UNIVERSITATEA LUCIAN BLAGA DIN SIBIU

Doctoral School of Engineering Sciences and Mathematics

Doctoral field:Industrial Engineering

PhD THESIS

Summary

RESEARCH ON THE OPTIMIZATION OF THE TECHNOLOGIES FOR OBTAINING ESSENTIAL OILS AND AROMATIC COMPOUNDS, WITH APPLICATIONS IN THE FOOD INDUSTRY

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SUMMARY

Through this PhD thesis, I wanted to explore a series of optimized industrial techniques and methods for the production of essential oils, simultaneously with the advancement of techniques and the development of analytical methods for their characterization.

The thesis focuses on two important related directions:research of industrial methods of production of natural essential oils (juniper fruit oil, oregano oil, thyme oil, coriander oil) and their use in the production of flavoring mixtures for various finished food products.

The documentary part includes 2 chapters in which are presented:

- The current state of knowledge in the field of essential oils and related legislation;
- Industrial applications of essential oils in the flavor industry and the perfumery industry;
- European legislative notices in the flavor and perfumery industry.Their impact in relation to natural essential oils;
- Presentation of the essential oils used in the food industry (oregano, coriander fruit, thyme, juniper;

The experimental part includes 4 chapters in which are presented:

- Materials and methods for the characterization of essential oils in order to determine the antibacterial potential;
- The use of essential oils in applications in the food industry (the potential of natural preservatives, flavoring preparations for sauces and meat products);
- Final conclusions
- Own contributions and future research and development directions;

The research was carried out during four years (2019 – 2023) and was carried out within the Solina Romania SRL and Esentivia SRL companies from Alba Iulia and data interpretation was carried out both within these companies and within the Center for Research in Biotechnologies and Food Engineers - Faculty of Agricultural Sciences, Food Industry and Environmental Protection from "Lucian Blaga" University

Content

INTRODUCTION

Essential oils are complex mixtures of various constituents, such as phenylpropanoids, esters, and mono-, sesqui-, di-, tri-, and tetraterpenes.in the current state of knowledge their therapeutic uses are related to the treatment of cancer, diabetes and cardiovascular and neurological diseases, in addition to having anti-aging, antioxidant and antimicrobial effects (Saljoughian et al. 2018;Benny and Thomas 2019;Bezerra et al. 2020b).Essential oils are obtained from plant materials, including flowers, roots, bark, leaves, seeds, bark, fruits, of whole plants (Hyldgaard et al., 2012).Throughout history, these volatile oils have been regarded with great interest, although many of their uses have been lost to time, it is generally accepted that humans have tried various methods and techniques to extract them from aromatic plants since the dawn of time. humanity.The applications of essential oils for different purposes are varied and include not only their use in the kitchen to enhance the taste of food, but also their use in the perfumery and cosmetics industry.

PURPOSE AND SCIENTIFIC OBJECTIVES OF THE DOCTORAL THESIS

The doctoral thesis "Research on the optimization of technologies for obtaining essential oils and aromatic compounds, with applications in the food industry" has the following objectives:

♦ Establishing analytical methods for characterizing essential oils (compositional, physico-chemical and organoleptic) in order to identify the optimal technological parameters for their industrial production.

♦Determination of the optimal technological parameters for the steam distillation process (entrainment time, steam flow rate, optimal amount of plant material/brash) in order to obtain improved entrainment yields and superior quality (compositional and organoleptic) for natural essential oils .

♦Determining the composition of natural essential oils:oregano, thyme, coriander seeds and juniper fruits, using specific analytical methods (gas chromatography GC-FID) and the identification of aromatic compounds with antimicrobial potential in their composition.

♦Determination of the main physico-chemical parameters (density, refractive index and rotational power) for essential oils and the influence of technological production parameters on them.

♦Analysis from an organoleptic point of view (appearance, smell) of natural essential oils with the aim of identifying the best qualities that will later be used in the preparation of flavoring mixtures.

♦Obtaining and characterizing mixtures of natural essential oils and investigating their potential antimicrobial properties.

♦ Structural, physico-chemical and organoleptic characterization of some flavoring preparations, obtained using natural essential oils.

♦Study of the influence of the origin of the vegetable raw material (geographical area) on the composition and physico-chemical and organoleptic properties, in the case of juniper essential oil obtained from juniper fruits harvested from the Balkans.

♦Obtaining and characterizing mixtures of natural essential oils used in the flavoring of some food products obtained on a pilot-industrial scale (pizza red sauce and "QSR" white sauce)

♦Establishing the optimal process parameters for the technological stages of industrial production of essential oils (oregano, thyme, coriander, juniper berries)

Keywords:essential oils, oregano, thyme, sauces, gas chromatography, industrial production.

CURRENT STATE OF KNOWLEDGE REGARDING THE OBTAINMENT, IMPORTANCE AND USES OF ESSENTIAL OILS.

Essential oils are complex mixtures of natural compounds, carotenoids, mainly monoterpenes and sesquiterpenes, different chemical groups of terpenes, aromatic hydrocarbons and their oxidized derivatives such as aldehydes, ketones, alcohols and esters obtained from aromatic plants.Many plant species synthesize and accumulate extractable organic substances in sufficient quantities used industrially as raw materials for various commercial applications.

Analytical methods and techniques used for the characterization of essential oils

The most common and classic specific analyzes to which essential oils are subjected are the following:

- Determination of physico-chemical parameters:relative density, refractive index, rotatory power, freezing point, evaporation residue, ethanol solubility, Acidity Index, Peroxide Index;
- Determination of composition:gas chromatography (GC) – most commonly used, thin layer chromatography (TLC), liquid chromatography (HPLC) – especially for the determination of furocoumarins in citrus oils.

Currently, however, the identification of essential oil components is usually performed by gas chromatography coupled to mass spectrometry (GC–MS) or flame ionization detector (GC-FID), using a capillary column (30 m × 0, 25 mm, with a film thickness of 0.25 μm).

aBased on average prices offered in 2007;

It can be concluded that the industrial use of essential oils is a very promising field and a steady annual growth will be seen in the future.Much research work will be undertaken both on the food safety of already existing products, but also on the development of new technologies and natural products (plant extracts) used as flavoring agents or as base products in the perfume industry.Both directions are equally important.Always over time there has been and will be a continuous trend to come up with something new in the industry, regardless of whether we are talking about food, perfumery, cosmetics, etc.Habits change, already established companies (eg:Coca Cola, McDonald's, Unilever, McKormick - flavors and others) will always be connected to the needs of the market and will continuously strive to come to the market with innovative, clean label products.This will undoubtedly lead to a constant development of the essential oil industry and an increase in their volumes, thus only bringing benefits to both the food and perfume and cosmetics sectors.

CHAPTER 2

PRESENTATION OF ESSENTIAL OILS USED IN FOOD INDUSTRY APPLICATIONS

2.1 OREGANO ESSENTIAL OIL (Origanum vulgare l.)

There are a large number of species worldwide that have been designated by the name of oregano, most belonging to the genera Origanum of the family Lamiaceae (vulgare, viride, virens, majorana and onites) and Lippia of the family Verbenaceae (graveolens) (Martínez-Rocha et al ., 2008).Oregano essential oil has been shown to be among the most effective

agents with antimicrobial and antioxidant properties (Fernández-Pan et al., 2012).

Table no. 1. Physico-chemical properties of oregano oils

Parameter	*O. vulgare*	*Th. capitatus*
αD - rotational power	-2 - +3o	-2 - +3o
nD20 – refraction index	1,502 – 1,528	1,500 – 1,510
d2020 – relative density	0,935 – 0,970	0,935 – 0,960
Solubility	1:2 in 70o ethanol (v/v)	
Content in phenols	65 - 75 %	60 – 75 %

2.2 CORIANDER ESSENTIAL OIL (Coriandrum Sativum l.)

Physico-chemical and organoleptic properties. Chemical composition

As physico-chemical and organoleptic characteristics, coriander fruit oil is a colorless or slightly yellowish liquid with a pleasant, sweet, woody-spicy and aromatic smell.The physico-chemical properties of the essential oil can be found in Table 11 below.Unlike the oil obtained from the fruit, the oil from the grass has a prominent intense aldehydic-fatty smell, reminiscent of the field bug.

Table no. 2. Physico-chemical properties of coriander fruit oil

Parameter	*Coriandrum sativum L.*
αD20 - rotational power	-8 - +15o
nD20 – refraction index	162 – 1,472
d2020 – relative density	0,862 – 0,878
Solubility	1:3 in ethanol 70% (v/v)
Acidity Index	Max. 3
Ester index	Max 22

2.3 ESSENTIAL OIL OF THYME (Thymus vulgaris l.)

As physicochemical and organoleptic characteristics, "red thyme" oil is a reddish-brown, reddish-orange or grayish-brown liquid with a rich and intense phenolic, sweet, warm herbaceous, slightly spicy and highly aromatic odor.

Table no. 3. Physico-chemical constants of "red thyme" oil, Thymus zygis

Parameter	*Thymus zygis*
αD20 - rotational power	-3 - 0o, difficult to measure
nD20 – refraction index	1,496 – 1,505
d2020 – relative density	0,915 – 0,935
Solubility	1:2 in 80% (v/v) ethanol
Content in phenols	38 – 56%

The wide variety of chemotypes that are known in the case of thyme oil is largely due to the influence of external factors, such as climate, soil, sun exposure, etc.

2.4 FRUIT JUNIPER ESSENTIAL OIL (Juniperus communis l.)

The essential oil of juniper fruit is a volatile oil widely used in industry, being a secondary metabolite of the plant juniperus communis l., a species that grows especially in alpine pastures, dry pine forests, river valleys or in areas with soil rich in moisture (Adams, 1998).The essential oil from juniper communis fruits is a mobile, colorless to slightly yellowish liquid, with a fresh, warm, rich, balsamic, woody-sweet smell and reminiscent of mountain pine.The tables below summarize the properties of this oil.

CHAPTER 3

PERSONAL CONTRIBUTIONS. EXPERIMENTAL PART OBTAINING AND CHARACTERIZING THE NATURAL ESSENTIAL OILS (OREGANO, THYME, CORIANDER, JUNIPER FRUIT).

3.2. Materials and methods

The protocol for the characterization of essential oils extracted from plant materials includes the stages shown in the block diagram in fig.1.

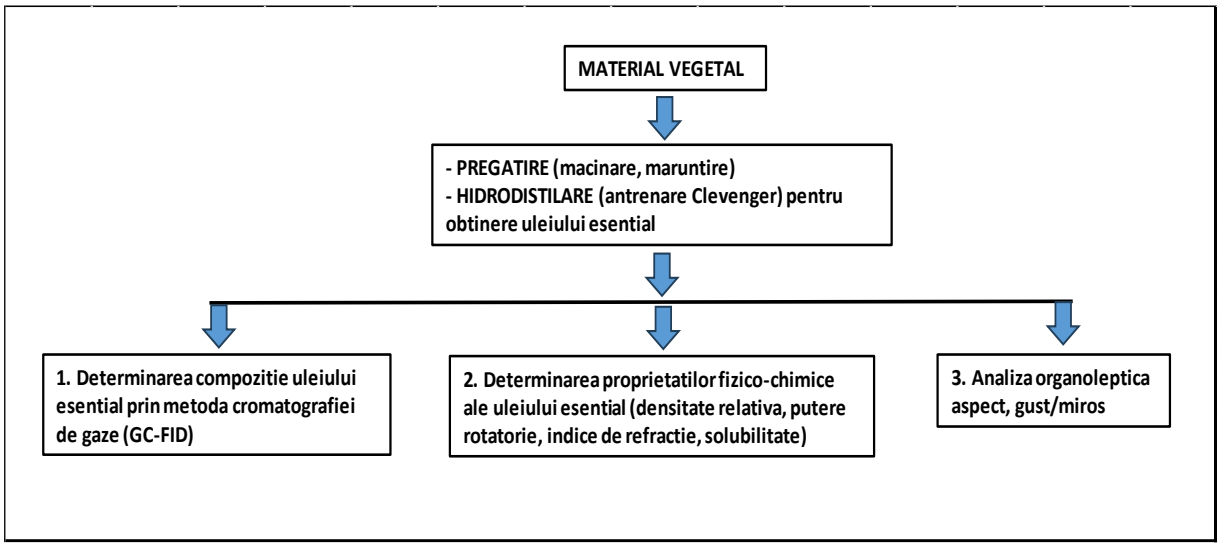

Figure no.1. The characterization protocol of essential oils

The plant material, as the case may be, oregano, thyme, coriander and juniper fruit, was obtained from the companies Solina Romania SRL, Esentivia SRL, from Romania, respectively Herba Natura doo - North Macedonia, respectively Alta Oils Ltd - Bulgaria.Solvents used:isopropyl alcohol, hexane, ethyl alcohol were purchased from Merck Milipore.

3.3. Results and discussion

3.3.1 Oregano essential oil (Origanum vulgare L.)

In the studied samples, analyzing the concentration of one of the main components, gamma-terpinene versus the variation of the concentration of the majority component carvacrol, a correlation between these parameters is observed with a good reliability factor R2 = 0.728.From a compositional point of view, if the technological process of steam entrainment respects the established parameters, the gamma terpinene content remains constant in relation to the carvacrol content.From the graph above, it can be seen that even with oils from plant material of different origins, the reliability coefficient R is a good one, the average concentration of gamma-terpinene being 5,060% and that of carvacrol 72,713% for the analyzed samples.

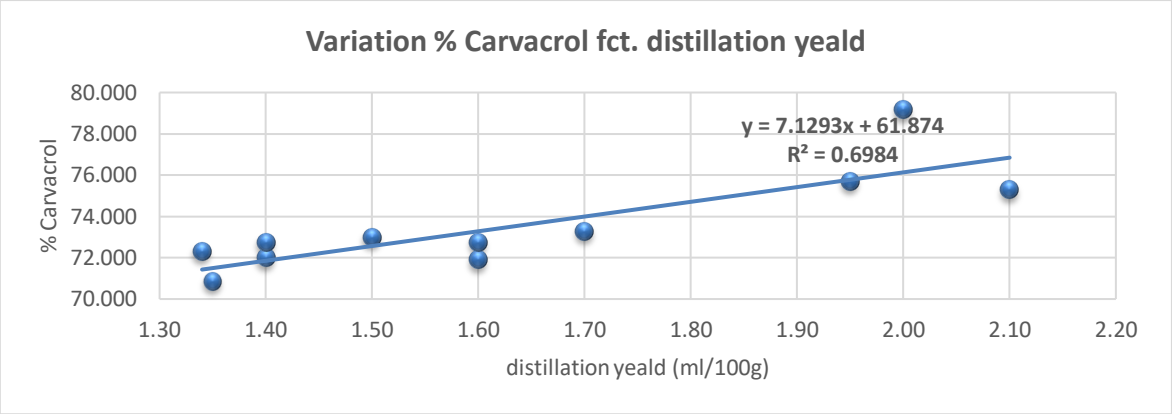

Figure no. 2. Gama Terpinen concentration correlation vs. Carvacol concentration
vulgare L.)

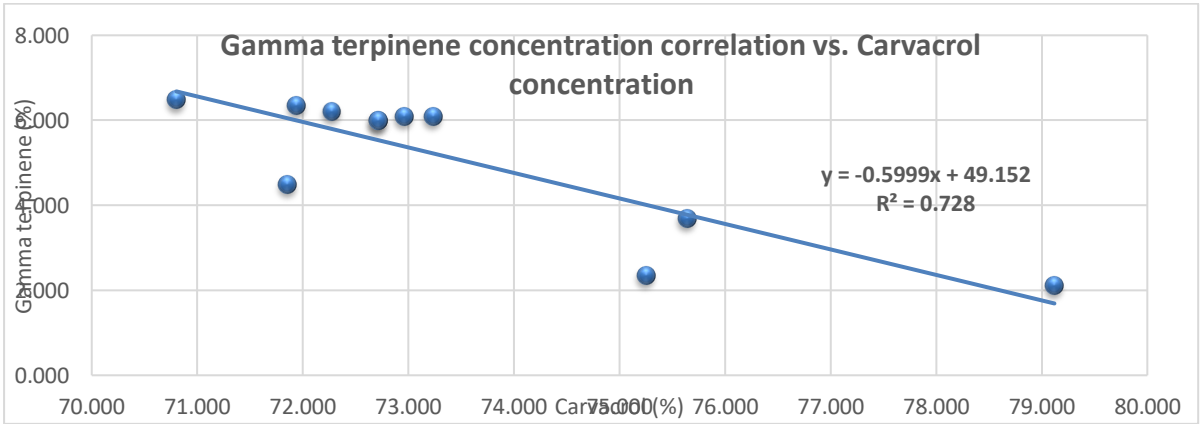

Figure no. 3. Variation of Carvacrol % according to distillation yeald

On the other hand, it is interesting to observe the correlation between the training yield values and the content of the major compound of interest (carvacrol).From the graph above, we can see this dependence with a good confidence factor, R2 = 0.6984.The samples obtained from the plant material of Turkish origin with the highest training yields (INAN, Manolya), but also the Macedonian material (MIN HERBA), also have the highest carvacrol concentrations.

Conclusions

Oregano essential oils obtained from raw materials originating in Turkey and Macedonia have the highest concentration of carvacrol, which is why their dosage in mixtures with antioxidant and antimicrobial properties can be reduced, this having a positive impact on the production cost of the recipe.

It was found that the raw material of Romanian origin has a concentration of carvacrol over 70%, but at the same time it also has a higher content of carvacryl-methyl-ether, the key component that gives the oil the possibility to be used in flavoring mixtures where the accentuation is desired the grassy, spicy note of the finished product.

3.3.2. Coriander essential oil (Coriandrum Sativum L.)

10 distinct batches of coriander oils were also analyzed, respectively:lot 834-RO, lot 804-RO, 7569-RO, 7606-RO, 7632-RO, 7609-RO, Star 180-RU, IND-RU, ARM100-RO, 793-RO.

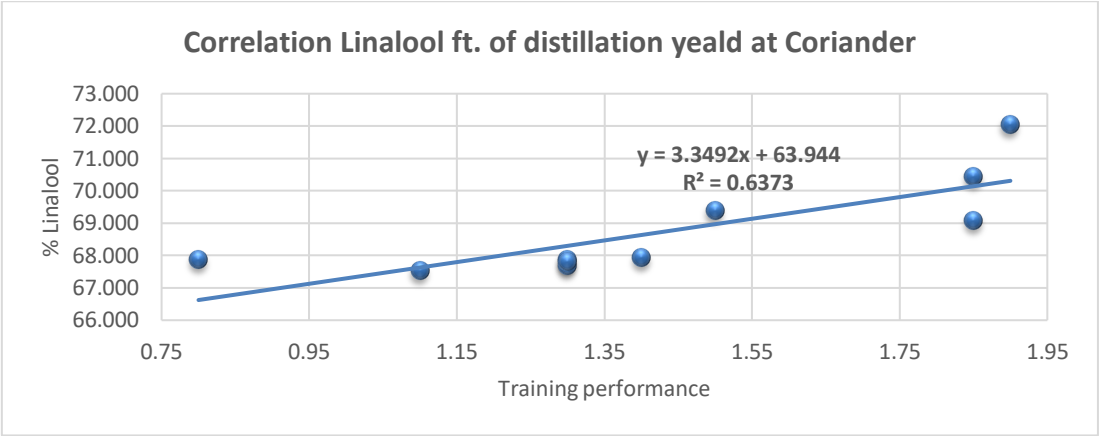

Figure no. 4. Correlation of Linalool as a function of coriander training yield

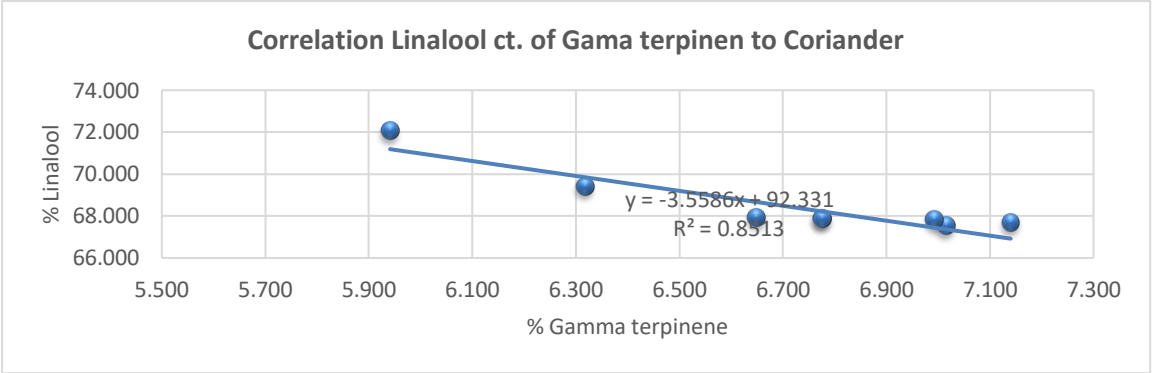

Figure no. 5. Correlation of Linalool according to Gamma terpinene in Coriander

From the point of view of the technological process of distillation, if the parameters remain constant, similar and constant structural and implicitly organoleptic profiles are obtained, this having a positive impact in the process of obtaining the flavoring preparation.

The characterization from the point of view of the physico-chemical and structural parameters shows that the plant material (coriander seeds) of Romanian origin (Galati area) offers an oil of the same quality as the one resulting from the Russian material (considered on the market as a standard of superior quality).This helps us to promote the production of essential oil from local sources, positively influencing the production costs (the Romanian material is about 40% cheaper than the Russian one).

The study on the vegetable raw materials to which we had access, helped us to obtain valuable information about the management of the distillation process and the quality of the thyme essential oil, thymol type obtained depending on the origin of the plant.From both a commercial perspective (clearly superior training yield for the Romanian oil, which implies a lower dosage in the recipe), but also from a compositional perspective (much higher thymol concentration), the Romanian oil is clearly superior and much more feasible in our flavoring applications and at the same time with antibacterial effects in the finished food product.

3.3.4. The essential oil of juniper fruit (Juniperus communis L.)

The essential oil of juniper fruits is obtained by steaming (water vapor) the ripe fruits of the plant juniperus communis l.The finished product obtained is widely used in the flavoring industry (gin flavor, in particular) but also in perfumery for its grassy, fresh notes.

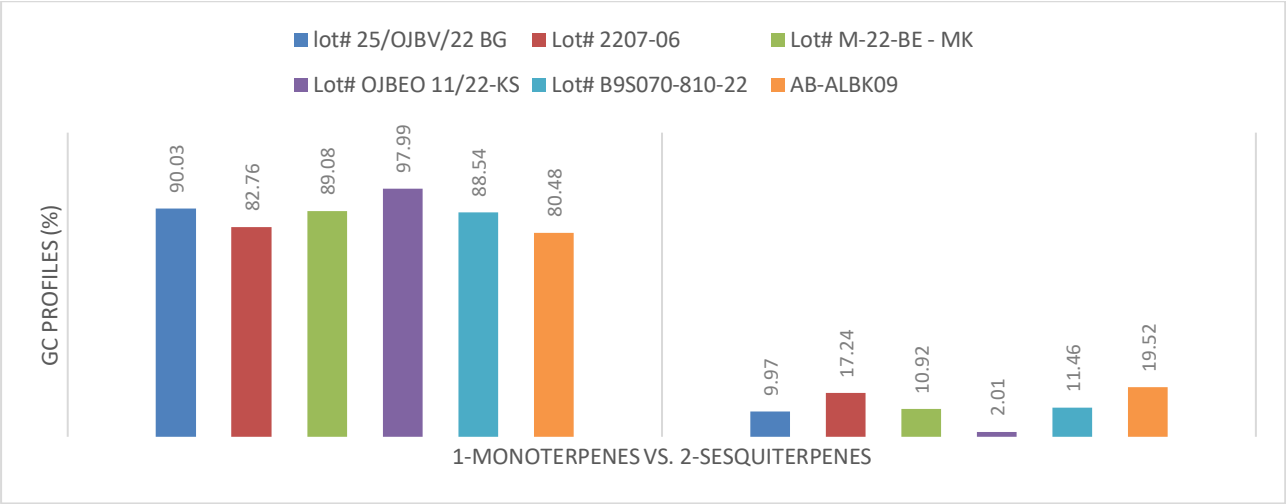

Figure no. 6. Graphic representation of the composition of the oil batches - monoterpenes vs sesquiterpenes

An important aspect in relation to the purpose of using juniper fruit oil assortments in obtaining flavoring preparations for the food industry, is the compositional structure from the point of view of monoterpenes and sesquiterpenes in the essential oil.

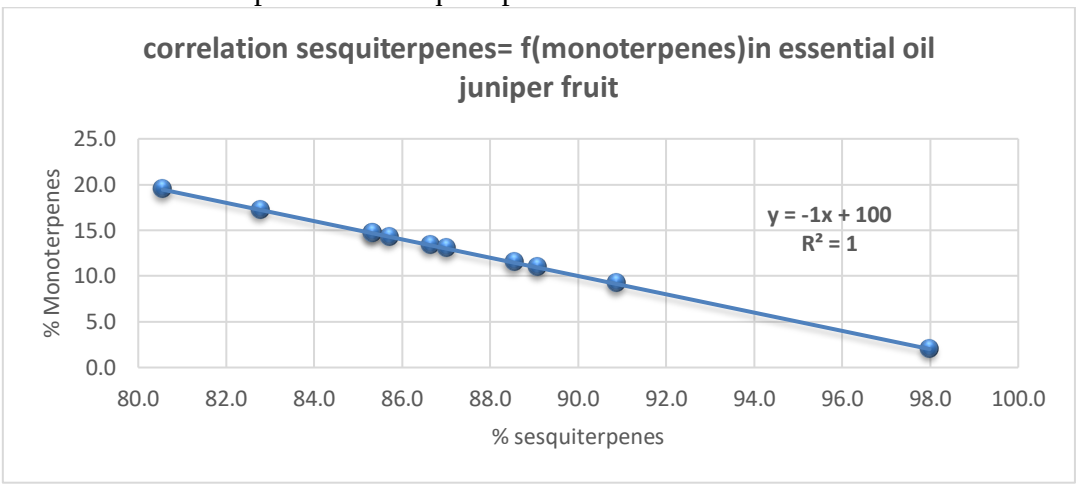

Figure no. 7. Correlation of sesquiterpenes = f(monoterpenes) in juniper fruit essential oil

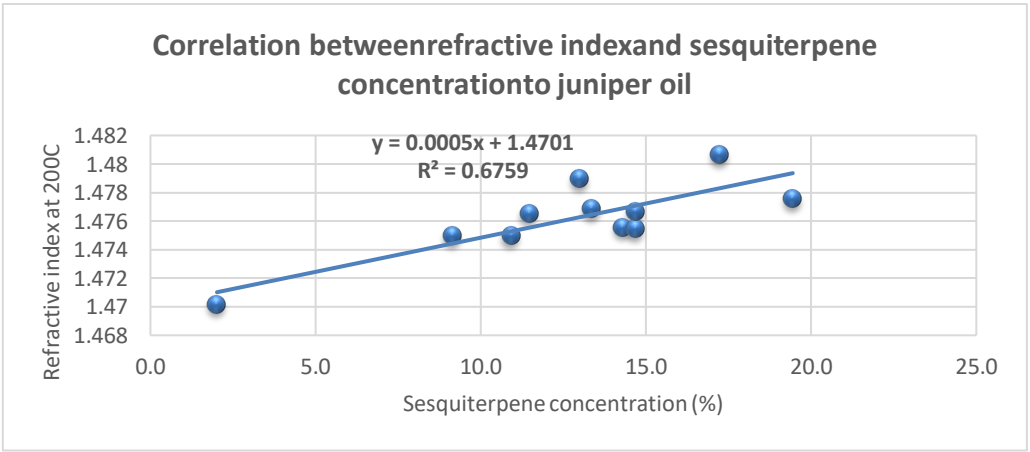

Figure no. 8. Correlation between refractive index and sesquiterpene concentration in juniper fruit oil

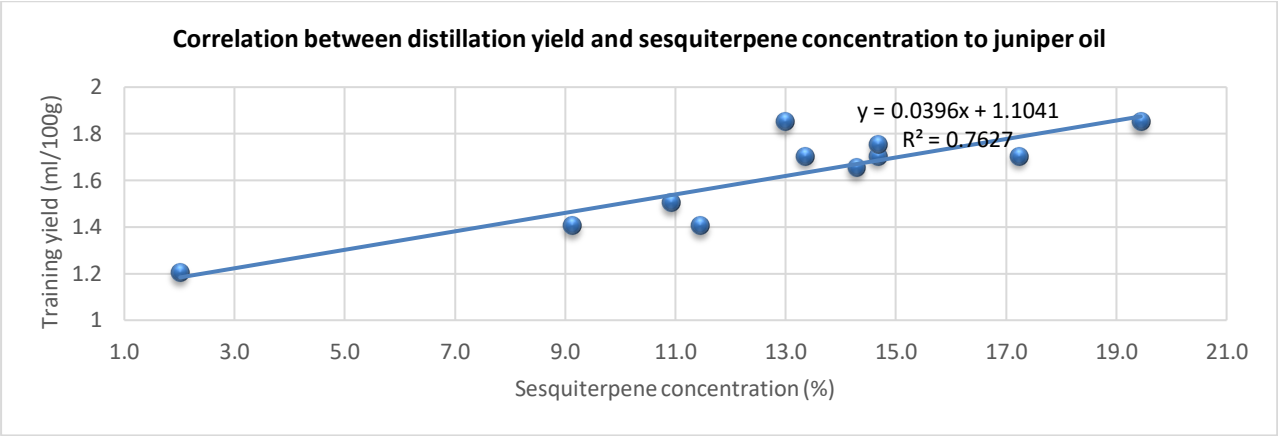

Figure no. 9. Correlation between training yield and sesquiterpene concentration in juniper fruit oil

The study shows us an assembly picture of the qualities of juniper essential oils available for use in the preparation of flavors for the food industry.

The geographical area where the plant grows and the specific weather conditions are the key parameters for the final quality of the finished product (essential oil), and on the occasion of this experiment we drew some valuable conclusions, namely that the juniper oils produced from local plant material rise to the level juniper oils from Albania from a qualitative point of view, some of them being even better from the point of view of sesquiterpene content (germacrene D), important compounds in applications for the production of natural flavors for the food industry.

CHAPTER 4

USE OF ESSENTIAL OILS IN THE FOOD INDUSTRY

4.1. Directions for using natural essential oils

Considering the organoleptic properties and the considerable antimicrobial/antioxidant activity of natural essential oils, as it appears from the specialized literature, we chose as the main direction of their use, which was also tested at an industrial level (pilot) and validated by potential clients of the Solina company:

- ***The use of thyme, coriander and oregano essential oils for the purpose of flavoring some products of interest (sauces) for a target customer, with special requirements, of Solina Romania.The products were obtained on the industrial equipment of the liquid department of Solina Romania.***
- ***The use of essential oils in order to produce flavoring preparations ("flavoring preparation") for various meat-based products (sausages, salami, sausages), etc.;***

4.2. The use of natural essential oils as flavoring agents in the industrial production of sauces

Technical infrastructure of the main work areas:

The main strengths and advantages of the product obtained through the optimized version proposed by us are:

- Shorter production time and lower energy consumption;
- Uniform appearance and taste throughout the meal, which do not depend on the correct homogenization or the agglomeration/sedimentation of the spices;
- High control and precise dosing of "flavor";
- Repeatability:the aroma is the same regardless of the batch of spices/harvest year/harvest area:essential oils can be standardized;
- all packages will have the same product:the taste given by the essential oils is found in the whole mass of the finished product;
- there are no differences in appearance between the packages that could appear due to the separation/agglomeration of spices;
- In the case of using the variant with essential oils, the possible discoloration of the spices over time is avoided.
- Finished "clean label" product, without preservatives.

4.3 Use of essential oils in flavoring preparations for various meat-based products

Considering the organoleptic properties and the considerable antimicrobial/antioxidant activity of natural essential oils, as it appears from the specialized literature, we have chosen two main directions of use:

- **Use for the purpose of producing flavoring preparations ("flavoring preparation") for various meat-based products (sausages, salamis, sausages), etc.;**
- **The purposeful use of preservatives - highlighting the antimicrobial, antioxidant properties of some essential oil mixes**

Figure no. 10. Sausage preparation

The mixture is kneaded according to the recipe and then sausages are obtained using the natural pork membrane. After obtaining the finished product, proceed to stage 2, cooking them.

Stage 2 – cooking the sausages flavored with CA smoked sausage flavor

In the Solina pilot food service station, an induction hob and a dedicated pan are used for frying sausages.Their preparation took about 10 minutes.The maximum temperature reached during cooking was 86 degrees Celsius in the product.

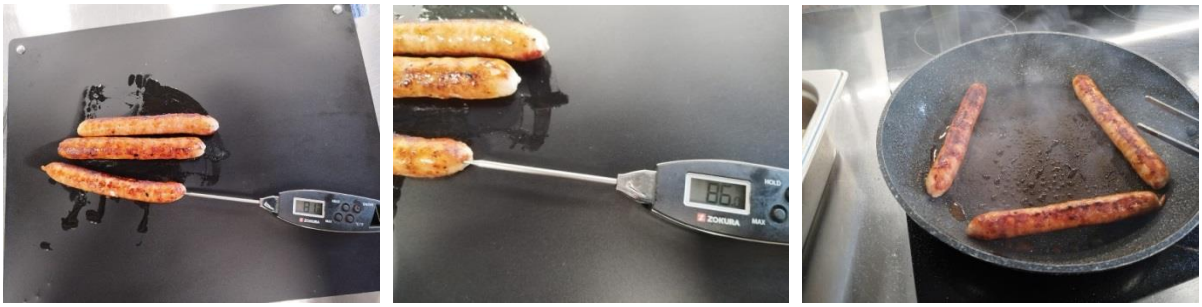

Figure no. 11. Cooking sausages

4.3.1. Discussions and interpretations

After the preparation and cooking of the fresh sausages, the tasting took place in the premises of the pilot food service station, which is an open-space restaurant type space, equipped with a professional kitchen dedicated 100% for product presentations and testing (tastings) in order to promote them to Solina customers .

At the tasting, tasting sheets were prepared according to the model below and a number of 20 people from the Solina R&D Laboratory, respectively the "Meat" pilot station, were present.

CHAPTER 5

FINAL CONCLUSIONS

Summarizing the main conclusions drawn as a result of the studies and research undertaken, the following conclusions can be outlined:

- Essential oils are complex mixtures of natural compounds, carotenoids, especially monoterpenes and sesquiterpenes, different chemical groups of terpenes, aromatic hydrocarbons and their oxidized derivatives such as aldehydes, ketones, alcohols and esters obtained from aromatic plants, being safe alternative sources for replacing synthetic products with antimicrobial, antiviral and antifungal activity.
- The most common specific analyzes for essential oil analysis are:
  - ✓ Determination of physico-chemical parameters:relative density, refractive index, rotatory power, freezing point, evaporation residue, ethanol solubility, Acidity Index, Peroxide Index;
  - ✓ Determination of composition:gas chromatography (GC) – most commonly used, thin layer chromatography (TLC), liquid chromatography (HPLC)
- Essential oils derived from plant raw materials are considered to be the most suitable alternative to replace synthetic food preservatives due to their strong antimicrobial, antioxidant properties, along with several advantages such as:lack of toxicity, products from the vegetable kingdom, eco-friendly, etc.
- Oregano essential oils obtained from raw materials originating in Turkey and Macedonia have the highest concentration of carvacrol, which is why their dosage in mixtures with antioxidant and antimicrobial properties can be reduced, this having a positive impact on the production cost of the recipe.
- From the point of view of the technological distillation process, if the parameters remain constant, similar and constant structural and implicitly organoleptic profiles are obtained, this having a positive impact in the process of obtaining the flavoring preparation.
- The characterization from the point of view of the physico-chemical and structural parameters shows that the plant material (coriander seeds) of Romanian origin (Galati area) offers an oil of the same quality as the one resulting from the Russian material (considered on the market as a standard of superior quality).This helps us to promote the production of essential oil from local sources, positively influencing the production costs (the Romanian material is about 40% cheaper than the Russian one).
- Having well-established and practically verified analytical methods at hand, we can continue the evaluation and research both in order to identify high-quality raw materials and in the direction of evaluating a wider range of flavoring mixtures with complex chromatographic profiles used in food solutions.
- The quality of oregano oils, depending on their field of use, is evaluated according to the content of carvacrol, a natural organic component responsible for excellent antimicrobial properties.Thus, the oils obtained from raw materials of Romanian origin (LOT 849 - RO, LOT 833 - RO, Lot 844 - RO, SAMPLE 7671 - RO, SAMPLE 7539 - RO) have an average carvacrol concentration of 72.80%, the one from Spain of 71.94% carvacrol, Turkey 75.44% carvacrol and North Macedonia 75.48%.An interesting aspect was observed in the MIN sample.HERBA-MK, which showed a carvacrol content (the highest of all analyzed samples) of 79.12%.

- Obtaining red "pizza" sauce and QSR sauce in the optimized version has proven to bring a number of advantages:
  - ✓ Shorter production time and lower energy consumption;
  - ✓ Uniform appearance and taste throughout the meal, which do not depend on the correct homogenization or the agglomeration/sedimentation of the spices;
  - ✓ High control and precise dosing of "flavor";
  - ✓ Repeatability:the aroma is the same regardless of the batch of spices/harvest year/harvest area:essential oils can be standardized;
  - ✓ the taste given by the essential oils is found in the entire mass of the finished product, so there are no differences in appearance and taste due to the separation/agglomeration of the spices;
  - ✓ In the case of using the version with essential oils, the possible discoloration of the spices over time is avoided.
  - ✓ The finished product is "clean label", without preservatives;

CHAPTER 6

OWN CONTRIBUTIONS AND FUTURE RESEARCH DIRECTIONS

The doctoral thesis sought to identify and establish the optimal parameters for obtaining natural essential oils widely used in the flavor industry, starting from the microproduction stage (laboratory) and reaching the industrial production stage.In this sense, the following was achieved:

- ➢ Obtaining and characterizing natural essential oils, namely oil of oregano, thyme, coriander seeds and juniper fruit, with the aim of obtaining reference data regarding their compositional and organoleptic profile, this being valuable information that will further help us to develop applications for food products .
- ➢ Establishing analytical methods for the characterization of essential oils (compositional, physico-chemical and organoleptic) in order to identify the optimal technological parameters for their industrial production.
- ➢ Determination of the optimal technological parameters for the steam distillation process (entrainment time, steam flow rate, optimal amount of plant material/brash) in order to obtain improved entrainment yields and superior quality (compositional and organoleptic) for natural essential oils.
- ➢ Determination of the composition of natural essential oils:oregano, thyme, coriander seeds and juniper fruits, using specific analytical methods (GC-FID gas chromatography) and the identification of aromatic compounds with antimicrobial potential in their composition.
- ➢ Determination of the main physico-chemical parameters (density, refractive index and rotational power) for essential oils and the influence of technological production parameters on them.
- ➢ Analysis from the organoleptic point of view (appearance, smell) of natural essential oils with the aim of identifying the best qualities that will later be used in the preparation of flavoring mixtures.
- ➢ Obtaining and characterizing mixtures of natural essential oils and investigating their potential antimicrobial properties.
- ➢ Development of an industrial technology optimized in terms of production yield and quality of the finished product obtained for juniper fruit essential oil.
- ➢ Major contributions have been made and new perspectives have been opened for the use of essential oils as flavoring agents in the industrial production of

sauces.

The topic addressed has great complexity and of course the ideas and results obtained by reaching the proposed objectives can be further continued in several directions, namely:

- The development of analytical methods with a superior accuracy (two-dimensional gas-chromatography methods GC x GC FID) for the determination of traces of volatile organic compounds (ppm) both in essential oils as such and in their mixtures.
- Characterization of essential oils and flavoring preparations by methods such as Gas Chromatography (GC) coupled to Time-of-Flight Mass Spectrometry (TOF-MS), which offer unique solutions for various analytical applications, including the analysis of food quality, authenticity markers and food safety.The coupling of gas chromatography (GC-FID) with mass spectrometry (MS) enables the identification and quantification of a wide range of trace compounds admissible by GC in complex matrices.
- Application of the analytical methods developed in order to identify the qualities of rosemary (rosmarinus officinalis), sage (salvia officinalis) oils, products with high potential for use in the food industry.
- The design of technological water vapor distillation installations with higher processing capacities (5 MC, 10 MC), and the quality of essential oils should not be affected by the industrial scaling stage.
- Research and development of a varied range of sauces and sauce bases using as flavoring and preservation agents various mixtures of essential oils obtained and qualitatively validated by the methods studied by us.

DISSEMINATION OF RESULTS

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