EXTRACTION OF ESSENTIAL OILS FROM PINE NEEDLES AND EVALUATION OF WOOD WASTE AS AN ALTERNATIVE RAW MATERIAL

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Abstract. This work presents a comprehensive analysis of the extraction of essential oils from pine (Pinus sylvestris) needles and the evaluation of wood waste as an alternative raw material. The main aim of the study is to explore the potential of transforming wood waste, which is harmful for the environment, into high value-added products under the “waste-to-value” concept. Hydrodistillation and Soxhlet extraction techniques were used to obtain essential oils and the efficiency of these techniques was comparatively evaluated in terms of yield, selectivity and compositional features of the outcomes. The experimental studies conducted proved that the method of extraction used influences significantly the quantitative indicators as well as the chemical composition of the essential oils. The physical and chemical properties of the obtained essential oils were studied and included the density, refractive index and spectral analysis, using modern analytical techniques. FTIR analysis indicated the presence of diverse functional groups (hydroxyl, carbonyl and aliphatic structures) within the samples, confirming the complex multi-component composition of the essential oils. The results indicate that hydrodistillation is more suitable for the selective separation of volatile compounds, such as low-molecular-weight essential oils, whereas Soxhlet extraction enables recovery of a wider range of higher-molecular-weight compounds in the organic extract. Such differences are clearly evident in the physicochemical parameters and spectral features of the obtained products.

Keywords: Essential oil, wood waste, pine needles, hydrodistillation, Soxhlet extraction.

Introduction

The considerable growth of industrial and forestry activities in recent years has led to a remarkable increase in wood waste. The poor management of these waste materials, especially open burning, releases toxic gases into the atmosphere and causes environmental pollution. In this connection, one of the pressing scientific and practical problems of our time is the alternative and efficient use of wood waste [1-4].

One of the modern approaches to solving this problem is the concept of "waste-to-value" which means the conversion of waste into valuable products. This method ensures an efficient utilization of resources while reducing waste in accordance with the principles of the circular economy [5–7].

Essential oils are volatile and biologically active compounds obtained from various parts of plants. They are widely used in the pharmaceutical, cosmetic, food, and aromatherapy industries. The composition of essential oils mainly includes terpenes, terpenoids, and various aromatic compounds, and these components determine their antioxidant, antimicrobial, and other biological properties [8–10].

For instance, the study of Muhammad Azri Amran et al. indicated the extraction of essential oils from agricultural wastes was economically and ecologically efficient. In another study, the article of Zhengyun Liang and collaborators used a new extraction method to obtain the essential oil from pine needles and studied the composition and biological activity of the oil, showing that the essential oils have antioxidant and antibacterial activity. Recent research shows that it is possible to extract essential oils from pine needles in an effective way using different methods such as hydrodistillation and microwave-assisted extraction and that the oils have high biological activity [11–14].

The pine (*Pinus sylvestris*) is one of the plants richest in essential oils, and its needles contain a variety of biologically active substances. However, a significant portion of this raw material in Azerbaijan remains unused as waste or is burned.

The primary objective of this study is to obtain essential oil from pine needles and to investigate the possibilities of utilizing wood waste as an alternative raw material. In addition, the effectiveness of different extraction methods is compared, and a basis is established for determining the future application areas of the obtained products.

Experimental Section

Pine (*Pinus sylvestris*) needles were selected as the object of study. The raw material was used in both fresh and dried forms. Dried samples were pre-ground and brought to a homogeneous state.

Several methods were employed for the obtaining of essential oils. The first method applied was hydrodistillation. For this purpose, 15 g of raw material was processed together with a defined volume of distilled water at a temperature of 98°C. During the process, water vapor passed through the plant material, carrying the essential oil components with it; the vapor was then condensed, resulting in the separation of the essential oil from the aqueous phase. The results obtained from fresh and dried raw materials were compared.

In the experiment carried out using the Soxhlet extraction method, 15 g of raw material was placed in filter paper and loaded into the Soxhlet apparatus, with 400 ml of ethanol used as the solvent. The extraction process lasted 8 hours. Since the obtained product was in the form of a mixture, the organic extract was separated by distillation, during which a certain degree of product loss was observed.

The target products obtained during the process -carried out at the appropriate temperature and time intervals — were analyzed using various methods. Specifically, FTIR analysis, density, and refractive index were determined for each product obtained, and the results were compared.

Results and discussion

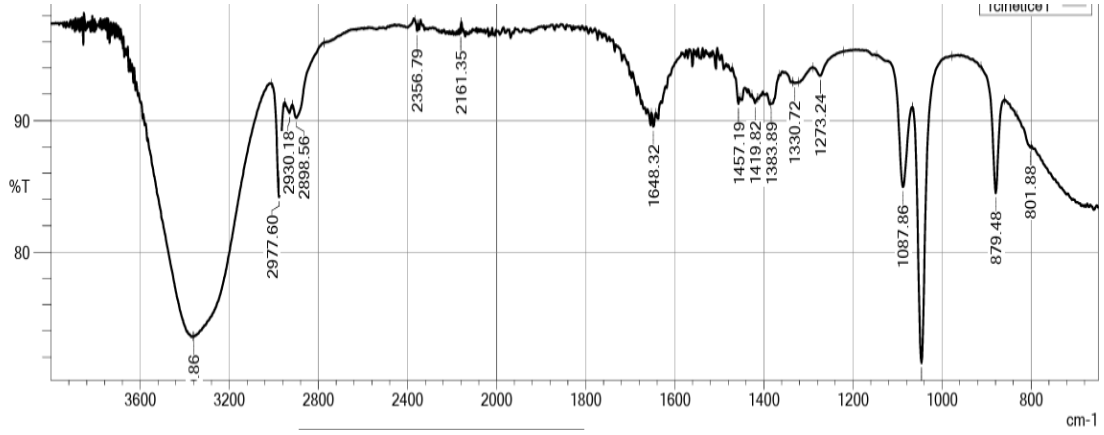
The results of the conducted research indicate that, in terms of obtaining essential oils from dried needle-bearing plant raw material, hydrodistillation is more effective and appropriate compared to Soxhlet extraction. This is attributed to the ability of the hydrodistillation process to ensure the selective separation of volatile components. Experimental observations demonstrated that during hydrodistillation, essential oil components are separated from the plant material via water vapor, subsequently condensed, and an aromatic aqueous phase is formed alongside the essential oil. The yield of products obtained by both methods was approximately 1.5–2%. Furthermore, the initial condition of the raw material has a significant effect on process efficiency; in the case of fresh (freshly harvested) and high-moisture raw material, the separation of essential oil components becomes more difficult, the yield decreases considerably, and in some cases is not observed at all.

In contrast, the Soxhlet extraction method — by employing organic solvents — leads to the extraction of not only volatile but also non-volatile and high-molecular-weight components. For this reason, the product obtained by this method

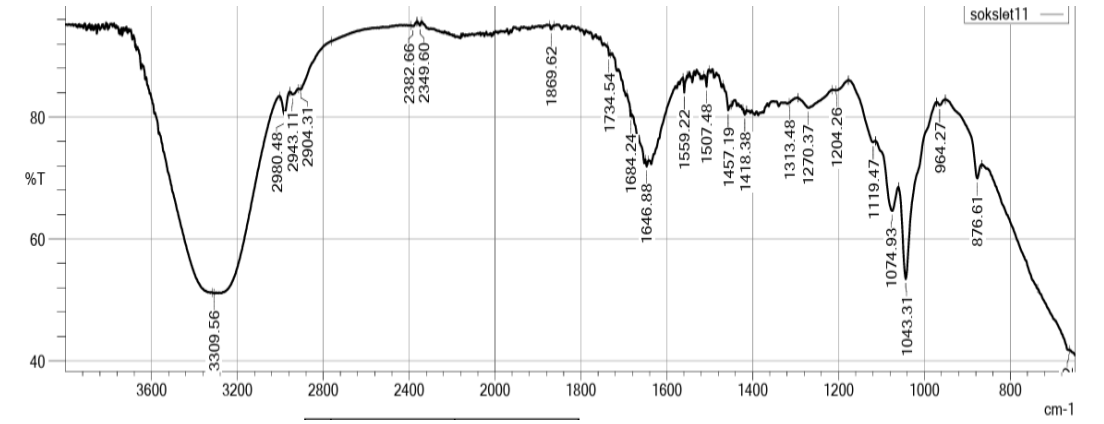
should be regarded as a complex plant extract rather than an essential oil.

Accordingly, the comparative analysis confirms that hydrodistillation is the more effective method for the purpose of obtaining essential oils and should be accepted as the primary method in this direction.

The comparison of FTIR spectra (Figure 1) and physical parameters of samples obtained by different methods made it possible to clearly determine the direction and nature of this effect.



(a)



(b)

Figure 1. FTIR spectra of samples obtained by hydrodistillation (a) and Soxhlet extraction (b)

The comparative results of the FTIR analyses (Figure 1) demonstrate that clear differences exist between the chemical composition and distribution of functional groups in the samples obtained by hydrodistillation and Soxhlet extraction methods.

In the spectrum of the sample obtained by hydrodistillation, volatile terpene and oxygenated terpenoid components — characteristic of essential oils — predominate. Specifically, the aliphatic C–H stretching vibrations in the 2977–2898 cm^{-1} range, C=C bonds at 1648 cm^{-1} and intense peaks assigned to C–O groups at 1087 cm^{-1} indicate the presence of monoterpene and sesquiterpene structures, while the absence of strong carbonyl peaks in the 1700–1750 cm^{-1} region suggest that the sample is rich in lighter and more volatile components.

The FTIR spectrum of the sample obtained by the Soxhlet extraction shows a broader and more intense distribution of functional groups. In particular, broad O–H bands around 3300 cm−1, signals assigned to carbonyl groups near 1700 cm−1 and strong C–O vibrations in the 1200-1000 cm−1 interval indicate the presence of oxygenated and relatively high-molecular-weight compounds in this sample, including phenols,

ketones, esters and other less volatile components. This demonstrates that the Soxhlet method extracts not only essential oil components, but also heavier and non-volatile substances.

The comparison shows higher selectivity of the hydrodistillation process, which ensures separation of mostly volatile components typical for essential oils, while the product of Soxhlet extraction has more complex chemical composition and does not fully correspond to the concept of essential oil. These differences are clearly visible both in the spectral distribution of functional groups and in the intensity of the peaks.

Thus, the results of FTIR analysis indicate that hydrodistillation is the more appropriate and selective method for obtaining essential oils, while Soxhlet extraction is more suitable for obtaining general plant extracts.

The analysis of physicochemical parameters complements the results of FTIR, and gives a more complete picture of the composition of the samples. The density of the essential oil obtained by hydrodistillation being 0.946 g /cm ^{3}, against 0.994 g/cm ^{3} for the Soxhlet extract, can be explained by the fact that the Soxhlet method extracts a wider spectrum of compounds including relatively less volatile and high molecular weight compounds. In the case of hydrodistillation, since primarily low-molecular-weight and more volatile terpenic components are separated, the density is correspondingly lower. The refractive index of the hydrodistillation sample — measured at 1.3653 — can be evaluated as an additional indicator reflecting the chemical structure and degree of unsaturation of the components present in the sample.

In general, the results of FTIR analysis were in agreement with the physico-chemical parameters, indicating that the extraction method influences not only the yield, but also the composition of the obtained product. Hydrodistillation is more selective in the separation of volatile components characteristic of essential oils, whereas Soxhlet extraction gives extracts richer in more complex, non-volatile components.

Conclusion

The conducted research shows that the hydrodistillation is the more effective and selective method to obtain essential oils from pine needles, since it provides the separation of mainly volatile components of terpenes and terpenoids, and finally the product is consistent with the definition of essential oil. Soxhlet extraction, on the other hand, removes a broader spectrum of substances — including non-volatile and high-molecular-weight compounds — and therefore yields a complex plant extract rather than an essential oil.

At the same time, this approach enables the conversion of wood waste into valuable products, thereby contributing to the reduction of negative environmental impact and the more efficient use of resources. Accordingly, hydrodistillation can be evaluated as the more appropriate method for obtaining essential oils from both a technological and an ecological standpoint.

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ŞAM İYNƏLƏRİNDƏN EFİR YAĞLARININ ALINMASI VƏ AĞAC TULLANTILARININ ALTERNATİV XAMMAL KİMİ QİYMƏTLƏNDİRİLMƏSİ

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Xülasə. Bu tədqiqat işində şam ağacının (*Pinus sylvestris*) iynəyarpaqlarından efir yağlarının əldə olunması və oduncaq tullantılarının alternativ xammal kimi dəyərləndirilməsi kompleks şəkildə araşdırılmışdır. Tədqiqatın əsas məqsədi ətraf mühitə mənfi təsir göstərən oduncaq tullantılarının "waste-to-value" konsepsiyası çərçivəsində yüksək əlavə dəyərə malik məhsullara çevrilmə potensialını müəyyən etməkdir.

Efir yağlarının alınması məqsədilə hidrodistillə və Soxhlet ekstraksiya üsulları tətbiq edilmiş, bu metodların effektivliyi çıxım, selektivlik və əldə olunan məhsulların tərkib xüsusiyyətləri baxımından müqayisəli şəkildə qiymətləndirilmişdir. Aparılan eksperimental tədqiqatlar göstərmişdir ki, istifadə olunan ekstraksiya üsulu efir yağlarının həm kəmiyyət göstəricilərinə, həm də kimyəvi tərkibinə əhəmiyyətli dərəcədə təsir edir.

Alınmış efir yağlarının fiziki və kimyəvi xüsusiyyətləri (sıxlıq, refraksiya göstəricisi və spektral analizi) müasir analitik üsullarla tədqiq edilmişdir. İQ-spektroskopiya (FTIR) analizi nəticəsində nümunələrin tərkibində müxtəlif funksional qrupların (hidroksil, karbonil və alifatik strukturlar) mövcudluğu müəyyən olunmuş, bu isə efir yağlarının kompleks və çoxkomponentli quruluşa malik olduğunu təsdiqləmişdir.

Nəticələr göstərir ki, hidrodistillə üsulu əsasən uçucu və aşağı molekul kütləsinə malik efir yağlarının selektiv ayrılması üçün daha əlverişli olduğu halda, Soxhlet ekstraksiyası daha geniş spektrli və yüksək

molekul kütləsinə malik üzvi ektraktların əldə olunmasına imkan verir. Bu fərqlər əldə olunan məhsulların fiziki-kimyəvi göstəricilərində və spektral xüsusiyyətlərində aydın şəkildə əks olunmuşdur.

Açar sözlər: Efir yağı, oduncaq tullantıları, şam iynəyarpaqları, hidrodistillə, Soxhlet ekstraksiyası.

ПОЛУЧЕНИЕ ЭФИРНЫХ МАСЕЛ ИЗ СОСНОВОЙ ХВОИ И ОЦЕНКА ДРЕВЕСНЫХ ОТХОДОВ КАК АЛЬТЕРНАТИВНОГО СЫРЬЯ

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Аннотация. В данной исследовательской работе комплексно изучено получение эфирных масел из хвои сосны (*Pinus sylvestris*) и оценка древесных отходов в качестве альтернативного сырья. Основная цель исследования состоит в определении потенциала переработки древесных отходов, оказывающих негативное воздействие на окружающую среду, в продукты с высокой добавленной стоимостью в рамках концепции «waste-to-value».

Для получения эфирных масел были применены методы гидродистилляции и экстракции по Сокслету, эффективность которых сравнительно оценивалась с точки зрения выхода, селективности и состава полученных продуктов. Проведённые экспериментальные исследования показали, что используемый метод экстракции оказывает существенное влияние как на количественные показатели, так и на химический состав эфирных масел.

Физические и химические свойства полученных эфирных масел — плотность, показатель преломления и спектральный анализ — были изучены с применением современных аналитических методов. В результате ИК-спектроскопический (FTIR) анализа в составе образцов была установлена наличие различных функциональных групп (гидроксильных, карбонильных и алифатических структур), что подтвердило сложный и многокомпонентный состав эфирных масел.

Результаты показывают, что гидродистилляция больше подходит для селективного разделения летучих соединений, таких как низкомолекулярные эфирные масла, тогда как экстракция по Сокслету позволяет извлекать более широкий спектр высокомолекулярных соединений из органического экстракта. Эти различия отчетливо видны в физико-химических параметрах и спектральных характеристиках полученных продуктов.

Ключевые слова: эфирное масло, древесные отходы, хвоя сосны, гидродистилляция, экстракция по Сокслету.