CZECH UNIVERSITY OF LIFE SIENCES PRAGUE

INSTITUTE OF TROPICS AND SUBTROPICS

M.Sc. thesis

Essentials Oil Analysis of Dried Vietnamese Coriander (*Persicaria odorata* Lour.) Grown in Thua Thien-Hue province, Vietnam

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2008

Declaration

I declare that I worked out the M.Sc. thesis "**Essentials Oil Analysis of Dried Vietnamese Coriander (*Persicaria odorata* Lour.) Grown in Thua Thien-Hue province, Vietnam**" on my own and all the quotations are mentioned in references.

In Pardubice 4.4.2008

Jan Ptáček

Acknowledgements

I express my sincere thanks to foundations **Nadání Josefa, Marie a Zdeňky Hlávkových** and funds of **Czech University of Life Sciences Prague** for the financial grants I was supported by during the work on my thesis.

I have a great pleasure to express my gratitude to supervisor **Ing. Jan Banout, Ph.D.** for his encouragement and constructive guidance during the run of our experiment and for his useful suggestions helping to compose my thesis. I am also very thankful for being given the opportunity to take part in "Sustainable Development in Phong My Commune" project.

I also thank to local project manager **Hien Le**, who helped us many times using his organizing skills and knowledge of local conditions and language. I gratefully acknowledge the **Hue University of Agriculture and Forestry** and the **NGO Tropenbos** for giving me the opportunity to work on my thesis in their premises.

I take this opportunity to acknowledge and thank to **Ing. Petr Ehl** for his personal affection and valuable help especially during the drying experiments and for his outstanding ability of solving any problem.

I express my sincere thanks to **Ing. Jaroslav Havlík, Ph.D.** and **Ing. Pavel Klouček, Ph.D.** for their help with essential oil analysis in the Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic and in laboratories of Faculty of Agrobiology, Food and Natural Resources. And Ing. Jaroslav Havlík, Ph.D. also for his invaluable help and leadership during results' evaluation.

ABSTRACT

The effect of drying on the essential oil (EO) constituents and yield in Vietnamese Coriander (*Persicaria odorata* Lour.), which is one of the typical flavours for Vietnamese cuisine, was studied.

Essential oil from fresh samples was compared to EO from double-pass solar-energy dryer and electric dryer. In the solar dryer, with the airflow powered by photovoltaic panel, the leaves were dried for 8 hours and the temperature ranged 35-63ºC; drying time in the electric dryer was set to 24 hours at 40ºC. Due to the beginning of rainy season the drying performance of the solar dryer varied strongly depending on the actual weather conditions. Final moisture content of the product reached by solar drying was in the average 16.11% (w.b.) while in the electric dryer 17.08% (w.b.) was reached. The essential oil of the fresh and dried leaves was isolated by hydrodistillation using modified Clevenger apparatus. The EO yield was higher from the fresh samples being 0.040% (w/w) while the yield from dried samples was slightly lower being 0.030 and 0.025% (w/w) for solar and electric dryer, respectively.

Refrigerated samples were transported from Vietnam to The Czech Republic and analysed by capillary gas chromatography and gas chromatography–mass spectrometry. In all samples, out of 24 constituents detected, 18 were identified presenting in average 96.15%. The main compound of the essential oil of Rau ram was *n*-dodecanal followed by decanal, dodecanol, decanol, bergamotene, α-humulene and undecanal. Out of the named main constituents drying had mild but statistically significant effect on four components – *n*-dodecanal, dodecanol, decanol, undecanal. The analyses proved notable difference in chemical composition between the two drying methods and fresh leaves. The difference in chemical composition was more remarkable when fresh and dried samples were compared.

Generally solar drying was found suitable for drying of *Persicaria odorata* with the positive fact, that in some aspects it had even milder influence on EO quality and composition than drying in electric dryer.

Keywords: *Persicaria odorata* Lour.; Vietnamese coriander; Rau ram; solar drying; essential oil; GC-FID; GC-MS; Vietnam

ABSTRAKT

Byl zkoumán vliv sušení na složení silice Rau ramu (*Persicaria odorata* Lour.), který je jedním z charakteristických koření vietnamské kuchyně.

Silice z čerstvých vzorků byly porovnávány se silicemi ze solární sušičky s dvojitým průchodem vzduchu a sušičky elektrické. V solární sušičce, ve které byl vzduch poháněn energií z fotovoltaického panelu, byly listy sušeny 8 hodin za teploty 35-63ºC; v elektrické sušičce byl čas sušení nastaven na 24 hodin a teplota na 40ºC. Kvůli začínajícímu období dešťů se výsledky dosažené solární sušičkou výrazně lišily v závislosti na aktuálních povětrnostních podmínkách. Konečná vlhkost produktu sušeného v solární sušičce dosáhla průměrné hodnoty 16,11%, zatímco v elektrické sušičce 17,08%. Silice z čerstvých i sušených vzorků byly extrahovány pomocí vodní destilace za použití Clevengerova aparátu. Výtěžek silic byl vyšší u čerstvých vzorků, kde dosahoval 0.040%, kdežto usušené vzorky vykazovaly mírně nižší obsah, který dosahoval hodnot 0,030% u solárního sušení a 0,025% u sušení v elektrické sušičce.

Zchlazené vzorky byly převezeny z Vietnamu do České republiky, kde byly analyzovány pomocí plynové chromatografie a hmotnostní spektrometrie. Ve všech vzorcích bylo z 24 zjištěných látek určeno 18, což představuje v průměru 96,15%. Hlavními látkami identifikovanými v silici Rau ramu byl *n*-dodecanal, dále decanal, dodecanol, decanol, bergamotene, *α*-humulene a undecanal. Sušení mělo mírný, ale statisticky prokazatelný efekt na čtyři látky - *n*-dodecanal, dodecanol, decanol, undecanal. Analýzami byly prokázány patrné změny v chemickém složení obzvlášť při porovnávání čerstvých a sušených vzorků.

Obecně můžeme říci, že solární sušení je pro *Persicaria odorata* vhodné a že navíc v některých aspektech mělo na kvalitu a složení silice dokonce mírnější vliv než sušení v elektrické sušičce.

Klíčová slova: *Persicaria odorata* Lour.; Rau ram; solární sušení; silice; GC-FID; GC-MS; Vietnam

PREFACE

Vietnamese cuisine is, in fact, based on original mixtures of herbs giving the meal typical and unique flavour. And Rau ram is one of really typical herbs being used mostly with poultry and fish. Usually it is used fresh and, to be honest, it is rare to hear of using dried Rau ram.

But people in developed countries often like exotic cuisine; they also try to have their dishes as authentic as possible. So they are consuming ever increasing amounts of fresh, frozen, processed and dried culinary herbs and spices, and this trend appears to be here to stay. There is a pressure from expanding ethnic populations demanding ingredients they are used to from their homelands. But the question is: “Are we able to produce the herbs indigenous in the tropics in acceptable quantity and quality within the temperate zone?” We guess not, and so suitable process of in situ processing of these herbs is to be discovered.

Drying is one of the oldest forms of food preservation methods known to man and is the most important process for preserving food since it has a great effect on the quality of the dried products. The major objective in drying agricultural products is the reduction of the moisture content to a level which allows safe storage over an extended period. Also, it brings about substantial reduction in weight and volume (Akpinar et al., 2006).

In recent times, energy demand has increased due to explosive growth of the world population, together with the increasing cost of conventional energy resources. Therefore, the need of the hour is development of low cost solar dryers (Tiwari et al., 1994).

Searching for methods of drying crops that consume less energy is also caused by increased costs during the past decade for fossil fuels used for drying crops (Abu-Hamdeh, 2003).

The most common system of drying crops in Thua Thien-Hue province is sun drying on the concrete patios or plastic sheets or simply on the road. Using solar dryer would bring higher product quality, less spoilage and accelerating the process.

If dried Rau ram was accepted by consumers, dried product from double-pass solar-energy dryer could become valuable source of income for the people in Thua Thien-Hue province.

ABBREVIATIONS USED IN THE THESIS

CFR	Crop and Food Research
CPEO	The Center for Public Environmental Oversight
CULS	Czech University of Life Sciences Prague
DM	Dry matter
EO	Essential Oil
FMC	Final Moisture Content
GC	Gas Chromatography
GC-FID	Gas Chromatography - Flame Ionization Detector
GC-MS	Gas Chromatography – Mass Spectrometry
GKSP	Gernot Katzer's Spice Pages
GM	Green matter
L4S	Library4Science
MC	Moisture Content
MPA	Mekong Protected Areas
NU	Newcastle University
PU	Purdue University
RH	Relative Humidity
RI	Retention Indices
RIRDC	Rural Industries Research and Development Corporation
WB	The World Bank

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1. INTRODUCTION

1.1 Vietnamese culinary herbs

Vietnamese cuisine has couple essential ingredients of which meat, rice or noodles and herbs are the most important. In fact, spices are quite rare and mostly are replaced by many kinds of herbs. Vietnamese people prefer communal food, which means it is prepared at the table together and usually one of the plates is full of mixture of fresh herbs.

The examples of the most common herbs used in Vietnam are as follows:

Coriander (*Coriandrum sativum*, fam. *Apiaceae*)

Coriander leaves (also called coriander green) are popular over the most part of Asia. The heartland of coriander leaf usage in South East Asia, however, is Vietnam. Particularly in South Vietnam, chopped coriander leaves appear as decorations on nearly every dish (sometimes combined with or substituted by peppermint or Rau ram) (GKSP, 2007).

The main compound of the seeds, which are used as spice, is linalool. Coriander seeds also stimulate enteric activity, though hey are used in folk medicine as carminative and stomachic (Valíček et al., 2002).

Spearmint (*Mentha spicata*, fam. *Lamiaceae*)

Spearmint has a distinct mint aroma and pleasing light flavour that is not pungent, warm or too antiseptic (RIRDC, 2001).

Basil (*Ocimum sp.*, fam. *Lamiaceae*)

Basil is one of the most pleasant spices, and indispensable for several Mediterranean cuisines. The sweet and aromatic fragrance is especially popular in Italy, but also often used in Vietnam (GKSP, 2007). Valíček et al. (2002) mentions the main producers being Mediterranean, USA, Madagascar, India and Thailand. Young leaves contain about 1,5% of essential oil with it's main constituents methylchavicol and linool. Leaves are used both fresh and dried (Valíček et al., 2002).

Perilla (*Perilla frutescens,* fam. *Lamiaceae*)

Aromatic but difficult to describe; the fragrance reminds of cinnamon and anise or licorice. The taste is similar, but with a marked astringent component. Leaves are, among other herbs, frequently served as a fragrant garnish to noodle soups and spring rolls (GKSP, 2007). Valíček et al., (2002) notices in East Asian cuisine young shoots and seeds are used as spice for rice, noodles and drinks flavouring.

Chameleon plant (*Houttuynia cordata,* fam. *Saururaceae*)

The Vietnamese name of the plant is diep ca [diếp cá], which is probably to be understood as “lettuce (smelling like) fish” (GKSP, 2007). According to Valíček et al. (2002) it comes from Himalaya where it is widely used in folk medicine as well as cooked leaves are eaten as a side-dish.

Piper sarmentosum Roxb. fam. *Piperaceae*

This plant is used much in Vietnamese cuisine as la lot, while no English name is known. It is also very common in Thailand for cuisine flavouring and is grown in many homegardens and crop plantations. Fresh leaves of *P. sarmentosum* are used to improve the flavour and smell of soups. Moreover, the leaves are generally eaten as vegetable (Chaveerach et al., 2006).

Lemongrass (*Cymbopogon citratus,* fam. *Poaceae*)

Vietnamese cookery makes use of lemongrass in several ways. A popular Vietnamese meal is bo nhung dam, often translated “vinegar beef” or “Vietnamese fondue”. At the table, each diner boils thin slices of beef in a vinegar-flavoured broth containing ample lemongrass. The beef is then, together with fresh vegetables and herbs (coriander, mint and Rau ram), wrapped in rice paper and eaten with spicy sauces based on fish sauce, lime juice, peanuts and chillies. This recipe demonstrates the Vietnamese preference for communal foods (prepared together at the table), for wrapped bits of food and for fresh herbs. Lemongrass is also used for Vietnamese curries, see rice paddy herb (GKSP, 2007).

The lemongrass is a plant of wide applications in tea preparations and its essential oil is used in perfume, food and pharmaceutical industries (Peisíno et al., 2005).

1.2 Characterization of *PERSICARIA ODORATA* (Rau ram)

Persicaria odorata belongs to family *Polygonaceae*. Synonym of *Persicaria odorata* is *Polygonum odoratum* Lour. We should also mention vernacular names as Rau ram (Vietnam), Vietnamese coriander (En).

1.2.1 Botanical characteristics

In popular horticulture handbooks confusion between *Persicaria odorata* and *P. Hydropiper* (L.) Spach (synonym: *Polygonum hydropiper* L.) is common. The synonym *Polygonum odoratum* is occasionally confused with *Polygonatum odoratum* (Mill.) Druce (*Liliaceae*) (de Guzman and Siemonsma, 1999).

Fig.1.1. *Persicaria odorata*

Source: de Guzman and Siemonsma, 1999

1.2.2 Origin

Rau ram (Fig. 1.1) is native to and is cultivated mainly in Indo-China. Since the 1960s its cultivation has spread with Vietnamese migrants, mainly to Australia, the Philippines and the United States (de Guzman and Siemonsma, 1999).

In addition to China, P. odoratum grows throughout Asia including Vietnam and Thailand where it is known as Pak pai or Vietnamese mint (Rafi and Vastano, 2007).

1.2.3 Uses

Rau ram is one of those herbs that give Vietnamese cuisine its unique flavour. The leaves are used both fresh and cooked; whereas fresh can be found especially in salads and also they are eaten as part of Vietnamese specialty called Trung long, which in fact is an incubated duck egg. Cooked leaves are mainly eaten with fish and chicken dishes, but also can be used with shellfish, turtle or frog meals.

It imparts a flavour reminiscent of lemon and coriander leaves with a slight radish-like pungent aftertaste. The flavour is destroyed by prolonged cooking. A few shoots of Rau ram and water dropwort (*Oenathe javanica* (Blume) DC.) are often added when preparing cabbage preserved in brine (like sauerkraut). Although relished by the Vietnamese, the flavour of Rau Ram is not universally admired, though it is liked by some people who do not appreciate the taste of coriander leaves.

Medicinally the leaves are used as a diuretic, stomachic, febrifuge and anti-aphrodisiac. Externally the crushed leaves are applied against fever, vomiting, ringworm and phagedaena. Juice prepared from the crushed leaves is taken as an antidote against poisonous snake bite and the bite is covered by the residue of the leaves. In Vietnam pregnant women avoid the use of Rau ram, since fresh leaves seem to have abortifacient properties (de Guzman and Siemonsma, 1999).

1.2.4 Properties

The nutritional value of Rau ram is unknown. The leaves contain a yellowish essential oil, consisting mainly of alkane aldehydes. Drimane sesquiterpenoids, which are under investigation for their antifungal and anticarcinogenic properties, are present in some *Persicaria* and *Polygonum* species, but have not been found in *P. odorata* (de Guzman and Siemonsma, 1999).

The roots of Rau ram have been used for a variety of therapeutic purposes in traditional Chinese medicine. It has been used as a crude medicinal agent in the treatment of analeptic and as a nutritious tonic in Asia. *P. odoratum* is a weed that grows throughout the southern United States and is commonly used as a condiment. *P. odoratum* alcohol extracts have been used as an immunopotentiator of mice injured by burns (Rafi and Vastano 2007).

1.2.5 Adulterations and substitutes

Besides coriander leaves, leaves of *Eryngium foetidum* L., known as sawtooth coriander or "Mexican coriander", are used as a substitute (de Guzman and Siemonsma, 1999).

1.2.6 Further description

It is a short-lived perennial, fragrant herb, somewhat glandular in all parts. Stem ascending, 30-35cm tall, 2-3mm in diameter, red, grooved; base trailing and forming roots at all nodes, much thicker than upright part. Leaves alternate; ocrea membranous, short, up to quarter of the length of the internode, loosely enveloping the stem, parallel veined, each vein culminating at apex in a long silky hair, with some glandular dots in horizontal lines; petiole attached to basal part of ocrea; blade entire, lanceolate to ovate-lanceolate, base attenuate, apex acuminate or obtuse, green, marked with red, margins and veins, especially the midrib, with appressed, fairly long hairs. Inflorescence an axillary, long, many-flowered, narrow spike, single or in pairs or in a small cluster; bracts long and funnel-shaped, with long hairs on margins; flowers hermaphrodite; perianth pentamerous, white to purplish-pink, persistent in fruit; stamens 8; styles 3. Fruit triangular, 1,5mm long, acuminate and both ends, smoth and shiny.

In Vietnam and Philippines flowering is profuse and starts in the first year. In Vietnam flowering occurs in August-September and fruiting in September-October (de Guzman and Siemonsma, 1999).

1.2.7 Ecology

Rau ram requires warm and humid growing conditions. Light frost isi probably tolerated. It grows best under partial shade, but full sunlight is tolerated if ample moisture is available. In Vietnam it can be grown and harvested year-round, but it grows best in spring. In cold and dry winters in northern Vietnam it may wither away. Fertile soils with adequate soil moisture are essential for optimal production. Under drier conditions, the stem base becomes woody and the leaves turn yellowish (de Guzman and Siemonsma, 1999).

1.2.8 Propagation and planting

Rau ram is usually and easily propagated by stem cuttings with 4-6 internodes (8-10cm long) taken from the top of mature stems. These are planted obliquely 5-6cm apart with a row spacing of 10-15cm in raised beds of light, well-manured soil and are watered well. Under warm and humid conditions cuttings start rooting after 3-5 days and growing after about a week. They are planted out in the field at a spacing of about 20cm x 20cm. In lowland Vietnam cuttings are sometimes planted directly in well-prepared and manured rice fields.

Rau ram can be rejuvenated by cutting back the stems (de Guzman and Siemonsma, 1999).

1.2.9 Diseases and pests

Occasional damage to Rau ram from diseases and pests is reported from Vietnam, but the casual agents are not known. In the United States fungal diseases and slugs sometimes affect Rau ram. Providing more sunlight can reduce damage. Caterpillars are the main problem in the Philippines (de Guzman and Siemonsma, 1999).

1.2.10 Harvesting, Yield and Handling after harvest

The first harvest of Rau ram is taken when plants are nearly two months old; subsequent harvests are every 12-15 days in Vietnam. In home gardens the leaves are picked when required. In commercial plantings whole tops are harvested.

Under very intensive cultivation in small market gardens yield of fresh tops of Rau ram may reach 1,3t/ha per harvest or about 15t/ha per year.

Leaves of Rau ram should be kept cool and moist to maintain their fresh appearance (de Guzman and Siemonsma, 1999).

1.2.11 Genetic resources and breeding

It is unlikely that germplasm collections of Rau ram are being maintained and there are no breeding programs (de Guzman and Siemonsma, 1999).

1.2.12 Prospects

Migration of Vietnamese people has greatly increased interest in Vietnamese cuisine in many countries, which will be a strong incentive to increase production of Rau ram. As people become more familiar with this flavour, Rau ram may become an alternative for coriander leaves, as many who do not like coriander find the taste of Rau ram more acceptable (de Guzman and Siemonsma, 1999).

1.3 Essential oil

Every essential oil is basically a mixture of different components / compounds. The percentage of these constituents in the oil plays an important part in determining its quality (Ahmed, 2005).

Essential oils are formed in special cells or groups of cells; and are generally found to predominate in one particular part or organ, such as leaves, flower calyces, fruit, roots, etc. These natural products are still used as raw materials in many fields, including perfumes, cosmetics, aromatherapy and phytotherapy, spices, and nutrition (Lahlou, 2004).

Crop and Food Research (2007) characterized essential oils as natural plant products which accumulate in specialized structures such as oil cells, glandular trichomes, and oil or resin ducts. Chemically, the essential oils are primarily composed of mono- and sesquiterpenes and aromatic polypropanoids synthesized via the mevalonic acid pathway for terpenes and the shikimic acid pathway for aromatic polypropanoids.

Essential oils are products, generally of rather complex composition comprising the volatile principles contained in the plants, and more or less modified during the preparation processes. They are also considered as complex mixtures of various aroma chemicals. So, each of these constituents contributes to the beneficial or adverse effects of these compounds. Synergistic as well as antagonistic actions are observed (Lahlou, 2004).

1.3.1 History

The essential oils which were regularly used in ancient Rome, Greece, and Egypt and throughout the Middle and Far East had, as a common feature, the essence of a plant; an identifiable aroma, flavour, or other characteristic that was of some practical use. They were used as perfumes, food flavours, deodorants, pharmaceuticals, and embalming antiseptics. Usually, plant material was steeped in a fatty oil or wine that acted as a solvent for the desired flavour or aroma. The extracts (usually impure and dilute) were used as oils or creams (CFR, 2007).

The quality and price of some oils are based on the percentage content of a single chemical component, so separation and measurement of individual components is very important. This is usually done using some form of chromatography; the most powerful is gas chromatography using capillary columns (CFR, 2007).

1.3.2 Essential oil production

The original approach involved collecting plants from wild populations and extracting oils for local use. Cheap manual labour of peasant or native workers was used.

The second approach is today's intensive cropping industry used in agriculturally "developed" countries. It uses all of the modern methods of mechanization and crop and financial management. Selections for high yields, high quality oil, and good agronomic features have greatly improved crop performance.

A third approach is still at the research stage. Artificial cultures of plant cells of a few species can produce some of the chemical compounds that characterize an essential oil. Attempts are being made to find and establish high producing lines of cells, using genetic manipulation together with tissue culture and liquid culture engineering. This system will probably produce single components rather than a complete essential oil. This is also true of production by synthetic chemical engineering which is now common for components used in cheaper products (CFR, 2007).

The essential oils from aromatic plants are for the most part volatile and thus, lend themselves to several methods of extraction such as hydrodistillation, water and steam distillation, direct steam distillation, and solvent. The specific extraction method employed is dependent upon the plant material to be distilled and the desired end-product. The essential oils which impart the distinctive aromas are complex mixtures of organic constituents, some of which being less stable, may undergo chemical alterations when subjected to high temperatures. In this case, organic solvent extraction is required to ensure no decomposition or changes have occurred which would alter the aroma and fragrance of the end-product. Newer methods of essential oil extraction such as using supercritical CO_2 which yield very high quality oils are commercially used, but are less common and beyond the financial means of most processors (CFR, 2007).

The recovery of non-volatile essential oils is also obtained by solvent extraction although the process is more difficult and complex than the recovery of the volatiles. This process yields an aromatic resinous product known as an oleoresin, which is more concentrated than an essential oil and which has wide application in the food industry (PU, 1990).

As a general rule, any essential oil should first be treated to remove metallic impurities, freed from moisture and clarified, and then be stored in well-filled, tightly closed containers, at low temperature and protected from light (Ahmed, 2005).

It is also clear that only a detailed knowledge of the mechanism of their action contributes to give more consideration of such compounds, and then it will lead to a proper use in different domains of application (Lahlou, 2004).

1.4 Distillation

Distillation may be characterized as a process in which a liquid or vapour mixture of two or more substances is separated into its component fractions of desired purity, by the application and removal of heat.

It is based on the fact that the vapour of a boiling mixture will be richer in the components that have lower boiling points. Therefore, when this vapour is cooled and condensed, the condensate will contain more volatile components. At the same time, the original mixture will contain more of the less volatile material.

It is also to be mentioned that:

- distillation is the most common separation technique
- it consumes enormous amounts of energy, both in terms of cooling and heating requirements
- it can contribute to more than 50% of plant operating costs

(NU, 1997)

Distillation accounts for the major share of essential oils being produced today. The choice of a particular process for the extraction of essential oil is generally dictated by the following considerations:

a) Sensitivity of the essential oils to the action of heat and water.

b) Volatility of the essential oil.

c) Water solubility of the essential oil.

As most of the essential oils of commerce are steam volatile, reasonably stable to action of heat and practically insoluble in water hence are suitable for processing by distillation.

1. Water or hydro distillation
2. Steam cum water distillation
3. Steam distillation (Ahmed, 2005)

Water / Hydro distillation

Water or hydro distillation is one of the oldest and easiest methods being used for the extraction of essential oils. In this method the plant material is fully dipped in the water.

In literature (Ahmed, 2005) there is mentioned a primitive method based on this principle: In the Bhapka method the distillation still is made of copper. The still is fitted on brick furnace and the plant material is filled in the still and entirely covered with water to the top. Another copper vessel with a long neck is placed in a water tank or natural pond to serve as condenser. A bamboo pipe is used to connect the vapour line and mud is used to seal the various joints. The oil vapour along with steam is condensed in the copper vessel and is separated. But this process suffers from serious drawbacks such as:

1. As the plant material near the bottom walls of the still comes in direct contact with the fire from furnace, there is a likelihood of its getting charred and thus imparting an objectionable odour (burning note) to the essential oil.
2. Prolong action of hot water can cause hydrolysis of some constituents of the essential oils such as ester, etc.
3. The process is slow and the distillation time is much longer thereby consuming more firewood / fuel (Ahmed, 2005).

Water cum Steam Distillation (Field Distillation Unit)

To remove the above drawbacks of water distillation the design has been improved where a perforated grid / net is introduced into the still just above the bottom. This type of unit is known as Field Distillation Unit. The main components of this process are:

1. Distillation still (usually made of stainless steel or food grade SS)
2. Condenser (usually tubular multitube condenser)
3. Oil separator / receiver
4. Brick furnace

The still / tank is made up of mild steel / stainless steel with a perforated grid and is fitted directly on a brick furnace. The plant material is kept on the grid and water is filled below it. The tank is connected to the condenser through a vapour line. The condensate oil / vapour mixture is separated in the oil separator.

Due to their very simple construction, low cost and easy operation the field distillation units are extremely popular. The furnace is fuelled by spent agro-waste, firewood. These types of units are currently being used for the distillation of citronella, lemongrass and agarwood, etc (Ahmed, 2005).

Steam Distillation

The main components of steam distillation unit are:

1. Distillation tank with steam coil
2. Condenser (usually multi-tube tubular)
3. Oil separator or receiver
4. Boiler

Steam distillation exploits the twin action of heat and moisture from steam to break down the cell walls of the plant tissues to liberate the essential oil. Steam is generated separately in a steam boiler and is passed through the distillation tank through a steam coil. The plant material is tightly packed above the perforated grid (false net). Steam along with oil vapours is condensed in the condenser and is separated in the oil receiver. Capital cost of putting up steam distillation unit is higher and also a trained person is required for operation of the boiler. Steam distillation is preferred where a lot of area is under cultivation and more than one unit is installed. Also for distillation of high boiling oils such of roots / woods for example agarwood chips), patchouli, etc (Ahmed, 2005).
Study of Chemat et al. (2006) enhanced steam distillation using microwave oven, which fastened the process (10 instead of 90 minutes) and saved energy.

Supercritical fluid extraction

Within this method instead of organic solvents, supercritical carbon dioxide (SC-CO_2) is used. Above its critical pressure and temperature, carbon dioxide behaves both like solvent and gas with higher diffusivity and density but lower viscosity (Wei, 2005).
Over the last two decades, supercritical fluid extraction has been well received as green and promising technology for extraction of natural products. The most significant advantages of using SC-CO_2 are its chemical inertness; its non-toxic, non-hazardous and non-inflammable characteristics; it leaves no solvent residue in the products and the process generates no waste, therefore it is environment benign technology (Wei, 2005).
El-Ghorab el al. (2004) stated that supercritical fluid extraction was about three times more efficient than hydrodistillation as yields of volatile compounds of marjoram leaves were compared.

1.5 Drying effect

The use of spices has increased significantly over the past few years, partly due to renewed interest in dishes that use a wide variety of spices. Consumers prefer high quality, which is mainly defined by essential oil content in the material. Spices need to be dried before they are stored but loss of essential oils is unavoidable during drying processes (Arabhosseini et al., 2006a).

Food drying is a traditional method of food preservation, which is also used for the production of special foods and food ingredients. Drying not only affects the water content of the product, but also alters other physical, biological and chemical properties such as enzymatic activity, microbial spoilage, viscosity, hardness, aroma, flavour and palatability of foods (Özbek and Dadali, 2007).

Drying prevents the growth of micro-organisms that may cause spoilage or food poisoning. The essential oil constituents of herbs can be adversely affected during drying. The effect of drying on the flavour and fragrance of different crops has been studied intensively; for example, Australian-grown ginger, basil, bay leaf, parsley, Roman chamomile, spearmint, thyme and sage and aromatic plants in general. These studies show that changes in the concentration of essential oils during the drying process depend on several factors like temperature and relative humidity of the drying air as well as the product properties (Arabhosseini et al., 2006a).

From the economic point of view drying also minimizes packaging requirements and reduces shipping weights. However, it has some problems related to the contamination with dust, soil, sand particles and insects, and being weather dependent. Also, the required drying time can be quite long (Doymaz, 2006).

Doymaz (2008) also mentions that drying enables storability of the product under ambient temperatures.

1.6 Solar dryers

Sun drying (without drying equipment) is the most widely practised agricultural processing operation in the world; more than 250 000 000t of fruits and grains are dried by solar energy per year. More sophisticated methods (solar drying) collect solar energy and heat air. Both solar and sun drying are simple inexpensive technologies. Energy inputs and skilled labour are not required and in the sun drying, very large amounts of crop can be dried at low cost (Fellows, 2000).

The introduction of solar crop dryers seems to be a way to lower mass losses compared to traditional drying methods and improves the quality of the product considerably (El-Sebaii et al., 2002).

In the literature there have been many successful experiments with solar drying described. Hellickson et al. designed a multi-purpose solar agricultural system which provided an energy equivalent of about 1.0 kWh/day/m^2 during field tests for grain drying as well as for air preheating for ventilation purposes. Calderwood developed a prototype system for rice drying by heating air with a solar collector array with a 96 m^2 area of roof space. Kok and Kwendakwema designed and built a small-scale forced convection indirect solar food dryer used for vegetable and meat drying during the Zambian winter season. The tested foods were dried to below 15% moisture in two or three days of the operating period. Raju et al. designed a forced convection drying system with a loading capacity of 500 kg of grapes per batch. The final yield was 120 kg of raisins and the drying time required was 56 hrs. The average system efficiency was 12.5%. Dillao et al. investigated the use of a natural ventilation solar dryer for drying fish. The overall drying rates indicated that a residential scale dryer could operate on a two-day cycle and provide quality dried products. All and Mathur designed and developed a solar-based one ton capacity grain dryer. The dryer consists of a fiat-plate air heating panel of a 4.5m x 4.5m area, two blowers, a drying chamber and two drying bins. The authors found that re-wetted maize grain at 20% moisture could dry up to 12% in one solar day. Lawrence et al. designed a small solar crop dryer, consisting of a drying unit, thermal storage, and solar collector for the climatic conditions of Raupua, New Guinea. The dryer was tested, and the detailed experimental studies were carried out for the drying of tapioca (Zahed and Elsayed, 1994).

1.6.1 *Solar energy*

Solar energy is one of the most promising renewable energy sources in the world. Compared to non-renewable sources such as fossil fuels, the advantages are clear: it is totally non-polluting, has no moving parts to break down, and does not require much maintenance. Solar generators can be installed in a distributed fashion, on each farm or building, using area that is already developed, and allowing individual users to generate their own power, quietly and safely (Abu-Hamdeh, 2003).

Solar radiation spans the spectrum from short-wavelength ultraviolet to visible light to long-wavelength infrared. Visible light is approximately 47 % of the incident extraterrestrial solar radiation. Visible wavelengths are from 380 to 780 nanometres. About 75 % of the sun's radiation passes through the Earth's atmosphere and reaches the surface (Fodor, 2005).

All the energy that we detect as light and heat originates in nuclear fusion reactions deep inside the sun's high temperature core. This core extends about one quarter of the way from the centre of the sun to its surface where the temperature is around 15 million ºC (Fodor, 2005).

Each day the sun rises, warms the Earth back up, and powers the entire biosphere. On a clear day, up to 1,000 Watts of solar energy are available for our use per square meter of the Earth's surface. Typical operating conditions for a solar dryer vary depending on weather, geographic location, season and time of day (Fodor, 2005).

Though the level of insulation at ground level varies greatly with time of the year, time of the day and atmospheric conditions, the level of extra-terrestrial radiation remains fairly constant. Many measurements have been made of the intensity of extra terrestrial radiation. This parameter is called the Solar Constant and its value as stated by Duffie and Beckman (1980) is 1,353 Wm^{-2}.

1.6.2 *The principle of solar drying*

Drying is simply the process of moisture removal from a product. It can be performed by various methods for a variety of different substances from solids to gases and even liquids. Drying can also be achieved mechanically by compression, centrifugal forces or gravity. Thermal drying, which is the form most commonly used for drying agricultural products, involves the vaporisation of moisture within the product by heat and its subsequent evaporation from the product (Ekechukwu, 1999).

Drying is a dual process of:

- heat transfer to the product from the heating source and
- mass transfer of moisture from the interior of the product to its surface and from the surface to the surrounding air.

Drying involves the extraction of moisture from the product by heating and the passage of air mass around it to carry away the released vapour. Under ambient conditions, these processes continue until the vapour pressure of the moisture held in the product equals that held in the atmosphere (Ekechukwu and Norton, 1999).

Solar dryers need some form of solar collector so that the air can be heated before directing it to the process of drying. This is usually done by fixed flat plate collectors, mounted at a certain slope and orientation (Bari, 2000).

Good ventilation is of crucial importance. It determines on the one hand the exchange of warmth from the absorbent surface to the air next to it and on the other hand the evaporation of the water on and in the product. Stronger ventilation leads to a lower average temperature but also to a more efficient overall transfer of warmth. This leads to reduction in the relative humidity and improved drying (Vanderhulst et al., 1990).

Much of the heat necessary for evaporation of moisture from a particle is supplied from the air by convection but conduction and radiation of heat to the particle can also be important, particularly so for those solar dryers with the drying chamber exposed to the sun (Brenndorfer et al., 1985).

It depends on climatic conditions, and requires a large surface and a long time of exposure to sun light and, in particular, to ultraviolet rays. This leads to deterioration of the dried product. Furthermore, this method does not enable the drying of large quantities (Bennamoun and Belhamri, 2003).

Amir et al., (1991) states the major problem in processing agricultural products, especially in the drying stage confronting farmers in tropical countries, is to remove the moisture from the products as fast as possible with very simple methods, to reach the final moisture content required for safe storage.

Seasonal changes of solar radiation suggest use of solar drying in the maximum radiation intensity season. Matching the drying process and the specific characteristics of solar radiation is also important in governing the investment costs. Because of small flux density of solar radiation, a high-temperature drying medium can only be provided with concentrating collectors. Such collectors are generally very expensive. Cheaper, flat-plate collectors, on the other hand, can be applied only for producing a moderate-temperature medium (usually under 60°C) and their efficiency improves with decrease in operation temperature (Mujumdar, 1987).

1.6.3 Basic division of dryers

Conventional drying systems are usually classified (according to their operating temperature ranges) into low and high temperature dryers. In the low temperature drying systems, the moisture content of the product is brought into equilibrium usually with the drying air by constant ventilation. High temperature dryers are used when fast drying is desired and crops require a short exposure to the drying air (Ekechukwu, 1999).

Different heat sources are employed for the drying of agricultural products, the most common being fossil fuels, electricity and solar energy. Since the mid 1950's, an extensive amount of work has been reported on the basic principles and fundamental theories of crop drying (Ekechukwu, 1999).

Fig. 1.2 illustrates a systematic classification of drying systems, indicating the sub-classes and the group lineage of solar drying systems (Ekechukwu and Norton, 1999).

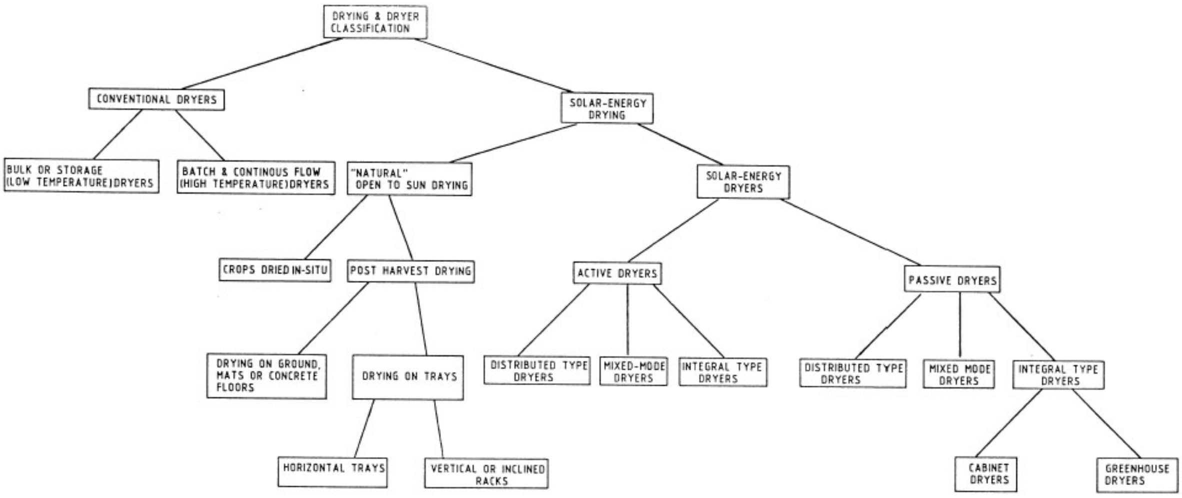


Fig.1.2. Classification of dryers and drying modes

Source: Ekechukwu, Norton, 1999

Strictly, all practically-realised designs of high temperature dryers are fossil fuel powered, while the low temperature dryers are either fossil fuel or solar-energy based systems (Ekechukwu and Norton, 1999).

High temperature dryers are usually employed when the products require a short exposure to the drying air. Their operating temperatures are such that, if the drying air remains in contact with the product until equilibrium moisture content is reached, serious over drying will occur. Thus, the products are only dried to the required moisture contents and later cooled. High temperature dryers are usually classified into batch dryers and continuous-flow dryers. In batch dryers, the products are dried in a bin and subsequently moved to storage. Thus, they are usually known as batch-in-bin dryers. Continuous-flow dryers are heated columns through which the product flows under gravity and is exposed to heated air while descending. Because of the temperature ranges prevalent in high temperature dryers, most known designs are electricity or fossil-fuel powered. Only a very few practically-realised designs of high temperature drying systems are solar-energy heated (Ekechukwu and Norton, 1999).

In low temperature drying systems, the moisture content of the product is usually brought in equilibrium with the drying air by constant ventilation. Thus, they do tolerate intermittent or variable heat input. Low temperature drying enables crops to be dried in bulk and is most suited also for long term storage systems. Thus, they are usually known as bulk or storage dryers. Their ability to accommodate intermittent heat input makes low temperature drying most appropriate for solar-energy applications. Thus, some conventional dryers and most practically-realised designs of solar-energy dryers are of the low temperature type (Ekechukwu and Norton, 1999).

1.6.4 Classification of solar-energy drying systems

Solar dryers can broadly be categorised into direct, indirect and specialised solar dryers (Kreider and Kreith, 1981, in Mwithiga and Kigo, 2006). Direct solar dryers have the material to be dried placed in an enclosure, with a transparent cover on it. Heat is generated by absorption of solar radiation on the product itself as well as on the internal surfaces of the drying chamber. In indirect solar dryers, solar radiation is not directly incident on the material to be dried.

Air is heated in a solar collector and then ducted to the drying chamber to dry the product. Specialised dryers are normally designed with a specific product in mind and may include hybrid systems where other forms of energy are also used (Mwithiga and Kigo, 2006).

Although indirect dryers are less compact when compared to direct solar dryers, they are generally more efficient (Mwithiga and Kigo, 2006).

Three distinct sub-classes of either the active or passive solar drying systems can be identified (which vary mainly in the design arrangement of system components and the mode of utilisation of the solar heat), namely:

- integral-type solar dryers;
- distributed-type solar dryers; and
- mixed-mode solar dryers (Ekechukwu and Norton, 1999).

Figure 1.3 shows the basic division of driers according to air convection (active, passive) and collector situation (direct, indirect and mixed).

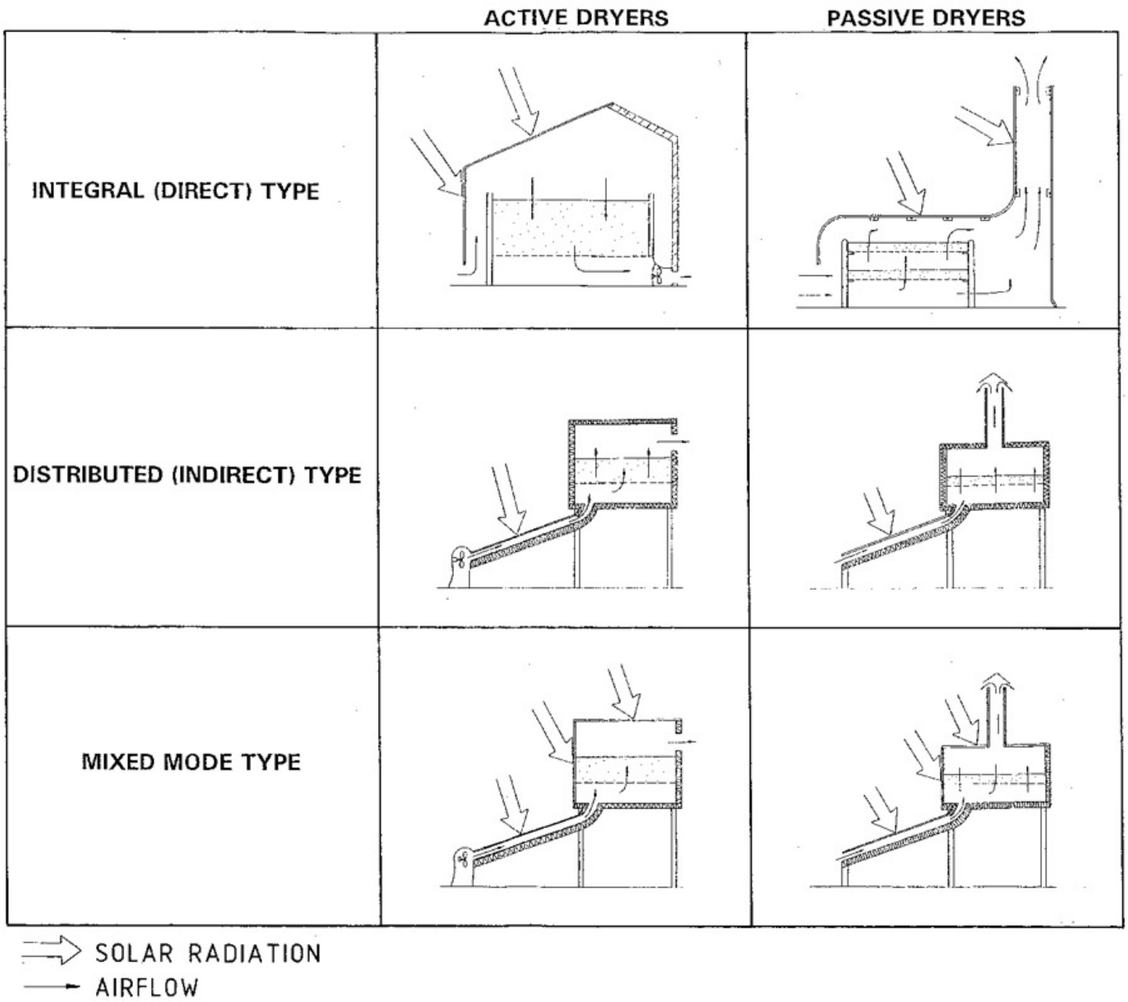


Fig.1.3. Typical solar energy dryer designs.

Source: Ekechukwu and Norton, 1999

The natural convection operation principle is based on the temperature difference, and consequently the difference in the density of the air inside and outside the drying chamber. This difference provides the necessary driving force for the air to flow through the drying chamber (Amir et al., 1991).

Active dryers are furnished by fans providing better and more reliable air convection. The fans are usually powered by electric or oil motors.

1.6.5 Key elements of solar dryers

The solar collector plays the part of primary energy source for a solar dryer. Essentially it has functions of energy conversion and energy transfer (Mujumdar, 1987).

As energy converter the collector converts the direct and diffuse radiation coming from the sun into heat. This energy transformation takes place in the so-called absorber of the collector (Fig. 4.). The radiation absorbed causes the inner energy of the absorber to grow and its temperature to rise. Than the radiation energy transformed into heat in the absorber is transferred to the working medium of the collector (Mujumdar, 1987).

The working medium is, with direct system solar dryers, the drying air itself; with indirect systems this is an appropriately chosen liquid (distilled water, oil or non-aqueous liquids) (Mujumdar, 1987).

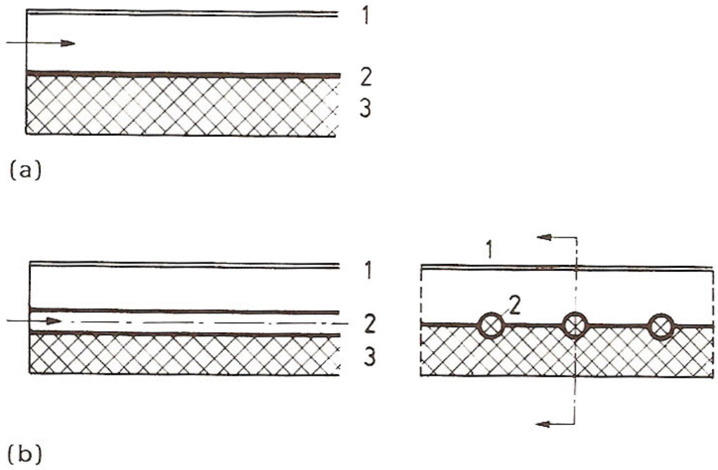
The simplest type of flat plate collector consists of an air duct, the uppermost surface of which acts as the absorber plate. This type of collector is widely used in crop drying operations and corrugated sheet roofs are frequently converted to collectors in this manner. Where a single cover flat plate collector cannot provide the required air temperature rise, further covers may be added to decrease heat losses. Due to the higher temperatures in double and triple-covered collectors more insulation is required than for single cover to minimize heat loss to the surroundings (Brenndorfer et al., 1985).

The choice of which of the types of single cover collector to use is made on the basis that as the temperature difference between ambient and heated air increases the need to insulate the air duct becomes greater (Brenndorfer et al., 1985).

Since the temperature of the covering is considerably lower than that of the absorber, the coating will also reduce the convective heat loss from the structure to the ambient air (Mujumdar, 1987).

Each square meter of solar panel can dry varying amounts of produce depending on the initial and final moisture contents. A solar dryer for seeds will handle approximately 12kg per day (Hollick, 1999).

Figure 1.4 shows the basic structure of solar collectors and the essential difference between the collector using air as the working medium and the other one using liquid as the working medium.



(1) covering, (2) absorber, (3) heat insulation; (a) air, (b) liquid as working medium

Fig.1.4. Setup of flat-plate collectors

Source: Mujumdar, 1987

Drying box is connected to a solar air heater, operating at low and medium temperature, respectively. The material holding capacity of each box depends mainly on the bulk density of the crop to be dried (Sharma el al., 1993).

As seen in the following chapter in certain designs do not require presence of separate collector and it is 'integrated' into the drying box.

1.6.6 Comparing solar and artificial drying

Tiris et al. (1996) have proposed a small scale solar dryer and have made an experimental comparison between natural sun and artificial drying. For this last, the air was heated using an electrical heater then a solar collector. It was found that artificial drying is more successful than natural sun drying. It was shown by graphs that artificial drying reduced the drying time significantly. In addition, it provided better product quality. Karathanos and Belessiotis (1997) presented a large scale solar dryer; 5000 kg capacity. For its artificial drying system, a burner operating with propane gas was used. Their comparison between sun and artificial drying has shown a considerable reduction in drying time for the last one. The disadvantage of this kind of dryers is the important quantity of energy required. In particular, in industrialized countries, between 7% and 15% of the industrial energy is used in drying (Bennamoun and Belhamri, 2003).

The efficiency of the system can be improved further

- by enhancing the absorber surface by way of introducing fins, matrix materials, etc.,
- by optimizing the air duct size,
- minimizing air leakage, and
- using the concept of two-pass flow, etc. (Sharma et al., 1993).

For further reduction in initial investment, it is worthwhile to use the concept of partial glazing. To have minimum payback period, the design of the system should be modified for multi-purpose use (Sharma et al., 1993).

Mujumdar (1987) suggests another possibility of using solar collector practically throughout the whole year, which is coupling a solar energy dryer to a farm's energy system, for example to produce hot water when the dryer is not in.

Sun drying has many problems - it is difficult to maintain consistent quality, losses are high from contamination, insects and animals, it requires large land areas and it takes a long time to dry. For these reasons, many commercial drying operations have switched to other types of drying and are able to justify the investment and high energy costs. These same operations would be ideal candidates for fuel switching from oil to solar. The best candidates for considering solar drying are those who dry all year long as they have longer utilization of the equipment and thus have a quicker payback time to recover their investment (Hollick, 1999).

Fellows (2000) considers the major disadvantages of solar drying in poor control over drying conditions, lower product quality and greater variability and lower drying rates as well. In addition, drying is dependent on the weather and the time of day.

This problem gave rise to the idea of storing part of the energy gained during radiation periods. This difficulty can be eliminated – aside from employing heat storage devices, only with the use of auxiliary energy source. Even radiation periods may produce certain difficulties. First, the intensity of incident radiation is a function of time. This is a circumstance that demands adequate control strategy and the means necessary for such control (Mujumdar, 1987).

Dryers need low grade heat to heat large volumes of air. Low cost solar collectors can provide solar heat for large volumes of air. Solar panels can be added to the roof or walls of buildings housing existing dryers and the panels would either heat or preheat the air entering the fan and dryer (Hollick, 1999).

There is also economic and environmental aspect of electric dryers mentioned in the literature. Drying is an energy intensive operation of some industrial significance. In most industrialized countries, the energy used in drying accounts for 7–15% of the nation's industrial energy, often with relatively low thermal efficiencies ranging from 25% to 50% (Akpinar et al., 2006). In contrary Singh et al. (2008) published that drying accounts for 10–25% of the total energy used in the manufacturing process worldwide.

2. AIM OF WORK

The main aim of this experiment is to compare the two types of drying of *Persicaria odorata* and to evaluate the impact of drying on the final product. The dried samples were compared to each other and to fresh samples through the analysis of the essential oils. The experiment was run in double-pass solar-energy dryer and electric dryer. Aerial parts of *P. odorata* were hydrodistilled and analysed in the laboratories of CULS. The other aim is to measure the drying characteristics of the solar dryer and to evaluate its suitability for this product.

3. MATERIALS AND METHODS

3.1 Site description

Thua Thien Hue Province is located in Central Vietnam, bordered on the east by the South China Sea and on the west by Lao PDR (Fig. 3.1). The province has an area of 5,053km^2 and is divided into nine administrative districts. The population is over one million; 300,000 people reside in or around the capital city, Hue.

Much of the province's infrastructure and industry lies in the coastal plain. The North-South Highway and the railway linking Hanoi to Ho Chi Minh City pass through Hue and effectively bisect the province. Roads extend to the district centres, including A Luoi, the relatively isolated westernmost district. Rivers in A Luoi District drain to the Mekong River, instead of directly to the sea, as in the rest of the province.

Most of the population lives on the coastal plain, within 25km of the coast. Much of the interior of the province has a population density of less than 50 people per sq. km (MPA, 2007).

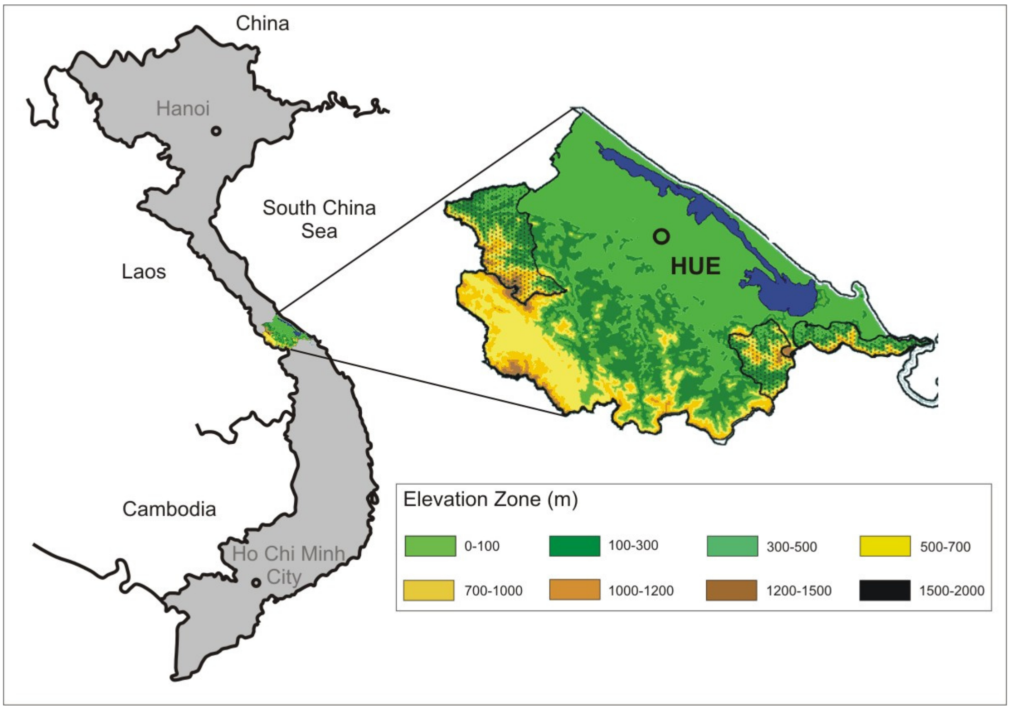


Fig.3.1. Thua Thien-Hue province situated in central Vietnam.

Source: MPA, 2007.

A large area in the West of Quang Tri and Thua Thien-Hue is mountains and hills. The mountains last along the west side of these provinces and have the elevation varying from 200-800m in Quang Th province, to 1000-1500m in Thua Thien-Hue province. Besides the mountains there is a large area of midlands having the elevation of 20-200m. The midlands and mountains occupy 50% of the total area of Quang Tri province and 70 % of one of Thua Thien-Hue province. Coastal plain is narrow lasting along the coast and occupying about 8% of total area of both these two provinces. The sandy dunes along the coast have an area of over 1,000km^2. Lagoons polders are mainly located in Thua Thien-Hue (WB, 1999).

Climate of the project area is tropical monsoon. There are two different seasons: the dry and rainy. However, because of topography the climate regime in the Section SI is relatively complicated. The Hai Van range as a natural frontier divides the area into two different climate zones. The section from Dong Ha to Hai Van Pass is influenced by North tropical monsoon, the other one from Hai Van Pass to Quang Ngai is by South tropical monsoon. There are two distinct periods for the rainy season: In the section from Dong Ha to the Hai Van Pass the rainy season lasts from October or November to March. In the section from Hai Van to Quang Ngai the rainy season lasts from the middle of September to the end of December.

Annual rainfall in this region is about 2,000-4,000mm, and differently varies in term of location. In coastal plains precipitation normally varies from 2,000mm to 2,400mm, but it is very high in mountains, where precipitation may occasionally reach 5,500mm per year. Over 85% of rainfall is concentrated in the rainy season. In the Southern part annual rainfall is much lower, which is only 1,500-1,800mm. Duration of the rainy season is only 3-4 months.

The typhoon season in this area occurs from July to November. Storms are usually accompanied by heavy rains, with up to 600mm rainfall over a 24 hour period in the coastal areas, sometimes creating heavy flood.

Due to the specific topography and climate the project area has abundant biological resources which comprise from two main ecosystems, including uplands and lagoons ecosystems. In the past, forests and forestry land in the provinces occupy more than 60% of the total area. Due to locating in a high rainfall, high humidity and high radiation forests in the central region contain tremendous diversity (WB, 1999).

3.2 Plant material

The aerial parts (stems and leaves) of *Persicaria odorata* were used for experimental tests in this study. Over 1kg of fresh herb (Fig. 3.2) was purchased every morning at a local market in Hue city (Annex A). The samples were obtained from the same seller every time to ensure the homogeneity. Plants were than washed with water to separate eventual soil residues or other foreign particles. The samples were identified by the plant taxonomists from the Hue University of Agriculture and Forestry.

Fig.3.2. Rau ram on the tray ready to be put into the dryer.
Source: Ptáček, 2008.

3.3 Dryers

During our experiment we have used a specialised double-pass solar-energy dryer (Fig. 3.3) designed by Ing. Jan Banout, PhD. and Ing. Petr Ehl, University of Life Sciences Prague.

The working principle of the dryer is closely linked to its "2 floor" construction (Annex B, C, D). Although from the outside it appears as one box, it is divided lengthwise to the collector and drying chamber. The upper (collector part) starting with air inlets and is made of black painted aluminium sheet equipped with eight (5cm high) straps in the direction of the passing air. The terminal 25cm of the collector were replaced by the air pass to the drying chamber. The surface of the collector (2x5m) was increased by the straps and decreased by the missing part replaced by air pass to 13,3m^2 and can be computed as:

$$S = w_D \cdot l_D + 16 \cdot h_S \cdot l_D - (16 \cdot h_S \cdot l_{AP} + w_D \cdot l_{AP})$$

Where:
S = collector surface
w_D = dryer width
l_D = dryer length
h_S = height of the strap
l_{AP} = air pass length.

The air inlets and outlets were protected by metal netting to prevent the insects or another unwanted particles from getting into dryer. This is especially important during drying of fruits or other attractive products.

The dimensions of the dryer are 5m in length and 2m in width being divided into five segments (2x1m). The transparent polycarbonate sheets' dimensions (2.1x1m) caused convex shape of the roof which ensured better water removal during rain. Each segment is equipped with two trays (each sized 1x1m) with high density polyethylene netting. The net hole dimensions are 0.5 x 0.5cm. Each tray was operated through own independent door.

The case of the dryer was made of four layers to ensure sufficient insulation and durability. The outer shell was made of 0,8mm galvanized metallic sheet followed by layer of 15mm polystyrene and 2mm cork layer. The inner shell was made of 0.5mm aluminium plate. The roof of the dryer was made of 8mm transparent polycarbonate panels coated with UV layer on the outer surface. The light transmissivity was 89%.

Being equipped with five 12V ventilators (Sunon KD1212PMBX-6A) the dryer is active type using overpressure as the ventilators are emplaced at the air inlet. The ventilators (120mm in diameter) are powered by SOLARTEC STR 36 - 55 / 12 photovoltaic panel (17.6V, 3A, 50W) consisting of 36 crystallic Si cells. The 17.6V optimal voltage is reduced by resistors (8.3Ω, 3W). For more details see the electric circuit scheme in Annex E. After passing the collector and drying part the air leaves the dryer through two rectangular air outlets (28x10cm).

For reference drying of Rau ram and for final moisture content determination the electric plant dryer (Ketonc 4000W) (Annex H) was used. The reference drying was done in the laboratory of NGO Tropenbos in Hue.

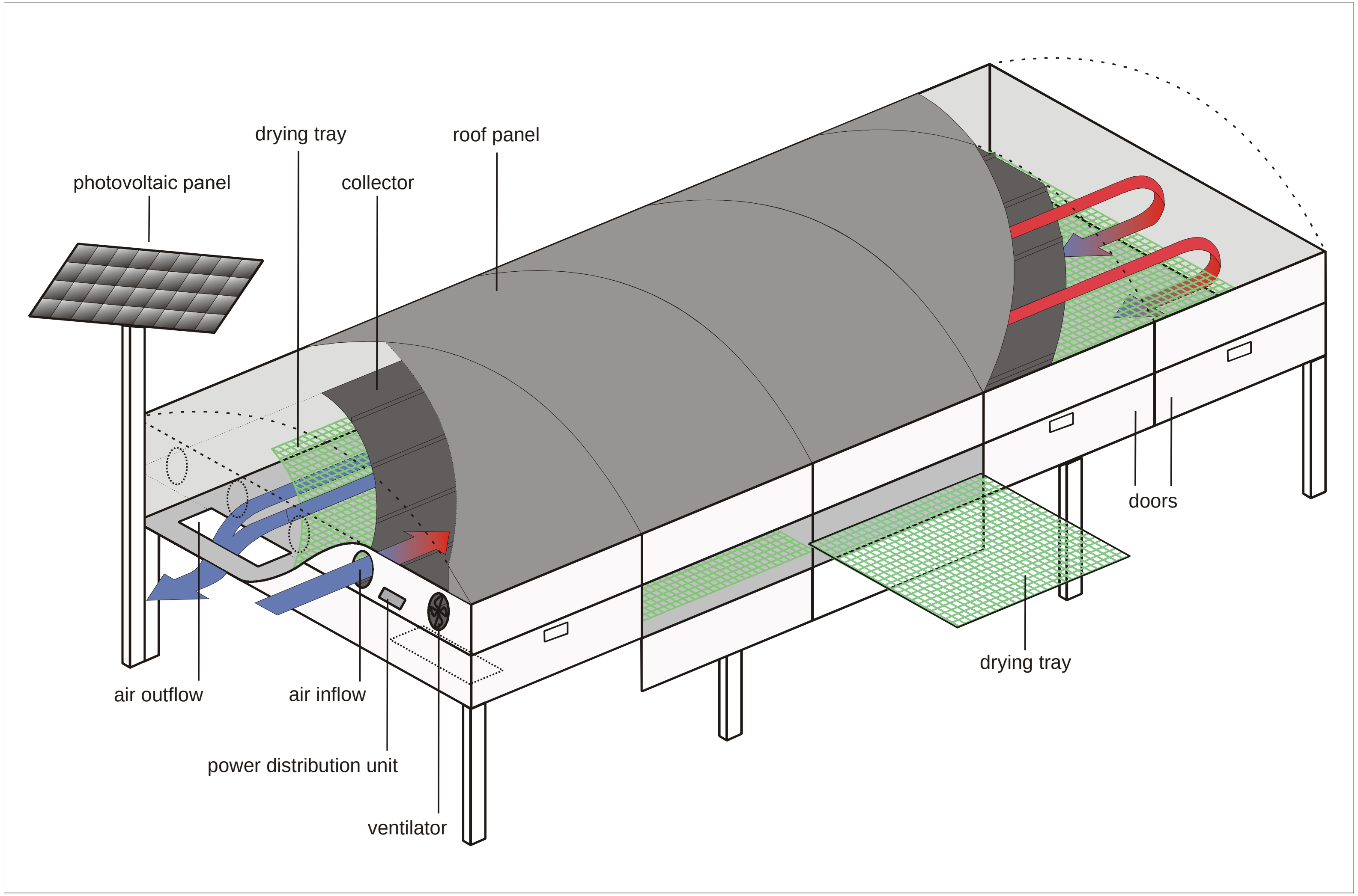


Fig.3.3. Scheme of double-pass solar-energy dryer.

Source: Ptáček, 2008.

3.4 Instrumentation and facilities

3.4.1 Hydrodistillation apparatus

During the experimental a special type stainless steel steam-proof pot with the volume of 5l was employed (Annex H). This vessel was combined with Clevenger apparatus trapping the condensed vapour while the aqueous phase and the essential oil formed into separated layers. Condensation of vapour was assured by water cooler (Fig. 3.4).

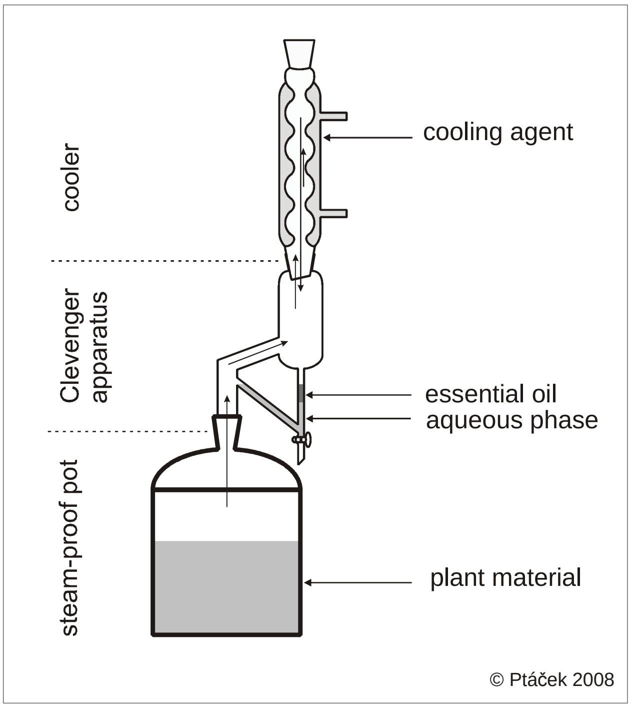


Fig.3.4. Scheme of modified hydrodistillation apparatus
Source: Ptáček, 2008.

3.4.2 Instrumentation

For measuring temperature and relative humidity in the drying chamber two Comet data loggers with S3121 sensors (Annex F) were employed. Temperature in the collector part was measured by two laboratory mercurial thermometers. The airflow inside the dryer and at the outflow was measured by thermic anemometer Testo 425.

Ambient conditions were observed with Kipp Zonen CMP 6 pyranometer measuring solar radiation supported by Kipp Zonen Solrad radiation integrator (Fig. 3.5). Ambient temperature and relative humidity was measured by Testo 605-H1 thermo-hygrometer (Fig. 3.6).

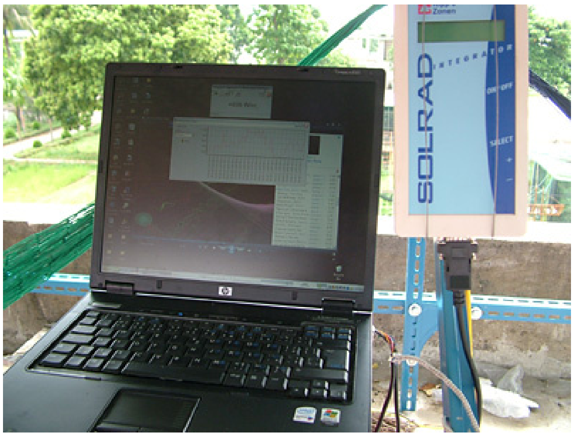


Fig.3.5. Data collection from pyranometer.

Source: Ptáček, 2008.

Fig.3.6. Measuring the air-flow in the drying chamber.

Source: Ptáček, 2008.

3.5 Experimental procedure

3.5.1 Drying procedure

One kilogram of fresh plant was equally spread on the tray and put into the dryer at 8:00am, when the drying process started. The drying period was set to 8 hours/day finishing at 4:00pm, which means one batch could be easily dried during one day in the case of sunny weather.

Drying in the electric dryer was processed under standard conditions, which means 40°C for 24 hours. Again 1kg of green matter was bought fresh at the local market, determined botanically, transported directly to the preheated dryer and left under constant conditions.

To have the most complex view of the drying conditions in the solar dryer, thermal and humidity conditions as well as air circulation inside the dryer were under close focus. Every hour temperature was measured at the initial and terminal part of the thermal collector, same interval was set for air circulation measuring. This was carried out at the central part of the thermal collector, at the central part of the drying chamber and at the air outflow. Detailed information was obtained for the drying chamber using five-minute interval for temperature and humidity data collection, which was measured at the initial part and at the terminal part of the drying chamber.

Outdoor conditions are playing crucial role in the performance of the solar dryer. Hence it is very important to know the relation between the weather conditions and the drying process. The ambient conditions were characterized by temperature, humidity and solar radiation, first two being measured hourly while solar radiation in five minute intervals.

Passing through the collector part the air is heated and the humidity decreases while in the drying chamber the process is reverse.

To every 1kg of the fresh herb a 50g control sample was add (Annex F). This reference sample after being dried either in solar or electric dryer had to undergo final moisture content determination. During this process the artificial dryer was set to 105°C for 24 hours. These samples were weighted before and after the drying process and the final moisture content was calculated.

Reference samples put into solar dryer were weighted every hour to find out the weight loss time behaviour.

3.5.2 Distillation procedure

Twelve different samples had to be run through the distillation process. Four in fresh condition, four dried in solar dryer and four dried in solar dryer. Regardless of being fresh or dried the plants were blended with a kitchen blender first, than put into the steam-proof pot, mixed with 3 litres of water and boiled for three hours. The growing of the essential oil was not linear, but had descending animus and the most of the essential oil was trapped in the first hour.

After the distillation process the essential oil was moved into the 1,5ml vial and after measuring the volume it was refrigerated (Fig. 3.7).

To prevent the essential oils from chemical changes or other undesirable deterioration the vials with samples were kept refrigerated from the time they were gathered in Hue, Vietnam until they were analyzed in Prague, Czech Republic. During the transport the optimal conditions were provided by an insulation box.

Once the samples were brought to CULS, they were inspected in laboratories of Faculty of Agrobiology, Food and Natural Resources. Primarily the samples were mixed with anhydrous sodium sulphate which creating crystal structures helped to remove the traces of water. The samples were diluted to 0.5% and 0.1% in hexane for GC-FID and GC-MS analysis respectively.

Fig.3.7. Vials with essential oil samples.

Source: Ptáček, 2008.

Fig.3.8. EO dilution.

Source: Ptáček, 2008.

3.5.3 Essential oil analysis

GC-FID analyses of essential oil samples were processed in laboratories of Faculty of Agrobiology, Food and Natural Resources, The Czech University of Life Sciences Prague. GC-MS analysis was held in laboratories of Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic.

The identification of individual components was based on comparison of their GC retention indices (RI) and comparison of their mass spectra by GC/MS to those described in the literature (Adams, 1995).

The flame ionization detector (FID) is a non-selective detector used in conjunction with gas chromatography. Because it is non-selective, there is a potential for many non-target compounds present in samples to interfere with this analysis and for poor resolution especially in complex samples. The FID works by directing the gas phase output from the column into a hydrogen flame. A voltage of 100-200V is applied between the flame and an electrode located away from the flame. The increased current due to electrons emitted by burning carbon particles is then measured. Except for a very few organic compounds (e.g. carbon monoxide, etc.) the FID detects all carbon containing compounds. The detector also has an extremely wide linear dynamic range that extends over, at least five orders of magnitude with a response index between 0.98-1.02 (L4S, 2005).

The oils were analyzed by GC using Agilent 6890 system. HP-5 column (30m x 0.32mm, film thickness 0.25µm) was used with nitrogen as carrier gas (1ml/min). The oven temperature was programmed from 60°C to 240°C at a rate of 3°C/min. The samples were diluted to 0.5% in hexane and injected in splitless mode, injection volume being 1 µl. The injector temperature was set at 250 °C. The percentage compositions were obtained from electronic integration measurements using flame ionization detection (FID), also set at 250°C. *n*-alkanes (C10 - C25) were used as reference points in the calculation of linear retention indices (LRI).

The Gas Chromatography-Mass Spectrometry (GC-MS) instrument separates chemical mixtures (the GC component) and identifies the components at a molecular level (the MS component). The GC works on the principle that a mixture will separate into individual substances when heated. The heated gases are carried through a column with an inert gas (such as helium). As the separated substances emerge from the column opening, they flow into the MS. Mass spectrometry identifies compounds by the mass of the analyte molecule. A “library” of known mass spectra, covering several thousand compounds, is stored on a computer. Mass spectrometry is considered the only definitive analytical detector (CPEO, 2002).

For identification of particular essential oil compounds, GC-MS analysis was carried out on a Finnigan Focus GC (Thermo Fischer Scientific, US) equipped with a J&W DB-5 fused silica column (30m × 0.25mm, film thickness 0.25µm, Agilent Inc., US) and interfaced with a quadrupole mass detector Fisons MD 800 (Fisons Instruments, now Thermo Fischer Scientific, US). The temperature program was the same as for GC-FID. Injector and transfer line temperatures were 220°C and 200°C, respectively; carrier gas He, constant flow 1ml/min; ionization energy 70eV; scan time 1s; mass range 30–600amu. The samples were diluted to 0.1% in hexane and injected in splitless mode, injection volume being 1µl.

The component identification was based on comparison of their retention indices indices (RI) (obtained using series of *n*-alkanes (C10–C25), retention times (RT) and mass spectra with those obtained from authentic samples (Sigma, US) and/or the NIST/NBS, Wiley libraries and the literature (Adams, 1995). Some minor peaks with a relative area below 1% were considered as unknown.

Effect of drying method on essential oil constituents was statistically evaluated using Statistica software Version 7.0 (Statsoft CR, CZ). The percentage composition of essential oils was used to determine the relationship among different drying methods using a hierarchical cluster analysis. Only those volatile constituents with relative peak area percentage higher than 1% were included in the cluster analysis. The cluster analysis was conducted on the basis of single linkage and Euclidean distance between the means of each treatment Analysis of variance and Fisher's LSD test were used to determine the difference between the groups.

4. RESULTS AND DISCUSSION

4.1 Solar dryer performance

4.1.1 Ambient conditions during drying

During the test drying runs there was typical humid tropic climate. The drying experimental was held in Hue in the period of August 14th to August 19th 2007. the typical weather was characterized by temperatures exceeding 30°C and high relative humidity. The mentioned time period was at the end of dry season and at the beginning of rainy season, though showers occurred during some of the drying runs. That gives the opportunity to compare the dryer's efficiency during the ideal conditions and during those disturbed by rain.

Tab.4.1. Summary of ambient conditions during 4 drying tests.

	Solar Radiation (W/m^2)			Temperature (°C)			Relative Humidity (%)		
	Min	Max	Mean	Min	Max	Mean	Min	Max	Mean
Drying I	302	842	576	32.0	38.9	36.2	40.0	63.5	49.3
Drying II	78	716	445	30.4	36.5	34.0	46.8	81.5	60.8
Drying III	82	1106	513	29.7	36.0	32.2	54.9	80.0	68.8
Drying IV	83	999	704	29.5	36.7	33.9	50.2	77.7	60.3

I-IV, drying run

The drying tests were taken during four days under various conditions (Tab. 4.1). The ambient temperature showed quite consistent values ranging 32.2-36.2°C in the average. During drying II and III the optimal drying conditions were disturbed by rain resulting in higher final moisture content (Tab. 4.2) of the product. The highest average relative humidity (68.8%) was observed during drying III when good drying conditions (1122W/m^2, 34.5°C, and 56.1% at 11:00) were followed by rain in the afternoon. Drying I resulted in final product with lowest final moisture content, although there was lower average solar radiation value observed (576 and 704W/m^2 for drying I and drying II respectively). Drying I gave best results because there was highest average ambient temperature (36.2°C) recorded and lowest average ambient relative humidity (49.3%). This is also connected with the fact that no rain occurred during drying I.

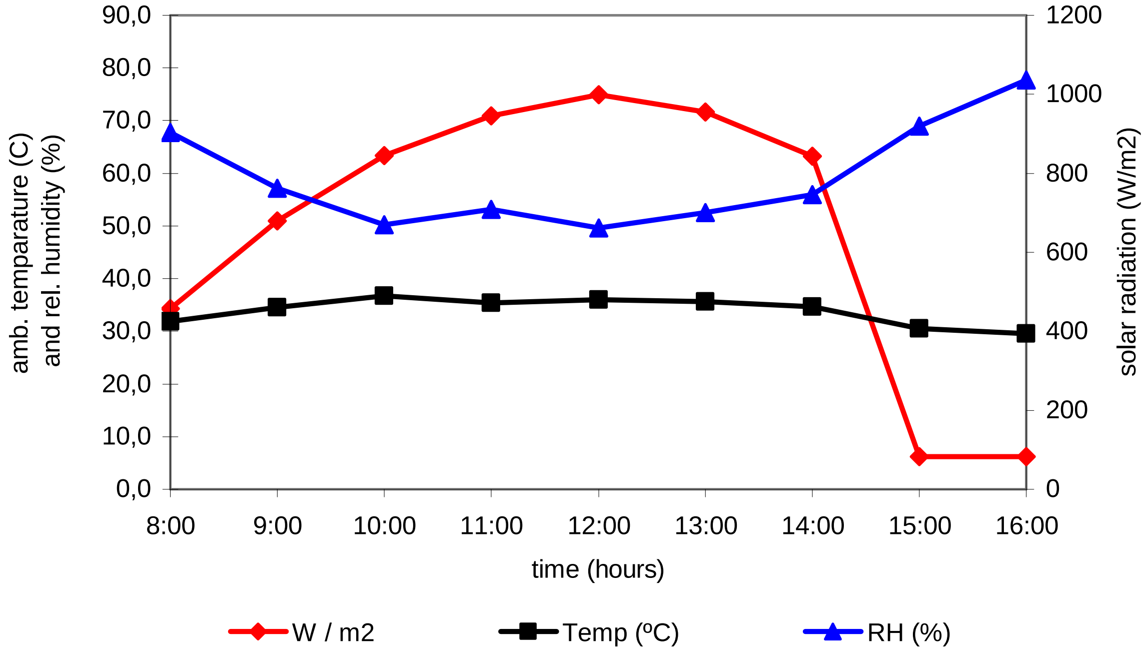


Fig.4.1. Example of ambient air conditions during drying experimental.

As obvious from figure 4.1 the temperature of the ambient air was in range 29.5 to 36.7°C with the maximum temperature oscillating between 10:00 and 12:00. Remarkable symmetry was observed during each drying run between the ambient air temperature and relative humidity. As seen in figure 4.1 relative humidity is decreasing as the values of temperature increase. In most of our measuring relative humidity had its minimum at the same point, where maximal temperature and solar radiation were recorded. The break point of solar radiation was observed at noon.

During the test run shown at figure 4.1 the values of solar radiation were 55-999W/m^2 with the maximum at 12:00 and minimum at 14:50, when the ambient conditions were changed by a sudden storm.

Generally solar radiation had typical parabola progress. The values during all our tests ranged 55-1122W/m^2. Values exceeding 800W/m^2 were observed from 10:00 to 14:00 in the average.

Drying efficiency i.e. during storms may be enhanced by using additional heating source. Bennamoun and Belhamri (2003) mention that adding a heater allows drying the product in unfavourable conditions, such as between 5 a.m. and 8 a.m. Using a heater has shown good improvement in the obtained results. It allows reaching in many studied cases the purposed moisture. Its use can present a rapid investment return.

Ekechukwu and Norton (1998) studied the variations in solar dryer efficiency during dry and rainy season, respectively. The performance of the integral type natural-circulation solar-energy dryer was found to be largely affected by seasonal weather variations. Drying conditions during the dry season were fairly constant. Variable cloud overcast, frequent rain and high relative humidity affected adversely the drying conditions during the wet season. The overall performance of the dryer was, thus, better during the dry season, requiring less than 2 days to reduce the moisture content of cassava chip to lower than the desired safe storage level. This was supported by our experiment. Since our dryings were run during the time when dry season was ending and rainy season was beginning, distinct variations between rainy and sunny days were observed. According to actual weather situations the final MC was ranging 11.6 to 23.5% (w.b.)(Tab. 4.2).

4.1.2 Drying conditions

An overview of the average conditions inside the dryer is shown in fig.4.2. Comparing the average airflow in the dryer, relative humidity and temperature in the drying chamber we can see, that temperature ranging 35.9-62.9°C and airflow ranging 0.0-1.35m/s reached their maximum at noon, while the lowest relative humidity (16.3%) was measured at 11:00. A dramatic change of weather can be observed at 14:00. As the temperature decreases, the relative humidity value rises. The temperature in the drying chamber fell from 52.5 to 41.9°C in half an hour. The fall of airflow rate is caused by sudden solar radiation decrease, since the airflow is powered through the photovoltaic panel directly by solar energy.

There is a distinct similarity between the progresses of airflow in figure 4.2 and solar radiation in figure 4.4, which shows very close relation between these two magnitudes and will be mentioned later.

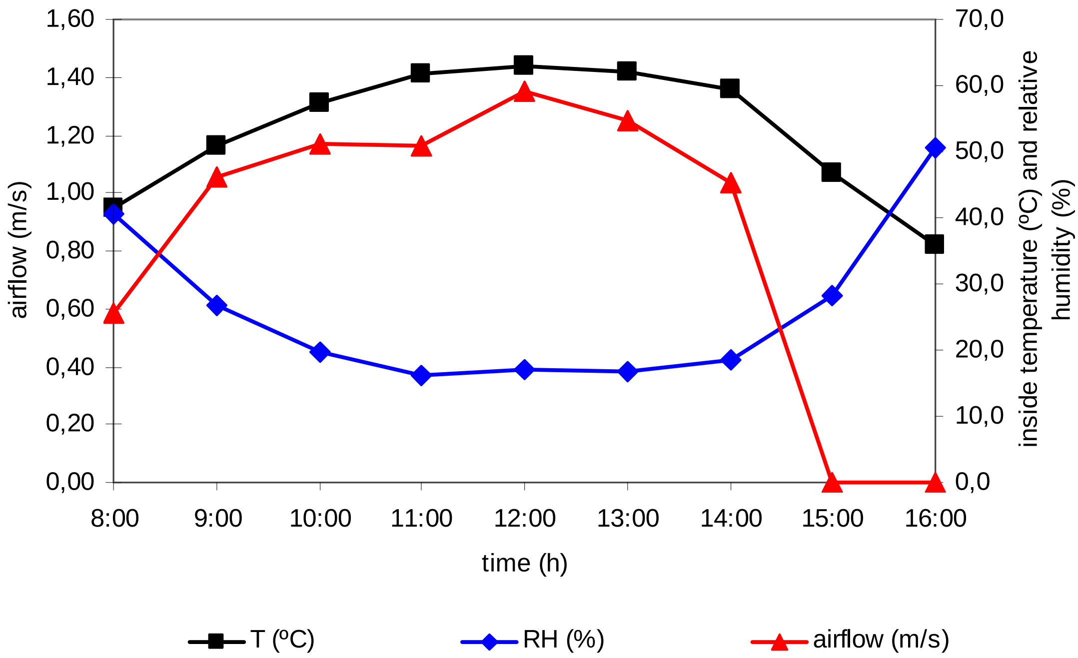


Fig.4.2. Relative humidity and temperature inside the solar dryer compared to airflow.

Detailed information on the temperature and relative humidity conditions inside the dryer is presented at fig.4.3, where initial and terminal part of the collector and initial and terminal part of the drying chamber are compared.

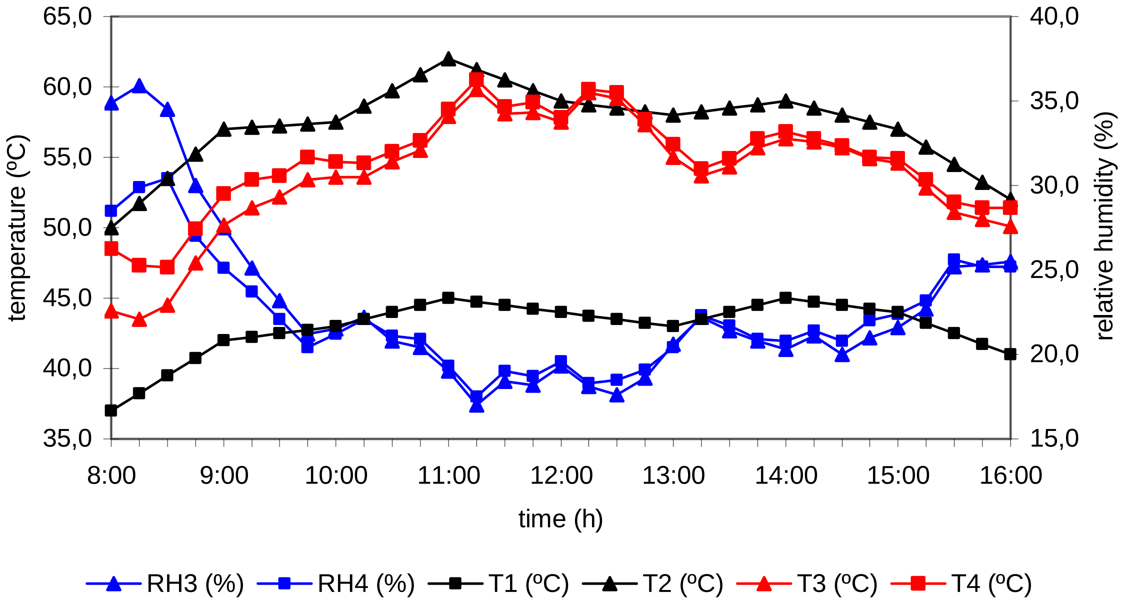
T1 measured in the initial part of the thermal collector was close to ambient temperature and ranged 37.0-45.0°C with the break point at 11:00. T2 measured in the terminal part of the collector was also the highest temperature within the whole solar dryer for the most of time. The range of T2 was 50.0-62.0°C; the top value recorded again at 11:00.

The collector efficiency can be described by the air temperature rise on the way from T1 to T2. The difference between these two values was smallest at 16:00 (11.0°C) and biggest at 11:00 (17.0°C) when the top temperatures were observed in the collector part.

Temperature was measured at the initial (T3) and terminal (T4) part of the drying chamber T3 ranging 44.1-59.8°C and T4 47.2-60.5°C both reaching top values at 11:15. For the most of drying time the difference between these two measuring points was less than 0.75°C, T4 being the higher value as the air is heated on the even way through the drying chamber. The biggest difference between T3 and T4 was at the beginning of drying time and the average difference was 1.0°C.

Relative humidity was measured at the initial (RH3) and terminal (RH4) part of the drying chamber. The ranges for RH3 and RH4 were 17.6-35.9 and 18.3-30.4% respectively. At the beginning of the drying RH3 was remarkably higher than RH4, equilibrium was reached at 10:15, than RH4 was slightly higher than RH3 for the rest of the drying time with the average difference of 0.3°C.

There is still an obvious relation between relative humidity and temperature and we can see that top temperature values were reached at about the same time, when relative humidity was minimal.



T1, temperature at initial part of collector; T2, temperature at terminal part of collector; T3, temperature at initial part of drying chamber; T4, temperature at terminal part of drying chamber; RH3, RH at initial part of drying chamber; RH4, RH at terminal part of drying chamber

Fig.4.3. Temperature and relative humidity within different parts of solar dryer.

Depending on the prevailing meteorological conditions and air flow rate reached, a useful temperature rise of 11-17°C with the average of 12°C can be achieved using double-pass solar-energy dryer. In contrast to our study Sharma et al. (1993) reported 10-27°C temperature rise using large scale indirect type solar dryer consisting of a fan, solar air heaters in two different configurations operating at low and medium temperature, and a drying chamber.

Our results were fairly in correlation with those published in literature. Low temperature is normally recommended for drying most of the agricultural products. The temperature rise of 15–20°C of air from ambient is sufficient to meet to the requirement of crop drying (Jain, 2007).

If the temperature of drying air is raised by only a few degrees, the relative humidity of even humid air are lowered enough to make it effective for crop drying (Tiwari et al., 1994).

Solar radiation is one of the most important factors within solar drying. Figure 4.4 presents the close relation between the solar radiation on one side and relative humidity and temperature in the drying chamber on the other. Temperature rises as the solar radiation values go up while the relative humidity has the opposite tendency. When there is a significant change of weather at 14:15, the solar radiation decrease causes immediate fall of temperature which is more than 10°C in 30 minutes. And the decrease of temperature reflected in rising relative humidity which rose more than 12% in 30 minutes.

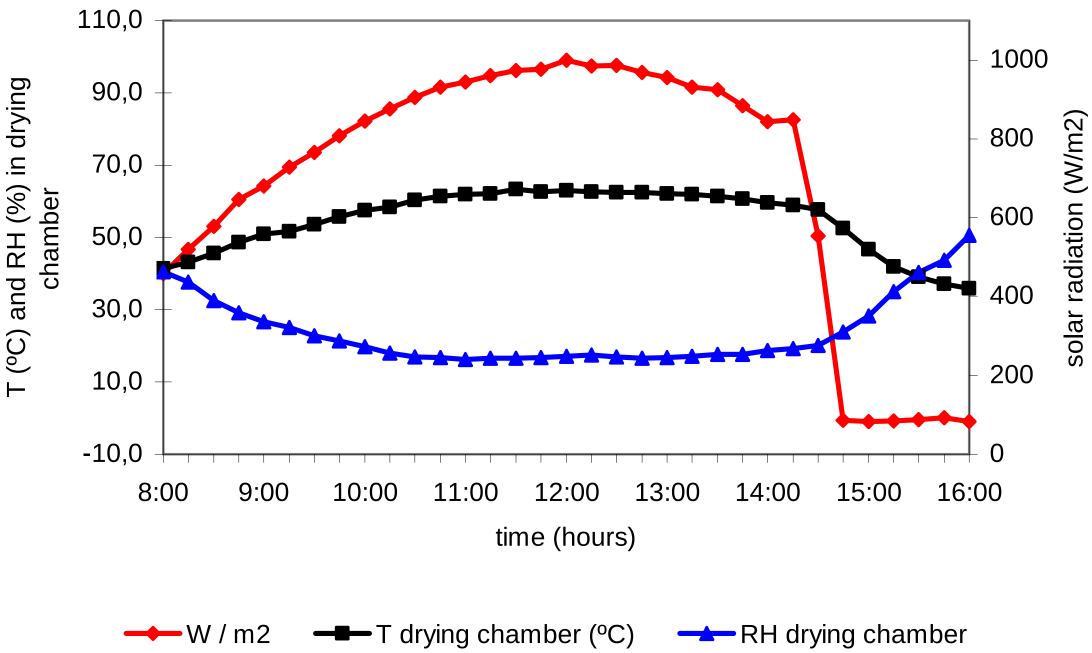


Fig.4.4. Drying chamber conditions in relation to solar radiation

Drying process of any products is mainly affected by air temperature, relative humidity and airflow. In the double-pass solar-energy dryer the airflow was powered by solar energy, so the ventilators' rpm was regulated directly by the amount of solar radiation.

In any passive solar dryer the airflow is mainly affected by solar radiation. In the double-pass solar-energy dryer the situation is the same, although the air is forced by five electric fans and the dryer is active type.

The airflow was measured in the drying chamber and at the outflow. During all the drying tests the average airflow in the drying chamber reached the values 0.2-0.6m/s. Top airflow values were observed from 10:00 to 14:00 as the solar radiation was most intensive.

Figure 4.5 shows that the airflow is in fact function of solar radiation. Figure 4.5 represents drying conditions disturbed by rain at 12:00. As you can see the clouds caused drop of airflow values (0.03m/s in the drying chamber) so the increased ambient relative humidity did not slow down the drying process much; in the average reference samples lost 3g of their weight from 11:00 to 13:00. During the test run shown in figure 4.5 the drying chamber airflow ranged 0.03-0.70m/s and 0.2-1.75m/s at the outflow.

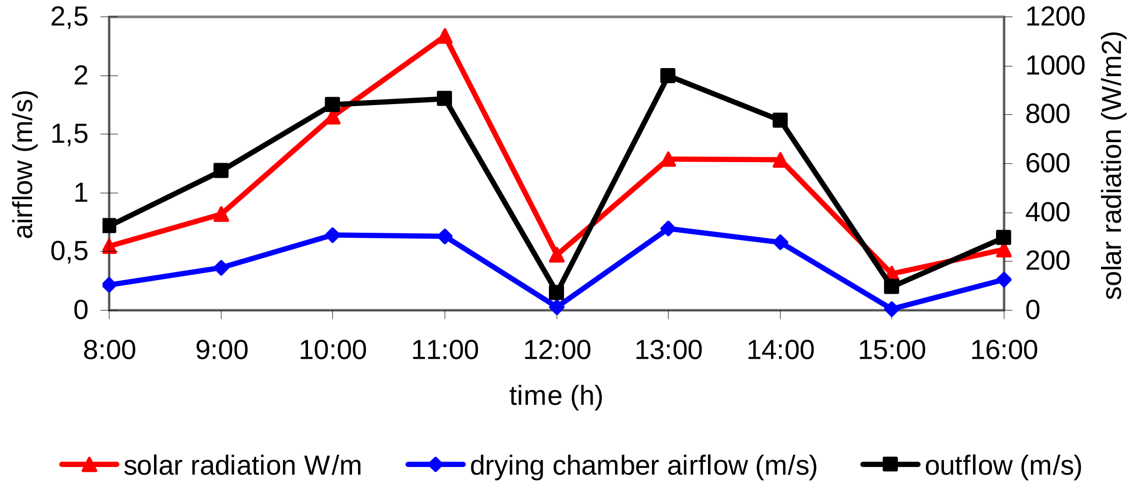


Fig.4.5. Solar radiation influence on the airflow.

During our measuring it was observed that airflow response to solar radiation is immediate as the photovoltaic collector reacted to the actual conditions.

When the solar radiation fell to 120W/m^2, the ventilators were hardly working and some of them stopped. At 80-90W/m^2 all of them stopped working and the airflow had fallen to zero. This may be taken as a positive effect of taking power from photovoltaic panel. If the ventilators were on during a sudden rain, the relative humidity inside dryer would rise much faster.

According to previous study drying efficiency increased when air speed is small and increased slowly when air speed is high. Lower air speed will result in low efficiency but higher air speed may result in an excessive pressure drop inside the collector (Abu-Hamdeh, 2003).

4.1.3 Drying kinetics

During every drying test there were three 50g reference samples added to the initial, central and terminal part of the drying chamber to investigate the difference in the drying conditions for each part of solar dryer.

As presented in figure 4.6 the solar dryer gave satisfactorily homogenous conditions in all its parts and the final weight of the 50g reference samples did not vary much (8.0, 8.5 and 8.6g for initial, central and terminal part of the dryer). The most significant differences were observed at the beginning of each drying run at the interval of 8:00 and 11:00 when the weight loss was much faster in the initial part of the drying chamber. After the first hour of drying the weights decreased from 50g to 21.2, 28.0 and 30.3g for initial, central and terminal part of drying chamber respectively. As the relative humidity in drying chamber decreased (falling from 40 to 16% in the mentioned interval) the differences became smaller. At 12:00 the difference was within 1g.

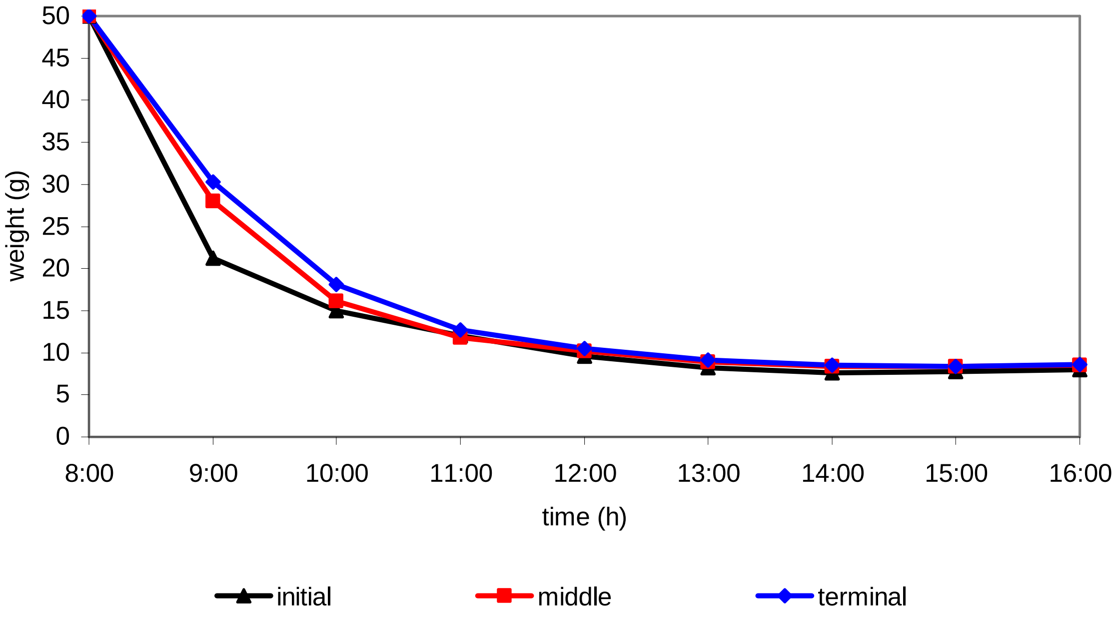


Fig.4.6. Weight losses during drying in different parts of drying chamber.

The study of Braga et al. (2004) confirms that high temperatures and low loads increased the drying kinetics. Therefore, the smaller the material load, the smaller is the amount of energy necessary for the process.

This is associated to the fact that more moisture content is removed when one works with smaller loads.

From a recent study on parsley it is apparent that drying rate decreases continuously with moisture content or drying time. However, the experimental results for forced convection drying showed that the drying air temperature has a significant effect on the evolution of the moisture content (Akpinar et al., 2006).

The average initial moisture content was 85.48% with the values ranging 84.4-88.2% (w.b.). Initial moisture was computed according to following formula:

$$M_i = \frac{W_i - W_{ad}}{W_{ad}} \cdot 100$$

The final moisture content of the plants was determined by the mass loss during the drying interval and was calculated in accordance to following formula:

$$M_f = 100 - \frac{W_{ad}}{W_d} \cdot 100$$

Where:

M_i = Initial moisture content (%)

M_f = Final moisture content (%)

W_i = Initial crop weight (g)

W_d = Dry crop weight (g)

W_{ad} = Absolute dry crop weight (%).

In comparison with the electric dryer the final moisture content of samples from solar dryer was approximately 1% lower (Tab. 4.2).

Tab.4.2. Initial and final moisture content during solar and electric drying.

	S					E				
	I	II	III	IV	Mean	I	II	III	IV	Mean
GM weight (g)	50,0	50,0	50,0	50,0	50,00	50,0	50,0	50,0	50,0	50,00
DM weight (g)	8,6	10,2	9,1	8,6	9,13	7,9	7,5	8,4	9,5	8,33
ADM weight (g)	7,6	7,8	7,6	7,5	7,63	7,0	5,9	7,0	7,7	6,90
Initial moisture content (% w.b.)	84,8	84,4	84,8	85	84,75	86,0	88,2	86,0	84,6	86,20
Final moisture content (% w.b.)	11,6	23,5	16,5	12,8	16,11	11,4	21,3	16,7	18,9	17,08

S, solar dryer; E, electric dryer; I-IV, drying run; ADM, absolute dry matter (105°C, 24h); w.b., wet basis

It was observed that the herbs can be dried at about 49°C reaching equilibrium moisture after 12 and 9.5h using the wire basket solar dryer and oven drying method respectively (Balladin and Headley, 1999).

Water content is certainly one of the most critical quality parameters of any herb, as it governs mould growth during storage and transport, which could lead either to the formation of mycotoxins or any unwanted flavours.

Correct drying of aromatic plants is necessary for high quality and stable products and the final moisture content should be 5–10% (w.b.). Drying prevents the growth of micro organisms that may cause spoilage or food poisoning. The essential oil constituents of herbs can be adversely affected during drying (Arabhosseini et al., 2006a). Tiwari et al. (1994) declares that for safe storage, agricultural crops must be dried to a moisture content of 9-13% (w.b.). Under optimal climatic conditions drying in solar dryer resulted in the product with FMC averaging 11.5-13% (Tab. 4.2). When the conditions were unfavourable, final moisture content was higher.

Both over-drying and under-drying are harmful for storage of agricultural products. Over-drying due to excessive exposure to sun or heat damages the seed coat and, thus, causes bleaching, scorching, discolouration, loss of germination power, and reduction in nutritional value. On the other hand, under-drying or slow drying results in deterioration of food quality due to fungal and bacterial action. Thus, drying under controlled conditions of temperature and humidity helps the crop to dry reasonably rapidly to a safe moisture content level and ensure a superior quality of the product. High temperatures could be used only in the beginning of the drying process (Sharma el al., 1993).

Doymaz et al. (2006) after undertaking drying experiments on dill and parsley stated that drying air temperature is effective parameter for the drying of leaves. As the air temperature increased, other drying conditions being same, moisture removal increases thus resulted into substantial decrease in drying time. This was also supported by our results. As the drying experiments were set to constant drying period, the most important factor was solar radiation, which is closely connected with temperature. As obvious from table 4.2 and table 4.1 the lowest moisture content was reached during the days with the highest average ambient temperature.

According to Reh et al. (2006) drying processes are mainly driven by temperature, particle diameter and partial pressure of water on the surface of the particle.

Experimenting with leafy vegetables Negi and Roy (2001) also notice that retention of the quality parameters of leaves was better at faster drying conditions.

4.2 Dried product properties

Since Rau ram is predominantly used fresh, no drying experiments on Rau ram were yet described in the scientific literature. Sensory properties evaluation of dried product was targeted on comparing the two types of drying to each other and to the fresh material.

Concentrating on the colour of the final product the results were satisfying in both dryers. The colour of the final product was celadon with no visible spots or colour abnormalities (Annex G). The only exception was noticed on the initial part of solar dryer's drying chamber. Due to the construction of the solar dryer there was small but noticeable part of drying trays exposed to direct sunlight. In total this area represents 3.5% of drying trays' surface. This was the area where the biggest, but still feasible changes occurred. The leaves were slightly bleached and the colour moved gently to brown.

The smell of Rau ram is dominated by strong citrus odour followed by some spicy odours, potato odour and two tropical fruit odours (Starkenmann et al., 2006).

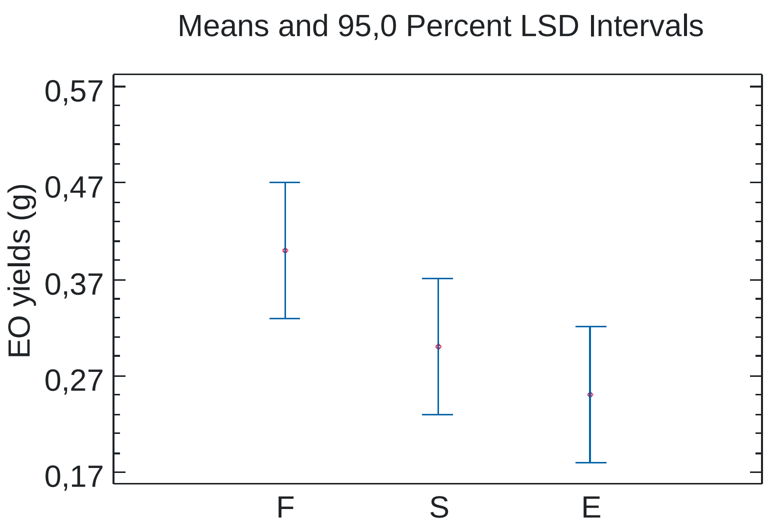
The fresh leaves of Rau ram (Annex G) are highly aromatic, all the dried samples kept the characteristic aroma. Both drying methods seemed to have similar effect on the aroma preservation. Regardless on the type of drying the odour was well apparent after grinding the leaves.

Investigating the drying influence on French Tarragon (*Artemisia dracunculus* L.) Arabhosseini et al. (2006b) investigated that drying of leaves at 45ºC resulted in small changes for colour while changes for the leaves dried at 60 ºC were large during drying. During our test drying temperature in the electric dryer was set to 40ºC and in the solar dryer ranged 47.8-54.6ºC and there was only slight change of colour where direct sunlight reached the leaves as described above.

It was assumed that moisture content also affects colour changes during drying. When moisture content is higher more changes occur. That is the reason for the strong quality changes in the initial phase of drying (Arabhosseini et al., 2006b).

4.3 Essential oil evaluation

Hydrodistillation of *P. odorata* gave light yellowish oil. According to the expectations the essential oil yields from dried samples were lower than those from fresh material. However solar drying had no statistically significant effect ($P<0.05$) on the essential oil yield in the contrary to drying in electric dryer (Fig.4.7).



F, fresh; S, solar dryer; E, electric dryer

Fig.4.7. Statistical evaluation of the effect of solar drying and drying in electric dryer on the EO yield.

As shown in Tab.4.3., the average essential oil yield for each tested drying method was 0.04, 0.03 and 0.025% (w/w) for fresh sample (F), solar dryer (S) and electric dryer (E), respectively.

Detailed information on essential oil yields is listed in Annex I.

Tab.4.3. The effect of solar drying and drying in electric dryer on the EO yield.

	F	S	E
	Mean	Mean	Mean
EO yield (ml)	**0,418**	**0,305**	**0,264**
EO yield (g)	**0,400**	**0,300**	**0,250**
EO content (% w/w)	**0,040**	**0,030**	**0,025**

F, fresh; S, solar dryer; E, electric dryer

There is lack of information about the influence of drying on the chemical composition of Rau ram essential oil in the literature so far. To our knowledge the only other data to be compared were published by Starkenmann et al. (2006). The study was focused on comparison of essential oils of *Persicaria odorata* and *Persicaria hydropiper* distilled from fresh material (Starkenmann et al., 2006).

Generally essential oil yields from our samples were higher than those obtained by Starkenmann et al. (2006). The yields were 0.012 and 0.040% (w/w) for Starkenmann and our experiment respectively.

The amount of the essential oil is influenced by cultivation conditions, climate and genetic factors. Starkenmann et al. (2006) analyzed herbs cultivated in a greenhouse in Switzerland, while our samples were grown in the tropics under natural climatic conditions.

Arabhosseini et al. (2006a) focuses on the temperature and relative humidity influence on French and Russian tarragon essential oil yield. The leaves dried at 45ºC and 90ºC showed higher yields for both French and Russian tarragon compared to the 60ºC treatments. The recovery of oil from Russian tarragon leaves was almost the same at 45 and 90ºC and the levels were higher than at 60ºC. At 60ºC most oil losses occurred before the leaves reached final MC of 35% (w/w) when dried with air of 7% RH and before MC of 25% (w/w) for the leaves dried at 18% RH.

In the case of solar dryer the temperature and humidity conditions are highly dependent on the ambient weather conditions. Fig. 4.8 shows the influence of drying conditions on the essential oil yield. Top yield (0.41ml) was reached when the average drying temperature was highest (54.6ºC) and relative humidity one of the lowest (23.9%).

For an equal duration of drying, product properties change more for higher temperatures and at a constant temperature longer drying time causes more changes (Arabhosseini et al., 2006a).

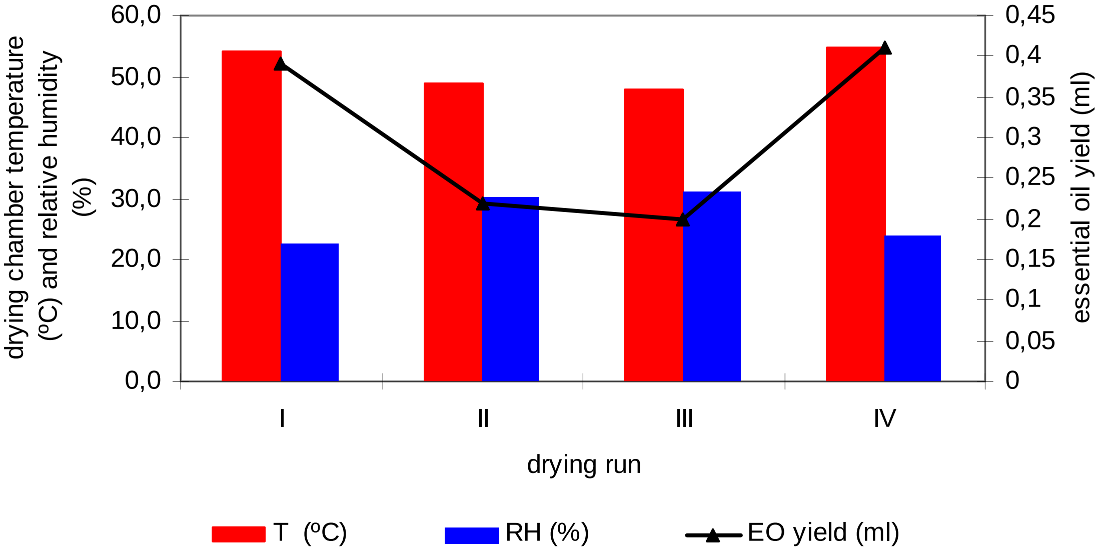


Fig.4.8. Influence of temperature and relative humidity in drying chamber on the essential oil yield.

From the experiment of Arabhosseini et al. (2006a) it is clear, that one of the key factors affecting EO yield and quality is drying time. Since our drying period was set to constant time, temperature and relative humidity are considered to be the main factors for the quality of dried product.

It appears that lower temperatures and shorter drying times yield more oil (Arabhosseini et al., 2006a). We have to add, that during solar drying where the drying period is the same, higher temperature has positive effect on EO yield.

Yiljep et al. (2005) reports very low yields ginger EO from the samples dried by sun, solar and natural air drying. This may be due to prolonged drying period which resulted in poor drying effect on the samples, thus, affecting the yield of EO. Our results support this thesis. The highest EO content in fresh Rau ram samples, the yield was lower in samples from solar dryer (8h drying period) and even lower in the samples from electric dryer (24h drying period).

Ahmed (2005) mentions that distillation process influences the EO yield significantly. In India natural gas supplied through underground pipeline is being used for commercial distillation of essential oils. It has been observed that besides lowering the cost of distillation, the oil recovery is also higher in shorter time.

Investigating the drying effect on long pepper (*Piper hispidinervium* C. DC.) Braga et al. (2004) mentions that the drying results in a higher essential oil yield for temperatures up to 50 ºC. The essential oil yield practically increased twice after drying. For drying air temperatures above 50ºC and drying residence times above 45 minutes, safrole content decreases about 20%.

Individual components were identified by comparison of both mass spectrum and their CG retention times, calculated using a homogenous series of *n*-alkanes, with those of authentic compounds previously analysed and by comparison of mass spectra with those published in literature (Adams, 1995).

In all samples, out of 24 detected, 18 were identified presenting in average 96.15%. Six compounds were though considered unknown or tentatively identified (Tab. 4.4).

The analyses proved notable difference in chemical composition between the two drying methods and fresh leaves. The difference in chemical composition was more remarkable when fresh and dried samples were compared, while the difference between the two drying types was little.

Essential oil of samples grown in Hue is characterized by the main constituents being as follows. The main compound of the essential oil of Rau ram was *n*-dodecanal averaging (48.3–65.5%) followed by decanal (10.6–14.3%), dodecanol (4.1–10.1%), decanol (4.1–10.2), bergamotene (3.0–3.6%), α-humulene (1.7–2.2%) and undecanal (1.5–2.0%).

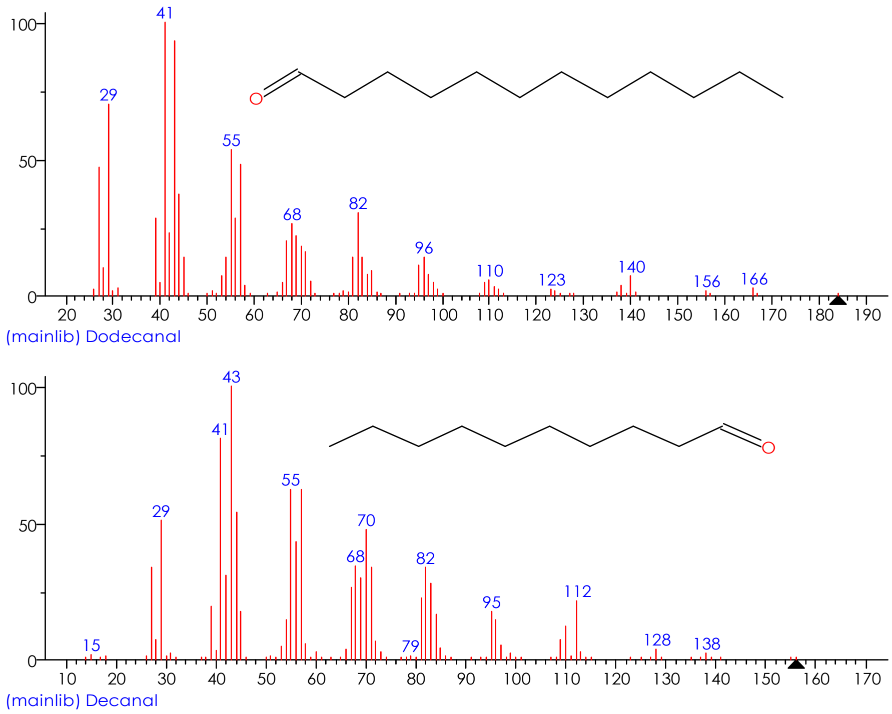


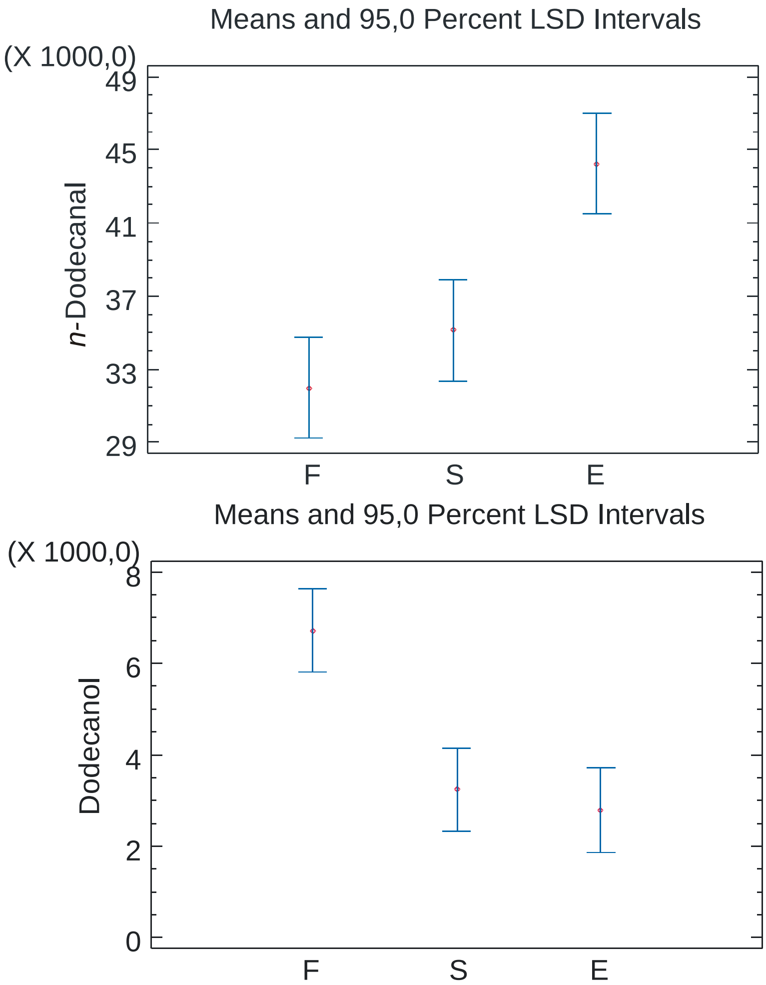
Fig.4.9. Chemical formula and MS spectra of *n*-dodecanal and decanal, the main constituents of the EO of Rau ram.

Out of the named main constituents drying had mild but statistically significant effect (P<0.05) on four components – *n*-dodecanal, dodecanol, decanol, undecanal.

In both cases drying increased *n*-dodecanal content (Fig. 4.10); the effect of solar drying was mild, while the electric dryer caused statistically significant difference (P<0.05). In fresh leaves the content of *n*-dodecanal was in the range 44–51.5%, while after drying in the electric dryer the values were even over 70% of total peak area.

There were no statistically significant changes (P<0.05) in decanal content. Highest variance of decanal content was recorded in leaves from the electric dryer with values between 5.3 and 17.7% in the contrary to fresh leaves and leaves dried in solar dryer, where more consistent: 9.5–12.5% and 12.2–17.0% for fresh and solar dried leaves, respectively.

The amount of dodecanol was decreased apparently by both types of drying. The decrease was statistically significant (P<0.05) in the samples from solar dryer as well as in those from electric dryer. Fresh leaves contained 10.2% of dodecanol compared to 5.3% content after solar drying and 4.1% after drying in the electric dryer (Fig.4.10).



F, fresh; S, solar dryer; E, electric dryer

Fig.4.10. The effect of drying in solar and electric dryer on *n*-dodecanal and dodecanol.

Decanol content was influenced in a similar way as for dodecanol during the drying which means the amount of decanol was decreased to one half in the average by both types of drying as well as dodecanol.

Beramotene and α-humulene content was slightly decreased by both dryers, but there was no statistically significant difference ($P<0.05$).

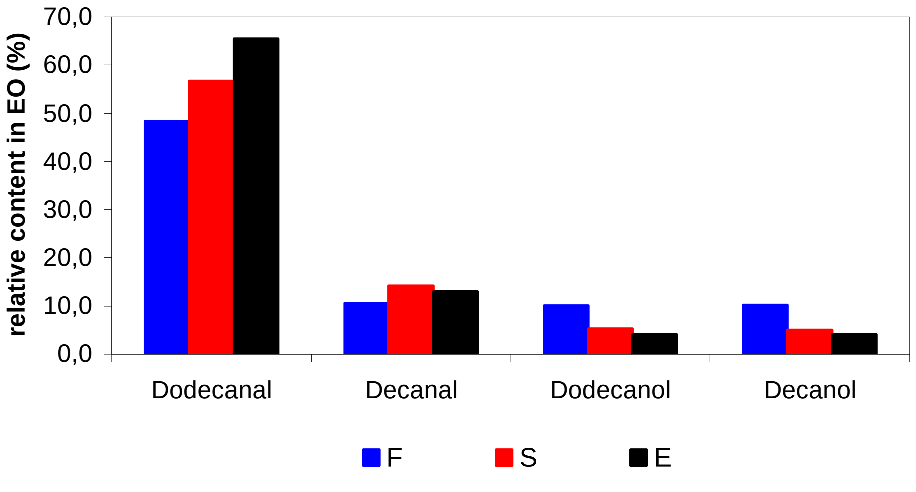
Electric drying had statistically significant effect ($P<0.05$) on undecanal resulting in notable increase of the amount, while during solar drying the increase was mild. Fresh samples show the average content of 1.5%, drying in electric dryer increased the average volume to 2.0%.

Remarkable changes were observed at the initial part of the chromatogram. Constituents unknown 2 and unknown 3 were observed only in the fresh samples while in both dried samples they were not detected. Constituent unknown 1 was again recorded in fresh samples also in two of four samples from electric dryer but in none of samples dried in the solar dryer.

There were also other monoterpenes detected as pulegone and menthone. Pulegone was detected in two of four fresh samples, it was also present in three of four samples from solar dryer, but none of the samples from electric dryer showed the presence of this constituent. For menthone the situation was alike, it was present in one fresh sample, in two samples from solar dryer but there was none in samples from electric dryer. It is an interesting but not surprising fact. Asekun et al. (2007) recorded similar results for pulegone and menthone during experiments with drying effect on *Mentha longifolia*. While in fresh, air-dried and sun-dried leaves the content of these two monoterpenes varied (18.4-35.0% for pulegone and 37.1-47.6% for menthone), in leaves dried in electric dryer there was none and trace amount for pulegone and menthone respectively.

Pulegone is reported to be a potent hepatotoxin, even at low concentrations. It is metabolised in the liver to menthofuran, a highly reactive metabolite which binds irreversibly to the components of liver cells in which metabolism takes place. It quickly destroys the liver (Asekun et al., 2007).

Due to the significant reduction of pulegone and menthone by drying in electric dryer, this type of drying could be recommended in order to reduce toxicity.



F, fresh; S, solar dryer; E, electric dryer

Fig.4.11. Relative content of main constituents in Rau ram essential oil under the influence of different drying types.

Tab.4.4. Constituents identified in Rau ram EO.

No.	Compound	RI	F			S			E		
			Mean	95% confidence interval		Mean	95% confidence interval		Mean	95% confidence interval	
1	**Unknown 1**	**n/a**	**1865,50^{a}**	**1662,83**	**2068,17**	**0,00^{b}**	**-202,67**	**202,67**	**54,75^{b}**	**-147,92**	**257,42**
2	**Unknown 2**	**n/a**	**305,50^{a}**	**249,50**	**361,50**	**0,00^{b}**	**-56,00**	**56,00**	**0,00^{b}**	**-56,00**	**56,00**
3	**Unknown 3**	**n/a**	**555,25^{a}**	**438,56**	**671,95**	**0,00^{b}**	**-116,70**	**116,70**	**0,00^{b}**	**-116,70**	**116,70**
4	Menthone	1155	253,75^{a}	-109,65	617,15	178,75^{a}	-184,65	542,15	0,00^{a}	-363,40	363,40
5	Decanal	1210	6987,25^{a}	4299,50	9675,00	8861,25^{a}	6173,50	11549,00	8736,50^{a}	6048,75	11424,30
6	Pulegone	1240	1111,75^{a}	-576,53	2800,03	1473,25^{a}	-215,03	3161,53	0,00^{a}	-1688,28	1688,28
7	**Decanol**	**1275**	**6718,75^{a}**	**5188,82**	**8248,68**	**3084,00^{b}**	**1554,07**	**4613,93**	**2769,75^{b}**	**1239,82**	**4299,68**
8	**Undecanal**	**1305**	**1001,00^{a}**	**864,30**	**1137,70**	**1170,00^{a,b}**	**1033,30**	**1306,70**	**1331,25^{b}**	**1194,55**	**1467,95**
9	***β*-Elemene**	**1381**	**548,25^{a}**	**442,86**	**653,64**	**447,75^{a,b}**	**342,36**	**553,14**	**347,75^{b}**	**242,36**	**453,14**
10	***n*-Dodecanal**	**1411**	**31978,20^{a}**	**28063,00**	**35893,50**	**35133,70^{a}**	**31218,50**	**39049,00**	**44233,50^{b}**	**40318,20**	**48148,80**
11	Bergamotene	1428	2233,25^{a}	1810,10	2656,40	2215,75^{a}	1792,60	2638,90	2005,50^{a}	1582,35	2428,65
12	Aromadendrene	1436	203,50^{a}	150,96	256,04	224,75^{a}	172,21	277,29	222,00^{a}	169,46	274,54
13	*α*-Humulene	1448	1442,50^{a}	1225,61	1659,39	1331,25^{a}	1114,36	1548,14	1136,25^{a}	919,36	1353,14
14	**Dodecanol**	**1477**	**6718,75^{a}**	**5422,25**	**8015,25**	**3240,00^{b}**	**1943,50**	**4536,50**	**2785,50^{b}**	**1489,00**	**4082,00**
15	Sesquisabinene hydrate	1536	257,50^{a}	207,62	307,38	284,50^{a}	234,62	334,38	267,25^{a}	217,37	317,13
16	**Decanediol**	**1554**	**219,50^{a}**	**171,88**	**267,12**	**330,75^{b}**	**283,13**	**378,37**	**282,50^{a,b}**	**234,88**	**330,12**
17	**Caryophyllene oxide**	**1569**	**293,00^{a}**	**234,84**	**351,16**	**392,25^{b}**	**334,09**	**450,41**	**373,75^{a,b}**	**315,59**	**431,91**
18	**Humulene epoxide II**	**1595**	**361,50^{a}**	**296,91**	**426,10**	**487,50^{b}**	**422,91**	**552,10**	**436,75^{a,b}**	**372,16**	**501,35**
19	Tetradecanal	1604	577,75^{a}	465,86	689,64	614,50^{a}	502,61	726,39	699,75^{a}	587,86	811,64
20	Unknown 4 (MW = 218)	1625	1385,00^{a}	1023,39	1746,61	1007,25^{a}	645,64	1368,86	930,00^{a}	568,39	1291,61
21	Bisabolol	1662	239,75^{a}	201,10	278,40	292,25^{a}	253,60	330,90	291,50^{a}	252,85	330,15
22	Pentadecanone	1830	507,50^{a}	412,18	602,82	397,75^{a}	302,43	493,07	434,75^{a}	339,43	530,07
23	Unknown 5 (MW = 223)	>2200	276,75^{a}	13,29	540,21	360,75^{a}	97,29	624,21	92,00^{a}	-171,46	355,46
24	**Unknown 6 (MW = 223)**	**>2200**	**128,25^{a}**	**68,94**	**187,56**	**236,00^{b}**	**176,69**	**295,31**	**50,75^{a,c}**	**-8,56**	**110,06**

RI, retention indices relative to C10 – C25 n-alkanes on DB-5 capillary column;F, fresh; S, solar dryer; E, electric dryer

Since the quantization is based upon GC-FID peak integration data, the accuracy is potentially limited by a number of factors, including co-elution of two or more compounds, possible sample discrimination during injection, peak bordering and differences in FID response factors among the components (Pino et al., 2003).

Gernot Katzer's Spice Pages (2007) discuss the composition of the essential oil, which is mainly made up of long-chain aldehydes e.g., decanal (28%), *n*-dodecanal (44%) and decanol (11%). Furthermore sesquiterpenes (α-humulene, β-caryophyllene) account for about 15% of the essential oil. Hence the information was published on the website, it was not possible to link it to a scientific publication.

Influence of climate on EO yield was already described above. Also the EO composition is dependent on such characteristics as the geographic character of the location from which the plant is obtained, seasonal variations and climate, production technique and purity. The effect of plant maturity at the time of oil production and the existence of chemotypic differences can also drastically affect the composition suggesting that ecological condition and or physiological states could interfere with the presence of biologically active compounds in the plant. These variations are of distinct importance for the study of biological and pharmacological activities of these natural products, because the value of an essential oil in aromatherapy has to be related to its chemical composition (Lahlou, 2004).

According to Starkemann et al. (2006) the strong citrus odour of Rau ram leaves attributes to aldehydes such as dodecanal and decanal. It is notable that the content of both compounds was increased by drying. For dodecanal the effect was mild in solar drying while drying in electric dryer had statistically significant effect. The amount of decanal was also was slightly increased by both types of drying.

In contrary to Starkemann et al. (2006) (Z)-3-hexenal, (E)-2-hexenal and (Z)-3-hexen-1-ol could not be determined by our method due to the negative interference with solvent peak, caused by the difference in split/splitless injection used by us and by Starkenmann.

Besides the constituents described in our study, there have been also another compounds previously identified in *P. odoratum* including quercitol, flavonoids, azetidine 2-carboxylic acid, mucous polysaccharides and steroidal compounds including furostanol glycoside and diosgenin (Rafi and Vastano, 2007).

During the experiments with French Tarragon (*Artemisia dracunculus* L.) Arabhosseini et al. (2006b) investigated that drying of leaves at 45 ºC resulted in small changes essential oil, and these qualities are well retained during storage. The changes at 60 ºC were large during drying but small during storage. Our results showed that essential oil composition was close for samples dried in the electric dryer (40ºC, 24h) and samples dried in the solar dryer (47.8-54.6ºC, 8h).

The drying effects, dominated by the air temperature and velocity, were studied by the differences in the composition of the essential oils of lemongrass extracted by hydrodistillation. The temperature varied from 40 to 60ºC and the air velocities investigated were 0.2, 0.5 and 0.8 m/s. The fresh parts contained fewer components than the dry ones. This can be explained by the fact that the water solvates the molecules and the components cannot be extracted. The differences between the air velocities did not influence the composition of the essential oils, but influenced the quantity of the components in the fractions and the time of the drying (Peisíno et al., 2005). Since the air velocity was in close correlation with solar radiation both were important factors in our study having the biggest influence on final moisture content and smaller influence on the essential oil composition.

In dried samples, an increase of terpenoids and phenylpropanoids was observed, when compared to fresh samples. In general, dried samples were slightly richer in oxidized components occurring at the later part of the chromatogram, originating from the thermal degradation. Their overall impact on the odour properties of essential oil is not clear, due to difficult compounds identification. These peaks are minor and based on our general essential oil sensory evaluation, and we consider them as not having a crucial influence on the overall odour.

5. CONCLUSION

The drying tests in our experiment were focused on the chemical composition and yield of the essential oil of Rau ram, on its final moisture content and sensory properties in relation to different types of drying. Interesting findings were made and can be concluded as follows:

1. In all samples, out of 24 constituents detected, 18 were identified. The main compound of the essential oil of Rau ram was *n*-dodecanal averaging (48.3–65.5%) followed by decanal (10.6–14.3%), dodecanol (4.1–10.1%), decanol (4.1–10.2), bergamotene (3.0–3.6%), α-humulene (1.7–2.2%) and undecanal (1.5–2.0%).

2. Out of the main constituents drying had mild but statistically significant effect ($P<0.05$) on four components – *n*-dodecanal, dodecanol, decanol, undecanal.

3. In both cases drying increased *n*-dodecanal content; the effect of solar drying was mild, while the electric dryer caused statistically significant difference ($P<0.05$).

4. In the case of toxic pulegone and menthone, drying in electrical dryer or longer drying interval in solar dryer would be recommended, in order to reduce toxicity.

5. The average essential oil yield for each tested drying method was 0.04, 0.03 and 0.025% (w/w) for fresh sample, solar dryer and electric dryer, respectively. Drying though did not cause considerable decrease of EO yield.

6. Final moisture content (% w.b.) of the product from solar dryer was highly dependent on actual weather conditions, reaching 11.6% FMC (% w.b.) in

ideal conditions and even 23.5% FMC (% w.b.) when the weather was cloudy and rainy.

7. Although the drying period in solar dryer was considerably shorter in comparison to electric dryer (8h, 24h), the product from solar dryer reached 1% lower final moisture content (% w.b.) than the product from electric dryer.

8. Solar drying in double-pass solar-energy dryer was found to be suitable for drying of Rau ram and it had lower effect than electric drying on the majority of compounds, reached suitable final moisture content of the product and caused no remarkable changes in sensory properties.

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7. Annex A.

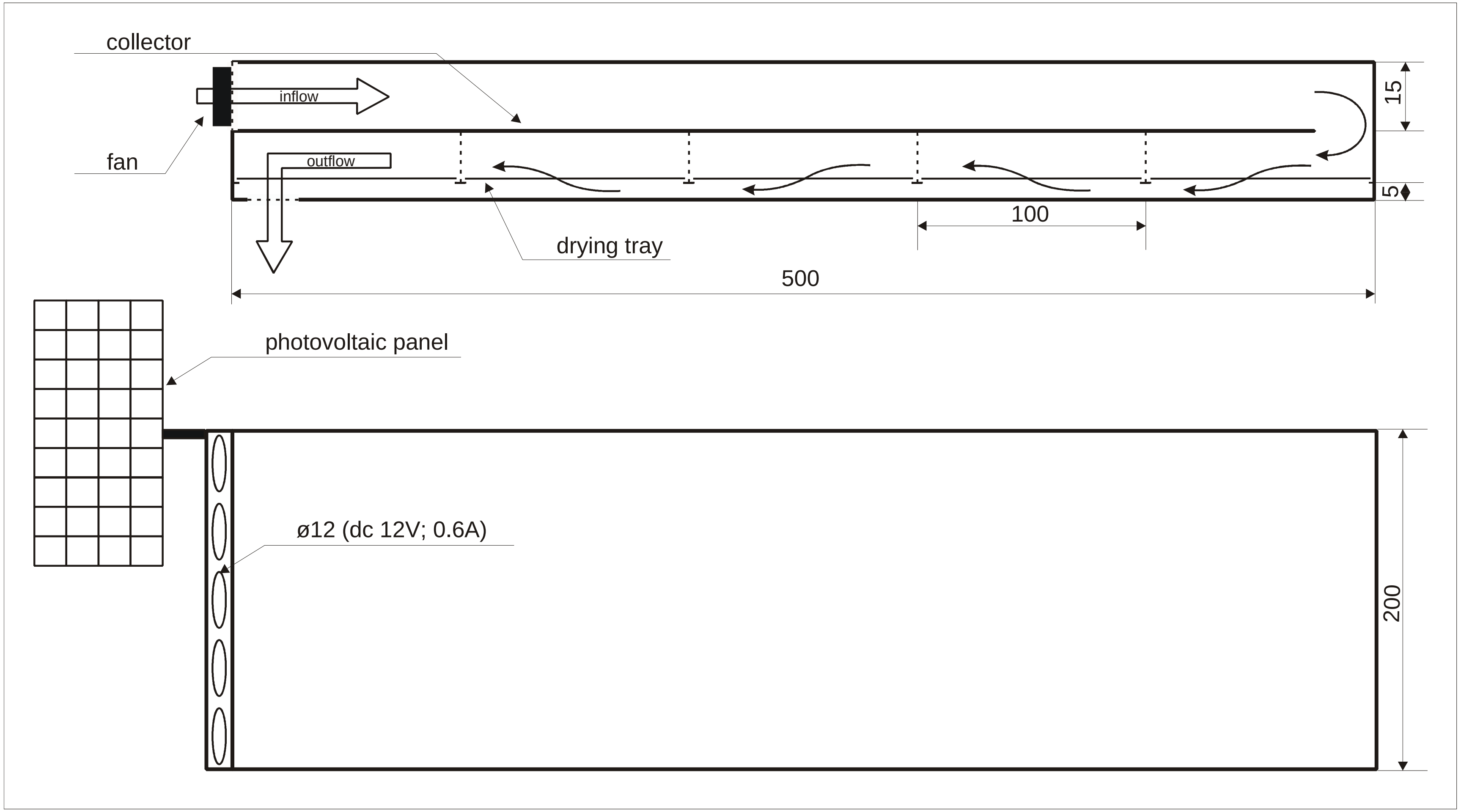
Rau ram sold at local market in Hue.

8. Annex B.

Construction of solar dryer.

Placing the insulation tape between two segments of solar dryer.

9. Annex C

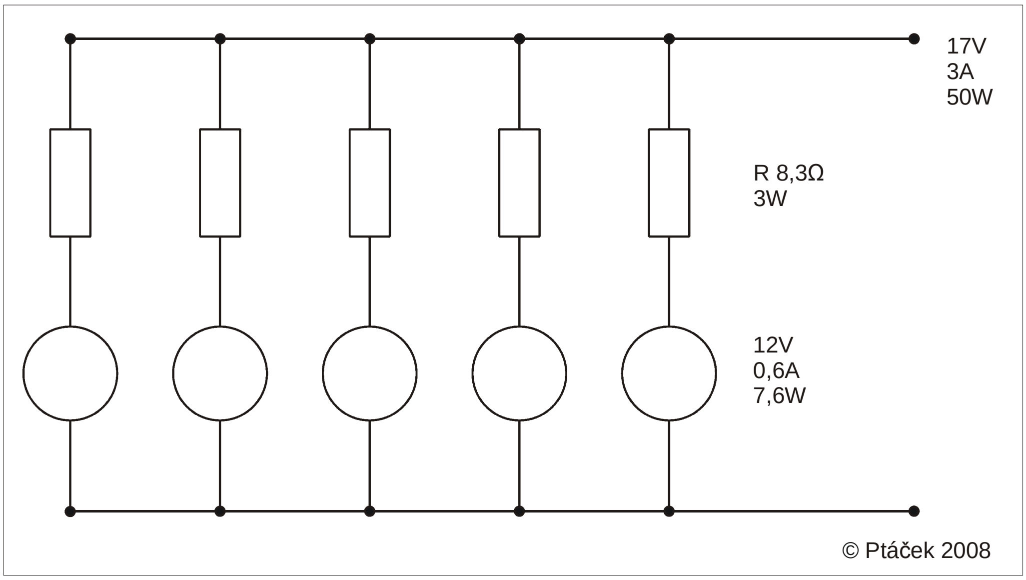


Scheme of double-pass solar-energy dryer (cm).

10. Annex D.

Working dryer.

11. Annex E.



Electric circuit scheme.

Working fans.

12. Annex F.

Reference sample on the drying tray with thermo-hygrometric sensor.

Taking the dried material out of the dryer at the end of drying process.

13. Annex G.

Fresh Rau ram.

Rau ram dried in the solar dryer.

14. Annex H.

Ketonc electric dryer.

Distillation apparatus.

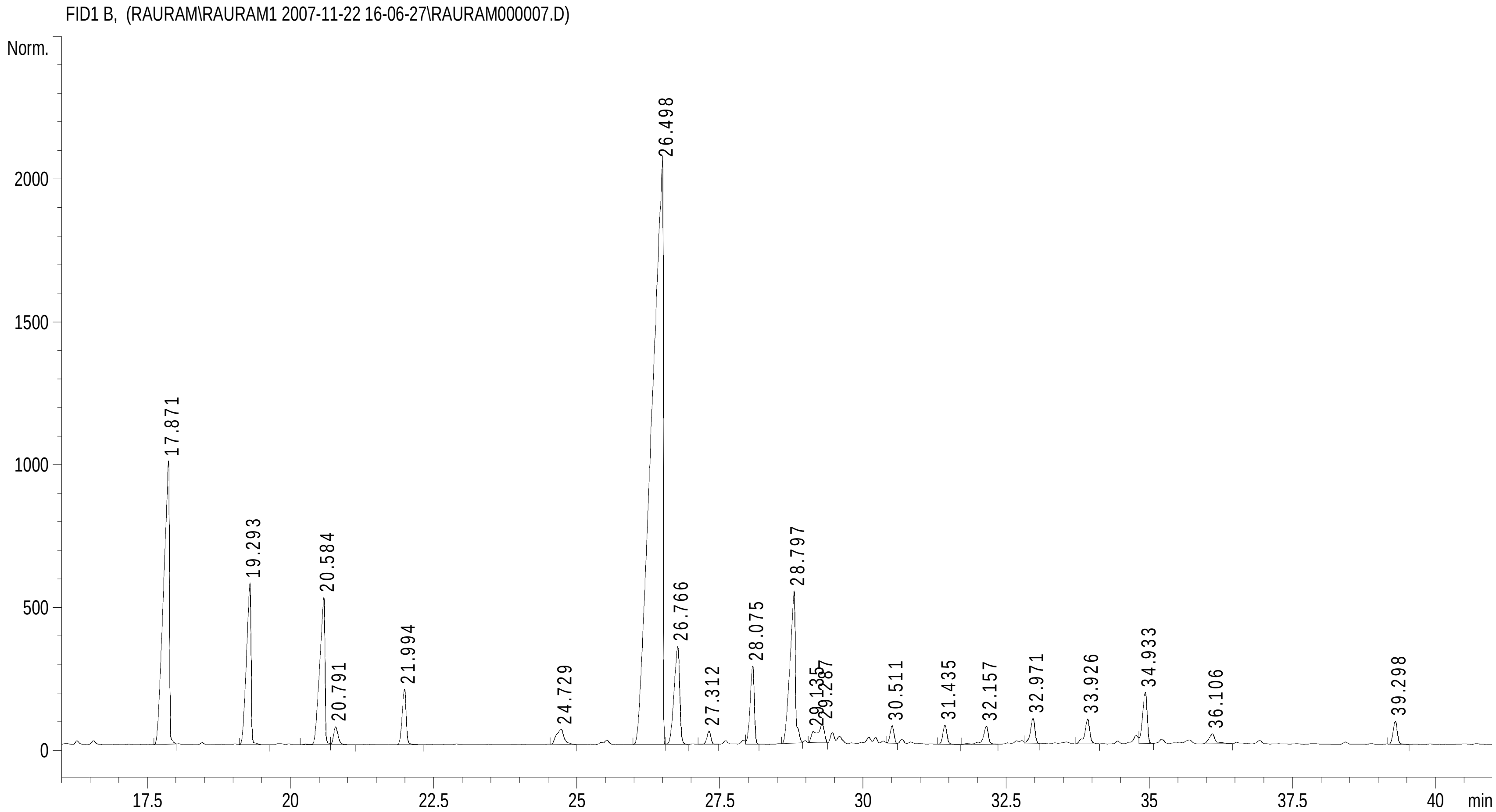
15. Annex I.

Comparison of EO yields.

	F					S					E				
	I	II	III	IV	Mean	I	II	III	IV	Mean	I	II	III	IV	Mean
EO yield (ml)	0,30	0,57	0,38	0,42	**0,418**	0,39	0,22	0,20	0,41	**0,305**	0,30	0,21	0,19	0,37	**0,264**
EO yield (g)	0,30	0,50	0,40	0,40	**0,400**	0,40	0,20	0,20	0,40	**0,300**	0,30	0,20	0,20	0,30	**0,250**
EO content %	0,03	0,05	0,04	0,04	**0,040**	0,04	0,02	0,02	0,04	**0,030**	0,03	0,02	0,02	0,03	**0,025**

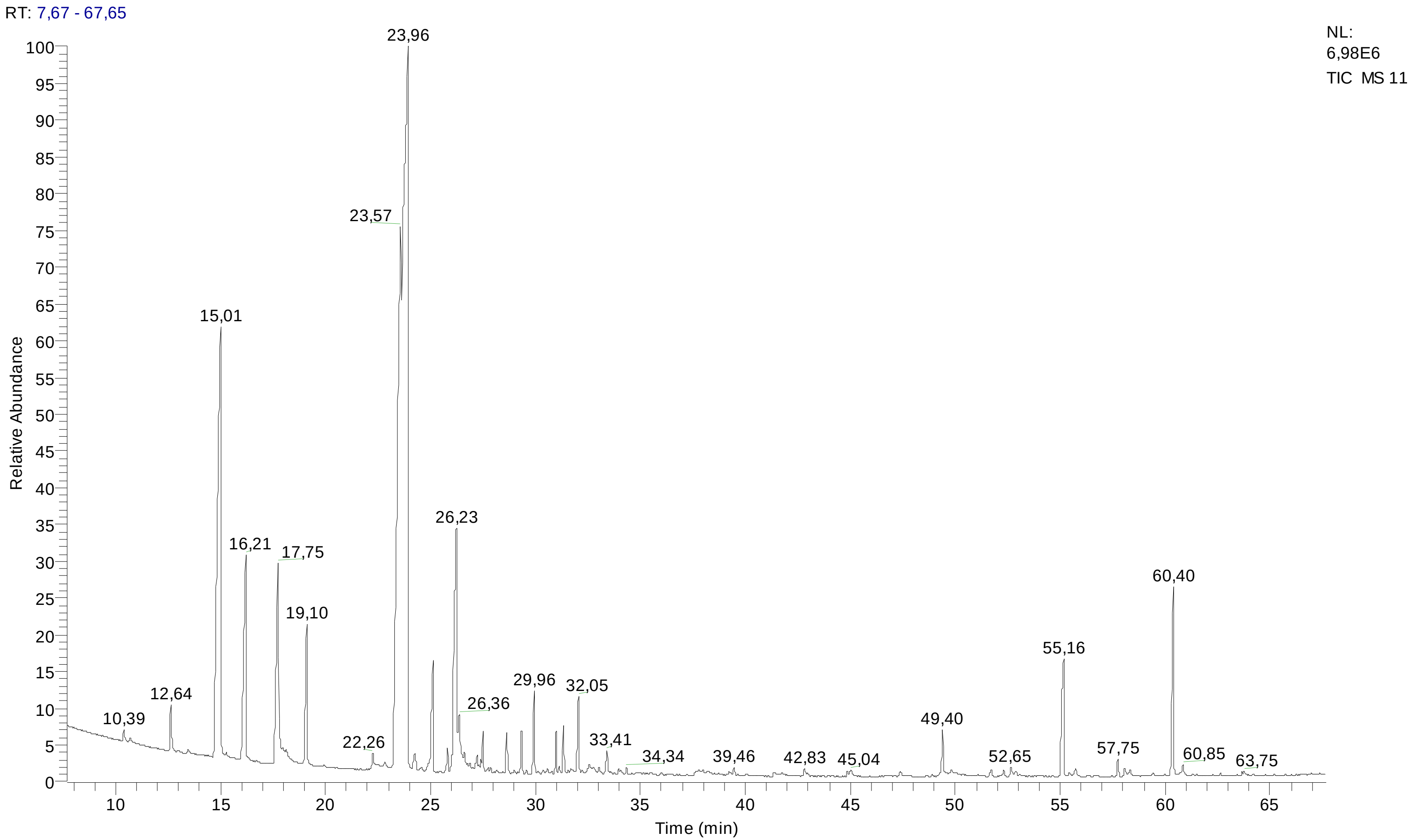
F, fresh; S, solar dryer; E, electric dryer

16. Annex J.



GC-FID chromatogram.

17. Annex K.



GC-MS chromatogram.