# STUDY OF VANILLA ESSENTIAL OIL EXTRACTION: HEAT AND WAVE PRINCIPLES

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#### ABSTRACT

To extract an essential oil the method used is extraction without using organic solvent. The conventional method used is hydro distillation (HD) that applies heat principle. However, a new green technique for essential oil extraction that is microwave which is applies wave principle was developed in recent years. Solvent-free microwave extraction (SFME) is a combination of microwave heating and dry distillation performed at atmospheric pressure without added any solvent or water. SFME with presence of carbonyl iron powder (CIP) was compared with HD for the extraction of essential oil from vanilla (Vanilla planifolia) with different type of raw material sizing. SFME extracted essential oil with higher value of percentage yield and concentration which allowed substantial savings of costs in terms of time, energy and plant material. SFME is an environmental friendly technique as it was rejected less carbon dioxide (CO<sub>2</sub>) compared to HD technique. The strength of aroma of extracted essential oil from both methods was compared by calculating the degree of concentration. The loss of the aroma depends on the degree of concentration. By using high performance liquid chromatography (HPLC) system the presence on vanillin in the extracted oil was identified and has been compared in terms of presence of impurities quantity and concentration. Vanilla treated by SFME and HD were viewed by scanning electron microscopy (SEM) and the results reveal that vanilla structure treated by SFME was being more ruptured compared to conventional HD technique.

Keywords: Essential oil; Vanilla planifolia; Vanillin; Solvent-free microwave extraction (SFME); Hydro distillation (HD).

#### **CHAPTER 1**

#### INTRODUCTION

#### **1.1 Background of study**

Vanilla beans are the fruits of Vanilla planifolia Andrews that is the group of Orchidaceae. The curing of green vanilla beans to obtain the well-appreciated vanilla aroma is a very laborious process. Every vanilla cultivating country has developed its own curing process but it generally consists of four steps that are scalding, sunning or sweating, drying and conditioning. Usually, scalding is followed by an autoclaving step. This means that the beans that have become hot after scalding are put into an airtight container overnight to retain as much heat as possible. It can be regarded as the first sweating step. The whole process normally takes more than 6 months (Dignum et al., 2002).

Vanilla planifolia has opposite, sessile leave of 10 to 23 cm long which are oblong in shape. Vanilla is a climbing plant whose stems may grow up to 30 m long. It has aerial roots that it sticks to the tree that serves it as support. It has fleshly leaves and large blackish-ochre pods with many seeds (Roger, 2002).

The curing of green vanilla beans to obtain the well-appreciated vanilla aroma is a very laborious process. The highest grade of cured vanilla is Black vanilla and the lowest grade is Red vanilla (Loeillet, 2003). Vanilla has stomachic, digestive and choleretic that increase the bile secretion, mildly invigorating and aphrodisiac properties. Although it's present use is only as a seasoning it is worth remembering its invigorating benefits to the digestive functions (Roger, 2002).



Figure 1.1 Vanilla beans

The main component of the vanilla aroma is vanillin (4-hydroxy-3ethoxybenzaldehyde) which was first isolated and identified in vanilla by Gobley et al, 1858. In the green bean important phenolic aroma compounds are present as glucosides. The curing process is meant to release the aglycons to set free the aroma compounds. In the last century, considerable research was devoted to the vanilla curing process. Many experiments have been conducted to determine the optimal conditions to obtain a good quality of cured vanilla, the important compounds and enzymes that are involved (Dignum et al., 2002).

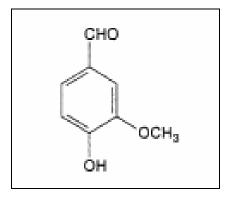


Figure 1.2 Vanillin

Vanillin is obtained from sulfite waste liquor by further alkaline hydrolysis of lignin. The same substance can be obtained from vanilla bean extract and is the common flavoring in foods and drinks. Natural and synthetic vanillin can be distinguished from each other by a slight difference for 13C in their structure since one is biosynthetic in the bean and the other is isolated from a second natural product, wood and hydrolysis from the lignin (Speight, 2000).

Natural vanilla is one of the most widely used and important flavoring materials worldwide. The source of vanilla is the bean or pod of the tropical Vanilla orchid that is principally Vanilla planifolia Andrews, synthetic V. fragrance. The Aztecs of Mexico cultivated Vanilla which was then brought to Europe by the Spaniards after 1520 and is now cultivated in a number of tropical countries. The major producers are Mexico, Madagascar, Tahiti and Indonesia. Vanillin in fact occurs in trace amounts in other plants including commercial products such as tobacco. However, the pods of the Vanilla orchid remain the only commercial source of natural vanillin (Walton et al., 2003).

A new technique to extract essential oils is microwave extraction that applies wave principle was developed in recent years. Solvent-free microwave extraction (SFME) is a combination of microwave heating and dry distillation performed at atmospheric pressure without adding any solvent. Microwave heating has already been widely applied in solvent extraction because of its main advantages like rapidity and high efficiency. SFME performed at atmospheric conditions without adding any solvent or water provided a new idea in the extraction of volatile compounds from fresh plant materials or prior moistened dried material. Besides, it made the whole process to be more simple, rapid and economic. Many scientists had applied SFME in extractions from aromatic herbs and spices (Wang et al., 2005).

#### 1.2 Objective

The objective of this research is to study the solvent-free extraction of vanilla essential oil through hydro distillation and solvent-free microwave extraction techniques.

#### 1.3 Scopes

The scopes of this study are:

- 1. To study the effect of raw material sizing based on presence of vanillin in extracted product from HPLC analysis.
- 2. To study the effect of raw material sizing based on extracted product concentration.
- 3. To study the effect of raw material sizing based on strength of aroma by calculating the product degree of concentration.
- 4. To study the energy consuming based on extraction time and electrical power.
- 5. To study the environmental effect by calculating  $CO_2$  rejected.
- 6. To study the vanilla structural changes by using Scanning Electron Microscopy (SEM).

#### **1.4 Problem statements**

Vanilla has very high demand and prices in the world for it's popular uses in flavoring ice creams and soft drinks. However, there is limited research on vanilla extraction in the world. Vanilla is also used in production of soaps, body lotions, floor polishes, air-fresheners, detergents, perfumes, cakes and pharmaceutical products.

Vanilla essential oil extraction industry has a big potential to grow up in Malaysia. At this moment, there is no vanilla essential oil extraction industry in Malaysia. Malaysia import the vanilla extracts from other country such as Indonesia for food manufacturing industry. This country is at tropical region and has hot climate as same as climate at Indonesia which is one of the largest and popular producer of vanilla in the world. Malaysia also safe from bad disaster such as hurricane. Hurricane at Madagascar in 1999 was destroying about 35% of the crop and 15% of the stocks.

Even though vanilla is quite expensive, the extraction of vanilla can yield not more that 5% of essential oil. Besides, the solvent extraction also not really safe for human health because it uses organic solvent such as methanol, ethanol, hexanol and others. It can cause hepatitis and kidneys malfunction. Solvent extraction also required higher cost of extraction because the uses of organic solvent.

The demand for increasingly clean and efficient chemical synthesis is continuously becoming more urgent from both an economic and an environmental standpoint. So-called green technologies are looking for alternatives yet they focus on large quantities of hazardous even toxic solvents. One could ever say that the best solvent is no solvent. Conventional method of extractions that are hydro and steam distillation that applies heat principle are the common methods that are always used. The methods will yield many impurities in the essential oil extracted, needs long period of extraction and yield the low strength of aroma. However, a new green technique to extract essential oils that applies wave principle that is solvent-free microwave extraction was developed in recent years that make the extraction simpler and save

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energy. The technique will yield better quality of essential oil aroma, higher percentage yield and reduce the number of impurities to be volatized during the extraction. Besides, this technique prevents solvent wastes, hazards and toxicity.

#### **CHAPTER 2**

#### LITERATURE REVIEW

Concentrated vanilla extracts have attained popularity with the manufacturing trade because they are cheaper than standard vanilla extract. There is true economy in a use of a concentrated extract because of the appreciable saving in alcohol and provided the concentrated product retains the full strength and quality of flavor of the beans from which it was made (Dignum et al., 2002).

#### 2.1 Essential oils and oleoresins

#### 2.1.1 Essential oils

An essential oil is a concentrated and hydrophobic liquid containing volatile aromatic compounds extracted from plants. It may be produced by distillation, expression or solvent extraction. Essential oils are used in perfumery, aromatherapy, cosmetics, incense, medicine, household cleaning products and for flavoring food and drink. They are valuable commodities in the fragrance and food industries. The essential oil has been obtained by steam distillation while the oleoresin is produced using solvent extraction (Povh et al., 2001)

Essential oils are mainly formed by hydrocarbon and oxygenated terpenes and by hydrocarbon and oxygenated sesquiterpenes. They can be extracted from seeds, roots, flowers, herbs and leaves using the hydro distillation technique. This is a very simple process but suffers of many drawbacks such as thermal degradation, hydrolysis and volatilization in water of some compounds that alter the flavor and fragrance profile of many essential oils extracted by this technique (Reverchon and De Marco, 2006).

Essential oil is also known as volatile oil and ethereal oil. It may also be referred as oil of the raw plant material from which it was extracted such as oil of clove. The term essential is intended to convey that the oil is an essence of the plant it is extracted from. It is not in the more common sense of being indispensable and do not confuse them with essential fatty acids. The conventional methods used for the preparation of essential oils and spice oleoresins are steam distillation and solvent extraction respectively (Simandi et al., 1998).

The essential oils of plants have usually been isolated by either hydro distillation or solvent extraction. The disadvantages of all these techniques are low yield, losses of volatile compounds, long extraction times, toxic solvent residues, degradation of unsaturated compounds and giving undesirable off-flavor compounds due to heat (Mostafa et al., 2004).

#### 2.1.2 Oleoresins

Resin is a hydrocarbon secretion of many plants valued for its chemical constituents and uses such as varnishes and adhesives. The term is also used for synthetic substances of similar properties.

Vanilla oleoresin is a semi-solid concentrate obtained by removing the solvent from the vanilla extract. A solution of isopropanol is frequently used instead of ethanol for the preparation. Some flavor and aroma is lost during removal of the solvent but it does contain essential oils. Vanilla oleoresin is used in non-food products. Unfortunately, it is not always stable in candle and soap making as it is considerably less expensive than vanilla absolute.

Vanilla oleoresins are employed as a base for the manufacture of flavors and have been articles of commerce for many years but the literature on flavoring materials indicates that food officials have given little attention to the detection of their adulteration (Wilson and Sale, 1926).

#### 2.2 Other raw materials of essential oil extraction

#### 2.2.1 Patchouli

Patchouli is both a plant and an essential oil obtained from the leaves of that plant. The scent of patchouli is heavy and strong. Many people find it offensive. It has been used for centuries in perfumes. Patchouli grown in the East and West Indies. Patchchouli is a word derives from the Tamil patchai means green and ellai means leaf.

Patchouli oil and incense underwent a surge in popularity in the 1960s and 1970s mostly among devotees of the free love and hippie lifestyles as it covers up the scent of marijuana. The Hare Krishna movement may have been responsible for this surge as the god Krishna was said to "inhabit" patchouli. Besides, it can be used as a hair conditioner for dreadlocks.

Despite its common association with an alternative lifestyle patchouli has found widespread use in modern industry. It is a component in about a third of modern highend perfumes including more than half of perfumes for men. Patchouli is also an important ingredient in East Asian incense. It is also used as a scent in products like paper towels, laundry detergents and air fresheners. The essential oil is obtained by steam distillation of the dried leaves of the plant. Steam distillation is a process that provides a relatively high yield of the oil. An important component of the essential oil is patchoulol.

During the 18<sup>th</sup> and 19<sup>th</sup> century silk traders from China traveling to the Middle East packed their silk cloth with dried patchouli leaves to prevent moths from laying their eggs on the cloth. Many historians speculate that this association with opulent eastern goods is why patchouli was considered by Europeans of that era to be a luxurious scent. This trend has continued to the present day in modern perfumery.

The patchouli plant is a bushy herb reaching two or three feet in height. The plant grows well in southern climates. It enjoys hot weather but not direct sunlight. If the plant withers due to lack of watering it will recover well and quickly once it has been watered. The seed-bearing flowers are very fragrant and bloom in late fall. The tiny seeds may be harvested for planting but they are very delicate and easily crushed. Cuttings from the mother plant can also be rooted in water to produce further plants (Roger, 2002).

#### 2.2.2 Lavender

The lavenders Lavandula are a genus of about 25 to 30 species of flowering plants in the mint family Lamiaceae. It is native from the Mediterranean region south to tropical Africa and east to India. The genus includes annuals, herbaceous plants, subshrubs and small shrubs. The native range extends across the Canary Islands, north and east Africa, south Europe and the Mediterranean, Arabia and India. Because the cultivated forms are planted in gardens worldwide they are occasionally found growing wild as garden escapes.

Lavender is one of the most useful medicinal plants. Commercially the lavender provides several important essential oils to the fragrance industry including soaps, colognes, perfumes, skin lotions and other cosmetics. In food manufacturing, lavender essential oil is employed in flavoring beverages, ice cream, candy, baked goods and chewing gum. The essential oils of Lavandula species are obtained by steam distillation of the fresh flowering spikes. Oil quality is assessed by oil chemical composition and by the organoleptic opinion of the flavorists (Chemat et al., 2005).

Lavenders are widely grown in gardens. Flower spikes are used for dried flower arrangements. The fragrant, pale purple flowers and flower buds are used in potpourris. Dried and sealed in pouches, they are placed among stored items of clothing to give a fresh fragrance and as a deterrent to moths. The plant is also grown commercially for extraction of lavender oil from the flowers. This oil is used as an antiseptic and for aromatherapy.

Lavender flowers yield abundant nectar that yields a high quality honey for beekeepers. Lavender honey is produced primarily in the nations around the Mediterranean and marketed worldwide as a premium product. Lavender flowers can be candied and are used as cake decoration (Roger, 2002).

#### 2.2.3 Cymbopogon

Cymbopogon that is also called as lemon grass, lemongrass, citronella grass or fever grass is a genus of about 55 species of grasses, native to warm temperate and tropical regions of the Old World. It is a tall perennial grass. Lemon grass is widely used as an herb in Asian and Caribbean cooking. It has a lemony flavour and can be used fresh, dried and powdered. The stalk itself is too hard to be eaten except for the soft inner part. However, it can be finely sliced and added to recipes. It may also be bruised and added whole as this releases the aromatic oils from the juice sacs in the stalk. The main constituent of lemongrass oil is citral.

Lemon grass is commonly used in teas, soups, and curries. It is also suitable for poultry, fish and seafood. It is often used as a tea in African countries.

The East-Indian lemon grass (Cymbopogon flexuosus) is native to India, Sri Lanka, Burma, and Thailand while the West-Indian lemon grass (Cymbopogon citratus) is assumed to have its origins in Malaysia. While both can be used interchangeably, Cymbopogon citratus is more suited for cooking. In India, Cymbopogon citratus is used both as a medical herb and in perfumes (Roger, 2002).

#### 2.2.4 Orange

Sweet orange is refers to the citrus tree Citrus sinensis and its fruit. The orange is a hybrid of ancient cultivated origin possibly between pomelo (Citrus maxima) and tangerine (Citrus reticulata). It is a small tree, growing to about 10 m tall with thorny shoots and evergreen leaves 4 to10 cm long. The word "orange" comes from Sanskrit "narang". Oranges originated in Southeast Asia in either India, Vietnam or southern China. The fruit of Citrus sinensis is called sweet orange to distinguish it from Citrus aurantium that is the bitter orange.

Oranges are widely grown in warm climates worldwide and the flavors of orange vary from sweet to sour. The fruit is commonly peeled and eaten fresh or squeezed for its juice. It has a thick bitter rind that is usually discarded, but can be processed into animal feed by removing water using pressure and heat. It is also used in certain recipes as flavoring or a garnish. The outer-most layer of the rind is grated or thinly veneered with a tool called a zester to produce orange zest. It is popular in cooking because it has a flavor similar to the fleshy inner part of the orange. The white part of the rind, called the pericarp or pith is a source of pectin (Roger, 2002).

#### 2.2.5 Ylang-ylang

Ylang-ylang or Cananga odorata is the flower of the cananga tree. The tree attains an average height of 12 meters grows in full or partial sun and prefers the acidic soils of its native rainforest habitat. The leaves are long, smooth and glossy. The flower is greenish yellow or pink, curly like a starfish and yields a highly fragrant essential oil.

The word derives from the Tagalog plant name ilang-ilang that means flower of flowers and does not allude to the pleasant scent of ylang-ylang flowers.

The fragrance of ylang-ylang is rich and deep with notes of rubber and custard, and bright with hints of jasmine and neroli. The essential oil of the flower is obtained through steam distillation of the flowers and separated into different grades according to when the distillates are obtained. The main aromatic component of ylang-ylang is methyl anthranilate.

Ylang-ylang essential oil is used in aromatherapy where it is believed to relieve high blood pressure, normalize sebum secretion for skin problems and considered to be an aphrodisiac. The oil from ylang-ylang is widely used in perfumery for oriental or floral themed perfumes. Ylang-ylang blends well with most floral, fruit and wood smells. In Indonesia, ylang-ylang flowers are spread on the bed of newlywed couples (Roger, 2002).

#### 2.2.6 Ginseng

Ginseng (*Panax Ginseng*) root has been continuously used for more than 4,000 years in China due to its invigorating properties. It was introduced in Europe during the eighteenth century and has bees the issue of many scientific studies due to its extraordinary virtues. Its scientific name *Panax* comes from the Greek words *pan* that is meaning of all and *axos* that is meaning of healing. For Chinese people, Ginseng is a true panacea that is able to heal a wide range of afflictions. Its aphrodisiac effects have given it a wide popularity in Western countries in which tobacco, alcohol and other drugs have become a continuous aggression to sexual performance.

The active components of ginseng root are so chemically complex that it has not been possible to synthesize them up to now. They are called ginsenosides and chemically these are steroid glycosides from the group of triterpenic saponins. Therapeutic properties of ginseng are due mainly to these subtances but are also enhanced by other components that are minerals and trace elements, sulphur, manganese, germanium, magnesium, calcium, zinc, vitamin B complex, phutosterol and enzymes.

Ginsenosides increase physical performance and endurance. This is not due to any excitant properties such as cocaine, coffee, tea or other drugs but to an improvement of metabolic processes. Ginseng speeds up the enzymatic process of glycogenesis that is the production of glycogen on the liver from sugar and glycogenolysis that is production of sugar from the stored glycogen decreases the concentration of lactic acid in muscles that causes stiffness.

Ginseng promotes spermatogenesis, stimulates sexual glands and increases hormone production. It increases sexual capability, improving both frequency and quality of male erection and promoting female genital organs excitation. Ginseng also promotes mental performance, increasing concentration and memory capabilities. Other than that, ginseng has anti-stress properties due to its adaptogenic properties, because it increases adaptation capabilities of the body to physical and psychological efforts. Research conducted on animals has proven that both hypophysis and suprarenal glands are stimulated with ginseng (Roger, 2002)

#### 2.3 Vanilla

Vanilla is the only edible fruit of the orchid family that is the largest family of flowering plants in the world. It is a tropical orchid and there are about 150 varieties of vanilla though only two types, Bourbon and Tahitian that are used commercially.

#### 2.3.1 Types of Vanilla

#### **2.3.1.1 Bourbon beans**

Bourbon beans are long and slender with a very rich taste and smell. Bourbon beans have thick oily skin containing an abundance of tiny seeds and have a strong vanilla aroma. Bourbon beans from Madagascar and the Comoros are described as having creamy, hay like and sweet with vanillin overtones. Bourbon beans from other regions will be similar if they are picked at peak ripeness and are properly cured.

#### 2.3.1.2 Mexican beans

Mexican beans are very similar to Bourbon beans though they have a mellower, smooth and a spicy, woody fragrance.

#### 2.3.1.3 Tahitian beans

Tahitian beans are usually shorter, plumper and contain a higher oil and water content than Bourbon beans. The skin is thinner and they contain fewer seeds. The aroma is fruity and floral. It often described as smelling like licorice, cherry, prunes or wine (Kroschwitz and Grant, 1997).

#### 2.3.2 Vanillin

Vanillin is a natural product that can be found as a glucoside that is glucovanillin in vanilla beans at concentrations about 2%. It can be extracted with water, alcohol or other organic solvents. Vanillin was observe long before it was reported in chemical literature as it crystallizes on the surface of vanilla beans after harvesting, processing and storage. The first report in the literature was probably made by Bucholtz in 1816. Some years later, Bley referred to vanillin from alcoholic solutions of vanilla bean extract and succeeded in obtaining it in a relatively pure form. He reported its composition to be  $C_{10}H_6O_2$ . Its correct analysis that is  $C_8H_8O_8$  was established in 1872 by Carles who gave its correct melting point that is  $81^{\circ}C$ .

Vanillin being an aldehyde is able to form acetals and hemiacetals. Therefore, in flavor formulations using high concentration of vanillin in conjunction with carriers such as propylene glycol, a glycol analysis often shows a reduced vanillin peak after storage of the compound flavor and the presence of new peaks indicating acetal formation. Addition of about 0.5% of water to the formula reverses the reaction for example there is a reduction of acetal and the reappearance of vanillin peaks (Kroschwitz and Grant, 1997).

## 2.3.3 Properties of vanillin

Synonyms	4-hydroxy-3-methoxybenzaldehyde			
Molecular Weight	152.15			
Chemical Formulas	C <sub>8</sub> H <sub>8</sub> O <sub>3</sub>			
Appearance	White to light yellow crystals			
Odor	Pleasant odor			
Solubility	Soluble in 125 parts water			
Specific Gravity	1.056			
рН	No information found			
Percentage Volatiles by volume or 21°C	0			
(70F)				
Boiling Point	285°C (545°F)			
Melting Point	81°C to 83°C (178°F to 181°F)			
Vapor Density (Air=1)	5.2			
Vapor Pressure (mm Hg)	0.0022 or 25°C (77°F)			
Evaporation Rate	No information found			
Stability	Slowly oxidizes in moist air and light			
	sensitive			
Hazardous Decomposition Products	Carbon dioxide and carbon monoxide may			
	form when heated to decomposition			
Hazardous Polymerization	Will not occur			
Incompatibilities	Strong oxidizers			

Table 2.1: Properties of Vanillin

#### 2.3.4 Uses of vanilla

#### 2.3.4.1 Food flavoring compounds

At least 30% of food-grade vanillin consumed in the world is through flavoring compounds. Flavoring compounding requires expertise to develop well-balanced and complex flavors such as fruit flavors. In the industrial production of dry cookies, cakes and pastries, the vanillin content ranges between 20 and 50 g per 100 kg of dough. Vanillin also is added during the chocolate manufacturing process in powder form in average amount of 20 g per 100 kg of the finished product. Although the vanillin concentration is a matter of taste depending on different factors in each individual case the following concentrations are generally accepted.

**Table 2.2:** Accepted vanillin concentration for dark chocolate and milk chocolate

Vanillin flavoring	g/100kg
Dark chocolate	15-60
Milk chocolate	5-30

In confections, the main applications are sugared almonds, caramel, nougat and sweets. For sugared almonds and caramel, vanillin is mixed into the sugar in the dry phase of the recipe. For nougat, vanillin is added during the liquid phase of manufacturing. In sweets, vanillin is added in the form of a 10% ethanol solution. The following may serve as a rough guide.

Vanillin flavoring	g/100 kg
Soft-center sweets	5-15
Other sweets	15-30
Caramel	15-55
Chewing gum	15-45
Nougat	40-55

 Table 2.3: Accepted vanillin concentration for various products

The main application of natural vanilla is for flavoring ice creams and soft drinks. It is estimated that nearly 300 tonnes of vanilla beans is used in USA every year in the preparation of cola type drinks. The major industrial purchasers of vanilla are pharmacy companies and soft drink companies like Coke and Pepsi. However, the fact remains that market for natural vanilla essence is today largely only confined to the West. There is no market in India now for vanilla essence. The domestic market in India is restricted to the green vanilla beans. In India, processing companies buy green vanilla beans from the farmers to process it and then export the same to foreign buyers who then do the extraction of vanilla essence (Kroschwitz and Grant, 1997).

#### 2.3.4.2 Animal feed

Vanillin is used as a palatability enhancer to make animal feed more appetizing by flavor-masking minerals with off-taste. Approximately 5 g of vanillin/100 kg of feed is added when preparing feed for lambs and pigs in order to increase feed intake and stimulate the growth of the animals. Vanillin is added during the manufacturing process either by mixing into the dry ingredients or in its liquid form. Increasingly vanillin is also used as a substitute for aniseed (Kroschwitz and Grant, 1997).

#### 2.3.4.3 Perfumes and cosmetics

In aldehylic perfumes, vanillin provides the powdery impressions given by the background smell usually up to 2% in the perfume concentrate. In fruity notes, vanillin enhances the fruity for instance a peach is not fully peach without vanillin. When vanillin is combined with some floral notes such as heliotrope and orchid that actually contain strong vanilla impressions, amounts of 2 to 5% are possible. However, with notes such as rose, orange flower and jonquil the addition of 0.1 to 2% vanillin can bring warmth and elegance. In woody families such as fougere and in spicy perfumes the harsh impression also needs the fine and smooth aroma provided by vanillin traces. In

detergent perfumes, the stability of vanillin is not always certain. It depends on the association made with other raw materials such as patchouli, frankincense, cloves, most of the animal notes and such chemicals as amyl salycate, methyl ionones and oak mosses. In some cases, these mixtures can cause discoloration effects. In cosmetics as in bath products most of the problems arising with the use of vanillin are related to the soap perfumery problems. The amount of perfume concentrate used in bath products is usually lower than that used in fine fragrances. Vanillin is also useful as a deodorant to mask the unpleasant odor of many manufactured goods. As a masking agent for numerous types of ill-smelling mass-produced industrial products particularly those of synthetic rubber, plastics, fiberglass and inks, vanillin find extensive use (Kroschwitz and Grant, 1997).

#### **2.3.4.4 Pharmaceutical products**

The single largest use of vanillin is as a starting material for the manufacture of an antihypertensive drug having the chemical name of Methyldopa or 3, 4dihydroxyphenyl-2-methylalanine. L-Dopa and Trimethoprim are two other drugs that can be made from vanillin. L-Dopa is used for the treatment of Parkinson's disease. Trimethoprim is an anti-infective agent used mainly for urinary tract infections and certain venereal disease. In addition, Mabeverine that is an antispasmodic agent and Verazide, a generic anti-tubercular agent are drugs that can be made from vanillin or its derivatives. Papeverine that is used to treat heart disease as a vasodilator is a drug that was originally made from vanillin but has since been made from veratrole and ortho-1, 2-dimethoxybenzene. Vanillin is also used as a pharmaceutical excipient (Kroschwitz and Grant, 1997).

#### 2.3.4.5 Agrochemical products

Hydrazones of vanillin have bees shown to have an herbicidal action similar to that of 2, 4-D and the zinc salts of dithiovanillic acid. Made by the reaction of vanillin and ammonium polysulfide in alcoholic hydrochloric acid, dithiovanillic acid is a

vulcanization inhibitor. A new potential use for vanillin is as a ripening agent to increase the yield of sucrose in sugarcane by the treatment of the cane crop a few weeks before harvest (Kroschwitz and Grant, 1997).

#### 2.3.4.6 Industrial applications

The anti-ultraviolet protection properties of vanillin have been patented and look promising for the plastics and cosmetics industries. Other uses of vanillin include the prevention of foaming in lubricating oils as a brightener in zinc coating baths, as an activator for electroplating of zinc, as an aid to the oxidation of linseed oil, as an attractant in insecticides, as an agent to prevent mouth roughness caused by smoking tobacco, in the preparation of syntans for tanning, as solubilizing agent for riboflavin, and as a catalyst to polymerize methyl methacrylate (Kroschwitz and Grant, 1997).

#### 2.3.5 Vanilla world production

World production is of the order of 5 400 tonnes (FAO data, 2002). It has increased strongly in recent years and is returning to the record level of the end of the 1980s. The collapse of production in 1990 and 1991 has been forgotten. Average annual growth has been about 4 percent over a 10-year period.

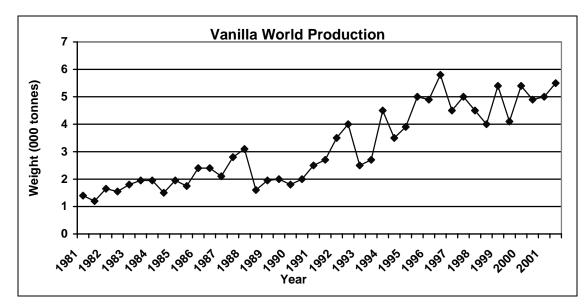


Figure 2.1 World production of vanilla 1981-2001 (Loeillet, 2003).

Vanilla demand can be estimated using customs data in the main consumer countries that are United States, European Union and Japan. Based on various sources the international market in 2001 would seem to have totaled some 2 300 tonnes excluding re-exports from non-producer countries and a figure that has been comparatively stable for three years. The United States has the lion's share with nearly two-thirds of world imports. The European Union takes 30 %. Japan and the rest of the world have equal shares in the remaining 6 %. Re-exports from non-producer countries total about 70 tonnes per year, with United States re-exporting 40 to 50 tonnes.

France is the main European Union importing country that is 258 tonnes, followed by United Kingdom with 151 tonnes and Germany that is 144 tonnes. These three countries account for three-quarters of European imports.

The present international demand from vanilla is about 19,000 tones. Practically all exports are from four countries, with Madagascar being the uncontested leader with 63 percent of international supplies in 2001. Indonesia and the Comoros are present on the market with 21 % and 9 % respectively. They are followed by modest suppliers like Uganda, India, Jamaica and Papua New Guinea.

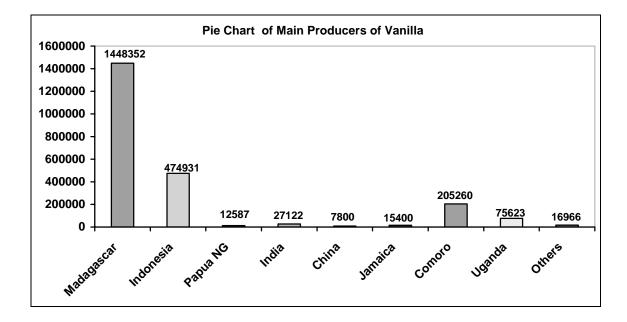


Figure 2.2 Main producers of vanilla (Loeillet, 2003).

India has just come into the market for production. Their production in 2002 was a merge 30 tonnes only. India is still a very insignificant player in vanilla. It will take some more time before they make their presence felt in the world markets. Presently Indian farmers are getting around Rs 150 per kg of green vanilla beans. The same green beans when they are processed fetch a price of around Rs. 1500 per kg.

However, processing technology in India for vanilla is still very primitive and many farmers are satisfied with just growing and supplying green beans. Considering the fact that cost of production is low, farmers are finding vanilla beans cultivation very attractive. In future, more farmers will take up this crop and the production and export figures of vanilla will increase (Loeillet, 2003).

Black vanilla	– Imports and	d re-exports	– Estimates 2	001	
In kg	Total	EU	USA	Japan	Others
Producer countries	2 284 041	707 000	1 444 900	70 599	61 542
Madagascar	1 448 352	546 000	807 300	53 706	41 346
Indonesia	474 931	19 000	447 700	600	7 631
Comoros	205 260	98 000	93 000	11 355	2 905
Uganda	75 623	16 000	58 600	1 023	0
India	27 122	4 000	22 800	0	322
Jamaica	15 400	15 000	400	0	0
Papua N.G.	12 587	5 000	4 300	23	3 264
China	7 800	2 000	100	100	5 600
French Polynesia	7 800	0	3 700	1 014	196
Tonga	3 818	0	3 000	696	122
Mexico	3 646	0	3 500	0	146
Reunion	1 946	0	0	1 946	0
Mauritius	1 000	1 000	0	0	0
Turkey	1 000	1 000	0	0	0
Costa Rica	410	0	400	0	10
Vanuatu	136	0	0	136	0
Taiwan	100	0	100	0	0
Re-exports	72 200	47 000	25 200	0	0
United States	43 000	43 000	0	0	0
Switzerland	2 000	2 000	0	0	0
Norway	1 000	1 000	0	0	0
Netherlands	5 000	0	5 000	0	0
France	5 100	0	5 100	0	0
Germany	5 100	0	5 100	0	0
Italy	100	0	100	0	0
Japan	100	0	100	0	0
Spain	800	0	8000	0	0
United Kingdom	9 000	0	9 000	0	0
Singapore	1 000	1 000	0	0	0

Table 2.4: Export and import of vanilla (Loeillet, 2003).

Vanilla is the most sought out flavor among the food lovers and it is recently voted as one among the top five flavors in US. According to ITC/UN Statistics, the total global demand for vanilla is projected at 19000 metric tonnes a year with the world market for vanilla beans highly concentrated in a few developed countries. The market for vanilla stood at 2200 tonnes per year in 2000. However, the arrival of vanilla into international market was badly hit as a devastating hurricane in Madagascar that is the world's biggest vanilla producer destroyed about 35 % of the crop and 15 % of the stocks. Due to this the prices have risen from USD 25 to USD 40/kg in 1999 to their present level of USD 200 to USD 230. US, France and Germany account for about 80 % of world imports, the US absorbing 50 to 60 % and France and Germany between 10