#### Резюмета на отпечатаните в пълен текст научни трудове

на

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Настоящата справка е съставена във връзка с участие в конкурс за заемане на една академична длъжност главен асистент по аналитична химия в област на висше образование 4. Природни науки, математика и информатика, професионално направление 4.2. Химически науки в сектор "Химия" на катедра "Химия и биохимия" при факултет "Фармация" обявен в ДВ бр.99/22.11.2024 г.

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# Synthetic Approaches toward the Benzo[a]quinolizidine System. A Review

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## Synthetic Approaches toward the Benzo[a]quinolizidine System. A Review

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#### Introduction

Over the recent years, the benzo[a]quinolizidine ring system  $\mathbf{1}$  (*Figure 1*) has become of great significance since it was found to be a key subunit in numerous natural and synthetic molecules possessing a variety of physiological activities or being promising candidates for such activity.<sup>1–10</sup>



Figure 1

The benzo[a]quinolizidine motif 1 is found in the alkaloids of the Schulzeines species (schulzeines A-C (2)) and Ipecac species (emetine (3), tubulosine (4)). (Figure 2) The schulzeines 2, isolated from the marine sponge Penares schulzei, are well studied and regarded as potential inhibitors of  $\alpha$ -glucosidases which may be crucial for treatment of diabetes, cancer and viral infections.<sup>11,12</sup> Emetine (3) has been used in traditional medicine, in the form of a root extract from Ipecac sp., for its antiparasitic and emetic activities. Later more intensive research was initiated to determine its ability to inhibit both ribosomal and mitochondrial protein synthesis and interfere with DNA and RNA synthesis. In their article "Biological Activities of Emetine" Akinboye and Bakare<sup>2</sup> showed the diverse biological properties of emetine (3) and some of its analogues, such

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# Physicochemical characteristics of seed oil of *Sambucus ebulus*, *Coriandrum sativum* L. and *Silybum marianum* L.

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The physicochemical properties of oil extracted from the seeds of *Sambucus ebulus, Coriandrum sativum L.* and *Silybum marianum L.* (0.5:1:1) were examined. The fatty acid composition of the oil mixture was found to contain oleic (51.35%) and linoleic acids (35.01%) predominantly. The characteristic peak in infrared spectrum at about 1740 cm<sup>-1</sup> was attributed to the C=O stretching in the carboxyl group of the fatty acids, oxidation products exhibit bands in 1730-1670 cm<sup>-1</sup> region. The O-H stretching of the monomer fatty acid appears at about 3550 cm<sup>-1</sup>, the signal around 3470 was attributed to the overtone of the CO stretching and the other peaks at 3535, 3621 and 3700 cm<sup>-1</sup> are related to the stretching vibration of the OH group. The oxidative stability index was determined to be 12.7 h, the total content of tocopherols is 1340 mg/kg and total content of sterols was found at about 0.46%. The plant sterols with highest content that were found in the oil were  $\beta$ -Sitosterol (58.4%) and  $\Delta$ 5-Avenasterol (19.3%). Two fluorescent signals were observed at 570 nm and 680 nm which were assigned to correspond to oxidation products and chlorophyll respectively. Concentration of the toxic elements Cd and Pb were below 5 ng.g<sup>-1</sup> and 50 ng.g<sup>-1</sup> respectively.

Key words: ATR FTIR spectroscopy, elements contents, fatty acids composition, fluorescence

#### **INTRODUCTION**

Non-traditional vegetable oils are increasingly used in traditional medicine due to their therapeutic properties unique phytochemical composition and antioxidant activity [1-4]. *Coriandrum sativum* L. has pharmacological applications due to its antioxidant [5], antidiabetic [6], anti-lipidemic [7] and antispasmodic [8] properties. The antioxidant properties of coriander are due to the high pigment content, especially carotenoids. The essential oil from coriander qualifies as a natural antioxidant [9].

The fruits of *Sambucus ebulus*, due to their high content of anthocyanins and polyphenols, have high antioxidant activity and have anti-cancer, immunostimulating, antibacterial, antiallergic, antiviral and anti-inflammatory properties [10] and their seeds can be used as raw materials for production of non-traditional oil. *Silybum marianum L*. is a herb, that has been used in European traditional medicine mainly for treatment of various liver diseases. [11]

The chemical properties and fatty acid composition for oils obtained from seeds of *Coriandrum sativum L.* [12], *Sambucus ebulus* [13] and *Silybum marianum L.* [14] were investigated by Nagella, Fazio and Khan.

The authors found no information about the elemental content, optical properties and DSC profile, which are connected with the presence of pigments and oxidative products. The main purpose of this work is to study the physicochemical properties of the oil.

#### MATERIALS AND METHODS Samples

The obtained oil was extracted from a previously prepared mixture of the seeds from *Sambucus ebulus* L, *Coriandrum sativum* L. and *Silybum marianum* L in 0.5:1:1 ratio respectively. This ratio dives the optimal fatty acid content in connection with the prospective use of the oil mixture as a dietary supplement.

The oil has been extracted with *n*-hexane in *Soxhlet* apparatus for 8 h. The solvent was partly removed in rotary vacuum evaporator, the residue was transferred in pre-weight glass vessels and the rest of the solvent was removed under stream of nitrogen to a constant weight to determine the oil content [15].

#### Used methods

Analysis of fatty acids. The fatty acid composition of oils was determined by gas chromatography (GC) after transmethylation of the respective sample with 20 g.kg<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub> in absolute CH<sub>3</sub>OH at 50°C [15]. Fatty acid methyl esters

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#### **CHARACTERIZATION OF OLIVE MEALS**

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#### ABSTRACT

The current communication describes results of olive meals characterization. The chemical composition in respect to nutrients (N, P, K, Ca, Mg, C), some microelements and heavy metals (Al, Co, Cu, Fe, Zn, Ni, Cd, Mn, Mo, Pb, Cr, As, Se, and Sr) is studied. FAAS, ISP-MS and ATR-FTIR spectroscopy methods are used. Parameters as moisture and contents of volatile compounds, raw oil, raw fibres, raw proteins, ash residues and insoluble ash are also examined. Adequate conclusions and recommendations in respect to olive meals utilization are advanced on the ground of the results obtained.

Keywords: olive meals; chemical composition; nutrients; FAAS, ICP-MS, ATR-FTIR spectroscopy.

#### INTRODUCTION

Olive oil production is one of the main agricultural branches in the Mediterranean region. The olive meal is among the by-products of the olive oil manufacturing process. It is obtained in large quantities. A few studies referring to this waste type have been carried out in comparison to the extensive one focused on meal residues from soybean (*Glycine max*), sunflower (*Helianthus annuus*), peanut (*Arachis hypogaea*) and cotton (*Gossypium*).

A recent study shows that olive meal could be added as a supplement to broiler feed with no harmful effect [1]. The high polyphenol content of the olive meal expressed mainly through hydroxytyrosol presence could be beneficial as an antioxidant supplement to animal fodder [2]. Olive meal and other by-products from olive oil production are a potential raw material for biofuels production [3]. Another alternative application of the olive meal could be referred to its utilization as a compost material. Composting is in fact regarded as one of the best waste management processes. Composts such as Mexican sunflower compost (MSC) and Cassava peel compost (CPC) have been successfully applied for the removal of lead and other heavy metals from soil [4]. Compost materials based on olive meals could be potentially used for soil amendment required by turfgrass cultures growing [5].

The aim of the present study is to evaluate the micro- and macro components, the organic and inorganic contents of olive meals as a preliminary stage towards its utilization as an additive to livestock fodder and compost materials.

#### **EXPERIMENTAL**

The olive meals used for the present investigation were obtained from a cold pressed olive oil installation in Aton, Greece. The sample collection and their handling were conducted according to BNS ISO 5500 and BNS ISO 5502 [6, 7].

The moisture and the volatile compounds content were determined according to the procedures described in the ref. [8]. The total ash content and the ash soluble in 10 % HCl were determined following the procedures Scientific Study & Research Chemistry & Chemical Engineering, Biotechnology, Food Industry

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ORIGINAL RESEARCH PAPER

## COMPOSITION AND PHYSICOCHEMICAL PROPERTIES OF SEED OIL OF RARELY GROWN VARIETIES OF GRAPES

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Abstract: The composition of grape seeds oil from a mixture of two varieties of red grape (Sangiovese and Pinot noir) from Varna region was investigated. Different analytical techniques have been used to determine the content of biologically active compounds, fatty acids, sterols and elemental compositions. It has been found that the oxidation stability of the oil is relatively low - about 4.5 hours. The fluorescence spectrum analyzed at an excitation wavelength of 350 nm contains peaks that are attributed to pigments, oxidizing products and vitamins. Transmission spectra were used to determine the content of  $\beta$ -carotene, chlorophyll and color characteristics in the CIELab colorimetric system. Infrared spectroscopic experiments (ATR and permeability) were used for confirmation of the fatty acid profile of the analyzed oil. The results show that this oil is a good source of various healthy nutrients.

**Keywords:** *chemical composition, color characteristics, fatty acids, sterols, fluorescence spectra, grape seed oil, IR spectra* 

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#### COMPARATIVE PHYSICOCHEMICAL ANALYSIS OF OILS DERIVED FROM NIGELLA SATIVA AND CORIANDRUM SATIVUM L

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#### ABSTRACT

The black cumin (Nigella sativa) and coriander (Coriandrum sativum L) are well-known representatives from Ranunculaceae and Apiaceae families, respectively. The aim of the present study is to evaluate the physicochemical properties of oils derived from black cumin and coriander. Their visible and fluorescent spectra have been recorded. The fatty acid profile for both oils has been obtained, indicating the predominant constituent of the oil to be linoleic acid (C 18:2) in 58.2 % and 42.29 %, respectively. The oils have a high content of polyunsaturated fatty acids,  $\gamma$ - and  $\alpha$ -tocopherols. Oxidative stability of coriander oil is lower (8.2 h) compared to that of the black cumin oil (12 h). Differential Scanning Calorimetry (DSC) was used to measure the enthalpy changes and phase transitions in both oils samples. The concentrations of some elements (essential and toxic) in the oils were determined after acidic decomposition by using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The low content of Pb, As and Cd makes the oils safe to be used.

<u>Keywords</u>: DCS analysis, fluorescence spectrum, colour characteristic, FT-IR spectrum, elemental analysis, vegetable oils.

#### INTRODUCTION

The oil and seeds from black cumin have been known for their physiological activity such as immunomodulating, antimicrobial and antioxidant properties and their effective use in treating respiratory conditions. The oil from coriander seeds has been known to alleviate rheumatoid, joint and gastro-intestinal conditions. The oil seeds from these two plants are important sources of oils of nutritional, industrial and pharmaceutical importance [1]. They have important meaning for human nutrition and health, because they contain valuable amounts of fat-soluble bioactives [2, 3].

The different parts of coriander are still used in folk medicine. Coriander is a commonly used domestic remedy, valued especially for its effect on the digestive system, treating flatulence, diarrhea and colic. It settles spasms in the gut and counters the effects of nervous tension. Beside, coriander fruits are also used as a diuretic by boiling equal amounts of coriander fruits and cumin seeds, then cooling and consuming the resulting liquid. The oil from its seeds is an ingredient in cosmetics, creams, lotions, emulsifiers, soaps, shampoos and detergents imparting a distinctive essence to the products [4, 5]. Black cumin seeds are used in medical applications [6, 7]. The cold pressed oil from seeds of black cumin is also in use for preparation of functional cosmetic and dietary products [6, 8].

The authors found no information about the element content, optical properties and DSC profile, which are connected with the presence of pigments and oxidative products. The main purpose of this work is to study the

# Fluorescence Spectra and Chemical Composition of High Oleic Sunflower Oils with Herbs Oil Additives

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Abstract. Fluorescence spectra and chemical composition of cold-pressed sunflower oleic oils with addition of oils from: 1) basil (Ocimum basilicum), 2) rosemary (Rosmarinus officinalis) and 3) oregano (Origanum vulgare) were studied. The aim of the study is to compare chemical composition of cold pressed oil with and without addition of different plant oils. The concentrations of some elements (essential and toxic) in oil were determined by using Inductively coupled plasma mass spectrometry (ICP-MS) after microwave digestion. Infrared spectroscopic experiments (ATR and transmittance) and NMR spectrum were used to study the fatty acid profile of the analysed oils.

#### 1. Introduction

Basil, rosemary and oregano have been known and used since ancient times as spices in food to improve the aroma and for medicinal purposes. In recent decades, their essential oils have been of great interest as they are a source of various natural products.

Various synthetic antioxidants are often used to prolong the storage and stability of industrially processed foods. For example, butylated hydroxytoluene (BHT) is widely used as an antioxidant in food, but some laboratory animal studies have questioned its safety [1]. Today, food consumers are increasingly concerned about the negative effects of synthetic additives on their health. That leads to an increasing need to find pure natural products with proven qualities to replace them [2, 3]. Because herbs are rich in antioxidants, vitamins, phenolic compounds, have antimutagenic, antibacterial and other properties, there is growing interest in their use as an alternative to synthetic additives. Oils of different types of herbs have low toxicity, are biodegradable, multifunctional and of low production value. Taking advantage of all these benefits can be a good start for their application in various fields - from agriculture and food production to medicine and pharmacy [4].

*Rosmarinus officinalis* L., family Lamiaceae, has a huge variety of applications in folk medicine. Infusions of stems and flowers are used to treat headaches and colds, as an analgesic and antispasmodic [5]. The leaves of this plant are considered to have one of the highest antioxidant activities [6]. Its essential oils have, in addition to antioxidant and antimicrobial properties against food pathogens [7, 8].

*Basil* (Ocimum basilicum L.) family Lamiaceae, one of the most popular herbs in cooking, is used both in flavouring and in food preservation [9]. In folk medicine, the anti-inflammatory, analgesic and

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# Capsaicinoids content in some Bulgarian varieties of *Capsicum annuum* L. obtained by RP-HPLC

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#### Abstract

Capsaicinoids are amides, a type of secondary metabolites in hot peppers, responsible for their hot taste also known as pungency. They possess many pharmacological properties with great potential for pharmacy like analgesic, blood glucose level reduction, insulin level improvement, reduction of triglycerides and cholesterol levels, etc. For the needs of this study a reliable and easy applicable RP-HPLC method with UV detection for determination of capsaicinoids was developed. Two traditional and two newly selected Bulgarian hot pepper varieties were studied. The concentrations of capsaicin and dihydrocapsaicin in the pericarp and the seeds were determined respectively. According to the Scoville heat unit (SHU) equivalence of the pericarps, the varieties Dzhulyunska shipka 1021, Zlatna shipka and Kehlibar show moderate pungency and present a potential value for the pharmaceutical and food industries. The current study contributes to the scientific database with regard to the pungency of *Capsicum annuum* L. varieties.

#### Keywords

capsaicine, dihydrocapsaicine, HPLC determination, Capsicum annuum

### Introduction

Chili peppers are world famous for their distinctive aroma, color and spiciness. They belong to the genus *Capsicum*, which comprises of some 30 species and more than 200 varieties. However, only five species are domesticated and these are *Capsicum annuum* L., *C. baccatum* L., *C. chinense* Jacq., *C. frutescens* L. and *C. pubescens* Ruiz &Pav. So far *C. annuum* appears to be the most economically valuable species with the largest distribution and demand worldwide (González-Zamora et al. 2013; Panayotov et al. 2017;

Batiha et al. 2020; Hernandez-Pérez et al. 2020). The chili peppers fruits are an important source of many health beneficial compounds such as ascorbic acid (vitamin C), carotenoids (provitamin A), tocopherols (vitamin E), phenolics, minerals, essential oils, etc. (González-Zamora et al. 2013; Hernandez-Pérez et al. 2020). Among these compounds the capsaicinoids particularly stand out for their unique distribution bound only to the *Capsicum* genus. They are synthesised and mainly accumulated in the placental tissue, but a substantial amount passes into the pericarp as well (Barbero et al. 2014). Capsaicinoids are

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#### Доклади на Българската академия на науките Comptes rendus de l'Académie bulgare des Sciences

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#### CHEMISTRY

Analytical chemistry

#### HPLC DETERMINATION OF BIPERIDEN IN SOLID DOSAGE FORMS

#### Trifon Milchev Angelov<sup>#</sup>, Biljana Slaveva Krastanova<sup>\*</sup>, Alexandar Svilenov Pashev, Maya Chavdarova Jotova

Received on September 6, 2022 Presented by B. Petrunov, Member of BAS, on December 20, 2022

#### Abstract

A rapid, new analytical method, based on liquid chromatography with UV detection, has been developed and utilised for the determination of biperiden hydrochloride in solid dosage forms. The chromatographic separation was performed on a conventional  $C_{18}$  reversed phase column with a mobile phase composed of acetonitrile-buffer mixture. Ultraviolet detection was carried out at 205 nm. A rapid extraction procedure using ultrasonic bath obtained good extraction yield values for the analyte ( $\geq 98.4\%$ ). The method was developed specifically for routine analysis of biperiden in solid dosage forms in the applicable concentration range. The linearity and range of the method investigated for the assay of biperiden is between 2–6 µg mL<sup>-1</sup>. The method is selective and sufficiently precise. Thus, the method developed is suitable for routine analysis of biperiden.

Key words: biperiden, HPLC-UV, assay, method development

**Introduction.** Biperiden is used to treat parkinsonism. It is a muscarinic receptor antagonist. Biperiden is also used in the treatment of arteriosclerotic and postencephalitic parkinsonism, and alleviates extrapyramidal symptoms induced by phenothiazine derivatives and reserpine.

Being a weak peripheral anticholinergic agent, biperiden has, therefore, some antispasmodic, antisecretory and mydriatic effects, and has a nicotinolytic activity [1,2].

 $<sup>^{\#} \</sup>rm Corresponding author. DOI:10.7546/CRABS.2023.02.02$ 

Nikolova K et al., JOTCSA. 2023; 10(2): 465-474.

**RESEARCH ARTICLE** 



#### Correlation between chemical characteristics and optical spectra of Spirulina commercially available on the Bulgarian market



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Abstract: The aggregate of various chemical substances useful for the functioning of the human body are known as nutrients. Spirulina has been present in human nutrition since ancient times, but in recent years the interest in it has been particularly increased due to the emergence of numerous alternative methods of nutrition. This study aimed to compare the functional and elemental composition as well as the optical properties of commercial Spirulina products available on the Bulgarian market. For this purpose, fluorescence spectroscopy in the ultraviolet and visible range, fourier transform infrared spectroscopy and inductively coupled plasma optical emission spectroscopy were used. The basic components of the analyzed Spirulina samples are proteins (1657 and 1537 cm<sup>-1</sup>) and carbohydrates (1069 and 1054 cm<sup>-1</sup>) and no meaningful differences between the IR spectra of the samples. Concentrations of important microelements Mg, Fe, Cu, Zn, and Mn varies with the manufacturer. The highest levels for Mg (6.69 g kg<sup>-1</sup>) were measured in samples from USA, while the *Spirulina* fabricated in Bulgaria exhibits the highest contents of Zn (242 mg kg $^{-1}$ ) and Cu (25.4 mg kg<sup>-1</sup>). All samples followed the tendency Mg>Fe>Mn>Zn>Cu. Making use of a fiber optic spectrometer the fluorescence spectra of the studied samples of Spirulina platensis for an excitation wavelength of 380 nm were measured. In these spectra we observe three fluorescence maxima: at 465 nm nicotinamide dinucleotide phosphate, 640 nm chlorophyll a, and 736 nm due to similar to chlorophyll pigments. A strong positive correlation between the contents of Zn and Cu on the one side and the second fluorescence peak ( $\lambda$ =640 nm) for excitation wavelength at 380 nm. In contrast, a high negative correlation for Fe and the third fluorescence maximum ( $\lambda=$  736nm) is observed for all excitation wavelengths. The correlation dependencies were obtained with the least squares method with a significance level of  $p \le 0.05$ .

Keywords: Spirulina, fluorescence spectra, infrared spectra, elements.

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#### CHEMICAL COMPOSITION AND OPTICAL PROPERTIES OF REFINED SUNFLOWER OIL WITH ADDED VARIOUS OILS

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DOI: 10.59957/jctm.v59.i4.2024.1

#### ABSTRACT

Some physicochemical characteristics and elemental composition of refined sunflower oil, as well as linseed oil added to it, were investigated; linseed oil and olive oil; truffle oil and rosemary oil. Fatty acid analysis shows substantial increases in monounsaturated fatty acids with the addition of truffle and rosemary oils (up to about 78 %). With the same supplements, a significant oxidative stability over 20 hours was also observed. High concentrations of chlorophyll were found with the addition of rosemary oils and oils of linseed oil and olive oil.  $\beta$ -carotene was affected three to six times in all supplements compared to the commonly refined oil. Eight elements (Mg, Cr, Mn, Zn, Ni, As, Pd and Cd) were analyzed in the studied oils, no presence of toxic elements As and Cd (< 0.02 mg kg<sup>-1</sup>), lead was up to 0.04 mg kg<sup>-1</sup>. The remaining elements vary in different concentrations depending on the additive oils used. The fluorescence spectra of the tested samples were obtained for excitation wavelengths of 380 nm, and the fluorescence maxima allowed to determine the relationship between the optical and chemical properties of the samples. In addition, infrared spectroscopic experiments (ATR and transmittance) were used to investigate the fatty acid profile of the analyzed oil samples.

Keywords: sunflower oil with additives, fluorescence, IR spectra, elements.

#### **INTRODUCTION**

The sunflower (*Helianthus annuus* L.) is an unpretentious plant suitable for different climatic conditions. Its seeds are the subject of much research due to the composition of fatty acids and secondary components it contains [1]. According to the production method, refined and unrefined oils can be obtained from its seeds (virgin sunflower oil). Unrefined sunflower oil

has a pleasant taste and aroma. The peculiarity of this oil is that it has almost no importance in the market and is most often used in the food industry [2]. Refined oil has no pronounced taste and aroma, its main use is in frying and baking. In Bulgaria, it is part of dressings, as well as used independently in different types of salads. Today, sunflower oil faces serious competition as its demand declines due to competition with other oil crops and changing consumer demand.





## Article Angular-Substituted [1,4]Thiazino[3,4-a]Isoquinolines: Biological Evaluation and In Silico Studies on DPP-IV Inhibition

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Abstract: Recent studies have discovered that aryl-substituted pyrido[2,1-a]isoquinolines have the potential to be highly active DPP IV inhibitors. In previous studies, we reported a novel synthetic approach for the construction of their sulfur-containing bioisosteric [1,4]thiazino[3,4-a]isoquinolines analogues, incorporating an additional aryl substituent. The present study aims to investigate the DPP IV inhibitory activity and cytotoxicity of the synthesized molecules by in vitro assay. The geometry optimization and molecular docking of the synthesized compounds were used to determine their binding modes to the active site of DPP IV. The docking analysis revealed that the energy-minimized poses of the studied compounds are close to the most important selectivity cliffs for DPP IV inhibition, forming hydrogen bonds and hydrophobic interactions with them. These results can be considered as a preliminary step towards further structural activity modifications.

**Keywords:** [1,4]thiazino[3,4-a]isoquinolines; DPP-IV inhibitors; type 2 diabetes; cytotoxicity; molecular docking; DFT

#### 1. Introduction

Dipeptidyl peptidase IV (DPP-IV) is a highly specific serine protease enzyme that has been widely explored for its potential to treat chronic metabolic type 2 diabetes mellitus (T2DM) [1–6]. Important endogenous physiological substrates, which are known to be cleaved by DPP-IV in vivo, are glucose-dependent insulinotropic polypeptide (GIP) and glucagon-like peptide-1 (GLP-1). These polypeptide hormones are released in the small intestine in response to the ingestion of carbohydrates, thus stimulating insulin secretion from the pancreas [6,7]. Subjects with T2DM exhibit a resistance towards the actions of GIP; therefore, this hormone is not a reliable glucose-lowering agent in such a pathological condition [8]. On the other hand, inhibition of DPP-IV in T2DM patients results in increased incretin levels of GLP-1, which, in turn, inhibits glucagon release, enhances insulin secretion, and improves glucose tolerance [9]. Recent findings suggest that DPP-IV inhibitors can also be used for the treatment of coronavirus disease 2019 (COVID-19) [10–12]. These findings have given rise to a drug class of DPP IV-inhibiting compounds that emerged on the market for treating type 2 diabetes [13–20].

Twelve DPP-IV inhibitors (sitagliptin, vildagliptin, saxagliptin, alogliptin, linagliptin, teneligliptin, gemigliptin, anagliptin, trelagliptin, evogliptin, omarigliptin, and denagliptin)



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# Short Note 9,10-Dimethoxy-4-oxo-1-phenyl-1,3,4,6,7,11b-hexahydro-[1,4]thiazino[3,4-a]isoquinoline-1-carboxylic Acid

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**Abstract:** The synthesis of the compound 9,10-dimethoxy-4-oxo-1-phenyl-1,3,4,6,7,11b-hexahydro-[1,4]thiazino[3,4-a]isoquinoline-1-carboxylic acid (**4**) was described for the first time using a reaction between 6,7-dimethoxy-3,4-dihydroisoquinoline and phenyl-substituted thiodiacetic anhydride **3**. The reaction proceeded in excellent yield and furnished the compound **4** as a single diastereomer. The structure and relative configuration of **4** was elucidated using a combination of spectroscopic techniques–<sup>1</sup>H, <sup>13</sup>C, COSY, HSQC, HMBC, and NOESY NMR spectra, as well as elemental analysis.

**Keywords:** 6,7-dimethoxy-3,4-dihydroisoquinoline; thiodiacetic anhydride; [1,4]thiazino[3,4-a]isoquinoline

#### 1. Introduction

The reaction between enolizable anhydrides and different imines is a versatile one-step process that can be used to obtain various nitrogen-containing polycycles with varying substitution patterns and molecular complexity [1–5]. Among the advantages of this synthetic approach are the fact that the process does not require any special catalysts and can be performed in a plethora of organic solvents, sometimes at room temperature. The advancements of synthetic methodologies have given rise to several one-pot varieties of this process [6–10], as well as the application of this reaction in the synthesis of biologically active compounds [2,11] and natural products [7,12].

Our research is focused on the reactions between cyclic imines with monocyclic anhydrides, such as glutaric, diglycolic, thiodiglycolic, and succinic anhydride [13]. This methodology was a previously unexplored approach to the construction of diastereomeric pyrido[2,1-a]isoquinoline derivatives as well as its oxygen and sulfur-containing analogues [13]. When 1-substituted-3,4-dihydroisoquinolines were used in the process, the expected products were obtained only in reaction with thiodiacetic anhydride [13,14]. The obtained sulfur analogues of the tricyclic pyrido[2,1-a]isoquinoline system–[1,4]thiazino[3,4a]isoquinoline derivatives were found to possess promising DPP-IV inhibitory activity in the sub-micromolar range [15].

In the course of our investigations, we attempted to access more complicated derivatives of the target heterocyclic system. For this reason, we focused on the reaction between 3,4-dihydroisoquinolines and phenyl-substituted monocyclic anhydrides. Among the published substituted monocyclic anhydrides, we chose the 3-phenyl-thiodiacetic anhydride because of its high reactivity even at room temperature [16]. Furthermore, since the reactions of this anhydride have been explored only with acyclic imines, the obtained results could provide useful information about the scope and limitations of the reaction. The



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# Essential oil composition of *Satureja kitaibelii* Wierzb. ex Heuff. from the central Danubian plain, Bulgaria

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Key words: essential oil, GC/MS, Satureja kitaibelii, Danubian plain

#### Short summary

This study investigates the essential oil composition of *Satureja kitaibelii* Wierzb. ex Heuff., a Balkan endemic species with notable medicinal properties, from the central Danubian Plain, an unexplored region in Bulgaria. Essential oils were extracted using a Clevenger apparatus and analyzed via GC/MS, revealing *p*-cymene, limonene, carvacrol, and borneol as dominant compounds. Differences between localities (Kaylaka and Kartozhabene) were identified, alongside similarities with populations from other parts of Bulgaria and Serbia. These findings contribute to understanding phytochemical variability and hold implications for standardizing *S. kitaibelii* essential oil for pharmaceutical and industrial applications.

#### Introduction

The species *Satureja kitaibelii* Wierzb. ex Heuff. is a Balkan endemic species, which is one of the five most common wild species of the genus in the Balkans and the second most common in Bulgaria (Đorđević et al. 2014; Euro+Med plantbase 2024).

The above-ground parts of the plants are used in traditional cuisine and medicine in the whole range of species distribution. As a folk remedy it is mainly used for diarrhea, nausea, indigestion, respiratory and infectious diseases, cramps, muscle pain, infertility and menstrual disorders (Acimovic et al. 2021). In the recent years many valuable properties of the species such as antioxidant, anti-inflammatory, hypoglycemic, antitumor and antimicrobial activity were reported (Acimovic et al. 2021; Dimitrijevic et al. 2023; Gopcevic et al. 2019). Considering the increasing resistance of microorganisms to antibiotics, the discovery of new bioactive molecules with high antimicrobial potential is very important (Uddin et al. 2021).

The literature references show that the essential oil composition of the species varies a lot (Dodoš et al. 2019, Slavkovska et al. 2001; Zheljazkov et al. 2022). Therefore, the research on the essential oil profiles of this species from different localities is important.

The aim of the presented study is to determine the essential oil composition of *Satureja kitaibelii* Wierzb. ex Heuff. from the previously unexplored region of the central Danubian plain.

#### Materials and methods

# **8 PHARMACIA**

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