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TECHNOLOGICAL AND BIOMATERIAL SCIENCE APPROACHES TO QUALITY ASSESSMENT OF ESSENTIAL OILS IN THE COSMECEUTICAL INDUSTRY

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Keywords: quality control, lavender essential oil, tea tree essential oil, fatty acid composition, acylglycerol composition, chromatographic profile.

Introduction

One of the most critical concerns for consumers in the perfume and cosmetic market is the efficacy of products. Protecting consumers from misleading claims regarding the effectiveness and other characteristics of cosmetic products is a cornerstone of global regulatory frameworks. Cosmetic products must deliver tangible benefits to users [1]. In Europe, Regulation (CE) No. 655/2013 [1] explicitly states: "*Claims concerning cosmetic products, whether explicit or implicit, shall be supported by adequate and verifiable evidence, regardless of the types of evidence used to substantiate them, including compliance.*" The evidence base for cosmetic product efficacy must consider real-world usage conditions, with studies being relevant to the raw materials, the product itself, and its claimed benefits. Additionally, research must adhere to well-designed and properly organized methodologies (valid, reliable, and reproducible) while complying with universally accepted ethical standards [2, 3]. The efficacy of a cosmetic product is directly dependent on the quality of its raw materials. This article proposes a comprehensive approach to assessing the quality and composition of essential oils, using tea tree oil and lavender oil as case studies. Use of Synthetic and Artificial Essential Oils (EOs) in cosmetology and cosmeceuticals is legally permitted and highly practical in formulation development, as it ensures compositional stability and consistent fragrance – attributes difficult to achieve with natural essential oils alone. However, synthetic and artificial oils often fail to deliver the desired clinical efficacy due to the absence of unique bioactive compounds found in natural EOs [4].

Adulteration of Natural Essential Oils refers to the deliberate alteration of a natural EO's composition for economic gain, achieved by introducing additives or selectively removing valuable components while maintaining the appearance of product quality. Adulterated EOs may also originate from falsified plant raw materials. Common adulterants include synthetic additives, volatile (so-called "turpentine") fractions of certain EOs, cheaper EOs, purified kerosene, vegetable fats, and even mineral oils. Typically, unethical producers engage in such practices to market substandard products, particularly with high-value EOs.

Quality Control of Essential Oils ensures that cosmetic products containing EOs as active ingredients meet declared specifications and deliver the promised benefits of the final product.

Study of Existing Solutions to the Problem

The use of essential oils in cosmetic products is widespread worldwide. Due to the unique content of biologically active substances in essential oils, cosmetic products possess a range of functional properties. For instance, cosmetic products intended for skin care and containing essential oils stimulate local blood circulation, exhibit firming and rejuvenating effects, relieve irritation, eliminate flaking, possess antiseptic and anti-inflammatory properties, create a natural protective barrier, demonstrate healing functions, and have the ability to penetrate deep into the subcutaneous layer of the epidermis, providing hydration and softening while reducing pigmentation [5–7]. The efficacy of a cosmetic product directly depends on the quality of the essential oil used. To prevent the use of low-quality or adulterated essential oils in cosmetic production, a series of methods for determining quality and assessing the composition of essential oils have been developed and implemented. This article proposes an examination of lavender essential oil and tea tree essential oil (supplied by Düllberg Konzentra GmbH & Co, Germany).

Lavender Essential Oil is obtained from the flowering spikes of narrow-leaved lavender (*Lavandula angustifolia* Mill.), which grows in many European countries, Canada, the USA, and Moldova. Lavender oil is a mobile, colorless or yellowish-green liquid with a bitter taste and the fresh floral scent of lavender with a woody undertone. Its chemical composition includes linalool (30–35 %), myrcene, α - and β -ocimene, γ -terpinene, α -pinene, caryophyllene, bergamotene, γ - and δ -cadinene, α -curcumene, farnesene, α -terpineol, geraniol, and camphor. The oil is extracted from the flower spikes through steam distillation, with a yield ranging from 0.78 % to 1.1% [2, 8, 9]. Lavender essential oil exhibits antiseptic, anti-burn, regenerative, and anti-inflammatory properties. It helps reduce skin redness and flaking, promotes the healing of wounds, cuts, and abrasions, prevents dandruff formation, is effective against pediculosis, and soothes skin after insect bites.

Tea Tree Oil is an essential oil obtained through hydrodistillation (steam distillation) from the leaves of the tea tree (*Melaleuca* spp.). It is a colorless or pale yellow liquid with a distinct camphoraceous odor. It is soluble in ethanol, vegetable oils, and propylene glycol; poorly soluble in glycerin; and insoluble in water. Tea tree oil contains over 100 terpene derivatives [3, 10]. The most effective antibacterial components of this oil are terpinen-4-ol, α -pinene, linalool, and α -terpineol. Lipophilic terpeneols penetrate the cell membranes of microorganisms, exerting a toxic effect on their membrane structure and function [6, 7].

Purpose and Main Objectives of the Study

The aim of this study is to assess the composition and quality of lavender essential oil (supplier – Düllberg Konzentra GmbH & Co, Germany) and tea tree essential oil (supplier – Düllberg Konzentra GmbH & Co, Germany) available on the cosmetic market, based on the parameters established in normative documentation [2, 3].

Materials and Methods

To verify the composition and quality of the essential oils, an integrated approach was employed using modern analytical equipment. This included gas-liquid chromatography with high-temperature columns and a set of methods such as the determination of fatty acid composition, acylglycerol composition, and obtaining the chromatographic profile of the essential oil [4, 5]. The quality control and composition analysis of tea tree essential oil were carried out according to the physicochemical parameters defined in applicable standards.

The fatty acid composition was determined according to DSTU ISO 5508-2001 "Animal and vegetable fats and oils. Analysis by gas chromatography of methyl esters of fatty

acids" [8, 11]. The acylglycerol composition was assessed in accordance with ISO/TS 17383:2014 "Determination of triacylglycerol composition in fats and oils – capillary gas chromatography method" [5, 12].

The study was conducted in the Scientific and Methodological Laboratory of Chromatographic Studies at SE "Ukrmetrteststandart" (Kyiv, Ukraine). The determination of acylglycerol composition was performed using a CP-3800 gas chromatograph (Varian), equipped with a flame ionization detector, electronic gas flow control system, a universal injector operating in both split and splitless modes, an autosampler (CP-8410 Varian), and Galaxy software. A MET-Biodiesel capillary column (length 14 m, internal diameter 0.53 mm, film thickness 0.16 μm) with an integrated 2-meter precolumn (internal diameter 0.53 mm) was used. Chromatographic separation was carried out under the following conditions: carrier gas flow rate – 2.5 mL/min, split ratio – 20:1, injector temperature – 390 °C, detector temperature – 400 °C, and a column oven temperature program with a gradual increase from 90 °C to 380 °C.

The fatty acid composition was analyzed using gas-liquid chromatography on an Agilent Technologies 7890 gas chromatograph with a Zebron FAME (Phenomenex) capillary column, 100 m in length, with an internal diameter of 0.25 mm and stationary phase thickness of 0.2 μm , under the following conditions: carrier gas flow rate – 1.2 mL/min, split ratio – 1:100, evaporator temperature – 280 °C, detector (FID) temperature – 290 °C, and column oven temperature program with a gradual increase from 60 °C to 230 °C. The injection volume was 1 μL .

For the identification of chromatographic peaks and chromatogram processing, a 37 Component FAME Mix of methyl esters of fatty acids from Supelco (Cat. No. 47885-U) was used. The chromatograms were recorded and processed using a personal computer equipped with ChemStation OpenLab software.

The chromatographic profile was obtained using an Agilent Technologies 7890 gas chromatograph with a vf-5ms capillary column, 25 m in length, internal diameter 0.25 mm, and stationary phase thickness of 0.33 μm , under the following conditions: carrier gas flow rate – 1.0 mL/min, split ratio – 1:20, evaporator temperature – 250 °C, detector (FID) temperature – 280 °C, and column oven temperature program with a gradual increase from 60 °C to 185 °C.

Research Results

The first stage of the study involved obtaining the chromatographic profile with the identification of aromatic components (data are presented in Table 1). It was established that, according to the chromatographic profile, the tested sample of lavender essential oil meets the requirements for lavender oil in accordance with ISO 3515:2017 "Oil of lavender (*Lavandula angustifolia* Mill.)". The normative requirements are presented in accordance with ISO 3515:2017 "Oil of lavender (*Lavandula angustifolia* Mill.)".

During the investigation of the chromatographic profile of tea tree essential oil, terpinen-4-ol was identified at a concentration of 16.6 %, although this compound is the principal active terpene component and should be present within the range of 35.0–48.0 % as specified by ISO 11024-1. The obtained results prompted further analysis, and the next stage of the research was the determination of the acylglycerol composition. The data revealed a relative mass fraction of triglycerides of 84.5 %, indicating the presence of vegetable oil in the essential oil sample. Subsequent studies focused on the identification of the oil based on its fatty acid composition. It was determined that the investigated essential oil contains soybean oil. Based on the results of gas-liquid chromatography, a summary table of the

complex physicochemical parameters of tea tree oil was compiled (Table 2). The normative requirements are provided in accordance with DSTU 4534:2006 “Soybean oil. Technical specifications” and ISO 4730:2017 “Essential oil of *Melaleuca*, terpinen-4-ol type (Tea tree oil)”.

Table 1 – Lavender essential oil (chromatographic profile)

Name of components	Regulatory documentation requirements	Research results	Uncertainty, U (k = 2, P = 0,95)	Regulatory documentation for test methods
Limonene, %	no more than 1	0,84	± 0,08	ISO 11024-1
1,8-Cineole, %	no more than 3	0,38	± 0,04	ISO 11024-1
beta-Phellandrene, %	no more than 1	not detected (<0,1)		ISO 11024-1
cis-beta-ocimene, %	no more than 10	0,62	± 0,06	ISO 11024-1
trans-beta-ocimene, %	no more than 6	0,27	±0,03	ISO 11024-1
3-Octanone, %	no more than 3	0,19	± 0,02	ISO 11024-1
Camphor, %	no more than 1.5	0,23	± 0,02	ISO 11024-1
Linalool, %	20-43	38,8	± 1,9	ISO 11024-1
Linalyl acetate, %	25-47	39,4	±2,0	ISO 11024-1
Lavandulol, %	no more than 3	1,9	±0,2	ISO 11024-1
Terpinen-4-ol, %	no more than 8	1,3	±0,1	ISO 11024-1
Lavandulyl acetate, %	no more than 8	7,2	±0,7	ISO 11024-1
Alpha-Terpineol, %	no more than 2	0,83	± 0,08	ISO 11024-1

The data obtained confirm the feasibility of using an integrated approach to assess the quality and composition of essential oils and indicate the need for more stringent control of essential oil quality at all stages of delivering this product to the end consumer.

Висновки

The study established that the content of the main aromatic compounds – linalool (38.8 %) and linalyl acetate (39.4 %) – according to the chromatographic profiles, complies with the requirements for lavender essential oil as set out in ISO 3515:2017 "Oil of lavender (*Lavandula angustifolia* Mill.)". The sample of tea tree essential oil, based on its chromatographic profile, did not meet the requirements of ISO 4730:2017 “Essential oil of *Melaleuca*, terpinen-4-ol type (Tea tree oil)”. The sample contained 84.5 % vegetable oil. According to its fatty acid composition, the vegetable oil present in the sample corresponded to the requirements for soybean oil defined in DSTU 4534:2006 "Soybean oil. Technical specifications". The obtained results confirm the necessity of a comprehensive approach in selecting analytical methods and designing research strategies for the quality assessment and composition analysis of essential oils, particularly for purposes of identification and detection of adulteration or falsification.

Table 2 – Tea tree essential oil. Physicochemical research indicators

Name of components	Regulatory documentation requirements	Research results	Uncertainty, U (k = 2, P = 0,95)	Regulatory documentation for test methods
<u>Fatty acid composition (relative mass fraction of fatty acids, %)</u>				
Palmitic (C _{16:0})	7,0–13	11,1	±0,4	DSTU ISO 5508
Stearic (C _{18:0})	2,0–6,0	4,3	±0,6	DSTU ISO 5508
Oleic (C _{18:1})	18,0–30,0	24,4	±1,0	DSTU ISO 5508
Linoleic (C _{18:2})	44,0–62,0	52,7	± 1,1	DSTU ISO 5508
Linolenic (C _{18:3})	4,0–11,0	5,9	±0,8	DSTU ISO 5508
<u>Acylglyceride composition</u>				
Relative mass fraction of triglycerides, %		84,5	± 4,2	ISO/TS 17383:2014
<u>Chromatographic profile of the aromatic component</u>				
Alpha-pinene, %	1,0–6,0	4,6	±0,5	ISO 11024-1
Sabinene, %	no more than 3,5	не виявлено (<0,1)		ISO 11024-1
Alpha-terpinene, %	6,0–12,0	14,2	± 1,4	ISO 11024-1
Limonene, %	0,5–1,5	4,2	±0,4	ISO 11024-1
Rho-cymene, %	0,5–8,0	7,0	±0,7	ISO 11024-1
1,8-cineole, %	no more than 10,0	10,0	±1,0	ISO 11024-1
Gamma-terpinene, %	14,0–28,0	10,6	± 1,1	ISO 11024-1
Terpinol, %	1,5–5,0	2,9	±0,4	ISO 11024-1
Terpinen-4-ol, %	35,0–48,0	16,6	± 1,7	ISO 11024-1
Alpha-terpineol, %	2,0–5,0	23,5	± 2,4	ISO 11024-1
Aromadendrene, %	0,2–3,0	not detected (<0,1)	–	ISO 11024-1
Delta-cadinene, %	0,2–3,0	not detected (<0,1)	–	ISO 11024-1
Globulol, %	no more than 1,0	not detected (<0,1)	–	ISO 11024-1
Viridoflorol, %	no more than 1,0	not detected (<0,1)	–	ISO 11024-1

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ТЕХНОЛОГІЧНІ ТА БІОМАТЕРІАЛОЗНАВЧІ ПІДХОДИ ДО ОЦІНКИ ЯКОСТІ ЕФІРНИХ ОЛІЙ У КОСМЕЦЕВТИЧНІЙ ПРОМИСЛОВOSTІ

У сучасній космецевтичній промисловості спостерігається стрімке зростання попиту на натуральні інгредієнти, особливо на ефірні олії, що обумовлює необхідність розробки надійних та стандартизованих методів оцінки їх якості, автентичності та безпеки. Дане дослідження представляє комплексний аналіз сучасних технологічних та біоматеріалознавчих підходів до контролю якості популярних ефірних олій, зокрема лавандової та чайного дерева, які широко застосовуються у косметичній, лікувально-профілактичній та фармацевтичній продукції.

Робота базується на вимогах сучасних європейських нормативних документів, зокрема регламенту CE 655/2013, який встановлює чіткі вимоги щодо надання науково обґрунтованих доказів ефективності, стабільності та безпеки косметичних інгредієнтів. Особливу увагу приділено проблемі фальсифікації ефірних олій, яка включає такі поширені методи як: розведення дешевшими рослинними оліями, часткову або повну заміну природних біоактивних сполук синтетичними аналогами, а також використання

технічних домішок для збільшення об'єму продукції. Результати досліджень виявили, що лавандова олія повністю відповідає вимогам міжнародного стандарту ISO 3515:2017 щодо хімічного складу та фізико-хімічних властивостей. На противагу цьому, у частині зразків олії чайного дерева було виявлено значні домішки соєвої олії, що суттєво суперечить вимогам стандарту ISO 4730:2017. Отримані результати демонструють високу ефективність запропонованого комплексного підходу, який поєднує передові хімічні, спектроскопічні та біологічні методи аналізу. Проведене дослідження має практичне значення для космецевтичної промисловості, пропонуючи алгоритм виявлення фальсифікованої продукції та підкреслюючи критичну важливість постійного моніторингу якості сировини на всіх етапах виробничого процесу. Результати роботи можуть стати основою для розробки нових, більш строгих стандартів контролю якості ефірних олій у косметичній, космецевтичній та фармацевтичній галузях, що сприятиме підвищенню безпеки та ефективності кінцевої продукції.

Ключові слова: контроль якості, ефірна олія лаванди, ефірна олія чайного дерева, жирнокислотний склад, ацилгліцериновий склад, хроматографічний профіль.

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TECHNOLOGICAL AND BIOMATERIAL SCIENCE APPROACHES TO QUALITY ASSESSMENT OF ESSENTIAL OILS IN THE COSMECEUTICAL INDUSTRY

The modern cosmeceutical industry is experiencing rapid growth in demand for natural ingredients, particularly essential oils, necessitating the development of reliable and standardized methods for evaluating their quality, authenticity, and safety. This study presents a comprehensive analysis of contemporary technological and biomaterial science approaches to quality control of popular essential oils, specifically lavender and tea tree oil, which are widely used in cosmetic, therapeutic, and pharmaceutical products.

The research is based on the requirements of current European regulatory documents, particularly Regulation (EC) No 655/2013, which establishes clear guidelines for providing scientifically substantiated evidence of cosmetic ingredients' efficacy, stability, and safety. Special attention is given to the problem of essential oil adulteration, which includes common practices such as dilution with cheaper vegetable oils, partial or complete substitution of natural bioactive compounds with synthetic analogues, and the use of technical additives to increase product volume. The study revealed that lavender oil fully complies with the international standard ISO 3515:2017 regarding chemical composition and physicochemical properties. In contrast, some tea tree oil samples contained significant adulterants of soybean oil, substantially deviating from the requirements of ISO 4730:2017. The obtained results demonstrate the high efficiency of the proposed integrated approach, combining advanced chemical, spectroscopic, and biological analytical methods. This research holds significant practical value for the cosmeceutical industry by providing a scientifically validated algorithm for detecting adulterated products and emphasizing the critical importance of continuous quality monitoring throughout all stages of the production process. The findings may serve as a foundation for developing new, more stringent quality control standards for essential oils in cosmetic, cosmeceutical, and pharmaceutical applications, ultimately enhancing the safety and efficacy of final products.

Keywords: quality control, lavender essential oil, tea tree essential oil, fatty acid composition, acylglycerol composition, chromatographic profile.