DEVELOPMENT OF SPRAY DRIED STRAWBERRY JUICE POWDER

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DEVELOPMENT OF SPRAY DRIED STRAWBERRY JUICE POWDER

JAGAN RAO A/L KRISHNAN

Thesis submitted to the Faculty of Chemical and Natural Resources Engineering in
Partial Fulfillment of the Requirement for the
Degree of Bachelor Engineering in Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

APRIL 2008

I declare that this thesis entitled "*Development of Spray Dried Strawberry Juice Powder*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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To my beloved father and mother

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ABSTRACT

This study explains the process and the optimum parameters to dry strawberry juice using lab scale spray dryer. The optimum parameters are the optimum drying temperature for minimum loss of vitamin C during drying process and optimum Maltodextrine 10 percentage for optimum colour, texture and taste of strawberry juice powder. There are 4 analytical experiments conducted on the strawberry juice powder produced which are vitamin C lost percentage, solubility analysis, effect of anti-caking and sensory evaluation. The strawberry juice is used as feed for spray drying process and production of strawberry powder. Firstly, strawberry juice is spray dried with spray dryer at various temperatures from 160 ^{o}C to 200 ^{o}C. Then, for the second experiment drying temperature is set constant at 170 ^{o}C and percentage of Maltodextrine is manipulated from 10% to 30%. Analytical experiment is conducted to measure the qualities of powder produced. Firstly, vitamin C loss studied before and after drying process to determine percentage of loss due to drying at different temperature. Then the solubility analysis conducted for powder produced with different percentage of Maltodextrine 10 addition. Then, the effect of anti-caking is analyzed by producing strawberry juice powder with addition of anti-caking agent (calcium carbonate). Lastly, sensory evaluation is conducted at cafeteria where people randomly choose to evaluate the qualities of reconstituted strawberry juice and powder produced. From the analysis, minimum loss of vitamin C observed at low temperature and it gradually increases as temperature increased. Solubility increases with increases of Maltodextrine 10 percentage. Anti-caking agent decreases hygroscopicity and increases the shelf life of powder. From all the analysis it is concluded that optimum temperature for drying is at 170 ^{o}C with optimum percentage of Maltodextrine 10 addition is 25%. High quality product is produced and it has high market potential in the future.

ABSTRAK

Kajian yang dijalankan menerangkan proses dan parameter optima bagi proses pengeringan jus strawberi mengunakan pengering semburan. Parameter optima yang dikaji adalah suhu optima bagi proses pengeringan dengan kehilangan vitamin C yang minima dan peratusan Maltodextrine 10 optima bagi tekstur, warna dan rasa yang baik. 4 analisis dijalankan terhadap serbuk strawberi yang diperolehi iaitu peratusan kehilangan vitamin C, analisis keterlarutan, kesan 'anti-caking' dan penilaian deria. Jus strawberi digunakan sebagai bahan mentah bagi proses pengeringan dan penghasilan serbuk strawberi. Eksperimen dimulakan dengan proses pengeringan jus strawberi pada suhu yang berlainan iaitu dari 160 ^{o}C ke 200 ^{o}C. Kemudian, process pengeringan dijalankan dengan suhu tetap pada 170 ^{o}C tetapi peratusan Maltodextrine 10 ditukar dari 10% hingga 30%. Eksperimen analitis dimulakan dengan menyukat kepekatan vitamin C sebelum dan selepas proses pengeringan. Peratusan kehilangan vitamin C pada setipa suhu dikaji. Kemudian, analisis keterlarutan dikaji bagi setiap serbuk yang dihasilkan dengan peratusan Maltodextrine 10 yang berbeza. Selepas itu, kesan 'anti-caking' terhadap serbuk dikaji dengan menghasilkan serbuk strawberi dengan tambahan kalsium karbonat. Akhir sekali penilaian deria dijalankan terhadap serbuk strawberi di kaferia pelajar. Beberapa pelajar dipilh secara rawak dan diberi jus dan serbuk strawberi bagi menilai kualiti merujuk kepada borang penilaian yang diedarkan. Analisis menunjukan kehilangan vitamin C yang sedikit yang pada suhu rendah dan ia meningkat pada suhu yang tinggi. Manakala, keterlarutan serbuk meningkat dengan meningkatnya peratusan Maltodextrine 10. 'Anti-caking' mengurangkan *hygroscopicity* dan meningkatkan tempoh penggunan. Suhu optima bagi proses pengeringan adalah pada 170 ^{o}C dengan peratusan optima Maltodextrine 10 adalah 25%. Produk yang mempunyai kualiti dan potensi pemasaran tinggi telah diperoleh.

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LIST OF ABBREVIATIONS

IU	-	International Unit
HPLC	-	High Performance Liquid Chromatography
SD	-	Spray Dryer
UV	-	Ultra-Violet
C-8	-	Octyl Chain
C-18	-	Octadecyl Chain
RI	-	Refractive Index
Near-IR	-	Near-Infra Red
MS	-	Mass Spectroscopy
NMR	-	Nuclear Magnetic Resonance
LS	-	Light Scattering
FDA	-	Food and Drug Association
U.S.A	-	United States of America
ASQC -	-	American Society for Quality Control
RSD	-	Relative Standard Deviation
UV-DAD	-	Ultra Violet Diode Array Detection
KK1	-	Kolej Kediaman 1

LIST OF SYMBOLS

μ	-	Micro
t	-	Time
r	-	Radius
d_L	-	Density of liquid
H_v	-	Latent heat of vaporization
m_i	-	Initial moisture content
m_f	-	Final moisture content
h	-	Film coefficient for heat transfer
ΔT	-	Temperature difference
D_p	-	Diameter of particle
A_i	-	Inlet cross section area
z	-	Depth of separator
D	-	Diameter of separator
V_o	-	Velocity of air
d_S	-	Density of particle
μ	-	Viscosity of fluid
ppm	-	Part per million
$CaCO_3$	-	Calcium Carbonate
KH_2PO_4	-	Potassium Dihydrogen Phosphate
RPM	-	Resolution per minutes
W	-	Watt
V	-	Voltage
MeOH	-	Methanol

CHAPTER 1

INTRODUCTION

1.1 Background

Fruits are important sources of vitamins and carbohydrates like fiber and sugar. They are low in calories and naturally sweet. Fruits and their juices are good sources of water, too. Different fruits contain different vitamins, so it is important to eat a variety of fruits. Mangoes, papayas, melons and citrus fruits, like oranges and grapefruit, are high in vitamin C **(Romero, M.A. Rodriguez, *et al, 1992*).** Cantaloupe, apricots, peaches, and nectarines are sources of vitamin A **(Bates CJ, 1995)**. Referring to food guide pyramid, fruits are second most important food in our daily life. There is no harm of taking lots of fruits because it will supply us all kind of vitamins which is needed by our body. These vitamins are very important for human being as a supplement and to avoid various kind of sickness.

Nowadays, the fast economic development has changed the trend of food consumption from calories assurance to diet nutrient enrichment. The consumers today are well educated and well aware of importance of vitamins. This scenario has increased the global market demand towards the fresh fruits. In order to meet the market demand throughout the year in all areas; the fresh fruits is preserved using different techniques. High moisture content in fruit leads towards decrease of quality due to water activity. So, drying of fruits used to remove the moisture content and decrease the water activity which will decrease the quality of the fruits. Drying is an ancient technique of preservation which still being applied in this modern world.

Nowadays, a lot more drying techniques such as spray drying, freeze drying, tray drying have invented to increase the productivity and achieve better control of process to increase the product quality.

Strawberry, scientific name *Fragaria* × *ananassa* and family Rosaceae (rose family), low herbaceous perennials with edible red fruits, native to temperate and mountainous tropical regions **(See G. M. Darrow, 1966)**. The name is derived from Old English *strēawberiġe* which is a compound of *streaw* meaning "straw" and *berige* meaning "berry". The major producer of strawberry is United States of America and Europe countries. It is reported USA produces 1,053,240 tonnes of strawberry in year 2005 **(USDA Economics, 2005)**. Fresh strawberry is delicious fruit and is one of the most nutritious fruit. The typical composition of strawberry is listed in Table 1.1 **(Ensminger AH, *et al,* 1983)**. It shows that strawberry contains high quantities vitamin C, fiber and potassium. The vitamin C content of some fruits is compared in Table 1.2 **(Romero, M.A. Rodriguez, *et al, 1992*)**. The table shows that strawberry is fourth highest vitamin C content among the fruits.

The fact that strawberries are available year round, offers us the perfect opportunity to add great taste and nutrition to our everyday meals. Research shows that 94% of Americans currently consume strawberries and it is strongly suggested that eating them more often will add to a person's overall long term health **(Hyson. D, 2002)**

In Malaysia strawberry is planted and produced at Cameron Highlands. It is the only place in Malaysia which has optimum weather and condition for plantation of strawberry. The cold and high humidity is the suitable place for plantation of the strawberry and it can be harvested the whole year. Currently, in market the following products are processed from strawberry; canned slices, concentrate, dehydrated products, jam, jelly, juice, puree, spread, syrup, flavors and yoghurt. Clarified and cloudy strawberry juice is at market. However, fresh strawberry juice powder is not available yet at market and will significant increases in demand of it in future.

Figure 1.1: Strawberry (*Fragaria × ananassa)*

Table 1.1: Composition of fresh Strawberry per 100g

Nutrient	Units	Amount
Proximates		
Water	g	91.570
Energy	kcal	30.000
Energy	kj	126.000
Protein	g	0.610
Total lipid (fat)	g	0.370
Carbohydrate, by difference	g	7.020
Fiber, total dietary	g	2.300
Ash	g	0.430
Minerals		
Calcium, Ca	mg	14.000
Potassium, K	mg	166.000
Vitamins		
Vitamin C, ascorbic acid	mg	56.700
Folate	mcg	17.700
Vitamin A, IU	IU	27.000

Table 1.2: Comparison of vitamin C content of some selected fruits

Fruits	Ascorbic Acid (mg/100g Fresh fruit)
Acerola	1300
Guava	300
Black Currant	210
Strawberry	57
Orange	50

1.2 Problem Statement

Strawberry (*Fragaria* × *ananassa)* the result of the hybridization of *F. chiloensis,* believed to be indigenous to Chile and to the mountains of W North America, with the wild strawberry (*F. virginiana*) of E North America **(See G. M. Darrow, 1966)**. The cultivated strawberries therefore big in size and have high water content. It is rich source of ascorbic acid (vitamin C) containing over 50 to 60 mg per 100g. The vitamin C is mainly found in the water content of the strawberry fruit. Therefore the strawberry juice should be consumed fresh in order to obtain all the nutrition. Clarified and cloudy strawberry juices are currently produced and may have greater market potential, but most of products are in liquid or cordial type. The drying technique also has been developed but process conditions for these products have not been determined to preservation purposes.

Preservation is very important because fresh strawberries are safe to be consumed within very short period. The strawberries are spoilt very fast and easily because of the water activity. Since strawberries have high water content, it is more prone to be spoilt if not stored and preserved well **(Ensminger AH, *et al,* 1983)**. Besides that, the geography condition of Cameron Highlands which is hilly and has moderate road system causes the transportation of the fresh strawberries not effective. The ineffective transportation system of strawberries in Malaysia leads to the fruit injuries. Since, the limited transportation available; a lot strawberries is

loaded in big lorries and transported to whole Malaysia. This indirectly causes injuries to fruits because of being stacked and transported through hilly roads.

Therefore the drying technique of preservation has been a very efficient and precise method to solve all the problems occurred. Drying has capable of the reduction of the vitamin C content in strawberry juice due to change of temperature and oxidation. However, the product produced (instant strawberry powder) has high potential to be used in formulated drinks, baby foods and other foods. The cost of transportation would reduce significantly when transporting the product to market places. Besides that, the fruit injuries problem due to the transportation would be reduces to zero and directly there would be no lost to the farmers.

However, information about strawberry powders does not exist in the literature. Strawberry has best nutritional properties and drying operations must be carefully designed to maintain these nutritional properties. Vitamin C retention in different temperature and after dried must be investigated to determine the effect of temperature in reduction of Vitamin C. Water activity level or moisture content effect on strawberry juice is investigated through experiment and to design best drying operating condition with minimum lost of vitamin C from the study.

1.3 Objectives

The objectives of the research project are:

1. to produce strawberry powder by spray drying method
2. to optimize the processing parameters for production of strawberry powder
3. to measure the qualities of spray dried strawberry powder

1.4 Scope

The scopes for this project are:

1. strawberry used as the source of pure juice
2. Maltodextrine 10 used as the carrier agent
3. determine effect of drying at different temperature on vitamin C (selected index)
4. optimum drying temperature for minimum lost of vitamin C determined
5. optimum Maltodextrine 10 percentage determined to obtain powder with optimum texture color and solubility rate
6. sensory evaluation conducted to determine the quality (appearance, flavor and texture) of the strawberry juice powder produced

CHAPTER 2

LITERATURE REVIEW

2.1 Food Preservation

Food preservation is the process which deals with the practical control of factors capable of adversely affecting the safety, nutritive value, appearance, texture, flavor, and keeping qualities of raw and processed foods. Since thousands of food products differing in physical, chemical, and biological properties can undergo deterioration from such diverse causes as microorganisms, natural food enzymes, insects and rodents, industrial contaminants, heat, cold, light, oxygen, moisture, dryness, and storage time, food preservation methods differ widely and are optimized for specific products. **(Borgstrom, 1968)**

Preservation usually involves preventing the growth of bacteria, fungi, and other micro-organisms, as well as retarding the oxidation of fats which cause rancidity. It also includes processes to inhibit natural ageing and discoloration that can occur during food preparation such as the enzymatic browning reaction in apples which causes browning when apples are cut. Some preservation methods require the food to be sealed after treatment to prevent recontamination with microbes; others, such as drying, allow food to be stored without any special containment for long periods. **(N. W. Desrosier, 1970)**

Common methods of applying these processes include drying, spray drying, freeze drying, freezing, vacuum-packing, canning, preserving in syrup, sugar

crystallization, food irradiation, and adding preservatives or inert gases such as carbon dioxide. Other methods that not only help to preserve food, but also add flavor, include pickling, salting, smoking, preserving in syrup or alcohol, sugar crystallization and curing. **(N.W. Desrosier, 1970).** Although there are many food preservation methods, our concern in this research project is drying preservation technique.

2.2 Drying

Drying is an ancient method of food preservation technique which defined as the application of heat under controlled conditions to remove the majority of the water normally present in a food by evaporation. It is a complicated process involving simultaneous heat and mass transfer in which heat penetrates into the product and moisture is removed by evaporation into an unsaturated gas phase.

Bacteria and micro-organisms within the food and from the air need the water in the food to grow. Drying effectively prevents them from surviving in the food. It also creates a hard outer-layer, helping to stop micro-organisms from entering the food. Drying method varies with the specific food and end products.

2.2.1 Drying Mechanism

The mechanism of moisture movement within the solid in drying process has received much attention in the literature and a significant number of drying theories have been developed. Mechanisms such as, molecular diffusion, capillary motion, liquid diffusion through solid pores, vapor diffusion in air-filled pores, Knudsen flow, vaporization condensation sequence flow and hydrodynamic flow were considered. These mechanisms are of particular importance for fruits and vegetables as product structure will influence the moisture movement. **Mujumdar** (1990)

reviewed theories on the mechanism of moisture migration. Generally, there appear to be four probable major modes of transfer:

1. Liquid movement caused by capillary forces;
2. Liquid diffusion resulting from concentration gradients;
3. Vapor diffusion due to partial pressure gradients;
4. Diffusion in liquid layers absorbed at solid interfaces.

Foods can be classified as *hygroscopic* and *non-hygroscopic*. The partial pressure of water vapor in hygroscopic food varies with the moisture content, while that of non-hygroscopic food is constant at different moisture contents. Thus, non-hygroscopic foods have a single falling-rate period, whereas hygroscopic foods have two falling rate periods. In the falling rate periods, the rate of moisture movement within the solid and the effects of external factors.

Moisture transfer in drying is a complex process where different mechanisms can occur at the same time. In the process of drying, mechanisms may vary considerably. A realistic model should consider as many as of the different phenomenon (e.g., simultaneous heat and mass transfer, multi-dimensional transfer, material shrinkage) occurring in the course of drying. It may not be possible to use same drying model for different foods or drying conditions.

There are many drying method available to dry strawberry juice puree. However the spray drying method has higher nutrient retention compare to the other methods. **(Robert Harris and Endel Karmas, 1975).** The juice or puree is dispersed or atomized to form droplets and sprayed into a heated chamber where it is dried and forms a “free-flowing” powder. The more common technologies such as convection, cabinet and drum drying are more costly, more labor-intensive, more complicated, and more likely to cause “powder burns.”

2.3 Spray Dryer

2.3.1 Introduction

A spray dryer is a device used in spray drying. It takes a liquid stream and separates the solute or suspension as a solid and the solvent into a vapor. The solid is usually collected in a drum or cyclone. The liquid input stream is sprayed through a nozzle into a hot vapor stream and vaporised. Solids form as moisture quickly leaves the droplets. A nozzle is usually used to make the droplets as small as possible, maximising heat transfer and the rate of water vaporisation. Droplet sizes can range from 20 μm to 180 μm depending on the nozzle.

Spray dryers can dry a product very quickly compared to other methods of drying. They also turn a solution or slurry into a dried powder in a single step, which can be advantageous for profit maximization and process simplification.

2.3.2 Basics of Spray Drying

2.3.2.1 Concentration of Puree

Feedstock is normally concentrated earlier before introduction into the spray dryer. The concentration stage increases the solids content thereby reducing the amount of liquid that must be evaporated in the spray dryer. The feedstock for conventional big scale spray dryer normally will be concentrated to 50%-60% before introduced to spray dryer. However the small scale laboratory spray dryer will have more diluted feedstock because it will be clogged easily if the feed have high viscosity.

2.3.2.2 Atomization

Atomization refers to the conversion of bulk liquid into a spray or mist (i.e. collection of drops), often by passing the liquid through a nozzle. Despite the name, it does not usually imply that the particles are reduced to atomic sizes. The liquid which sprayed through nozzle will increase the surface area of the liquid which later will be contacted to hot air and dried into powder. The nozzle size may differ according to the size of spray dryer. Droplet sizes can range from 20 μm to 180 μm depending on the nozzle. Smaller spray dryer occupies smaller nozzles and reverse for the industrial scale spray dryer.

2.3.2.3 Droplet-air contact

The important component of spray dryer is the chamber; here the sprayed droplet is contacted with the hot air for the drying process. Air, which is, normally the drying media used, is heated to a predefined temperature depending upon the characteristics of the feed fluid and hot air is heated by the heating element which situated before entering the camber. This hot air is brought in contact with the spray droplets in one of the following ways through the air distributor.

1. Co-current-Air and particles move in the same direction.
2. Counter-current-air and particles move in the opposite direction.
3. Mixed flow - particles are subjected to co-current and counter-current phase.

The thermal energy of the hot air is used for evaporation and the cooled air pneumatically conveys the dried particles in the system. The contact time of the hot air and the spray droplets is only a few seconds, during which drying is achieved and the air temperature drops instantaneously. The nozzle which increases the contact area of droplet and hot air influences in huge heat transfer between the droplet and hot air. The hot air evaporates the moisture content in the droplet and changes it into powder form. The dried particle never reaches the drying air temperature. This

enables efficient drying of heat sensitive materials without thermal decomposition. The most efficient way of spray droplet and hot air brought in contact is counter-current.

2.3.2.4 Droplet Drying

The droplet drying takes place in three periods. During the first period, the temperature increases to the wet bulb temperature. In the second period, a concentration gradient builds up in the drop and water activity at the surface decreases, thus causing the surface temperature to rise above that of the of the wet bulb temperature. In the final period, internal diffusion becomes limiting. Critical moisture content is eventually reached below which the surface becomes impenetrable. **(Karel, 1975)** Different products have differing evaporation and particle-forming characteristics. Some expand; others contract, fracture or disintegrate. The resulting particles may be relatively uniform hollow spheres, or porous and irregularly shaped.

The drying time for a single droplet may be estimated by the following equation:

$$t = \frac{r^2 \, d_L H_V \, (m_i - m_f)}{3 \, h \, (\Delta \mathrm{T})(1 + \, m_i)}$$

The typical drying time for an average milk droplet of 40 μ is only a fraction of a second. However, because of the great initial velocity, the particle will have traveled a considerable distance from the atomizer before it is dry (13.5 cm for average conditions). It should be noted, that the drying time is proportional to the square of the radius; thus, for larger droplets the drying time may become so long that the droplet reaches the wall of the dryer while still wet. This problem is often encountered in small scale dryers. The above equation also stresses that the drying time can be shortened by reducing the initial moisture content by pre-concentration of the liquid. **(Buma, 1971)**

2.3.2.5 Separation of Dry Particles

Separation is carried out partly within the drying chamber itself and partly in secondary separation equipment. In general, it is easy to remove 90% or more of the powder, but removal of the remainder becomes problematic. Cyclone separators operate on the 'momentum separation' principle (centrifugal action) and are extensively used in large scale dryers for removal of fines.

Charm (1971) has given an equation which relates the dimensions of a cyclone to the smallest particle (Dp) which can be separated:

$$D_p^2 = \frac{3.6\, A_i\, D_0 \mu}{ZDV_0 d_s}$$

From the equation it appears that in designing a cyclone the depth and diameter should be as large as possible. Increasing the air velocity is also important. Industrial experience has shown that efficiency is also affected by the powder concentration in the air stream. For this reason, it is better to use several cyclones in parallel than just one single separator.

2.3.2.6 Powder Collection

As the powder is separated with the cold air by the cyclone separator, the dried powder will drop through the opening at the bottom of cyclone. So, there will be a container which is attached to the cyclone for recovery of the powder produced. The powder primary recovered from the drying chamber and collected into separate container. The conventional spray dryer is equipped with hammer which will give vibration to the wall of the drying chamber and drop all the powder that stick at wall of the chamber. The small scale sprayer does not facilitate with such advantage and it is done manually.

2.3.3 Advantage of Spray Dryer

In the world of industrial dryers, there are few types that accept pump able fluids as the feed material at the inlet end of the process and produce dry particulate at the outlet. Spray drying is unique in its ability to produce powders with a specific particle size and moisture content without regard for the capacity of the dryer and the heat sensitivity of the product. This flexibility makes spray drying the process of choice for many industrial drying operations. Advantages of spray drying are as follows:

- Able to operate in applications that range from aseptic pharmaceutical processing to ceramic powder production.
- Can be designed to virtually any capacity required. Feed rates range from a few pounds per hour to over 100 tons per hour.
- Powder quality remains constant during the entire run of the dryer.
- Operation is continuous and adaptable to full automatic control.
- A great variety of spray dryer designs are available to meet various product specifications.
- Can be used with both heat-resistant and heat sensitive products.
- As long as they are can be pumped; the feedstock can be abrasive, corrosive, flammable, explosive or toxic.
- Feedstock can be in solution, slurry, paste, gel, suspension or melt form.
- Product density can be controlled
- Nearly spherical particles can be produced.
- Material does not contact metal surfaces until dried; reducing corrosion problems.

2.3.4 Schematic Diagram of Laboratory Scale Spray Dryer

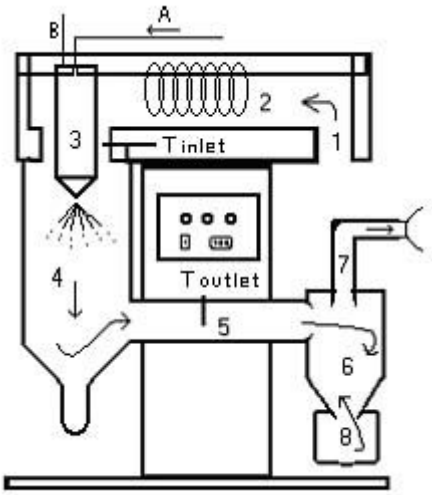


Figure 2.1: Laboratory Scale Spray Dryer

Table 2.1: Components of Spray Dryer

Label	Element
1	Air Inlet
2	Air Heating Element
3	Atomizer
4	Drying Chamber
5	Connector
6	Cyclone Separator
7	Cold Air Outlet
8	Container (Powder Recovery)
A	Compressed Air
	Feed

2.4 High Performance Liquid Chromatography (HPLC)

2.4.1 Introduction

High-performance liquid chromatography (or High pressure liquid chromatography, HPLC) is a form of column chromatography used frequently in biochemistry and analytical chemistry to separate, identify, and quantify compounds. HPLC utilizes a column that holds chromatographic packing material (stationary phase), a pump that moves the mobile phase(s) through the column, and a detector that shows the retention times of the molecules. Retention time varies depending on the interactions between the stationary phase, the molecules being analyzed, and the solvent(s) used **(Lloyd R. Snyder and John W. Dolan, 2006)**

2.4.2 Basics of HPLC

2.4.2.1 Mobile Phase

The mobile phase in HPLC refers to the solvent being continuously applied to the column, or stationary phase. The mobile phase acts as a carrier for the sample solution. A sample solution is injected into the mobile phase of an assay through the injector port. As a sample solution flows through a column with the mobile phase, the components of that solution migrate according to the non-covalent interactions of the compound with the column. The chemical interactions of the mobile phase and sample, with the column, determine the degree of migration and separation of components contained in the sample. For example, those samples which have stronger interactions with the mobile phase than with the stationary phase will elute from the column faster and thus have a shorter retention time, while the reverse is also true. The mobile phase can be altered in order to manipulate the interactions of the sample and the stationary phase. **(Snyder, 1983)**

2.4.2.2 Stationary Phase

The stationary phase in HPLC refers to the solid support contained within the column over which the mobile phase continuously flows. The sample solution is injected into the mobile phase of the assay through the injector port. As the sample solution flows with the mobile phase through the stationary phase, the components of that solution will migrate according to the non-covalent interactions of the compounds with the stationary phase. The chemical interactions of the stationary phase and the sample with the mobile phase, determines the degree of migration and separation of the components contained in the sample. For example, those samples which have stronger interactions with the stationary phase than with the mobile phase will elute from the column less quickly and thus have a longer retention time, while the reverse is also true. Columns containing various types of stationary phases are commercially available. Some of the more common stationary phases include: Liquid-Liquid, Liquid-Solid (Adsorption), Size Exclusion, Normal Phase, Reverse Phase, Ion Exchange, and Affinity. However our concern for the research project is Reverse Phase stationary phase. **(Snyder, 1983)**

2.4.2.2.1 Reverse Phase

Reverse Phase operates on the basis of hydrophilicity and lipophilicity. The stationary phase consists of silica based packings with n-alkyl chains covalently bound. For example, C-8 signifies an octyl chain and C-18 an octadecyl ligand in the matrix. The more hydrophobic the matrix on each ligand, the greater is the tendancy of the column to retain hydrophobic moieties. Thus hydrophilic compounds elute more quickly than do hydrophobic compounds. **(Snyder, 1983)**

2.4.2.3 Injector

Samples are injected into the HPLC via an injection port. The injection port of an HPLC commonly consists of an injection valve and the sample loop. The sample is typically dissolved in the mobile phase before injection into the sample loop. The sample is then drawn into a syringe and injected into the loop via the injection valve. A rotation of the valve rotor closes the valve and opens the loop in order to inject the sample into the stream of the mobile phase. Loop volumes can range between 10 µl to over 500 µl. In modern HPLC systems, the sample injection is typically automated.

Stopped-flow Injection is a method whereby the pump is turned off allowing the injection port to attain atmospheric pressure. The syringe containing the sample is then injected into the valve in the usual manner, and the pump is turned on. For syringe type and reciprocation pumps, flow in the column can be brought to zero and rapidly resumed by diverting the mobile phase by means of a three-way valve placed in front of the injector. This method can be used up to very high pressures **(Willard, 1988)**

2.4.2.4 Pumps

There are several types of pumps available for use with HPLC analysis, they are: Reciprocating Piston Pumps, Syringe Type Pumps, and Constant Pressure Pumps.

2.4.2.4.1 Reciprocating Piston Pumps

Reciprocating Piston Pumps consist of a small motor driven piston which moves rapidly back and forth in a hydraulic chamber that may vary from 35-400 µL

in volume. On the back stroke, the separation column valve is closed, and the piston pulls in solvent from the mobile phase reservoir. On the forward stroke, the pump pushes solvent out to the column from the reservoir. A wide range of flow rates can be attained by altering the piston stroke volume during each cycle, or by altering the stroke frequency. Dual and triple head pumps consist of identical piston-chamber units which operate at 180 or 120 degrees out of phase. This type of pump system is significantly smoother because one pump is filling while the other is in the delivery cycle.

2.4.2.4.2 Syringe Type Pumps

Syringe Type Pumps are most suitable for small bore columns because this pump delivers only a finite volume of mobile phase before it has to be refilled. These pumps have a volume between 250 to 500 mL. The pump operates by a motorized lead screw that delivers mobile phase to the column at a constant rate. The rate of solvent delivery is controlled by changing the voltage on the motor.

2.4.2.4.3 Constant Pressure Pumps

In Constant Pressure Pumps the mobile phase is driven through the column with the use of pressure from a gas cylinder. A low-pressure gas source is needed to generate high liquid pressures. The valving arrangement allows the rapid refill of the solvent chamber whose capacity is about 70 mL. This provides continuous mobile phase flow rates.

2.4.2.5 Detectors

The detector for an HPLC is the component that emits a response due to the eluting sample compound and subsequently signals a peak on the chromatogram. It is positioned immediately posterior to the stationary phase in order to detect the compounds as they elute from the column. The bandwidth and height of the peaks may usually be adjusted using the coarse and fine tuning controls, and the detection and sensitivity parameters may also be controlled (in most cases). There are many types of detectors that can be used with HPLC. Some of the more common detectors include: Refractive Index (RI), Ultra-Violet (UV), Fluorescent, Radiochemical, Electrochemical, Near-Infra Red (Near-IR), Mass Spectroscopy (MS), Nuclear Magnetic Resonance (NMR), and Light Scattering (LS). Here we concentrate more on Ultra-Violet (UV) detector.

2.4.2.5.1 Ultra Violet Detectors (UV)

Ultra-Violet (UV) detectors measure the ability of a sample to absorb light. This can be accomplished at one or several wavelengths:

a) Fixed Wavelength measures at one wavelength, usually 254 nm
b) Variable Wavelength measures at one wavelength at a time, but can detect over a wide range of wavelengths
c) Diode Array measures a spectrum of wavelengths simultaneously

UV detectors have a sensitivity to approximately 10-8 or 10 -9 gm/ml. Therefore, we have chosen the UV detector to conduct the analysis on the nutrient retention. So, more accurate results can be obtained.

2.4.2.6 The Whole HPLC Analysis Process

2.4.2.6.1 A Flow Scheme of HPLC

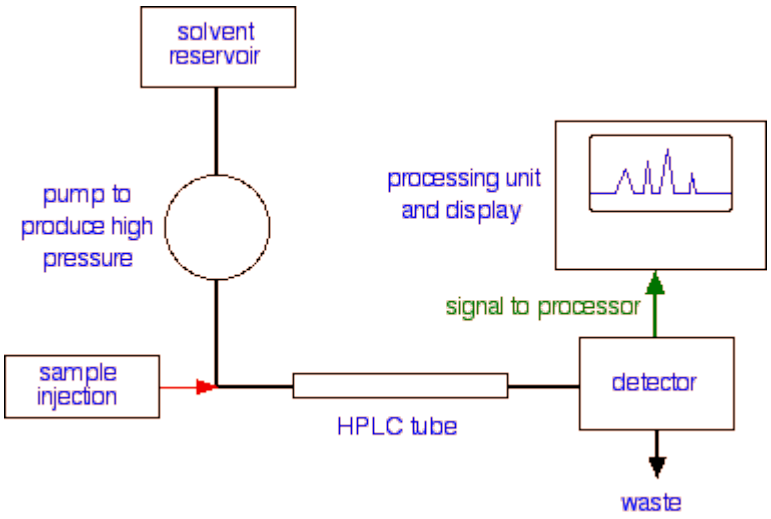


Figure 2.2: Flow Scheme of HPLC

2.4.2.6.2 Retention Time

The time taken for a particular compound to travel through the column to the detector is known as its retention time. This time is measured from the time at which the sample is injected to the point at which the display shows a maximum peak height for that compound.

Different compounds have different retention times. For a particular compound, the retention time will vary depending on:

- the pressure used (because that affects the flow rate of the solvent)
- the nature of the stationary phase (not only what material it is made of, but also particle size)
- the exact composition of the solvent

- the temperature of the column

That means that conditions have to be carefully controlled if retention time's method used as a way of identifying compounds.

2.4.2.6.3 The Detector

Many organic compounds absorb UV light of various wavelengths. If we have a beam of UV light shining through the stream of liquid coming out of the column, and a UV detector on the opposite side of the stream, we can obtain a direct reading of how much of the light is absorbed.

The amount of light absorbed will depend on the amount of a particular compound that is passing through the beam at particular time.

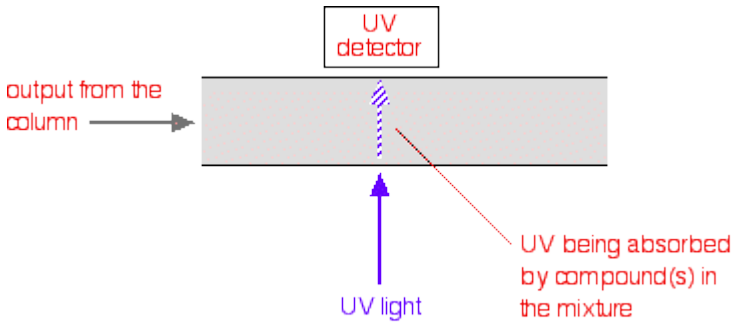


Figure 2.3: Schematic of Detector

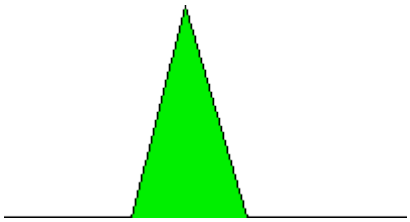
The solvent used do absorb the UV light but different compound absorb most strongly in different wavelength of UV spectrum. Methanol, for example, absorbs at wavelengths below 205 nm, and water below 190 nm. If methanol-water mixture used as the solvent, we would therefore have to use a wavelength greater than 205 nm to avoid false readings from the solvent. The optimum wavelength used for vitamin C (ascorbic acid) detection is 254 nm.

2.4.2.6.4 Interpreting the Output from Detector

The output will be recorded as a series of peaks - each one representing a compound in the mixture passing through the detector and absorbing UV light. As long as the condition of the column is controlled carefully, we could use the retention times to help to identify the compounds present – provided.

However, the peaks also can be used as a way of measuring the quantities of the compounds present. Let's suppose that the interested particular compound is X. If solution containing a known amount of pure X injected into the machine, not only it could record its retention time, but also relates the amount of X to the peak that was formed.

The area under the peak is proportional to the amount of X which has passed the detector, and this area can be calculated automatically by the computer linked to the display. The area it would measure is shown in green in the (very simplified) diagram.



If the solution of X was less concentrated, the area under the peak would be less - although the retention time will still be the same. Therefore, area under the graph indicates the concentration of X in solution. For example:

This means that it is possible to calibrate the machine so that it can be used to find how much of a substance is present - even in very small quantities. The calibration curve is developed using 5 standard solutions (known concentration). Later a calibration curve drew using the amount area under graph versus concentration. The calibration curve will give the equation (Y=mX + B) which related the area under graph and concentration. Then, from the equation and the area under the graph which obtained for particular compound, the concentration of the particular compound in pure juice can be determined. The X in equation indicated concentration of compound and Y indicated area under graph.

2.5 Nutrient Retention

By the definition of *ANSV* ASQC standard (ASQC, 1986), quality means "the features and characteristics of a product or service to the ability of that product or service to satisfy given needs". In other words, it is the acceptability of the buyers, or the customers, that finally determine the degree of quality. In the present day, the majority of consumers either has a high level of educational background or get information from the guide of the authorities concerned, such as Nutritional Labeling Education of FDA in U.S.A, **(Block and Langseth, 1994).** They would rather choose natural or minimally processed food than food that contains artificial additives, such as saccharine, nitrite, etc., or "over" -processed food **(King et aI., 1989).** Such thought is somewhat correct as nutrient loss always occurs in processing and storage.

The nutrient retention of processed foods at the time of purchasing or consumption depends mainly on the compositions of the raw materials, the pre-processing or pre-storage conditions, the processing method, and the storage conditions in transportation, distribution, wholesale and retail before consumption. For the majority of food products in which the quality decreases with time, it follows that there will be a limited period of time, which is so-called the shelf-life, before the product becomes unacceptable. From the viewpoint of food processors, by the definition of Institute of Food Technologists in the USA, shelf-life is "the period

between the manufacture and the retail purchase of a product, during which time the product is in a state of satisfactory quality in terms of nutritional value, taste, texture and appearance" **(Robertson, 1993).** From the definition, it encompasses the chemical, biochemical, microbiological, sensorial and nutritional stability attributes to the quality of foods. The nutrient retention will occupy a more important position in the scope of shelf-life, other than the texture, flavor and microbiological change. Hence, more attention has been paid by the food processors to determine the nutrient aspect of shelf-life of their products. The North Central Region Committee on Food Losses and Conservation (NCR-122) of U.S.A. established definitions of yield, loss and waste, in food processing and might be applied in storage as well. "Loss" was classified into two categories - avoidable waste "that loss which is avoidable by use of the best practical technology", and unavoidable waste, "nutrients". Now the challenge that a modern food technologist should face is to find a solution to convert the nutrient unavoidable losses to avoidable losses as much as possible not only in the processing but also in the storage to meet with the requirement of the customers. Therefore, it is important to focus the research in minimizing nutrient loss in processing and storage.

2.5.1 Vitamin C Retention

Fresh fruit and vegetables, with the living and respiring tissues, are perishable or can be destroyed. Thus, they have limited shelf-life. Certain extent of processing is necessary to prolong their shelf-life, such as drying. The nutritional advantage of fruits and vegetables is that they contain a plentiful amount of micronutrients and are rich sources of vitamins.

Processing and preservation generally involve deterioration in vitamins, because of their susceptibility to degradation. **Marks, J.S (1983)** showed that food processing causes changes in vitamin in many ways, such as:

1. losses during preprocessing operations (cleaning, peeling, trimming, milling, mechanical
2. damage, fermentation, leaching in water blanching, etc.);
3. changes due to the specific preservation process (sterilization, freezing, dehydration) degradation during storage.

Among all groups of vitamins, L-ascorbic acid or vitamin C (L-threo-2-hexenono-1 A-lactone) is one of the most vulnerable to chemical degradation and has been studied in great detail. It is can easily be oxidized. Factors that influence the degradation of vitamin C include temperature, water activity or moisture content, pH, and metal traces, especially copper and iron. Different ways may give different products affecting the overall degradation rate. The reaction may proceed under aerobic and anaerobic conditions, or through catalyzed or uncatalyzed aerobic pathways **(Villota and Hawkes, 1992).** Thus, vitamin C degradation becomes a complicated process involving many parameters and different pathways. However, the degradation process can be simplified for analysis purpose:

Ascorbic acid $\longrightarrow$ Degraded Products

If vitamin C, being the most unstable among the nutrients, is retained well, the other nutrients are generally assumed to be well retained. Thus, vitamin C may be used as a quality index (QA) for the degradation of vitamin C rich product. So, Vitamin C (L-ascorbic acid) was selected as the quality index and its retention during drying and storage was monitored.

CHAPTER 3

METHODOLOGY

3.1 Introduction

For this research the type of method that is used is experimental method. Drying process is the main process to convert strawberry powder from strawberry juice. There are two phase of this experiment. First phase is drying process of strawberry using spray drier at different temperature. Second phase is analytical analysis on the qualities of strawberry juice powder. The qualities that measured and analyzed are vitamin C reduction percentage after spray drying process at different temperatures, influence of maltodextrine towards solubility, anti-caking agent and sensory evaluation on powder produced.

The main part of this research is to prepare strawberry powder from strawberry puree and determination of optimum temperature for minimum lost of vitamin C.

3.2 Material

3.2.1 Strawberry Puree

The fresh samples for this phase of experiment were first bought from Giant at Kuantan. Later the strawberry fruits are blended without any additional of water to produce strawberry juice puree. The strawberries bought are California breed and it is big in size which contributes to high water content. The strawberries are blended using the centrifugal juice blender. The solids of strawberries were retained by a filter while juice is collected separately in a container. However, there was still very small size strawberries' solid in the juice; solids are removed by filtering using cloth filter. The sample vitamin C is determined initially because study shows blending and cutting can contributes to metal catalyzing of deterioration of vitamin C but it is very low effect and negligible.

3.2.2 Maltodextrine

Maltodextrin is based products line; cover the dextrose equivalent (DE) range from 10 to 40. Each product differs in its degree of hydrolysis, providing wide range of functionalities and properties for special applications. Maltodextrin often use as bulking agent, dispersant, carrier agent, binding agent, processing aid and texture improver in a variety of food and beverage products. Maltodextrin also plays part to decrease the puree solubility and hygroscopicity.

Maltodextrin 10 were used for this study. This product is sponsored by AAA San Soon Seng Food Industries Sdn. Bhd (Sungai Buloh, Selangor). Maltodextrine does not have any taste and no impact on typical flavor of the juice. Maltodextrine is the product which produced from starch like corn starch, tapioca starch and etc. Hence the final spray dried product contains fruit solids and hydrolyzed maltodextrine solids, and in liquid solution contribute the typical flavor and color of the original fruit. Maltodextrine play part only as a medium to spray dry. The

maltodextrine causes the powder produced will be free flow and very fine which retain colour of the strawberry juice. The specification of the Maltodextrine 10 is shown in table below.

Table 3.1: Specification of Maltodextrine 10

Specification	
Dextrose Equivalent	9 - 12
Moisture, %	Max. 5.0
pH (20% Solution)	4.5 - 5.5
Sulphur Dioxide, ppm	Max. 10
Colour (O.D.)	Max. 2.0
Bulk Density (tapped), g/l	450 - 600
Shelf life	2 years
Raw material	Tapioca Starch
Storage condition	Cool & dry condition

3.2.3 Anti-Caking Agent

Anti-caking agents are used in such things as table salt to keep the product from forming lumps, making it better for packaging, transport, and for the consumer. An anti-caking agent in salt is denoted in the ingredients for example as "anti-caking agent (554)", which is sodium aluminosilicate, a man-made product. This product is present in many commercial table salts as well as dried milks, egg mixes, sugar products, and flours. In Europe, sodium ferrocyanide (535) and potassium ferrocyanide (536) are more common anti-caking agents in table salt. Natural anti-caking agents used in more expensive table salt include calcium carbonate and magnesium carbonate. In this research project the natural anti-caking agent calcium carbonate is used.

Anti-caking agents are also used in non-food items such as road salt, fertilizers and cosmetics, and in manufacturing applications.

3.2.3.1 Calcium Carbonate

Calcium carbonate is a chemical compound with the chemical formula $CaCO_3$. It is a common substance found as rock in all parts of the world, and is the main component of shells of marine organisms, snails, and eggshells. Calcium carbonate is the active ingredient in agricultural lime, and is usually the principal cause of hard water. It is commonly used medicinally as a calcium supplement or as an antacid, but high consumption can be hazardous.

Calcium Carbonate added to finely powdered or crystalline food products to prevent caking, lumping, or agglomeration. The specification of the anti-caking agent (calcium carbonate) is shown below:

Table 3.2: Specification of Calcium Carbonate

Specification of Calcium Carbonate	
Cas Number	471-34-1
Molar Mass	100.09 g/mol
Appearance	White powder
Density	2.71 g/cm^3 (calcite) 2.83 g/cm^3 (aragonite)
Solubility in Water	0.00015 mol/L (25°C)

Figure 3.1: Calcium Carbonate

3.3 Equipments

3.3.1 Spray Dryer

The laboratory scale spray dryer used for to spray dry the strawberry juice. The powder is produced by continues process and free flow powder is produced. The picture of the spray dryer used is illustrated below:

Figure 3.2: Laboratory Scale Spray Dryer

The specification of the spray dryer is shown in the table below:

Table 3.3: Specification of Spray Dryer

Specification of spray drier	
Type/Brand	Lab Plant SD 06
Capacity	50-200 g/hr
Vacuum Pressure	1 psig
Serial No.	399
Manufacturing Code	2504

3.3.2 Fruit Juice Blender

The centrifugal mechanism house hold Fujiu fruit juice blender is used to prepare strawberry fruit juice. The blender operates according to the centrifuge mechanism where the blendered strawberries is rotated with high rpm; the centrifugal rotation cause the solid and juice to be thrown to the wall of blender (sieve). The sieve later will retain the solid and allow the juice to go through the sieve. So, the juice is separated and collected with different container. The picture of the blender is shown below:

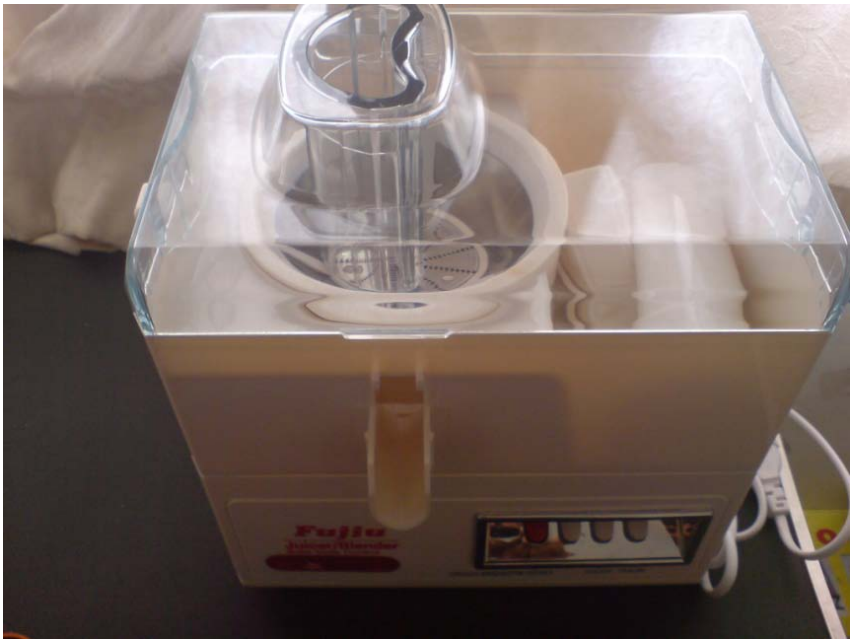


Figure 3.3: Fruit Juice Blender

Specification of the blender is shown below:

Table 3.4: Specification of Blender

Specification of Blender		
Power Supply	220 V – 250 V, Local supply rating 50-60 Hz	
Power Consumed	300 W	
Speed	11000 – 22000 rpm (blender)	
Blender Container	Maximum 1000-1600 mL	
Weight	3 kg	
Accessory	Meat Mincer	Dry Mill

3.3.3 High Performance Liquid Chromatography (HPLC)

High Performance Liquid Chromatography (HPLC) is used for the analytical measurement of the vitamin C before and after the strawberry juice spray dried. HPLC is essential equipment for the measurement of nutrients' concentration in strawberry juice because it is very sensitive and able detect very low concentration. The picture of HPLC used for the analysis is shown below:

Figure 3.4: HPLC

The specification of the HPLC at FKKSA analytical laboratory is shown below:

Table 3.5: Specification of HPLC

Specification of HPLC	
Instrument No.	G1311-90107
Injection Range	0.1-100 µl in 0.1ul increments
Replicate injections	1 - 99 from one vial
Precision	Typically < 0.5% RSD of peak areas from 5 - 100 µl Typically < 1% RSD of peak areas from 1- 5 µl
Minimum sample volume	1 µl from 5 µl sample in 100 µl microvial, or 1µl sample in 300 µl microvial
Carryover	Typically < 0.1 %, < 0.05% with external needle cleaning
Sample capacity	100 x 2-ml vials in 1 tray 40 x 2-ml vials in 1/2 tray 15 x 6-ml vials in 1/2 tray
Injection cycle time	Typically 50 s depending on draw speed and injection volume
Detector	Ultra Violet (190 – 600 nm)

Source: Agilent Technology

3.4 Method of Research

3.4.1 Strawberry Sample Preparation

After the strawberry juice is blendered and filtered; it is must be prepared further for spray drying process. The Maltodextrine 10 is added according to the percentage (mass maltodextrine/Volume of sample) and stirred until all the Maltodextrine dissolve and mixed well. The sample is later spray dried to produce free flow powder at specific temperature.

3.4.2 Drying Process

The prepared sample is dried using the Lab Plant SD 06 spray dryer. All the components of the spray dryer must be washed, dried and fixed. Before strawberry juice spray dried always run distilled water to make sure all the moisture to be dried and to heat up the spray dryer.

The power cable for spray dryer and pump is fixed and switched on. Later switch ON the main switch on the SD 06 spray dryer. Then switch on fan, compressor and the heater according to the appropriate screen and green 'START' button. The heater will not be operating unless the blower is running. This is a delay on the start up before the heater switches on. Using the controller on screen 'FAN SETTINGS' adjust the fan speed to 20. Later use SET TEMP screen to set the inlet temperature at 160 ^{o}C. The actual Inlet Temperature and Outlet Temperature are displayed all times on SET TEMP screen. Then wait until the spray dryer inlet temperature and outlet temperature to reach desired temperature and stabilizes.

Using the 0.5mm jet maximum backpressure is approximately 2.8 Bar and this reduces when the bigger diameter jets used. For this research project 0.5mm jet is used to spray the sample and increase the contact area between the sample droplet and hot air. After the temperature stabilizes on screen 'PUMP SETTING' pump set to 5 and deblocker set at fast. Spray dryer is allowed to warm up for a while until it shows 'Sytem Ready'. For first trial water is run through the spray dryer for 5 minutes. This ensures the silicone tube to clean and to prepare the spray dryer for drying process.

Transfer the silicone from water to sample and allow the pump to carry the sample through the tube. When the sample reaches the jet the Spray Drying Process should commence and dried powder should be observed spiraling down the cyclone into the collection bottle.

The exact spray drying procedure is carried out with different temperature. Later the same procedures followed to produce powder for different amount Maltodextrine 10. Different percentage of Maltodextrine is added to strawberry juice sample before spray dying. The operating temperature is maintained at 170 ^{o}C and only percentage of Maltodextrine which varies from 10% to 30% is added to the sample. This experiment conducted to observe the effect of the Maltodextrine 10 towards the quality of the powder produced. The powder produced in kept at dry place for further analytical experiment.

3.4.3 Analytical Experiment

3.4.3.1 Vitamin C measurement using HPLC

For this vitamin C analysis two type of mobile phase need to be used. Firstly, 0.05M potassium hydrogen phosphate (KH_2PO_4) is prepared and named as mobile phase A. For the second solvent 70 percent of HPLC grade methanol and 30 percent of 0.05 potassium hydrogen phosphate is mixed and named as mobile phase B. 500 ml of mobile phase A and B syringe filtered and filled into solvent flask. 250 mL HPLC grade methanol is syringe filter and put into solvent flask. Another 250 mL solvent flask is filled with ultra pure water to be used as solvent.

After that 5 standard solution of ascorbic acid of 20mg/l to 100mg/l were prepared to develop calibration curve and made comparison with the strawberry sample. The prepared standard solutions were put into vial after syringe filtered. The standard solution analyses were performed with column C18 with mobile phase A and B. Ultra Violet HPLC detector is used to detect the ascorbic acid. The UV wavelength is set at 254 nm and the absorbance is later observed. The calibration curve is developed by plotting area under graph versus concentration of the standard solution. The calibration curve later used to calculate liner equation which relates the area of peak and concentration. The equation is later used to calculate the concentration of vitamin C in strawberry juice.

After that, sample solutions being prepared from the maltodextrine added strawberry juice. The pure strawberry juice is diluted 10 times and syringe filtered before filled into vial. Then the pure strawberry juice analysis was performed with column C18 with mobile phases A and B. The average mass of strawberry juice obtained for 200 ml of strawberry must be determined first before HPLC analysis for powdered samples. The result of spray drying shows that 200 mL Strawberry puree capable to produce around 40 gram of strawberry powder in same moisture content level from the amount added maltodextrine. Hence, the reconstituted of strawberry puree made by adding 10 gram of the powder to the 100 mL water to get same concentrated product as the original strawberry juice. The strawberry puree diluted for 10 times and syringe filtered before filled into vial. Then again the sample analyses were performed with column C18 with mobile phase A and B. The comparison is made between original strawberry pure juice and reconstituted strawberry juice which produced at different temperature. Percentage of vitamin C loss is calculated and optimum operating temperature is determined.

Table 3.6: HPLC analysis parameter

Parameter	Settings
Column	C-18(octadecyl ligand)
Detector	Ultra Violet (254 nm @ DAD signal)
Injection	1 µL
Mobile Phase	Mobile A - 0.05M potassium hydrogen phosphate (KH_2PO_4) Mobile B – 70% MeOH + 30% Mobile A
Flow Rate (mobile phase)	0.45 mL/ min
Temperature	Not controlled

3.4.3.2 Effect of Anti-Caking

The effect of anti-caking is experimented by producing a different strawberry powder. The weight of the 500 mL strawberry sample is determined. Referring to

Minister of Health and Welfare under Foodstuffs, Cosmetics and Disinfectants Act, 1972 of United States; the proposed ratio of anti-caking agent addition is 5000mg/kg solution; the specific amount according to ratio is added to the prepared sample. Calcium Carbonate had been identified as anti-caking agent for the research project. Hence, the specific amount Calcium Carbonate is added to the sample and stirred well. Later the exact procedures for strawberry spray drying are followed and new strawberry powder is produced. The powder obtained is kept at dry place and left for 3 weeks. After three weeks the texture and properties of the powder is observed.

3.4.3.3 Solubility Analysis

Solubility is determined according to the **Eastman and Moore method (1984)** and modified by **Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005).** According to method 1g of powder were added to the 100 mL distilled water and mixed at high velocity using the aid of stirrer for 5 minutes. Later the solution is placed in a centrifuge tube and centrifuged at 2600 rpm for 5 minutes. 25 mL of the centrifuged supernatant is placed in a previously weighed Petri dish and immediately oven dried at 105 ^{o} C for 5 hours. Solubility percentage is calculated by weight difference of the solid retained in the solution and mass of the powder added. The equation used to calculate the solubility of powder is shown below:

$$Solubility\ Percentage = \left(\frac{Mass\ of\ solid\ retained\ after\ drying}{Mass\ of\ Solid\ added\ X\ \frac{1}{4}} \right) X\ 100\%$$

Mass of the solids retained after drying is determined by the mass difference before and after drying. Mass of solids added is 1 g, which is added to the 100 mL distilled water. The one quarter is multiplied with the mass of solids added because only 25 mL of solution is used to be dried in oven. Thus, the solid retained indicates the amount of solids which soluble in 25mL of solution.

3.4.3.4 Sensory Evaluation

Sensory evaluation is last part of analytical experiment. Sensory evaluation conducted to determine the qualities of the powder produced. The evaluation is done by random probability. The important criteria of the powder which should be evaluated are determined and a sensory evaluation form is constructed. From the Sensory Evaluation Manual by Associate Professor Richard Mason from The University of Queensland, the sensory evaluation criteria are determined. The criteria are dividing into three parts which are appearance, texture and flavor. The sensory evaluation is conducted randomly at Universiti Malaysia Pahang KK1 cafeteria. The strawberry powder and the reconstituted strawberry juice is given randomly to the people at cafeteria and asked to evaluate the product according to criteria stated in the evaluation form. Randomly 10 people are selected and evaluation is conducted. All the results are collected for further analysis. The evaluation form of the Sensory Evaluation is attached in the appendix.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Spray Drying

4.1.1 Change of Temperature

Lab Plant Spray Dryer used to produce ready to dilute strawberry juice powder. All the condition or parameter have been controlled and set constant. The powder has been produced at different temperature above 160 ^{o}C by evaporating the water content in the sample. The results are shown below:

Table 4.1: Parameters of Spray Dryer

Set temp (^{o}C)	Inlet temp (^{o}C)	Outlet temp (^{o}C)	Volume (mL)	Time taken to dry(min)
160	160	87	200	55
170	170	91	200	70
180	180	96	200	69
190	190	103	200	71
200	200	105	200	72

The time taken for the drying of the powder is almost constant because it is influenced by the pump setting. Since, the pump setting is constant the time taken for the drying is almost the same. The time taken for the drying at 160 ^{o} C is lesser

than others because it is stopped earlier before sample finishes. This is because heat intensity at 160 ^{o} C is not sufficient to dry the sample and almost half of the sample still left as liquid with very high concentration and hygroscopic. The minimum sufficient heat for spray drying is obtained at 170^{o} C; almost all the sample is dried and free flow powder is produced. Since, spray drying mechanism allows a very short time of contact between the sprayed droplets and hot air, it is necessary to supply enough heat by hot air to dry all the droplets and change it into powder form. So, from the experiment is discovered that for laboratory scale spray dryer, 170 ^{o}C is minimum temperature for 99% of powder transformation from juice.

The amount of the powder obtained is strongly influenced by the amount of the Maltodextrine added. This is because the solid exist in the strawberry juice powder is removed completely. So, without the additional of the Maltodextrine there will be very few powder would have been obtained and very high viscosity paste like liquid is obtained. The amount of Maltodextrine added is made constant for this experiment which is 20% or 20 g per 100 mL of strawberry juice. The results of amount powder obtained are shown below:

Table 4.2: Amount of strawberry powder obtained

Sample	Inlet temp (^{o}C)	Amount of Maltodextrine	Amount of Strawberry
1	160	20 g	24.30
2	170	20 g	44.0
3	180	20 g	39.30
4	190	20 g	40.94
5	200	20 g	23.74

The amount of powder obtained varies with temperature used. This is because temperature had big influence in the process of spray drying. Hot air temperature is the only supply of heat which used to evaporate the water content and change the liquid sample into powder. As the temperature increased the heat supply carried by the hot air increased. The powder obtained at 160 ^{o} C is very less compare

to the amount of powder obtained at 170 ^{o} C. This is because the amount of heat supplied for the drying process is not sufficient to dry the continuous feed of strawberry juice. It is observed that almost half of the strawberry juice sample remains as liquid form in the drying chamber.

However, the highest amount of powder is obtained at 170 ^{o} C, this is because the heat supply is sufficient to evaporate the water content of droplet and produce free flowing strawberry juice powder. As the temperature increased it is observed that the amount of powder obtained is decreases. The lowest amount of powder obtained at 200 ^{o} C. This phenomenon happens due to the excessive heat supply for the drying process. When very high heat supplied for the drying process, the powder produced have high potential to burn because there is huge amount of heat which left after the water evaporated. Therefore, the balance heat is used to burn off the powder and it indirectly decreases the yield of the process. At temperature 180 ^{o} C and 190 ^{o} C it is observed almost same amount of powder produced. However it powder obtained is still less relative to powder obtained at 170 ^{o} C.

4.1.2 Change of Maltodextrine 10 percentage

Maltodextrine 10 which used to as carrier agent had direct influence towards the amount of powder produced. So, the amount of Maltodextrine 10 is set as manipulated variable to observe the effect of it toward the qualities of powder produced. All the other parameters are made constant including pump setting, fan setting and also temperature. It is set at 5, 20 and 170 ^{o}C respectively. The amount of powder obtained is illustrated in the table below:

$$\%\ of\ Maltodextrine = \frac{Mass\ of\ maltodextrine\ (g)}{Volume\ of\ Sample\ (mL)}\ X\ 100\ \%$$

Table 4.3: Amount of powder produce when percentage of Maltodextrine 10 varied

Sample	Inlet temp (^{o}C)	Amount of Maltodextrine	% of Maltodextrine	Amount of Strawberry
6	170	10 g	10	8.63 g
7	170	15 g	15	16.24 g
8	170	20 g	20	43.96 g
9	170	25 g	25	23.7 g
10	170	30 g	30	42.82 g

General trend shows that the amount of powder obtained is increases when percentage of Maltodextrine 10 increases. This is because the amount of solid in the sample increases when more Maltodextrine 10 is added. From the observation it is observed that Maltodextrine 10 has significant effect on the colour intensity of the powder obtained. As the percentage of Maltodextrine 10 increases the powder's colour intensity become more fade. This is because when small amount of Maltodextrine 10 used there is huge number of strawberry molecules available to be carried together and become powder. Meanwhile, when percentage of Maltodextrine 10 increases the ratio between the amounts of strawberry molecules available to be carried and Malotodextrine 10 decreases and so the colour intensity decreases.

From the observation when the amount of Maltodextrine 10 increased, the ability of the sample to dissolve Maltodextrine decreases because the sample becomes very saturated with Maltodextrine 10. 25 % and 30% of Maltodextrine 10 shows very high difficulties in dissolving although with the aid of stirrer. There is some amount of Maltodextrine 10 not actually dissolved and left without dissolving it further. From the past experience, the viscosity of the sample should not be high because it will cause clogging problem in the atomizer. Hence, 30 % of Maltodextrine 10 is not recommended for the future analytical experiments.

Figure 4.1: Strawberry Powder and reconstituted strawberry juice

4.2 Analytical Experiment

4.2.1 Vitamin C retention studies

Results of vitamin C retention in strawberry during drying and initial condition are discussed in this section. All the HPLC experimental data are presented in Appendix. The concentration of the Vitamin C before and after spray drying is measured to calculate the percentage of the loss of vitamin is the spray drying process. The powder from spray drying process at different temperature will be reconstituted and amount of vitamin C retained is observed. The calibration curve developed from the standard solution is shown below:

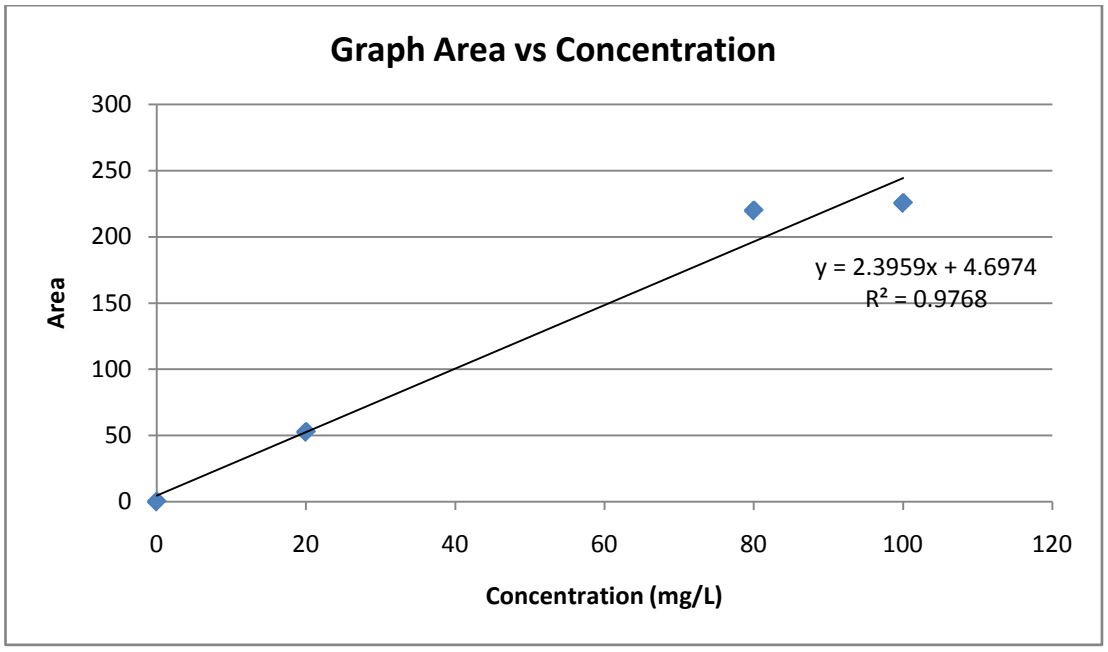


Figure 4.2: Graph Area versus Concentration

Due problem occurs in the column of the HPLC few results for the calibration is neglected. Only area under peak for 20mg/l, 80 mg/L and 100 mg/L is used to develop the calibration curve. The calibration curve is used to determine the concentration vitamin C in the reconstituted powder sample and pure sample. From the calibration equation X indicates the concentration of the sample and Y indicates the area of the peak developed during the HPLC analysis.

The amount of the vitamin C retained during the process of the spray drying at different temperature is shown in the table below at next page:

Table 4.4: Concentration of vitamin C in pure and after spray dried

Sample	Drying Temperature (^{o}C)	Amount (mg/L) of vitamin C in 10 times diluted sample	Concentration (mg/L) of vitamin C in Original strawberry puree	Amount of Vitamin C deterioration (mg/L)
Pure		113.49	1134.9	0
1	160	98.48	984.78	150.12
2	170	95.11	951.07	183.83
3	180	92.83	928.34	206.56
4	190	89.83	898.27	236.63
5	200	87.98	879.81	255.09

The vitamin C is very sensitive and most unstable nutrient found in strawberry juice. Thus, it is very sensitive to high heat or temperature. So, when strawberry juice is spray dried using high heat, it would lead to the loss of vitamin C. From the analysis it shows that the vitamin C deterioration increases as the temperature of hot air used for the drying increases. The vitamin C lost at 160 ^{o}C shows the minimal lost which are 150.12 mg/L and it increases as the temperature is increased. The maximum loss of vitamin C occurs when hot air temperature set at 200 ^{o}C which is 255.09 mg/L. The percentage of vitamin C loss is calculated and illustrated in the table below:

Table 4.5: Percentage loss of vitamin C after spray dried

Sample	Drying Temperature (^{o}C)	Concentration (mg/L) of vitamin C in Original strawberry puree	Percentage of Vitamin C loss (%)
Pure		1134.9	
1	160	984.78	13.20
2	170	951.07	16.19
3	180	928.34	18.20
4	190	898.27	20.85
5	200	879.81	22.47

The deterioration of vitamin C at 200 ^{o}C is very high because the heat supplied by the hot air is excessive until it can burn off the powder produced. Vitamin C which is very sensitive to heat is exposed to the excessive heat and so the deterioration is results of it. However, at 160 ^{o}C minimum vitamin C loss is observed because the heat supplied for the spray drying process is very less. It is observed that heat supply at 160 ^{o}C is insufficient to transfer the entire liquid sample into powder form. Therefore, at that temperature not much vitamin C loss is observed. The loss increases as the temperature increases where at 170 ^{o}C it shows 16.19 % of loss. At this temperature almost the entire liquid strawberry sample is changed into powder, however is shows just the second lowest vitamin C loss. Hence, 170 ^{o}C shows advantage for the both spray drying and also vitamin C retention. It has produced maximum yield of strawberry powder and second lowest vitamin C deterioration. Meanwhile 180 ^{o}C and 190 ^{o}C shows deterioration of 18.20 % and 20.85 % respectively.

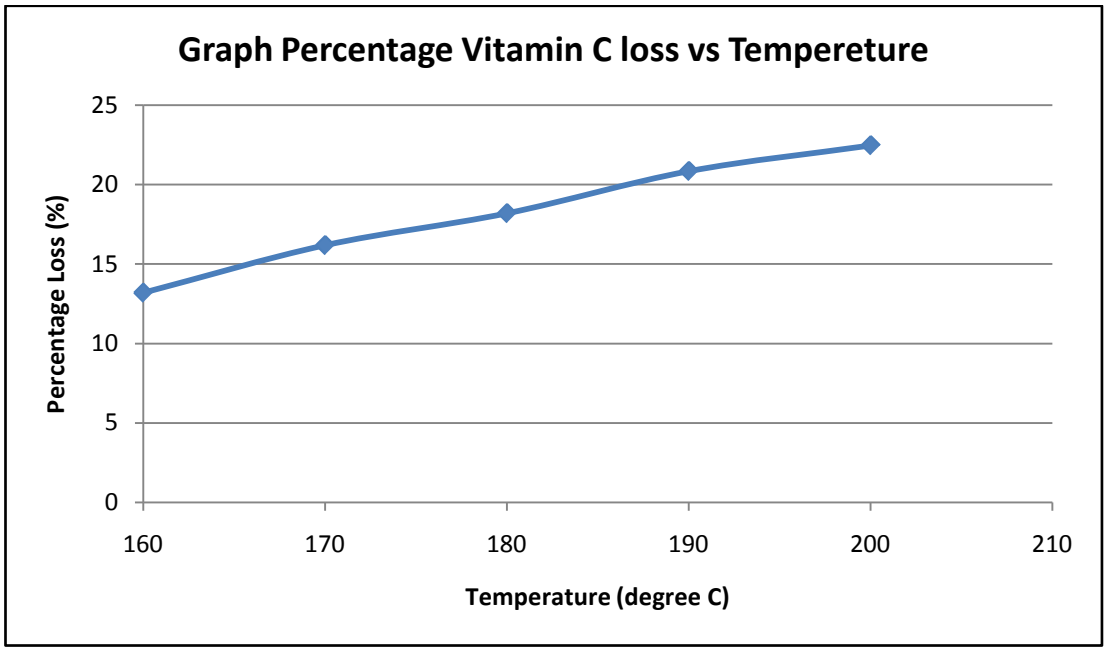


Figure 4.3: Graph Percentage Vitamin C loss versus Temperature

From the graph it is observed that the deterioration increases almost constantly as 10 ^{o} C of temperature increased. This means the amount of vitamin C deterioration each time temperature increased is the same. This is because the pump setting and fan settings are constant and the overall time taken for the spray drying

process is almost the same for all the temperatures. Hence, the contact time of the sprayed droplet and hot air is almost the same for all the experiments.

4.2.2 Anti-Caking analysis

The anti-caking analysis does not need and experiment. It needs just physical observation. The powder produced by adding the Calcium Carbonate and powder produced without adding is stored at dry place and leaved for three weeks. The results after three weeks are shown in the picture below:

Figure 4.4: Powder without anti-caking

Figure 4.5: Powder with anti-caking

From the picture we can observe that the powder without anti-caking agent become hard due very hygroscopic and looks like paste. Meanwhile the powder produced with the addition of anti-caking agent is still the same and does not become lump and absorb water. So, it is shows that the anti-caking agent helps in the storing of the powder for long time and increases the shelf life. However, the colour intensity has decreased and looks pale compare to the powder produced without anti-caking agent.

4.2.3 Solubility analysis

Solubility analysis is conducted for the powder produced using the different percentage of the Maltodextrine 10. The solubility test is conducted to evaluate the ability of the powder to dissolve in the water and effect of the Maltodextrine towards the ability of the powder to dissolve in the water. The results of the solubility analysis are illustrated in the table below:

The results of the solubility analysis show that the ability of the powder to dissolve in water is increases. This is because the mass of powder retained after drying is increases as the percentage of Maltodextrine 10 increased. The percentage of the solubility is calculated using the Eastman and Moore method (1984). The percentage of solubility for different amount of Maltodextrine 10 is illustrated in the table below.

Table 4.6: Percentage of solubility for different amount of Maltodextrine 10

Sample (percentage of Maltodextrine 10) %	Weight of boat + weight of supernatant after drying	Weight of solids retained after drying	Percentage of solubility %
10	1.512	0.195	78.0
15	1.517	0.201	80.4
20	1.497	0.211	84.4
25	1.536	0.225	90.0
30	1.521	0.226	90.4

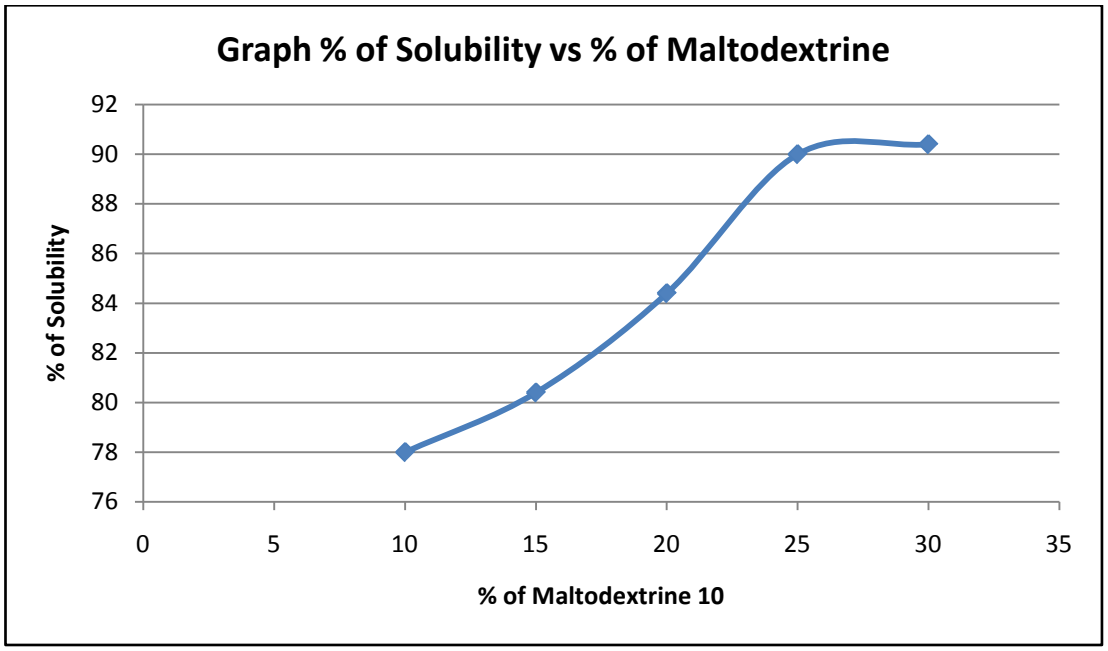


Figure 4.6: Graph percentage of solubility versus percentage of Maltodextrine 10

From the graph it is observed that the percentage of solubility increase quite rapidly as the percentage of Maltodextrine 10 is increased. The percentage of solubility become stable as it reaches the percentage of 25 and 30. This is because the strawberry juice samples become extremely saturated with Maltodextrine 10 and do not able to dissolve more of it. Hence, the amount of solids in the strawberry samples with 25 % and 30% of Maltodextrine 10 does not have much different and becomes almost the same. Therefore the percentage of solubility is almost the same to each other.

4.2.4 Sensory Evaluation

The results from the random sensory evaluation are collected and all the results are illustrated in the table below:

Table 4.7: Frequency of response for sensory evaluation

Criteria	Score Frequency				
					
Appearance					
Color			1	2	7
Size			2	4	4
Shape			2	3	5
Flavor					
Odor		1	4	3	2
Taste		1	2	2	5
Texture					
Mouth Feel		1	2	2	5
Viscosity				5	5
Total		3	13	21	33

From the results obtained from the random sensory evaluation, we can observe that the distribution of marks given for the powder produced is concentrated at highest mark which is 5. So, it is concluded that all the commercial qualities of the powders is at more than satisfactory range. The powder produced might have high potential at market since it satisfies the customers for all the required qualities for a ready to reconstituted instant strawberry juice powder. The percentage of response from the sensory evaluation survey is illustrated in the table below:

Table 4.8: Percentage of response for sensory evaluation

Score	Frequency for all criteria	Percentage (%)
1	0	0
2	3	4.3
3	13	18.6
4	21	30
5	33	47.1
Total	70	100

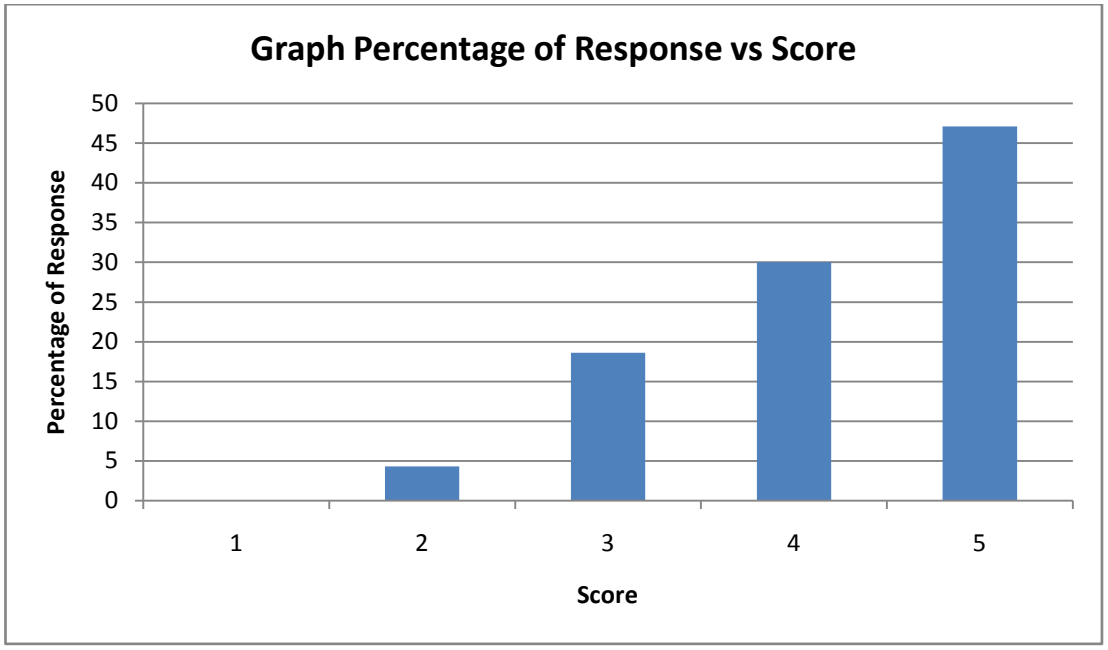


Figure 4.7: Graph percentage of response versus score

From the histogram we can observe that the score given by students for the strawberry juice powder is concentrated at score 4 and 5. This shows that the powder and the reconstituted strawberry juice powder have all the qualities with more than average range. Therefore, we can conclude that the product have high potential of being good product at market.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

At this chapter the conclusion from the experimental analysis and the results obtained will be discussed. The optimum parameter for every experiment will be selected according to the significant towards results and economical values.

5.1.1 Spray drying process

Spray drying process is divided to two sections which is drying of strawberry juice into powder at different temperature and drying of strawberry juice with addition of different percentage of Maltodextrine 10. The drying process at different temperature is to determine the effect of drying temperature on deterioration of vitamin C. Meanwhile, drying with different percentage of Maltodextrine 10 is to determine the effect of Maltodextrine 10 on solubility of the powder and also towards the texture, colour and taste of strawberry juice obtained.

5.1.1.1 Optimum Temperature

The strawberry powder produced by spray drying method is very stable at room temperature and it retained the colour of the pure strawberry juice. The reconstituted strawberry juice looks exactly the same as the pure juice. The optimum temperature for the production of the strawberry juice is at 170 ^{o} C. Maximum amount of powder have been obtained and there is no problem of powder burn addressed during the experiment. Therefore, hot air at 170 ^{o} C has provided sufficient heat for the drying process of the strawberry juice.

There is vitamin C deterioration have been identified during the spray drying process. This is because vitamin C is most unstable nutrient which found in the strawberry juice. The vitamin C concentrations have been studied and the effect of temperature has been identified towards the deterioration of vitamin C during spray drying process. It is observed that lowest vitamin C deterioration occurs at temperature 160 ^{o} C and maximum temperature deterioration observed at 200 ^{o} C. The percentage of loss of vitamin Cat 160 ^{o} C and 200 ^{o} C is 13.20% and 22.47%. As the preferences the temperature with lowest vitamin C deterioration must be selected as the optimum temperature. However, according to the experiment conducted it is observed that at 160 ^{o} C around half of the strawberry juice remains as the liquid and did not change to powder. Meanwhile, at 170 ^{o} C maximum strawberry powder is obtained which is 44 g for 200 mL of strawberry juice. It is observed that vitamin C lost at 170 ^{o} C is 3% more than observed at 160 ^{o} C.

Since, strawberry spray drying at 170 ^{o}C has advantage of getting more yields and only 3% more vitamin C deterioration, so, 170 ^{o} C is the optimum temperature for spray drying for and produces strawberry powder with maximum quality.

5.1.2 Effect of Maltodextrine 10

5.1.2.1 Effect on amount of powder obtained

Maltodextrine 10 is an important substance which used for the spray drying process. Without the additional of it strawberry juice powder can be produced. However, different percentages of Maltodextrine 10 addition to strawberry sample do have some effect to the final product. As the percentage of Maltodextrine 10 addition increased the amount of strawberry powder increases together. Meanwhile, low amount of powder obtained when lesser percentage of Maltodextrine 10 added. This is because the amount solids in the strawberry juice increase with addition of Maltodextrine 10 and so more powder obtained.

Furthermore, Maltodextrine 10 influences the colour texture of the powder produced. The powder is brighter when more Maltodextrine 10 added and darker when less Maltodextrine 10 added. So, from the experiment and observation 25% of Maltodextrine 10 is selected as the optimum percentage for the spray drying of pure strawberry juice

5.1.2.2 Effect on Solubility

Maltodextrine 10 has high influence towards the solubility of the strawberry powder. As the percentage of Maltodextrine 10 increased in the strawberry juice, the percentage of solubility also increases. This is because the nature of Maltodextrine 10 which have high solubility in water. Thus, when more Maltodextrine 10 presences in strawberry juice powder, it enables the powder to be more soluble as and solubility increases. When the Maltodextrine 10 dissolves in the water it carries together all the strawberry molecules and makes the powder to be more soluble. From the experiment it is observed that strawberry juice powder with 25 % and 30% of Maltodextrine 10 have the highest percentage of solubility. Since strawberry powders with 25% and 30% of have almost same percentage of solubility which is

90%, 25% of Maltodextrine 10 is selected as the optimum percentage of Maltodextrine for the production of strawberry juice powder.

5.1.3 Anti-caking

Calcium Carbonate has been chosen as the anti-caking agent for this research project and effect of it on shelf life has been studied. The powder without the anti-caking become lumps and absorbs huge amount of water. So, the shelf life of the powder is very short due to the presences of the water may spoilt the powder. Meanwhile the powder with the additional of anti-caking agent shows that is have extremely low hygroscopic and did not form into lump or paste. Therefore, there are no presences of water that can spoil the powder. So, indirectly the shelf life of the pure strawberry juice has been extended with retaining all the qualities of the pure strawberry juice. Besides that, is also has reduced the problem of fruits injuries and transportation cost significantly.

5.1.4 Sensory evaluation

Sensory evaluation is conducted to identify the response of people towards the strawberry juice powder produced. From the sensory evaluation it can be concluded that the strawberry juice powder produced have meet the requirement of the customers and have potential of being marketed in the future. All the qualities are above the satisfactory range and preservation technique using spray drying is an essential method.

5.2 Recommendation

Food preservation is an important method to increase the shelf life of the food. There are varieties of methods available for the preservation purposes according to the characteristic of the food. However, all the techniques faces problem of retaining the freshness and the qualities of food as it is fresh. Among the problem the crucial part in food preservation is nutrient retention, it is important to minimize the nutrient loss during the food processing. There are a lot of factors that can cause nutrient lost namely high temperature, mechanical processing, additional of chemicals and etc.

Anti-caking is an important material used to increase the shelf life of the powder produced. However the usage of anti-caking should be correct and suitable with the characteristic of the food. Therefore, further research on effect of anti-caking on the nutrient should be conducted. This is to identify the specific anti-caking agent that should be used according to characteristic of the food. Now, it is recommended some anti caking agents which is suitable for a group of food and not the specific food like strawberry juice. Therefore, a further research on effect and suitability of specific anti-caking is essential to produce high quality preserved food.

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APPENDIX A
HPLC RESULTS

Data File C:\CHEM32\1\DATA\VIT C 08\150109000001.D
Sample Name: std 1

Ascorbic acid 20mg/l

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Injection Date  : 1/16/2009 10:31:28 AM          Seq. Line :   1
Sample Name     : std 1                          Location : Vial 1
Acq. Operator   : idayu/Jaganrao                     Inj :   1
Acq. Instrument : Instrument 1                 Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 4/1/2008 3:11:47 PM by idayu/nabila
Analysis Method : C:\CHEM32\1\METHODS\VIT C 08.M
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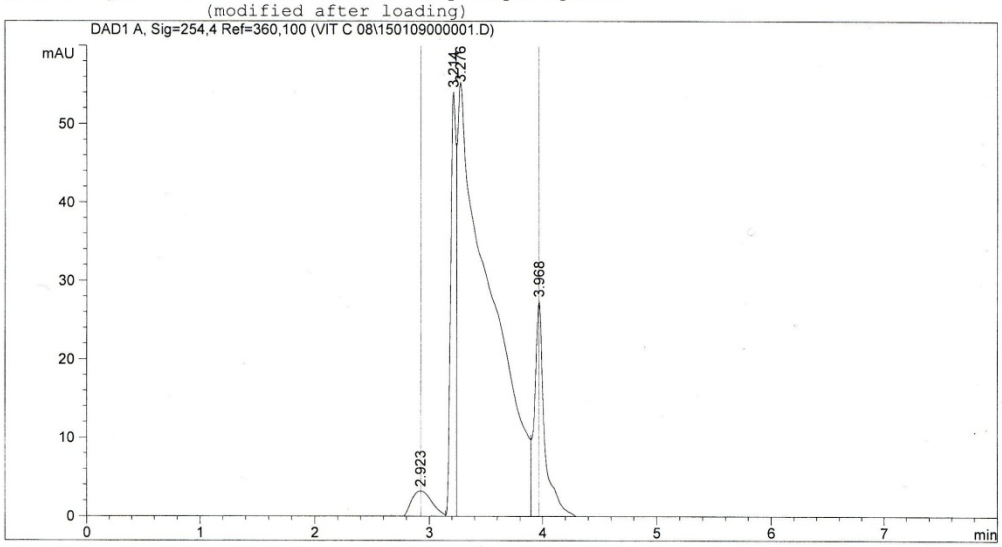

Area Percent Report

```
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.923	BV	0.2142	50.70716	3.69375	3.2963
2	3.214	VV	0.0503	179.55830	54.66808	11.6723
3	3.276	VV	0.2468	1117.18445	55.97575	72.6235
4	3.968	VB	0.0930	190.87442	28.17492	12.4079
Totals :				1538.32433	142.51248	

Appendix A1: HPLC results for 20mg/L standard Ascorbic Acid solution

Data File C:\CHEM32\1\DATA\VIT C 08\150109000002.D
Sample Name: std 2

Ascorbic acid 40mg/l

Injection Date	: 1/16/2009 10:44:27 AM	Seq. Line :	2
Sample Name	: std 2	Location :	Vial 2
Acq. Operator	: idayu/Jaganrao	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
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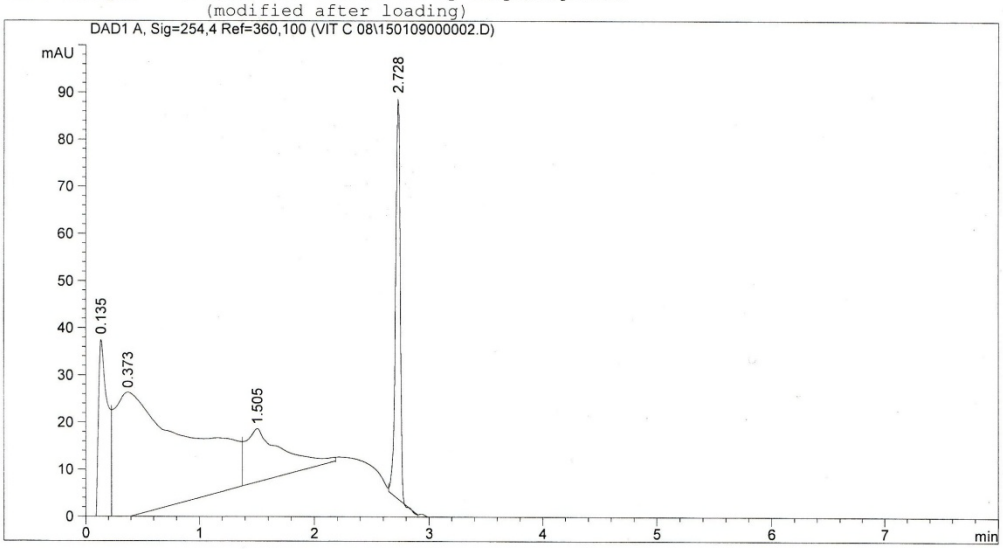

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.135	BV	0.0798	222.67352	39.46986	11.9560
2	0.373	VV	0.5475	1147.97253	26.49661	61.6382
3	1.505	VB	0.2775	250.52965	11.35154	13.4517
4	2.728	BV	0.0450	241.26262	85.26096	12.9541
Totals :				1862.43832	162.57897	

*** End of Report ***

Appendix A2: HPLC results for 40mg/L standard Ascorbic Acid solution

Data File C:\CHEM32\1\DATA\VIT C 08\150109000003.D
Sample Name: std 3

Ascorbic acid 60mg/l

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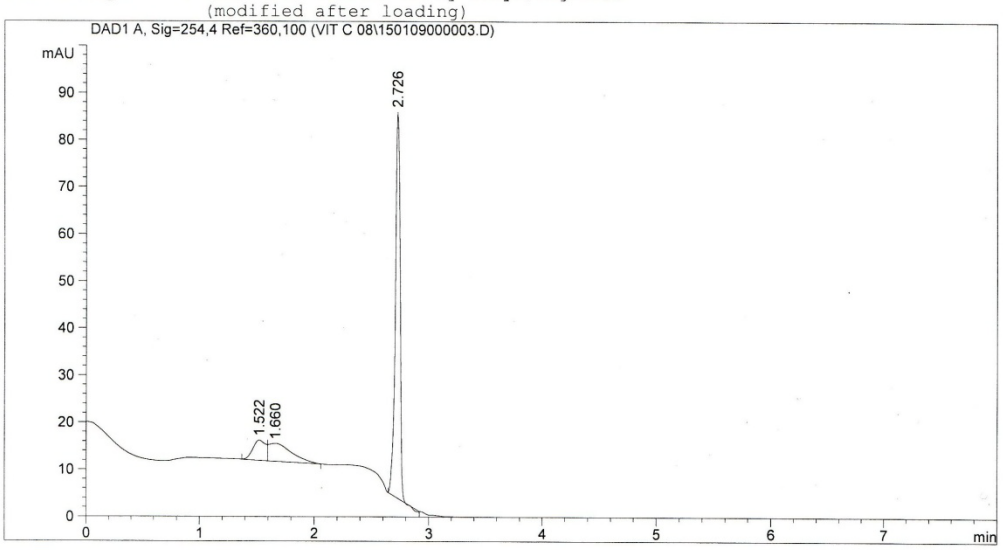
Acq. Instrument	: Instrument 1	Inj Volume :	1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:38:17 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.522	BV	0.1136	33.06215	4.30620	10.2325
2	1.660	VB	0.2021	52.68423	3.79186	16.3054
3	2.726	BB	0.0457	237.36343	82.16055	73.4622
Totals :				323.10982	90.25862	

*** End of Report ***

Appendix A3: HPLC results for 60mg/L standard Ascorbic Acid solution

Data File C:\CHEM32\1\DATA\VIT C 08\150109000004.D
Sample Name: std 4

Ascorbic acid 80mg/l

```
Injection Date  : 1/16/2009 11:10:26 AM          Seq. Line :   4
Sample Name     : std 4                          Location : Vial 4
Acq. Operator   : idayu/Jaganrao                     Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 4/1/2008 3:11:47 PM by idayu/nabila
Analysis Method : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 1/22/2009 9:38:17 AM by idayu/Jaganrao
                  (modified after loading)
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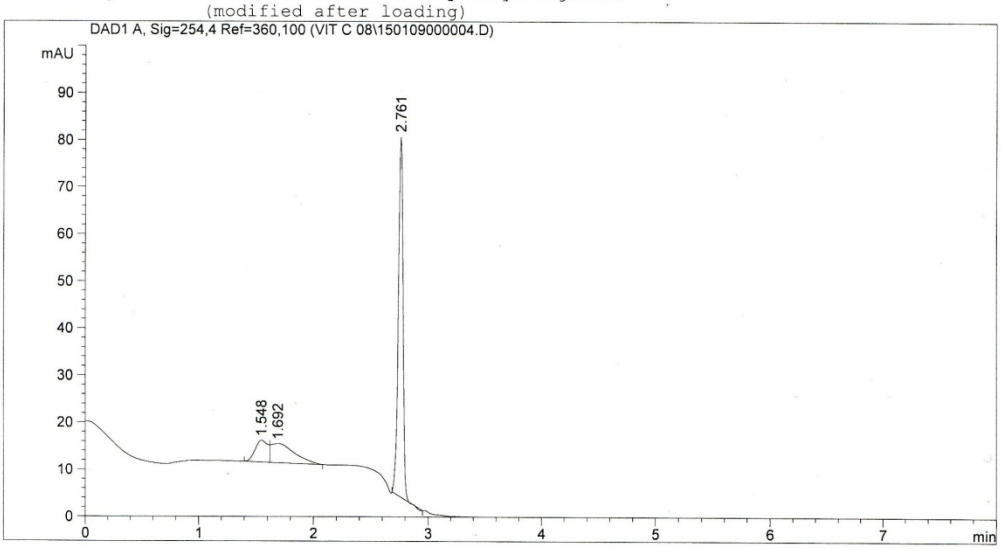

Area Percent Report

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Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.548	BV	0.1141	35.97667	4.66081	11.4669
2	1.692	VB	0.2049	57.90204	4.09539	18.4552
3	2.761	BB	0.0455	219.86513	76.66154	70.0779
Totals :				313.74384	85.41773	

*** End of Report ***

Appendix A4: HPLC results for 80mg/L standard Ascorbic Acid solution

Data File C:\CHEM32\1\DATA\VIT C 08\150109000005.D
Sample Name: std 5

Ascorbic acid 100mg/l

```
Injection Date  : 1/16/2009 11:23:26 AM          Seq. Line :   5
Sample Name     : std 5                          Location : Vial 5
Acq. Operator   : idayu/Jaganrao                     Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 4/1/2008 3:11:47 PM by idayu/nabila
Analysis Method : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 1/22/2009 9:38:17 AM by idayu/Jaganrao
                  (modified after loading)
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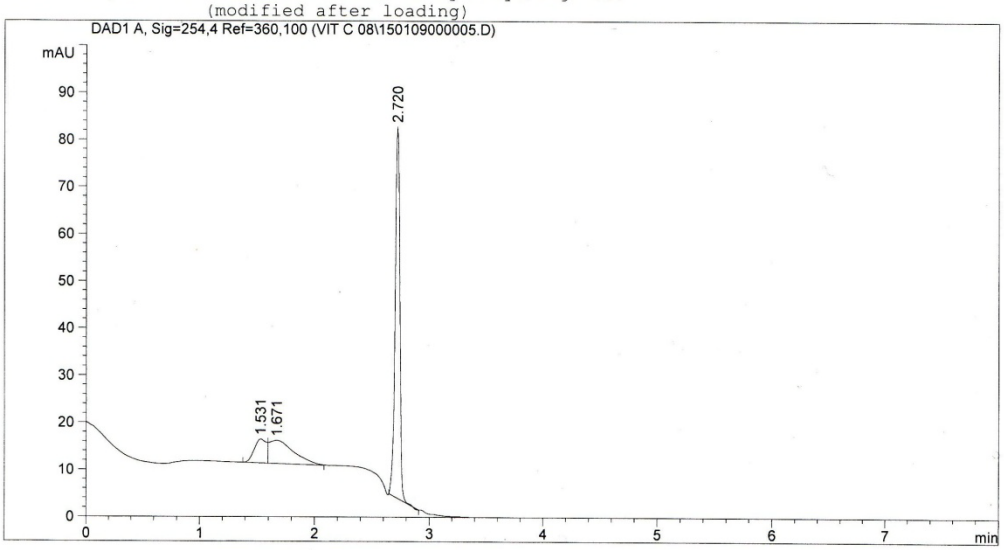



```
                          Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   1.531 BV    0.1087   36.95130    5.08952  11.0164
   2   1.671 VB    0.2143   72.97704    4.94165  21.7568
   3   2.720 BB    0.0454  225.49400   78.88040  67.2269

Totals :                    335.42234   88.91157


                          *** End of Report ***
```

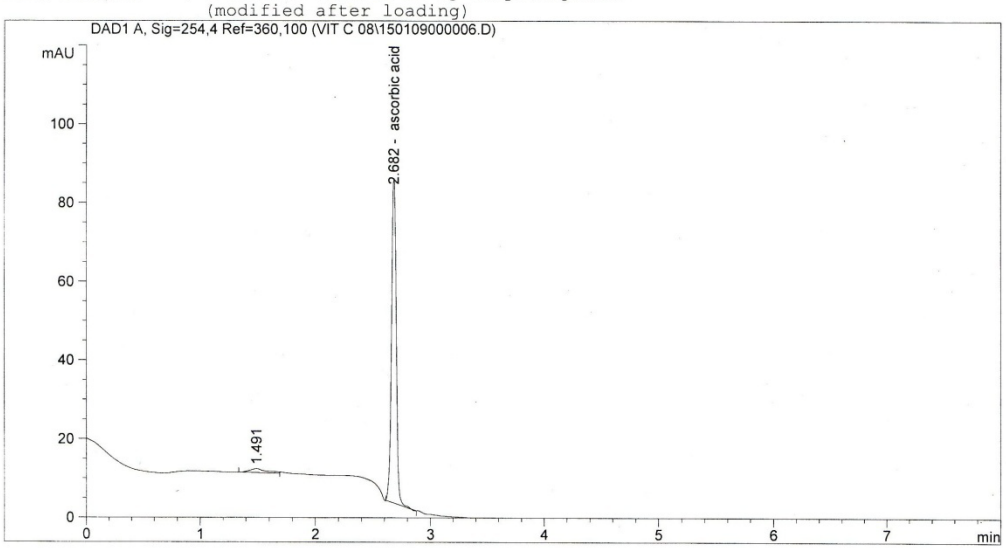
Instrument 1 1/22/2009 9:39:14 AM idayu/Jaganrao Page 1 of 1

Appendix A5: HPLC results for 100mg/L standard Ascorbic Acid solution

Data File C:\CHEM32\1\DATA\VIT C 08\150109000006.D
Sample Name: SAMPLE 1

Pure Strawberry Juice

Injection Date	: 1/16/2009 11:36:26 AM	Seq. Line :	6
Sample Name	: SAMPLE 1	Location :	Vial 6
Acq. Operator	: idayu/Jaganrao	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:53:32 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By : Signal
Calib. Data Modified : 1/22/2009 9:53:18 AM
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	1.491	BB	0.1407	10.12117	4.1023	?
2	2.682	BB	0.0453	236.59897	95.8977	ascorbic acid
Totals :				246.72013		

*** End of Report ***

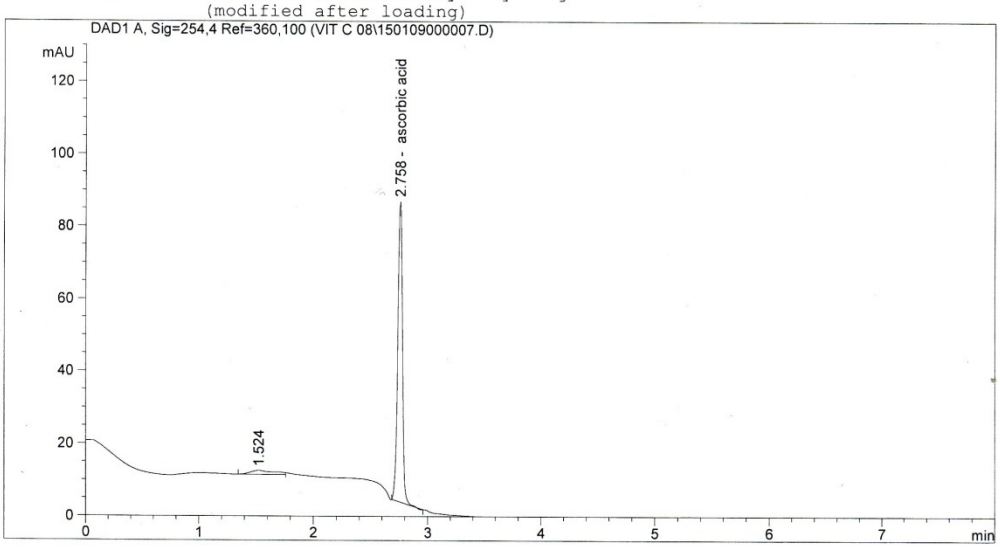
Instrument 1 1/22/2009 9:53:45 AM idayu/Jaganrao Page 1 of 1

Appendix A6: HPLC results for pure strawberry juice

Data File C:\CHEM32\1\DATA\VIT C 08\150109000007.D
Sample Name: SAMPLE 2

Strawberry Juice at 160 Degree C

Injection Date	: 1/16/2009 11:49:26 AM	Seq. Line	: 7
Sample Name	: SAMPLE 2	Location	: Vial 7
Acq. Operator	: idayu/Jaganrao	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:54:29 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Calib. Data Modified	:	1/22/2009 9:53:18 AM
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	1.524	BB	0.1922	15.79431	6.1591	?
2	2.758	BB	0.0477	240.64255	93.8409	ascorbic acid
Totals :				256.43686		

*** End of Report ***

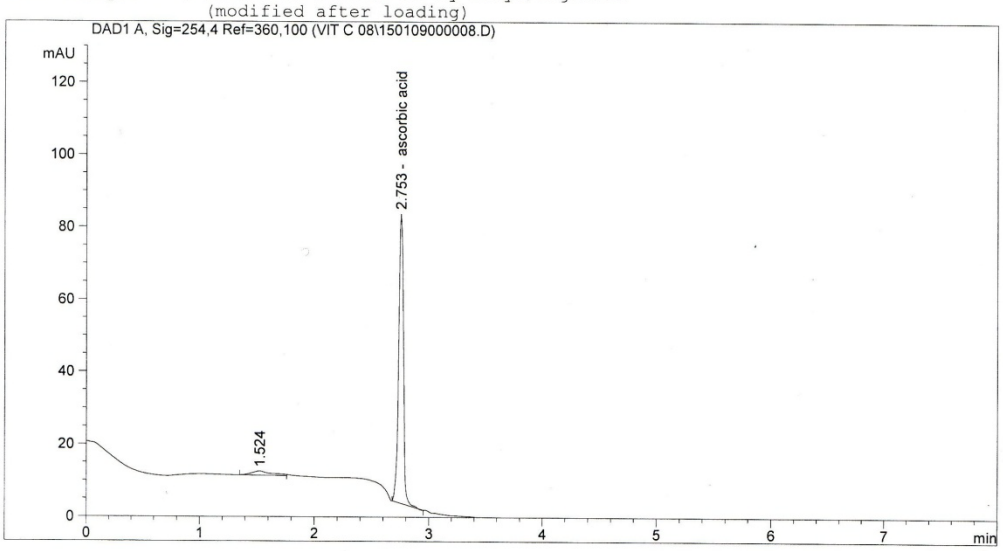
Instrument 1 1/22/2009 9:55:25 AM idayu/Jaganrao Page 1 of 1

Appendix A7: HPLC results for powder spray dried at 160 ^{o}C

Data File C:\CHEM32\1\DATA\VIT C 08\150109000008.D
Sample Name: SAMPLE 3

Strawberry Juice at 170 Degree C

Injection Date	: 1/16/2009 12:02:27 PM	Seq. Line :	8
Sample Name	: SAMPLE 3	Location :	Vial 8
Acq. Operator	: idayu/Jaganrao	Inj :	1
Acq. Instrument	: Instrument 1	Inj Volume :	1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:54:29 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By : Signal
Calib. Data Modified : 1/22/2009 9:53:18 AM
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	1.524	BB	0.1671	13.89168	5.6366	?
2	2.753	BB	0.0459	232.56531	94.3634	ascorbic acid
Totals :				246.45699		

*** End of Report ***

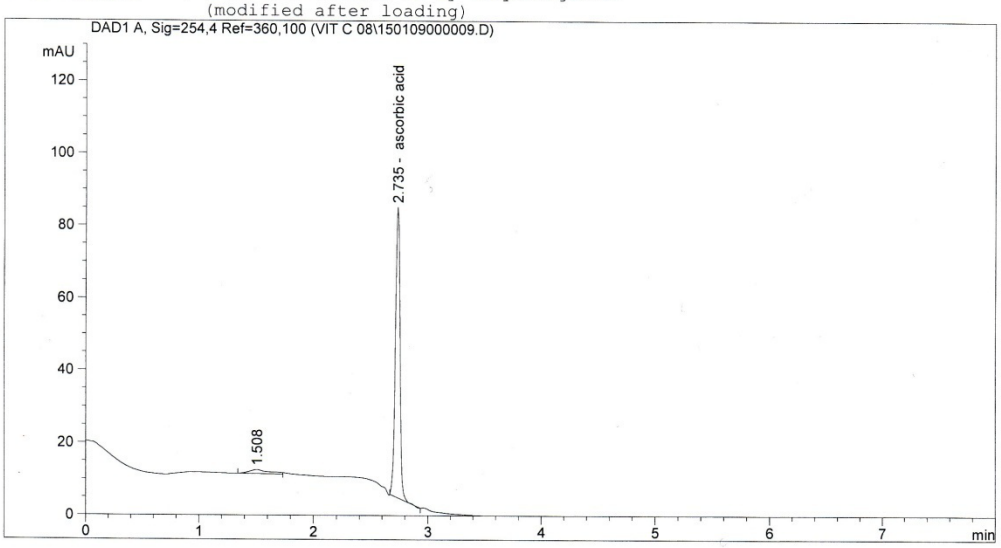
Instrument 1 1/22/2009 9:55:58 AM idayu/Jaganrao Page 1 of 1

Appendix A8: HPLC results for powder spray dried at 170 ^{o}C

Data File C:\CHEM32\1\DATA\VIT C 08\150109000009.D
Sample Name: SAMPLE 4

Strawberry Juice at 180 Degree C

Injection Date	: 1/16/2009 12:15:26 PM	Seq. Line	: 9
Sample Name	: SAMPLE 4	Location	: Vial 9
Acq. Operator	: idayu/Jaganrao	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:54:29 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By	:	Signal
Calib. Data Modified	:	1/22/2009 9:53:18 AM
Multiplier	:	1.0000
Dilution	:	1.0000

Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	1.508	BB	0.1716	13.32963	5.5434	?
2	2.735	BB	0.0450	227.13068	94.4566	ascorbic acid
Totals :				240.46031		

*** End of Report ***

Appendix A9: HPLC results for powder spray dried at 170 ^{o}C

Data File C:\CHEM32\1\DATA\VIT C 08\150109000010.D
Sample Name: SAMPLE 5

Strawberry Juice at 190 Degree C

```
==========================================================================
Injection Date  : 1/16/2009 12:28:27 PM          Seq. Line :  10
Sample Name     : SAMPLE 5                        Location : Vial 10
Acq. Operator   : idayu/Jaganrao                      Inj :  1
Acq. Instrument : Instrument 1                 Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 4/1/2008 3:11:47 PM by idayu/nabila
Analysis Method : C:\CHEM32\1\METHODS\VIT C 08.M
Last changed    : 1/22/2009 9:58:41 AM by idayu/Jaganrao
                  (modified after loading)
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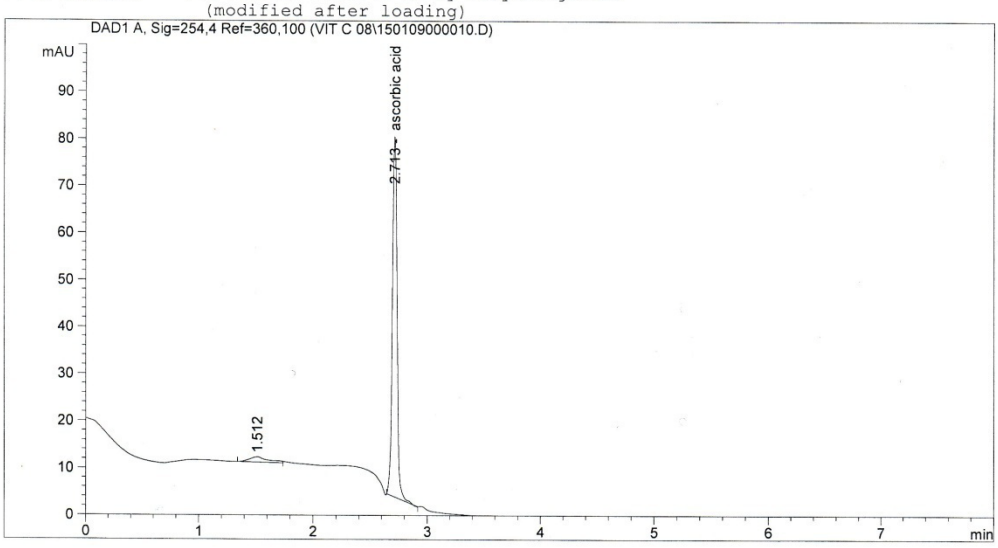



```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Calib. Data Modified  :      1/22/2009 9:53:18 AM
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime  Type   Width     Area      Area    Name
  #   [min]          [min]   [mAU*s]       %
----|-------|------|-------|----------|--------|--------------------
   1   1.512 BB      0.1603   12.83126   5.5130 ?
   2   2.713 BB      0.0453  219.91539  94.4870 ascorbic acid

Totals :                    232.74665

==========================================================================
                          *** End of Report ***
```

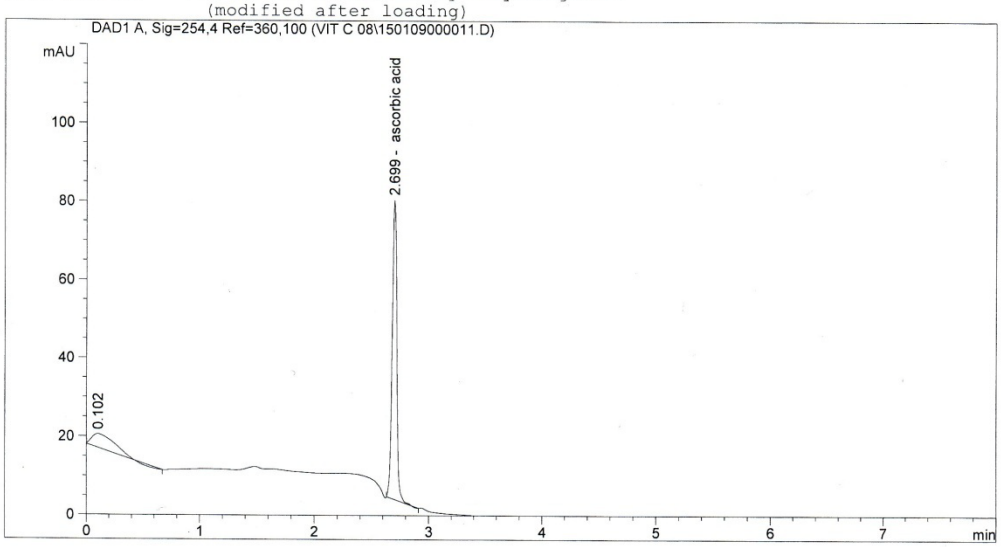
Instrument 1 1/22/2009 9:58:47 AM idayu/Jaganrao Page 1 of 1

Appendix A10: HPLC results for powder spray dried at 190 ^{o}C

Data File C:\CHEM32\1\DATA\VIT C 08\150109000011.D
Sample Name: SAMPLE 6

Strawberry Juice at 200 Degree C

Injection Date	: 1/16/2009 12:41:28 PM	Seq. Line	: 11
Sample Name	: SAMPLE 6	Location	: Vial 11
Acq. Operator	: idayu/Jaganrao	Inj	: 1
Acq. Instrument	: Instrument 1	Inj Volume	: 1 µl
Acq. Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 4/1/2008 3:11:47 PM by idayu/nabila		
Analysis Method	: C:\CHEM32\1\METHODS\VIT C 08.M		
Last changed	: 1/22/2009 9:59:41 AM by idayu/Jaganrao (modified after loading)		



Area Percent Report

Sorted By : Signal
Calib. Data Modified : 1/22/2009 9:53:18 AM
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Area %	Name
1	0.102	BB	0.1832	47.64671	18.1070	?
2	2.699	BB	0.0448	215.49272	81.8930	ascorbic acid
Totals :				263.13943		

*** End of Report ***

Instrument 1 1/22/2009 9:59:46 AM idayu/Jaganrao Page 1 of 1

Appendix A11: HPLC results for powder spray dried at 200 ^{o}C