DEHYDRATION OF LIME JUICE AND SOME OBSERVATIONS ON THE HEAT STABILITY OF ASCOBIC ACID IN THE PRODUCT

BY

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DECLARATION

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DEDICATED TO

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ABSTRACT

Lime is one of the most common ingredients in many parts of Asia The flavour and acidic base make lime a favourite among various Asian cuisines. In Sri Lanka, the annual production 150 millions of limes and about 50% of this annual production is wasted due to marketing problems. So far, no proper system has been introduced to preserve lime The purpose of this study is to develop a powder from lime with an extended shelf life, so that this wastage could be minimized.

The process involves extracting the juice, mixing with cornstarch and drying in an oven at 60 ^{0}C for three hours to obtain a powder. Three samples were prepared by fortifying the powder with juice twice, thrice and five times respectively. Analysis was carried out in each sample to determine the moisture content, citric acid concentration and ascorbic acid concentration. A sensory evaluation test was carried out to find out the best sample out of the three.

Although limejuice is rich in ascorbic acid, it is destroyed by heating at higher temperatures for longer periods. Observations were made on the destruction of ascorbic acid at temperatures 70^{0}C, 80^{0}C and 90^{0}C and the activation energy for the reaction was calculated using the data obtained. These data were used in determining the amount of ascorbic acid destroyed during the production of lime powder (theoretically), which were compared with practical figures. The results were also used in predicting the concentration of ascorbic acid in the samples after a specific storage period.

From the findings of this study, it can be concluded that lime can be preserved in the form of a powder without the addition of any artificial preservatives When acid taste is considered, the powder with the highest concentration of juice constituents was preferred to the other two samples

The product will provide an answer to the huge wastage of lime plucked during the season. Results obtained in observing the heat stability of ascorbic acid could be used to maximize the retention of ascorbic acid in limejuice during thermal processing.

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CHAPTER 1

1.1 Introduction

Fruits are recommended to include in balanced diets for mainly two reasons; firstly they contain more vitamins and secondly they possess organic acids like citric acid and other fruit acids. Citrus family is a family that provides fruits, which have a higher nutritive value as well as a good flavour. Therefore, including citrus fruits in the diet will provide many benefits to the consumers.

The lime, *Citrus aurantifolia* is one of the citrus species and belong to the citrus family. Being originated in the tropical Asia, the crop is adoptable to a wide range of soils, terrain, planting and cultural practices. Lime is widely cultivated in India, Sri Lanka, Malaysia, West Indies and Florida.

Lime is a fruit that has a considerably higher Vitamin C content. Table 1.1 shows the amount of ascorbic acid present in some fruits.

Table 1.1 Ascorbic Acid Content of Some Fruits

Fruit	Ascorbic acid content (mg/100g)
Mango	16
Orange	30
Lime	63
Lemon	45
Papaya	57
Passion fruit	25
Pine apple	30
Wood apple	3

Source: Perera (1989), Tables of food composition for use in Sri Lanka

Ascorbic acid plays an important role in human nutrition. Helping to build connective tissues, improving the metabolism of amino acids and cholesterol and building up resistance to infections are among the important functions of vitamin C inside the human body.

On the other hand, the high amount of citric acid and volatile compounds present in lime impart a pleasing odour and a good flavour to the juice. Lime is an everyday ingredient of the foods in East Asia. Juice is added to curries, salads, *sambols* and many other dishes as a flavouring agent. Lime makes a delicious drink, which is rich in vitamin C.

The lime is a common fruit tree in Sri Lanka and is grown mainly in wet intermediate and dry zone areas. The tree is grown mainly as a plant in home gardens. Although the tree is grouped under ever bearing fruits, it is grown as an annual in Sri Lanka. A peak production exists in the months of February, March and April. Wholesale and retail prices of lime fluctuate throughout the year, depending on the local supply. The prices fall during the season and rise drastically during the off-season. In areas like Moneragala, tonnes of lime are wasted without being consumed. If a preservation method can be introduced to extend the shelf life of lime, this wastage could be prevented.

In this research, an attempt is made to extend the shelf life of lime by means of dehydration. Fresh limejuice was oven dried to obtain a powder and cornstarch was used as a drying aid. The product was targeted to be added to curries as a flavouring agent (mainly). But it can be used in various other dishes such as salads, soups and *sambols*. The dissolved powder can also be added to other fruits such as papaw and avocado, to enhance their flavour.

As the product can be added to the foods directly, it would save the time of housewives.

The product will provide a better way to utilize the loads of lime produced during the season. Therefore, the wastage during this time can be minimized. As the product has an increased shelf life, it can be made available during the off seasons, and will be a good substitute for lime at a remunerative price

Reduction of post harvest losses, minimum storage requirements and reduced storage and transportation costs are some other benefits that can be obtained.

1.2 Objectives

- To develop a powder from lime with an extended shelf life.
- To observe the heat stability of ascorbic acid of the product.

CHAPTER 2

REVIEW OF LITERATURE

2.1 Lime Cultivation

Limes can be classified into three species.

(1) The small fruited group – *Citrus aurantifolia* Swingle

(2) The large fruited group – *Citrus latifolia*

(3) The sweet lime – *Citrus limettoides* Tan

Table 2.1 describes the major characteristics of these three species.

Table 2.1
Lime Varieties

Variety	Other Names	Locations	Flavour	Colour
Small-fruited limes	Mexican Key	Brazil California China Egypt Florida India Iran Malaysia Mexico	Highly acidic, Distinct aroma	Greenish yellow
Large fruited limes	Bearss seedless Persian	Florida Australia Brazil California	Very acidic, True lime flavour	Greenish yellow
Sweet limes	Palestine	Florida California Egypt India	Insipidly Sweet Slightly bitter aftertaste	Straw yellow

From these three varieties, *Citrus aurantifolia* swingle (small-fruited variety) is the one that is common to Asia and also it is the most commercially important variety. It is grown in tropical and sub tropical areas around the world.

2.1.1 Origin and Distribution

Lime, *Citrus aurantifolia* is one of the citrus species and belongs to the citrus family. According to Rajput and Haribabu (1993), lime is native to India and Southeast China. Verheij and Coronal (1991) identifies India and the adjoining parts of Burma as the place of origin. Bonavia and Tanka believed that lime originated in the Malayan region. The lime is now widely distributed and naturalised in tropics and in warm sub tropical areas (Verheij and Coronal, 1991;Rajput and Haribabu, 1993). Outside these areas, the lime has never been successful since it is the tenderest of all citrus fruits.

2.1.2 Botany of the Plant

Lime is considered as a shrub, which reaches up to 1.5-3.5 m. According to Verheij and Coronal (1991), the tree is a small, irregularly branched, evergreen tree, which grows up to a height of 5m. The simple leaves are elliptic to oblong ovate in shape, with margins scalloped or crenulated. Petioles are narrowly winged and the leaves are aromatic when crushed (Verheij and Coronal, 1991; Ashton et al., 1997).

The twigs are armed with stiff, short, sharp spines. The tree bears small, white flowers. The fruit, a globose ovoid berry, is 3 to 6 cm in diameter and is green to greenish yellow in colour. The fruit is sour. Seeds are small, plump and smooth with white embryos (Verheij and Coronal, 1991, Rajput and Haribabu, 1993)

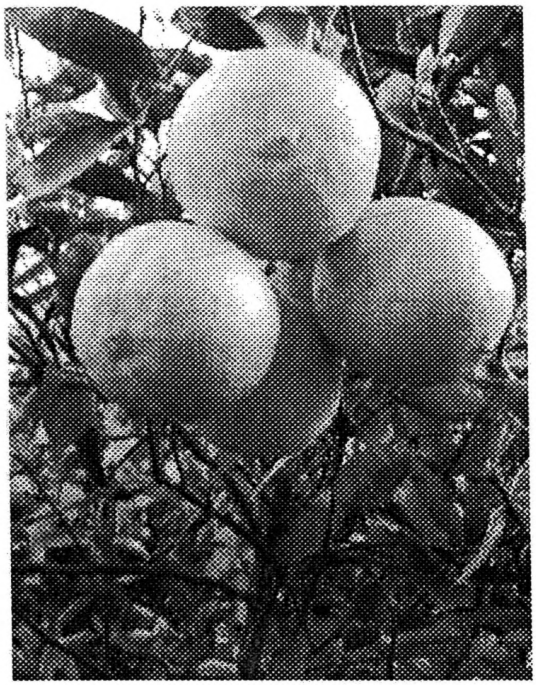


Figure 2.1 Lime tree with fruits

2.2 Environmental Requirements

2.2.1 Climatic Conditions

Climate is a deciding factor about success or failure of a given crop at a given place. Therefore, one should give utmost importance to it. According to Rajput and Haribabu (1993), acid lime, being tender to frost than other citrus fruits, require tropical climate for its successful performance. Verheij and Coronal (1991) further add that the tree is sensitive to cold but is quite drought resistant.

Situations, which are warm, moderately humid and free from strong winds and frost, are ideally suited for lime cultivation. It grows successfully even up to elevations of 900m above sea level, provided the humidity in the regions is not high. In the more humid areas where the rainfall is above 125cm, the lime become highly susceptible to citrus canker, which makes the trees unproductive and short-lived (Rajput and Haribabu, 1993).

2.2.2 Soil Requirements

Acid limes grow well on a wide variety of soils from heavy clay to very light soils. Heavy soils, under good drainage conditions yield bumper crops, while very light soils, on the contrary, due to lack of fertility and moisture, produce low yields. In shallow soils, the trees remain stunted and die in their prime due to malnutrition. Trees are particularly more sensitive to higher concentrations of salts and cannot stand waterlogged conditions for any length of time. The ideal soil thus seem to be a medium or light loam with a slightly heavier sub soil, with a depth of 2 to 3m and pH of 6.0 to 8.0 (Rajput and Haribabu, 1993).

2.3 Lime Production and International Trade

According to FAO statistics, the world produced 6 million t of limes and lemons in 1998, the figures showing a rising trend. The lime crop is by far, the smaller of the two, but since lemons are hardly grown in South East Asia, the following figures must refer to lime.

Cambodia	– 1000t
Laos	– 8000t
Malaysia	– 3000t
Thailand	– 1000t

Large-scale production for the international trade in juice and oil is mainly found in Central America, Dominica and Ghana (Verheij and Coronal, 1991).

2.4 Local Production of Lime

The lime is a common fruit tree in Sri Lanka and grows mainly in the wet intermediate and dry zone areas. The tree is grown mainly as a plant in home gardens

2.4.1 Major Producing Districts

About 28% of the total extent of lime is grown in the Moneragala district. Kurunegal (15%), Ampara (10%) and Ratnapura (10%) are the other major growing areas and accounts 35% of the total extent grown. About 14% of the total extent is grown in Badulla, Hambanthota and Anuradhapura districts and the balance (23%) is grown in other districts.

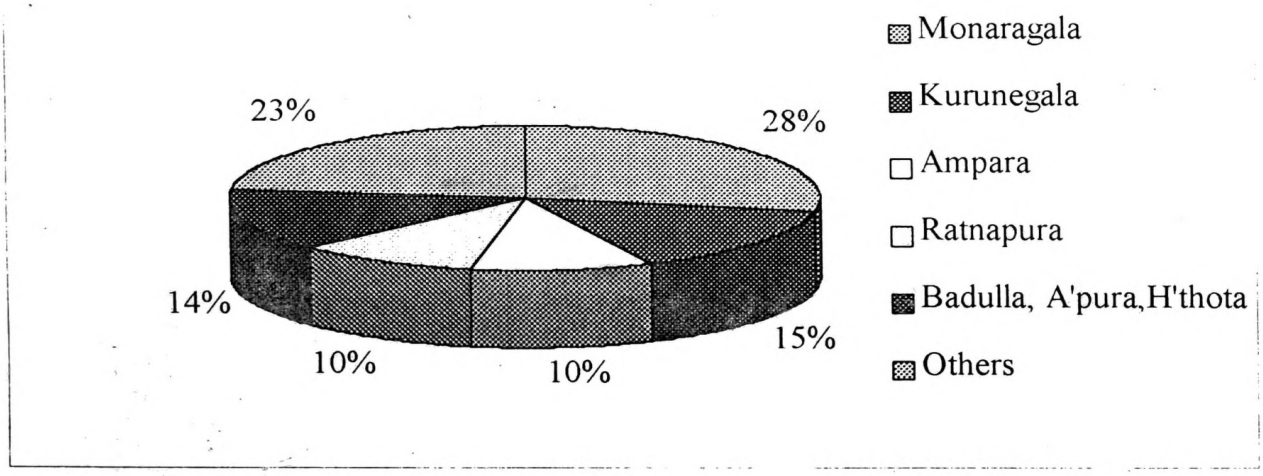


Figure 2.2 Distribution of Lime Among the Major Producing Districts

2.4.2 Extent Cultivated, Production and Yield

The total extent cultivated with lime is 6,999Ha (2001) and is largely concentrated in the intermediate and dry zone areas. In the past twenty years, (1981 – 2001) the total extent cultivated has increased from 3,908 Ha in 1981 to 6,999 Ha in 2001. Table 2.2 and figure 2.3 show the distribution of cultivated areas over the period

Total production has decreased considerably during the consecutive period (1981 – 2001) from 351,855 Ha in 1981 to 131,733 Ha in 2001. The decrease is very significant in 1991 but has once again increased gradually. Table 2.2 and the figure 2.4 show the changes of the total production during the period. The average yield per hectare has also decreased

by 76% during the same period. The notable fact is that the average production and yield has deceased instead of increasing with the extent grown. Table 2.2 and figure 2.5 illustrate the change of average yield over the past twenty years.

Table 2.2

Extent, Production and average yield of lime – Sri Lanka

YEAR	EXTENT (Hectares)	PRODUCTION (000 No)	AVERAGE YIELD (No/Ha)
1981	3,908	351,855	90,035
1982	3,883	251,601	64,796
1983	3,704	360,972	97,455
1984	3,953	208,011	52,621
1985	4,074	252,902	62,077
1986	5,212	474,501	90,953
1987	4,937	304,305	61,638
1988	5,041	117,238	23257
1989	5,194	119,832	23,071
1990	4,964	113,167	22,798
1991	5,154	103,742	20,129
1992	5,788	125,102	21,614
1993	6,773	155,911	23,019
1994	6,894	175,080	25,396
1995	6,860	172,009	25,074
1996	6,691	151,459	22,636
1997	6,854	136,225	19,875
1998	6,803	115,723	17,010
1999	6,955	117,663	16,918
2000	7,337	129,295	17,622
2001	6,999	131,733	18,822

Source: Department of Census and Statistics

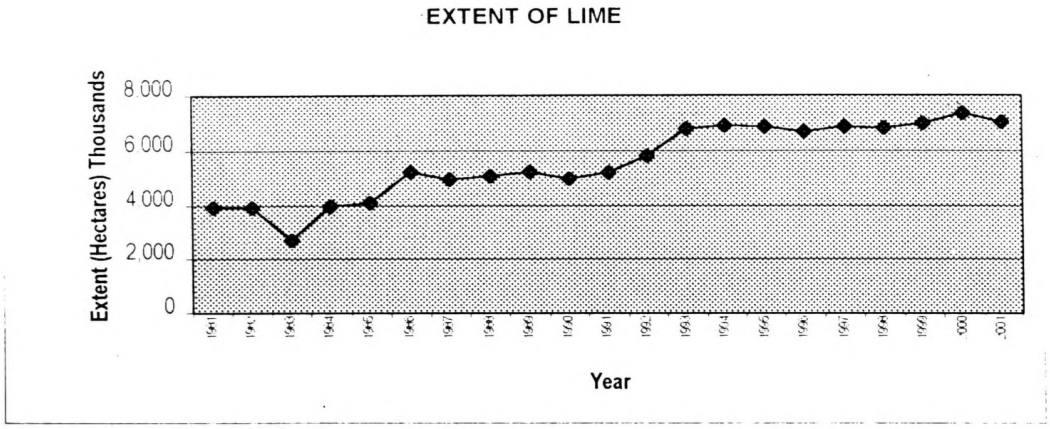


Figure 2.3 Extent of Lime

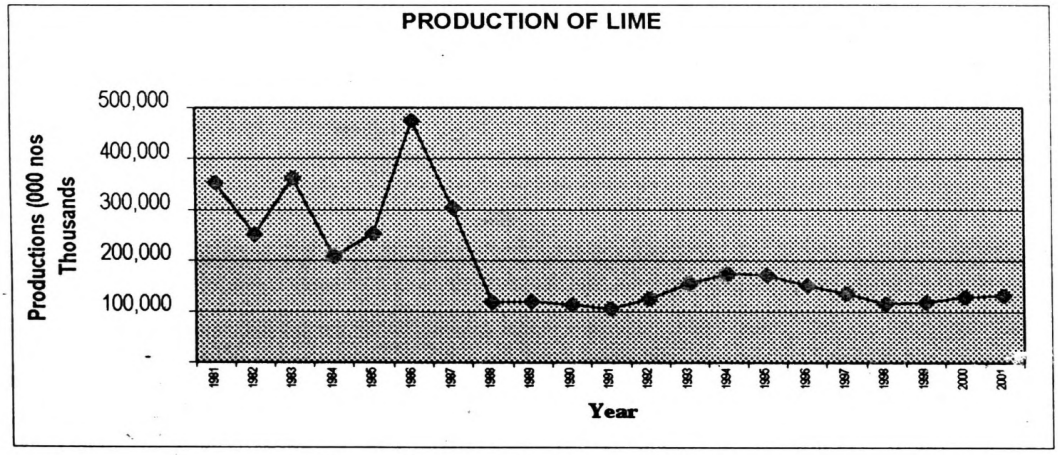


Figure 2.4 Production of Lime

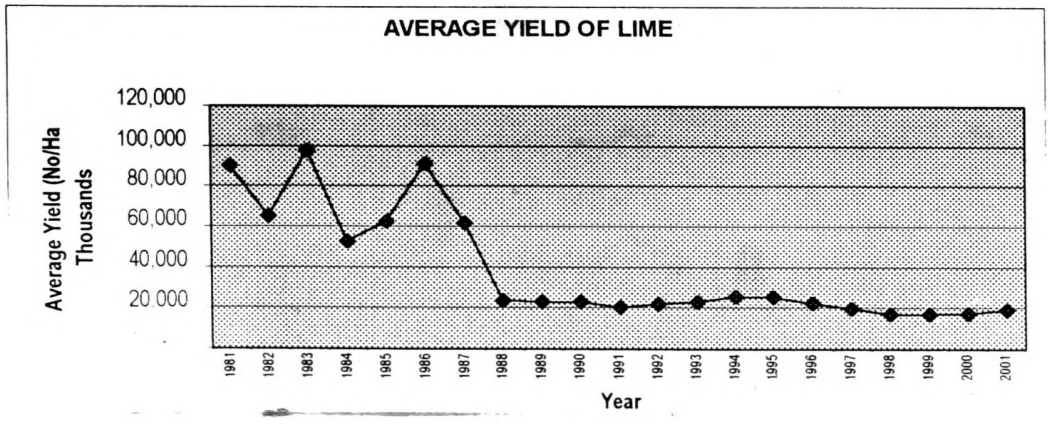


Figure 2.5 Average Yield of Lime

2.4.3 Marketing

Limes grown in rural areas are brought by local collectors and sold at weekly fair. Usually, area collectors collect the produce through local agents and send it to the terminal markets in Colombo and Kandy.

The average wholesale price has increased from Rs 20.59 in 1997 to Rs 36.79 in 2000,but dropped to Rs 22.49 in 2001. Wholesale prices fluctuate throughout the year depending on the local supply. Limes fetch a low price in April and May and high price in June and July. Table 2.3 shows the wholesale price of lime during the past five years.

Table 2.3

Average wholesale prices of lime at Pettah market

Month	1997	1998	1999	2000	2001
January	11.05	29.20	11.55	14.09	8.15
February	22.06	21.91	9.55	8.73	5.17
March	10.52	24.38	8.28	7.04	6.22
April	26.24	65.42	6.17	6.97	9.46
May	30.24	114.06	5.19	13.20	11.08
June	32.57	122.78	6.11	32.36	15.47
July	19.24	89.03	16.64	47.22	19.50
August	10.25	36.68	34.66	74.90	18.37
September	10.20	23.54	91.39	117.41	40.41
October	15.55	29.74	91.39	70.60	57.22
November	24.000	24.02	54.53	32.69	54.00
December	35.22	14.76	25.06	16.27	24.86
Average	20.59	49.63	30.04	36.79	22.49

Source. Marketing and Food Policy Division of HARTI

The monthly average retail price was Rs 73.08 per kg in 2001, which was a 44% increase over the prices in 1997. Retail prices fluctuate during the year and vary from Rs 34.50 in March to Rs 127.51 in November. Prices remained low during the months of February, March and April. Table 2.4 shows fluctuations of retail prices over the years.

Table 2.4

Monthly Average Retail Prices of Lime in Colombo and Suburbs

Month	1993	1994	1995	1996	1997
January	42.94	39.62	42.53	50.88	37.54
February	38.93	38.56	36.34	15.84	26.70
March	28.65	33.82	37.42	38.85	29.51
April	30.54	34.67	84.42	45.28	59.86
May	44.99	48.34	94.06	52.81	68.50
June	50.75	77.91	94.29	70.93	70.12
July	44.55	90.12	75.89	63.13	54.19
August	37.83	122.10	61.02	60.14	39.30
September	37.98	127.38	63.12	74.4.	37.34
October	40.53	119.54	89.48	99.16	42.64
November	40.41	92.41	111.94	101.20	54.69
December	40.71	62.19	76.84	65.04	74.27
Average	33.07	42.50	59.94	57.60	41.05

Source: Marketing and Food Policy Division of HARTI

2.5 The Structure of the Lime Fruit

The true lime is a small, thin-skinned fruit. The shape of the fruit is round to oblong. The apex of the fruit is rounded and slightly nippled. The fruit does not vary distinctly from the general structure of the citrus fruit.

2.5.1 Parts of Citrus Fruits

From outside to inside, the major parts of the citrus fruit are the peel or the rind (epidermis, flavedo, oil glands, albedo and vascular bundles); segments or sections (section wall, juice vesicles and seeds); and the core (vascular bundles and parenchyma tissue).

2.5.1.1 Peel

Epidermis – The epidermis of a citrus fruit consists of an epicuticular wax layer in platelets, the cutin matrix, the primary cell wall and the epidermal cell. The wax layer is deposited slowly during the development of the fruit. The wax and cutin are unevenly deposited and the wax coating is thicker immediately around the stem. The epidermal cells have few Chloroplasts.

Flavedo –The flavedo cell area is characterised by a green, yellow or orange colour, interspersed oil glands and no vascular bundle system. The flavedo consists of a few layers of small cells, slightly elongated tangentially to the surface and several layers of large cells that fill the areas between the oil glands. The cells have chloroplasts, which convert to chromoplasts as the fruit matures.

Oil glands – The adjacent cells of an oil gland are the sources of essential oils that accumulate in oil glands. The walls of these cells are very thin and fragile at maturity More oil glands per unit area are present on the stem end of the fruit.

Albedo – The albedo tissue is composed of loosely packed, many branched, tube like cells that form a continuous network with the greatest part of the tissue volume. The albedo cells do not possess chloroplasts or chromoplasts. Albedo is an important source of limonin. Albedo and flavedo are highest in pectin, compared to other parts of the fruit.

2.5.1.2 Segments (Sections)

The segments (carpels), approximately ten in citrus, are thought to have evolved by the folding of individual leaves with the lower "stomatal" surface outward and the "non-stomatal" surface inward.

Segment wall – The inner side of the segment wall (septum) has an epidermis with a cuticular surface. The septa are composed of six to eight compact cell layers next to each epidermis and two or three more loosely packed mesophyll cell layers at the centre between the adjacent segments. Toward the outside of the fruit, these mesophyll cells blend into the albedo. Septa may contain bitter compounds. Septa of the grapefruit have the highest amount of limonin of all anatomical parts.

Juice vesicles – Localised areas on the inner surface opposite the ovules (seeds) become merisematic and form protuberances. These continue to expand by cell division and enlargement, to form juice vesicle stalks and vesicle sacs. All the cells of the stalk are greatly elongated. Surface of the juice vesicle stalk and the sacs have a waxy cuticle.

Juice vesicle contents – The outer juice sac epidermis forms a strong covering, but the internal cells have very thin walls that are easily ruptured to release juice. Citrus juice consists of the liquid expressed from the cytoplasm and vacuoles of the internal cells within the vesicles, with the latter predominating. Most of the sugars, essentially all the titrable acids and some other soluble materials are dissolved in the vacuolar sap The cytoplasm of the juice cells contains proteins. lipids, the carotenoids, limonoids. some soluble solids, and phenolics. Experiments have shown that some amount of oil is present inside juice vesicles, but this amount is less than the amount in the peel.

2.5.1.3 Core

The central core of the fruit is composed of several vascular bundles, with loose, spongy mesophyll tissue surrounding the bundles. Some of the bundles end in the ovules (seeds), while others continue to the stylar end of the fruit. Core mesophyll is similar to that of the albedo and excessive extraction treatments will introduce undesirable compounds from this tissue to processing juice.

2.6 Composition

Wardowski et al. (1986) state that over 400 different constituents have been isolated from fruits of various species. The principal components of a fruit are carbohydrates, which include mainly sucrose, glucose and fructose. These constitute about three fourth of the total soluble solids (TSS). Organic acids, mainly citric and malic, with traces of others constitute somewhat less that 16% of the TSS and the free amino acids plus nitrogenous bases and glutathionine, about 6%. The remaining compounds include inorganic ions, vitamins, flavanoids and lipids.
(Wardowski et al.,1986)

The juice extract is about 41% of the fruit weight (Verheij and Coronal, 1991). According to Kimball (1999), the lime resembles lemon in composition and the composition of juice varies considerably with the variety of fruit and the location of the fruit growing area. The composition of lime is listed below in the table 2.5

Table 2.5

Composition of Lime Juice

Constituent	Number of samples	Content per 100g	
		Range	Average
Protein (total N *6.25)	11	0.3-0.7g	0.4g
Fat	-	0.00-0.11g	Trace
Soluble solids, total (^{0}Brix)	93	8.3-14.1g	10.0g
Acid, total as anhydrous citric	129	4.94-8.32g	11.2g
Sugar, total as invert	13	0.00-1.74g	5.97g
Non-reducing sugar	7	0.02-0.26g	0.72g
Ash, total	5	0.25-0.40g	0.14g
Calcium	2	4.50-10.4mg	0.35g
Phosphorous	2	9.3-11.2mg	7.0mg
Iron	2	0.19-0.92mg	10.0mg
Carotene	2	0.003-0.005mg	0.60mg
Thiamine	2	0.011-0.028mg	0.004mg
Riboflavin	2	0.011-0.018mg	0.020mg
Niacin	5	0.090-0.275mg	0.015mg
Ascorbic acid (Vitamin C)	13	23.6-32.7mg	0.19mg
pH	-	2.0	-
Food energy (Calories)		24-33	-

Source: Kimball, 1999

2.7 Nutritional value of lime

Macronutrients

Macronutrients mainly carbohydrates, proteins and lipids are supplied in the diet by serials, meat products and butter and oils respectively. But fruits and vegetables also contribute in this area significantly.

Carbohydrates

Most of the caloric or energy contribution of citrus products comes from the content of available carbohydrates. The major carbohydrates present are glucose, sucrose and fructose. The sugars are distributed with the ratio of sucrose, glucose and fructose of 2:1:1.

Organic acids – the organic acids (excluding amino acids) are usually considered with the carbohydrates, since they are significant as caloric sources. Citric and malic acid account for over 80% of the acid present. Oxalic, succinic, malonic and similar acids have also been identified. These compounds are easily metabolised by the body, as they are parts of the major metabolic pathways.

Proteins

The amount of protein in the edible portion of citrus is not too high, 0.4g in the average lime (per 100g edible potion). Like other plant proteins, this is scored lower than ideal as source of the essential amino acids required for building human proteins.

Lipids

The essential oils and lipids of citrus fruits are present in only small amounts and any nutritional benefits must be minimal. In a diet in which the levels of the various lipids are too high, the low level found in citrus must be considered a plus

2.8 Processing of Lime

The processing of lime includes many products, limejuice, canned fruits etc. The most common products of citrus industry are given below.

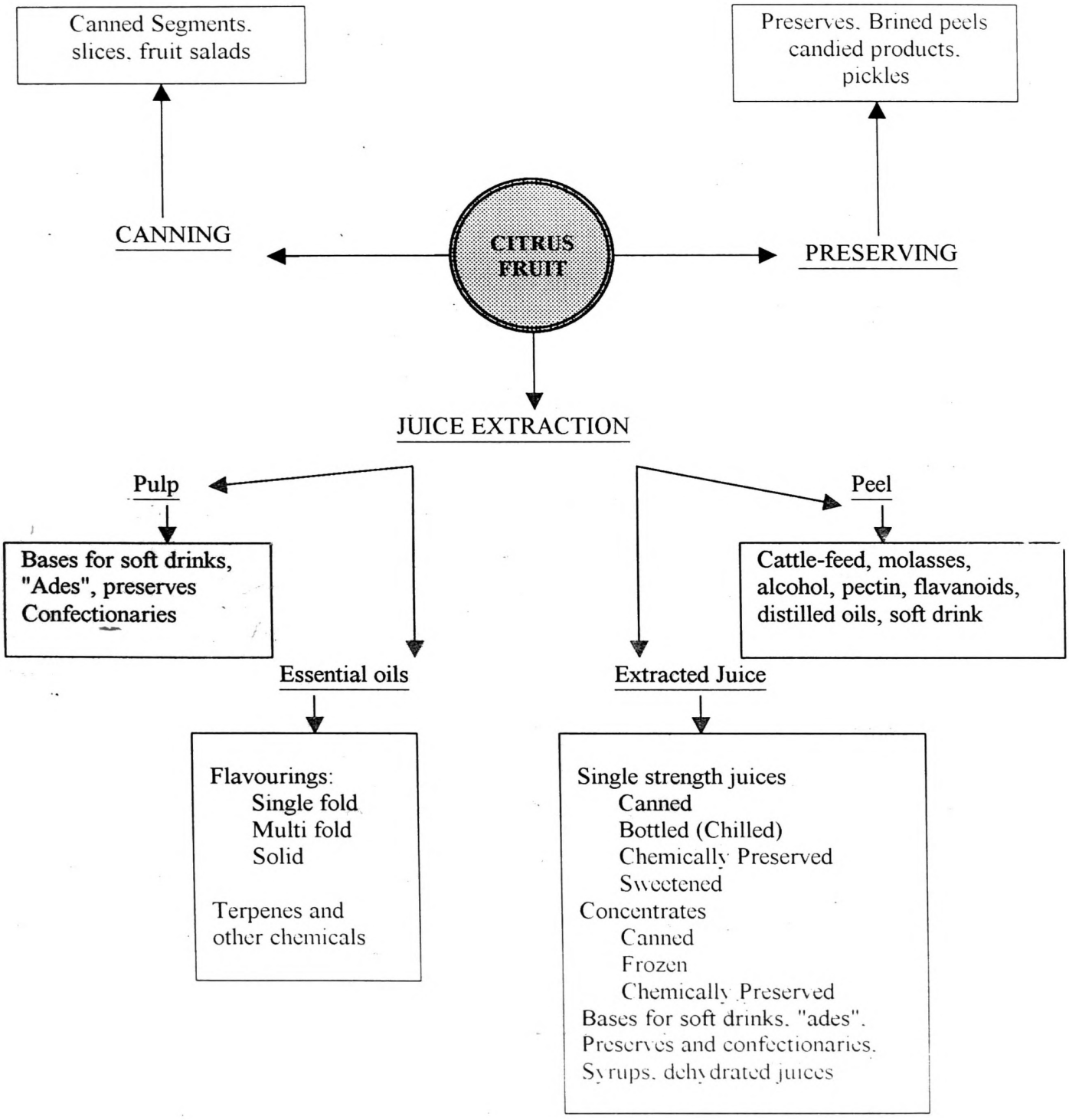


Figure 2.6 The Most Common Products of Citrus Industry

2.8.1 Powdered Citrus Juice

The dehydration or drying of citrus juice to a powdered form has been of considerable interest for many years because of the numerous advantages of such a product. Unfortunately, the technological difficulties encountered are tremendous.

Since citrus juices consist largely of water, it is not economical to attempt to dry them directly to a powder. A more economical procedure is first to concentrate the juice in a vacuum evaporator (United States Department of Agriculture [USDOA], 1967).

Most early processes were based on vacuum drying. The final stages of drying were extremely slow and the product turned to a sticky mass under this technology. A significant break through came with the foam-mat drying technology (United Nations Industrial Development Organization, 1989).

USDOA (1967) states that citrus juice concentrate has been converted to powdered form by spray-, drum-and freeze-drying. It further adds that the dehydration was accomplished by the adding of drying aids to promote the rate of drying, to counteract the hygroscopicity of the product and to reduce the tendency of the powder to cake during the storage. Carboxymethyl cellulose, glyceryl mono stearate and corn syrup solids are some of the drying aids used. A powder is produced by mixing the juice with corn syrup and spray drying, which contains about 20% of lemon juice solids and 80% of corn syrup solids. Some lemon oil and crystalline ascorbic acid are added to the dry powder and the product is used as a flavour base for the confectionary and soft drinks trades.

2.8.2 Dry Packaged Products

Wardowski et al. (1986) describe two methods involved in the production of citrus crystals. In one method, citrus concentrate is applied with a foaming agent, to a moving stainless steel belt enclosed in a large horizontal cylinder under high vacuum. The dehydrated citrus crystals are removed from the belt.

In the other method, foam-mat drying process is used. A liquid citrus concentrate is mixed with a foaming agent and whipped into thick foam. The moisture is removed by passing hot air over and through it, while it is spread out in a thin layer on a continuous belt.

In both processes, crystals are removed from the belt when the moisture content falls below 2.5 – 3.0% and are packaged under controlled humidity of 10%.

2.9 Ascorbic Acid (Vitamin C)

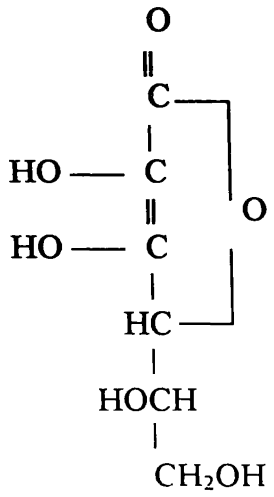
Ascorbic acid is known for centuries as antiscorbutic vitamin, as it prevents the disease scurvy. According to West et al. (1974), the best sources of vitamin C are citrus fruits, berries, melons, tomatoes and leafy green vegetables.

Chemistry

This vitamin is a white crystalline substance with a very acidic taste. It has a structural resemblance to hexoses. It is a strong reducing substance on account of its enediol structure. The vitamin is stable in the solid form and in acidic solutions, but is rapidly destroyed in alkaline solutions (West et al., 1974).

Ascorbic acid is soluble in water and is insoluble in typical fat solvents such as ether, benzene and chloroform.

Structure

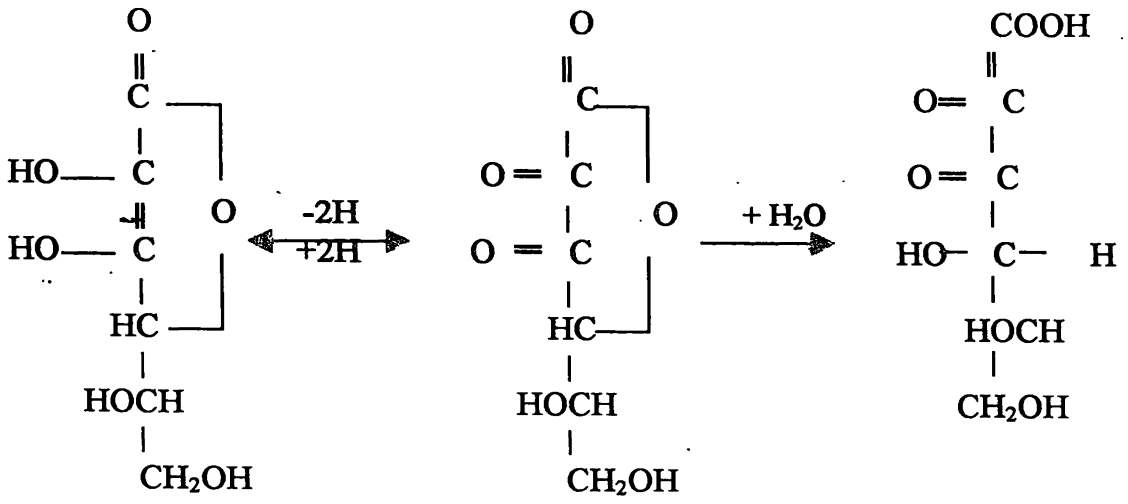


Antia (1966) states that carbon atoms 4 and 5 are asymmetric, therefore the compound is optically active. He further states that Levo form is an active antiscorbutic substance whereas the Dextro form is not.

As can be seen in the structural formula, ascorbic acid does not contain a free carboxyl group, but is a lactone forming a five membered ring of the furan type. The acidic character of vitamin C is due to the presence of enol configuration (Braverman, 1949).

Oxidation

Ascorbic acid is a reducing agent with a hydrogen potential of +0.08V, making it capable of reducing such compounds as molecular oxygen, nitrate and cytochromes a and c. Oxidation of ascorbic acid yields dehydroascorbic acid. While the conversion of ascorbic acid to dehydroascorbic acid is a reversible change, the further hydration of this compound to l-diketogulonic acid is irreversible.



Ascorbic acid Dehydroascorbic acid L-Diketogulonic acid

2.9.1 Nutrient Losses Encountered With Processing

Hulme (1974) describes reduction in vitamin C potency under two categories.

(1) Disappearance of vitamin C prior to heat processing.
(2) Changes in vitamin C during heat processing.

2.9.1.1 Disappearance of Vitamin C Prior to Heat Processing

(i) Peeling losses

The peel of the citrus fruits contains a significant amount of vitamin C. In an analysis of several varieties of Italian oranges, the following values have been reported by Hulme (1974).

Flavedo (yellow peeling): 175 – 292 mg/100g
Albedo (white peeling) : 86 – 194 mg/100g
Endocarp : 44 - 74 mg/100g

A similar distribution has been found in limes, oranges and grapefruits from Florida. Generally, the processed citrus products contain a very little amount of the flavedo or albedo portions of the fruit. This is undesirable in the nutritional point of view, as the concentration gradient of ascorbic acid decline from flavedo towards the endocarp.

The effect of lye peeling on ascorbic acid retention has not received much attention. As vitamin C is extremely unstable to heat under alkaline solutions, the lye treatment used should be only sufficient to remove the outer carpellary membrane of the fruit.

(ii) Enzymic Changes

Ascorbic acid is oxidised when exposed to air. The oxidation process is catalysed by oxidases, which are present in the cells of foodstuffs. The rate of oxidation increases rapidly with the temperature and with the presence of an alkali (Ramsden, 1995).

Enzymes like ascorbic acid oxidase, cytochrome oxidase and peroxidase can lead to oxidation of vitamin C. In intact fruits, these enzyme systems are balanced due to reductases. However, when the enzyme balance is disturbed by cellular disruption, the oxidases can destroy all of the vitamin C unless they are inhibited (Hulme, 1974). When fruits are chopped, grated or bruised, the oxidases get liberated and the destruction of vitamin C will proceed.

(iii) Non-enzymatic Changes

Various substances catalyse the oxidation of ascorbic acid *in vitro*. Hulme (1974) states that traces of copper will catalyse the oxidation of ascorbic acid and the effect will be enhanced by iron. This non-enzymatic reaction gives dehydroascorbic acid. Although this retains full vitamin C activity, it is more thermo labile and would be easily destroyed during heat processing. Thus, keeping juice in stainless steel or glass-lined containers is essential in vitamin C retention.

2.9.1.2 Changes in Vitamin C During Heat Processing

(a) Aerobic Destruction

Oxidative destruction can take place during heat processing. This is mainly a non-enzymic process as the oxidase systems are destroyed by heat under acidic conditions.

(i) Type of the Container

Type of the container has an important influence on the retention of Vitamin C potency. Loss of Vitamin C in enamel-lined cans is greater than in plain tin cans. In the latter case, a part of the residual oxygen reacts with tin. Hence, the amount of residual oxygen left for the reaction with vitamin C is less. Glass-packed juice loses about 10% of its content after four months at refrigerated temperatures whereas the plastic and fibre-board packed products can lose ~ 20% in 3 – 4 weeks. In contrast to glass containers that are hermetically sealed, plastic bottles and fibreboard cartons are permeable to oxygen, thus they may lower the vitamin retention (Charalambous, 1986).

(ii) Headspace Oxygen

Oxygen in the headspace of the can has a detrimental effect on the ascorbic acid concentration. A small headspace and a low oxygen tension will have a beneficial effect on vitamin C retention. Therefore, de-aeration is essential for the stabilisation of vitamin C (Hulme, 1974).

(iii) pH in Fruit Juice

Ascorbic acid readily destroys on neutral and alkaline conditions, but in citrus products where the highest pH is about 4.0, the vitamin is relatively stable (Hulme, 1974). The greater stability of ascorbic acid in an acid solution is associated with decreased trend to hydrolyse the lactone ring at reduced pH. In alkaline solutions, the hydrolysis is fairly rapid (West et al.,1974).

(b) Anaerobic Destruction

After a period of relatively rapid aerobic degradation of vitamin C, the anaerobic destruction begins. The rate of degradation hardly varies from pH 1 – 11. However, a small but maximum occurs at pH range 3 – 4. The reaction is accelerated by fructose and sucrose. Lead and Aluminium are the most powerful catalysts of anaerobic degradation, but are effective only at concentrations that are far in excess of those found in fruits (Hulme, 1974).

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Sample Preparation

Materials

1. Fresh limes
2. Cornstarch
3. Knife
4. Cutting board
5. Muslin cloth
6. Analytical balance
7. Measuring cylinders (250ml)
8. Thermostatic hot air oven (maintained at 60 °C)

Method

Fresh limes were obtained from the local market in bulk. Damaged and spoiled ones were removed and the limes were washed thoroughly in cold water to remove dirt, dust and insects if any.

Limes were cut into slices and the juice was extracted by squeezing the slices. Juice was filtered using a muslin cloth to remove seeds and juice vesicles.

Cornstarch was added to the juice in the ratio of 130g cornstarch/100ml juice and a paste was obtained. The paste was transferred to a muslin cloth and was oven dried at 60 °C for three hours and a dry powder was obtained. Fresh juice was added to the powder again in a ratio of 98ml juice/100g of powder and the resultant paste was oven dried. The process was repeated and three samples were obtained.

S_1 – Original powder fortified twice with limejuice

S_2 – Original powder fortified thrice with limejuice

S_3 – Original powder fortified five times with limejuice

Figure 3.1 The Oven Used in the Drying Process

3.2 Product Evaluation

3.2.1 Determination of the Moisture Content of Lime Powder

The final moisture contents of the three samples were determined by the oven drying method.

Apparatus

1. Moisture dishes made of porcelain
2. Oven maintained at 105 °C
3. Analytical balance
4. Desiccator

Method

A small amount (about 5g) of each sample was weighed to the nearest milligram, in moisture dishes previously dried and weighed. Dishes were placed in the oven for four hours and were removed, cooled in the desiccator and were weighed. The drying, cooling and weighing process was repeated at 30 minutes intervals until the difference between the two consecutive weighing did not exceed one milligram and the loss of weight for each sample was recorded.

$$\text{Moisture\%} = \frac{m_2 - m_3}{m_1 - m_3} \times 100$$

m_1 – Initial weight of the sample with the dish

m_2 – Final weight of the sample with the dish

m_3 – Weight of the empty dish

3.2.2 Determination of the Citric Acid Content

Reagents

1. Distilled water
2. 0.10N NaOH – prepared by dissolving 4g of NaOH pellets in 100 ml of distilled water.
3. Phenolphthalein indicator

Apparatus

1. Analytical balance
2. Petri dishes
3. Conical flasks
4. Burette
5. Pipettes (5ml, 10ml)
6. Funnel
7. Measuring cylinders (10ml, 100ml)
8. Beakers
9. Volumetric flasks (50ml, 100ml)

Method

For fresh juice :

10ml of limejuice were diluted to 50ml using distilled water. 10ml of the diluted solution was taken to a conical flask and was titrated with 0.10 N NaOH in the presence of 1% Phenolphthalein. Titre value was recorded.

For samples :

A known weight of each of the three samples was dissolved in hot distilled water and cooled and the volume was made up to 100ml using recently boiled distilled water. Aliquots of 5ml of each sample were titrated with 0.10 NaOH, using a few drops of 1% Phenolphthalein solution as indicator. Titre values were recorded.

$$\% \text{ Titrable Acidity} = \frac{\text{Titre} \times \text{Normality of Alkali} \times \text{volume made up} \times \text{Eq. Weight of acid} \times 100}{\text{Volume of sample taken for estimation} \times \text{Weight or volume of the sample taken} \times 1000}$$

3.2.3 Determination of the Ascorbic Acid content of the Product

Ascorbic acid content of the fresh juice and the final samples were determined by 2,6 – dichlorophenolindophenol visual titration method.

Reagents

1. 5% Trichloro Acetic acid (TCA) – Prepared by dissolving 5g of TCA in 100ml of distilled water.
2. Standard Ascorbic Acid Solution – 10mg of Ascorbic acid was dissolved in 50ml of 5% TCA.
3. 2,6 – dichlorophenolindophenol (2,6-DCIP) – 50mg of dye was dissolved in 150ml of distilled water containing 0.2mg of Sodium bicarbonate and diluted into 200ml with distilled water.

Apparatus

1. Analytical balance
2. Petri dishes
3. Conical flasks
4. Burette
5. Pipettes (5ml)
6. Funnel
7. Measuring cylinders (100ml, 250ml)
8. Beakers (50ml, 100ml)
9. Volumetric flasks

Method

Standardization of the dye

5ml of standard Ascorbic acid was added to 5ml of 5%TCA and was titrated against the dye in a burette till the faint pink colour persisted for 15 seconds. The ascorbic acid equivalent for 1ml dye solution was calculated.

Dye factor = 1/Titre

Preparation of the Vitamin C extract

For fresh juice:

Juice was extracted from about five whole fruits and the volume was noted. About 30ml of 5% TCA was added to the juice and the volume was made up to 250ml using distilled water.

For powdered lime samples:

About 10g of each sample was weighed accurately and was dissolved in 10ml of hot distilled water and the volume was made up to 50ml using 5% TCA.

Estimation of Vitamin C

5ml of each prepared sample was taken to a conical flask and 5ml of 5% TCA was added. These were titrated with the standardized dye. (The end point is colourless to pink).

Titrations were carried out for the three samples of lime powder and for fresh juice and the ascorbic acid content of each sample was calculated and was expressed as mg of ascorbic acid/100g or ml of sample.

$$\text{mg of Ascorbic acid/100g or ml} = \frac{\text{Titre x Dye factor x Volume made up x 100}}{\text{Aliquot of extract taken for estimation} \times \text{Weight or volume of the sample taken for estimation}}$$

3.2.4 Sensory Evaluation

Preparation of the Samples

Two tablespoons each of Sample 1, sample 2 and sample 3 were dissolved in 5ml, 7ml and 10ml of water respectively. A coconut *sambol* was prepared and was divided into three equal portions. The prepared samples of juice were added to the three portions and the portions were labelled accordingly.

Method

A hedonic test was conducted to determine whether a significant difference exists between the three samples of lime powder, using a five-point category scale. A group of 30 untrained panellists were selected and the testing method and the procedure was explained.

Three samples were presented in three identical containers to the panellists. The sample containers were coded with three digit random numbers.

The code numbers were:

356 – Sample treated with the powder S_1

802 – Sample treated with the powder S_2

475 – Sample treated with the powder S_3

All the samples were simultaneously presented to each panellist in a balanced order with a ballet paper and re-tasting of the samples were allowed. Panellists were instructed to evaluate the coded samples for degree of liking. They evaluated by checking a category on the scale that ranged from like extremely to dislike extremely.

For data analysis, the categories were converted to numerical scores ranging from 1 to 5 where 1 represented dislike extremely and 5 represented like extremely. The numerical scores for three samples were tabulated and analysed by Kruskal-Wallis test.

3.3 Observations on the Heat Stability of Ascorbic Acid

Reagents

1. 5% Trichloro Acetic acid (TCA) – Prepared by dissolving 5g of TCA in 100ml of distilled water.
2. Standard Ascorbic Acid Solution – 10mg of Ascorbic acid was dissolved in 50ml of 5% TCA.
3. 2,6 – dichlorophenolindophenol (2,6-DCIP) – 50mg of dye was dissolved in 150ml of distilled water containing 0.2mg of Sodium bicarbonate and diluted into 200ml with distilled water.

Apparatus

1. Analytical balance
2. Petri dishes
3. Burette
4. Pipettes (5ml, 10ml)
5. Funnel
6. Measuring cylinders (100ml, 250ml)
7. Beakers (100ml, 250ml, 500ml)
8. Volumetric flasks
9. Water bath
10. Thermometer
11. Stopwatch

Method

Sample Preparation

About 120mg of ascorbic acid was dissolved in distilled water and was diluted to 300ml using distilled water. (About 30 ml of 5% TCA was added to the solution to keep the stability of ascorbic acid). A 10ml sample was withdrawn prior to heating, using a pipette and 5ml of 5% TCA was added to the solution.

The rest of the solution was heated for three hours in a water bath at 70^0C. Samples of 10ml from the heating solution were withdrawn at 30-minute intervals and 5ml of 5% TCA was added to each sample.

This process was repeated at 80^0C and 90^0C and samples (10ml) were taken at 30-minute intervals, during heating.

Estimation of Vitamin C

Each prepared sample was transferred to a conical flask and was titrated with standardized 2,6 – DCIP dye solution and the amount of ascorbic acid (mg/100ml) of sample was calculated for each sample, as described in Section 3.2.3.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Product Evaluation

4.1.1 Yield of the Product

Table 4.1

Yield of the Product

Sample	Powder (g)	Fresh fruits (kg)	Juice (ml)
S_1	100	0.719	272.92
S_2	100	0.957	370.92
S_3	100	1.433	566.92

Figure 4.1 Sample 1

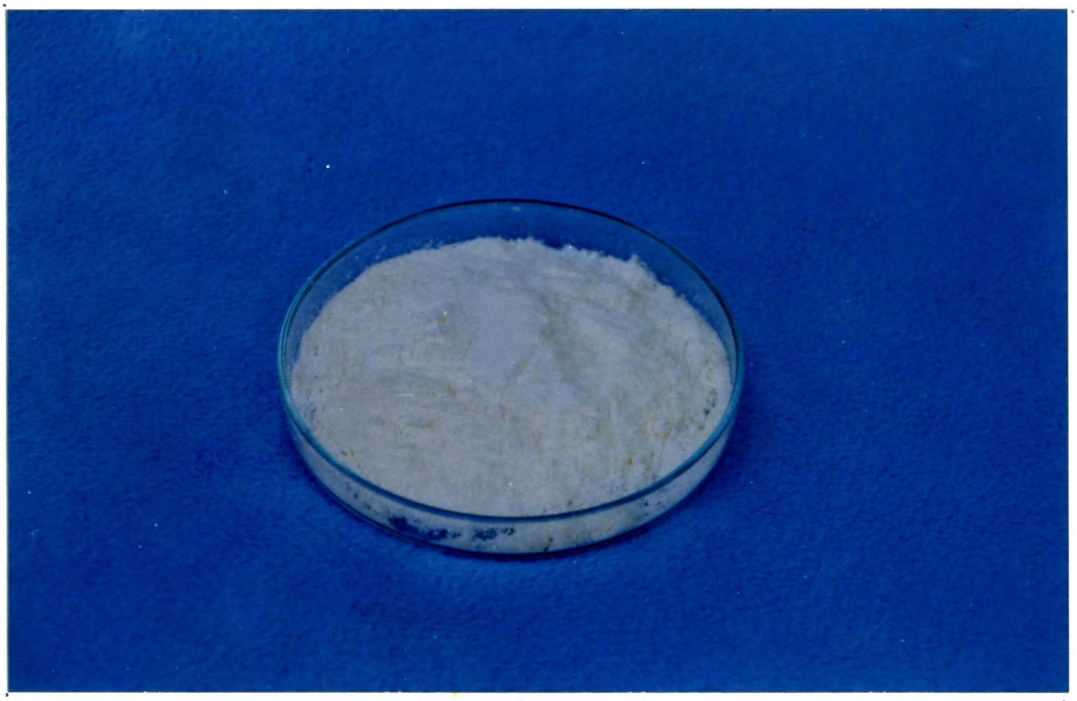


Figure 4.2 Sample 2

Figure 4.3 Sample 3

4.1.2 Moisture Content of the Product

Table 4.2 shows the moisture content of the three samples

Table 4.2

Moisture Content of the Samples

Sample	Initial weight (g)	Final weight (g)	Weight loss	% Moisture
S_1	11.363	11.066	0.297	2.614%
S_2	7.428	7.227	0.201	2.705%
S_3	10.235	9.969	0.266	2.599%

The moisture content of foods varies greatly according to the type of food and water is a major constituent of most food products. Expected moisture content of a food can affect the choice of the method of measurement and accuracy of determining the moisture content while this also depends on proper techniques and personal experience.

Oven drying method was used to determine the moisture content of the samples. The values obtained were highly depended on the type of oven used, conditions in the oven and the time and temperature of drying.

The moisture content of the three samples was in the range of 2 – 3%. The literature states that the product should be dried until the moisture content falls below 2.5 – 3.0% The packaging material used has an influence on the moisture content of the final product upon prolonged storage. Therefore, to maintain the moisture content constant during storage a packaging material, which provides a strong barrier to moisture, must be selected.

4.1.3 Citric Acid Concentration of the samples

Citric acid is the main compound that is responsible for the acidic flavour of limejuice The citric acid concentration of fresh juice and the three samples are listed in the table 4.3.

Table 4.3

Citric Acid Concentration of the Samples

Sample	Titre (ml)	Sample weight or volume	Normality of alkali	Volume made up (ml)	Volume of sample taken (ml)	% Citric Acid
Fresh juice	18.65	10ml	0.10	50	10.0	5.97%
S_1	2.94	3.140g	0.10	100	5.0	11.98%
S_2	4.01	3.191g	0.10	100	5.0	16.08%
S_3	5.53	3.103g	0.10	100	5.0	22.81%

Citric acid is not thermo labile and the boiling point of the acid is in the range of 120^0C Therefore the acid is not destroyed during the drying process and is concentrated in the powder, with the evaporation of water at each stage of drying. Accordingly, the highest concentration is recorded in the powder that was fortified five times.

As citric acid is concentrated at each stage of drying, the concentration of each sample can be calculated using the citric acid concentration of fresh juice, and the theoretical values and practical values should parallel. This is shown in the table 4.4 and in the figure 4.4.

Table 4.4

Practical and Theoretical Values Obtained for the Citric Acid Concentration of the Samples

Sample	% Citric acid (Theoretical)	%Citric acid (Practical)
S_1	12.49%	11.98%
S_2	17.03%	16.08%
S_3	26.03%	22.81%

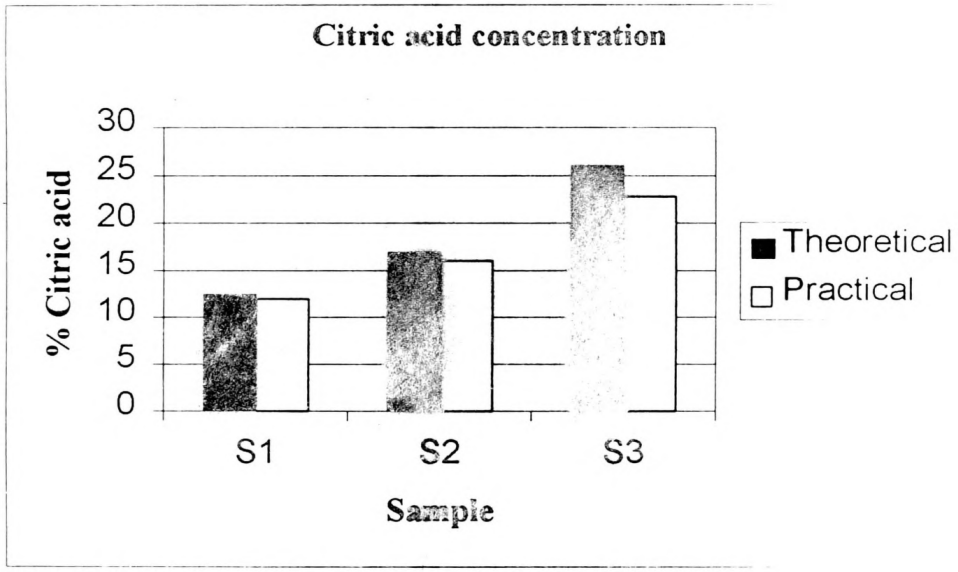


Figure 4.4 Practical and Theoretical Values Obtained for the Citric Acid Concentration of the Samples

4.1.4 Ascorbic Acid Concentration of the samples

Table 4.5

Ascorbic Acid Concentration of the Samples

Sample	Sample weight/volume	Volume of dye (ml)	Dye factor	Ascorbic acid (mg)
Fresh juice	86.5ml	4.190	0.1754	42.49/100ml
S_1	10.075g	5.745	0.1754	100.01/100g
S_2	9.131g	6.770	0.1754	130.05/100g
S_3	6.325g	6.500	0.1754	180.25/100g

4.1.5 Results of the Sensory Evaluation

P = 0.000

Table 4.6

Average Ranks for the Three Samples

Sample	Average rank
S_1	23.3
S_2	39.1
S_3	74.1

The P value for the test is less than 0.05. This implies that a significant difference exists among the three samples. When rank values are considered, S_3 has the highest rank value

Therefore, the best sample out of the three is S_3. The results obtained for the test is included in appendix 2.

4.2 Observations on the Heat Stability of Ascorbic Acid

Table 4.7, 4.8 and 4.9 show the ascorbic acid concentrations at 70 ^{0}C, 80 ^{0}C and 90 ^{0}C respectively.

Table 4.7

Ascorbic Acid Concentration at 70^{0}C

Time of Heating (min)	Titre (ml)	Concentration (C-mg/5ml)	C/C_0	ln (C/C_0)
0	3.76	0.8530	1.0000	0.00000
30	3.82	0.8498	0.9963	-0.00365
60	3.50	0.7780	0.9116	-0.09250
90	3.40	0.7550	0.8856	-0.12150
120	3.26	0.7250	0.8504	-0.16200
150	3.14	0.6970	0.8167	-0.20250

Table 4.8

Ascorbic Acid Concentration at 80^0C

Time of Heating (min)	Titre (ml)	Concentration (C-mg/5ml)	C/C_0	ln (C/C_0)
0	5.34	1.125	1.0000	0.0000
30	4.78	1.006	0.8942	-0.1118
60	4.21	0.887	0.7885	-0.2376
90	4.14	0.871	0.7738	-0.2564
120	3.32	0.699	0.6218	-0.4752
150	2.95	0.621	0.5521	-0.5940

Table 4.9

Ascorbic Acid Concentration at 90^0C

Time of Heating (min)	Titre (ml)	Concentration (C-mg/5ml)	C/C_0	ln (C/C_0)
0	5.56	5.835	1.0000	0.0000
30	4.55	4.776	0.8186	-0.2001
60	2.84	2.989	0.5122	-0.6691
90	2.28	2.645	0.4102	-0.8912
120	1.44	1.301	0.2592	-1.3501
150	1.03	1.076	0.1845	-1.6901

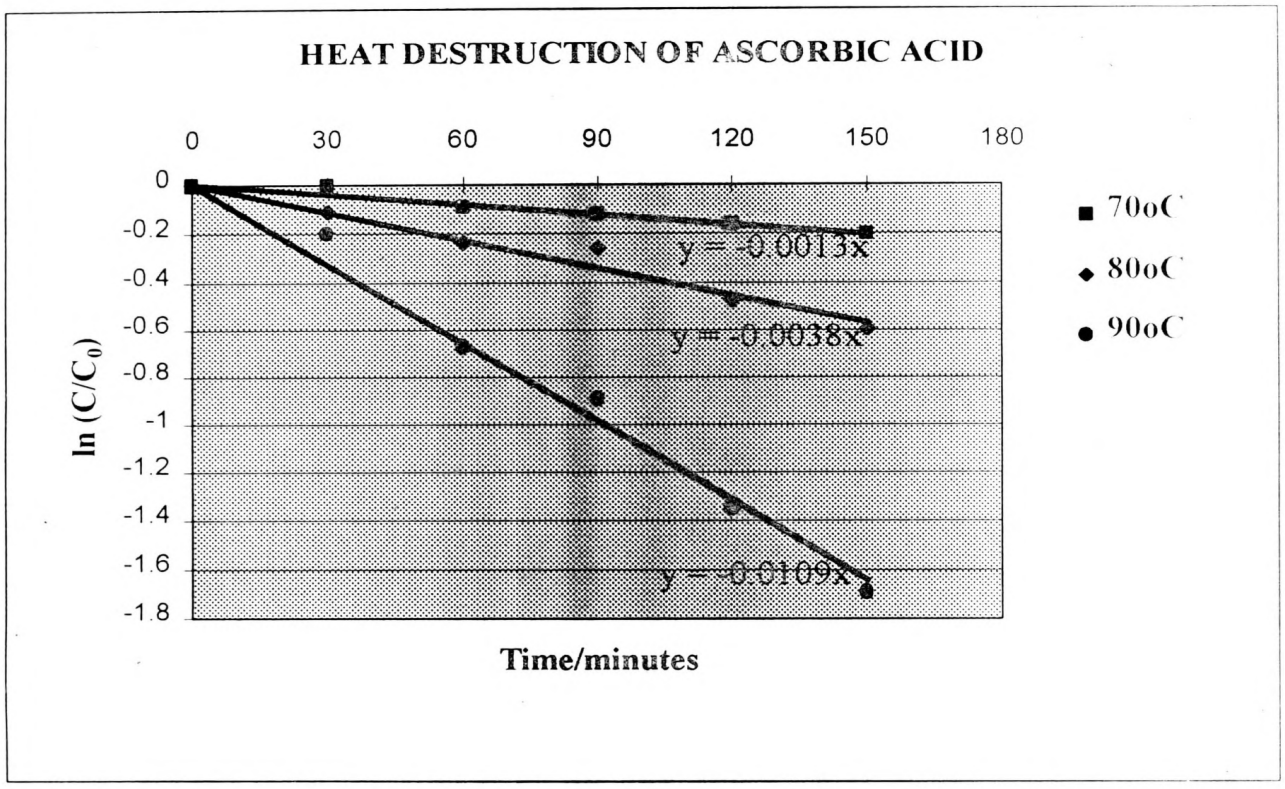


Figure 4.5 Heat Destruction of Ascorbic Acid

1/T (K)	ln k
0.00275	-4.52
0.00283	-5.57
0.00291	-6.64

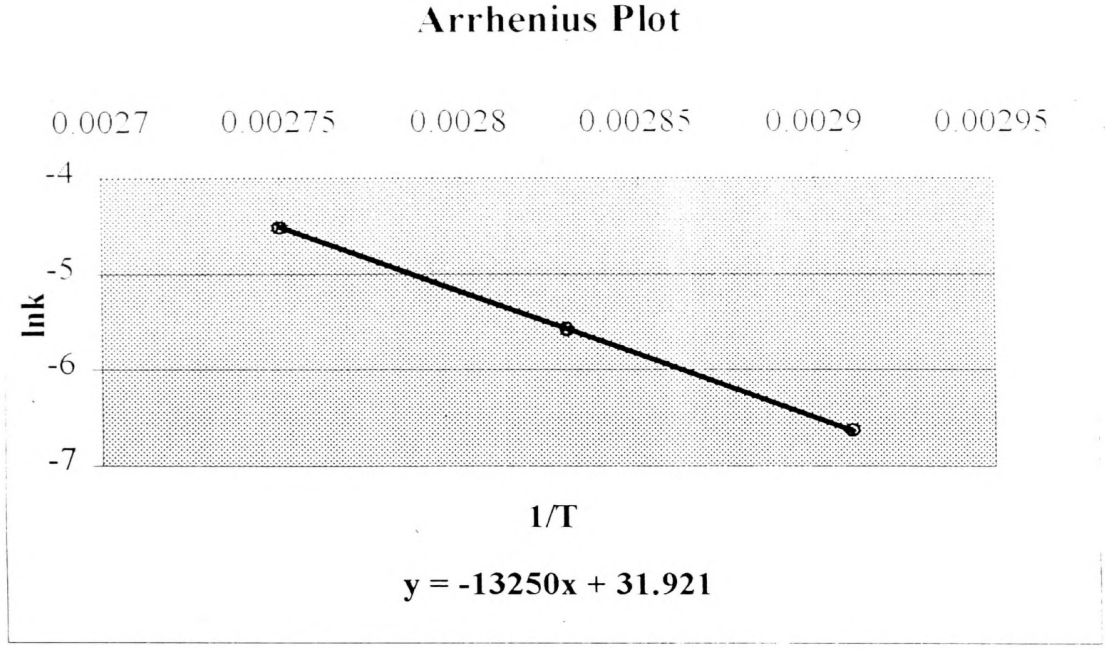


Figure 4.6 Arrhenius Plot

Arrhenius equation:

$$\ln k = \ln A - (E_a/R) / T$$

k = Rate constant

A = Arrhenius factor

E_a = Activation energy

R = Gas constant

T = Absolute temperature

The gradient of the graph = -13250

Activation energy of the reaction = 26328 Cal/mol

Using the activation energy of the reaction, the amount of ascorbic acid destroyed during the drying process can be calculated. The theoretical values and the practical values for the destruction reaction are shown in the table 4.10 and the figure 4.7

Table 4.10

The Amount Of Ascorbic Acid Destroyed During The Drying Process

Sample	Amount of ascorbic acid destroyed (mg) (Theoretical)	Amount of ascorbic acid destroyed (mg) (Practical)
S_1	13.866	15.955
S_2	23.750	27.550
S_3	49.083	60.630

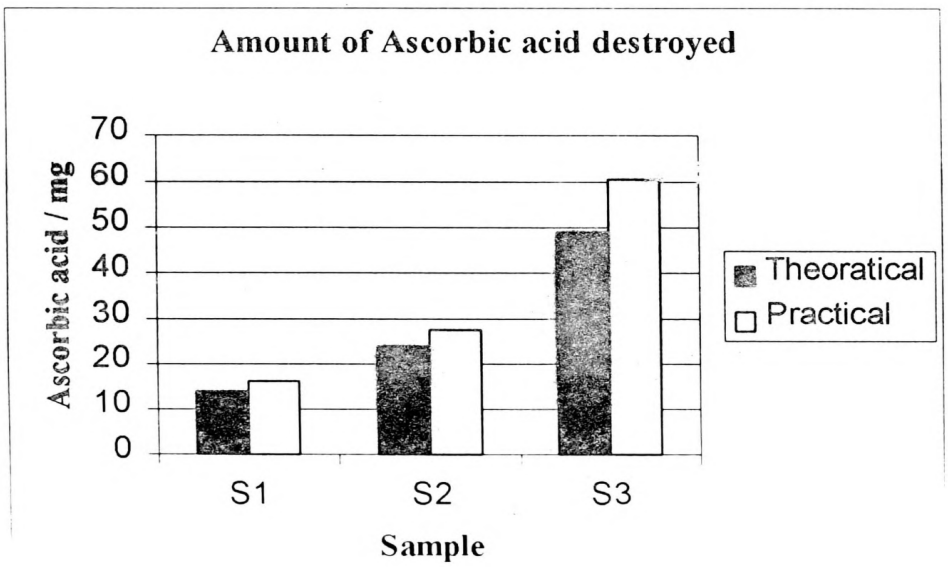


Figure 4.7 Practical and Theoretical Values Obtained for the Amount of Ascorbic Acid Destroyed During the Process

The theoretical values have deviated slightly from practical values. For the studies on heat stability of ascorbic acid, pure ascorbic acid was used instead of using limejuice. When fresh limejuice is considered, there are external factors other than high temperatures that are responsible for the destruction of ascorbic acid. The presence of Oxygen, Cupric ions and Fe as well as the activity of ascorbic acid oxidase enzyme system will accelerate the destruction of vitamin C. these factors are not counted in the experiment. This may be the cause for this small deviation that exists between the theoretical and practical values.

4.2.1 Ascorbic Acid Concentration of the Product After Storing for Three Months

The concentration of ascorbic acid in each sample of powder after a storage period of three months at 27^0C was calculated using the activation energy of the reaction. The concentration for each sample is listed in the table 4.11.

Table 4.11
Ascorbic Acid Concentration of the Samples After Storing for Three Months

Sample	Amount of ascorbic acid (mg)
S_1	53.66
S_2	69.77
S_3	96.70

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study was focussed on the development of a powder from lime with an extended shelf life. According to the results, it can be concluded that lime can be preserved by oven drying method in the form of a powder. The results obtained from the sensory evaluation revealed that the best sample is S_3, i.e. the sample that was fortified five times with fresh lime juice.

5.2 Suggestions

This preservation method can be made use of, to preserve lime during the season, so that the huge wastage of the produce can be prevented. The product will provide an answer to the price fluctuation throughout the year, and will provide a good substitute to lime in the lean seasons at a remunerative price.

Results obtained in observing the heat stability of ascorbic acid could be used to maximize the retention of vitamin C in lime juice and other citrus juices during thermal processing.

5.3 Recommendations for Further Studies

In this research, cornstarch was used as a drying aid in the drying process. Addition of cornstarch will reduce the solubility of the resultant powder. If a water-soluble compound is used instead of cornstarch, a powder with a higher solubility could be obtained Studies should be done with other water-soluble drying aids.

In this experiment, a conventional air dryer was used to dehydrate lime juice. Exposure to high temperatures for longer time periods will destroy ascorbic acid as well as the volatile compounds in the juice. Studies should be done using vacuum dryers, in which, the drying process is carried out at lower temperatures.

Shelf life evaluation of the product was not included in this research. Changes such as the oxidation of the flavour compounds and colour changes may be occur, upon prolonged storage. Therefore, further studies in the area of shelf life evaluation are recommended, in order to obtain a product with a better quality.

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Appendix 1

The Ballot Paper Used in the Sensory Evaluation Test

Name:
Date :

Please taste each of the samples in order listed below. Assign the rank values for the samples considering the acid flavour, according to the category scale given below.

Code	Rank assigned
356	.....
475	.....
802	.....

Category scale:

1 – Dislike extremely
2 – Dislike slightly
3 – Neither like nor dislike
4 – Like slightly
5 – Like extremely

Appendix 2

Results Obtained for the Sensory Evaluation Test

Kruskal-Wallis Test on Flavour

Sample	N	Median	Ave Rank	Z
1	30	3.000	23.3	-5.70
2	30	4.000	39.1	-1.64
3	30	5.000	74.1	7.34
Overall	90	45.5		

H = 59.42 DF = 2 P = 0.000

H = 64.69 DF = 2 P = 0.000 (adjusted for ties)

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