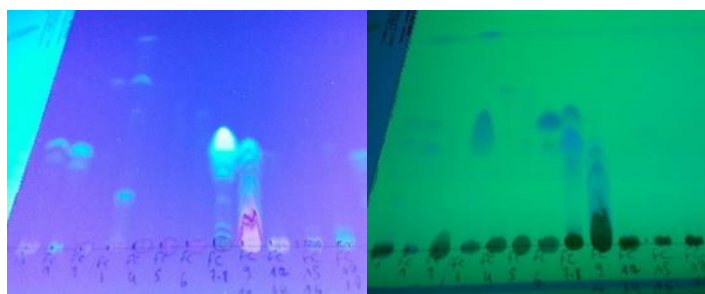
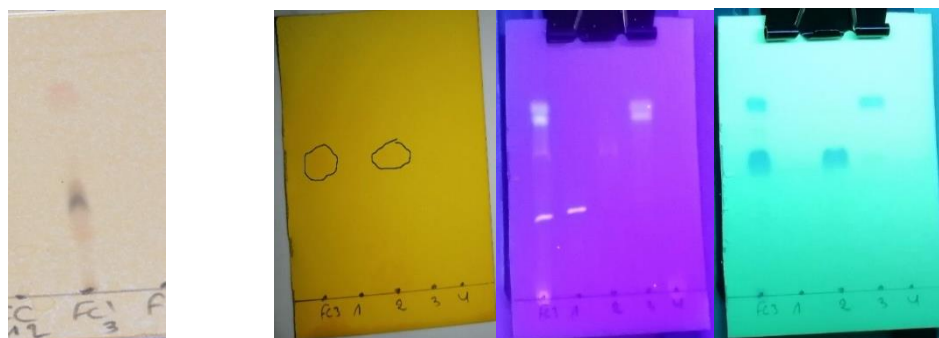


Figure 42: The TLC profile of C' fractions "QD"

Resultas and Discussion

From the TLC profile of C' fractions (Figure 43), the FC3 illustrates the presence of five separate compounds. The FC3 fraction was washed with n-H and then the precipitate was separated with PTLC to obtain 4 compounds (Figure 44).



A: FC3 Before washing

B:FC3 after washing and separation “QF”

Figure 43: The TLC profile of FC3 before separation and after separation A and B

A yellow oily isolated compound from FC3-2 with 1.3 mg was revealed as an alkaloid compound.

B. Identification of alkaloid compounds in D fraction

The HPLC analysis was used to identify the alkaloid compounds in the study fractions by the comparison of retention times and surfaces of six alkaloid controls with the fractions, chromatograms recorded for identification and quantification of their content. (Figure 45).

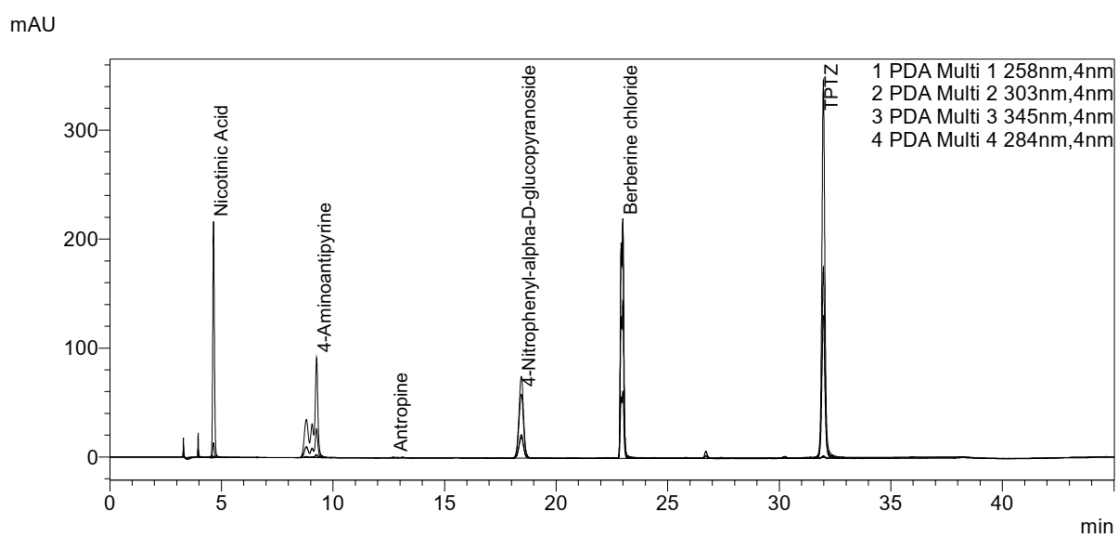


Figure 44: HPLC chromatogram of the six studies alkaloid control

Three alkaloid compounds were identified in the D fraction compared to the controls (4-Aminoantipyrine, Atropine, and 4-Nitrophenyl- α -D-

Resultas and Discussion

glucopyranoside) (Figure 46), the atropine illustrated as a main compound 1243.93mg/kg MS (mass sample) (96.3%) (Table 17).

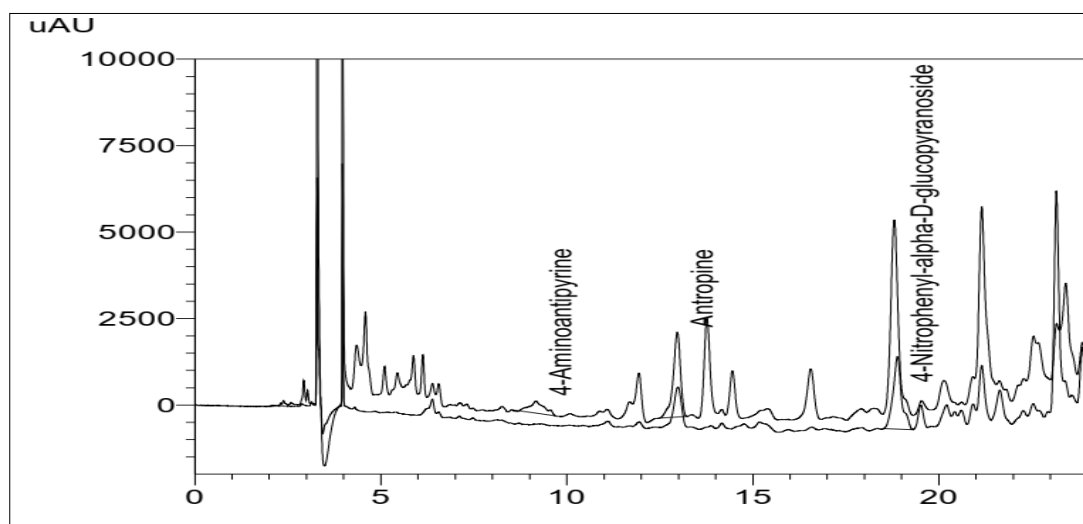


Figure 45: The HPLC chromatogram with the identified alkaloid in D fraction

C. Identification of alkaloid compounds in E fraction

Two alkaloid compounds were identified in the E fraction (4-Aminoantipyrine and Atropine) (Figure 47), with the Atropine revealed as a main compound (96%) (Table 17).

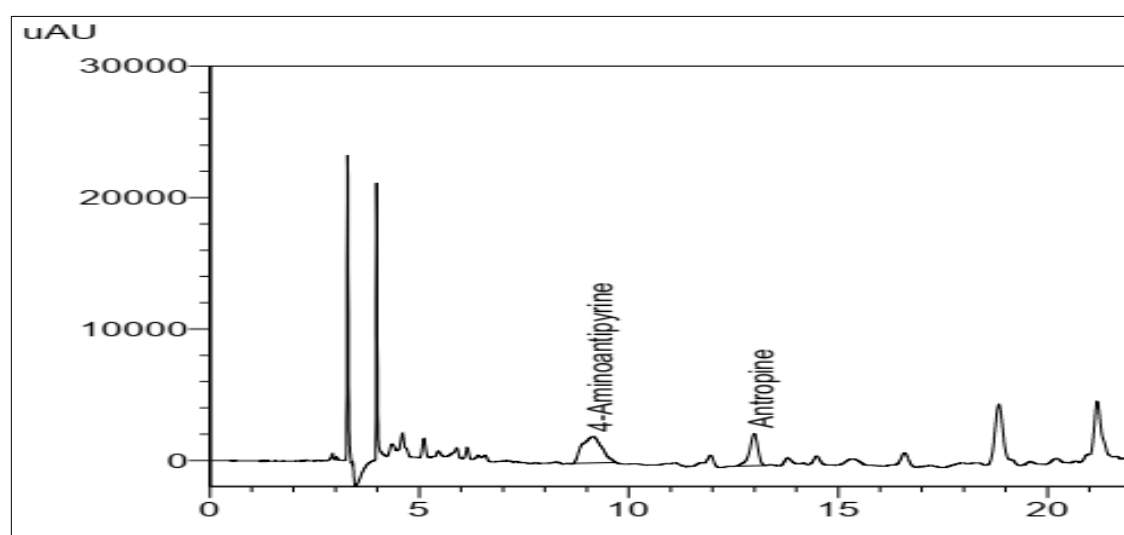


Figure 46: The HPLC chromatogram with the identified alkaloid in E fraction

Resultas and Discussion

D. Identification of alkaloid compounds in F fraction

Two alkaloid compounds were identified (4-amino antipyrine and atropine) (Figure 48)

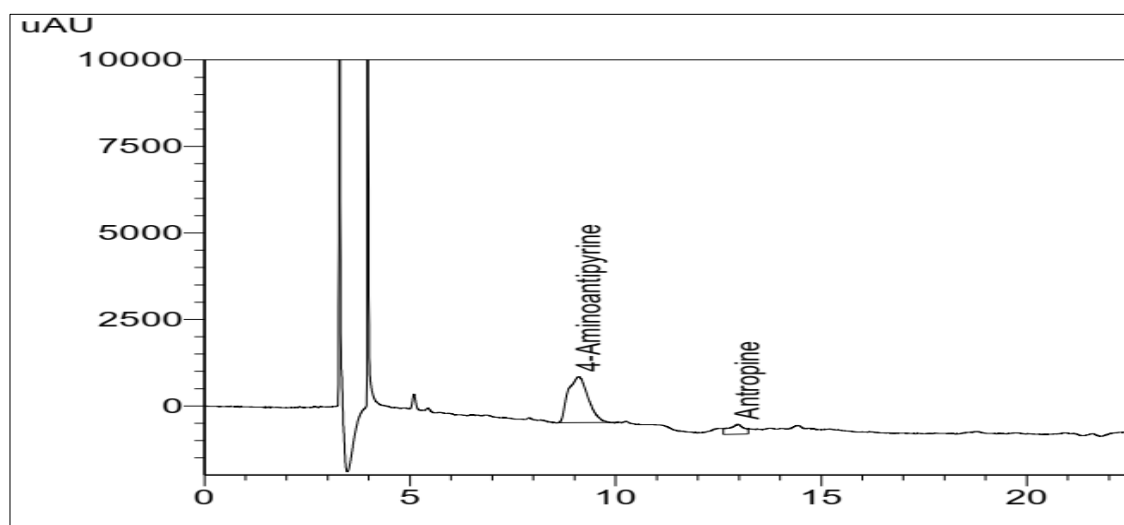


Figure 47: The HPLC chromatogram with the identified alkaloid in F fraction

Table 17: The identified alkaloid compounds in D, E, and F fractions.

N ^o	R.T	Compound	Concentration mg/kg MS			λ nm
			D	E	F	
1	9,15	4-Aminoantipyrine	45.03	230.43	109.5	258
2	12,96	Atropine	1243.93	5418.29	165	258
3	18,88	4-Nitrophenyl- α -D-glucopyranoside	3.23	-	-	303

R.T: retention time

The qualitative and quantitative results of alkaloid compounds analyzed by HPLC illustrated that the D, E, and F fractions demonstrate the presence of three alkaloid compounds in the D fraction and two in both the E and F fractions, with the atropine identified as a main compound at 1243.93, 5418.29, and 165 mg/kg MS, respectively. The E fraction exhibits the highest concentration of atropine at 5418.29 mg/kg MS.

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III.3.2.2. Identification in Polar solvent EA extract

The precipitation method of EA extract is given 4 sub-fractions (Figure 17). The EA-M fraction shows the presence of alkaloid compounds (Figure 50). At the same time, the HPLC results confirmed this presence by identifying berberine chloride and 4-amino antipyrine as alkaloid compounds with berberine chloride as the main compound (Figure 49, Table 18).

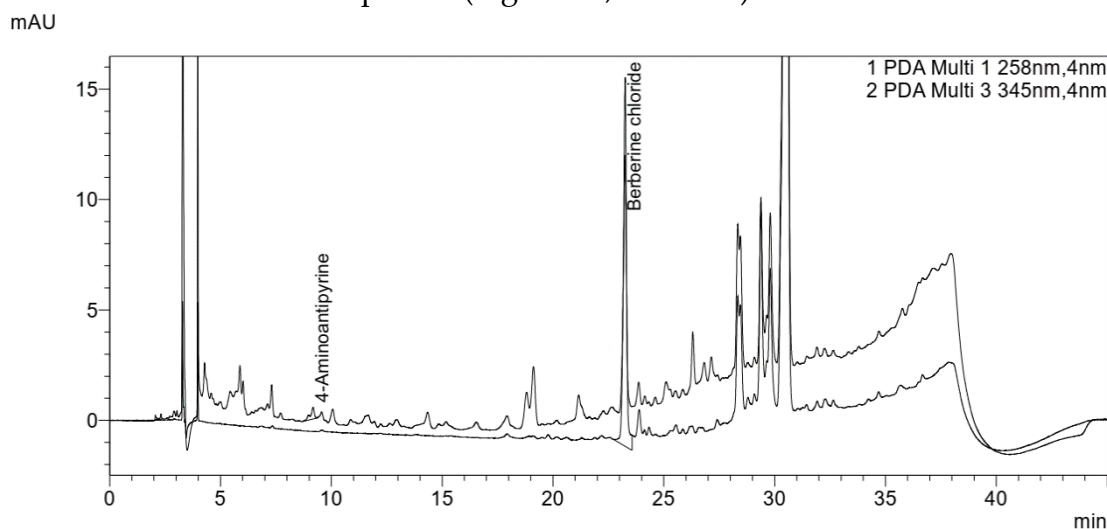


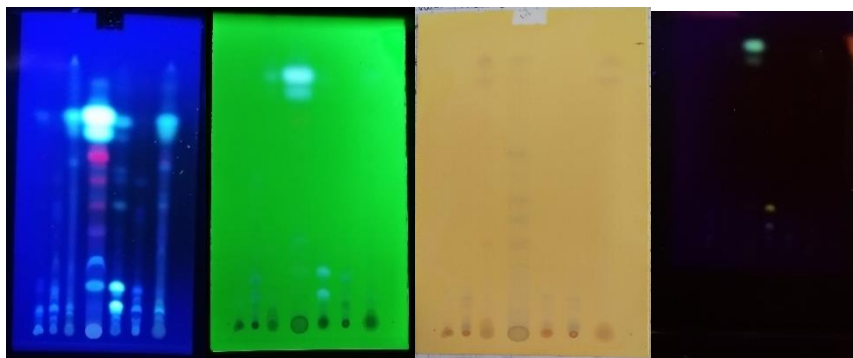
Figure 48: The HPLC chromatogram with the identified alkaloid in the EA-M fraction

III.3.2.3. Identification in Free Alkaloid Extract

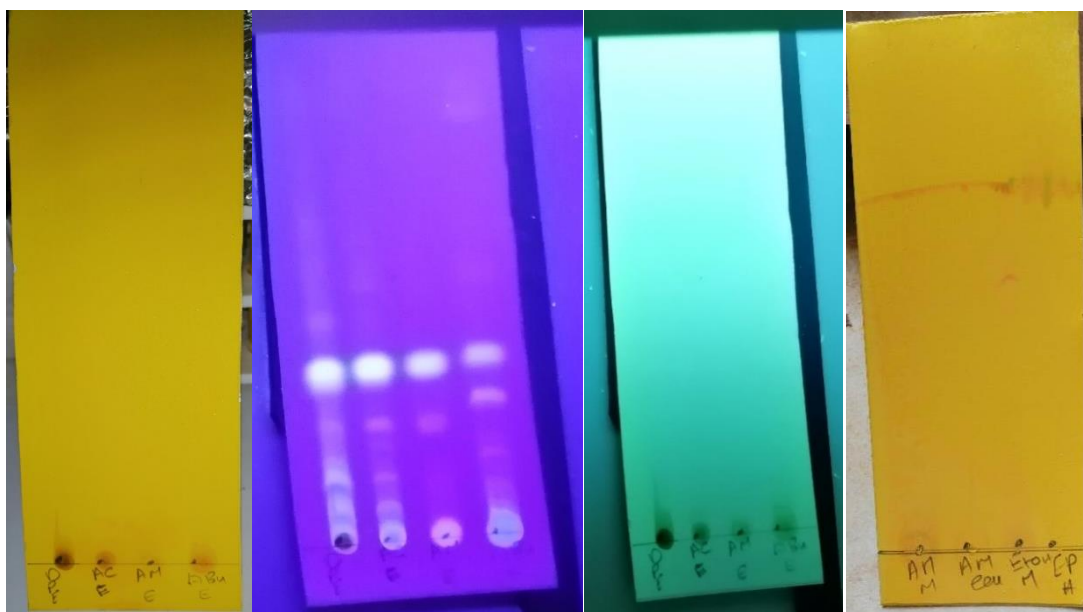
❖ In FAEDE-M, FAEA-M, and FAEB-M

The HPLC analysis allowed us to identify berberine chloride as an alkaloid compound in the **FAEDE-M**, **FAEA-M**, and **FAEB-M** fractions (Figures 51, 52, 53) with a concentration of 1.09 mg/kg (Table 18). The 4-Nitrophenyl-alpha-D-glucopyrnsoside was identified only in the FAEA-M fraction. The quantification analysis illustrated the berberine chloride with a remarkable quantity in FAEA-M and FAEB-M compared to FAEDE-M (Table 19). The HPLC analysis confirmed the TLC results and showed the presence of berberine chloride with a yellow fluorescence (figure 27-B of B fraction). These findings confirmed the TLC analysis where it's show the presence of alkaloid compound such as berberine with a yellow fluorescence (Figure 54).

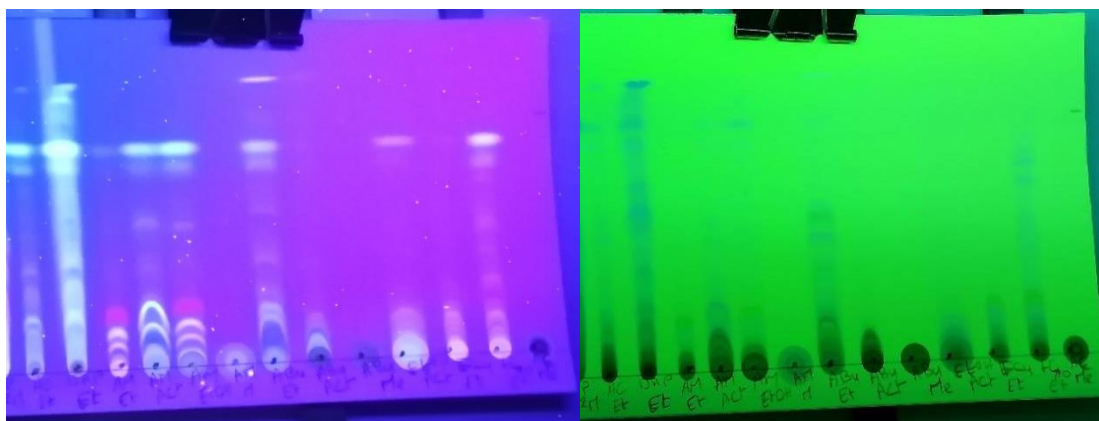
Resultas and Discussion



A: TLC profile of free alkaloid extracts before and after Dragendorff reagent “QC”

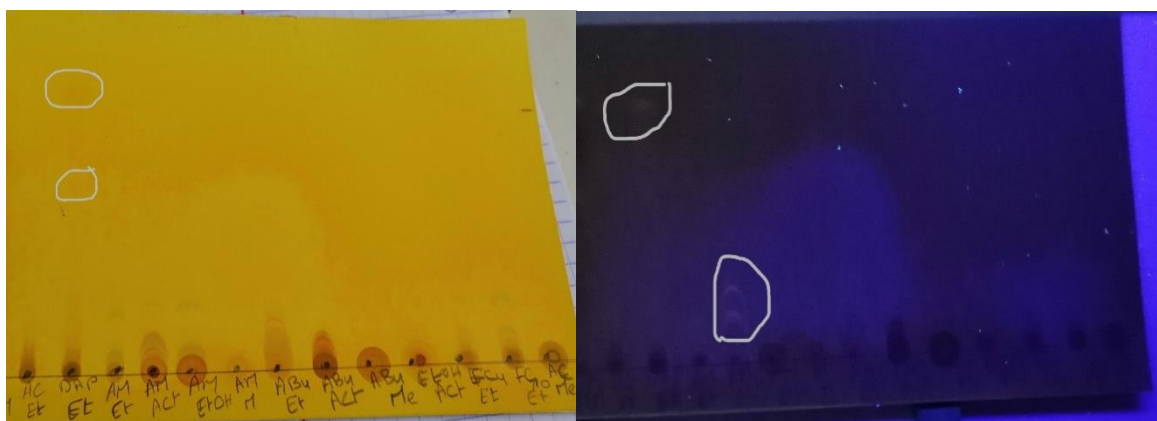


B: TLC profile of free alkaloids fractions **FAED-E**, **FAEA-E**, **FAB-E** before and after Dragendorff reagent “QF”

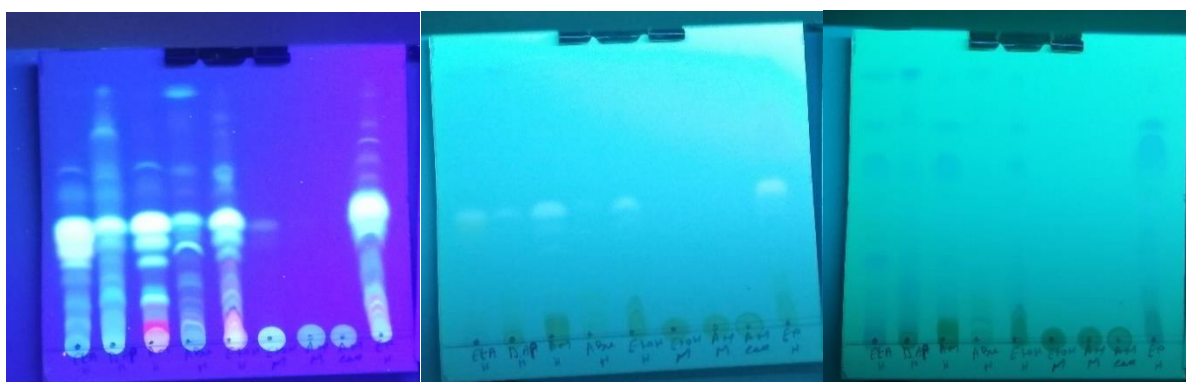


C : TLC profile of different fractions of EA and free alkaloid fractions 254 and 366nm “QF”

Resultas and Discussion



D: TLC profile of different fractions of EA and free alkaloid extracts after Dragendorff reagent “QC”.



E:The TLC profile of the FAED and FAEB fractions of free alkaloids extracts “QF”.

Figure 49: The TLC profiles (A, B, C, D and E) of EA and Free alkaloid fractions and extracts with different eluent and state.

Resultas and Discussion

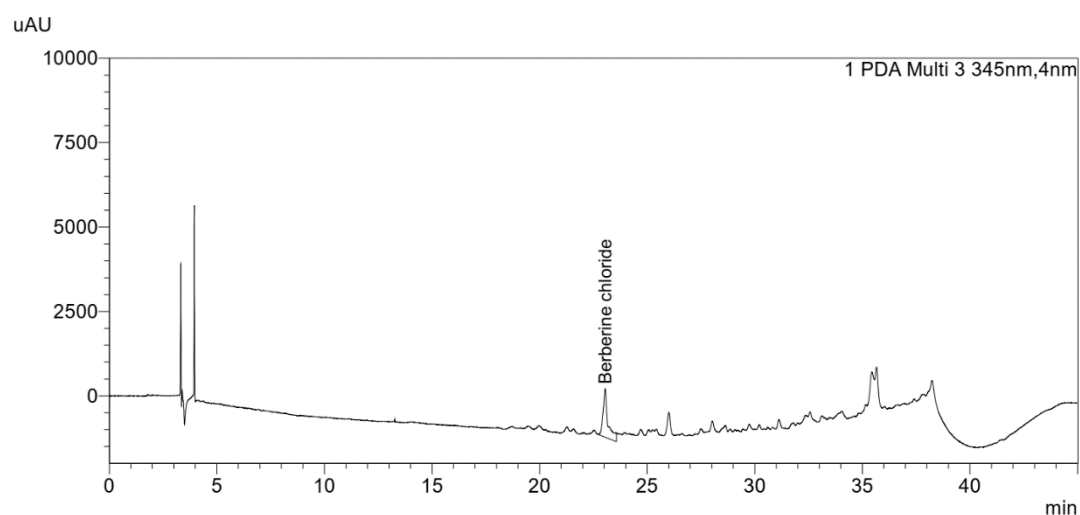


Figure 50: The HPLC chromatogram of alkaloid identified in FAEDE-M

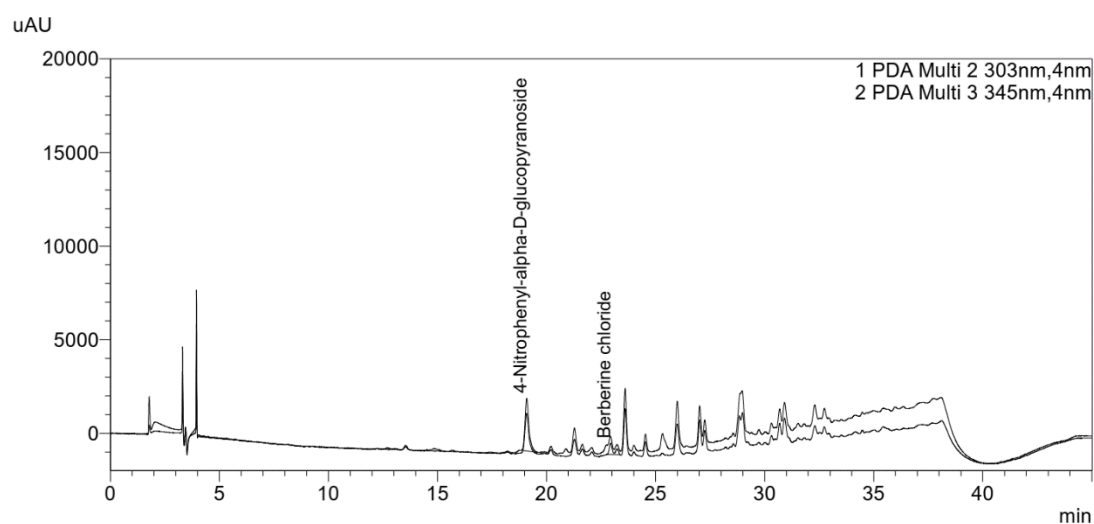


Figure 51: The HPLC chromatogram of alkaloid identified in FAEA-M

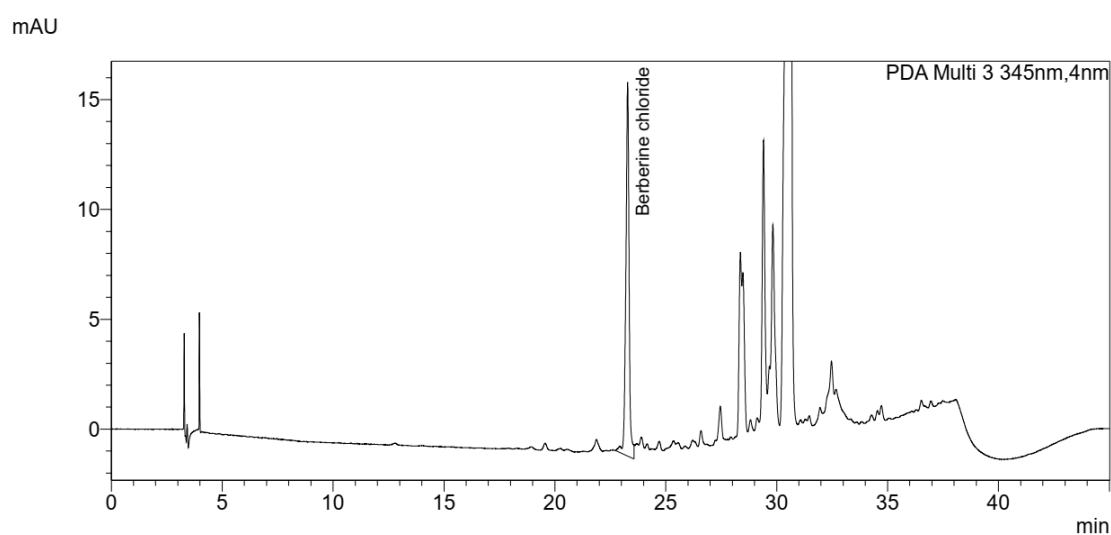


Figure 52: The HPLC chromatogram of alkaloid identified in FAEB-M

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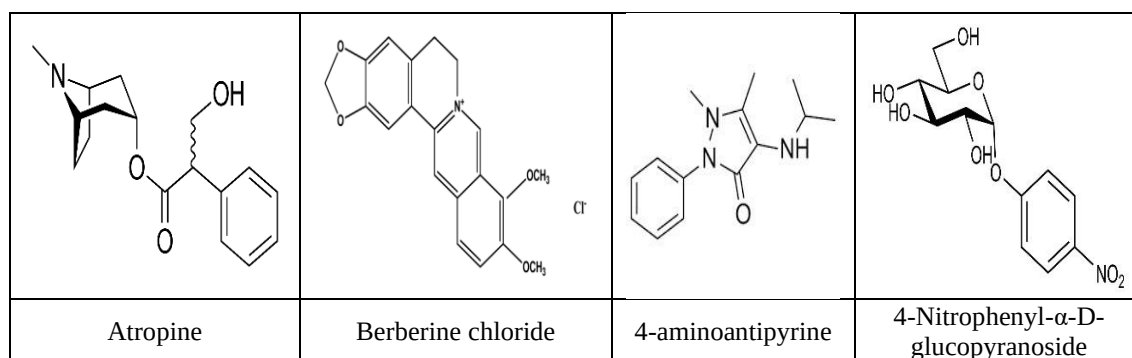


Figure 53: The structure of the identified alkaloids

Table 18: The identified alkaloid compounds in free alkaloid fractions.

N°	R.T	Compound	Concentration mg/kg MS			
			FAEDE-M	FAEA-M	FAEB-M	EA-M
01	9.151	4-Aminoantipyrine	/	/	/	6.19
02	19.09	4-Nitrophenyl-α-D-glucopyrnoside	/	1.48	/	/
03	23.25	Berberine chloride	1.09	7.26	8.10	5.40
Total			1.09	8.74	8.10	11.59

R.T: Retention time

The identification of four alkaloid compounds in the different fractions/ extracts with different solvents (non-polar, semi-polar, and polar) indicated that the plant contains more than one (group-family) type of fixed alkaloids. Where the 4-amino antipyrine was identified especially in the semi-polar and polar fractions with the high concentration in the fraction of 25:75% DCM: MeOH. In addition, the atropine was identified in these type of fractions with the high concentration in the same fraction 25:75% DCM: MeOH. While the 4-Nitrophenyl-alpha-D-glucopyrnoside was identified just in the semi-polar fractions, the berberine chloride was identified in all the solvent types with a high concentration in the FAEB and FAEA respectively.

These results inform us with the presence of interesting alkaloid groups in this study plant. This identification allows us to detect the presence of alkaloid compounds from the isoquinoline and tropane alkaloids such as berberine and atropine respectively [139]. On the other hand, the identification of alkaloid compounds in this plant allow us to identify some volatile with an interesting concentration and 4 fixed alkaloid compounds for the first time with an

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important concentration such as Atropine[60]. Where they exhibit important pharmacological properties such as anti-diabetic, anti-Alzheimer, bio-insecticide, and antioxidant activities[12], [18], [140], [141].

The HPLC analysis allow as to identify three alkaloid compounds in the fractions of free alkaloid extract. The berberine chloride was identified in all these fractions except the EA-M fraction, with 4-amino antipyrine as the main compound. The identification of alkaloid compounds in this species allows us to identify some volatile and 4 fixed alkaloid compounds for the first time with an important concentration such as Atropine. Our results were in agreement with Niko Radulovic et al., (2011) reported the presence of 2-Pentylpiperidine as a volatile alkaloids in *Conium maculatum* L. Apiaceae family plants[41].

III.4. Identification of Phenolic compounds

III.4.1. In fractions of Basic alkaloid extracts

A. Identification in C fraction

Three phenolic compounds were identified in the C fraction (Figure 55); two phenolic acids (p-coumaric acid, cinnamic acid), and one flavonoid (naringenin). The quantitative analysis shows that naringenin is the main compound (Table 19).

B. Identification in D fraction

Five phenolic compounds were identified in the D fraction by HPLC analysis (Figure 56), two phenolic acids (4-Hydroxybenzoic acid, and p-Coumaric acid), and three flavonoids ((-)-Epicatechin, Apigenin 7-glucoside, and Quercetin), the quantitative analysis shown the (-)-Epicatechin as a major compounds (Table 19).

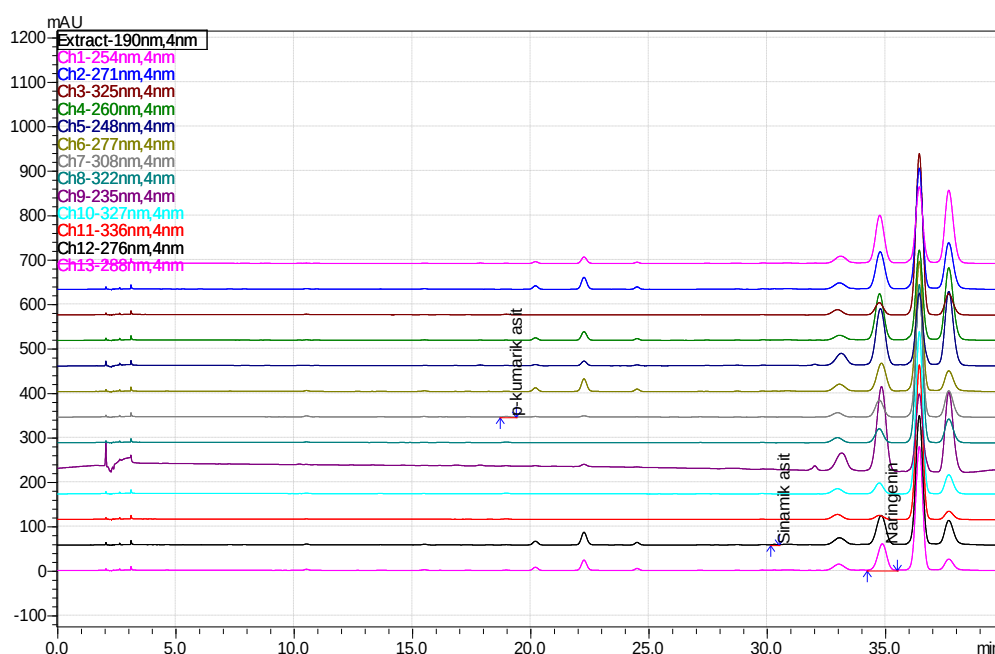


Figure 54: HPLC chromatogram of phenolic compounds identified in C fraction

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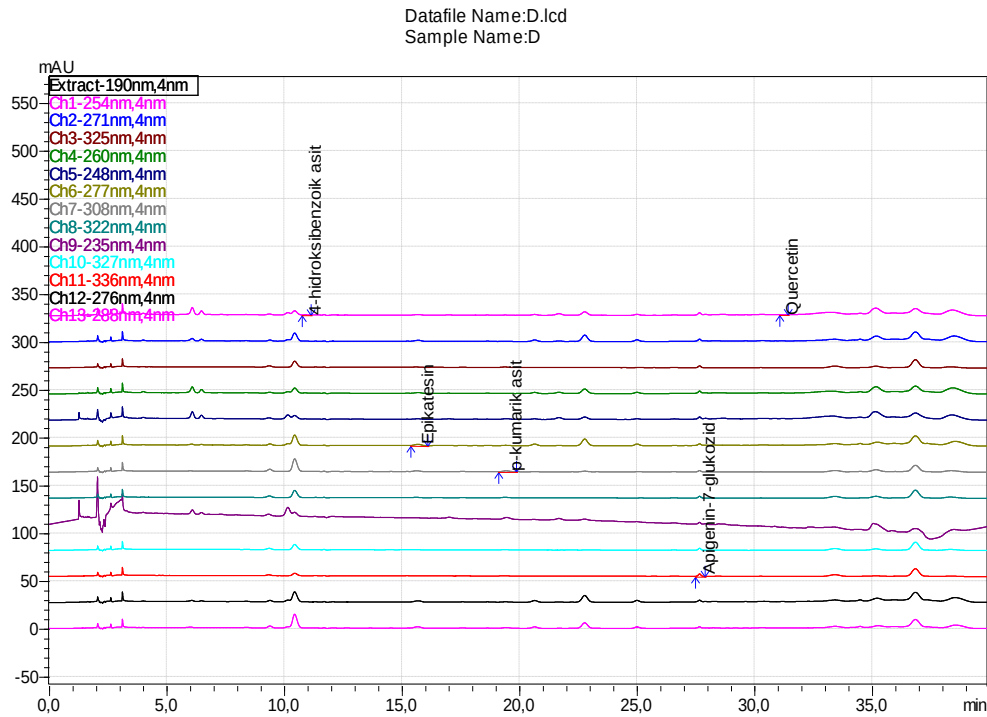


Figure 55: HPLC chromatogram of phenolic compounds identified in D fraction

C. Identification in E fraction

Two phenolic compounds were identified; flavonoid (Apigenin 7-glucoside) and phenolic acid (Cinnamic acid) (Figure 57, Table 19).

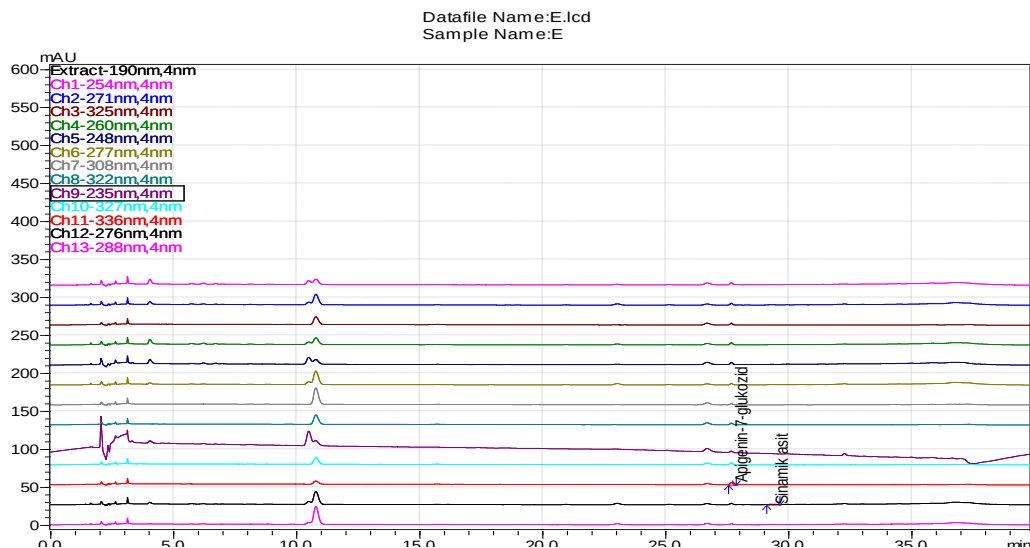


Figure 56: HPLC chromatogram of the phenol compounds identified in E fraction

Resultas and Discussion

D. Identification in F fraction

Two phenolic compounds were identified; phenolic acid (Cinnamic acid), and flavonoid (Quercetin) (figure 58, Table 19).

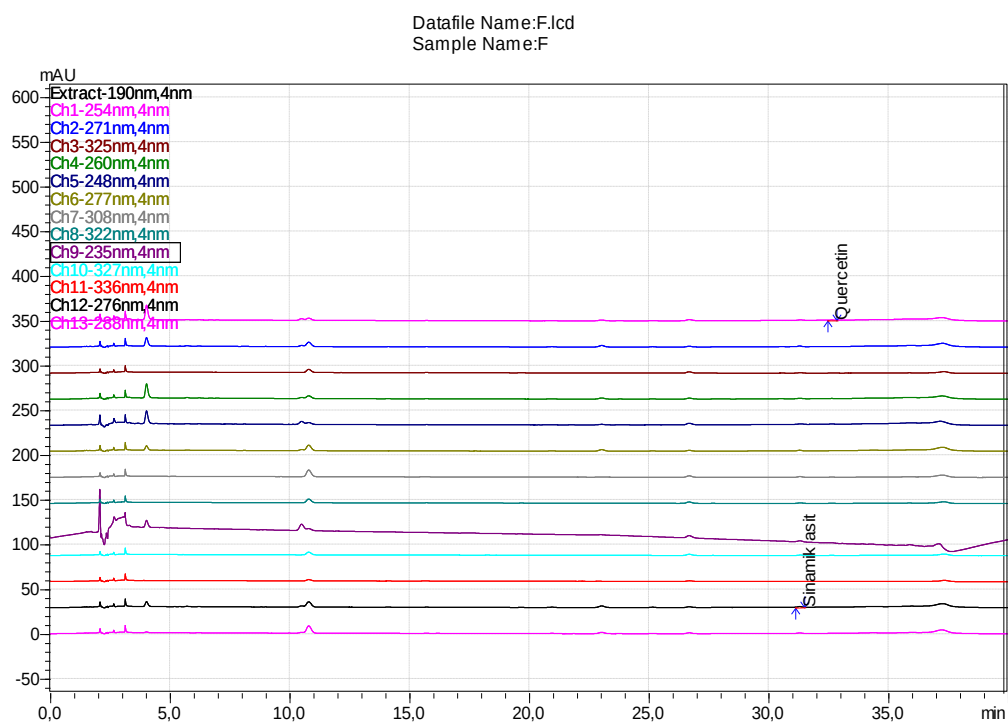


Figure 57: HPLC chromatogram of phenol compounds identified in F fraction

Table 19: The identified phenolic compounds in C, D, E, and F fractions by HPLC analysis

N	R.T	Compound	Concentration mg/kg MS				
			B	C	D	E	F
2	10,91	4-Hydroxybenzoic acid	-	-	0.14	-	-
4	15,71	(-)-Epicatechin	-	-	10.67	-	-
6	19,22	p-Coumaric acid	-	0.19	0.8	-	-
7	27,68	Apigenin 7-glucoside	-	-	1.36	6.81	-
8	30,32	Cinnamic acid	-	0.08	-	0.9	0.61
9	31,93	Quercetin	-	-	0.16	-	0.6
10	34,89	Naringenin	-	30.06	-	-	-

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E. Identification in Polar solvent EA-M

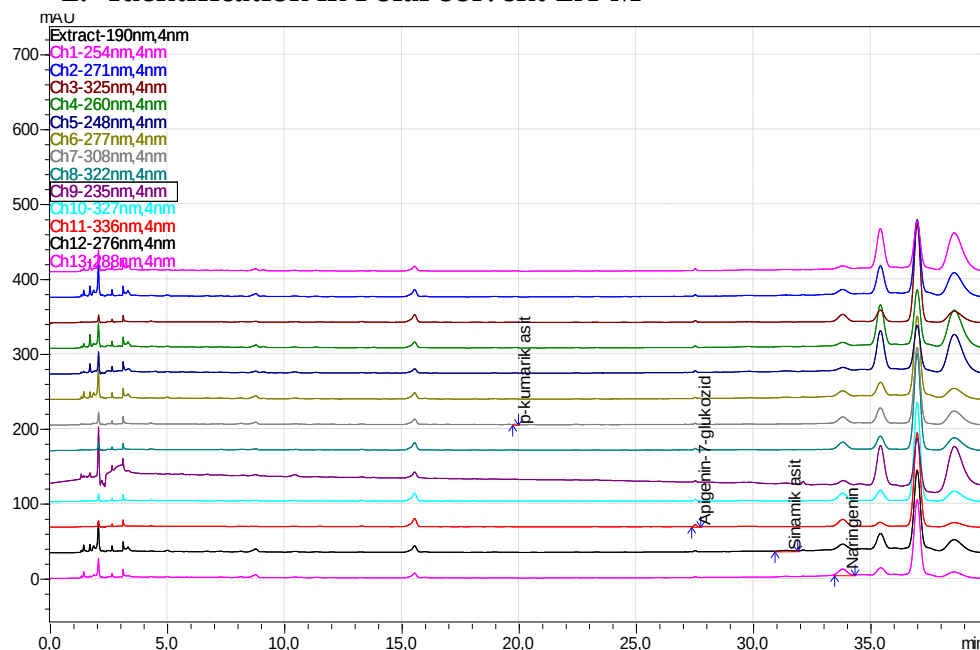


Figure 58: HPLC chromatogram of phenolic compounds identified in EA-M fraction

Table 20: The identified phenolic compounds in EA-M fraction by HPLC analysis

N	R.T	compound	Concentration mg/Kg MS
			EA-M
1	19.84	P-coumaric acid	0.007
2	27.54	Apigenin-7-glucosid	0.080
3	29.97	Cinamic acid	0.041
4	33.83	Naringenin	0.911

III.4.2. Phenolic in Acidic alkaloid extracts

A. In AWAED and AWAEF fractions

Four phenolic compounds were identified in AWAED-CN, and five compounds were identified in AWAEF-CN. Epicatechin and chlorogenic acid were revealed as the main compounds in AWAED-CN and AWAEF-CN respectively (Figure 60-61, Table 21).

Results and Discussion

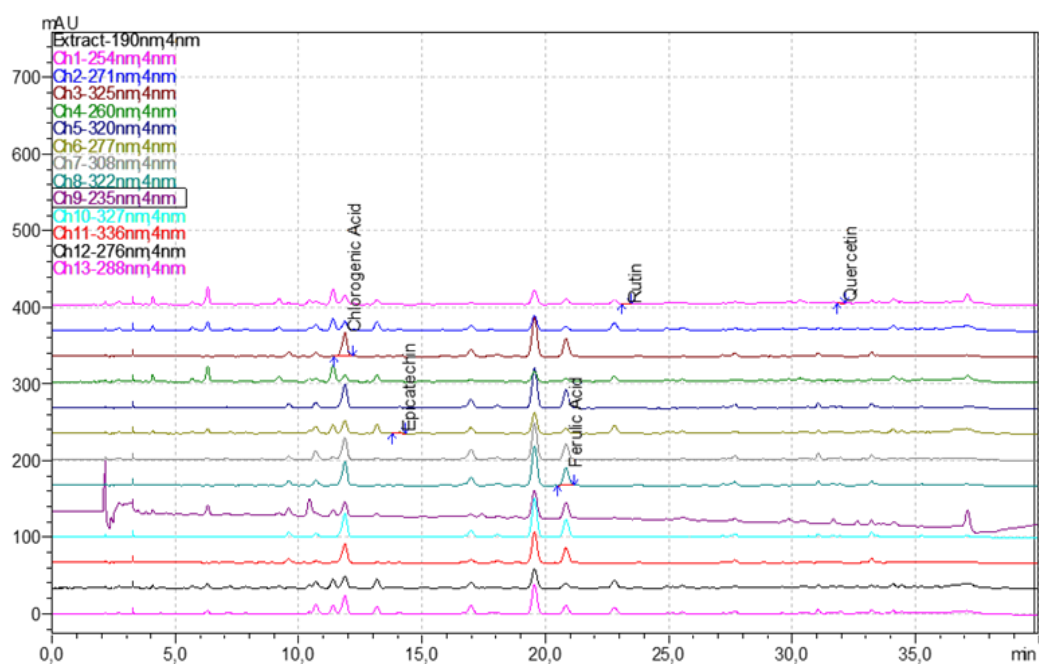


Figure 59: The HPLC chromatogram of phenolic compounds identified in AWAE-CN fraction

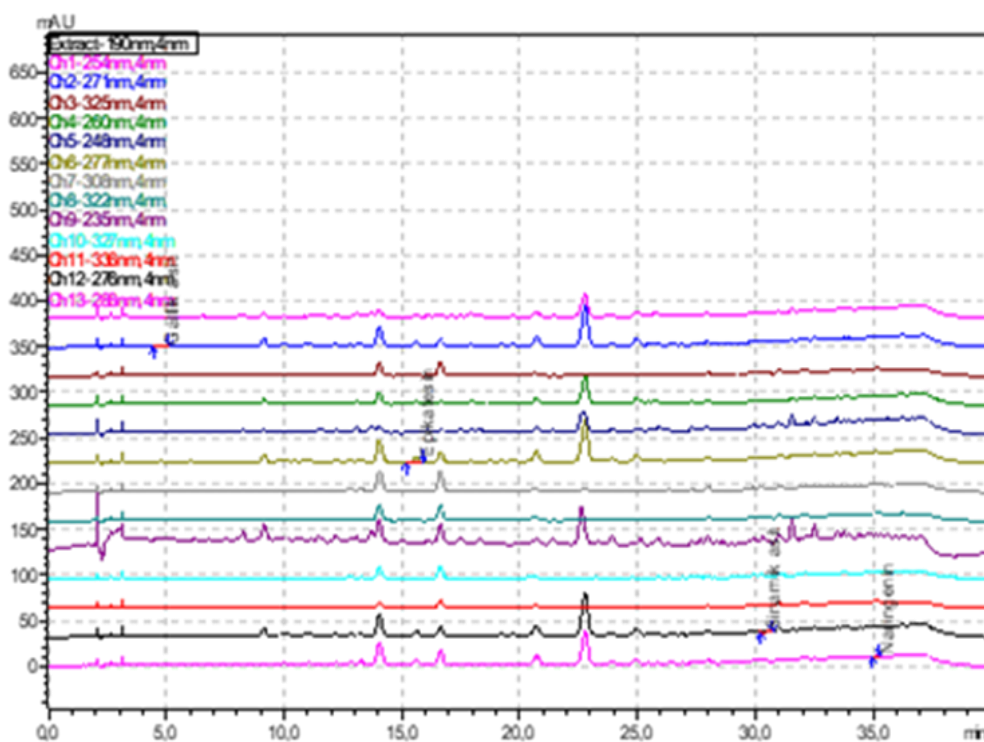


Figure 60: HPLC chromatogram of phenolic compounds identified in AWAED-CN fraction

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B. In MAAE-CN fraction

Ten phenolic compounds were identified in the MAAE-CN fraction with chlorogenic acid, Epicatechin, Eriocitrin, and chicoric acid were identified with high concentrations. The Eriocitrin (eriodictyol glycoside) compound was identified particularly in this fraction (Figure 62, Table 21).

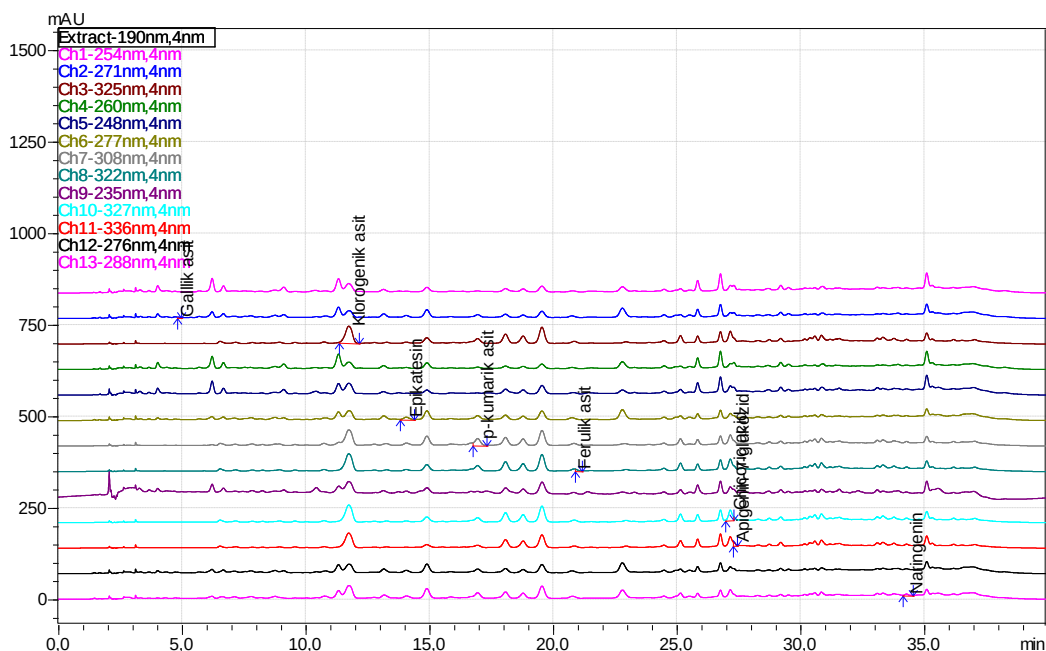


Figure 61: HPLC chromatogram of phenolic compounds identified in MAAE-CN fraction

Table 21: identified phenolic compounds in AWAEEs' fractions and MAAE-CN fraction by HPLC.

N	R. T	Compound	Concentration mg/kg MS		
			AWAED	AWAEE	MAAE-CN
1	4.744	Gallik acid	1.59	/	0.73
2	11.723	Chlorogenic acid	/	22.40	73.16
3	14.084	Epicatechin	45.07	6.39	34.14
4	18.315	p-Coumaric Acid			9.92
5	20.467	Ferulic Acid	/	9.5	1.68
6	20.784	Eriocitrin			58.94
7	23.711	Rutin	/	0.19	
8	27.16	Chicoric acid			67.19
9	27.54	Apigenin-7-glucosid			3.46
10	30.185	Cinamic acid	1.16	/	0.55
11	32.017	Quercetin	/	0.50	
12	34.853	Naringenin	0.80	/	2.07

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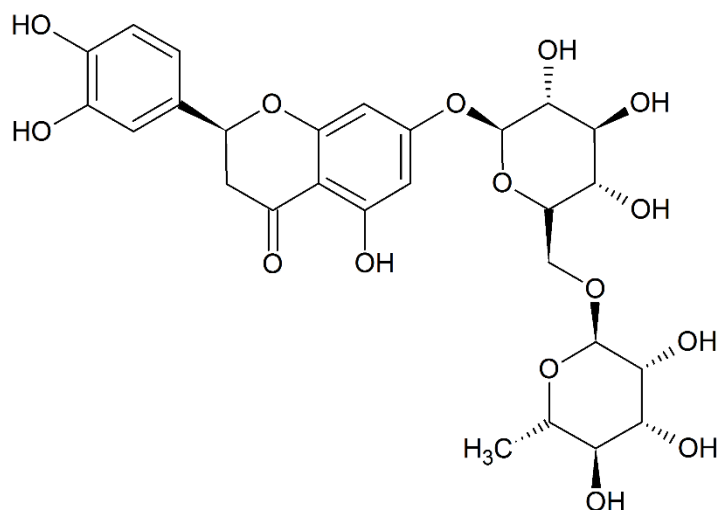


Figure 62: The eriocitrin-identified compound

III.4.3. Phenolic in free Alkaloid Extract

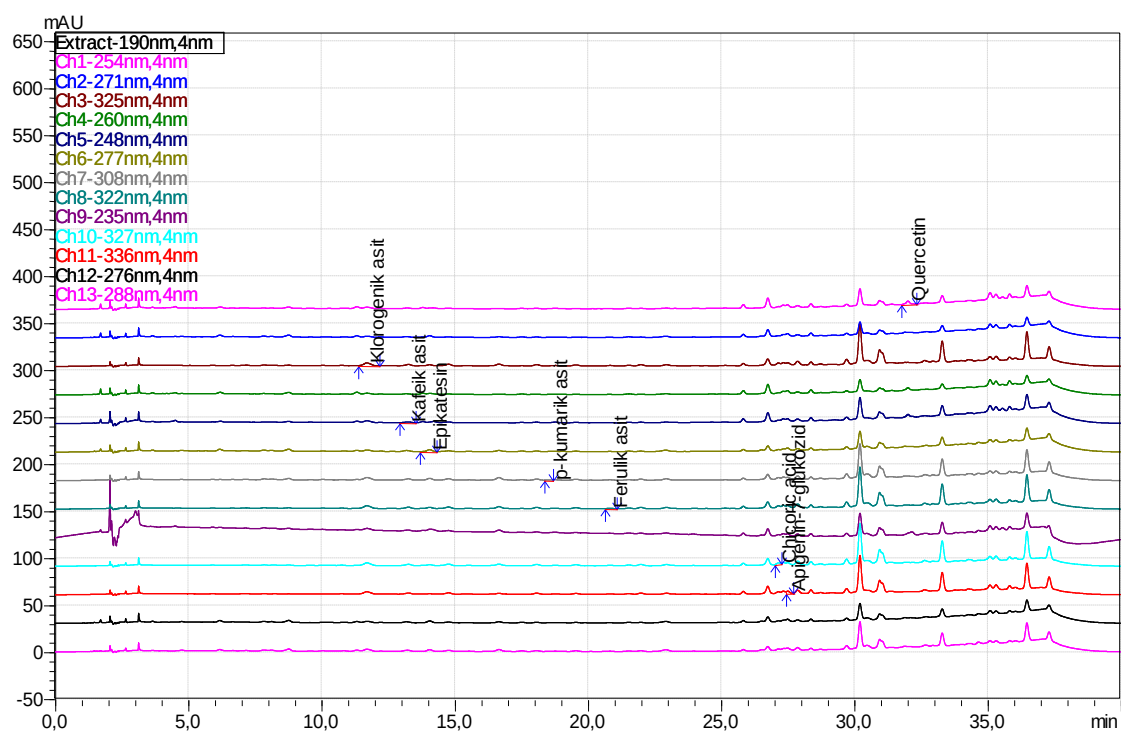


Figure 63: HPLC chromatogram of phenolic compounds identified in FAEB-CN fraction

Resultas and Discussion

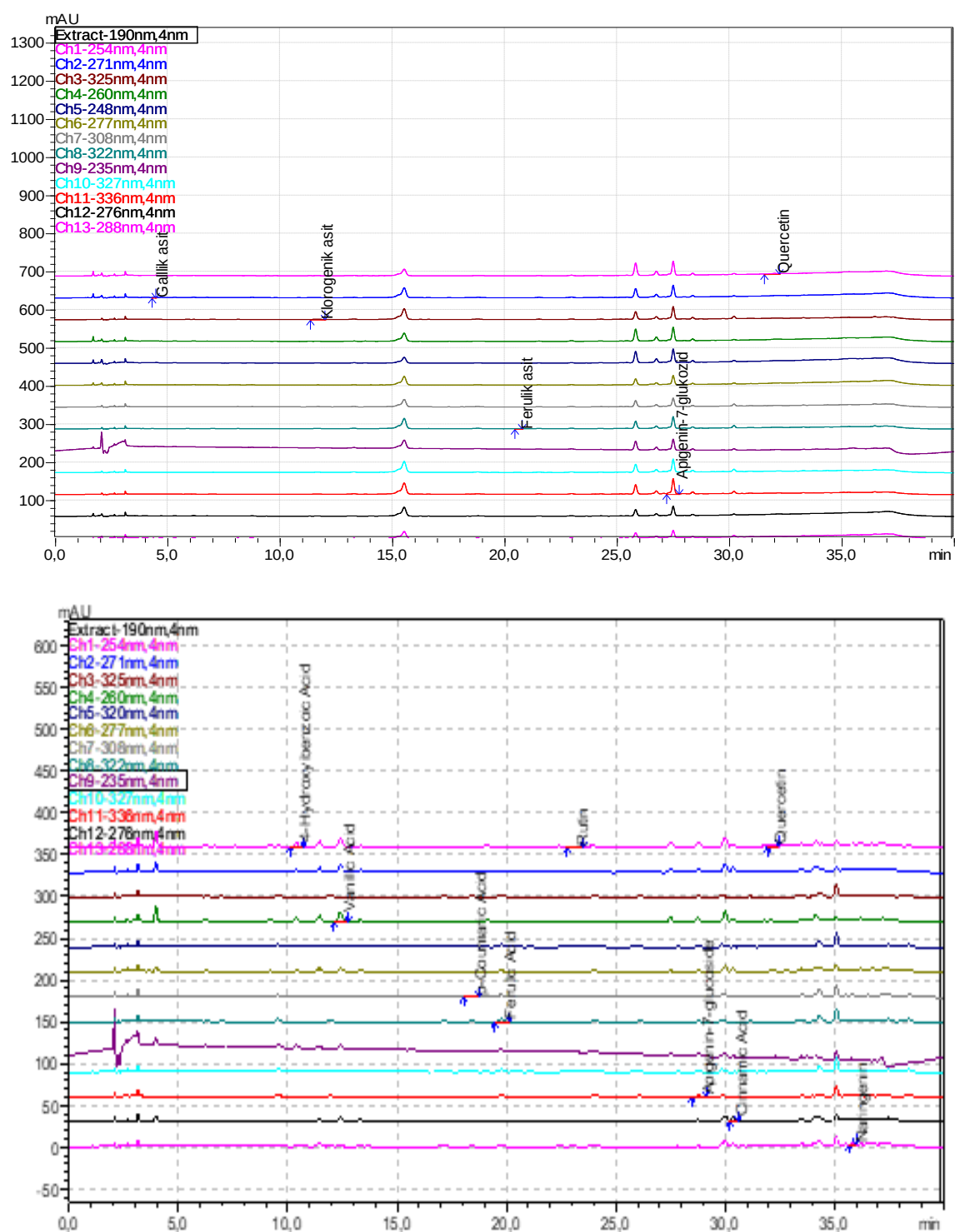


Figure 65: HPLC chromatogram of phenolic compounds identified in FAEDE-E fraction

Results and Discussion

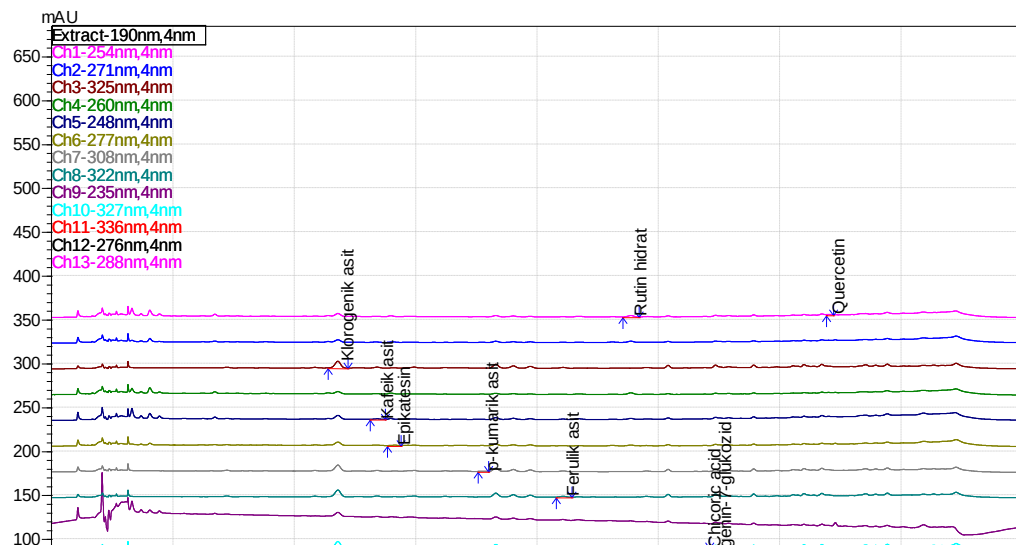
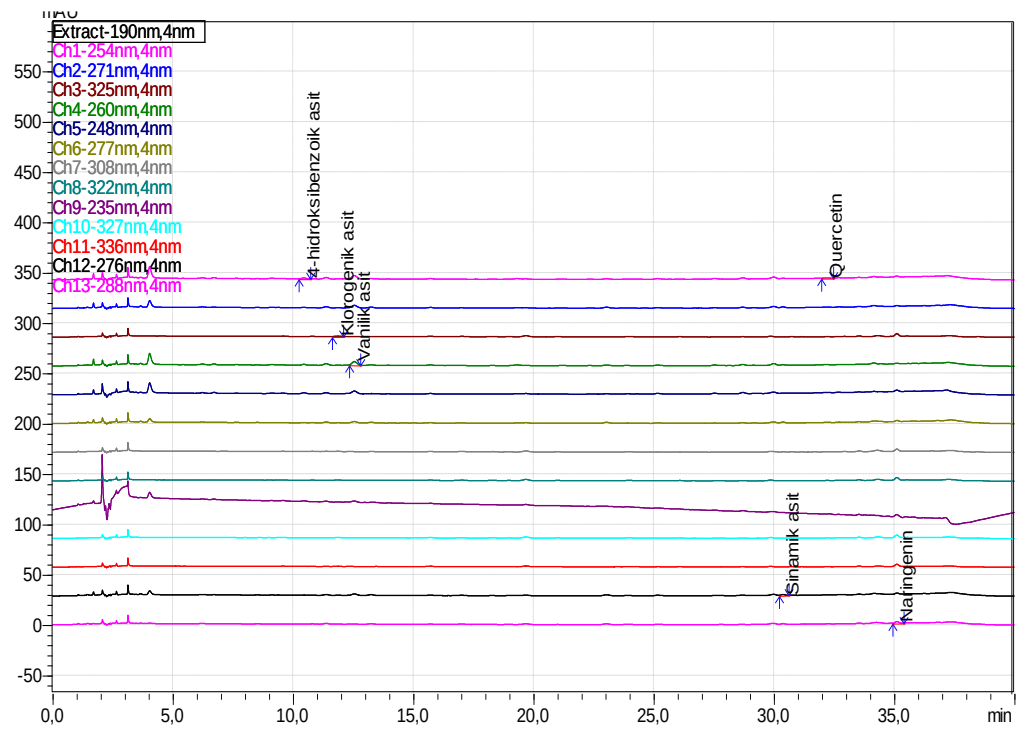


Figure 67: HPLC chromatograms of phenolic compounds in fractions of free alkaloids extract

Resultas and Discussion

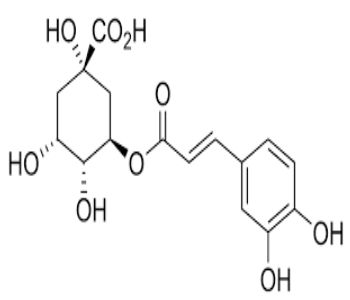
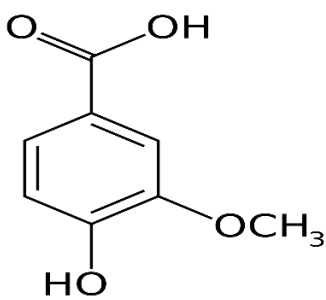
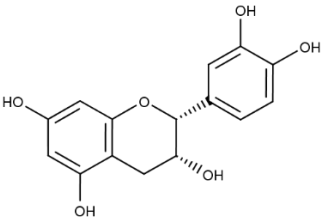
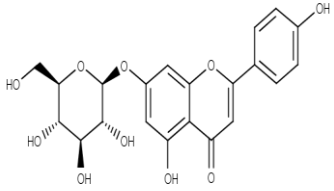
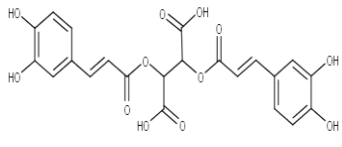
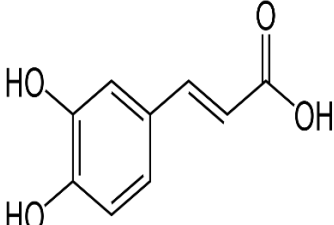
		
Chlorogenic Acid	Vanillic Acid	Epicatechin
		
Apigenin-7-glucoside	Chicoric acid	Caffieic acid

Figure 68: The main phenolic compounds identified in several fractions

Table 22: HPLC profile of phenolic compounds in free alkaloid extract/fractions

N	R.T	compound	Concentration mg/kg MS				
			FAEDE-E	FAEDE-M	FAEA-M	FAEB-CN	FAEB-M
1	4.37	Galllik acid	/	/	/	/	0.016
2	10.39	4-Hydroxyibenzoic Acid	0.62	0.24	/	/	
3	11.71	Chlorogenic Acid	/	0.22	5.019	3.423	0.258
4	12.33	Vanillic Acid	2.21	1.31	/	/	/
5	13.26	Caffieic acid	/	/	0.714	1.531	/
6	14.18	Epicatechin	/	/	1.142	8.149	/
7	18.57	p-coumaric acid	0.12	/	0.048	0.126	/
8	19.78	Ferulic Acid	0.53	/		0.215	0.024

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9	24.8 0	Rutin	0.56	/	0.344	/	/
1 0	27.1 6	Chicoric acid	/	/	1.631	2.081	/
1 1	27.5 1	Apigenin-7-glucoside	0.30	/	0.395	1.089	4.681
1 2	30.3 5	Cinnamic Acid	0.34	0.14	0.923	/	/
1 3	32.2 1	Quercetin	/	0.31	0.365	2.693	0.504
1 4	34.8 5	Naringenin	/	0.57	/	/	/
Total compound			7	6	9	8	5

The results show the presence of fourteen phenolic/flavonoid compounds (Gallik acid, 4-Hydroxyibenzoic Acid, Chlorogenic Acid, Vanillic Acid, Caffiec acid, Epicatechin, p-coumaric acid, Ferulic Acid, Rutin, chicoric acid, Apigenin-7-glucoside, Cinnamic Acid, Quercetin, and Naringenin) distributed between FAEDE-E, FAEDE-M, FAEA-M, FAEB-CN, and FAEB-M fractions of free alkaloid extract (Figure 64- 86) with a different concentrations, with the epicatechin was observed as a main compound in FAEB-CN fraction. While Gallik acid and Naringenin were identified particularly in FAEB-M and FAEDE-M fractions respectively (Table, 22) (Figure 69).

The plant contain an important polyphenol, flavoind contents in different extracts/ fractions that what was explained by our results, where was identified a number of phenolic compounds with an important concentration. These results were in agreement with the study of Lilya et al., (2018) they revealed that the aqueous extract of *Deverra scoparia* Coss.& Dur. contained bioactive phenolic compounds such as salicylic and rosmarinic acids as well as apigenin which possess analgesic and anti-inflammatory properties.[79]. While Touil et al., (2006) reported that tamarixetin-3-O-glucoside, apigenin-6,8-di-C-glucoside (vicenin-2), isorhamnetin- 3-O-rutinoside and isorhamnetin-3-O-glucoside were isolated and identified from *P. Chloranthus*[142]. On the other hand, rutin has previously been reported to occur in the flowers of *F. vulgare* a plant from Apiaceae in addition to six flavonol glycosides [143]. From the n-butanol extract of *P. scoparius* stems four compounds were isolated such as D-mannitol[144] . In addition, the study of Djeridane et al., (2008) isolated one flavone from *D. scoparia*[93]. The fruits and leaves of *F. vulgare* have been shown to contain several flavonols (quercetin, isoquercetin, kaempferol 3-glucuronide, kaempferol 3-arabinside) and *Pimpinella anisum* has been shown to contain rutin, isoorientin and isovitexin[145]. Quercetin and apigenin appeared to be the main flavonoid aglycones and rutin was one

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of the major quercetin glycosides in the extracts of *A. sylvestris*[146]. *A. graveolens* was reported to contain the flavonols rutin and quercetin[147]. identified six flavonoids in the extracts and exudates of Ri T-DNA – transformed root of *D. carota*: three flavonols (kaempferol, quercetin, rutin) and three flavones (apigenin, chrysin, and luteolin). Data in the present study are in accord with previous studies and extend the observation of quercetin, together with its glycosides, rutin and quercetin 3-O-glucoside, to seventeen additional species of Apiaceae[148].

III.5. Isolated compounds

III.5.1. Identification of the compound B1-1

The column chromatography separation of the B fraction allows us to obtain 6 regrouped sus-fractions The B1 was obtained as a yellow fraction followed by precipitation with n-hexane to obtain a white crystal B1-1 Figure 70. The identification result of B1-1 sus-fraction by a GC-MS analysis (Figure 71) shows B1-1 as a fatty acid compound “Nonacosan-10-one”.

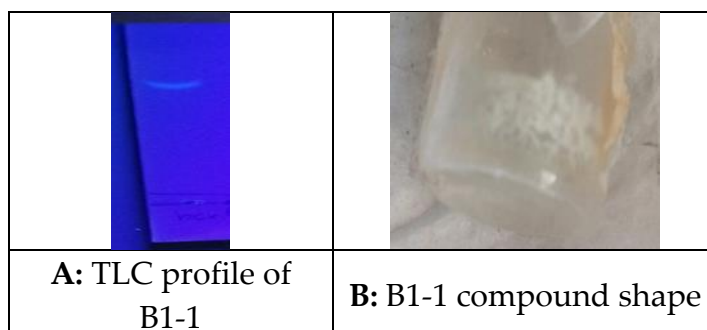


Figure 69: The isolated compounds B1-1

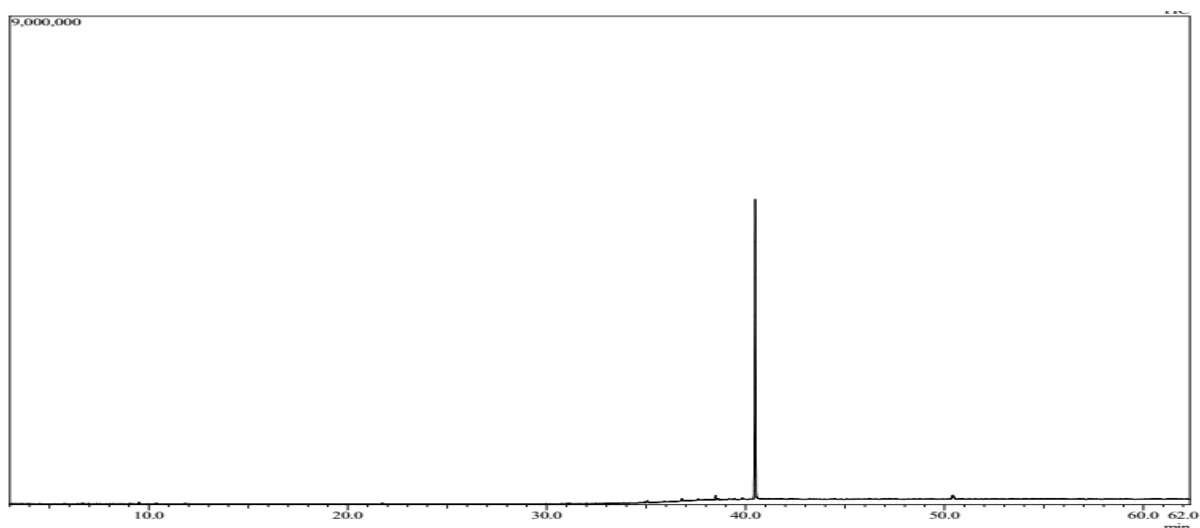


Figure 70: GC-MS Chromatogram of compound B1-1

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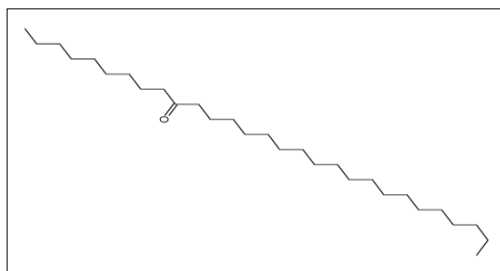
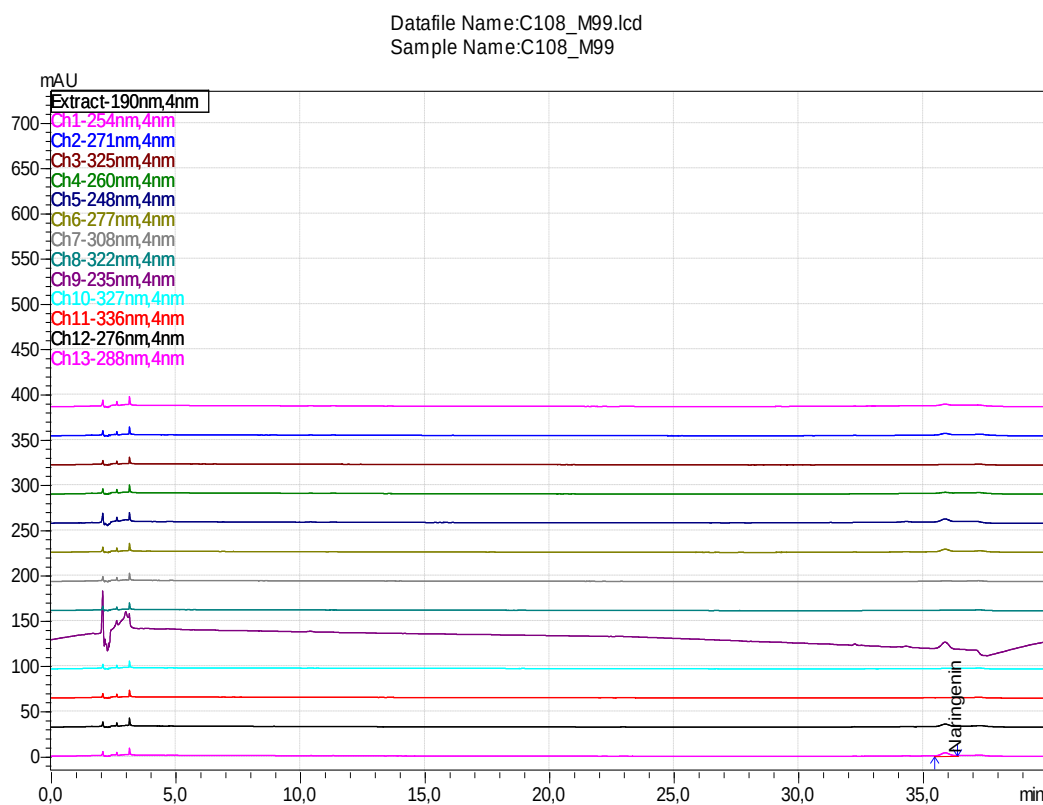


Figure 71: The structure of Nonacosan-10-one compound

III.5.2 Identification of the compounds C99-108 p

A white amorphous compound was identified by HPLC analysis (Figure 73) as a naringenin compound 906.31 mg/g MS (Figure 74, Table 23) isolated from a C99-108 M fraction.



Resultas and Discussion

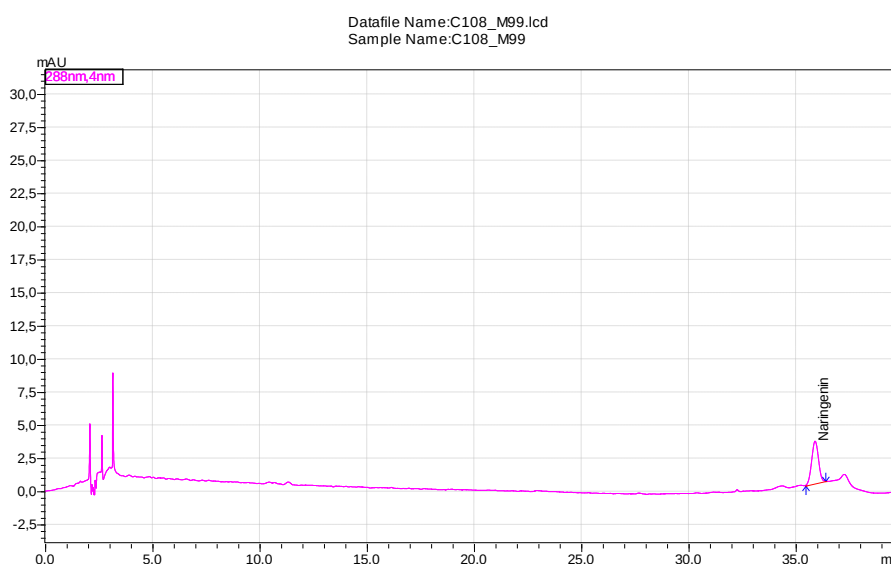


Figure 72: The HPLC chromatogram of the isolated compound C99-108 p

Table 23: The identification of C99-108 p

N	R.T	compound	mg/g
1	35,910	Naringenin	906.31

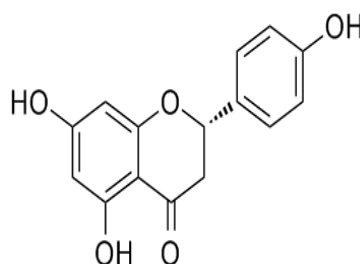


Figure 73: The Naringenin structure

III.5.3. Identification of the compound MAAE-FCR

A white crystal isolated MAAE -FCR after separating a precipitate from MAAE-M, washed with MeOH, identified as Apigenin-7-glucoside 4.362 mg/g MS a pure compound by HPLC analysis (Figure 75, 76).

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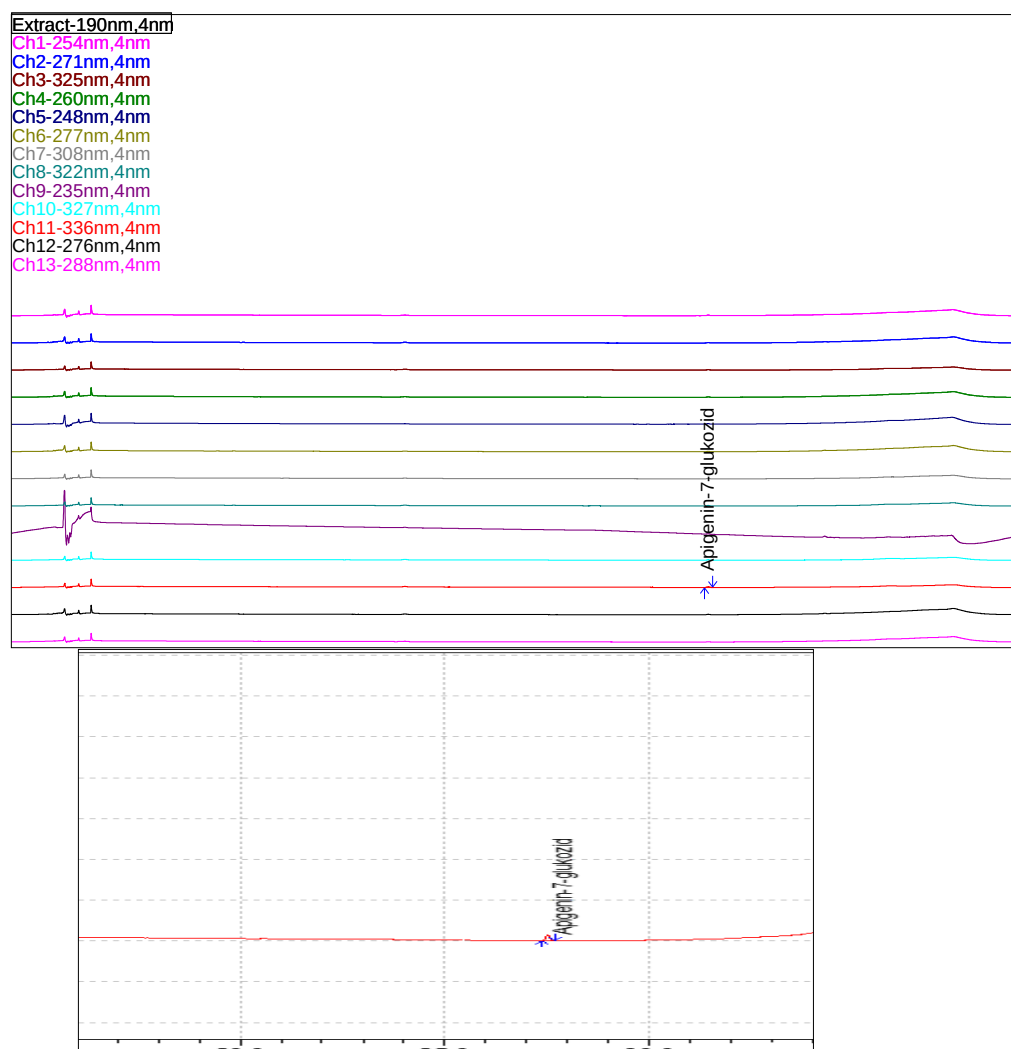


Figure 74: The HPLC chromatogram of the separate compound MAAE-FCR

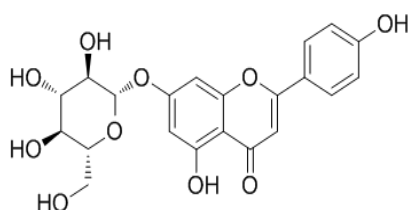
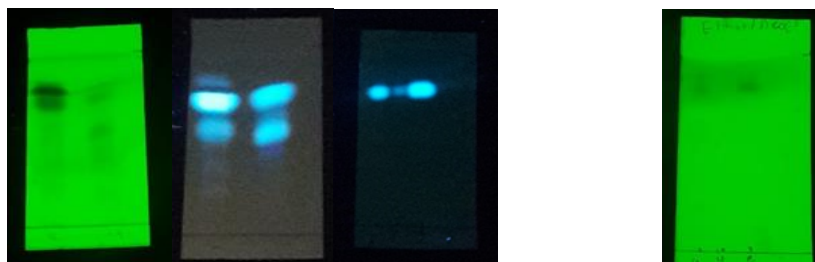


Figure 75: The Apigenin-7-glucoside structure

Resultas and Discussion

I. The SFC2 and SFC4

The SFC2 and 4 were separated by PTLC to isolate 1 compound from SFC2, SFC2-1, and two compounds from SFC4, SFC4-1, and SFC4-2 (**Figure 77**).



A: SFC2, 4 Before PTLC (254, 365nm) **B:** SFC4-1, 4-2, and SFC2-1(254, 365nm)

Figure 76: The TLC profile of SFC2 and 4 before and after separation and purification “ QF”.

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III.6. Pharmacological screening

In the present research, the antioxidant, antidiabetic, anti-urease, and insecticidal activity *in vitro* of the different extracts of the alkaloids of the investigated medicinal plant *Deverra scoparia* were evaluated with a view to possible valorization.

III.6.1. Antioxidant activity

Antioxidant activity is defined by the power of all bioactive compounds, whether extracted or pure compounds, to inhibit free radicals or block oxidation. Different methods estimate antioxidant activity. These methods are based on the staining and discoloration of the solution at a well-defined wavelength. In the present study, the antioxidant potential was evaluated using DPPH, phenanthroline, and β -carotene methods.

III.6.1.1.DPPH assay

The antioxidant potential of the alkaloidal extracts of *Deverra scoparia* and that of the BHA and BHT controls was evaluated by the DPPH H' free radical scavenging method. The latter is a stable radical that can be reduced by a compound with antioxidant properties, which results in a discoloration measurable with a spectrophotometer at 517 nm. This test makes it possible to determine the capacity of the extracts to scavenge DPPH and to compare it with that of the controls used BHA, and BHT. The results obtained for the different alkaloidal extracts are expressed as inhibition percentages of the DPPH radical as a function of each fraction concentration (Figure 78). The values obtained will be expressed as the 50% radical inhibitory concentration (IC₅₀) from the contents of the fractions tested and represented in the form of a histogram (Figure 79).

The evaluation of the DPPH free radical scavenging test gives the results (figure 79, Table 24) showing that the tested fractions have an efficiency to scavenge the DPPH radical, however, the percentage of radical inhibition varies according to the fraction studied. The fractions of the acid alkaloid extract have the highest percentages of the order of 85.28 and 86.9 % for both fractions, followed by the fractions of free alkaloid extracts FAE with a percentage of 80.57 and 85.39 % compared to controls BHA and BHT with a percentage of 87.13 and 94.97 % respectively at a concentration of 200 μ g/ml. On the other hand, the fractions of the volatile alkaloid extract AV have the lowest inhibition rates of 30.12 and 37.51%.

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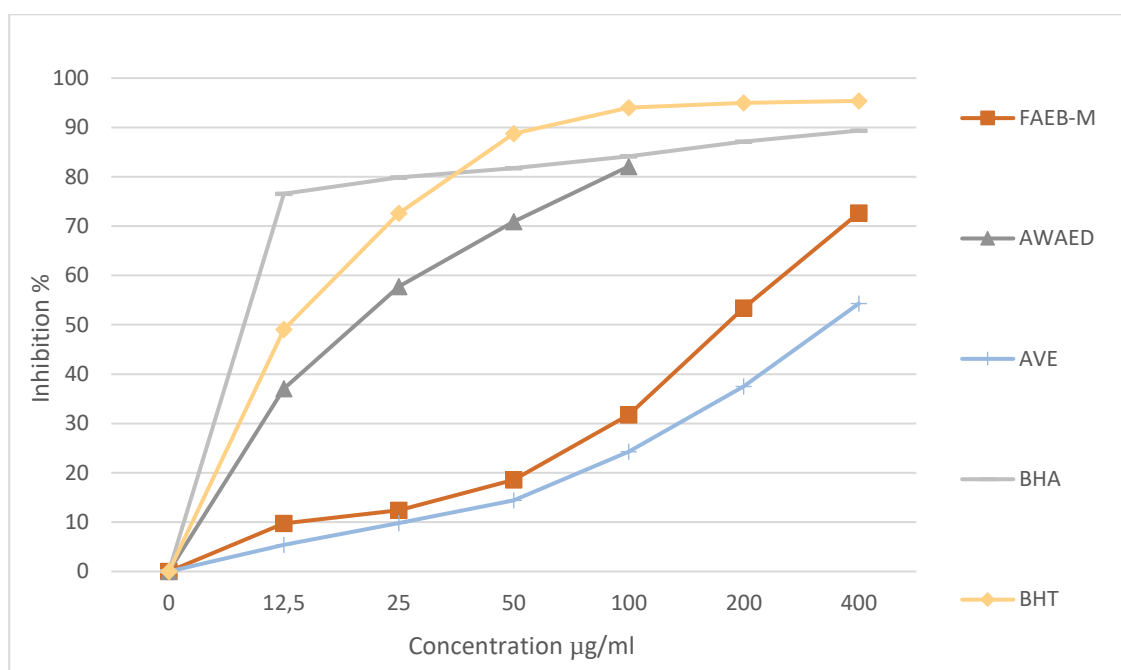


Figure 77: DPPH inhibition rate with different concentration

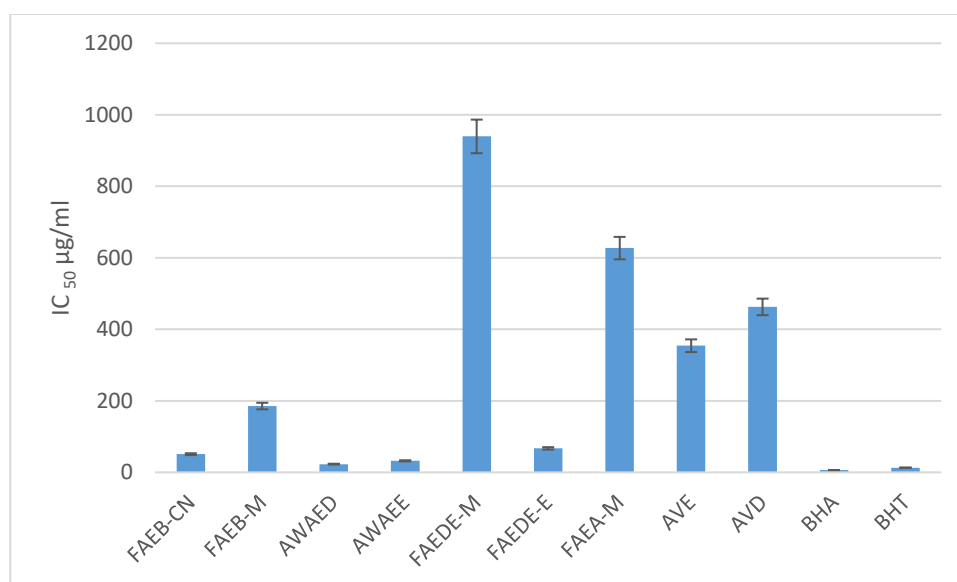


Figure 78: The IC₅₀ DPPH of different fractions

AVD; alkaloid volatile DCM fraction/ **AVE**; alkaloid volatile Et₂O fraction/ **AWAEE**; acidic water alkaloid extract Et₂O fraction/ **AWAED**; acidic water alkaloid extract DCM fraction/ **CA**; crude alkaloid extract/ **EA**; ethanol alkaloid extract/ **FAEDE**; free alkaloid extract with DCM-EtOH/ **FAEA**; free alkaloid extract with ACoET/ **FAEB**; free alkaloid extract with BuOH and **MAAE**; acidic alkaloid extract with MeOH.

The results showed that, compared to the control studied with high antioxidant power, the fraction with the highest antioxidant activity is that of the fractions of the acidic alkaloid extract; AWAED (IC₅₀ = 23.01± 0.38 µg/mL), AWAEE (IC₅₀ =

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32,21± 1,32 µg/mL), in comparison with BHA and BHT (IC₅₀ = 6.14± 0.41 and 12.99 0.41 µg/mL). The Fractions of the free alkaloid extracts FAEB-CN and FAEDE-M presented a remarkable IC₅₀ equal to 50.89 ± 0.70 and 67.15 ± 2.23 µg/mL, respectively. Finally, the fractions of the volatile alkaloid extract AVE and AVD (IC₅₀ = 354.24 ± 2.20 and 462.76± 2.38 µg/mL) in order (Figure79).

III.6.1.2. Phenonthroline (PHT) assay

The ability of *D. scoparia* to reduce the Fe³⁺ ion using the phenanthroline assay is shown in Figure 80. The antioxidant activity of fractions determined by this method decreases in the following order: BHA (A_{0.5} = 2,01± 0,16 mg/mL) > AWAED (A_{0.5} = 8,28±0,79 mg/mL) > BHT(A_{0.5} = 8,82± 0,87 mg/mL) > AWAEE (A_{0.5} = 14,51±0,48 mg/mL) > FAEB-CN (A_{0.5} = 16,93±0,30mg/mL) > FAEB-M (A_{0.5} : 43,47±1,15 mg/mL) > FAEA-M (A_{0.5} = 43.65±1,14 mg/mL) > FAEDE-E (A_{0.5} = 59,31±1,03 mg/mL) > FAEDE-M (A_{0.5} = 59,64± 1,62mg/mL). Thus, the AWAED fraction showed the highest chelation activity (A_{0.5}: 8,28±0,79 mg/mL) even compared with BHT control, in addition, we see that all the tested fractions have a close chelation activity on each other (Figure 81, Table 24).

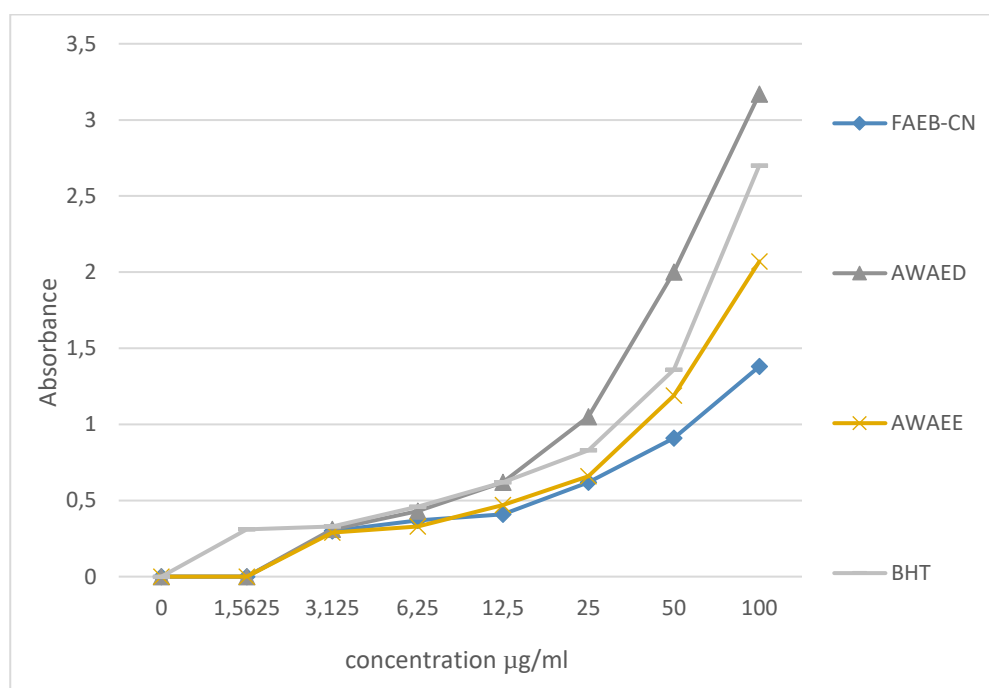


Figure 79: Phenanthroline absorbance with different concentrations

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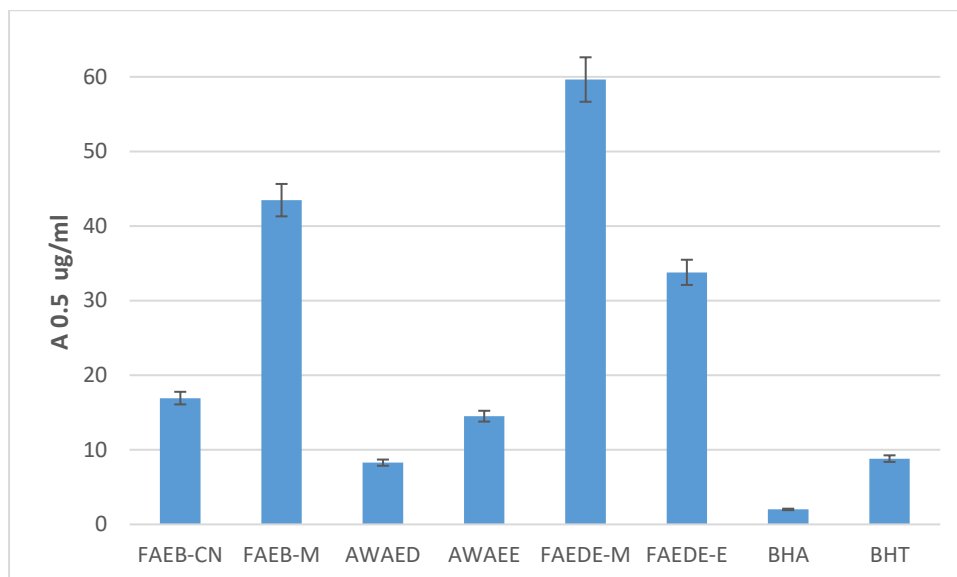


Figure 80: Phenanthroline A0.5 of different fractions

AVD; alkaloid volatile DCM fraction/ **AVE**; alkaloid volatile Et₂O fraction/ **AWAEE**; acidic water alkaloid extract Et₂O fraction/ **AWAED**; acidic water alkaloid extract DCM fraction/ **CA**; crude alkaloid extract/ **EA**; ethanol alkaloid extract/ **FAEDE**; free alkaloid extract with DCM-EtOH/ **FAEA**; free alkaloid extract with ACoET/ **FAEB**; free alkaloid extract with BuOH and **MAAE**; acidic alkaloid extract with MeOH.

III.6.1.3. β -Carotene bleaching by linoleic acid assay

The assay for β -carotene/linoleic acid establishes the ratios of inhibition for the oxidation of linoleic acid, which is utilized to investigate the capacity of various alkaloid extracts to impede lipid peroxidation.

Figure 82 shows the inhibition percentage results of the studied fractions of different extracts, BHA, and BHT controls. The inhibition percentage of fractions ranged from 39.57 to 72.76%, while the control BHT was 95.58% and BHA was 99.66%. The relatively highest antioxidant activity with a low IC₅₀ was obtained in the non-polar fraction of non-polar free alkaloid extract FAEDE-E equal to 52.51 ± 1.78 $\mu\text{g/ml}$, followed by the fractions of acidic alkaloid extract AWAE (E-D) and AVE fraction of volatile alkaloid extract (IC₅₀= $54,37 \pm 1,26$, 75.9 ± 2.63 , and 67.60 ± 2.10 $\mu\text{g/ml}$) respectively, compared to BHT and BHA as control (IC₅₀= 0.91 ± 0.01 and 1.05 ± 0.03 $\mu\text{g/ml}$). (Figure 83, Table 24)

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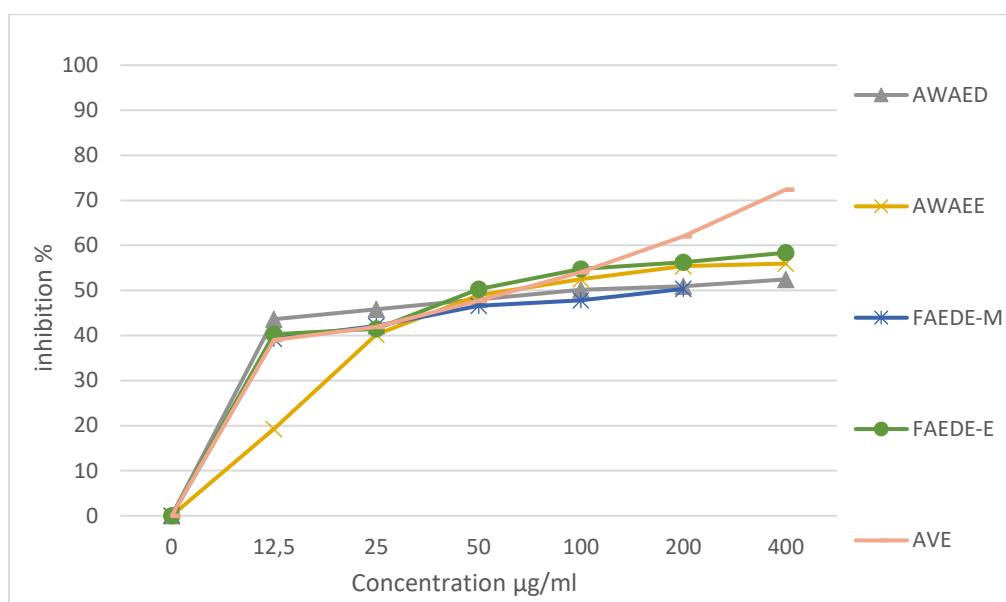


Figure 81: B-carotene inhibition rate with different concentrations

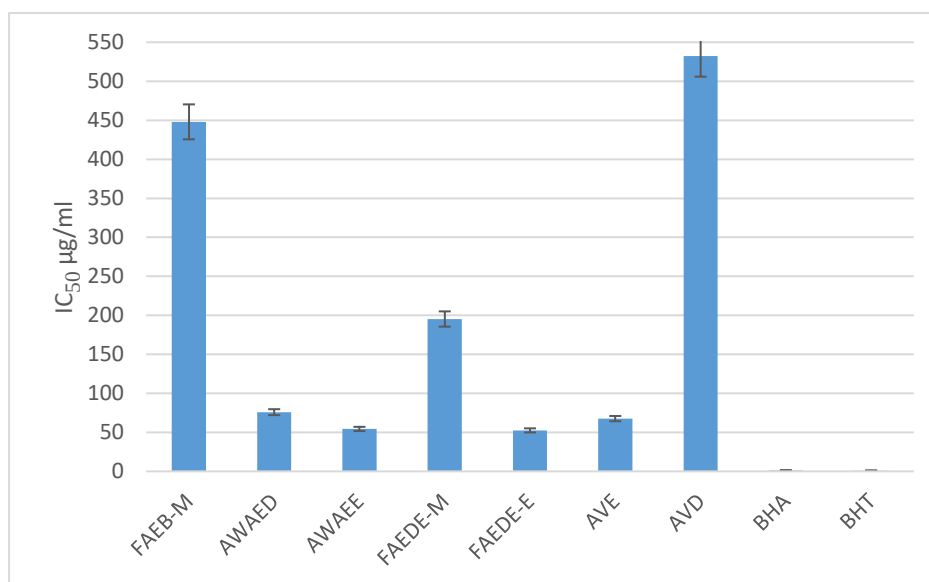


Figure 82: β -carotene IC₅₀ of different fractions

AVD; alkaloid volatile DCM fraction/ **AVE**; alkaloid volatile Et₂O fraction/ **AWAEE**; acidic water alkaloid extract Et₂O fraction/ **AWAED**; acidic water alkaloid extract DCM fraction/ **FAEDE**; free alkaloid extract with DCM-EtOH/ **FAEA**; free alkaloid extract with ACoET/ **FAEB**; free alkaloid extract with BuOH.

Different antioxidant activity methods can be categorized into various mechanisms, such as evaluating lipid peroxidation, radical scavenging, binding of transition metal ion catalysts, and reducing capacity. Accordingly, we combined a variety of complementary assay methods to evaluate the antioxidant effect. The results from the methods above showed that AWAED and AWAEE

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fractions exhibit potential antioxidant activity because of the increasing presence of phenolic and flavonoids. Since the intimate relationship between antioxidant activity and total phenol content has been discovered [88], [93].

The O-phenanthroline assay can be measured by its Fe (III) reducing power, resulting in an intense red color. The AWAED fraction showed the highest activity of *D.scoparia*, indicating its potential as a Fe³⁺ ion-reducing agent. It is worth noting that our study is the first to explore this aspect currently.

The rapid discoloration of β -carotene is due to the absence of antioxidants. Our results showed that IC₅₀ values of FAEDE-E, and AWAEF fractions indicated a powerful protector on β -carotene. This may be due to various antioxidants: those neutralizing peroxy radicals, generated free radicals of linoleate, and other compounds in the system. In the present study, FAEDE-E, and AWAEF fractions indicated the presence of some phenolic compounds with a remarkable concentration and were more attracted to the polarity of solvents, which slowed reduced lipoperoxidation, accordingly, the oxidation of β -carotene. Where there's no previous study based on the estimation of the β -carotene-linoleic acid system for the alkaloid extracts of *D. scoparia*, while a few reports evaluated the essential oil potential [78].

Benarfa et al., (2020) reported that the potential of bioactive compounds was influenced by the extraction time and extraction temperature all of these affected the antioxidant activities results [149]. On the other hand, chlorogenic acid was reported as an essential compound due to its multiple biological properties including antioxidative, antihypertensive, antibacterial, anti-tumor, and anti-inflammatory properties and its ability to resist HIV-1 [150], where it noted that the lipophilicities of the chlorogenic acid derivatives were found to be positively correlated with their antioxidant activities [151]. Both alkaloids and biogenic amine were reported with their remarkable antioxidant potential [152], [153], [154] such as atropine and berberine chloride were noted as an important source of oxidative stress potential [155], [156].

III.6.2. Anti-Diabetic (α -amylase inhibition)

Monitoring postprandial blood glucose levels using naturally sourced carbohydrate hydrolyzing enzyme inhibitors is considered an economically interesting therapeutic approach in the treatment of type 2 diabetes since the use of synthetic drugs is often associated with gastrointestinal side effects [157].

Most studies on plant extracts or phytoconstituents that may contribute to the treatment of diabetes have focused on the inhibition of α -amylase, which catalyzes the digestion of carbohydrates into glucose [52].

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To find a natural inhibitor of α -amylase, we studied the effect of different alkaloid extracts of medicinal plants on activity at varying concentrations, to evaluate the inhibition of α -amylase, starch was chosen as the most important substrate for the convenience of performing the assay by photometric methods. The Figure 84 illustrates the inhibition percentage of α -amylase in the different extracts studied.

To determine the inhibitory concentration of the enzyme activity at 50%, the IC₅₀ was calculated based on the inhibition percentage shown in the Figure 85.

The results show that the nonpolar fraction of nonpolar extract of free alkaloid and the AVD fraction showed a strong inhibitory activity with a low IC₅₀ (260.81±1.87 and 1630.69±1.41 μ g/ml, respectively) compared to the control used, acarbose (IC₅₀ = 3650.93±1.7 μ g/ml) on the other hand the nonpolar fraction of acidic alkaloid extract shows activity with (IC₅₀ = 3730.90±0.58 μ g/ml) of close equality to the control studies (Figure 85, Table 24).

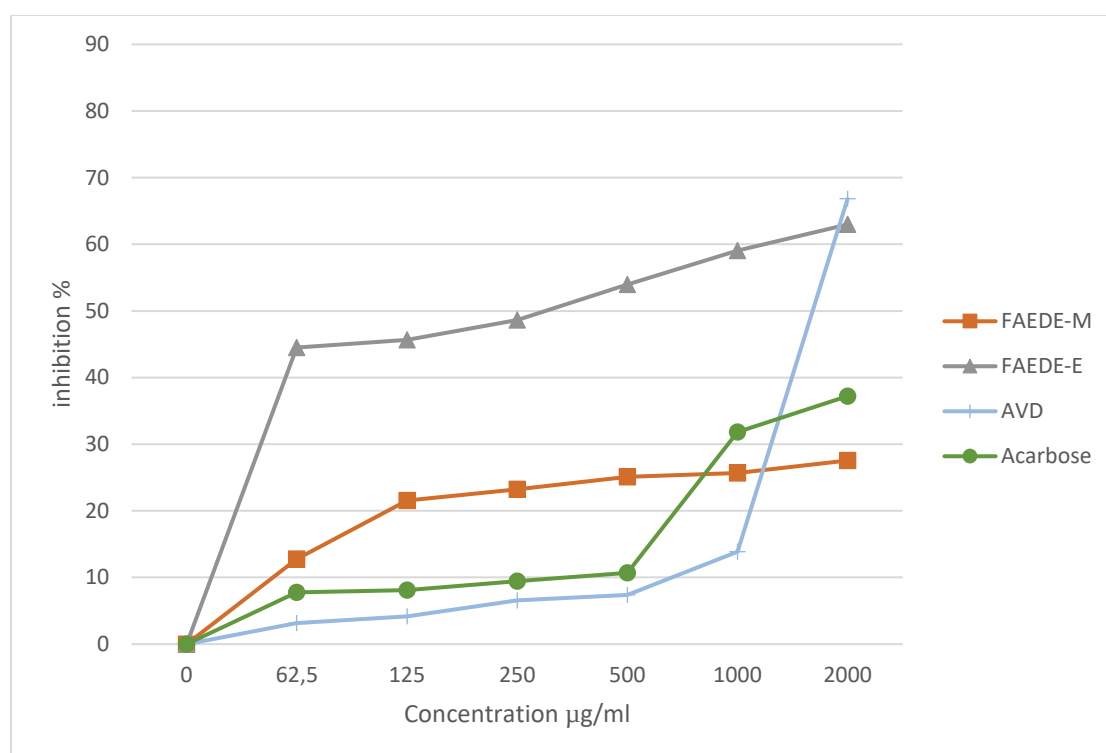


Figure 83: a-amylase inhibition rate of fractions with different concentrations

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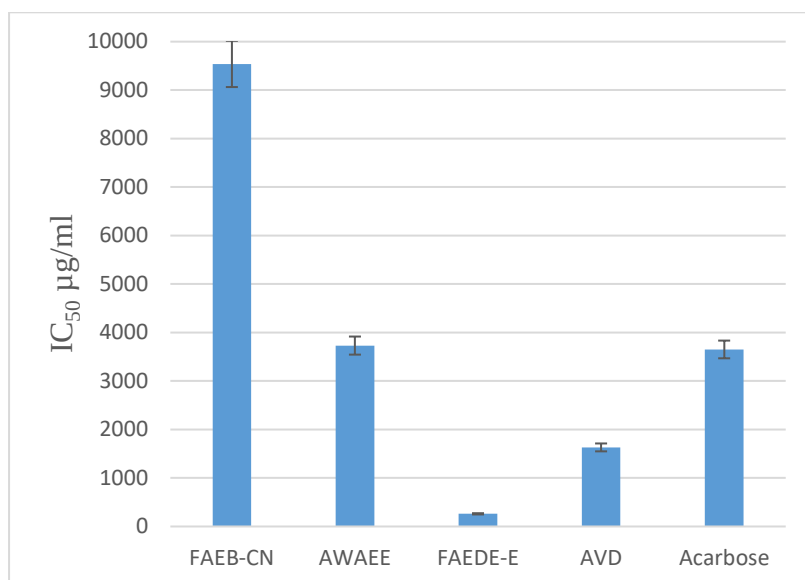


Figure 84: The IC₅₀ of different fractions

AVD; alkaloid volatile DCM fraction/ **AVE**; alkaloid volatile Et₂O fraction/ **AWAEE**; acidic water alkaloid extract Et₂O fraction/ **AWAED**; acidic water alkaloid extract DCM fraction/ **FAEDE**; free alkaloid extract with DCM-EtOH/ **FAEA**; free alkaloid extract with ACoET/ **FAEB**; free alkaloid extract with BuOH.

In the examination of *D.scoparia*, different alkaloid extracts can inhibit the α -amylase enzyme based on the potential of the alkaloid compounds on the inhibition of the enzymes[34], [158], which illustrates that alkaloid compounds showed important pharmacological properties such as phenyl ethyl amine alkaloid, piperidine, and indole which are identified by GC-MS analysis in AVD fraction. Furthermore, the anti-diabetic effect calculated in the FAEDE-E fraction was more powerful than that of acarbose, with an interesting inhibitory effect against α -amylase explaining its constitution presents bioactive compounds that assure this inhibition compared to other fractions, which are less or not active against this enzyme despite their high antioxidant activity. Moreover, some literature on *Pituranthos chloranthus*, *D. tortuosa*, recorded the anti-diabetic potent of this genus[159], [160], [161], as well as Tapan Behl et al., represented that alkaloid compounds were able to control diabetes [68] and other studies were noted that alkaloids exhibit an important potential to inhibit diabetic enzymes[111], [162], [163]. Hamdy Kashtoh et al.,(2023) reported that the isolated berberine from the *Cardiospermum halicacabum* plant was found able to retard the activity of α -amylase by 72% at 10 μ g/mL[164]. All these findings are extremely consistent with our results. On the other hand, these results can explain that the variation in chemical composition is strongly related to the pharmacological potential of fractions. Regarding that, it should be evaluated for the anti-diabetic potential of isolated alkaloids, which will be highlighted in this plant in future research. This is a

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confirmation of several studies, that evaluated the antidiabetic potential of some *Deverra* species and reported the importance of this species as a positive control of antidiabetic[165], [166], [167], [168], [169], [170].

III.6.3. Anti-Alzheimer (Butyrylcholinesterase inhibition)

The different alkaloid extracts of the plant include a remarkable inhibitory potential against butyrylcholinesterase compared to that of galanthamine (Figure 86, Table 24). In general, it is known that alkaloids are responsible for blocking the breakdown of enzymes in BuChE and AChE [171], [172].

The results for BChE inhibition were represented in (Figure 86) as inhibition percentage for all applied concentrations, and (Table 24) illustrates the IC₅₀ values. All alkaloid extracts/fractions inhibited BChE. The volatile alkaloid extract and acidic alkaloid extracts of non-polar solvents are the most potent inhibitors, the AWAEF inhibited BChE with an IC₅₀ value of 1.81 ± 0.87 µg/ml. The AWAED, AVD, and AVE fractions exhibited the most important activity with a better effect than galanthamine against BChE with an IC₅₀ <3.125 µg/ml with an inhibition percentage of 59.21, 66.64, and 67.71% respectively compared to galanthamine 52.26% at 3.125 µg/ml. These results agreed with the L. Gali et al., findings where the alkaloid extract exhibited an important potential against AChE and BChE compared to ethanol extract and fractions[173]. Sung-Woong Kim et al.,2013 reported that the essential oil of 10 species from Apiaceae their essential oil exhibits an important AChE inhibition on the adult rice weevil insect[174]. Indeed, the study based on the acetylcholinesterase inhibition of the alkaloid extract of leaves, fruits, root bark, and trunk bark shows that the alkaloid extract has an interesting inhibition potential[175].

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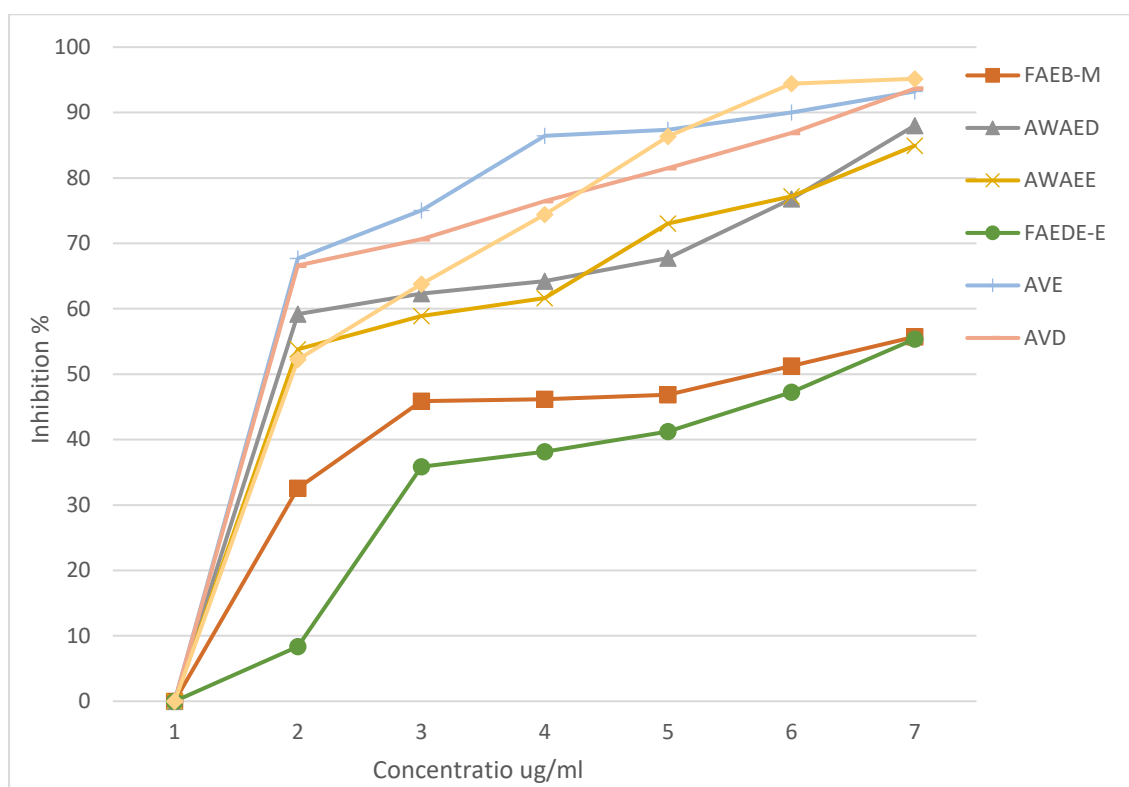


Figure 85: The BChE inhibition rate with different concentration

Table 24: The IC₅₀ of different study tests with different fractions and control

Fractions	IC ₅₀ µg/ml				
	Antioxidants			Enzymatic inhibition	
	DPPH	PHT	β-Carotene	α-amylase	BChE
FAEB-CN	50,89±0,70	16,93±0,30	>800	9540.16± 2.25	116.39± 0.55
FAEB-M	185,49±1,76	43,47±1,15	448.02± 1.75	>4000	43.52± 2.34
AWAED	23,01±0,38	8,28±0,79	75.9± 2.63	>4000	<3.125
AWAEE	32,21±1,32	14,51±0,48	54,37±1,26	3730.90± 0.58	1.81± 0.87
FAEDE-M	939.55±0.12	59,64±1,62	195,29± 2,49	>4000	>200
FAEDE-E	67,15±2,23	59,31±1,03	52.51± 1.78	260.81± 1.87	67.34± 1.71

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FAEA-M	627,24± 3,00	43.65±1,14	>800	>4000	>200
AVE	354,24± 2,20	NT	67.60± 2.10	>4000	<3.125
AVD	462,76± 2,38	NT	532.50± 2.34	1630.69± 1.41	<3.125
BHA	6.14±0.41	2,01± 0,16	1.05±0.03	NT	NT
BHT	12.99±0.4 1	8,82± 0,87	0.91±0.01	NT	NT
Acarbose	NT	NT	NT	3650,93±10,7 0	NT
Galantamine	NT	NT	NT	NT	1.33± 0.93

AVD; alkaloid volatile DCM fraction/ **AVE**; alkaloid volatile Et₂O fraction/ **AWAEE**; acidic water alkaloid extract Et₂O fraction/ **AWAED**; acidic water alkaloid extract DCM fraction/ **FAEDE**; free alkaloid extract with DCM-EtOH/ **FAEA**; free alkaloid extract with ACoET/ **FAEB**; free alkaloid extract with BuOH.

As a consequence of the pharmacological potential of the studied fractions, we note that the fractions of volatile and acid extracts had a high inhibitory capacity compared to control extracts, and this capacity could be due to the high amount of alkaloids in the volatile fractions, in addition to the number of compounds they contain. This was confirmed by the results obtained by quantitative and qualitative determination. While for acid fractions, despite the low alkaloid content, there is a strong inhibition capacity, which is explained here by the presence of a high-efficiency compound or group of compounds, despite their low concentration, their presence alongside other compounds allows them to show a synergistic effect between them.

III.6.4.Sun protection factor SPF

Sunscreens protect the skin from UV radiation using various organic and inorganic filters. Inorganic filters, such as particulate oxides, reflect or scatter UV radiation. Organic filters, which are aromatic compounds, absorb high-energy photons from UV radiation. When these molecules absorb energy, they become excited, and upon returning to their ground state, they release this energy as heat or light in a non-harmful wavelength. Both organic and inorganic filters are commonly found in commercial sunscreens to enhance their effectiveness. In recent years, a new range of hypoallergenic cosmetics has been developed to

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cater to individuals with sensitive or allergic skin, aiming for more skin-friendly cosmetic actives[176].

The use of plant bioactives in cosmetic formulations is increasing due to the demand for new sustainable sources of raw materials. There are significant safety concerns and potential social impacts associated with the improper use of natural resources. Plant extracts have been extensively studied in various industrial sectors[32], [177], [178]. The potential use of Saharan plant extracts in cosmetic formulations has not been thoroughly explored yet. We evaluated the sunscreen potential of our study plant fractions by estimating their sun protection factor, considering their impact on public health and the possibility of using their extracts in cosmetics as bio-cosmetic sources. The SPF values of the *D. scoparia* alkaloid extracts were computed to assess their photoprotective efficacy. According to Mansur, et al., method, the values of 2–12, 12–30, 30–50, and >50 in the SPF rating are thought to correlate to low, moderate, and high levels of sun protection action, respectively[32]. The investigations' alkaloid extracts and fractions of *D. scoparia* have SPF values ranging from $9,45 \pm 2.14$ to $46,11 \pm 0.00$, as shown in Figure 87. These findings suggest that photoprotective properties are present in all alkaloid extracts and fractions. However, a part of these fractions/extracts exhibit interesting values compared to the other extracts, where they have a high photoprotective impact. In contrast, the free alkaloid FAEB fractions show a low photoprotective effect. The SPF values can be classified in their photoprotective potential as follows: FAEA-M ($46,11 \pm 0.00$), FAEDE-M ($46,11 \pm 0.00$) > AWAED ($45,82 \pm 0,50$) > AWAE (45,06 ± 1.82) > FAEDE-E ($32,49 \pm 0.03$). This important potential is in agreement with the study results recorded by Ahlem .,(2020) where the plant was found used in dermatology treatment with a high percentage of 30% compared to the other diseases of *D.scoparia* growing in Setif city[179][180].

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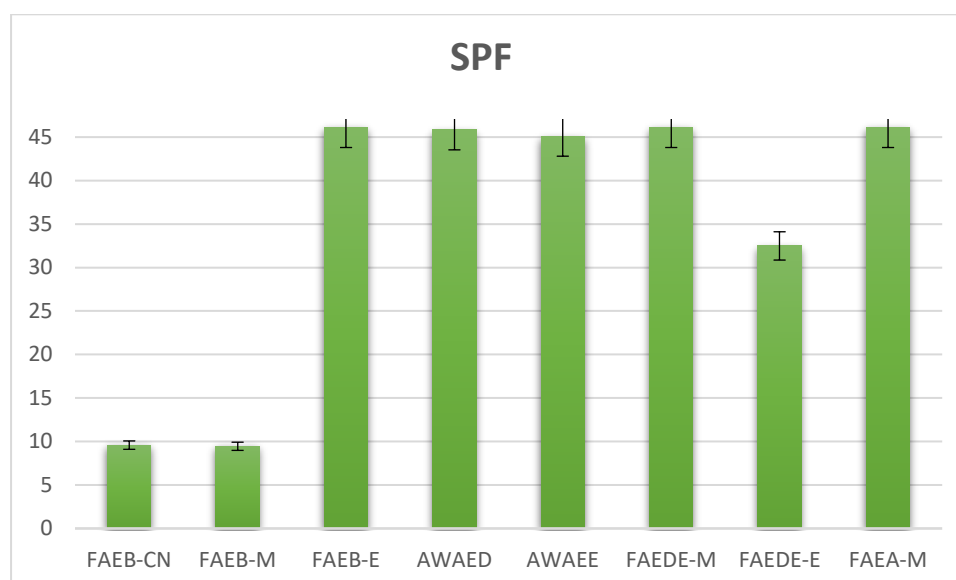


Figure 86: The calculated SPFs of different fractions

III.6.5.Bio-insecticidal assay

Insects are a major issue for agricultural industries as they cause damage to crops during growth and after harvest. Numerous studies have been carried out to address this problem. Synthetic chemicals, commonly used as insecticides, can be harmful to the environment, living organisms, and humans who consume contaminated food[181]. Scientists are now focusing on natural products as potential solutions, aiming to develop non-toxic and environmentally friendly insecticides[182]. The effect of the alkaloid extract and its fraction was calculated After 24h of the experience the results were expressed by the mortality rates related to the five tested concentrations to determine the lethal doses (50 and 99%) LD₅₀ and LD₉₉ on the insect population of the white scales.

Five concentrations for each fraction were used for the test. Figure 88 shows that there is a proportional correlation between the mortality rate and the five concentrations where it represents that the highest mortality rate for all fractions mentioned is the concentration 5 mg/ml which is the highest concentration, the same correlation is shown in the probit regression line corresponding to the percentages of the corrected mortalities as a function of the logarithms of the concentrations in the applied fraction figure 89. The LD₅₀ and LD₉₉ of the white scale population for the raw extract are 1.50 ±0.22 and 4.08 ±0.58 mg/ml respectively whereas for the different fractions, the LD₅₀ values are between 1.60 ±0.33 and 1.50 ±0.22 mg/ml. The D fraction has the lowest intake concentration LD₅₀ = 1.50 ±0.22 mg/ml followed by the F fraction. While the LD₉₉ we have the E

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fraction have the lowest concentration equal to 4.08 ± 0.58 mg/ml, all the lethal dose values are reported in (Figure 90).

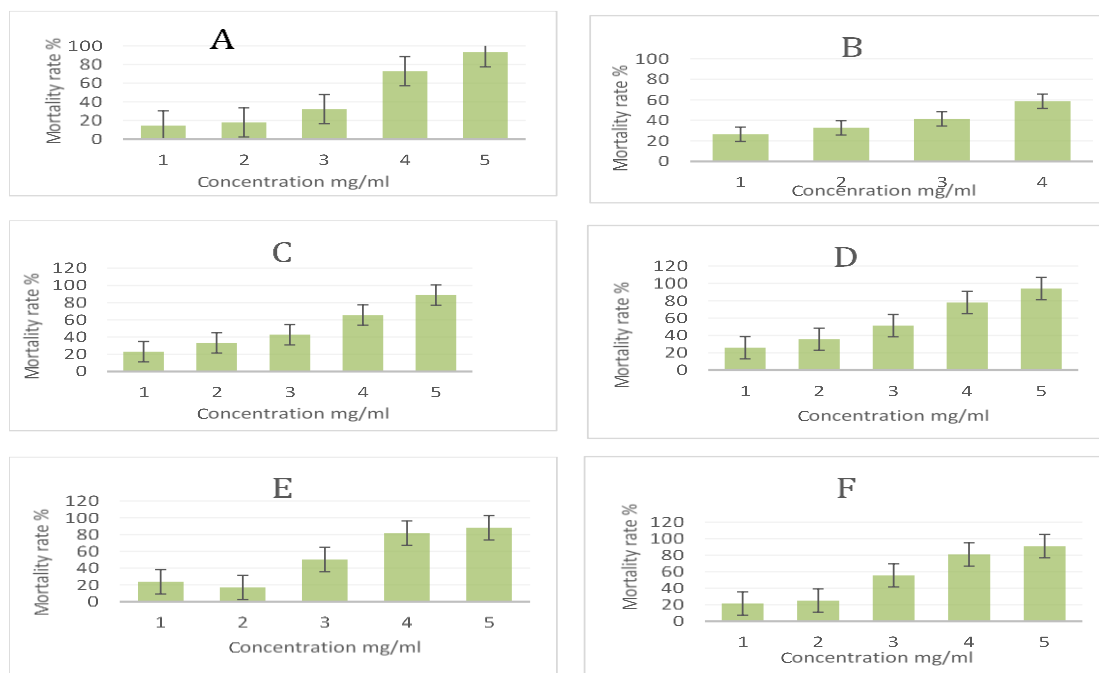


Figure 87: The mortality rate of WS insects with different concentrations and fractions.

A: crud alkaloid extract, B: fraction 100% CH₂Cl₂, C: fraction 25%-75% (MeOH-CH₂Cl₂), D: fraction 50%-50% (MeOH-CH₂Cl₂), E: fraction 75%-25% (MeOH-CH₂Cl₂), F: fraction 100% MeOH.

-CH₂Cl₂: Dichloromethane / -MeOH: Methanol.

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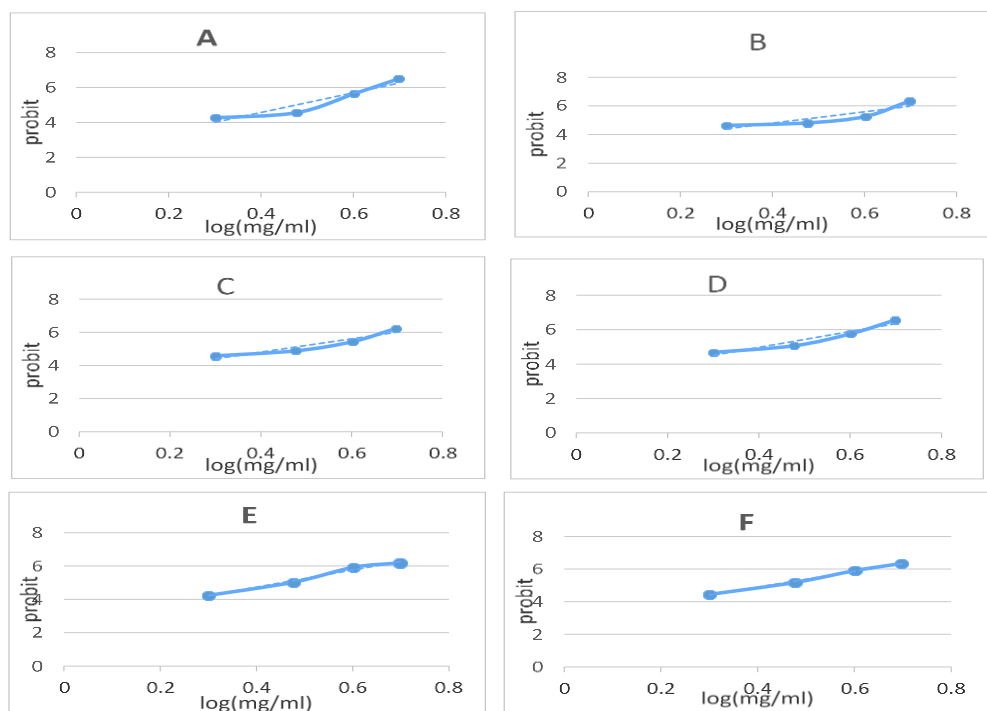


Figure 88: Probit as a function of applied concentrations

*A: crud alkaloid extract, B: fraction 100% CH₂Cl₂, C: fraction 25%-75% (MeOH-CH₂Cl₂), D: fraction 50%-50% (MeOH-CH₂Cl₂), E: fraction 75%-25% (MeOH-CH₂Cl₂), F: fraction 100% MeOH.

-CH₂Cl₂: Dichloromethane / -MeOH: Methanol.

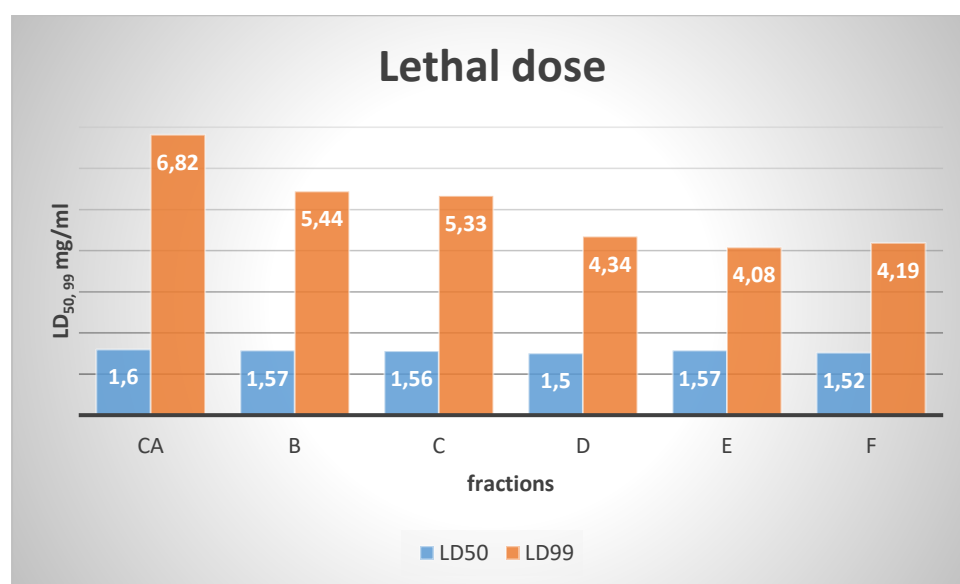


Figure 89: the leathal dose of applied fraction

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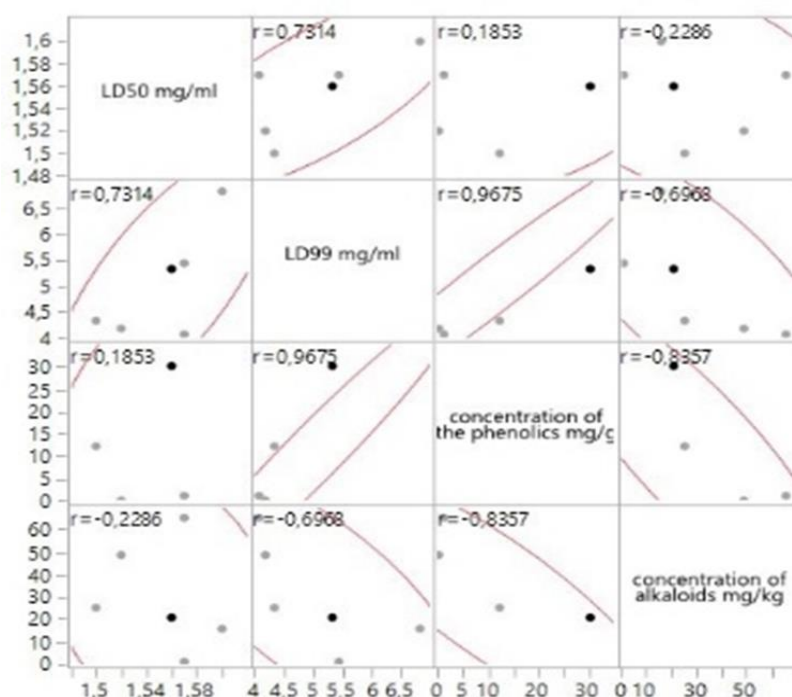


Figure 90: The correlation results of the LD₅₀, LD₉₉, phenolic, and alkaloid content concentrations.

According to the obtained results we mentioned that there is an observable insecticide activity of all the fractions, and this potential was related to the applied fraction's concentration positively each time the concentration is increased the mortality rate is increased too, with an observed correlation between the lethal dose and phenolic and alkaloid concentration figure 91 and a height mortality rate was registered at 5 mg/ml concentration. The E fraction showed the presence of alkaloids with a strong concentration according to the other secondary metabolites compared with the other fraction, which increases the insecticidal potential of E because of possible synergistic effects between the alkaloids that increase their overall toxicity[183].

Based on the acaricidal potential of the essential oil constituents from *D. scoparia* (Tunisia), the study evaluated that all oils' constituents present a synergy effect, increasing the acaricidal property more than the individual compound. Where the toxicity of blends of selected constituents indicated that the presence of all constituents was necessary to reproduce the toxicity level of the natural oil.[83]. The E fraction showed the presence of alkaloids with an intense concentration and a higher atropine concentration according to the other secondary metabolites and compared with the different fractions. This makes the insecticidal potential of E increase cause of the possibility of synergistic effects between the alkaloids that increase their overall toxicity [183]. While the remarkable mortality rate of the crude extract compared with the other fractions shows that, the compounds in this extract have an antagonistic effect on each

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other. Based on the results of our study we believe that there are residues with different concentrations depending on the polarity of the solvents used. This explains the difference of LD₅₀ of each fraction compared with LD₉₉ also which in turn affects the selected insects to study.

The [184] reported the 24 species extract from the Apiaceae family were tested for their potential as insecticidal and fungicidal activities while giving a remarkable potential with a high percentage against insects. It also showed the high potential of the alkaloid and phenolic compounds as a bio insecticides with percentages equal to 30% and 20% respectively. That was confirmed with the identification analysis of the alkaloids and phenolic compounds in the fractions extract of the plant *D. scoparia*. Whereas fraction D showed very significant effects due to its containment of the three alkaloid compounds in addition to the epicatechin compound, this gave fraction D the lowest value of the LD₅₀ compared to the rest of the fractions. While atropine and epicatechin are known for their ability to kill insects, the study of [45][185]. about the epicatechin potential confirmed that the high ability of the antioxidant and insecticidal activity of the ethanolic extract is due to the high phenolic content, which was characterized by the presence of epicatechin, which gave the extract this ability, which makes it a compound of high importance in the pharmaceutical and agricultural industries. While the presence of apigenin in the E fraction in addition to the high alkaloid content showed its significant effectiveness by giving the lowest value for the concentration in LD₉₉ compared to the other fractions besides the crude extract. Where the compound apigenin is considered a compound with anti-oxidant, anti-inflammatory activity, antimicrobial, and chemoprevention [186], [182],[187], and it is a common compound between fractions D and E, also A which is the crude extract that contains all the compounds combined, in which there is an interaction between them in addition to the considered alkaloid content This explains the values obtained between LD₅₀ and LD₉₉. The polar extract of total alkaloid from *Cynanchum mongolicum* (Asclepiadaceae) plant showed an important insecticidal alkaloid, the alkaloid Antofine N-oxide mentioned as a toxic one among the three alkaloids isolated, the study effect of these three alkaloids against *Spodopteralitura* (Kaltenbach) showed an inhibition in the development of insect and stomach toxicity [188].

The extract of the root bark of *T.wilfordii* (Celastraceae)hook f. after fractionated to give seven sesquiterpenes alkaloid 1-desacetyl wilforgine and 1-desacetyl wilforine reported as an insecticidal for the first time, this study carried out against larvae of *C. pipiens pallens*coquillet and adults of *M. domestica*, the wilforine alkaloid mentioned as a main insecticidal constituent among the other alkaloids isolated [189].

The two-isolated alkaloid from the n-butanol fraction of polar extract from *Macleayacordata* (Papaveraceae) seeds plant showed a remarkable decrease in the

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survival of aphids *Aphis gossypii* Glover, the presence of these alkaloids in the extract plant and the alcohol extract of *M. cordata* make it reported to be toxic to several insects such as the mosquito. Therefore, the stronger insecticidal activities of the sub-fractions over those of the isolated alkaloids might be due to the synergistic or additive effects of all of the alkaloids included in the seeds of *M. cordata* [190]. Alkaloids, terpenes, phenolic, and flavonoids were reported with their interest in insecticidal potential evaluated from several plant families such as apiaceae[184].

The bioactive compounds contained in the study plant *D. scoparia*, are illustrated by the presence of volatile compounds such as a biogenic amine[191], [192], [193] with terpenes, oxygenated terpenes, and fatty acids,[84], [172], [194], [195], in addition to the existence of phenolic and flavonoid compounds such as chlorogenic acid, epicatechin, vanillic acid, naringenin, apigenin, and eriocitrin,[5], [196], [197], [198], [199], [200], moreover, the alkaloid compounds like atropine, 4-amino antipyrine, and berberine[201], [202], [203], [204] all these bioactive compounds can produce their pharmacological potential individually or have synergistic effects in each other evaluate the uses of the plant.

Conclusion

Conclusion

Conclusion

The study of alkaloid extracts of the plant *D. scoparia* in the south Algerian Sahara «Hoggar» in the Tamanrasset region leads to the following conclusions: The maximum extraction yield of alkaloids was obtained with acidic alkaloid extract with polar solvent ethanol EA (11.46 %), while the fraction AVD of the volatile alkaloid extraction gave the lowest yield (0.0078%). The total alkaloid content (TAC) of the plant extracts was measured using the bromocresole colorimetric method. TAC range between 1645.83 and 62895.83 mg/kg DS with the higher content registred for the fraction AVD of volatile alkaloids extract 62895.83 mg/kg DS.

In the chemical study, we used different methods to extract alkaloids, separation, and identification. Using open-column chromatography and several chromatography techniques and methods revealed the presence of the volatile alkaloids, fixed alkaloids such as 2-(tetrahydrofuran-2-yl) piperidine, triethanolamine (Sterolamide), 3-methyl-2-propanoyl-1H-Indole, N2-[(2,4-dichloro phenoxy)acetyl]- L-Lysine, [(2-fluorophenyl)methyl] 1H-Purin-6-amine, 1-decyl-1H-imidazole-2-methanol, atropine, berberine chloride, 4-amino antipyrine, and phenolic compounds; Epicatechin, Apigenin-7-glucoside, Chlorogenic acid, Vanillic Acid and Naringenin, their identified by the uses of chromatographic analysis HPLC chromatograms and GC/MS spectra and comparing with literature data.

In the pharmacological study, we investigated *in vitro* antioxidant activity of plant alkaloid extracts. The fractions of acidic alkaloid extract AWAED and AWAEF showed significant antioxidant activity. They were found to be effective scavengers against DPPH radicals with IC₅₀ values of 23.01± 0.38 and 32.21± 1.32 µg/ml for AWAED, and AWAEF respectively, and interested IC₅₀ values of 8,28±0,79 and 14,51±0,48 respectively for the phenanthroline test. Conversely, the fraction of the free alkaloid extract FAEDE-E and the acidic fraction AWAEF presented their effective potential to inhibit linoleic acid oxidation with IC₅₀ values 52.51± 1.78 and 54,37±1,26 in order.

The *in vitro* antidiabetic activity of *D. scoparia* revealed a significant effect of the FAEDE-E fraction of the free alkaloid extract and the AVD fraction of the volatile alkaloid extract for inhibition of the α -amylase enzyme with an IC₅₀ equal to (260.81 1.87 µg/ml) and (1630.69 1.41 µg/ml), relative to acarbose (3650.93 µg/ml), respectively. The FAEDE-E fraction exhibits an important inhibition of α -amylase enzyme estimated fourteen times higher than positive control galantamine.

Conclusion

The fractions of volatile plant alkaloid extract appear to have a very high inhibitory effect against butyrylcholinesterase $IC_{50} < 3.125 \mu\text{g/ml}$ compared to galanthamine compared to the other fractions tested.

The study fractions exhibit an important protection capacity from the sun light such as the fractions of the acidic alkaloid and the free alkaloid extracts, presented protection was determined .

The evaluation of the bio-insecticidal capacity of the fractions of the basic alkaloid extract (non-polar solvent) against *Parlatoria blanchardi* insect at different concentrations presented the ability of the fractions to control the growing insectes.

Our results confirm that *Deverra scoparia* is an important source of antioxidant, antidiabetic, anti-Alzheimer, and insecticidal agents and could be used as a bio-resource. Further phytochemical analysis is required to isolate the elements of the plant that show a broad spectrum of pharmacological activity. These results demonstrate the high pharmacological properties of the volatile alkaloid extract of *Deverra scoparia*, suggesting the importance of volatile alkaloid compounds and other nitrogen compounds in this study plant. However, the isolation and characterization of these compounds will help establish structure-activity relationships when discovering new drugs. These results might support some of the traditional uses of *Deverra scoparia*. Future studies will be *in vivo*.

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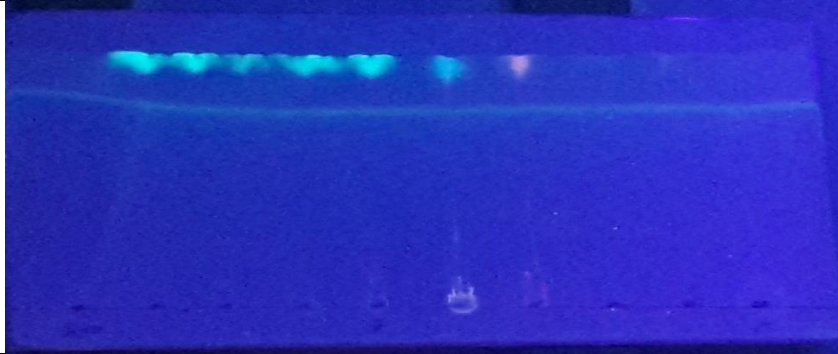
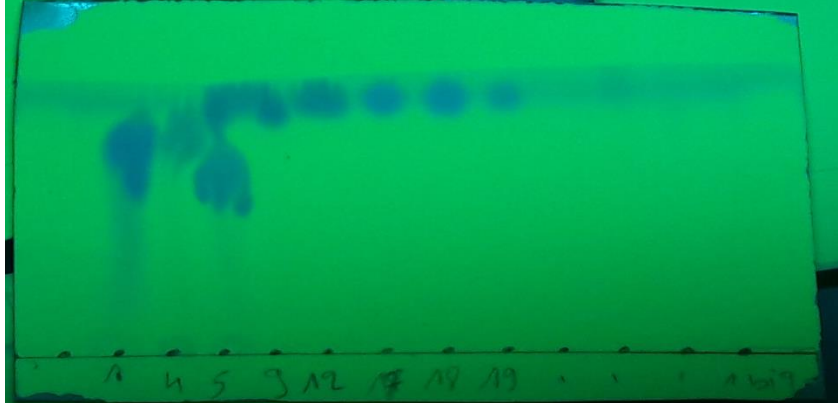
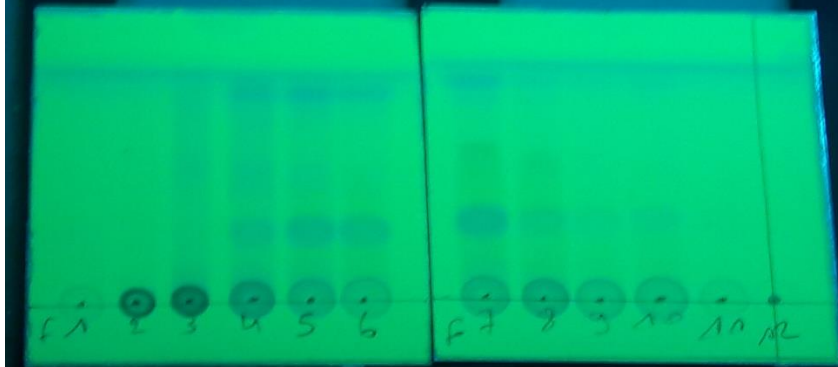
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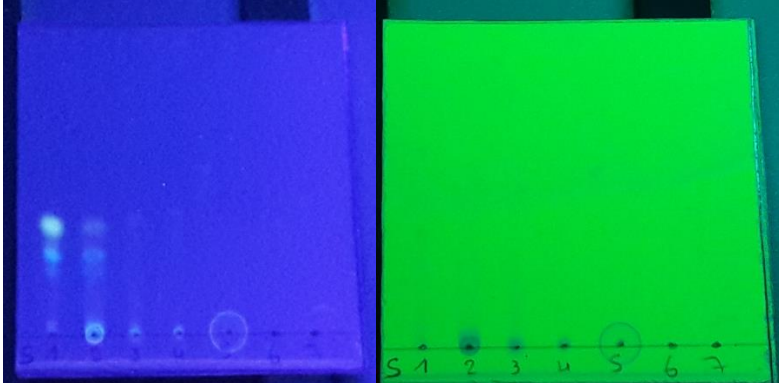
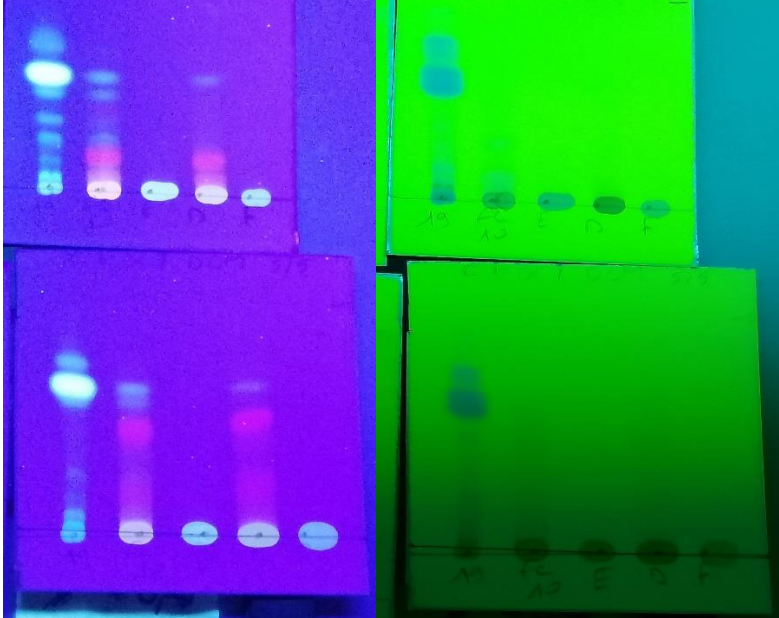
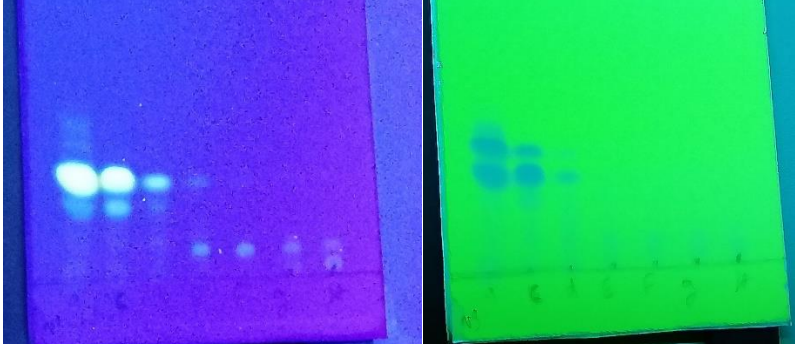
Annexes

Annexes

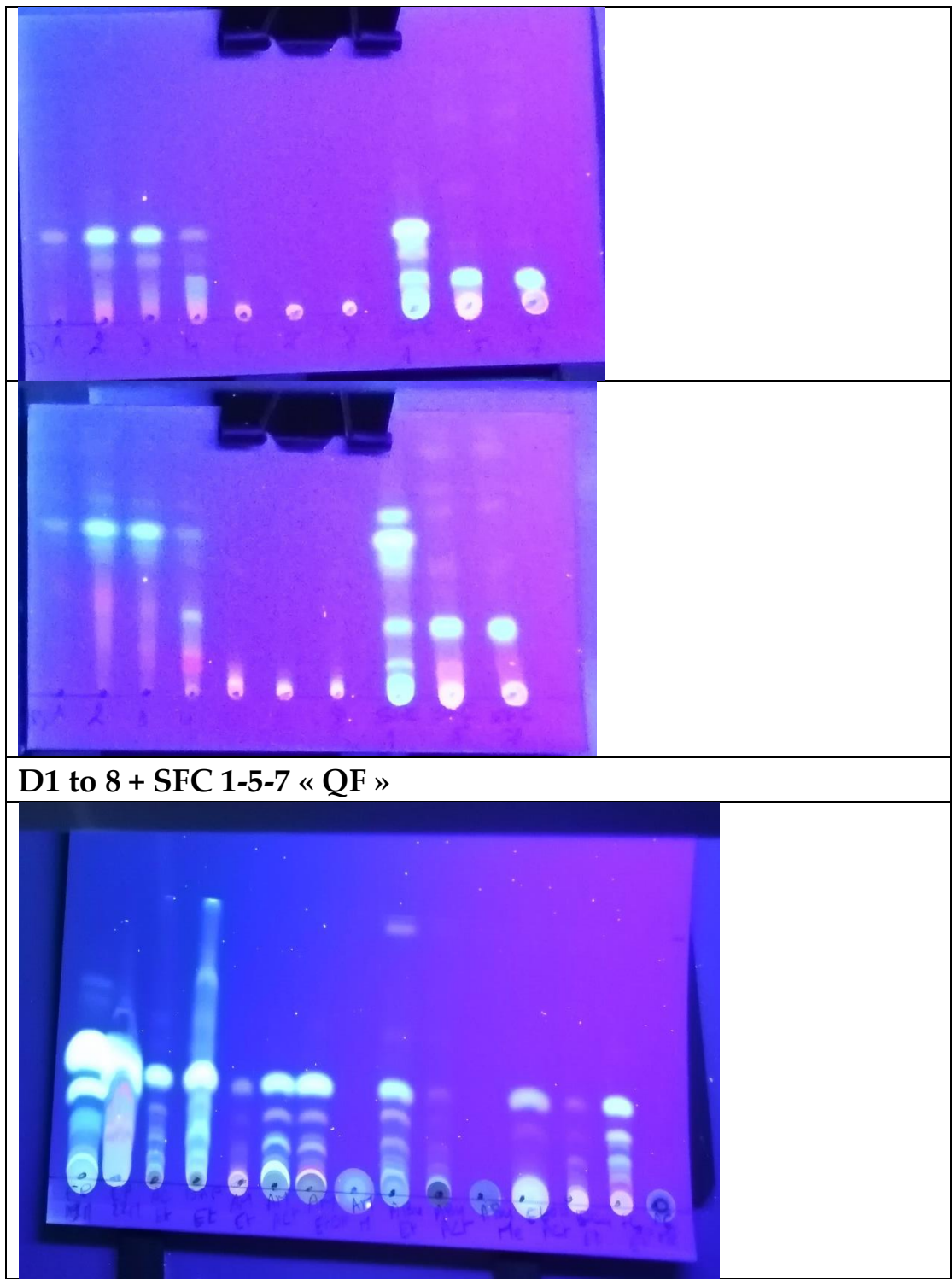
Some TLC profiles

	
	
B- hexane fractions (from column of silica-gel) "QC"	
	
	
B- p-hexane (from column of Sephadex) « QC"	

Annexes

	
C7-8 hexane (from column of Silica-gel) « QF''	
	
b19-Fc10-E-D-F 254 « QC »	
	
frac b19 « QF »	

Annexes



Annexes

