THE EFFECTS OF PROCESS PARAMETERS ON CONCENTRATION OF DRAGON FRUIT JUICES VIA FREEZE CONCENTRATION TECHNIQUE

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ABSTRACT

Dragon fruit is one of the most nutritious and exotic fruit that is available in Malaysia. In this research, dragon fruit juice was concentrated by using freeze concentration process. Concentrated fruit juice can be preserved longer than non concentrated fruit juice. Besides, due to its small capacity storage, concentrated fruit juice also can minimize the transportation cost to import or export it. Freeze concentration process was used because it eliminates water from the juice at temperature below the water's freezing point, thus preserving the aromatic compound that originally contained in the fruit juice. In addition to research objective, two process parameters and its effect on the fruit juice final concentration were studied. The process parameters were stirring rate and cooling medium temperature. The apparatus used to done the freeze concentration process was crystallizer unit. Dragon fruit juice that was blended and standardized its initial concentration was put into the crystallization unit. The fruit juice spent 12 hours in the crystallizer unit. During the time spent in the crystallizer unit, stirring rate and cooling medium temperature were varied in the range of (600, 800, 1000, 1200 and 1400) rpm and (-6, -8, -10, -12 and -14) °C. To determine the best stirring rate, the temperature of the cooling medium in the crystallizer unit was constant at -10 °C while the stirring rate of the fruit juice was varied. Then, the best stirring rate was used to determine the best cooling medium temperature. The dragon fruit juice initial concentration was standardized at 7 °Brix. After 12 hour spent in the crystallization unit, the concentration of the fruit juice increased. For the first study where the temperature of the cooling medium was remaining constant, the highest concentrated fruit juice was 17 °Brix. The best stirring rate to produce that concentrated fruit juice was 1200 rpm. For the second study where the stirring rate was remaining constant, the highest concentrated fruit juice also was 17 °Brix. The best cooling medium temperature to produce that particular concentrated fruit juice was -10 °C. As a conclusion, the highest concentrated fruit juice produced was 17 °Brix. The best stirring rate was 1200 rpm while the best cooling medium temperature was -10 °C.

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ABSTRAK

Buah naga merupakan salah satu buah yang paling berkhasiat dan eksotik yang boleh didapati di Malaysia. Kajian ini berkisar tentang kaedah pemekatan jus buah naga dengan menggunakan teknik pembekuan. Objektif kajian ini adalah untuk mengkaji kesan kadar kacau serta suhu medium penyejukan tehadap kepekatan akhir yang boleh dicapai oleh jus buah naga. Jus buah yang telah dipekatkan boleh disimpan lebih lama berbanding juas buah yang belum dipekatkan. Selain itu, kos pengangkutan untuk eksport atau import jus buah yang dipekatkan dapat dikurangkan kerana ia memerlukan ruang yang sedikit untuk menyimpannya berbanding jus buah yang belum dipekatkan. Teknik pembekuan digunakan untuk memekatkan jus buah kerana teknik in menyingkirkan air daripada jus buah pada suhu dibawah takat beku air. Justeru, khasiat jus buah dapat dipelihara dengan cara ini. Dalam kajian ini,kesan dua parameter terhadap kepekatan akhir jus buah telah dikaji. Kedua - dua parameter tersebut adalah kadar kacau dan suhu medium penyejukan. Unit pengkristalan telah digunakan untuk mengakaji kedua - dua parameter tersebut. Sebagai permulaan, buah naga segar telah dikisar dan ditapis untuk memperolehi jusnya. Setelah itu, air suling telah ditambah kedalam jus buah tersebut untuk mematikan kepekatan awal jus buah seragam pada 7 °Brix. Setelah itu, jus buah akan dimasukkan ke dalam unit kepekatan dan dibiarkan didalamnya selama 12 jam. Selama berada didalam unit pengkristalan, jus buah naga disejukkan pada suhu yang seragam iaitu -10 °C. Selain itu, jus buah juga dikacau dalam kadar (600, 800, 1000, 1200 and 1400) putaran seminit. Selepas selesai, kadar kacau yang terbaik akan digunakan untuk eksprimen kedua dimana, kadar kacau tersebut akan dimalarkan pula. Sementara itu, suhu medium penyejukan akan dimanipulasikan pula dalam julat (-6, -8, -10, -12 and -14) °C. Hasil daripada kajian, kepekatan tertinggi jus buah yang dicatatkan adalah 17 °Brix. Kepekatan ini diperolehi pada kadar kacau 1200 putaran seminit. Justeru, kadar kacau terbaik adalah 1200 putaran seminit. Sementara itu, suhu medium penyejukan yang terbaik adalah -10 °C kerana pada suhu ini, jus buah dapat dipekatkan pada kepekatan tertinggi iaitu 17 °Brix. Kesimpulannya, kepekatan jus buah yang tertinggi dapat dicatatkan adalah 17 °Brix. Kadar kacau yang terbaik adalah 1200 putaran seminit manakala suhu medium penyejukan yang terbaik adalah -10 °C.

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LIST OF SYMBOLS

°Brix	Degree of Brix
°C	Degree of Celsius

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LIST OF ABBREVIATIONS

$C_2H_6O_2$	Ethylene glycol
HPLC	High-Performance Liquid Chromatography
KH_2PO_4	Potassium hydrogen phosphate
Ppm	Part per million
Rpm	Rotation per minute
SOLTEQ	Equipment for Engineering Education & Research

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Dragon fruit is one of the most nutritious and exotic fruit that is available in Malaysia. It has been plant commercially in Kuala Pilah and Sitiawan region (Peter, 2008). It can be eaten as a desert or raw. Sometimes, people also make a juice with it. Although it can be considered as a healthy fruit, it is not very popular among Malaysian consumer. The most famous product of dragon fruit is production of wine. Despite that, there are variety types of product that can be produced from the dragon fruit. For example, the fruit can be blend to get the fruit's juice. Then it can be concentrated to produce other different products such as jams, canned fruits, beverages and some baked goods.

Based on 2010, 89.8% of tropical fruits that has been exported from Malaysia to Europe are come from dragon fruit and star fruit. Furthermore, the production value of this fruit per year on 2006 is Rm 12,670,755 and is expected to continue rising for 2010 (Cheah and Zulkarnain, 2008). Based on this fact, it shows an increasing demand of dragon fruit due to its very nutritious content. This fruit can be considered as other alternative for natural antioxidant consumption for human body. This shows that this fruit has a very high commercial value if it has been marketed wisely.

There are many types of processes that can concentrate a non concentrated liquid product. Industry nowadays used processes such as freeze concentration process and vacuum evaporation process. Both of this process can be considered as the crystallization

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technique based on its technique that crystallizes the water particle to produce a concentrated product. The differences of these two processes are that freeze concentration process operated at low temperature in order to freeze the water particle while vacuum evaporation operated at high temperature. Hence, in order to preserve the aroma and volatile compound of the liquid product, freeze concentration process can be considered as the best concentration technique to get the optimal product quality of heat-sensitive food (Sanchez et al., 2010).

1.2 PROBLEM STATEMENT

The conventional technique used to concentrate the fruit juice is by using vacuum evaporation process. This technique operated at high temperature in order to vaporize the water content of fruit juice to concentrate it. Thus, due to its high operating temperature, some of the volatile component in the fruit juice are lost to the vapor phase and must be added back to the fruit juice. Other method that has been developed to overcome this problem is by using membrane process. This process operated at normal temperature and pressure. However, the major drawback of this process is the needs of frequent replacement. The membrane needs to be replaced in order to maintain the efficiency of the membrane to produce the product with high quality.

To tackle the entire above major problem, freeze concentration technique is used. Freeze concentration process offer significant advantages over the other concentrating method such as membrane process and vacuum evaporation. Freeze concentration operated at low temperature and requires only simple equipment to run it (Hartel, 2001). Besides, it works at normal atmospheric pressure and has average energy consumption. The major advantage of this technique is that there is no losses of volatile flavor component as well as no thermal degradation occur compared to other technique (Ashurst, 2005 and Hartel, 2001). Thus, freeze concentration process can be described as an economic process for aroma-rich liquid foods.

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1.3 RESEARCH OBJECTIVES

The purpose of this research is to examine the effect of process parameters which are stirring rate and cooling medium temperature during concentrating dragon fruit juice by using freeze concentration technique.

1.4 RESEARCH SCOPE

In order to achieve the objectives of this research, several scope of study has been determined. The scopes of study are:

i) To determine the effect of stirring rate on the concentration of dragon fruit juice by varying the stirring rate in the range of 600 to 1400 rpm.

ii) To determine the best operating temperature yield the highest concentration of dragon fruit juice by varying the temperature in the range of -6 to -14 °C.

iii) To analyze the active component in fruit juice by using high-performance liquid chromatography (HPLC).

1.5 RATIONALE AND SIGNIFICANCE

There are several advantages of the concentrated fruit juice product. For example, there are many other products that can be produced from the concentrated fruit juice such as jams and beverages. For the seasonal fruit, the production of the fruit can be limited for several month of the year. Hence, to counter the drawback, the industrial processing this fruits into juices as well as concentrated it as an alternative to enabling their production during their non-production season. Furthermore, concentrated fruit juices also minimize the storing capacity as well as the transportation cost due to their small capacity storage.

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Moreover, concentrated liquid product can be preserve longer than the non concentrated product due to their low of water content compared to the non concentrated product.

Dragon fruit also a seasonal fruit. Thus, by concentrating the fruit juice, it can be preserve for a longer time to provide a backup for the increasing demand in the market. This fruit contain high level of antioxidant content such as Vitamin C in it. Thus, it required proper technique to concentrate it. Without the proper technique, the original characteristics of the fresh fruits can be diminish, thus lower the quality of the concentrated product. Hence, freeze concentration process is chose as the method to concentrate the dragon fruit juice.

Freeze concentration process is used in this study to concentrate the dragon fruit juice. It is widely recognized that this process is a low-temperature operation that causes no thermal degradation reactions to occur as well as the flavor quality is much more superior to the flavor of evaporated product. This process can retain the original characteristics of the fruit juice, thus produce the concentrated liquid product with high quality. By doing this study, the best working environment for freeze concentration process can be studied so that it can lead in mass production of concentrated dragon fruit juice in the future.

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CHAPTER 2

LITERATURE REVIEW

2.1 OVERVIEW OF DRAGON FRUIT

Dragon fruit, also known as pitaya fruit or pitahaya fruit, is among the most nutritious and wonderful exotic fruit. It is a climbing vine cactus species which has received worldwide recognition, first as an ornamental plant and then as a fruit crop. It is native to Central America and requires tropical climate to be grown successfully and fruitful. Thus, based on the nation climate, growing dragon fruit commercially is common in country like Colombia and Nicaragua. Malaysia and Vietnam is another example of Asian countries that grows the dragon fruit for commercial purpose. Usually, dragon fruit only bloom at night and can be harvested around June to December as it is a seasonal type fruit (Peter, 2008).

Generally, there are three most common varieties of dragon fruit that is being cultivated for commercial purposes which are:

i) Hylocereus undatus: Dragon fruit with red skin and white flesh.

ii) Hylocereus polyrhizus: Dragon fruit with red skin and red flesh.

iii) Hylocereus megalanthus: Dragon fruit with yellow skin and white flesh.

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(a) *Hylocereus undatus* (b) *Hylocereus polyrhizus* (c) *Hylocereus megalanthus*

Figure 2.1: Colour features distinguishing the Hylocereus varieties

Source: Ariffin et al. (2009)

Figure 2.1 shows the colour features distinguishing the Hylocereus varieties. Dragon fruit taste mildly sweet and can be eaten raw as a fresh fruit, where 70-80% of the fruit is edible. All dragon fruit varieties have flesh filled up with tiny black seedlings and the seeds are edible with the flesh. Thus, eating the fruit is likened to kiwi fruit in terms of their flavor as well as their fruit pulp texture. According to Peter (2008), dragon fruit also "can be processed into a range of industrial products such as juice, sherbets, jam, syrup, ice cream, yogurt, jelly, preserve, candy and pastries". In Malaysia, dragon fruit is famous in the wine making industry and is widely used to produce a high quality wine.

2.1.1 Nutritional Information

There are so many health benefit of this fruit that make this fruit can be considered as "health fruit". For example it is rich with antioxidant. "It prevents colon cancer and diabetes, neutralizes toxic substances like heavy metal, reduces cholesterol and high blood pressure" (Peter, 2008). On top of that, dragon fruit also contain highly concentrated Vitamin C that can improve human body's general immune system. Besides that, dragon fruit also can help controls human blood glucose level and will make a perfect diet for a diabetes patient. Other health benefit is that it can help in the human's digestion system, help to develop strong teeth and bones, and helping lowering the bad cholesterol. The

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typical nutritional value per 100g of raw dragon fruit (of which 55g are edible) is shown in Table 2.1.

Table 2.1: Typical nutritional value per 100g of raw dragon fruit

Nutritious	Weight
Water	80-90 g
Carbohydrates	9-14 g
Protein	0.2-0.5 g
Fat	0.1-0.6 g
Fiber	0.3-0.9 g
Ash	0.4-0.7 g
Calories	35-50 g
Calcium	6-10 g
Iron	0.3-0.7 mg
Niacin (Vitamin B_3)	0.2-0.45 mg
Ascorbic acid (Vitamin C)	4-25 mg
Carotene (Vitamin A)	traces
Riboflavin (Vitamin B_2)	traces

Source: Ariffin et al. (2008)

2.2 CRYSTALLIZATION THEORY

According to Geankoplis (2003), "Crystallization is a process where solid particles are formed from the homogeneous solution". This process can occur in the formation of snow particle from a vapor and the formation of solid particles from a liquid solution (Geankoplis, 2003). Crystallization process consists of two major processes which are nucleation (nucleus formation) and crystal growth. These two processes occur concurrently in the crystallization process. When nuclei are formed and grow, crystals are created. The

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kinetic processes of nucleation and crystal growth require supersaturation. It can be thought of as the concentration of solute in excess of solubility. It is the driving force of the crystallization, thus, the rate of nucleation and growth is driven by the existing supersaturation in the solution. Generally, there are three ways to achieve supersaturation which are, first, by a change in temperature (cooling or heating), second, by removing the solvent (usually by evaporation), and lastly by adding a drowning-out agent or reaction partners. Further explanation of supersaturating state will be discussed in Section 2.4.3.

Nucleation process can be divided into two categories which are homogeneous and heterogeneous nucleation. Homogeneous nucleation involves the spontaneous formation and subsequent growth of small particles of the new phase in a supersaturated solution (birth of a new crystal). This process involves only the one material, thus it is intrinsic to the material. Compared to homogeneous, heterogeneous nucleation occurs when the new phase is initiated on a foreign material such as particle or a surface layer. As for crystal growth, it is the subsequent growth of the nuclei that succeed in achieving the critical cluster size.

Based on this theory, crystallization technique is widely used by industry to concentrate the fruit juice. There are several industrial scale concentration techniques that are been used based on this theory including freeze concentration process and vacuum evaporation process (Hernandez et al., 2009).

Concentrated liquid food have several advantages such as minimizing the storage size, lowering the transportation cost due to its small storage size and for making a powder product, it is better to use the high concentrated liquid food rather than using the lower one. Other than that, concentrated liquid product also can be considered as a way of preserving food because it contain low amount of water compared to the non concentrated liquid product, thus reducing the water activity in that liquid product (Onsekizoglu et al., 2010).

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In the commercial crystallization, there are several things that are often desirable by the industry such as the yield and the purity of the ice crystal. Other than that, to minimize the caking in the package as well as for ease in washing and filtering, it is often desirable that the crystals that are formed to be uniform in size (Geankoplis, 2003). Vacuum evaporation process can give higher concentrated product compared to freeze concentration technique (Ribeiro et al., 2007 and Garg et al., 2009). On the other hand, freeze concentration technique can produce the more quality product based on its method compared to vacuum evaporation technique (Ashurst, 2005). Thus, for producing a high quality concentrated liquid product, freeze concentration technique is the most promising technique to be used.

2.3 FRUIT JUICE CONCENTRATING METHOD

2.3.1 Freeze Concentration Process

"Freeze concentration is based on the separation of soluble solids from a liquid phase by means of freezing the water content of the liquid" (Hernandez et al., 2009). This process requires a very low temperature to operate and can be considered as the best concentration technique to get the optimal product quality of heat-sensitive food such as fruit juice (Aider & de Halleux, 2009). The common temperature for this process to operate is below 0 °C as it is below the water freezing point. Thus, it will crystallize the water particle at this temperature and leaves behind the unfreezing solution. Fruit juices contain the volatile flavor compound that can be loss when heated. By using this process, no loss of volatile component happen as well as no thermal degradation occur (Sanchez et al., 2010).

Generally, there are two basic methods to concentrate solution by freezing. The first method is suspension freeze concentration while the second method is progressive freeze concentration (Sanchez et al., 2010). Suspension freeze concentration method is a method where many small ice crystals are formed and their size is being enlarged by the Oswald ripening mechanism. Then, the ice crystals are separated from the concentrated product in

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the washing tower under pressurized condition. Due to the complicated separation technique used, this method is reported to be the most expensive method compared to the other concentrating method (Aider & de Halleux, 2009). Thus, the food industry rarely used this method to concentrate the fruit juice. However, the second method (progressive freeze concentration), although it is still not widely used, offers the less expensive ways to concentrate the fruit juices. This method freeze the water content of a solution that is in direct contact with the cooling medium. The freezing water forms a large ice crystal that grown on the cooling surface, thus make it easier to separate the ice crystal with the concentrated solution (Sanchez et al., 2010 and Aider & de Halleux, 2009). Figure 2.2 shows the main distinguishing characteristics for these two methods.

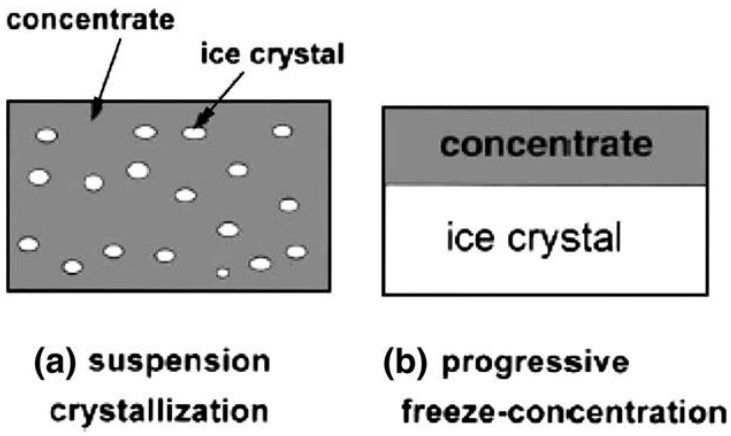

Figure 2.2: Two methods for concentration by freezing

Source: Hernandez et al. (2009)

2.3.2 Vacuum Evaporation Technique

Vacuum evaporation technique is a conventional technique used in the industry to concentrate fruit juice (Onsekizoglu et al., 2010). This technique operates at high temperature, in order to vaporize the water content of the fruit juice. Usually, the industrial concentration of fruit juices is performed by multistage vacuum evaporation processes in

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order to achieve the desired solid contents of 50-65 wt. % (Ribeiro et al., 2007). This technique is an improved technique of thermal evaporation technique, in which, vacuum is used to lower the operating temperature of the process by lowering the pressure inside the tank below the vapor pressure of the water. Thus, the water can be evaporated at lower temperature. However, the operating temperature is still high enough to destroy the volatile component of the fruit juice such as vitamin C (Chin et al., 2009). Furthermore, these techniques also degrade the colour of the original fruit juices as well as give a flat taste due to the thermal effect. Despite the major drawback, there is one advantage of this technique which is can yield a higher concentrated product (45–65 °Brix) compared to the other technique (Cassano et al., 2004).

2.3.3 Membrane Process

Membrane process is a technique that permits concentration and separation without the use of heat. Particles are separated on the basis of their molecular size and shape with the use of pressure and specially designed semi-permeable membranes. Membrane process has been widely known as one of the technique used to concentrate the fruit juice (Jiao et al., 2004; Vaillant et al., 2001 and Hongvaleerat et al., 2008).

The conventional membrane process is reverse osmosis concentration, where pressure in which higher than the osmotic pressure is applied to the compartment that contained the high concentration solution, hence, forces the water to pass through the membrane in the direction reverse to that of osmosis. In this manner, water is passed through the membrane to the other compartment leaving behind the dissolved solid in the other compartment (Jiao et al., 2004 and Kucera, 2010). The mechanism of this process is shown in the Figure 2.3.

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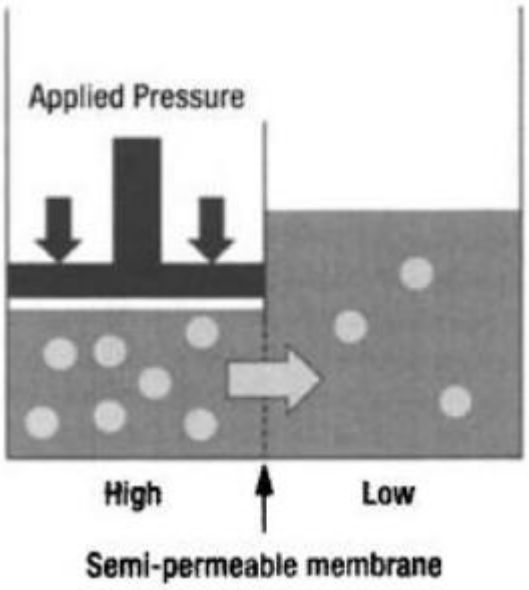

Figure 2.3: Reverse osmosis process

Source: Kucera (2010)

Despite the use, there are several disadvantages of the membrane process that limit its efficiency to concentrate the fruit juice. Single-stage reverse osmosis concentration cannot reach concentration larger than 25-30 °Brix (Jiao et al., 2004). Other than that is membrane fouling that decrease the efficiency of the membrane, thus requires frequent replacement of the membrane. "Membrane fouling refers to the attachment, accumulation, or adsorption of foulants onto membrane surfaces and/or within the membrane, deteriorating the performance of the membranes over time". In the reverse osmosis concentration, membrane fouling refers to the increase in the membrane resistance due to the formation of fouling layer on the membrane surfaces (Wang et al., 2011).

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2.4 FACTORS INFLUENCE FREEZE CONCENTRATION TECHNIQUE

2.4.1 Temperature

Temperature of the cooling medium is the main factor that influences the freeze concentration technique. This is because the coolant temperature controls the ice crystal front growth rate. The higher the rate of the crystal growth rate, the product will be more concentrated. According to the study done by Sanchez et al. (2010), the growth rate of the ice crystal is increased when the temperature is more lowered. Thus, it means that the lower the cooling medium temperature, the product will be more concentrated.

2.4.2 Stirring Rate

The stirring rate is another factor that influences the freeze concentration technique. "The time required for nucleation was reduced by wave stirring, but increased by rotary stirring" (Yaoi et al., 2004). In other words, the nucleation is stimulated by wave stirring. Thus, this indicates that the ice crystal growth is strongly dependant on the stirring method used and the solution flow. Therefore, it is very essential to do the process under optimized stirring conditions for producing high quality concentrated product.

2.4.2 The nature of the solute

A saturated solution can be defined as the maximum amount of dissolved solute in a solvent at given temperature and pressure. Thus, the solubility of a solution can be defined as the amount of solute that must be added to a given volume of solvent to form a saturated solution. When the solute concentration exceeds its solubility limit, it becomes supersaturated or in metastable state. (Tung et al. 2009). Supersaturation is the driving force for crystallization to occur. In other words, crystallization will not occur if the solution is not in the supersaturated state. Figure 2.4 provides further explanation on why

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crystallization occurs and what type of process that might the most suitable to be used for production of particular substance.

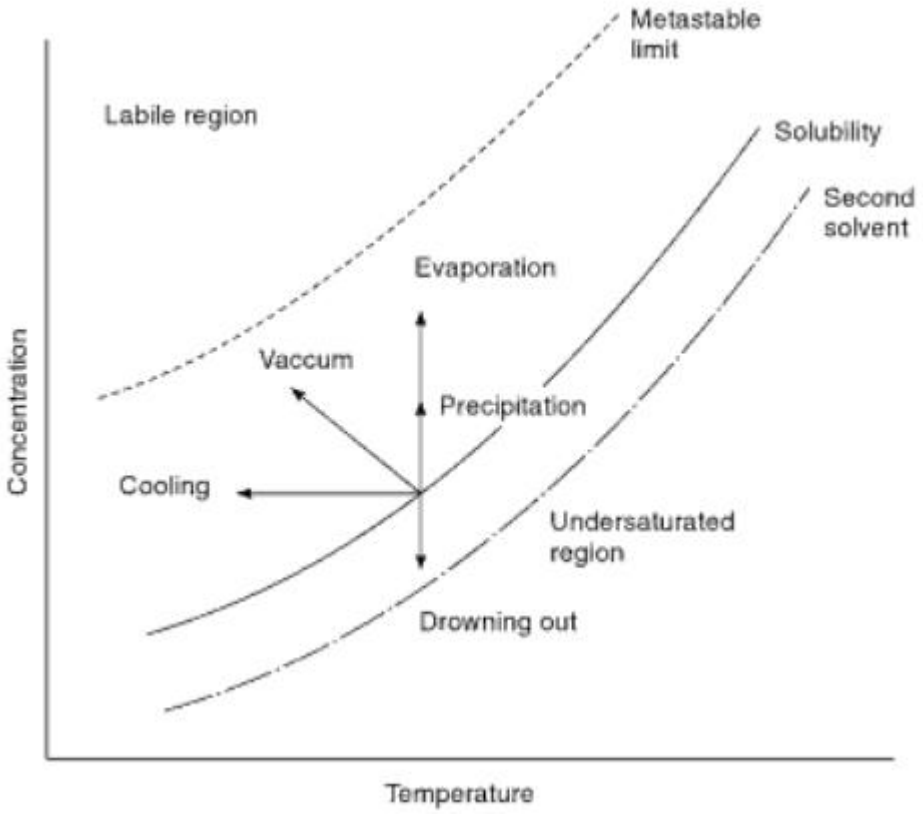

Figure 2.4: Solubility-supersolubility diagram

Source: Jones (2002)

According to the Figure 2.4, undersaturated region represent the state where all the crystal present in a solution will dissolve. Metastable region, or supersaturated region, represents the region in which crystal will grow. Lastly, labile region represent the region in which a solution will nucleate spontaneously.

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