

ABSTRACT

BI, XIANGYU. Design and Testing of an Induction-Heated, Vacuum-Assisted Evaporation System. (Under the direction of Dr. K.P. Sandeep and Dr. Josip Simunovic).

Vacuum evaporation is widely used to increase the solid content of foods and to change the color of liquid foods by partial removal of water by boiling. However, commercial vacuum evaporators take a long time to concentrate liquid foods and this may result in color change, flavor change, and degradation of bioactive and thermally sensitive ingredients. As a potential solution to this problem, an induction heated vacuum assisted evaporation system was designed and tested with sucrose solutions, bovine milk, infant formula (Similac), and aqueous extracts of purple sweet potato. The goals of this study were to 1) design and build an induction heated vacuum assisted evaporation system, 2) evaluate the capability and performance of the system, 3) evaporate different bioactive and thermally sensitive liquid products and assess retention rate of targeted compounds in the concentrated products.

An induction heated vacuum assisted evaporation system was designed and built using a BUCHI rotavapor system and a commercial induction cooker. Solutions containing 10%, 30%, and 50% sucrose were evaporated under 130 mbar, 160 mbar, and 190 mbar pressure for performance testing and then compared with a hot plate heating system in terms of energy efficiency. Bovine milk, reconstituted infant formula powder (Similac), and pre-mixed formula (Similac) were evaporated for 30% and 60% volume reduction under 130 mbar and then tested for fat and solid content. Aqueous extracts of purple sweet potato were evaporated for 2X, 3X, 4X, and 5X concentration under 150 mbar pressure and then tested for anthocyanin retention in the concentrated products.

The results showed that the induction heated vacuum assisted evaporation system could be operated under a minimum pressure of 130 mbar and that it had a significantly higher energy

efficiency compared to that of the hot plate vacuum evaporation system. The system was capable of concentrating bovine milk and infant formula under 130 mbar pressure. It was also able to concentrate aqueous extracts of purple sweet potato and significantly increase the anthocyanin content. However, bumping (product being drawn into the condenser) occurred during evaporation of bovine milk and aqueous extracts of purple sweet potato. Although installation of a bump trap could prevent product from being drawn into the condenser, bumping still affected the final volume of concentrate. Nevertheless, this system has the potential to serve as a cost effective and rapid system for evaporation and small scale concentration of thermally sensitive materials.

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Design and Testing of an Induction-Heated, Vacuum-Assisted Evaporation System

by
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DEDICATION

I dedicate this thesis to my parents and the rest of my family.

To everyone who supported me and brought love to my life.

BIOGRAPHY

Xiangyu Bi was born in Taiyuan, Shanxi, China. He decided to study abroad after a British orchestra played in his high school, which later inspired him to explore the world and learn different cultures.

In 2013, Xiangyu began his bachelor's degree in Food Science at NC State University. He joined the University Scholar Program in his Freshman year and then started working as an undergraduate tutor for two semesters. He was actively involved in several committees in the food science club and volunteered as an ambassador for incoming international students. Xiangyu also participated in an undergraduate research project with a goal of extracting phospholipids from cocoa butter to replace soy lecithin in dark chocolate, and later presented the findings of the study in the undergraduate research symposium. Upon graduation, he received three scholarships, completed one university honors program, and successfully graduated with the honor of Magna Cum Laude. While he received admission to the graduate school at NC State University, he began working as a lab technician under Dr. Van-den Truong and Ms. Rong Reynolds in the USDA-ARS unit in Schaub hall during the last summer of his undergraduate career. During this time, he was involved in extracting and analyzing the chemical constituents of purple sweet potatoes.

In 2017, Xiangyu began his Master of Science degree program in Food Science under the direction of Dr. Sandeep and Dr. Simunovic. He worked to design and test an induction heated vacuum assisted evaporation system. In summer 2018, Xiangyu interned at Takasago corp. in Rockleigh, New Jersey, where he spent two months analyzing flavor trends and worked with potential customers. He attended two annual IFT meetings and received two scholarships from

the food science club. Xiangyu plans to work for a year before making the decision of whether to pursue a PhD degree.

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CHAPTER 1. Introduction

Evaporation is an important industrial process to increase the solid content of foods and to change the color of liquid foods by partial removal of water by boiling. The benefits of evaporation include prolonging product shelf life, producing nutrient enriched concentrates, and reducing cost associated with packaging, transportation, and storage (Cassano, et al., 2003; Onsekizoglu, Bahceci, & Acar, 2010).

Evaporation is accomplished at atmospheric pressure or under vacuum. Evaporation at atmospheric pressure is simple but time consuming and not energy efficient. Furthermore, since most food products are thermally sensitive, prolonged exposure to high temperature could cause undesired color changes, nutrient losses, development of off flavors, and degradation of overall quality (Toledo, Singh, & Kong, 2018). Therefore, vacuum evaporation is used to minimize the above undesired changes. However, there are associated issues such as degradation of aroma compounds due to long process time and low energy efficiency of the process (Sabanci, Cevik, Cokgezme, Yildiz, & Icier, 2019). In recent years, researchers have focused on development of alternative concentration techniques such as freeze concentration, reverse osmosis, membrane concentration, as well as improving the efficiency of energy usage by implementing multiple effect vacuum evaporator systems and electro heating methods such as ohmic heating. However, several factors restrict the use of these techniques in the food industry. Some of the limitations include low final concentration value, long process time, and high installation and operating costs (Sabanci, Cevik, Cokgezme, Yildiz, & Icier, 2019; Barbe, Bartley, Jacobs, & Johnson, 1998).

Induction heating is an electromagnetic non-contact heating technology that has been used in metal processing, medical applications, and cooking (Geetha & Sivachidambaranathan,

2019). It has advantages such as temperature uniformity, safety, reliability, energy efficiency, flexibility, and compactness of system (El-Mashad & Pan, 2017). Although induction heating could be a desirable heating source for evaporation due to its advantages, the food industry is still in its early stages of using this technology. Induction heating could potentially result in energy savings of up to 50% for cooking flat products on a conveyor belt, and 20% energy savings in evaporation processes that use electrical furnaces (Levacher, Bethenod, & Hartmann, 2009). However, little information could be found on using induction heating for evaporation of food products.

The purpose of this research is to design and test an induction heated vacuum assisted evaporation system which is capable of concentrating bioactive materials such as anthocyanins extracted from purple sweet potato and blueberries, and concentrating thermally sensitive food ingredients such as milk. The first part of this work will involve the determination of the capability of the induction heated vacuum assisted evaporation system in concentrating sucrose solutions at different pressures. The next part will focus on comparing energy usage by evaporation using a hot plate and the induction heating system. The last two parts of this work will focus on analyzing the capability of the system to concentrate bioactive and thermally sensitive food products such as bovine milk, infant formula, and anthocyanin extracts from purple sweet potatoes.

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CHAPTER 2. Literature Review

2.1. Evaporation

2.1.1. Potential benefits of evaporation process

In the food industry, the process of evaporation is used primarily as a means of bulk and weight reduction for fluids since most liquid foods have a relatively low solids content. For instance, bovine milk contains 12.5% solids and fruit juice has 12% solids. This mass and volume reduction could bring several benefits to the food industry. Firstly, it helps in reducing cost of packaging, transportation, and storage. Secondly, it can boost nutrient content per unit serving. Thirdly, it can be used for preparation for subsequent treatment such as crystallization of sugar, precipitation of pectin, coagulation of cheese, and dehydration of milk and whey. Lastly, it can be used as a preservation method by reducing water activity as well as building a desired consistency for jellies and jams (Berk, 2009).

2.1.2. Vacuum evaporation

Long time exposure of liquid foods to high temperatures is likely to cause changes in the color and flavor of the liquid. In some cases, these changes may be acceptable, but in the food industry, most of these changes are undesirable. Processing of thermally sensitive products such as milk and fruit juices and bio-active compounds such as antioxidants may undergo severe heat degradation and hence produce undesirable attributes. To minimize such heat damage, vacuum pumps, steam ejectors, and different kinds of condensers (direct and indirect) are used to reduce the pressure of the liquid in the evaporator (Brennan, 2006). Reducing pressure in the evaporator leads to a lower boiling point of the liquid because a liquid boils when the vapor pressure it

exerts equals the external pressure above it. Typically, the evaporation temperatures are in the range of 40 – 95 °C, with corresponding pressures of 7.5 – 85 kPa (Brennan, 2006).

2.1.3. Factors affecting boiling point

Since vacuum evaporation could reduce the off-flavors produced in an evaporation process, determining the liquid boiling temperature becomes necessary. There are several factors affecting the liquid boiling temperature in an evaporation process. Pressure is a primary factor that affects boiling temperature. For instance, water boils at 100 °C at atmospheric pressure of 101.35 kPa, but it boils at 50 °C at 12.349 kPa. The second factor that affects boiling temperature is the total soluble solids content in a liquid. Higher the soluble solids content in a liquid, higher its boiling point. The mathematical relation between boiling temperature and vapor pressure is given by the Clausius-Clapeyron equation:

$$\frac{dp}{dT} = \frac{L}{T\Delta v}$$

Where $\frac{dp}{dT}$ is the slope of the tangent to the coexistence curve at any point, L is the specific latent heat of evaporation, T is the temperature, and Δv is the specific volume change of the phase transition.

In addition to pressure change and presence of soluble solids, the depth of the liquid could also affect the boiling point. Especially in some long tube evaporators, the rise of boiling point can lead to overheating of the liquid, hence causing heat damage (Brennan, 2006). The pressure exerted by the depth of the liquid can be described as:

$$P = \rho gh$$

where ρ is density of the liquid, h is depth of the liquid, and g is acceleration due to gravitation.

2.1.4. Potential problems during vacuum evaporation

Bumping and foaming are two major problems during vacuum evaporation that lead to product loss through the vacuum vent, contamination of condensate, as well as decreasing of evaporation efficiency. Bumping occurs when a product boils rapidly, forming bubbles that causes the sample being concentrated to flash out through the vacuum vent. This is generally caused by too much heating and a high degree of vacuum (Tennenhouse, 2017). Foaming, on the other hand, caused by agitation and aeration, could also lead to decreased efficiency of evaporation as well as loss of product and product quality (Girton, Macneil, & Anantheswaran, 1999)

Tennenhouse (2017) suggested the use of an anti-foam agent, a large vessel, and a glass foam brake to prevent foam entering into the receiving container and thereby contaminating the system. Bumping is not easy to prevent, although slowly increasing heat and decreasing pressure could help ease the situation, especially when a specific pressure and temperature combination is required for processing. To solve bumping, bump trap is often used in a laboratory scale vacuum evaporation system. It is placed between the vapor tube and evaporation flask to prevent contents of the flask from being drawn into the condenser. The inner vapor tube is sealed from the top to prevent product entry into the condenser, whereas holes at the bottom of the inner vapor tube help the product drain back into the evaporation flask.

2.2. Evaporators used in the food industry

Evaporators usually consist of a heat exchanger, a separator, and a condenser that are all built of stainless steel. Evaporators can be categorized into two groups, single-effect evaporators and multiple-effect evaporators.

2.2.1. Single-effect evaporator

There are several single-effect evaporators that are commonly used in the food industry. They include vacuum pan evaporator, long tube evaporator, plate evaporator, and agitated thin film evaporator.

2.2.1.1. Vacuum pan evaporator

Vacuum pan evaporator is the simplest type of batch vacuum evaporator used in the food industry. It consists of a hemispherical pan with a steam jacket and sealed lid that is connected to a vacuum pump (Figure 2.1). It could take hours for a product to reach a certain solids content because larger the pan, lower the heat transfer area to volume ratio. Although smaller pan could have a higher heat transfer area to volume ratio, it limits the throughputs per unit time. Vacuum pan evaporator is usually used for products that need to be frequently changed such as jams, sauces, soups and gravies (Brennan, 2006).

2.2.1.2. Long tube evaporator

Long tube evaporator often consists of tubes that are 3-15 m long and 25-50 mm in diameter. Tubes are contained in a vertical shell where steam is introduced and provides the heat of evaporation (Brennan, 2006). There are two models of such evaporators, namely the rising film evaporator and falling film evaporator.

In the rising film evaporator, the preheated feed is pumped into the bottom of the tube bundle (Figure 2.2). Evaporation starts as the feed rises along the tube. As vapor generates and expands, ideally, a thin film of liquid will be pushed up and rise with the vapor. The liquid becomes more concentrated and it was collected at the top cyclone together with the vapor. The

vapor is either condensed, or used as heating source for the next stage of evaporation, in a multiple-effect system. The rising film evaporator is suitable for low viscosity and heat sensitive materials such as milk and fruit juices and its advantages include high rates of heat transfer and short residence time (Brennan, 2006).

In the falling film evaporator, the preheated feed is evenly distributed at the top of the tube bundle. As the feed descends (Figure 2.3), a thin film of the liquid forms and flows down the inner surface of each tube. Steam heats from the outside of the tubes and vapor is generated as the feed concentrates downwards in the tubes. The liquid-vapor mixture is collected at the bottom and then separates into two streams where vapor is condensed or pumped into another heat jacket, in a multi-effect evaporator, and concentrate is collected as product or recycled to another effect for further concentration. Falling film evaporator is suitable for concentration of high viscosity and heat-sensitive materials. The advantages include short contact time between the feed and heating surface, high heat transfer coefficient, minimal pressure drop, minimal pressure head, and small process fluid holdup (Alhousseini, Tuzla, & Chen, 1998).

2.2.1.3. Plate evaporator

Similar to a plate heat exchanger, which is commonly used in milk pasteurization, a plate evaporator consists of assembled plates where steam and liquid flow in alternate spaces between plates (Figure 2.4). Spacing is larger than that in the pasteurization process to accommodate both liquid and vapor. Like in other evaporators, the liquid-vapor mixture leaving the plates enters a cyclone separator. The vapor is then condensed or used as heating source in another effect. The concentrate is collected as product or is further concentrated. This type of evaporator cannot handle high viscosity materials and has low throughput but has a high rate of heat transfer, with

short residence time, and low possibility of fouling for the product. Its compact design and ease of dismantling provide unique advantages to the food industry in comparison to those affected by tube evaporators (Brennan, 2006).

2.2.1.4. Agitated thin film evaporator

The component that distinguishes an agitated thin film evaporator from other types of evaporators is a rotating shaft (Figure 2.5). The shaft has blades on its side which can wipe away the fouling material on the inner surface of the shell so that a high rate of heat transfer can be maintained throughout the process. Since the agitated thin film evaporator has a high initial cost and low throughput, it is not commonly used in initial bulk volume reduction but often used as the last evaporator when high solid content is required. Tomato paste, milk products, sugar products, gelatin solution, and coffee extracts are the commonly processed in this type of evaporator (Brennan, 2006).

2.2.2. Multiple-effect evaporator

Evaporators can be operated in series to create a multiple-effect evaporator. Each evaporator is referred to as an effect. The product coming out from one evaporator becomes the feed for the next effect, and the high temperature vapor produced in one effect is used to heat the lower temperature product in the next effect (Best available techniques in the food, drink and milk industries, 2006). In order for this arrangement to work, the evaporation temperature of one effect has to be lower than that in the preceding effect. This can be achieved by gradually lowering the pressure in each effect.

In the food industry, the purpose of using a multiple-effect evaporator is to reduce the steam consumption for large water removal because steam can be costly. In a single effect

evaporator, it takes 1.1-1.3 kg of steam to evaporate 1 kg of water. In a double effect evaporator, the steam consumption decreases to 0.55-0.70 kg of steam to evaporate 1.0 kg of water. In a triple effect evaporator, the steam requirement further reduces to 0.37-0.45 kg per 1.0 kg of water (Brennan, 2006). The steam consumption can be further decreased by applying mechanical or thermal vapor recompression (Best available techniques in the food, drink and milk industries, 2006). Although steam consumption can be largely reduced with the increasing number of effects, the capital cost of a multiple-effect evaporator increases with the number of effects. For example, a triple effect evaporator costs approximately three times that of a single effect evaporator. It is also important to note that a multiple-effect evaporator does not increase the product throughput in comparison to that in a single effect evaporator. Therefore, alternative heating methods such as microwave heating, ohmic heating, and induction heating are getting more and more attentions.

2.3. Induction heating

2.3.1. Principle of induction heating

In food processing and cooking, induction heating heats food indirectly by conduction through a ferrous material. Ferrous materials that mainly contain iron include steel, stainless-steel, pig iron, and iron alloys. When ferrous materials are placed in an alternating magnetic field produced by an alternating current in an induction coil, it generates eddy currents that have a similar frequency as the produced magnetic field. As eddy currents flow through the ferrous material, heat is generated by the resistance of the material and it then conducts heat to the contacting food material. Aside from heat generated by eddy current, heat is also generated by magnetic hysteresis loss, which is an internal friction created when magnetic parts pass through the inductor (El-Mashad & Pan, 2017).

Induction heating is a non-contacting heating method that combines several complex interactions including electromagnetics, heat transfer, and metallurgical phenomena (El-Mashad & Pan, 2017). Applying alternating magnetic field or voltages to an electrically conducting object will induce eddy current flow in that object. Eddy current flows through the resistance of the material and produces heat by Joule effect (I^2R) (Rudnev, Loveless, & Cook, 2017).

2.3.2. Benefits of induction heating

There are three areas in which induction heating is commonly applied: industry, domestic, and medical (Geetha & Sivachidambaranathan, 2019). In industry, induction heating is mainly used in the metals industry and also in several other industries such as glass and quartz processing, semi-conductor fabrication, and chemical synthesis of liquid and gases. The major advantages of utilizing induction heating in these industries include ability of in-depth heat generation and highly controllable heat intensities on the workpiece, which lead to high productivity and repeatable quality (Rudnev, Loveless, & Cook, 2017).

Compared to most other heat sources including gas-fired furnaces and electric stoves, induction heating can reduce heat loss to the outside environment which makes it more environmentally friendly and more energy efficient. Induction heating system is also easy to clean since any fumes and surface contaminants produced during the induction heating process can be easily removed from the surface. Safety and low operational cost also make induction heating competitive and attractive to other heat sources since no combustion is involved, no environmental contaminants are produced, and less labor cost needed (Rudnev, Loveless, & Cook, 2017).

In the domestic area, the conventional way of heating food using gas stoves has drawn concerns over the past few decades. The increasing awareness of environmental deterioration and global warming effects is convincing people to switch from gas stoves to electric coils and induction cookers which have both been proven to have a significantly higher heating efficiency without consuming non-renewable energy sources and producing CO₂ and other chemical byproducts that are unfriendly to the environment (Meng, Cheng, & Chan, 2011).

In domestic scale testing, although several research (El-Mashad & Pan, 2017; CenturyLife.org, n.d.) have shown that induction heating does not necessarily lead to higher energy efficiency when compared to conventional electric coils in the long run, others conclude that induction heating has an energy efficiency around 80-90 %, the corresponding values for electrical coil heating and gas heating are 74-77 % and 40%-50% respectively (Meng, Cheng, & Chan, 2011). The efficiency of the induction system depends on parameters such as size of the pan, area of heating surface, thickness of the induction coil, material of the coil, design of the power circuit topology (Geetha & Sivachidambaranathan, 2019). With the current design of induction cooker, induction heating can achieve an efficiency of 90% (Lucía, Maussion, Dede, & Burdío, 2014). Beyond efficiency testing, induction heating system also requires far less start-up and shutdown time (Rudnev, Loveless, & Cook, 2017).

Beyond heating conductive materials, induction heating is also capable of heating non-conductive materials by using of a susceptor, such as a graphite crucible. Applications includes melting refractory materials, glasses, quartz, sintering carbides, semi-conductor fabrication, heating liquids, and heating gases during chemical synthesis (Vairamohan, Bran, & Bellefon, 2011).

2.3.3. Commercial induction cooker

Application of induction heating in domestic and commercial cooking has gained increasing attention in the past 20 years. Nowadays, restaurants are considering switching from the conventional stove to induction cooker due to numerous benefits including high heat efficiency, cleanliness, and controllability (Meng, Cheng, & Chan, 2011).

A typical induction cooker system consists of power circuit, control, and load. The power circuit is often referred to as the rectifier and the inverter. The rectifier converts alternating current to direct current by only allowing it to flow in one direction and then the inverter increases the main frequency to anywhere between 20-100 kHz (Meng, Cheng, & Chan, 2011). The load often refers to the induction coil and the pan. When high frequency current flows through the coil, it produces an alternating magnetic field, which causes eddy current and hysteresis to heat up the pan. The power switch and power level system are performed by the control part (Figure 2.7).

Induction heating has several primary components such as power electronic circuit, magnetic components, and modulation and control strategies (Geetha & Sivachidambaranathan, 2019). Research on the power electronic circuit has shown improved performance (Lucia, Carretero, Burdio, Acero, & Almazan, 2012). Specifications of magnetic components such as the thickness of the copper coil, type of the coil, and shape of the coil contribute to the amount of energy produced in the heating system (Sivachidambaranathan, 2015). The modulation and control system is crucial to the performance of the commercial induction cooker since different power circuit topologies and power regulation schemes can have both good and bad impacts on the system performance (Selvamuthukumar, Satheeswaran, & Babu, 2015; Meng, Cheng, & Chan, 2011).

2.3.4. Application of induction heating in food processing

The food industry uses a lot of energy in the form of steam and hot water. However, energy wasted during heating the steam and water and then transferring heat to the food products results in a low energy efficiency in processing. Therefore, many alternative heating technologies such as infrared, ohmic, and microwave heating have gained popularity in recent years (Pan & Atungulu, 2010). Induction heating could be another alternative technology due to its advantages such as high energy efficiency, high safety, controllability, and compactness of the heating system.

Although there have not been many applications of induction heating in the food industry, research and patents show some potential benefits of applying such technology. A study showed that using induction heating could save 50% of the energy demand for cooking flat products on a conveyor belt (Levacher, Bethenod, & Hartmann, 2009). Another study showed that induction heating could save 20% energy when compared to that using an electrical furnace for evaporation of organic and inorganic liquids (Kuzmichev & Tsybulsky, 2011). However, few studies could be found in evaporation of food products using induction heating.

There are several patents describing induction heating systems used for heating of food materials. One is an inclined cylindrical metallic barrel design where food enters and is heated and rotated along the longitudinal axis. Heat is induced throughout the wall of the barrel and is then transmitted into the food material as the food material passes through (United States of America Patent No. US4265922A, 1979). Another patent utilizes induction heating in a device made of meal trays one above each other. The trays have ferric metal on the bottom so that food placed on top of the tray can be heated by magnetic induction. The food to be heated will be covered by an insulating dish cover and the device will switch on/off based on the presence of

the dish covers to optimize the energy efficiency of the entire system (United States of America Patent No. 5628241, 1997).

Another patent shows the ability of induction heating in extending shelf life of frying oil during frying (United States of America Patent No. US8076620B2, 2008). It was suggested that induction heating could supply electrons during frying and resulted in reducing oxidation of oil. Later research compared this patent and conventional frying of French fries and showed that the reduction of the formation rate of free fatty acid and aldehyde by utilizing induction heating could increase the shelf life of frying oil (Wenstrup, Plans, & Rodriguez-Saona, 2014). Free fatty acid is an indicator for frying oil replacement and aldehyde is response for the off-flavor in fried products (Pike & O'Keefe, 2017).

Research has also shown the possibility of applying induction heating for extraction of pectin from citrange albedos to yield higher volume of pectin when compared to that by conventional heating (Zouambia, Ettoumi, Krea, & Moulai-Mostefa, 2014). Atyhanov and Sagyndikova developed a grain dryer using induction heating (2013) and Tomita et al. developed a superheated steam generator using induction heating (2009). Although there are many patents and studies on the application of induction heating in food processing, little information is found on the design, operation, and energy efficiency of induction heating systems for different food processes. There is also little information available on the effect of induction heating on functional components in various food ingredients.

2.4. Food materials containing thermally sensitive bioactive ingredients

A common issue associated with evaporation is product quality loss due to high temperature and longtime processing, especially for food materials that containing thermally sensitive and bioactive ingredients such as anthocyanins, proteins, lactose, and lipids.

2.4.1. Anthocyanins

Anthocyanins are water soluble bioactive compounds that belong to a flavonoid group termed polyphenolic pigments and they are responsible for many of the red, orange, blue, and violet colors present in plant parts such as fruits, flowers, and leaves (Wallace & Giusti, 2015).

Anthocyanins are known as natural antioxidants due to their ability to donate protons to quench highly reactive free radicals at the site of oxidative events (Sadilova, Carle, & Stintzing, 2007). Anthocyanins are also found to be effective in preventing further generation of free radicals and protecting cells from oxidative damage which has often been associated with aging and various diseases caused by oxidative stress (Boldt, Meyer, & Erwin, 2014). Recent studies have also shown support for consuming anthocyanin-rich fruits such as blueberry, cranberry, and bilberry in promoting health, but their low bioavailability (<1%) makes them highly unlikely to extend life span and prevent chronic diseases (Glover & Martin, 2012). Although anthocyanins are not one of the essential nutrients and there have not been any deficiency disorders associated with the lack of anthocyanin consumption, effectively and efficiently extracting and concentrating a large amount of anthocyanins as natural oxidants can have potential benefits (Wallace & Giusti, 2015).

Anthocyanins can be extracted through numerous methods. The classic approach is to extract anthocyanins similar to how phenolic compounds are extracted, by subsequent solvent

extraction with polar solvents such as methanol, ethanol, acetone, and even water with acidified organic or inorganic acids (Silva, Costa, Calhau, Morais, & Pintado, 2017; Barnes, Nguyen, Shen, & Schug, 2009; Metivier, Francis, & Clydesdale, 1980; Vatai, Škerget, & Knez, 2009). New methods such as supercritical fluid extraction, ultrasound-assisted extraction, pressurized liquid extraction, microwave-assisted extraction, and ohmic heating-assisted extraction are reported to be effective and efficient in anthocyanin extraction (Silva, Costa, Calhau, Morais, & Pintado, 2017).

Given that anthocyanins can provide potential benefits to human health by serving as natural antioxidant and nutritive food additives, ensuring their stability during storage and processing becomes a particular of interest (Sui, 2017). Several factors affect the stability of anthocyanins. These include temperature, pH, light intensity, oxygen availability, presence of enzymes, ascorbic acid, sugars, metal ions, and also structure of the anthocyanins (Roobha, Saravanakumar, Aravindhan, & Devi, 2011). Temperature is one of the major factors affecting anthocyanin degradation during food processing (Sui, 2017).

Extracted anthocyanins can be added into food products and dietary supplements to not only serve as natural antioxidants, but also promote health and prevent aging.

2.4.2. Proteins

Protein is one of the most important components in foods and it is crucial to human body development for structure and function. Most proteins consist of 20 kinds of amino acids that are linked through peptide bonds. Although the human body is capable of producing certain amino acids, there are 9 amino acids termed essential amino acids that cannot be made by human body and have to be ingested from diet.

Aside from being an important nutrient source, protein is also widely utilized in various food applications due to its functional properties. Common functionality includes solubility, foamability, viscosity, emulsification, elasticity, water holding, gelation, and fat and flavor bonding (Lin, Wu, & Wang, 2012). Products including sausage, cake, salad dressing, noodles, and ice cream require the involvement of the above functionalities to achieve the desired characteristics. Therefore, with the increasing demand of proteins in human diet, discovering new sources of protein, and also extracting and isolating proteins can be beneficial to the food industry.

Muscle protein, casein, whey protein, wheat protein, and soybean protein are the most common protein sources for humans. However, due to the increasing population and rising demands of proteins, researchers are switching to exploiting new protein resources such as insect proteins, leaf proteins, single-cell protein, and oil-crop proteins (Lin, Wu, & Wang, 2012). For example, the protein content of many insects exceeds 50% on a dry basis. Crickets and ants have protein content close to 70%.

Proteins can be extracted by numerous methods including alkaline extraction, acid extraction, salt extraction, reverse micellization, and aqueous two-phase extraction (Cunha & Aires-Barros, 2002; Boye & Barbana, 2012). The extracted proteins need to be recovered through various methods for purification and precipitation. Since most proteins precipitate at pH values close to their isoelectric point, isoelectric precipitation becomes a frequently used method in the food industry. Microfiltration and ultrafiltration are pressure-driven filtration methods that are frequently used as alternative methods to isoelectric precipitation and are extensively used to separate protein from milk, soybeans, peas, and chickpeas (Boye & Barbana, 2012). Extracted and isolated proteins are extensively used in protein supplements for improving muscle growth.

Besides extracting proteins from food materials, concentration of protein-containing material provides another way of acquiring the high nutrient value of protein. Evaporated milk and condensed milk are two applications of concentration of proteins in milk. The resulted products not only have longer shelf life, but are also high in nutrient content.

2.4.3. Lactose

Lactose is a disaccharide which is widely found in milk and non-fermented dairy products. Among mammals, human milk has the highest lactose concentration with lactose counting for 60% of the total osmolarity (Slupsky, et al., 2017). Human milk contains twice as much lactose in comparison on to that in cow milk, lactose making up around 2-8% by weight. However, people who lack lactase cannot digest or metabolize lactose. Once their intake of lactose exceeds 20-50 g on an empty stomach, they may suffer from abdominal pain, bloating, diarrhea, nausea, acid reflux, and flatulence (Wang, sun, Huang, Zhou, & Sun, 2012; Balieiro, et al., 2016). Lactose intolerance is estimated to affect 65% or more of the total human population with an increase in intolerance with age and variation by ethnic groups (Law, Conklin, & Pimentel, 2010).

Technologies that are able to reduce lactose content or completely remove lactose from milk products are developed to satisfy people who suffer from lactose intolerance. Lactose hydrolysis, capillary electrophoresis, ultrafiltration, ion-exchange chromatography, and molecularly imprinted polymers are used to remove lactose (Bargeman, 2003; Marín-Navarro, Talens-Perales, Oude-Vrielink, Cañada, & Polaina, 2014; Mohammad, Ng, Lim, & Ng, 2012; Balieiro, et al., 2016).

By removing lactose from human milk, preterm infants and babies will be able to avoid lactose intolerance symptoms. By decreasing lactose content of cow milk, lactase deficiency consumers can start drinking milk again, which ultimately will enhance milk production and consumption in the long run. Lactose free milk can also be used in other food and dairy applications without causing further concerns.

2.4.4. Lipids

Lipids are naturally-occurring molecules that exist in all animals and plants consumed by humans. Lipids are not only the major energy source in human body, but also protect human body from mechanical injury and heat loss. Lipids are also carriers of various fat-soluble vitamins such as vitamin A, D, E, and K, and the precursors of many bioactive molecules such as sex hormone and epinephrine (Shen, Wang, & Das, 2012). Although humans are capable of synthesizing and metabolizing lipids through various pathways, there are certain lipids termed essential fatty acids that have to be ingested from the diet.

Most vegetables and fruits contain very little lipids, about 0.3%, except avocados which contain 20% edible lipids. Lean beef, fish, white poultry, and shellfish contain about 2% lipid content in the muscle tissue, whereas in fatty pork and fish, the lipid content can exceed 30%. Oil-bearing nuts and seeds are also a good source of lipids within soybeans containing 20% lipids and in walnuts containing 65% lipids.

In the food industry, lipids can be extracted through mechanical and chemical methods. Traditionally, vegetable oil is extracted from seeds using an oil mill. Nowadays, the food industry uses solvent extraction (mainly by n-hexane) to achieve a higher yield (Phan, Brown, White, Hodgson, & Jessop, 2009). Microwave-integrated Soxhlet extraction is developed and

successfully shortens the process of extraction from 8 hours to 32 min in comparison to the conventional Soxhlet method (Virot, Tomao, Colnagui, Visinoni, & Chemat, 2007). However, the high cost of organic solvents, increasingly stringent regulations, and rising awareness of environmental concerns necessitate the food industry to adopt clean technologies for such processing (Mohamed & Mansoori, 2002). Supercritical carbon dioxide extraction has advantages including being non-toxic, non-flammable, non-polluting, recoverable, and its ability to solubilize lipophilic substances. Researchers have reported that supercritical carbon dioxide extraction of fatty acids is able to produce better retention of aromatic compounds without producing solvent residue, as well as saving time and money, compared to the conventional solvent extraction (Sahena, et al., 2009; Norulaini, et al., 2009; Herrero, Cifuentes, & Ibanez, 2006; Zaidul, Norulaini, Omar, & Smith, 2006).

Aside from being a nutritional ingredient and used in cooking oil applications, lipids are also effective in reducing risk of chronic disease by serving as functional ingredients (Moreau, 2001). Functional lipids including omega-3 and omega-6 fatty acids, conjugated linoleic acids, medium chain triglycerides, and phytosterols are also found to have positive effects on obesity, bone health, and in treating and managing blood pressure, depression, and cardiovascular health (Alabdulkarim, Bakeet, & Arzoo, 2012). New methods of extraction and concentration of lipids, especially the functional lipids, can bring potential benefits to the general population in the near future.

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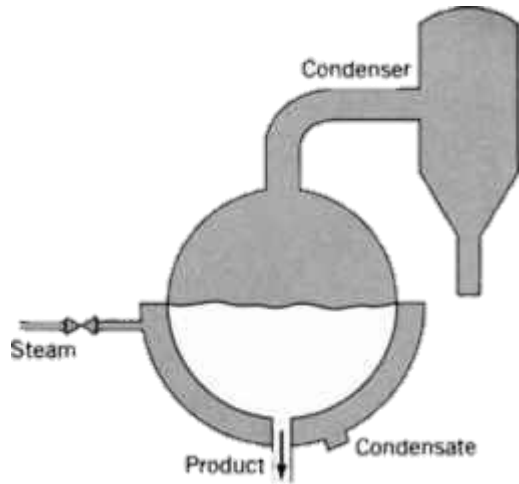


Figure 2.1. Vacuum pan evaporator (Barnard Health Care, 2019)

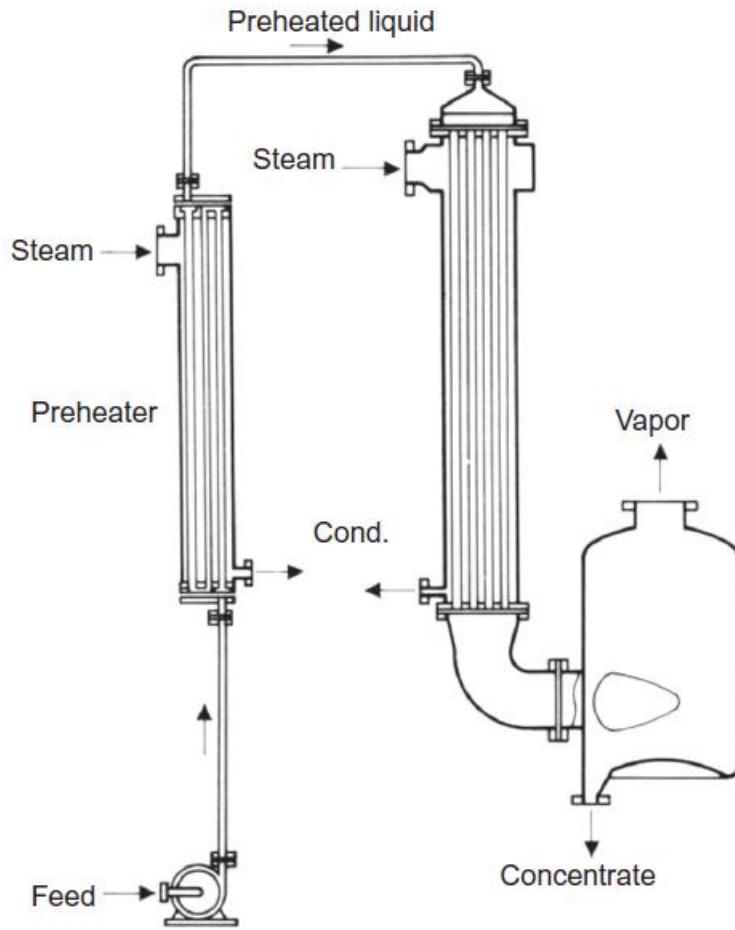


Figure 2.2. Rising film evaporator (Berk, 2009)

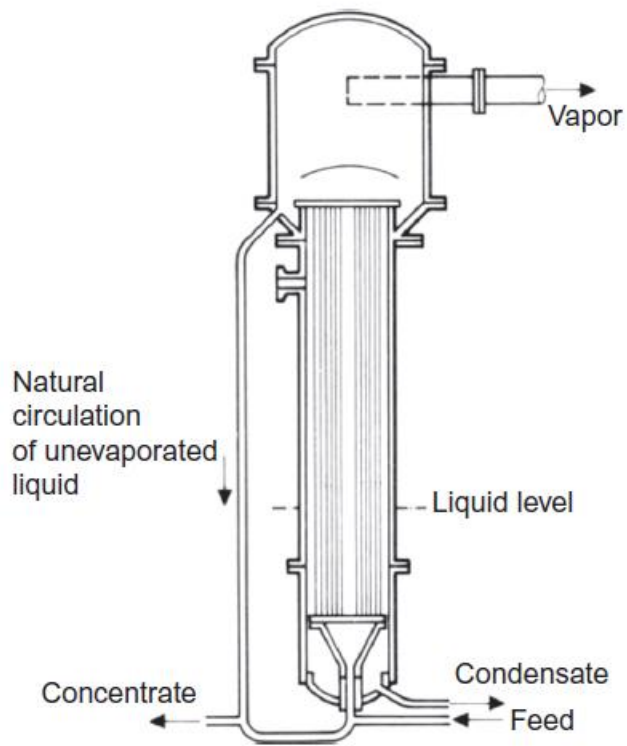


Figure 2.3. Falling film evaporator (Berk, 2009)

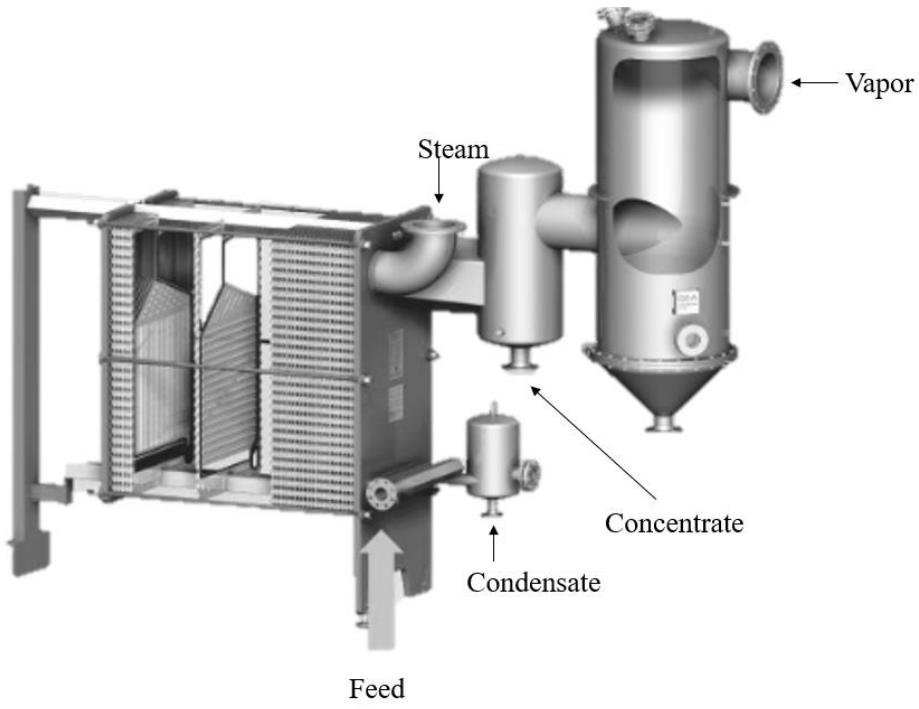


Figure 2.4. Plate evaporator (GEA, n.d.)

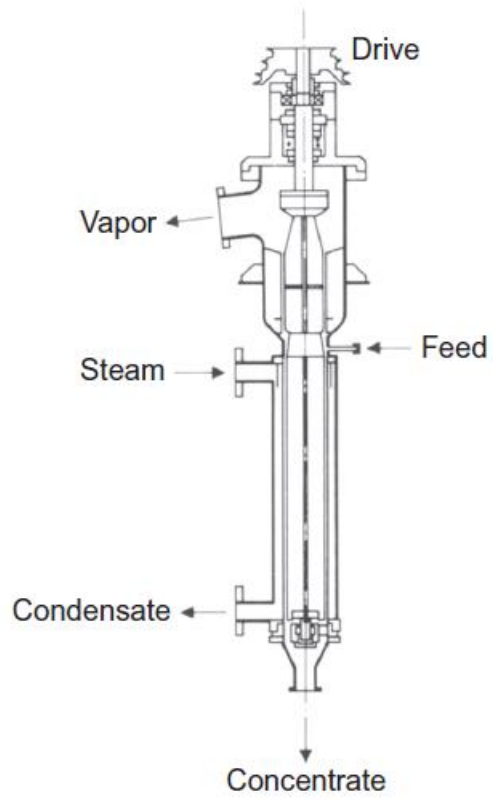


Figure 2.5. Agitated thin film evaporator (Berk, 2009)

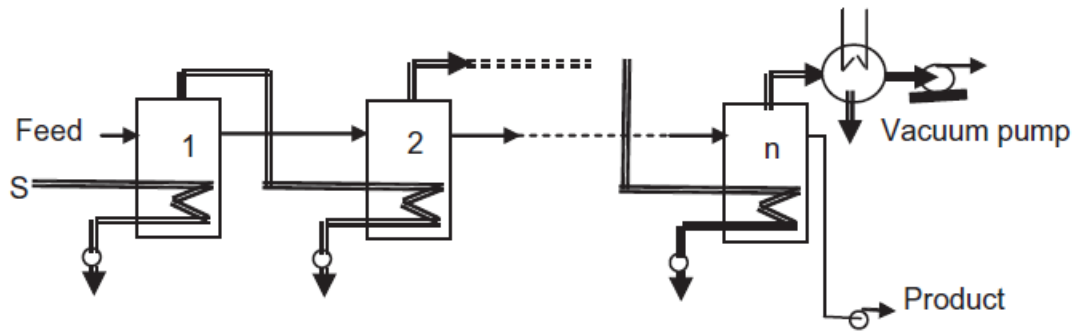


Figure 2.6. Forward feed multiple effect evaporator (Berk, 2009)



Figure 2.7. Exploded view of induction heating appliances (Lucia, O., Acero, J., Carretero, C., & Burdio, J. M., 2013)

Chapter 3. Design and Testing of An Induction Heated Vacuum Assisted Evaporation System and Testing with Sucrose Solutions

3.1. Abstract

Vacuum evaporation is widely used to increase the solid content of foods and to change the color of foods through partial removal of water from liquid foods by boiling. However, it has some disadvantages such as high cost of steam, degradation of aroma compounds, and low energy efficiency. Induction heating has advantages such as high energy efficiency, scalability, reliability, and precise temperature control which makes it a potential heating method for evaporation. Induction heated vacuum assisted evaporation system (IHVAES) was designed and tested with sucrose solutions and protein polyphenol complex. 10% w/v, 30% w/v, and 50% w/v sucrose solutions were evaporated under 130 mbar, 160 mbar, and 190 mbar pressure. Boiling temperatures, rates of evaporations, and energy efficiency were determined under each concentration-pressure combination. Hot plate was used as an alternative heat source for comparison of energy efficiency. The results showed that the lowest boiling temperature was 54.6 ± 0.2 °C at 130 mbar pressure and the highest rate of evaporation was 11.4 ± 0.4 ml/min at 130 mbar pressure. IHVAES had energy efficiency of 1012.8 ± 25.2 ml/kWh, whereas the hot plate had an efficiency of 923.3 ± 3.2 ml/kWh. Evaporation of protein polyphenol materials was not successful because product fouling occurred at the bottom of the pot and the texture also changed. Since IHVAES was capable of heating and evaporating sucrose solutions at reduced temperature under vacuum at a higher energy efficiency compared to that by a hot plate, it was concluded that this system might have good potential for serving as a cost effective, small scale evaporation system for thermally sensitive materials.

3.2. Introduction

In the food industry, the process of evaporation is mainly used for volume and weight reduction for fluids since most fluid foods have a relatively low solids content. This weight and volume reduction can not only reduce costs associated with transportation, storage, and packaging, but also enable the concentrated food products to have higher resistance to microbial and chemical deterioration as a result of water activity reduction (Onsekizoglu, Bahceci, & Acar, 2010).

Evaporation is accomplished under atmospheric pressure or under vacuum. Atmospheric evaporation is simple but time consuming and not energy efficient (Toledo, Singh, & Kong, 2018). In addition, most food products are thermally sensitive, and hence prolonged exposure to high temperature could cause undesired color changes, nutrient losses, off flavors, and degradation of overall quality (Toledo, Singh, & Kong, 2018). Therefore, vacuum evaporation is commonly utilized in the food industry. Vacuum evaporation can be achieved by various evaporators such as vacuum pan evaporator, climbing film evaporator, falling film evaporator, plate evaporator, and agitated thin film evaporator. Although each evaporator has its own advantages regarding different processing applications, they all face a common problem, which is the high volume of steam consumption. Steam can be costly, especially in the evaporation process for large amount of water removal. To overcome this problem, the multiple-effect evaporator was invented to reduce steam consumption. In a single effect evaporator, it takes 1.1-1.3 kg of steam to evaporate 1 kg of water. In a double effect evaporator, the steam consumption decreases to 0.55-0.70 kg of steam to evaporate 1.0 kg of water. In a triple effect evaporator, the steam requirement further reduces to 0.37-0.45 kg per 1.0 kg of water evaporated (Brennan, 2006). Although steam consumption can be largely reduced with increasing the number of

effects, the capital cost of a multiple-effect evaporator increases with the number of effects. For example, a triple effect evaporator costs approximately three times that of a single effect evaporator. In recent years, researchers have focused on development of alternative concentration techniques such as freeze concentration, reverse osmosis, membrane concentration, as well as improving the efficiency of energy usage by implementing multiple effect vacuum evaporator systems and electro heating methods such as ohmic heating and microwave heating (Sabanci, Cevik, Cokgezme, Yildiz, & Icier, 2019; Sabanci & Icier, 2017; Bozkir & Baysal, 2017). However, limitations such as high installation and operation costs, final concentration value, and long process time restrict their usage in the food industry (Sabanci, Cevik, Cokgezme, Yildiz, & Icier, 2019).

Induction heating is an electromagnetic (non-contact) heating technology and it has been used in domestic applications for more than 50 years. It has advantages such as precise temperature control, temperature uniformity, reliability, energy efficiency, flexibility, and compactness of the heating system (El-Mashad & Pan, 2017). Although induction heating could be a desirable heating source for evaporation due to its advantages, the food industry is still in its early stages of applying this technology. According to literature, using induction heating could save up to 50% energy in cooking flat products on a conveyor belt, and save up to 20% energy in evaporation processes that use electrical furnaces (Levacher, Bethenod, & Hartmann, 2009). However, little information could be found on the use of induction heating for evaporation of food products. Thus, the purpose of this research was to (1) design an induction heated vacuum assisted evaporation system, (2) test its capability in evaporating water from sucrose solutions, and (3) compare the energy efficiency of the induction heating system to that of a hot plate heating system.

3.3. Materials and methods

3.3.1. Induction heated vacuum assisted evaporation system (IHVAES)

The IHVAES was built using a BUCHI Rotavapor R-215, BUCHI vacuum controller V-855, and BUCHI vacuum pump V-700 as the starting point. Heating bath and evaporation flask were removed and replaced with a high power commercial induction cooker (Puda Electric, model HZD 5KW-PX, Foshan, China) and a modified pressure cooker (Cook's Essentials, item # 918060796, West Chester, PA). Tap water was used as the cooling agent in the condensation tube. The schematic of an IHVAES was shown in Figure 3.1.

The lid of the pressure cooker was modified as shown in Figure 3.2. The handle was removed, and the smaller hole was covered by plastic tape. Barbed hose fitting and hose were added and connected to the larger hole. Silicone sealer (Mega Black Silicone Gasket Maker, Hartford, CT) was added around the hose fittings to prevent leakage. PTFE was added to the outside and inside of the lid to reduce friction of the magnetic coupling connected to an agitator. The agitator components are shown in Figure 3.3. The agitator (A) was printed using a 3D printer (AON3D, model AON-M2, Montreal, Canada) with ULTEM™ 9085 as the material. The handles (B) and connecting shaft (C) were printed by a 3D printer (WANHAO, model Duplicator 6, Jinhua, China) in Polylactic Acid. Four magnets, two on top and two on bottom of handles B, were used to transfer agitating forces from an outside mixer (OMNI, Kennesaw, GA) through magnetic coupling on the lid. This design could achieve continuous agitation without undermining the vacuum conditions in the scaled vessel. The clear hose labeled with B (McMaster-Carr 5393K44 Vacuum-Rated PVC Tubing) and the red connecting hose labeled with A (McMaster-Carr 5543K68 Abrasion-Resistant Gum Rubber Opaque Tubing) connected the modified pressure cooker to the vapor duct of the Rotavapor.

A receiving flask of volume 2 L was used for collection of vapor condensate. A labeled scale with 50 ml increments in volume was affixed to the flask so as to serve as a visual (quantitative) indication of the rate of evaporation.

3.3.2. Products tested

Sucrose solutions were prepared with granulated sugar (Domino, Premium pure cane, New York, NY) and tap water at three different concentrations, 10% w/v, 30% w/v, and 50% w/v. 10% w/v sucrose solutions were made by combining 100 g of sucrose and 935 ml of water in a 1 L volumetric flask, which yielded a total volume of 1 L solution. 30% w/v sucrose solutions were achieved by mixing of 300 g of sucrose with 807 ml of water. 50% w/v sucrose solutions were achieved by mixing of 500 g of sucrose with 680 ml of water. This process was repeated 3 times and a total of 3 L sucrose solutions were obtained for one evaporation experiment.

Three samples (weighted 591 g, 588.5 g, and 580.8 g) of protein polyphenol complex were obtained from SinnovaTek (Raleigh, NC) and consisted of mainly egg white and blueberries. Agitator was installed in this set of experiments to keep a uniform heating and prevent burning at the bottom.

3.3.3. Evaporation process

Three concentrations (10% w/v, 30% w/v, and 50% w/v) of sucrose solutions were prepared and 3 pressure levels (190 mbar, 160 mbar, and 130 mbar) were selected for testing the capability of the system. The power level of the induction cooker was set to the minimum during heating and evaporation because a higher power level led to over production of steam which

exceeded the capability of condensation of Rotavapor R-215 system. 10% w/v, 30% w/v, and 50% w/v sucrose solutions were evaporated for 220 min, 180 min, and 150 min, respectively. Each of 3 concentrations was tested under each of the 3 pressures. These experiments were conducted in triplicate, and in total, 27 experiments were conducted.

Hot plate (Thermo scientific, model SP131635, Waltham, MA) was chosen as an alternative heating source to compare energy efficiency to that of the induction heating system. The temperature was set at 400 °C to best match the rate of heating and evaporation with that by induction heating. 10% w/v sucrose solutions at 190 mbar and 50% w/v sucrose solutions at 130 mbar were tested as the two extreme conditions to facilitate comparison. Similar to what was done in the induction assisted evaporation system, 10% w/v and 50% w/v sucrose solutions were evaporated for 220 min and 150 min, respectively. Triplicate testing was performed, and in total, 6 experiments were conducted.

Three samples of protein polyphenol complexes were evaporated at 130 mbar, which was the highest degree of vacuum the IHVAES could achieve, to prevent potential protein degradation. The agitation system described earlier was incorporated to facilitate evaporation and prevent aggregation of proteins.

3.3.4. Temperature measurement

Two type K thermocouples (Omega, Norwalk, CT) were inserted into the bottom and top of the evaporation vessel for monitoring temperatures. The lower thermocouple was inserted 3 cm above the bottom of the pot and the tip of the thermocouple was placed horizontally at the center. The upper thermocouple was inserted 16 cm above the bottom of the pot and tip of the thermocouple was also placed horizontally at the center.

Temperature was measured every 5 minutes during the first 20 minutes and then every 10 minutes for the rest of the time. Temperature was also recorded when condensation started to form in the spherical receiving flask.

3.3.5. Energy measurement and determination of efficiency

Electricity usage was measured in kWh using a power meter (PUUCAI, model P06S-100, Ningbo, China) and recorded along with temperature measurements. Total electricity usage was also recorded at the end of each experiment. Energy efficiency was calculated for both heating methods by dividing volume of condensate by the electricity consumption (kWh).

3.3.6. Statistical analysis

Statistical analysis was performed using JMP[®], Version 14.2 (SAS Institute Inc., Cary, NC). A one-way analysis of variance (ANOVA) in conjunction with a Tukey's HSD test was used to determine the statistical significance for changing of boiling temperature and rate of evaporation among different concentrations and pressures at a confidence level of 95%. A one-way analysis of variance (ANOVA) in conjunction with a Student's T-test was used to determine the statistical significance of energy efficiency between IHVAES and hot plate assisted evaporation system at a confidence level of 95%.

3.4. Results and discussion

3.4.1. Evaporation of sucrose solutions

Electromagnetic interference occurred when type-K thermocouple was used for temperature measurement because the electromagnetic field generated by the induction cooker could induce voltage in the thermocouple wires, and hence affect the temperature reading. To overcome this problem, temperatures were only recorded when the induction cooker was periodically turned off, since the induction cooker (at the minimum power level) switched on for about 5 seconds and off for 3 seconds.

The boiling temperature data for different concentrations of sucrose solutions are shown in Table 3.1. The boiling temperature of all three concentrations of sucrose solutions decreased significantly as pressure decreased ($p < 0.05$). For example, a 10% w/v sucrose solution had boiling temperatures of 54.6 ± 0.2 °C, 58.7 ± 0.4 °C, and 61.5 ± 1.0 °C under pressures of 130 mbar, 160 mbar, and 190 mbar, respectively (Figure 3.4). Additionally, the boiling temperature of sucrose solution at 130 mbar, 160 mbar, and 190 mbar increased significantly as its concentration increased ($p < 0.05$). For example, the boiling temperatures of 10% w/v, 30% w/v, and 50% w/v sucrose solutions under pressure of 190 mbar were 60 °C, 60.4 °C, and 61.1 °C, respectively (Figure 3.5). Time-temperature results also showed that boiling point continued to rise as evaporation occurred. These boiling point elevations in both cases were caused by the increasing of total soluble solids content (sucrose molecules in this case) as the solvent evaporated and molality of the solution increased.

The results for average rate of evaporation (RoE) for different concentrations of sucrose solutions at different pressure are shown in Table 3.2. Figure 3.6 depicts the RoE of sucrose solutions increased as pressure increased, though the increase was not statistically significant.

The RoE for the 10% w/v sucrose solution under 190 mbar, 160 mbar, and 130 mbar pressure were 10.8 ± 0.3 ml/min, 11.1 ± 0.2 ml/min, and 11.4 ± 0.4 ml/min, respectively. Figure 3.7 shows that RoE decreased as concentration of sucrose solution increased from 10% w/v to 50% w/v at 190 mbar. From Table 3.2 it can be seen that the only significant difference in RoE was between the sucrose solution of low (10% w/v) concentration and the solution of high (50% w/v) concentration.

3.4.2. Comparison of energy efficiency for evaporations using induction heating versus hot plate

Energy efficiency (EE) results for evaporation using induction heating and hot plate are shown in Table 3.3. This does not include the energy consumption by the vacuum pump. The average EE using the hot plate were 923.3 ± 3.2 ml/kWh and 939 ± 45.1 ml/kWh for 10% w/v sucrose solution at 190 mbar pressure and 50% w/v sucrose solution at 130 mbar pressure, respectively. The average EE using induction heating were 1012.8 ± 25.2 ml/kWh and 1000 ± 25.2 ml/kWh for 10% w/v sucrose solution at 190 mbar pressure and 50% w/v sucrose solution at 130 mbar pressure, respectively. The results showed that induction assisted vacuum evaporation system had a significantly higher EE than that of a hot plate assisted vacuum evaporation system ($p < 0.05$). Two reasons might contribute to this result. First, induction heating induced eddy currents only at the bottom of the pot, thus induction heating directly heated the ferrous bottom of the pot through Joule (I^2R) effect and then heated the sucrose solution (Rudnev, Loveless, & Cook, 2017). However, the hot plate had to heat up the hot plate surface, then the bottom of the pot, and finally conduct heat to the sucrose solution. Second, the hot plate used in this experiment had a surface area larger than that of bottom of the pot. Heat generated at the areas exposed to air was lost to the air and therefore wasted. Although a smaller

heating surface area could improve the efficiency of hot plate, heat would still be lost from the side area that exposed to air because of the thickness of the hot plate surface. Figure 3.8 and Figure 3.9 depict the energy consumption and condensate volume throughout the experiment. These graphs indicate that induction heated evaporation had a higher energy efficiency than that by hot plate heated evaporation.

3.4.3. Evaporation of protein polyphenol complex

Three samples of protein polyphenol complexes were tested using IHVAES with an agitator installed. However, undesirable texture change (protein denaturation) and aroma (burnt smell) was observed in these experiments. Several reasons could be attributed to these observations. First, for all three samples, the agitator stopped turning during the first 10 min. Although the top (outer portion) of the agitator was still rotating, the bottom (inner portion) of the agitator stopped turning as the viscosity of the protein polyphenol complex increased. Second, although vacuum helped lower the boiling temperature, the boiling temperature of the lowest pressure level (130 mbar) was still higher than the denaturation temperature of egg protein. Third, the magnetic attraction between the two components on either side of the lid was too weak. Once the resistance to mixing exceeded the magnetic attraction, agitation inside the pot stopped while rotation on the outside continued. Last but not least, the bottom part of the agitator did not touch the bottom of the pot, and hence protein was denatured at the bottom and later resulted in being burnt because of excessive heat. This effect progressed upwards and resulted in protein denaturation and burning throughout the product.

3.5. Conclusion

The induction heated vacuum assisted evaporation system was capable of heating and evaporating sucrose solutions at reduced temperature under vacuum. This system could evaporate sucrose solutions at a rate of 9.9 ml/min at 54.6 °C at a pressure of 130 mbar and at a rate of 11.4 ml/min at 66 °C at a pressure of 190 mbar. This system also had a higher energy efficiency of 1012.8 ml/kWh compared to that of hot plate assisted heating 923.3 ml/kWh. Therefore, this system may have good potential for serving as a rapid and cost effective, small scale processing system, for low temperature evaporation of thermally sensitive materials.

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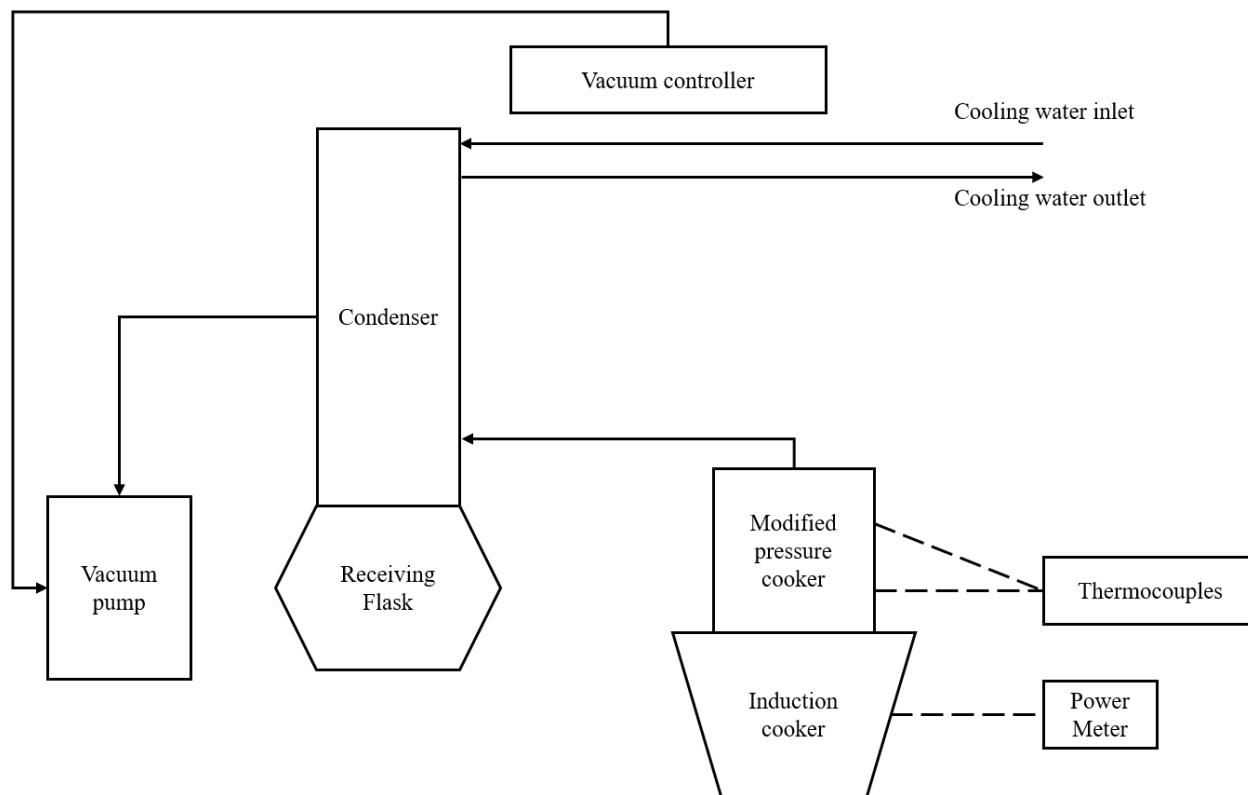


Figure 3.1. Schematic of IHVAES

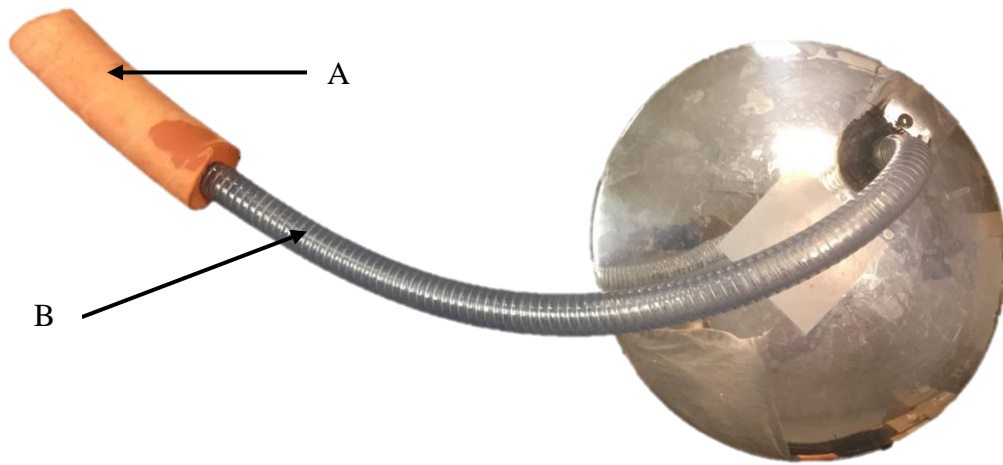


Figure 3.2 Lid of pressure cooker

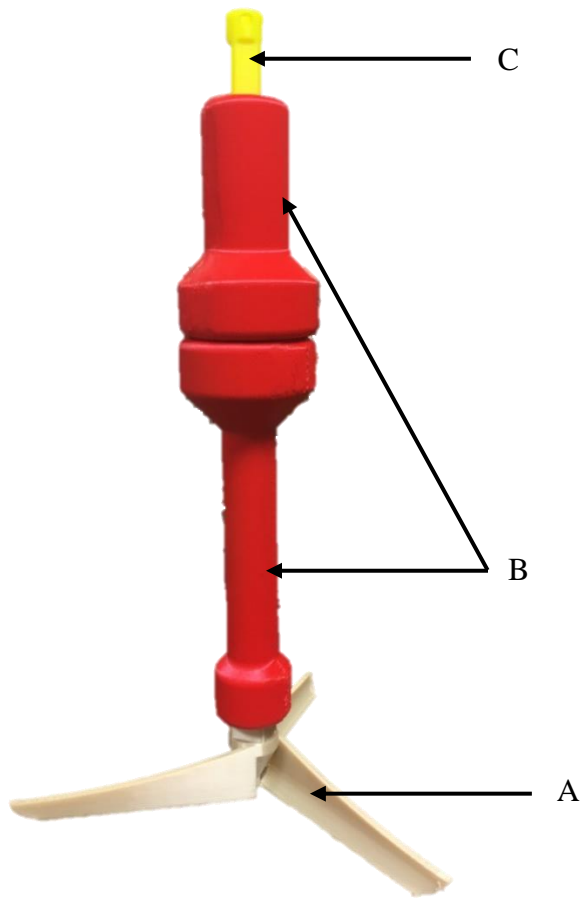


Figure 3.3. Agitator

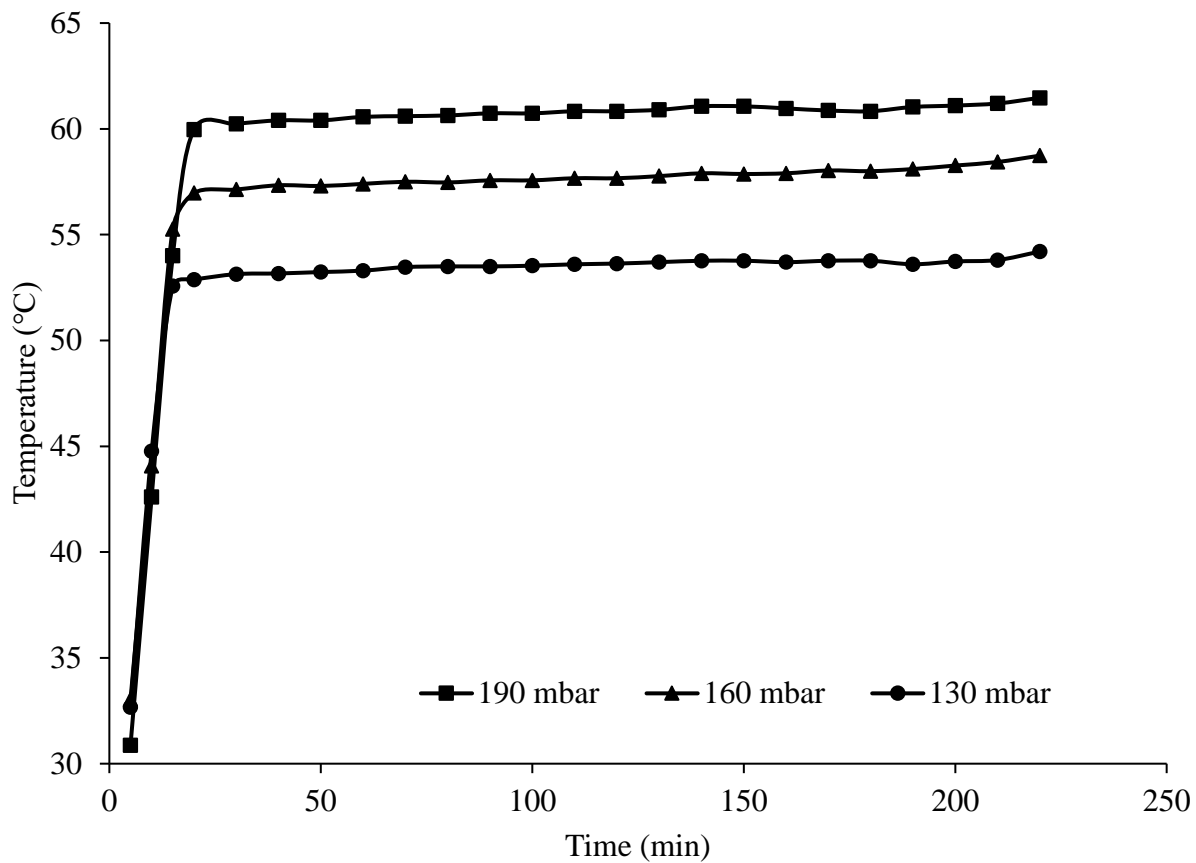


Figure 3.4. Time-temperature history for 10% w/v sucrose solutions under different pressures

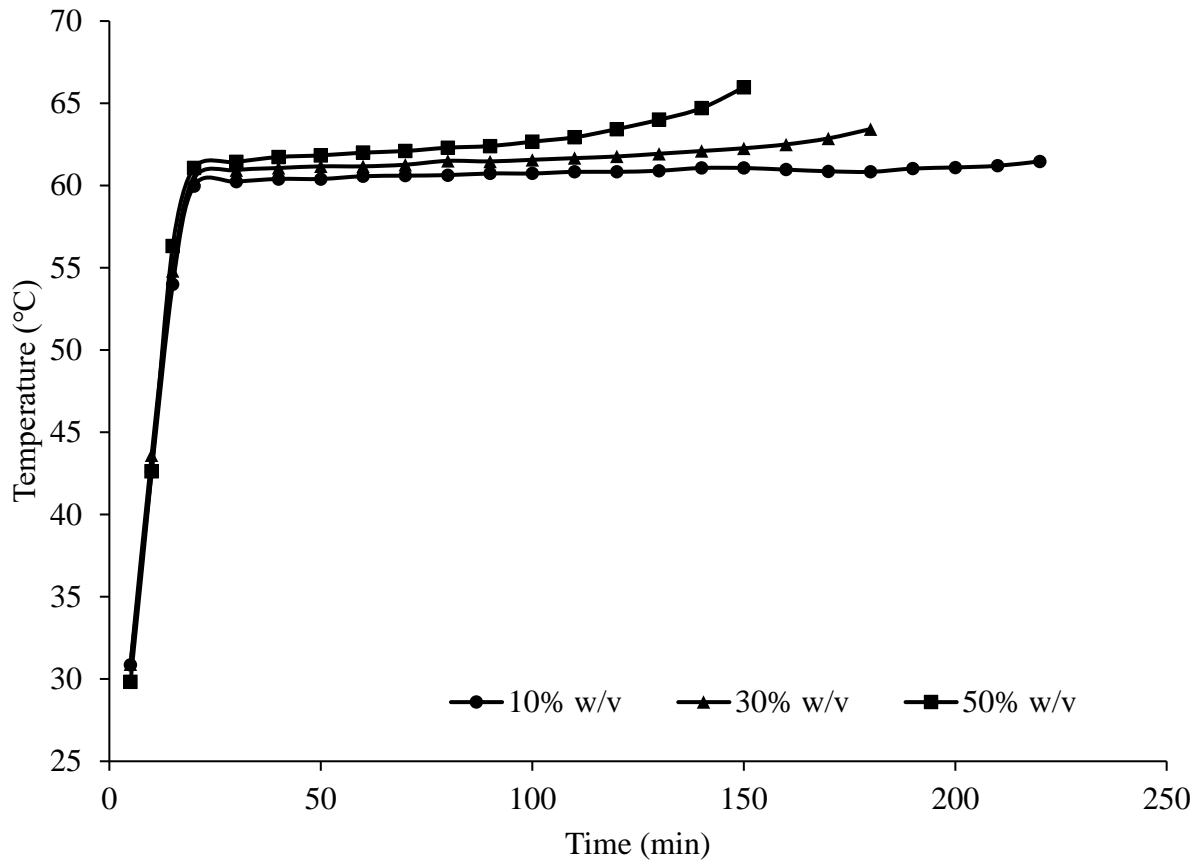


Figure 3.5. Time-temperature history for different concentrations of sucrose solutions under 190 mbar pressure

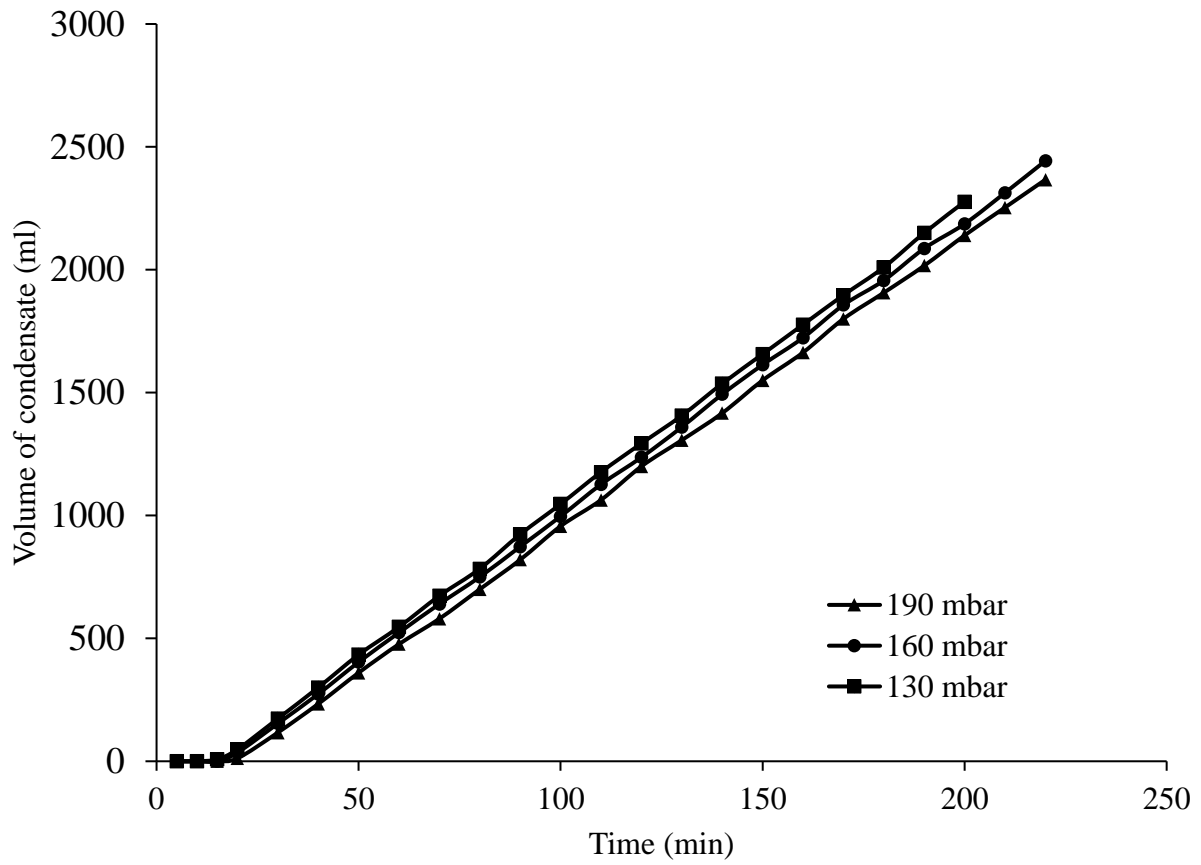


Figure 3.6. Rate of evaporation under different pressures for 10% w/v sucrose solutions

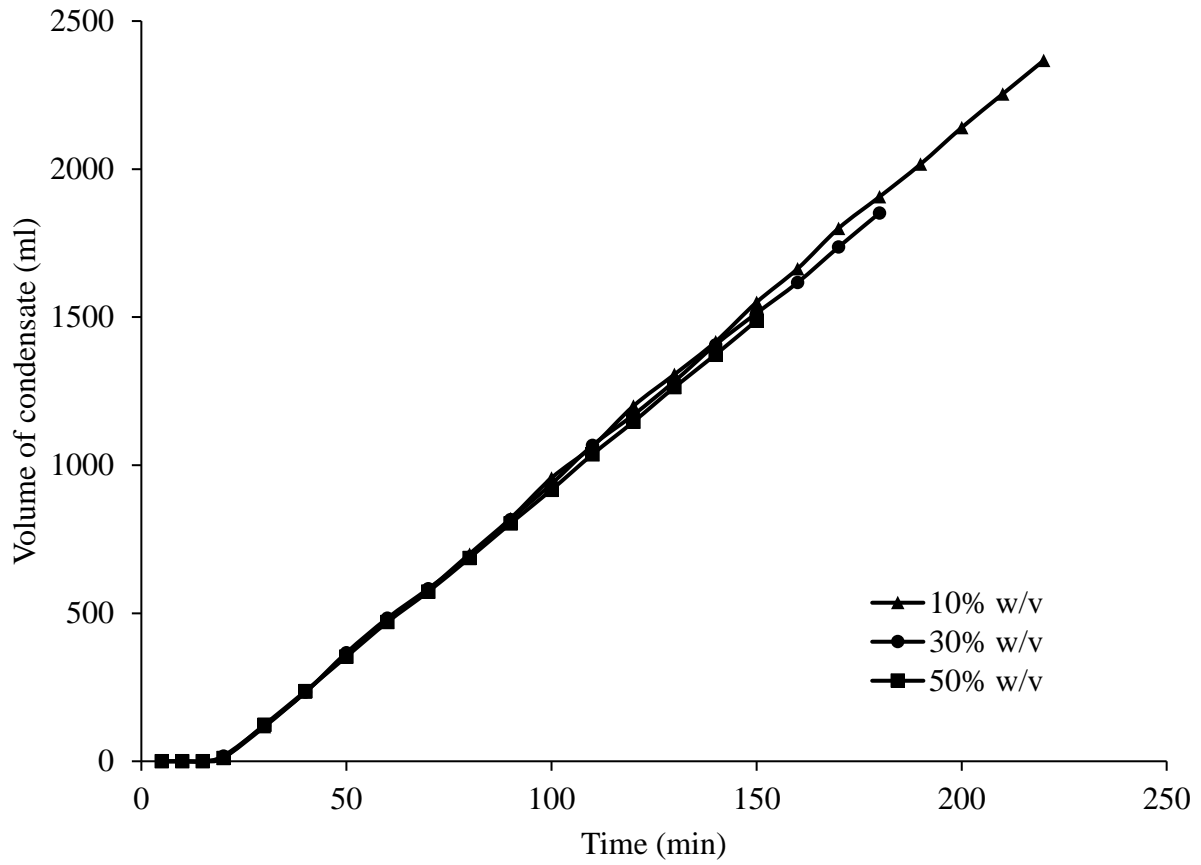


Figure 3.7. Rate of evaporation for different concentrations of sucrose solutions under 190 mbar pressure

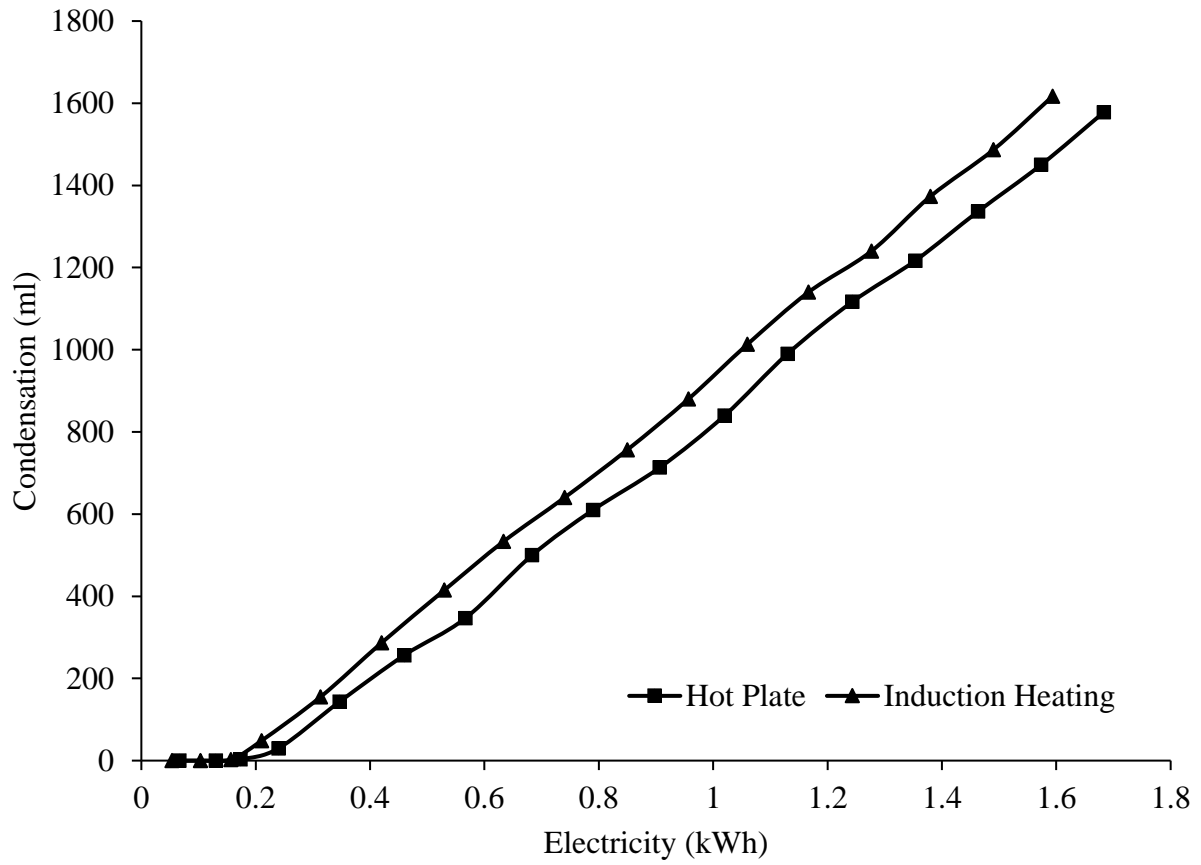


Figure 3.8. Electricity consumption of hot plate and induction heating system for evaporation of 50% w/v sucrose solutions under 130 mbar pressure

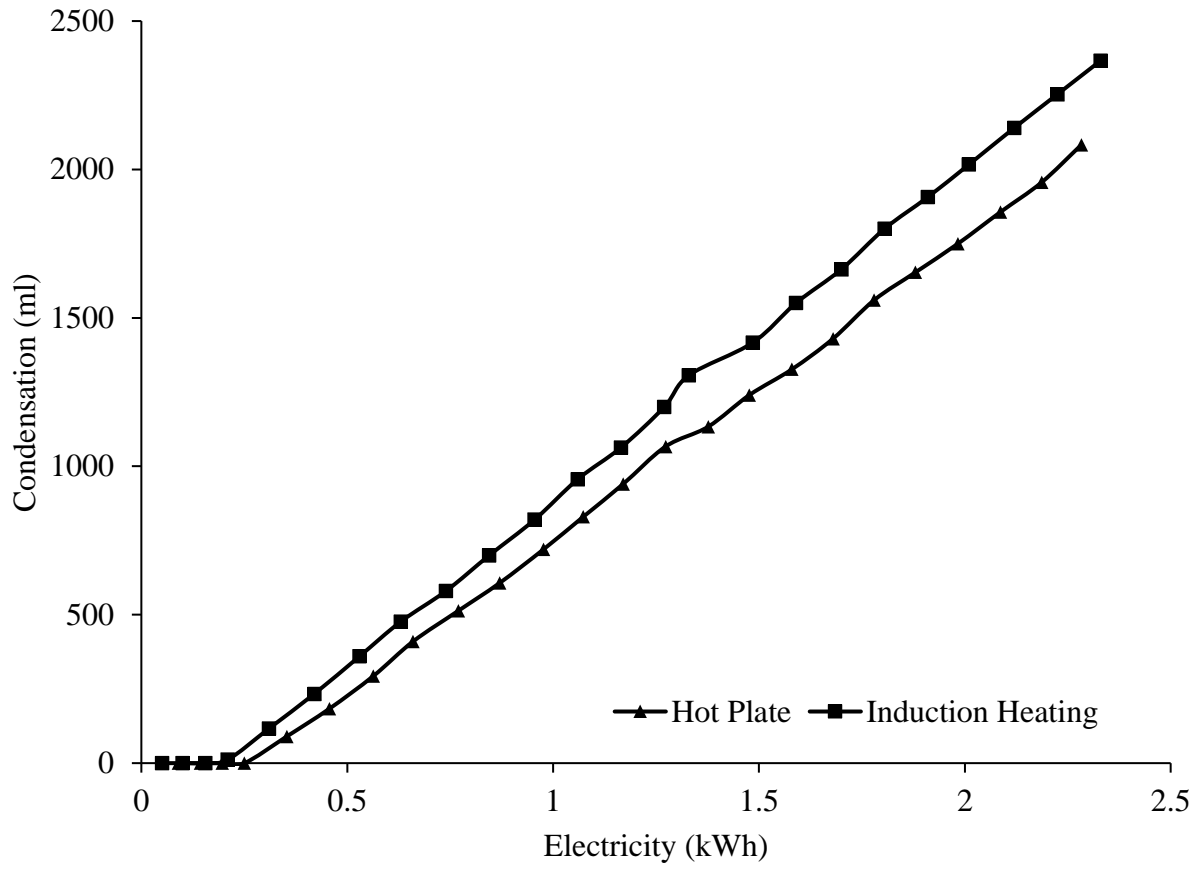


Figure 3.9. Electricity consumption of hot plate and induction heating system for evaporation of 10% w/v sucrose solutions under 190 mbar pressure

Table 3.1. Boiling temperatures of different concentrations of sucrose solutions under various pressures

	Concentrations	190 mbar	Std.	160 mbar	Std.	130 mbar	Std.
Boiling temperature (°C)	10% w/v	61.5	1.0	58.7	0.4	54.6	0.2
	30% w/v	63.4	0.4	59.8	0.5	56.4	0.5
	50% w/v	66.0	0.6	62.6	0.9	60.0	0.9

Table 3.2. Rate of evaporation of different concentration of sucrose solutions under various pressures (levels not connected by same letter are significantly different)

	Pressure	10% w/v	Std.	30% w/v	Std.	50% w/v	Std.
Rate of evaporation (ml/min)	130 mbar	11.4 ^a	0.4	11.0 ^{ab}	0.1	10.6 ^b	0.3
	160 mbar	11.1 ^a	0.2	10.4 ^{ab}	0.3	10.1 ^b	0.4
	190 mbar	10.8 ^a	0.3	10.3 ^{ab}	0.4	9.9 ^b	0.2

Different letters indicate there was significant difference between different concentrations of samples as per Tukey HSD test ($p < 0.05$).

Table 3.3. Energy efficiency comparison of evaporation assisted by hot plate and induction heating

Hot Plate					
Samples	Volume (ml)	Electricity consumption (kWh)	Energy Efficiency (ml/kWh)	Avg. EE (ml/kWh)	Std.
10% w/v 190 mbar	2115	2.29	923.6	923.3	3.2
	2070	2.25	920.0		
	2140	2.31	926.4		
50% w/v 130 mbar	1575	1.60	984.4	939.0	45.1
	1565	1.75	894.3		
	1595	1.70	938.2		
Induction Heating					
Samples	Volume (ml)	Electricity consumption (kWh)	Energy Efficiency (ml/kWh)	Avg. EE (ml/kWh)	Std.
10% w/v 190 mbar	2365	2.35	1006.4	1012.8	25.2
	2435	2.34	1040.6		
	2300	2.32	991.4		
50% w/v 130 mbar	1630	1.59	1025.2	1014.7	13.1
	1600	1.60	1000.0		
	1620	1.59	1018.9		

Chapter 4. Evaporation and Concentration of Bovine Milk and Infant Formula Using an Induction Heated Vacuum Assisted Evaporation System

4.1. Abstract

Preterm infants usually require a higher amount of protein and energy for the early development of their immune system and organs, which human milk cannot provide (Ballard & Morrow, 2013; Hay, 2018). Commercial fortified human milk is expensive and hence there is a need for concentration of human milk to meet the nutrient requirement of preterm infants. Bovine milk (BM), reconstituted Powder formula (PF), and Pre-mixed formula (PMF) as alternative model materials were concentrated by an induction heated vacuum assisted evaporation system (IHVAES) because of the limited supply of human milk. 500 ml of each material was evaporated under 130 mbar pressure for 30% and 60% volume reduction. The concentrate of each sample was collected and analyzed for fat and solids content. The results showed that BM boiled at 55 ± 1 °C under 130 mbar pressure whereas PMF and PF boiled at 53.5 ± 0.5 °C. The results also showed that evaporation of PF and PMF consumed significantly less energy and time compared to that of BM. Meanwhile, rate of evaporation was also higher for PF and PMF than that of BM. The results also showed that all samples had a significant increase in fat and solids content. Therefore, this study showed that the IHVAES was capable of concentrating BM, PF, and PMF and may have a good potential in concentrating human milk for preterm infants at a relatively low cost.

4.2. Introduction

Milk is sometimes referred to as a complete food because of its rich nutrient content of proteins, fat, carbohydrates (lactose), vitamins and minerals. Bovine milk contains about 3% proteins, 4.5% fat, 4.1% lactose, 0.8% vitamins and minerals, and the rest is water (Crowley, O'Mahony, & Fox, 2017). Human milk contains less protein (1%), more lactose (7%), and similar fat content (4.8%). However, there are abundant bioactive molecules in human milk which make it superior for infants in helping against inflammation and infection, as well as developing organs and immune system (Ballard & Morrow, 2013). Therefore, human milk is the optimal food for newborns. Preterm infants, on the other hand, require a higher protein and energy intake for body development, which human milk alone cannot provide (Hay, 2018). Although commercial fortified human milk can provide more protein and energy for preterm infants, it is expensive. Therefore, there is a need to increase the nutrient density of human milk.

Evaporation can increase the solids content of milk through partial removal of water by boiling. Vacuum is utilized during evaporation to minimize potential color changes and production of off flavors. A falling film evaporator is commonly used in production of milk powder and condensed milk to increase solids content because it is suitable for concentration of high viscosity and heat-sensitive materials (Park & Drake, 2016; Alhusseini, Tuzla, & Chen, 1998). However, steam as heating source can be expensive, especially in the evaporation process for large amount of water removal. Induction heating, on the other hand, is an electromagnetic non-contact heating technology which has advantages such as temperature uniformity, safety, reliability, energy efficiency, flexibility, and compactness of heating system (El-Mashad & Pan, 2017). However, little information could be found on using induction heating for evaporation of food products.

An induction heated vacuum assisted evaporation system (IHVAES) was designed and has proven its capability in evaporation and concentration of sucrose solution (Bi, 2019). Since human milk is a premium commodity and unavailable in a large quantity, bovine milk and infant formula (Similac) were used as alternative materials for testing because of their similar nutrient compositions. The goal of this study was to 1) test the functionality of IHVAES in concentration of dairy foods and 2) determine the optimum operating conditions for processing these products.

4.3. Materials and methods

4.3.1. Experimental setup

Induction heated vacuum assisted evaporation system (IHVAES) was designed and built (Bi, 2019). In addition, a glass bump trap (Chemglass, 250 ml, Vineland, NJ) was installed between the connect hose (A) and the vapor duct (B) of the Rotavapor as shown in Figure 4.1 because bumping (liquid product being drawn into the condenser) occurred during preliminary experiments of evaporating dairy products. A bump trap can prevent product from the pressure vessel from being drawn into the condenser. The inner vapor tube of the bump trap was sealed from the top to prevent product entry into the condenser, whereas holes at the bottom of the inner vapor tube helped the product drain back into the evaporation vessel.

4.3.2. Products tested

Four gallons of bovine milk (Pasteurized vitamin D milk) were obtained from a local grocery store (Food Lion, Raleigh, NC) and stored in a refrigerator before use. Similac Advance (Abbott, Chicago, IL) was bought in both powder and liquid form. Pre-mixed formula (PMF) was stored in a refrigerator before use. Powder formula (PF) was prepared as per instructions on

the package, mixing 1 scoop (8.7 g) of powder with 2 fl oz of water. To meet the minimum volume requirement (500 ml) of IHVEAS, PF was prepared before each experiment by mixing 16 fl oz (474 ml) of water and 8 scoops (69.6 g) of powder, which yielded a total volume of 520 ml. 20 ml was removed to facilitate comparison of time, energy consumption, and rate of evaporation (RoE) between BM and PF.

4.3.3. Experimental methodology

BM, PF, and PMF were evaporated for 30% and 60% volume reduction. These reduction levels were selected because they covered a relatively wide concentration range. All experiments were tested under 130 mbar pressure, which was the maximum degree of vacuum the system could attain. 500 ml of BM, PF, and PMF were prepared for each experiment. These volumes were selected because of the price and availability of materials. Evaporation of BP, PF, and PMF were stopped once 150 ml (30% volume reduction) and 300 ml (60% volume reduction) of condensate were obtained. Experiments were conducted in triplicate for bovine milk and powder formula at both concentration levels. PMF was only tested once for each volume reduction because of its high cost and similarity in composition with PF. After each evaporation, the concentrate was filled into plastic centrifuge tubes and stored in a refrigerator after initial cooling.

Bumping occurred only during the evaporation of bovine milk. Preliminary experiments were conducted on bovine milk, and it was found that shutting down the induction cooker for 3 minutes once bumping started and then restarting it could prevent bumping from happening again. Therefore, the induction cooker was shut down for 3 minutes once bumping started. Bumping did not occur after restarting the induction cooker.

Determination of solids and fat content was conducted using a microwave oven (CEM, model BR601050, Matthews, NC) and a rapid nuclear magnetic resonance fat analyzer (CEM, Oracle, Matthews, NC), respectively. These analyses were conducted in triplicate.

4.3.4. Statistical analysis

Statistical analysis was performed using JMP[®], Version 14.2 (SAS Institute Inc., Cary, NC). A one-way analysis of variance (ANOVA) in conjunction with a Tukey's HSD test was used to determine the statistical significance of evaporation of BM, PF, PMF regarding time, RoE, energy consumption, fat, and solids content at a confidence interval of 95%.

4.4. Results and discussion

4.4.1. Evaporation conditions

Bovine milk (BM), powder formula (PF), and pre-mixed formula (PMF) were evaporated for 30%, and 60% volume reduction under 130 mbar pressure. Experiment involving 30% and 60% volume reduction were conducted in triplicate except for PMF. PMF and PF are the same product except that PMF is ready to drink but PF needs to be properly mixed before consuming.

Under 130 mbar pressure, the boiling temperature of BM were 55 ± 1 °C with a maximum of 2 °C rise in temperature during evaporation, whereas the boiling temperature of PF and PMF was 53.5 ± 0.5 °C with a maximum of 2 °C rise in temperature during evaporation. Results of energy consumption (excluding energy used by the vacuum pump), time, and rate of evaporation are shown in table 4.1. Evaporation of PF and PMF took significantly less energy and less time and had a significantly higher rate of evaporation compared with evaporation of BM for both volume reductions ($p < 0.05$). Compositional differences between BM and PF resulted in the

difference in energy consumption, time and RoE. Results of PF and PMF were expected to be similar because the composition was the same for both products.

4.4.2. Fat and solids content analyses

Results of solids content and fat content are shown in Table 4.2 and Table 4.3. The initial fat and solids content were significantly different among BM, SMF, and PF ($p < 0.05$). The results showed that IHVAES successfully increased fat and solids content of BM by 45% and 49%, respectively, for 30% volume reduction, and 140% and 141%, respectively, for 60% volume reduction. Fat and solids content of PF were increased by 49% and 45% for 30% volume reduction, and 169% and 149% for 60% volume reduction. For PMF, fat and solids content were increased by 50% and 43% respectively for 30% volume reduction, and 162% and 143% respectively for 60% volume reduction. Results also showed that PMF and PF had higher fat and solids content than BM did. Evaporated BM, PF, and PMF contained significantly higher fats and solids compared to their control group (Figure 4.2). Thus, IHVAES was capable of concentration of BM, PF, and PMF.

4.4.3. Bumping during evaporation

Bumping only occurred during evaporation of bovine milk. To prevent product loss by bumping, the induction cooker was turned off for 3 min. This method effectively stopped further bumping. According to literature, bumping occurs when a product boils rapidly, forming bubbles that cause the sample being concentrated to flash out through the vacuum vent. In short, bumping occurs because of too much heat and a high degree of vacuum (Tennenhouse, 2017). Since evaporation at a relatively low temperature is desired, the pressure was not changed. Instead,

heat was shut off for a short duration (2 minutes in this case), which prevented further bumping. It was hypothesized that product was not heated uniformly, which caused formation of hot spots and resulted in bumping. However, preliminary experiments were conducted and preheating of product to boiling temperature under 130 mbar pressure did not prevent bumping. In addition, bumping did not occur during evaporation of PF and PMF. It is thus hypothesized that compositional differences between bovine milk and infant formula caused the differences in bumping.

4.5. Conclusion

The IHVAES was capable of concentrating BM, PF, and PMF and significantly increased the fat and solids content corresponding to the volume reduction. Therefore, this system has potentials to be used to concentrate human milk and may have a good potential for producing nutrient enriched human milk at a lower price in comparison to that of commercially available fortified human milk for preterm infants. Overall, this information assures the nutrient retention in bovine milk and infant formula, and could be useful in predicting the optimum operation conditions for evaporation of dairy products for achieving different volume reductions.

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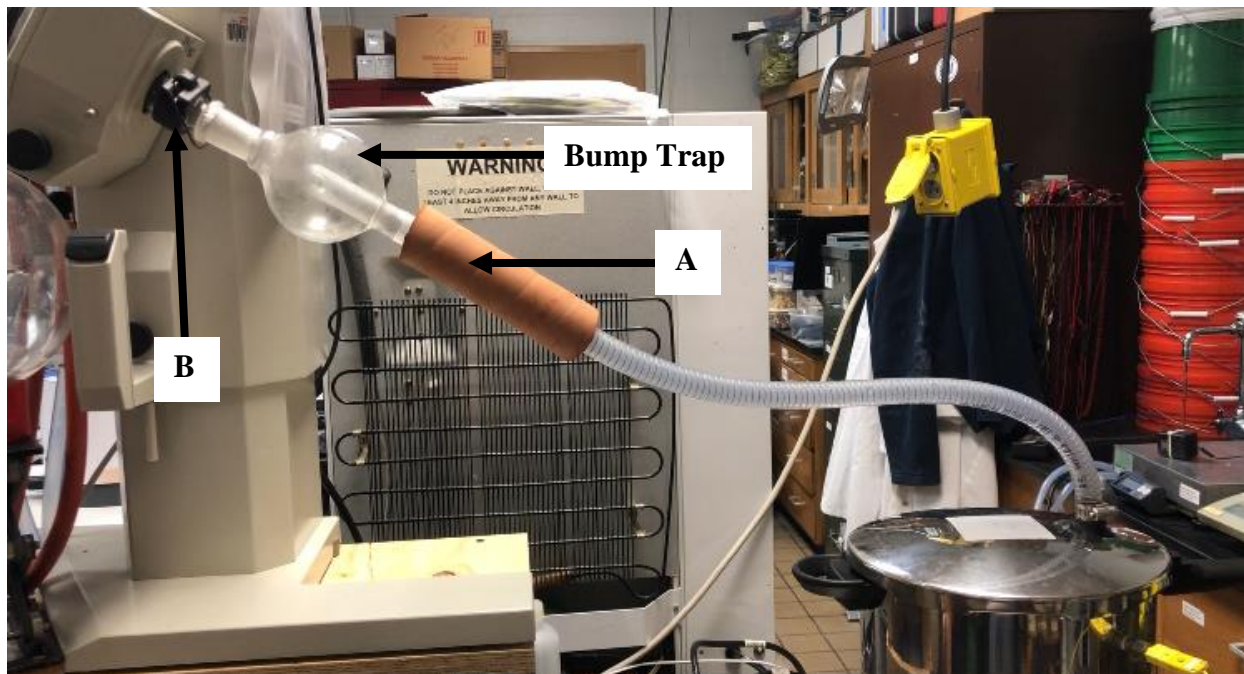
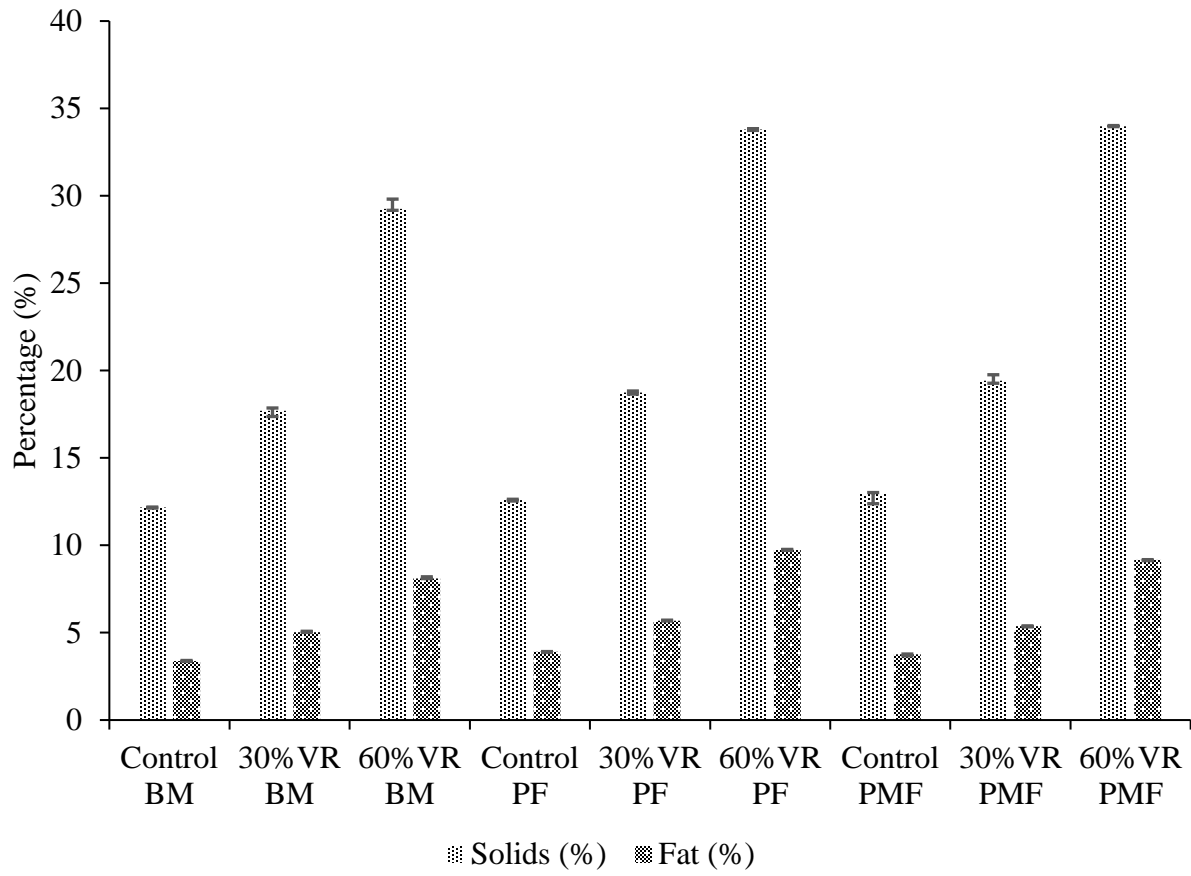


Figure 4.1. Illustration of bump trap



VR: Volume Reduction

Figure 4.2. Comparison of fat and solids content for different products

Table 4.1. Energy consumption, time, and rate of evaporation for 30% and 60% volume reduction of BM, PF, and PMF

	BM		PF		PMF	
	30% VR	60% VR	30% VR	60% VR	30% VR	60% VR
Energy (kWh)	0.2	0.33	0.18	0.31	0.18	0.31
Time (min)	19	32	17	30	17	30
RoE (ml/min)	7.9	9.4	9.2	10.33	9.2	10.33

(VR: volume reduction, and RoE: rate of evaporation)

Table 4.2. Solids analysis of bovine milk, powder formula, and pre-mixed formula

Volume Reduction (VR)			R1	R2	R3	Average
Bovine Milk (BM)	0% BM	Solids (%)	12.17 (± 0.02)			12.17 (± 0.02)
	30% BM	Solids (%)	17.89 (± 0.01)	17.54 (± 0.04)	17.69 (± 0.04)	17.70 (± 0.15)
	60% BM	Solids (%)	29.80 (± 0.03)	29.41 (± 0.05)	28.53 (± 0.04)	29.24 (± 0.57)
Powder Formula (PF)	0% PF	Solids (%)	12.58 (± 0.05)			12.58 (± 0.05)
	30% PF	Solids (%)	18.83 (± 0.03)	18.63 (± 0.04)	18.76 (± 0.02)	18.74 (± 0.09)
	60% PF	Solids (%)	33.80 (± 0.08)	33.79 (± 0.03)	33.80 (± 0.05)	33.79 (± 0.05)
Pre-mixed (PMF)	0% PMF	Solids (%)	12.94 (± 0.08)			12.94 (± 0.08)
	30% PMF	Solids (%)	19.43 (± 0.33)			19.43 (± 0.33)
	60% PMF	Solids (%)	33.97 (± 0.05)			33.97 (± 0.05)

Data are expressed as mean \pm SD of triplicates. R1, R2, and R3 are different replicates. Each replicate was measured 3 times for fat and solids content tests.

Table 4.3. Fat analysis of bovine milk, powder formula, and pre-mixed formula

Volume Reduction (VR)			R1	R2	R3	Average
Bovine	0% BM	Fat (%)	3.37 (± 0.04)			3.37 (± 0.04)
Milk	30% BM	Fat (%)	5.08 (± 0.04)	4.99 (± 0.02)	5.04 (± 0.03)	5.03 (± 0.05)
(BM)	60% BM	Fat (%)	8.18 (± 0.08)	8.14 (± 0.01)	8.00 (± 0.02)	8.11 (± 0.09)
Powder	0% PF	Fat (%)	3.91 (± 0.01)			3.91 (± 0.01)
Formula	30% PF	Fat (%)	5.71 (± 0.03)	5.67 (± 0.02)	5.70 (± 0.02)	5.69 (± 0.02)
(PF)	60% PF	Fat (%)	9.70 (± 0.06)	9.74 (± 0.04)	9.74 (± 0.02)	9.73 (± 0.04)
Pre-	0% PMF	Fat (%)	3.77 (± 0.01)			3.77 (± 0.01)
mixed	30% PMF	Fat (%)	5.38 (± 0.01)			5.38 (± 0.01)
(PMF)	60% PMF	Fat (%)	9.16 (± 0.02)			9.16 (± 0.02)

Data are expressed as mean \pm SD of triplicates. R1, R2, and R3 are different replicates. Each replicate was measured 3 times for fat and solids content tests.

Chapter 5. Evaporation and Concentration of Aqueous Extracts of Purple Sweet Potato Using an Induction Heated Vacuum Assisted Evaporation System

5.1. Abstract

Purple sweet potatoes are considered to be a healthy food source because of the antioxidant functionality of the anthocyanins in them. However, the low bioactivity (<1%) of anthocyanins in purple sweet potatoes makes it difficult to achieve health benefits. The goal of this study was to concentrate anthocyanins in aqueous extracts of purple sweet potato using an induction heated vacuum assisted evaporation system (IHVAES) and determine the optimum operating conditions for the process and the corresponding anthocyanin content. Aqueous extracts of purple sweet potato were obtained and evaporated to achieve 2X, 3X, 4X, and 5X concentrations. Results showed that IHVAES was capable of concentrating aqueous extracts of purple sweet potato at 57.2 ± 1 °C under 150 mbar pressure at a low power level. In addition, rate of evaporation and energy efficiency decreased as final concentration increased. Results also showed that the heat process resulted in 7% - 37% degradation of anthocyanins. IHVAES can thus be used to produce concentrated anthocyanins with minimal degradation, which can serve as a functional ingredient and colorant in the food industry.

5.2. Introduction

Purple sweet potatoes used to be cultivated as they are a good source of natural colorants, but they are now gaining popularity as a dietary source of antioxidants because of their abundant anthocyanin content (Steed, 2008). Anthocyanins are water soluble bioactive compounds that belong to a flavonoid group termed polyphenolic pigments and are responsible for many of the red, orange, blue, and violet colors present in plant parts such as fruits, flowers, and leaves (Wallace & Giusti, 2015). In addition, anthocyanins are also known to be natural antioxidants due to their ability to donate protons to quench highly reactive free radicals at the site of oxidative events (Sadilova, Carle, & Stintzing, 2007). Anthocyanins have been found to be effective in preventing further generation of free radicals and protecting cells from oxidative damage which has often been associated with aging and various diseases caused by oxidative stress (Boldt, Meyer, & Erwin, 2014). Recent studies have also shown support for consuming anthocyanin-rich fruits such as blueberry, cranberry, and bilberry in promoting health, but their low bioavailability (<1%) makes it highly unlikely to extend life span and prevent chronic diseases (Glover & Martin, 2012). Although anthocyanins are not essential nutrients and there are no deficiency disorders associated with the lack of anthocyanin consumption, effectively and efficiently extracting and concentrating a large amount of anthocyanins as natural oxidants can have potential benefits to the food and dietary supplement industries (Wallace & Giusti, 2015). For example, in the food industry, purple sweet potatoes can be processed into food colorants, paste, and flour and further incorporated into several food and beverage products such as noodles, bakery products, confectionery, juices, and soups (Suda, et al., 2003).

Given that anthocyanins can provide potential benefits to human health as natural antioxidants and nutritive food additives, ensuring their stability during extraction, concentration,

processing, and storage is of interest (Sui, 2017). Several factors (temperature, pH, light intensity, oxygen availability, presence of enzyme, ascorbic acid, sugar, metal ion, and structure of the anthocyanins) affect the stability of anthocyanins (Roobha, Saravanakumar, Aravindhan, & Devi, 2011). Temperature is one of the major factors affecting anthocyanin degradation during food processing (Sui, 2017). Therefore, developing an appropriate thermal method to prevent degradation of anthocyanins during concentration is critical.

The induction heated vacuum assisted evaporation system (IHVAES) has proven its ability in rapidly and efficiently concentrating products (Bi, 2019). Thus, the goals of this study were to 1) determine the optimum operating conditions for evaporation of aqueous extracts of purple sweet potato, and 2) determine the anthocyanin content of the concentrated products.

5.3. Materials and methods

5.3.1. Experimental setup

An induction heated vacuum assisted evaporation system (IHVAES) was used in this study (Bi, 2019). In addition, a glass bump trap (Chemglass, 250 ml, Vineland, NJ) was installed between the red connecting hose (A) and the vapor duct (B) of the Rotavapor as shown in Figure 4.1 because bumping (product being drawn into the condenser) occurred during preliminary experiments. A bump trap can prevent liquid product from the pressure vessel from being drawn to into the condenser. The inner vapor tube of the bump trap was sealed from the top to prevent product entry into the condenser, whereas holes at the bottom of the inner vapor tube helped the product drain back into the evaporation flask.

5.3.2. Products tested

Aqueous extracts of purple sweet potato (anthocyanin content of 20.87 ± 0.79 mg/g) were obtained by microwave assisted extraction (Bhatia, 2019). Materials were then stored in a freezer and were thawed for 30 min in a water bath before use.

5.3.3. Experimental methodology

Preliminary experiments were conducted on 500 ml (minimum product volume required for the IHVAES) of aqueous extracts (based on limited availability) of purple sweet potato. Four different concentration levels of 2X, 3X, 4X, and 5X were selected and tested under 150 mbar pressure, which was determined to reduce the occurrence of bumping based on preliminary experiments. The power of induction cooker was set to the lowest level as higher power levels yielded higher amount of steam, which exceeded the maximum condensing capacity of IHVAES. Concentrates were collected in dark centrifuge tubes and stored in a freezer to prevent degradation caused by heat and light. Experiments were conducted in triplicate for each concentration level. A spectrophotometer was used to determine the anthocyanin content in the products.

5.3.4. Quantification of total anthocyanins

The total anthocyanin content was determined using the pH differential method as described by Giusti and Wrolstad (2001). The following equations were used to calculate the total anthocyanin content:

$$A = (A_{530} - A_{700})_{pH1.0} - (A_{530} - A_{700})_{pH4.5}$$

where A_{530} and A_{700} are the absorbance of the solutions measured at 530 nm and 700 nm respectively. The yielded absorbance (A) was then used to determine the total anthocyanin content:

$$\text{Total anthocyanin content (mg/L)} = \frac{A \times MW \times DF \times 1000}{\epsilon \times I}$$

Here, molecular weight (MW) = 449.2 g/mol, dilution factor (DF) = 5, path length (I) = 1 and extinction coefficient (ϵ) = 29600 L·mol⁻¹·cm⁻¹. The total anthocyanin content was reported as mg/100g of purple sweet potatoes.

5.3.5. Statistical analysis

Statistical analysis was performed using JMP[®], Version 14.2 (SAS Institute Inc., Cary, NC). A one-way analysis of variance (ANOVA) in conjunction with a Tukey's HSD test was used to determine the statistical significance of aqueous extracts of purple sweet potato regarding RoE, energy efficiency, and anthocyanin content on at a confidence interval of 95%.

5.4. Results and discussions

5.4.1. Evaporation conditions

Aqueous extracts of purple sweet potato were evaporated at a pressure of 150 mbar instead of 130 mbar because preliminary experiments indicated that occurrence of bumping was lower at a pressure of 150 mbar. The boiling temperature of anthocyanin extract under 150 mbar pressure was measured to be 57.2 ± 1 °C. Results of time, volume of condensate, rate of evaporation, and energy consumption at each concentration are shown in Table 5.1. It was observed that the volume of concentrate was always less than the desired volume. For example, a 2X concentration of 500 ml sample should yield 250 ml condensate and 250 ml of concentrate.

In reality, when 250 ml of condensate was collected, there would always be less than 250 ml left in the pressure vessel. Approximately 10 - 20 ml of water vapor was trapped and further condensed (by losing energy to the outside environment) in the bump trap and condenser. This volume was not accounted for, in the condensate, and hence the volume of concentrate was always less than the desired volume.

Rate of evaporation of 2X concentrate was 9.2 ml/min and increased to 9.4 ml/min, 9.8 ml/min, and 9.9 ml/min for 3X, 4X, and 5X concentrates respectively. Energy efficiency for the 2X concentrate product was 868 ml/kWh, and increased to 888 ml/kWh, 928 ml/kWh, and 941 ml/kWh for 3X, 4X, and 5X concentrate product, respectively. These results showed that rate of evaporation and energy efficiency seem to increase as product concentration increased. However, these increases were not statistically significant possibly due to inaccurate measurement of condensate volume as a result of bumping.

5.4.2. Anthocyanins analysis

The anthocyanin content in the concentrated products are shown in Table 5.2. The anthocyanin content of the original aqueous extract of purple sweet potato was 20.87 ± 0.80 mg/100g. On average, the 2X, 3X, 4X, and 5X had significantly higher anthocyanin contents of 38.70 ± 1.96 mg/100 g, 48.25 ± 2.94 mg/100 g, 52.94 ± 0.50 mg/100 g, and 77.37 ± 3.99 mg/100g respectively ($p < 0.05$). Degradation occurred during evaporation and it was determined that 2X, 3X, 4X, and 5X concentrated products had anthocyanin degradation of 7%, 23%, 37%, and 26% respectively.

Figure 5.2 shows that the degradation of anthocyanins was not linear across different concentrations, despite previous study reported a first-order reaction model for the degradation

of monomeric anthocyanins from different products (Kırca, Özkan, & Cemeroğlu, 2007; Yang, Han, Gu, Fan, & Chen, 2008). 3X concentration and 5X concentration had a similar level of anthocyanin degradation, while 4X concentration had a much higher level of anthocyanin degradation. Previous studies have indicated that temperature, pH, light intensity, oxygen availability, presence of enzyme, ascorbic acid, sugar, and metal ion could affect the stability of anthocyanins (Roobha, Saravanakumar, Aravindhana, & Devi, 2011). In this study, temperature and processing time were likely the main factors which caused degradation of anthocyanins. Additionally, storage temperature, exposure to light, and oxygen affect the bioavailability of anthocyanin during storage. Higher anthocyanin degradation of the 3X and 4X samples may be due to inaccurate reading of condensate volume and variation in initial anthocyanin content of the products.

5.4.3. Bumping during evaporation

Bumping occurred when evaporating aqueous extracts of purple sweet potato. It could be seen that a purple color fluid entered the bump trap and was retained in it or flowed back into the pressure cooker. Purple fluid droplets were also seen on the underside of the lid and inside wall of the pressure cooker after evaporation (Figure 5.3). Although majority of the concentrate was collected and further analyzed, product droplets on the wall were not salvaged. Anthocyanins contained in those droplets might be more or less concentrated than the final concentrate, and could hence affect the accuracy of the data.

5.5. Conclusions

This study investigated the effect of concentration using an IHVAES under 150 mbar pressure on the anthocyanin content. The results showed that IHVAES was capable of evaporating water and concentrating anthocyanins in aqueous extracts of purple sweet potato at a pressure of 150 mbar and at a boiling temperature of 57.2 ± 1 °C. It was seen that the overall rate of evaporation and energy efficiency increased as final concentration increased. In addition, degradation during concentration of anthocyanins using an IHVAES was not linear and ranged from 7% - 37%. Therefore, this system has the potential to be used to concentrate other bioactive thermally sensitive food materials such as fruit juices, purees, sauces, and probiotic foods.

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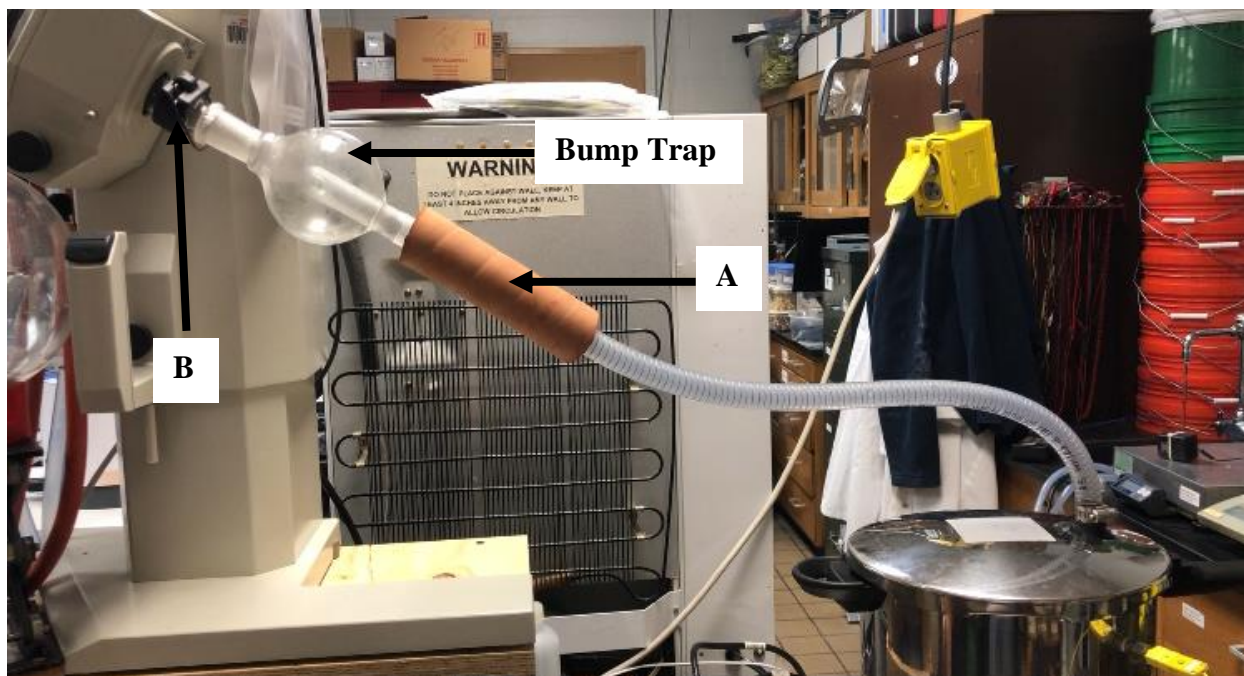


Figure 5.1. Illustration of bump trap

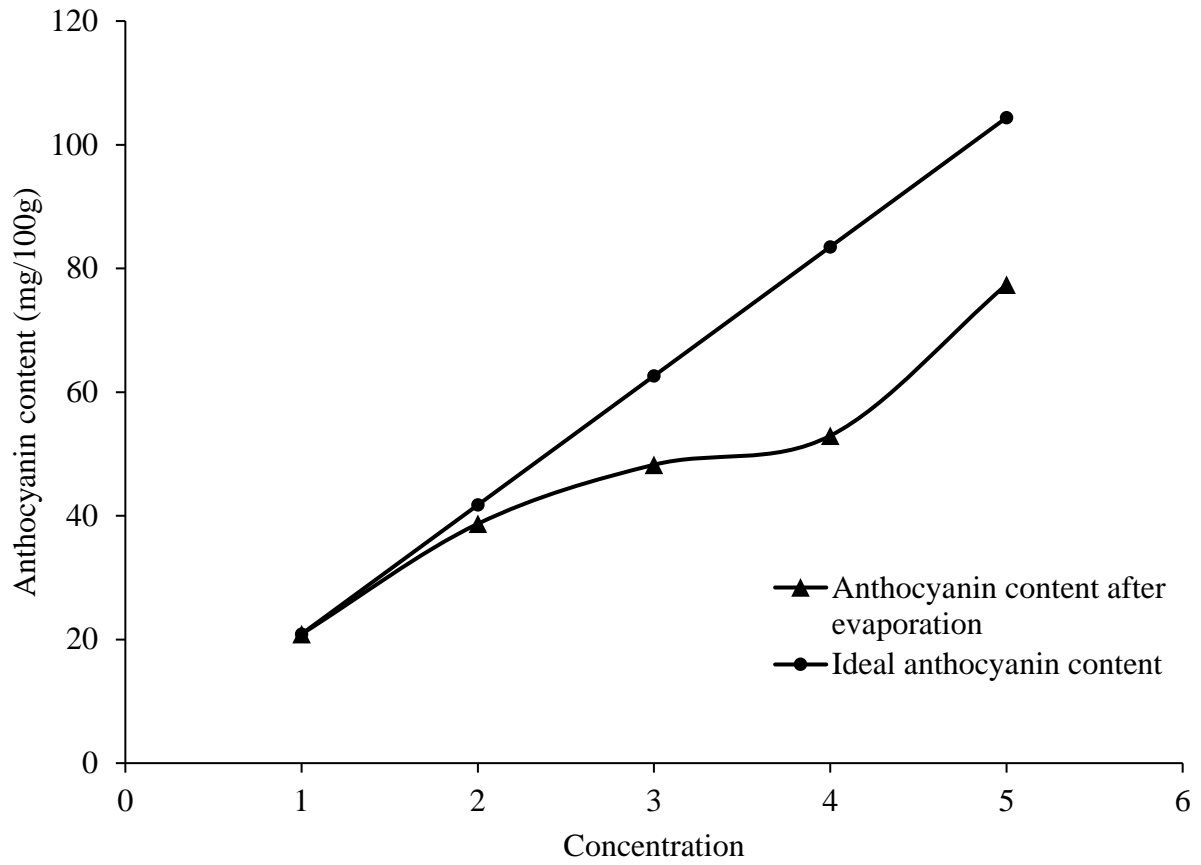


Figure 5.2. Degradation of anthocyanins at different concentrations



Figure 5.3. Anthocyanin residue after bumping

Table 5.1. Results of evaporation for different concentrations

	2X	3X	4X	5X
Time (min)	27.3	33.7	35.0	37.3
Volume of condensate (ml)	252	317	343	370
Rate of evaporation (ml/min)	9.2	9.4	9.8	9.9
Energy consumption (kWh)	0.29	0.36	0.37	0.39
Energy efficiency (ml/kWh)	868	888	928	941

Table 5.2 Anthocyanin content in different samples

Samples	Anthocyanins (mg/100 g)	Average (mg/100 g)	Std. (mg/100 g)
1X-Rep 1	21.49		
1X-Rep 2	21.16	20.87	0.80
1X-Rep 3	19.97		
2X-Rep 1	40.91		
2X-Rep 2	37.99	38.70	1.96
2X-Rep 3	37.19		
3X-Rep 1	45.54		
3X-Rep 2	47.84	48.25	2.94
3X-Rep 3	51.37		
4X-Rep 1	52.74		
4X-Rep 2	52.57	52.94	0.50
4X-Rep 3	53.50		
5X-Rep 1	78.03		
5X-Rep 2	73.10	77.37	3.99
5X-Rep 3	80.99		

Chapter 6. Recommendations for Future Work

IHVAES was only operated and tested at a low power level due to limited capability of the Rotavapor in condensing vapors. A large condenser could handle more steam and enable operation at a higher power level, which could potentially accelerate the evaporation process and reduce loss in product quality. The results of this research also showed that temperature readings were not stable because of electromagnetic interference (EMI) caused by induction heating. Fiber optic probes are unaffected by EMI and could be used to monitor temperature. In addition, power consumption of the vacuum pump needs to be measured in the future to better compare energy consumption and efficiency with other heating sources. Furthermore, the agitator should incorporate more magnets to overcome the resistance of high viscosity materials.

To better understand the capability of IHVAES for concentration of dairy products, the protein and lactose content of the concentrated product should be determined. Future research is also needed to understand the differences between bovine milk and infant formula to better understand the cause of bumping and then accordingly modify the IHVAES to minimize or prevent bumping without interfering with the concentration process. Since bovine milk and infant formula performed well in the IHVAES, human milk may also be concentrated with the IHVAES for production of nutrient enriched human milk. This may turn out to be better than the commercially fortified human milk.

To obtain accurate product concentration, a method of volume measurement should be developed to accurately monitor the volume of the product in the pressure vessel. Furthermore, since degradation of anthocyanins was uneven across different concentrations, future research is needed to determine the factors that affect degradation of anthocyanins during vacuum

evaporation. Last but not least, future research is needed to minimize or eliminate bumping which occurred during evaporation of bovine milk and aqueous extracts of purple sweet potato.

APPENDICES

Appendix A

Appendix A. Raw data of solids and fat analyses of bovine milk, powder formula, and pre-mixed formula

Volume Reduction			R1			R2			R3		
Bovine Milk (BM)	Control	Solids (%)	12.17	12.16	12.19						
	BM	Fat (%)	3.37	3.33	3.41						
	30% VR	Solids (%)	17.89	17.88	17.89	17.49	17.56	17.57	17.73	17.67	17.66
	BM	Fat (%)	5.05	5.06	5.12	4.97	4.99	5.00	5.07	5.03	5.01
	60% VR	Solids (%)	29.76	29.82	29.82	29.41	29.36	29.45	28.57	28.52	28.49
	BM	Fat (%)	8.10	8.18	8.25	8.13	8.15	8.15	8.02	7.98	7.99
Powder Formula (PF)	Control	Solids (%)	12.54	12.57	12.64						
	PF	Fat (%)	3.91	3.92	3.91						
	30% VR	Solids (%)	18.80	18.86	18.82	18.60	18.63	18.67	18.76	18.75	18.78
	PF	Fat (%)	5.74	5.69	5.70	5.67	5.69	5.66	5.70	5.71	5.68
	60% VR	Solids (%)	33.71	33.85	33.83	33.76	33.78	33.82	33.85	33.80	33.75
	PF	Fat (%)	9.65	9.70	9.76	9.78	9.70	9.74	9.72	9.74	9.75
Pre- mixed Formula (PMF)	Control	Solids (%)	12.89	12.91	13.03						
	PMF	Fat (%)	3.76	3.76	3.78						
	30% VR	Solids (%)	19.81	19.27	19.20						
	PMF	Fat (%)	5.39	5.38	5.37						
	60% VR	Solids (%)	33.93	33.97	34.02						
	PMF	Fat (%)	9.16	9.15	9.18						

R1, R2, and R3 indicate three replicates.

Appendix B

Appendix B. Raw data of anthocyanin content in different samples

Samples	Abs 530 nm	Abs 700 nm	Net Abs 1	Abs 530 nm	Abs 700 nm	Net Abs 2	Final Absorbance	Anthocyanins (mg/L)	Anthocyanins (mg/100g)	Average (mg/100g)	Std. (mg/100g)
	pH1	pH1	pH1	pH4.5	pH4.5	pH4.5					
1X-Rep 1	0.3015	0.0023	0.2992	0.0507	0.0089	0.0418	0.2574	21.4915	21.4915	20.8736	0.7986
1X-Rep 2	0.2990	0.0019	0.2971	0.0531	0.0094	0.0437	0.2534	21.1575	21.1575		
1X-Rep 3	0.2887	0.0026	0.2861	0.0538	0.0069	0.0469	0.2392	19.9719	19.9719		
2X-Rep 1	0.2961	0.0110	0.2851	0.0640	0.0239	0.0401	0.245	20.4561	40.9123	38.6969	1.9600
2X-Rep 2	0.2826	0.0115	0.2711	0.0606	0.0170	0.0436	0.2275	18.9950	37.9900		
2X-Rep 3	0.2801	0.0139	0.2662	0.0614	0.0179	0.0435	0.2227	18.5942	37.1884		
3X-Rep 1	0.2275	0.0086	0.2189	0.0489	0.0118	0.0371	0.1818	15.1793	45.5379	48.2514	2.9396
3X-Rep 2	0.2318	0.0064	0.2254	0.0448	0.0104	0.0344	0.191	15.9474	47.8423		
3X-Rep 3	0.2431	0.0059	0.2372	0.0444	0.0123	0.0321	0.2051	17.1247	51.3741		
4X-Rep 1	0.2336	0.0251	0.2085	0.0788	0.0282	0.0506	0.1579	13.1838	52.7351	52.9355	0.4987
4X-Rep 2	0.2356	0.0266	0.209	0.0829	0.0313	0.0516	0.1574	13.1420	52.5681		
4X-Rep 3	0.2421	0.0272	0.2149	0.0829	0.0282	0.0547	0.1602	13.3758	53.5032		
5X-Rep 1	0.2753	0.0361	0.2392	0.0849	0.0326	0.0523	0.1869	15.6051	78.0255	77.3715	3.9856
5X-Rep 2	0.2726	0.0370	0.2356	0.0956	0.0351	0.0605	0.1751	14.6199	73.0994		
5X-Rep 3	0.2830	0.0318	0.2512	0.0913	0.0341	0.0572	0.194	16.1979	80.9896		

R1, R2, and R3 indicate three replicates.