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## Summaries

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# LOSSES OF ESSENTIAL OILS AND ANTIOXIDANTS DURING THE DRYING OF HERBS AND SPICES. A REVIEW

# STRATY OLEJKÓW ETERYCZNYCH ORAZ ZWIĄZKÓW O WŁAŚCIWOŚCIACH PRZECIWUTLENIAJĄCYCH PODCZAS SUSZENIA ZIÓŁ I PRZYPRAW. PRZEGLĄD

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**Summary:** Drying is the most common and also the simplest method of preserving fresh plant materials. This process greatly extends the life of the product by the removal of water, which decreases the rate of chemical and enzymatic reactions, or even inhibits them. The drying step can cause a change in appearance, taste, color and consistency, as well as reduce the quantity of essential oils, polyphenols, pigments and vitamins. These changes can be significantly reduced by using suitable drying techniques, depending on the material. The choice of the technique should be based on knowledge of the biological, physical and chemical characteristics of the raw material. The paper presents the effects of drying on the quantitative loss of essential oils and compounds with antioxidant properties, as well as changes in the antioxidant properties of selected herbs and spices dried by different methods. Both, the food industry and the cosmetics or pharmaceuticals industry generates a demand for high-quality raw materials, and hence research is being conducted both on preservation methods and on their impact on the quality of the obtained material. It seems that the new technology of drying material in a fluidized bed, greatly reduces or even eliminates the disadvantages of current procedures.

**Keywords:** drying, herbs, essential oils, polyphenols.

**Streszczenie:** Suszenie jest najczęściej stosowaną i zarazem najprostszą metodą konserwacji świeżych surowców roślinnych. Proces ten znacznie przedłuża trwałość produktu, poprzez usunięcie wody, co zmniejsza szybkość bądź nawet hamuje reakcje chemiczne i enzymatyczne. Etap suszenia może powodować zmiany w wyglądzie, smaku, kolorze czy konsystencji, jak również obniżać ilość olejków eterycznych, polifenoli, barwników czy witamin. Zmiany te można w znacznym stopniu ograniczyć, stosując odpowiednie techniki suszenia w zależności od suszonego materiału, których dobór powinien wynikać ze znajomości biologicznych, fizycznych i chemicznych właściwości surowca. W pracy przedstawiono wpływ suszenia na straty olejków eterycznych i związków o właściwościach przeciwutleniających oraz zmiany właściwości antyoksydacyjnych wybranych ziół i przypraw, suszonych różnymi metodami.

Zarówno przemysł spożywczy jak i kosmetyczny czy farmaceutyczny, wykazuje zapotrzebowanie na surowce wysokiej jakości, i stąd wskazane jest prowadzenie badań zarówno nad metodami utrwalania jak i nad określeniem ich wpływu na jakość uzyskanego surowca. Wydaje się, że nowa technologia suszenia surowców w złożu fluidalnym w znacznym stopniu ogranicza a nawet eliminuje wady obecnie stosowanych procedur.

**Słowa kluczowe:** suszenie, zioła, olejki eteryczne, polifenole.

## 1. Introduction

Plant materials contain many beneficial substances, such as essential oils, polyphenols, anthocyanins, vitamins, minerals, and fatty acids, which significantly affect human health [Zheng, Shiow 2001]. Many herbs and spices not only impart a characteristic flavor to food products, but also have some therapeutic and preventive effects. The latter include the increased secretion of digestive juices and peristalsis, and protection against diseases of the gastrointestinal system. The natural antioxidants contained in plants play an important role in the treatment of many diseases, including diabetes, atherosclerosis, heart disease, and cancer [Makała 2010; Kaefer, Milner 2008]. Herbs are currently widely used in the food, pharmaceutical, and cosmetic industries [Zheng, Shiow 2001; Fatouh et al. 2006]. Herbs and spices are also important ingredients in the bakery, dairy, meat, fruit and vegetable, and spirit industries, and essential oils are widely used in the confectionery and cosmetic applications. Herbs are also used as raw materials for the production of seasonings and in coloring or flavoring of foodstuffs. The food industry generates a demand for stabilized high-quality raw materials, and hence research is being conducted both on different preservation methods and on their impact on the quality of the obtained material.

Drying is the most common and also the simplest method of preserving fresh plant materials [Deng, Zhao 2008; Lewicki 2006; Vega-Mercado et al. 2001]. The process increases the storage life of herbs and spices and enables their use after a prolonged period of time. The procedure reduces some adverse processes occurring in the plant material, as the removal of water decreases the rate of chemical and enzymatic reactions, or even inhibits them.

Drying may be classified into two categories: natural (floor drying) and artificial (in drying chambers). All the technologies currently used for preserving herbs, despite their advantages (long shelf life, high sensory quality) lead to losses of valuable plant components, including essential oils [Omidbaigi et al. 2004; Venskutonis 1996]. The new and innovative procedure of removing water from herbs involves drying them in a fluidized or dynamic bed with a closed air cycle and a heat exchanger, which makes it possible to obtain products at a specified moisture content and hence with a higher concentration of biologically active compounds. Additionally, a natural component known as Fluidolat® is produced in this way. Fluidolat is condensed herbal water containing most of the volatile compounds that are normally lost in conventional processes [Śmigielski et al. 2012, Śmigielski et al. 2014].

## 2. Traditional and innovative methods of drying plant material

The drying process is widely used in the fruit and vegetable industry (drying of fruits, vegetables, mushrooms), meat and fish industry, milk industry (production of powdered milk, buttermilk, and nutritional supplements), egg industry (powdered eggs and egg yolks), potato industry (production of potato flakes, potato purée, and potato starch), cereal and milling industry (production of oatmeal, rice flakes, and pasta), as well as food concentrates (concentrates of coffee, tea, supplements, dry soups).

The drying process significantly extends the life of the product, but does not protect against autoxidation of fats, protein denaturation, fiber crystallization, and the loss of essential oils and vitamins. The drying method should provide both microbiological safety as well as high quality of the physical, chemical, biological, and sensory properties of the final product.

Drying is divided into two categories – natural drying, using solar radiation and the heat contained in the air (mainly in countries with warm climates) and artificial drying in dryers. Artificial methods include contact, convection, radiant, fluidized bed, microwave and freeze drying.

The traditional way of preserving plant materials, despite its many advantages, such as low cost and simple technology, results in the loss of many valuable food ingredients, both essential oils and antioxidants [Capecka et al. 2005; Omidbaigi et al. 2004; Schweiggert et al. 2007; Śmigielski et al. 2011]. The use of low temperature and shorter process times in lyophilization better preserves the biological value of the raw material. However, the high costs associated with the use of low temperature prevent the widespread use of this method [Ozcan et al. 2005; Díaz-Maroto et al. 2002]. Infrared radiation is an attractive method of preserving plant materials in terms of both economic and quality considerations, thanks to which it is used increasingly often [Boehm et al. 2002]. Progress in the vacuum-microwave drying method enables cost reduction and leads to products of higher quality, without the negative effects of local overheating and excessive destruction of the product structure. The advantages of fluidized-bed drying over the traditional and some modern methods primarily include excellent heat transfer and transfer conditions (reducing drying time) and good mixing of material in the fluidized bed (ensuring the same heat treatment conditions for all particles of the material). Consequently, the obtained products are uniformly dried and of higher quality. A variant of this method is a fluidized-bed setup with closed-loop circulation of the drying agent with a heat exchanger system [Śmigielski et al. 2012; Śmigielski et al. 2014].

New trends in dehydration aim both to increase thermal performance and speed of drying, as well as to increase the safety and efficiency of the process and to ensure high quality of the resulting products.

## 3. Effect of drying on the loss of the essential oils

Essential oils are secondary metabolites of plants obtained from flowers, leaves, stems, roots, bark, seeds, or pericarp. The major constituents of essential oils are terpenes/terpenoids and aromatic and aliphatic compounds, which are characterized as low-molecular-weight aroma chemicals. Generally, essential oils are comprised of two or three major components in relatively high concentrations (20–95%) and other components present in trace levels [Betts 2001; González-Burgos et al. 2011; Pichersky et al. 2006]. They may be extracted by steam distillation or hydrodistillation (expect citrus − squeezing). The drying method used affects the amount of essential oils remaining in the raw material, and thus the sensory quality of the components obtained in this way [Huopalahti et al. 1985; Venskutonis 1996].

The losses of essential oils contained in the plant material depend to a significant extent both on the process parameters and on the biological characteristics of the plant material. As reported in a paper by Sellami et al., Laurel (*Laurus nobilis* L.) leaves dried at ambient temperature afforded an essential oil yield of 1.13%. Upon infrared drying at 45°C the yield of essential oil was lower by 31%, while convective drying at 45°C or 65°C led to losses of up to 83% as compared to the amount of essential oil extracted from the raw material dried at ambient temperature. Following convection drying, essential oils exhibited a loss of monoterpene hydrocarbons, which was probably due to chemical rearrangements of compounds or their volatility with steam [Sellami et al. 2011]. The process of drying lavender (*Lavandula angustifolia* L.) flowers in a well-ventilated and shaded place prior to the hydrodistillation step led to a loss of essential oil amounting to over 40% as compared to the quantity of essential oil obtained from fresh raw material. No significant differences in the qualitative composition of the essential oils of lavender were observed. The drying process reduced the content of monoterpene esters by about 13% and increased the amount of monoterpene alcohols by about 4%. This is probably due to the activation of enzymes belonging to the group of hydrolases, and hence the change in the quantitative composition of essential oils [Śmigielski et al. 2011].

Roman chamomile (*Chamaemelum nobile* L.) flowers dried in the shade afforded an essential oil yield of about 1.9%. Oven-drying at 40°C caused essential oil losses of 52.6% . However, the highest losses were recorded when the drying process was conducted in the sun (79%). The results show that different drying methods have no effect on the presence of the main essential oil components, but significantly influence their percentage content [Omidbaigi et al. 2004].

Microwave-vacuum drying of parsley (*Petroselinum crispum*) caused little change in its essential oil content (approximately 10%) as compared to the fresh material. However, the material dried by the traditional method exhibited a loss of up to about 70%. Furthermore, microwave-vacuum drying led to products with a favorable color and an aroma better than that obtained by conventional methods

[Boehm et al. 2002]. A change in the drying air temperature from 30°C to 70°C caused a decrease in essential oil yield from dried parsley leaves. The highest essential oil content (2.27 [mg·100 g DW-1]) was recorded at 30°C, while drying at 70°C resulted in a 38% loss of essential oils [Rudy et al. 2011].

Convection drying at 60°C or microwave-vacuum drying, or convection pre-drying combined with microwave drying of oregano leaves (*Origanum vulgare*) reduces the amount of essential oils contained in them. The smallest loss was found for microwave-vacuum drying (about 15%) while the largest after convection drying (about 69%), which may indicate both chemical instability and volatility of the chemical compounds contained in the essential oil [Figiel et al. 2010]. On the other hand, microwave-vacuum drying of rosemary (*Rosmarinus officinalis* L.) led to losses of essential oils reaching about 54%, while convection drying resulted in losses of only about 35%. Microwave drying, preceded by convection drying led to a product with improved sensory properties, since the content of essential oils was reduced only by approximately 26%, which was probably due to the short duration of the process (30 min). The presented analyses show that the content of essential oils remaining in the material depends not only on the procedure used but also on the nature of the fresh plant material [Szumny et al. 2010].

The drying method affects not only the amount of essential oils, but also often their chemical composition. In a study by Asekun et al., the main component of the essential oil extracted from fresh mint leaves (*Mentha longifolia*) was pulegone (47.9%), while menthone (38.3%) was the predominant compound of essential oil isolated from mint leaves treating the air drying (in a well-ventilated room), and limonene (40.8%) was the most abundant compound after oven drying at 40°C. Menthone and pulegone were not found in essential oils obtained from leaves dried in the oven at 40°C, which indicates poor stability of these compounds at the process temperature. Given the lack of menthone and pulegone (potentially harmful to humans) in the essential oils obtained from oven-dried leaves at 40°C, this technique should be considered the most appropriate [Asekun et al. 2007]. Drying the aerial parts of sweet wormwood (*Artemisia annua* L.) at different temperatures caused a loss of essential oils. The higher the drying temperature used, the lower the yield. When the process was conducted at ambient temperature, the yield of essential oil amounted to 1.12%, while drying at 65°C led to losses of up to 67% as compared to the yield obtained from raw materials dried at ambient temperature. The main component of the oil extracted from the raw material dried at room temperature or at 45°C was 1,8-cineol, while following drying at 35°C or 55°C the prevailing compounds were both 1,8-cineol and camphor. The predominant compounds of the essential oil obtained from the aerial parts of sweet wormwood dried at 65°C were β-caryophyllene and germacrene D [Khangholil, Rezaeinodehi 2008]. The effects of the drying temperature indicate instability of the volatile organic compounds contained in the essential oil of sweet wormwood [Sellami et al. 2011].

**Table 1.** Characteristics of selected plants and obtained from them essential oils

| Name | The main compounds of essential oil | Properties and applications | References |
|---|---|---|---|
| Laurel (*Laurus nobilis* L.) | cineol, pinen, felandren, terpineol, eugenol | bactericidal, choleretic, improved blood circulation cosmetic and medicinal plant | [Sellami et al. 2011] |
| Lavender *(Lavandula angustifolia* L.) | Linalool, linalyl acetate, borneol, lawandulol | sedative, analgesic, antimicrobial, antioxidant, a medicinal, cosmetic, honey plant | [Śmigielski et al. 2011; Śmigielski et al. 2014] |
| Roman chamomile *(Chamaemelum nobile* L.) | chamazulen, α-bisabolol, mircen, spiroeter | anti-inflammatory and anti-edema, antibacterial and antifungal, neutralizes toxins, stimulates metabolism | [Omidbaigi et al. 2004] |
| Parsley (*Petroselinum crispum)* | apiol, myristicin, limonen | antiseptic, carminative stimulants, analgesics | [Boehm et al. 2002; Rudy et al. 2011] |
| Oregano (*Origanum vulgare)* | tymol, carvacrol | antidiarrheal, antispasmodic, expectorant, disinfectant, diuretic, carminative | [Figiel et al. 2010] |
| Rosemary (*Rosmarinus officinalis* L.) | cyneol, pinen, cymen, borneol | antiseptic, stimulant, strengthening the memory | [Szumny et al. 2010] |
| Mint (*Mentha longifolia)* | mentol, mentofuran, menton, menthol acetate | relaxant, soothes an upset stomach, clears the respiratory tract, sedative, antiemetic, disinfecting | [Asekun et al. 2007] |

## 4. Effect of drying on the antioxidant properties and the loss of the compounds of anti-oxidant activity

The health-promoting properties of herbs and spices are largely determined by the oxidants they contain, that is, vitamins, polyphenolics, and carotenoid pigments. Both the method of drying and the species of the plant affect the antioxidant activity of plant materials. Increased temperature and water removal generally cause a loss of chemical compounds with antioxidant properties; however, products of enzymatic and non-enzymatic browning reactions may retain their antioxidants [Manzocco et al. 2001].

### 4.1. Effect of drying on the antioxidant properties

Comparing the antioxidant properties of oregano (*Origanum vulgare*), melissa (*Melissa officinalis* L.), and mint (*Mentha* L.), it was found that the plant with the highest capacity to prevent the oxidation of linoleic acid (expressed as total antioxidant capacity) was oregano (95%), followed by lemon balm (69%), and mint (62 %). Convection drying led only to a slight reduction in the antioxidant properties of the tested plants: by 10% in lemon balm, by 12% in mint, and by 5% in oregano [Capecka et al. 2005].

The antioxidant potential of oregano (*Origanum Majorana* L.) was determined using DPPH by Newerli-Guz [Newerli-Guz 2012]. The study involved 16 samples of oregano grown in Poland and Egypt. The ability to scavenge the DPPH radical, expressed in %, was a similar level and ranged from 88.70% to 90.66% for Egyptian oregano and 87.95–89.19% for Polish oregano.

FRAP and DPPH assays were used to determine the antioxidant properties of the dried spearmint. The antioxidant assays used are based on different mechanisms − DPPH assay is based on the ability of antioxidants to act as radical scavengers and FRAP assay measures the ability of antioxidants to perform as reducing agents. Both antioxidant assays showed that freeze-drying was the most effective drying method (FRAP 120 mg trolox/g MD, DPPH 90 mg trolox/g MD ) and the least effective was the microwave drying (FRAP 50 mg trolox/g MD, DPPH 5 mg trolox/g MD) [Orphanides et al. 2013].

## 4.2. Effect of drying on the loss of the compounds of anti-oxidant activity

### 4.2.1. Phenol content

The method of drying the raw material may have a negative impact on the content of antioxidants in the dried material, and thus on its antioxidant properties [Prabhanjan et al. 1995]. Total phenol content in ethanol extracts of dried leaves and stems of microwave-dried oregano (*Origanum vulgare*) and dried leaves of basil (*Ocimum basilicum*) was slightly reduced for oregano (about 21%), while a much greater decline was observed in the case of basil (70%), as compared to extracts from fresh raw material. Extracts from dried basil had a lower content of polyphenols, which means that the compounds contained in that plant are not very resistant to the action of high temperature, radiation, or oxygen [Nowacka et al. 2011]. In the case of ethanol extracts of microwave-vacuum dried or convection dried oregano, the amount of phenolic compounds depends on the process temperature. When convection drying was carried out at 60 or 70°C, the loss of polyphenols in the extracts amounted to over 80%. The use of microwaves resulted in a reduced loss of phenolic compounds in the studied extracts, and the higher the power used, the smaller the loss was, even by about 24% when power was increased from 240 W to 480 W, due to the shortening of the process [Jałoszyński et al. 2008].

Total phenol content in ethanol extracts of peppermint (*Mentha x piperita* L.) was higher by 16% in the case of drying under natural conditions (20–30°C) and by 45% in the case of microwave drying than in extracts from fresh material. This is the result of radiation destroying the tissue structure and facilitating the diffusion of polyphenolic compounds to the solvent. In contrast, convective drying at 50°C resulted in a reduction of polyphenol content in extracts by as much as 53%, indicating instability of these compounds at the process temperature or their enzymatic degradation [Arslan et al. 2010]. This can be limited by the use of microwave radiation, since during this process the contact time of the material with oxygen is shorter [Kramkowski 2001].

#### 4.2.2. Ascorbic acid

Ascorbic acid (vitamin C) is one of the best studied vitamins with antioxidant properties. It can neutralize the noxious activity of free radicals and lipid peroxides. Although ascorbic acid exhibits lower antioxidant potential than tannins, anthocyanins, and catechins, it should be stressed that its activity is multidirectional.

The drying process greatly affects the content of ascorbic acid (vitamin C) in dried raw materials. Following the process of convection drying, the content of vitamin C was reduced by 91% and 94% in the leaves of peppermint (*Mentha × piperita* L.) and melissa (*Melissa officinalis*), respectively, and by 82% in oregano (*Origanum vulgare*) [Capecka et al. 2005]. Drying leaves of spinach (*Spinacia* L.) at room temperature for three days led to a tenfold decrease in vitamin C content [Diplock et al. 1998]. Smaller losses of ascorbic acid were found after microwave drying: approximately 30% in marjoram (*Origanum majorana* L.) and 56% in rosemary (*Rosmarinus officinalis*) [Singh et al. 1996]. Microwave power has a significant impact on vitamin content during drying. Ozkan et al. found that increasing microwave power (from 90 W to 1000 W) led to retaining almost a double amount of ascorbic acid in spinach (41.79 as compared to 23.30 mg 100-1g-1) [Ozkan et al. 2007].

#### 4.2.3. Dyes: chlorophylls and carotenoids

While extending the shell life of products, the drying process can change their color.

Convection drying of leaves of amaranth resulted in less loss of chlorophylls than drying in the sun [Negi, Roy 2001]. Microwave drying allowed for better retention of chlorophyll a and b oregano (*Origanum vulgare*) and basil (*Ocimum basilicum)*, than convection drying [Witrowa-Rajchert et al. 2009].

During microwave-convective drying at 40°C the leaves of basil (*Ocimum basilicum*), lovage (*Levisticum officinale*) , oregano (*Origanum vulgare*), parsley (*Petroselinum crispum*) or arugula (*Eruca sativa*) resulted in a degradation chlorophylls, and its level was different for each species of herbs. High retention of chlorophyll a was observed in the lovage and basil, while losses of chlorophyll b was the higher at mint. Only in the case of lovage drying does not affect the outcome of the designation of chlorophyll a, while the content of chlorophyll b has not changed significantly in the basil, lovage and mint. For both types of chlorophyll pigments the greatest losses were observed in the arugula [Śledź, Witrowa-Rajchert 2012].

Convection drying has a significant impact on the degree of conservation of carotenoids in peppermint (*Mentha×piperita* L.), melissa (*Melissa officinalis* L.) and oregano (*Origanum vulgare*). The largest loss was observed in melissa (55%) and oregano (50%) and the greatest stability of carotenoids in mint (a decrease of 44%). This would indicate a significant effect of species on the loss of carotenoids in the plant material [Capecka et al. 2005].

The influence of the drying temperature (50 and 60 °C) and the different pretreatments (without pretreatment, osmotic dehydration and vacuum osmotic dehydration) on carotene content on parsley (*Petroselinum crispum*), were evaluated by Mahecha. The results showed that drying at 60 °C without pretreatment is the best process for drying parsley because the process time (68 min) and the moisture content (4,3%) are lower and carotene contents −12.66% losses of carotenoids (36.95 mg carotene/100 g sample) are less affected. The carotene content decreases at a higher rate in the process that takes more time to reach equilibrium moisture content due to its thermosensitivity [Mahecha et al. 2010].

The carotenoid in fresh, stored and processed spinach (*Spinacia oleracea* L.) was compared by Bunea et al. The stability of these compounds was evaluated during storage at 4 and 18 °C and after blanching, freezing and boiling. The major carotenoids were lutein (37–53 g/kg),b-carotene (18–31lg/kg), violaxanthin (9–23 lg/kg) and neoxanthin (10–22lg/kg). The content of carotenoids was best preserved after storage for one day at 4°C. Among individual carotenoids, lutein was most stable, followed by b-carotene, while violaxanthin being more polar and soluble became susceptible to degradation [Bunea et al. 2008].

## 5. The influence of impurities on the quality of the dried material

The antioxidant, bactericidal and bacteriostatic properties of herbs are increasingly being used in food processing. That is why microbiological status of added raw materials is very important. The causes of microbial contamination can be different − wrong way of harvesting and drying or inadequate packing, which lead to mold growth and mycotoxin production, which are toxic to humans [Makała 2010; Janda-Ulfig, Ulfig 2008]. The appendix of contaminated herbs cannot only contribute to the deterioration of product quality and shorten the shelf life, but it also poses a threat to the health and even the life of a man [Kunicka, Śmigielski 2011].

Water content in the raw material has significant influence on the development of microflora reducing the amount of results in the inhibition of enzymatic and non-enzymatic transformations and microbial inactivation. The presence of microorganisms can endanger the health of a consumer and affect the organoleptic and changes in the chemical composition of dried raw materials. The presence of *Bacillus pumilus* degrades pectin and sugars, *Pseudomonas sp.* metabolize essential oils, and the mold of the genus *Chaetomium, Fusarium* and *Aspergillus* decomposes phenolic compounds, which is the source of carbon for them. The occurrence of *Aspergillus* and *Penicillium* leads to the decomposition of the alkaloids, and the bacteria of the genus *Agrobacterium* and *Pseudomonas* metabolize glycosides [Asano, Komeda, Yamada 1993; Kunicka, Śmigielski 2011; Święcicka, Hauschild 1996].

## 6. Conclusions

Herbs and spices have a long history. They are natural antioxidants, and not only impart sensory qualities (flavor, aroma) to food, but also improve its microbiological stability [Wargovich et al. 2001; Hinneburg et al. 2006]. The observed increase of interest in herbs, both in culinary applications and in the cosmetic and food industries, has provided a stimulus for scientists to search for new drying methods, preserving the highest amount of biologically active compounds possible, as well as minimizing changes in the appearance, taste, color, and texture of the materials [Kaefer, Milner 2008; Sloan 2004; Lisiewska et al. 2006]. After drying, the amount of essential oils may be reduced by up to 70% (parsley), polyphenols by 70% (basil) [Boehm et al. 2002; Nowacka et al. 2011] and ascorbic acid by 90% (lemon balm, mint) [Asekun et al. 2007] as compared to the content of these components in fresh plant material.

The type and parameters of the drying process must be selected individually for each fresh plant material to obtain final products with sensory and functional properties comparable to those of the starting material.

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