Spray Drying of Liquorice (Glycyrrhiza glabra) Extract

A Master Thesis
in
Food Engineering
University of Gaziantep

Supervisor
Assist. Prof. Dr. A. Coşkun DALGIÇ

by

İrem KARAASLAN

December 2010

T.C.
UNIVERSITY OF GAZİANTEP
GRADUATE SCHOOL OF
NATURAL & APPLIED SCIENCES
FOOD ENGINEERING

Spray Drying of Liquorice (Glycyrrhiza glabra) Extract
İrem KARAASLAN
26.11.2010

Approval of the Graduate School of Natural and Applied Sciences

Prof. Dr. Ramazan KOÇ
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science

Prof. Dr. Ali Rıza TEKİN
Chairman of Department

I certify that I have read this thesis and that in my opinion it is fully adequate , in scope and quality, as a thesis for the degree of Master of Science.

Assist. Prof. Dr. A.Coşkun DALGIÇ
Supervisor

Examining Committee in Charge:

Prof. Dr. Medeni MASKAN (Chairman) .....
Assist. Prof. Dr. Hasan VARDİN .....
Assist. Prof. Dr. Çiğdem SOYSAL .....
Assist. Prof. Dr. K. Bülent BELİBAĞLI .....
Assist. Prof. Dr. A. Coşkun DALGIÇ .....

ABSTRACT

SPRAY DRYING OF LIQUORICE (GLYCYRRHIZA GLABRA) EXTRACT

KARAASLAN, İrem
M. Sc. In Food Engineering
Supervisor: Assist. Prof. Dr. A. Coşkun DALGIÇ
December 2010, 73 pages

Liquorice root was first leached in water. Three leaching tempratures (4 °C, 25°C, and 37°C) and liquorice/water ratios (1/5 w/w, 1/4 w/w, and 1/3 w/w) were selected. Leaching at 25 ˚C by using the 1/3 as the ratio of liquorice/water was observed as the optimum extraction procedure. The extract obtained was then evaporated by using rising film evaporator. After evaporation, spray drying process was used to determine the optimum processing conditions that yield minimum color change, and acceptable moisture content, bulk density, hygroscopicity, acidity & pH, solubility. Maltodextrin as drying agent was developed. 12 DE, 18 DE, and 19 DE maltodextrins were used as drying agents. Liquorice extract was spray dried at inlet air temperatures of 110 °C, 120 °C, and 130°C and maltodextrin concentrations of %20, % 15, and %10. Data for the yields was obtained and the powders were analyzed for moisture content, bulk density, color change, hygroscopicity, acidity & pH, solubility. Increases in inlet air temperature were caused an increase in yield, pH , solubility and a decrease in moisture content, bulk density, hygroscopicity, L*, a*, b*, acidity. Increases in maltodextrin concentrations were caused an increase in yield, L*, b*, acidity and a decrease in moisture content, bulk density, hygroscopicity, a*, pH, solubility. Increases in DE maltodextrins were caused an increase in bulk density, hygroscopicity, L*, Ph and a decrease in yield, moisture content, a*, b*, acidity, solubility.

Key words: Liquorice, Liquorice powder, maltodextrin, stickness

ÖZ

MEYAN ÖZÜTÜNÜN PÜSKÜRTMELİ KURUTUCU İLE KURUTULMASI

KARAASLAN, İrem
Yüksek Lisans Tezi, Gıda Mühendisliği Bölümü
Tez Yöneticisi: Assist. Prof. Dr. A. Coşkun DALGIÇ
Aralık 2010, 73 sayfa

İlk olarak meyan kökünün suda özütü çıkarılmıştır. Bu işlem için üç sıcaklık (4 °C, 25°C ve 37°C) ve üç meyan/su oranı (1/5, 1/4, and 1/3) belirlenmiştir. Meyan özütünün çıkarılması ile ilgili 1/3 oranında meyan/su karışımının 25°C de bekletilmesi en etkin metod olarak tespit edilmiştir. Çıkarılan özüt daha sonra evapore edilmiştir. Evaporasyon sonrasında, meyan özütü, asgari renk değişimine, kabul edilebilir seviyede nem, yoğunluk, nem tutma kapasitesi, asitlik, pH, çözünürlük değerine sahip ideal işleme koşullarının tespit edilebilmesi için püskürtmeli kurutucu ile kurutulmuştur. Kurutma yardımcı maddesi olarak DE 12, DE 18, DE 19 değerli maltodekstrinler kullanılmıştır. Meyan özütü; 110°C, 120°C ve 130°C hava giriş sıcaklığında , maltodekstrin konsantrasyonu %20, %15 ve %10 olacak şekilde kurutulmuştur. Elde edilen ürünle ilgili (verim) veriler toplandı ve toz ürünler nem, yoğunluk, renk değişimi, asitlik & pH için analiz edildi. Hava giriş sıcaklığındaki artış, verim, pH ve çözünürlükte artışa; nem, yoğunluk, su tutma kapasitesi, L*, a*, b* ve asitlikte azalmaya sebep olmuştur. Maltodekstrin konsantrasyonundaki artış,verim, L*, b*, asitlikte artışa ; nem, yoğunluk, su tutma kapasitesi, a*, pH ve çözünürlükte azalmaya sebep olmuştur. Maltodekstrin DE değeri arttıkça, yoğunluk, su tutma kapasitesi, L*, pH artmış ; verim, nem, a*, b*, asitlik, çözünürlük azalmıştır.

Anahtar kelimeler: Meyan, Meyan tozu, maltodekstrin, yapışkanlık

ACKNOWLEDGEMENT

I wish to express my deepest gratitude to my supervisor Assist. Prof. Dr. A. Coşkun DALGIÇ for his patient supervision, continuous guidance and encouragement. I always fell indebted him due to his supports through my M. Sc. studies as perfect scientist. Invaluable help of his will always be living along this thesis.

I would like to take the opportunity to thank Prof. Dr. Medeni MASKAN, Assist Prof. Dr. Hasan VARDİN and Assit Prof. Dr. K. Bülent BELİBAĞLI for their advices and for sharing their expertise about spray drying, methods and analysis done. Many thanks to Prof. Dr. Sevim KAYA and Assist. Prof. Dr. Çiğdem SOYSAL for allowing me the use of their labaratoary tools and for making my study easier with their help.

A special note of appreciation goes to my family for always believing in me, their continuous help, and their encouragements.

To my family

Reşat, Gülseren, and Eren

TABLE OF CONTENTS

	Page
ABSTRACT……..	iii
ÖZ…….....	iv
ACKNOWLEDGEMENTS…….	v
TABLE OF CONTENTS…….....	vii
LIST OF TABLES…….....	ix
LIST OF FIGURES……..	x
1. INTRODUCTION…….....	1
1.1. Liquorice……..……..…….....	1
1.2. Production of Liquorice Concentrate…….....	4
1.2.1. Solid Liquid Extraction (Leaching)…….....	4
1.2.2. Evaporation…….....	5
1.3. Drying……...…….....	7
1.4. Quality Parameters of Liquorice Powders.....	12
1.4.1. Moisture Content …….....	12
1.4.2. Bulk Density…….....	13
1.4.3. Hygroscopicity …….....	14
1.4.4. Color …….....	15
1.4.5. Acidity & pH .....….....…….....…….....	16
1.4.6. Solubility.....	17
1.5. The Aim of the Thesis……...…….....	17
2. MATERIALS AND METHODS…….....	19
2.1. Materials…….....	19
2.2. Solid Liquid Extraction (Leaching)…….....	19
2.3. Evaporation…….....	20
2.4. Spray Drying…….....	20
2.5. Analysis…….….....	20
2.5.1. Yield.....	20

2.5.2. Moisture Content ……..... 22
2.5.3. Bulk Density..... 22
2.5.4. Hygroscopicity……..... 22
2.5.5. Color ……..... 22
2.5.6. Acidity & pH ……..... 22
2.5.7. Solubility..... 23
2.6. Statistical Analysis 23
3. RESULTS AND DISCUSSION……..... 24
3.1. Solid Liquid Extraction (Leaching)……..... 24
3.2. Spray Drying…….....…..... 26
3.2.1. Effect of Parameters on Yields..... 28
3.2.2. Effect of Parameters on Final Product Moisture Content. 30
3.2.3. Effect of Parameters on Final Product Bulk Density..... 33
3.2.4. Effect of Parameters on Final Product Hygroscopicity..... 36
3.2.5. Effect of Parameters on Final Product Color..... 39
3.2.6. Effect of Parameters on Final Product Acidity & pH..... 44
3.2.7. Effect of Parameters on Final Product Solubility..... 48
CONCLUSION…….....……..... 50
REFERENCES……..... 51
APPENDICES A..... 56
APPENDICES B..... 65

LIST OF TABLES

	Page
Table 2.1. Experimental design for solid liquid extraction (leaching)….....	19
Table 2.2. Evaporation operation conditions ...…...….....	20
Table 2.3. Experimental design for spray drying	21
Table 3.1. Brix measurements at different time intervals for different liquorice/water ratios at 25 ° C	25
Table 3.2. Results of the spray drying yield and final product analysis	26
Table B.1. One-way ANOVA and LSD Test for yield analysis.....	66
Table B.2. One-way ANOVA and LSD Test for moisture content analysis.....	68
Table B.3. One-way ANOVA and LSD Test for bulk density analysis.....	69
Table B.4. One-way ANOVA and LSD Test for hygroscopicity analysis.....	70
Table B.5. One-way ANOVA and LSD Test for acidity analysis.....	72
Table B.6. One-way ANOVA and LSD Test for solubility analysis.....	73

LIST OF FIGURES

	Page
Figure 1.1. The liquorice plant……...…….....	1
Figure 1.2. Structure of Glycyrrhizic acid (GA)…….....	3
Figure 1.3. Schematic diagram of rising film evaporator.....	8
Figure 1.4. Schematic diagram of drying process.....	9
Figure 1.5. Schematic representation of spray dryer.....	11
Figure 1.6. The diagram of Hunter L, a, b color space	16
Figure 3.1. The graph of brix (°Bx) measurements at 4°C, 25 °C, and 37 °C.....	24
Figure 3.2. Leaching curve of liquorice root at 25 °C	25
Figure 3.3. Graphs of yield (%) for different inlet air temperatures (°C).....	28
Figure 3.4. Graphs of yield (%) for different maltodextrin concentrations (%)..	29
Figure 3.5. Graphs of yield (%) for different maltodextrin dextrose equivalents (DE).....	30
Figure 3.6. Graphs of moisture content (% wet basis) for different inlet air temperatures (°C).....	31
Figure 3.7. Graphs of moisture content (% wet basis) for different maltodextrin concentrations (%).....	32
Figure 3.8. Graphs of moisture content (% wet basis) for different maltodextrin dextrose equivalents (DE).....	33
Figure 3.9. Graphs of bulk density (g/mL) for different inlet air temperatures (°C).....	34
Figure 3.10. Graphs of bulk density (g/mL) for different maltodextrin concentrations (%).....	35
Figure 3.11. Graphs of bulk density (g/mL) for different maltodextrin dextrose equivalents (DE).....	36
Figure 3.12. Graphs of hygroscopicity (g/100g) for different inlet air temperatures (°C).....	37

Figure 3.13. Graphs of hygroscopicity (g/100g) for different maltodextrin concentrations (%)..... 38
Figure 3.14. Graphs of hygroscopicity (g/100g) for different maltodextrin dextrose equivalents (DE)..... 39
Figure 3.15. Graphs of color change for different inlet air temperatures (°C) (a) L* (b) a* (c) b*..... 41
Figure 3.16. Graphs of color change for different maltodextrin concentrations (%) (a) L* (b) a* (c) b*..... 42
Figure 3.17. Graphs of color change for different maltodextrin dextrose equivalents (DE) (a) L* (b) a* (c) b*..... 43
Figure 3.18. Graphs of acidity (%) for different inlet air temperatures (°C)..... 44
Figure 3.19. Graphs of acidity (%) for different maltodextrin concentrations (%)..... 45
Figure 3.20. Graphs of acidity (%) for different maltodextrin dextrose equivalents (DE)..... 45
Figure 3.21. Graphs of pH for different inlet air temperatures (°C)..... 46
Figure 3.22. Graphs of pH for different maltodextrin concentrations (%)..... 47
Figure 3.23. Graphs of pH for different maltodextrin dextrose equivalents (DE)..... 47
Figure 3.24. Graphs of solubility for different inlet air temperatures (°C)..... 48
Figure 3.25. Graphs of solubility for different maltodextrin concentrations (%) 49
Figure 3.26. Graphs of solubility for different maltodextrin dextrose equivalents (DE) 49
Figure A.1. Graphs of yield (%) at different inlet air temperatures (°C) , maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE 57
Figure A.2. Graphs of moisture content (%) at different inlet air temperatures (°C) , maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE 58

Figure A.3. Graphs of bulk density (g/mL)at different inlet air temperatures (°C) , maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c)..... 59
Figure A.4. Graphs of hygroscopicity (g/100g) at different inlet air temperatures (°C), maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE..... 60
Figure A.5. Graphs of color change at different inlet air temperatures (°C), maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE..... 61
Figure A.6. Graphs of acidity (%) at different inlet air temperatures (°C), maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE..... 62
Figure A.7. Graphs of pH at different inlet air temperatures (°C), maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE..... 63
Figure A.8. Graphs of solubility at different inlet air temperatures (°C), maltodextrin concentrations (%), DE maltodextrins (a) DE 12 (b) DE 18 (c) 19 DE..... 64

CHAPTER I
INTRODUCTION

1.1 Liquorice

The liquorice plant (Figure 1.1) has a long and storied history of use in both Eastern and Western cultures pre-dating the Babylonian and Egyptian empires. The genus name *Glycyrrhiza* is derived from the ancient Greek word for 'sweet root' (Gr. g*lykos* (sweet) + *rhiza* (root)), which was later Latinized to *liquiritia* and eventually to liquorice (Isbrucker and Burdock, 2006).

Figure. 1.1. The liquorice plant

Native to Asia and the Mediterranean region, liquorice (*Glycyrrhiza glabra*) is a tall shrub of the Leguminosae family . There are about 14 species known, although most commercial liquorice is extracted from varieties of *G. Glabra* grown in southern and central Europe (var. *typica*), in central and southern Russia (var. *glandulifera*), and in Iran and Iraq (var. *violacea*). Liquorice also grows in the United States (var. *lepidota*) and England (var. *typica*) but neither represents a significant contribution to world production. Commercially important sources are Spain, Iraq, Iran, Turkey, Russia and China, and although there are no known prohibitions against use of any species, variety or country of origin, some types are not sweet enough to have commercial value. Chinese liquorice (*G. uralensis* and *G. pallidiXora*) are somewhat smaller, related plants, regarded as separate species of *Glycyrrhiza* (Isbrucker and Burdock, 2006).

Liquorice is used in two primary forms: roots (rhizomes) and extracts (Ariño et al., 2007).

Liquorice root contains about 20% of water-soluble extractives, and much of this typically 3–5% of the root—is composed of glycyrrhizin. The sweet taste of the root comes from the substance glycyrrhizin, reputed to be 50 times sweeter than refined sugar. The bright yellow color of liquorice root is provided by Xavonoids, particularly liquiritin, isoliquiritin and their corresponding aglycones, which typically comprise 1–1.5% of the water soluble extract. The harvesting of liquorice root occurs in the autumn of its third or fourth year of growth .The roots are dug up, washed and transported to warehouses for bailing, sorting and drying. The dried roots are crushed by millstones and the pulp is boiled to make the extract (Isbrucker and Burdock, 2006).

Glycyrrhizin is a water-soluble pentacyclic triterpenoid glycoside responsible for the sweetness of liquorice and its aglycone is responsible for various medicinal attributes and clinical applications in the treatment of spleen, sore throat, bronchitis, liver, kidney, and ulcer.

The glycoside usually occurs in a combined calcium or potassium salt form of glycyrrhizic acid (GA) which is a weak acid containing.The acid form of glycyrrhizic

acid is not particularly water-soluble, but its ammonium salt is soluble in water at pH greater than 4.5. The mono-ammonium salt of GA is used as an anti-inflammatory and anti-allergic remedy for the treatment of bronchial asthma, eczemas and other diseases (Mukhopadhyay and Panja, 2008).

Glycyrrhizinic acid (GA) (Figure 1.2) has two stereo-isomers and glycyrrhetinic acid (aglycone, which hydrolysis from glycyrrhizin) also has two stereo-isomers in liquorice root (Pan et al., 2000).

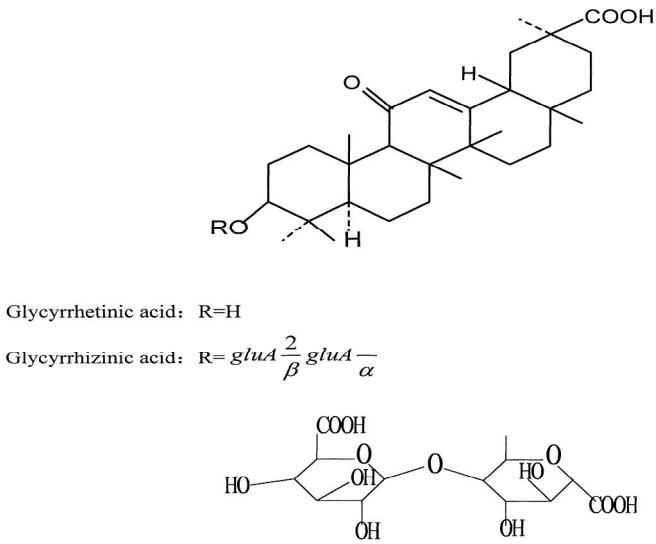


Figure 1.2. Structure of Glycyrrhizic acid (GA).

Liquorice extract is produced by shredding and extracting the root with water in a steam extraction plant. The extracted liquor is filtered and then concentrated to produce a solid liquorice block or spray-dried to obtain liquorice powder. Liquorice extract is also sold in liquid form (a syrup-like material) with water content of 12–15% (Ariño et al., 2006).

Liquorice extract contains reducing and nonreducing sugars, starch, plant gums, resins, essential oils, inorganic salts and low levels of nitrogenous constituents such as proteins, individual amino acids, and nucleic acids (Isbrucker and Burdock, 2006).

Liquorice is extensively used in anti-inflammatory, anti-viral, anti-allergic, anti-oxidant, gastro-protective, and anticancerous properties, in food, confectionery

and pharmaceutical products, such as cough syrups, herbal supplements, chewing gums, drinks, and candy.

Liquorice is largely used as a flavouring and sweetening agent, but has been proposed also for various clinical applications (Fiore et al ., 2005).

Liquorice extract is added to certain types of chewing gum to insure a flexible texture and to certain chocolate candies to stabilize the fat dispersion.Especially in the United States much liquorice is used by the tobacco industry in cigarettes, cigars, smoking mixtures, chewing tobacco, and even snuff. Liquorice added to tobacco imparts a sweet taste and characteristic flavor and also enhances the mildness of a mixture. Liquorice in beer increases the foaminess of the beverage. In other drinks, such as root beer, porter, and stout, liquorice is added for flavor. Liquorice may also be used as a Brown coloring matter. Liquorice has been used medicinally for many centuries mainly to mask the bitter or acrid taste of other drugs or as a soothing remedy for affections of the respiratory tract. Liquorice is also a popular ingredient in herbal tea and in some botanicals, consumed to improve overall health and to alleviate a wide range of diseases (Ariño et al ., 2006).

1.2 Production of Liquorice Concentrate

1.2.1 Solid Liquid Extraction (Leaching)

Extraction is a term used for an operation in which a constituent of a liquid is transferred to another liquid (solvent). Solid-liquid extraction (leaching) is the process of removing a solute or solutes from a solid by using of liquid solvent. The operation is widely used in chemical industries where mechanical and thermal methods of separations are not possible or practical. Extraction of sugar from sugar beets, oil from oil bearing seeds, production of a concentrated solution of a valuable solid material are typical industrial examples of solid liquid extraction (leaching).

Leaching always involves two steps:

1. Contact of the solvent with the solid to be treated so as to transfer the soluble constituent (solute) to the solvent;
2. Separation or washing of the solution from the residual solid.

The most important example is the medicinal plant sector, from which the active principles with pharmacological properties for the treatment of several human pathologies and illnesses are obtained; similar fields are herbalist, cosmetics and the perfume industry; even in these cases the principal ingredients of theirs products are obtained by submitting parts of plants such as flowers, leaves, roots etc. to solid-liquid extraction. Also in other industrial sectors like the beverage industry, a solid-liquid extraction is used to get alcoholic extracts from the peel of citrus fruit, flowers, leaves etc. that are then mixed to water and sugar to obtain the finished product. The list could continue mentioning many other industrial applications (Naviglio et al., 2007).

1.2.2 Evaporation

Evaporation is an operation used to concentrate a solution of a nonvolatile solute and a volatile solvent, which in many cases is water. A portion of the solvent is vaporized to produce a concentrated solution, slurry or thick, viscous liquid.

Evaporation is used to remove a liquid from a solution, suspension, or emulsion by boiling off some of the liquid. It is thus a thermal separation, or thermal concentration, process. Evaporation process can be defined as one that starts with a liquid product and ends up with a more concentrated, but still liquid and still pump-able concentrate as the main product from the process (Lee and Lee, 1999; Riberio et al., 2007).

Evaporation is one of the most important unit operations in food processing. Large quantities of fruit and vegetable juices, sugar, and syrups are concentrated in several types of commercial evaporators (Saravacos et al., 1970). Typical evaporator applications are product concentration, dryer feed pre-concentration, volume reduction, water / solvent recovery, crystallization.

In the beverage industry, a frequently used process is the evaporation under vacuum. Freshly pressed fruit juice from stone, pomaceous and soft fruit as well as juice from citrus and tropical fruit is concentrated and is in this way preserved. In the fruit juice industry, evaporation is used for the concentration of extract based on different starting materials. For example, juice residues and oil can be extracted from

the pulp of the fruit or from the peels of citrus fruit. The recovered extract can be concentrated by evaporation and can be reused, and different types of oil can be separated. Pectin can be extracted from the pomace of apples and pears and from the peels of beet and citrus fruit. The extract is concentrated in an evaporation plant, and the pectin is then precipitated from the concentrate by means of, for example, alcohol. The alcohol can be efficiently recovered in a combined evaporation and distillation process.

Evaporation plants are also used in other fields of the beverage industry, e.g. in the brewing industry for the concentration of malt extract, brewer‘s yeast, yeast extract, hop extract, grain press water and wort. The dealcoholization of beer in a falling film evaporation plant is a particularly gentle process, which guarantees the characteristic taste of beer with a residual alcohol content of less than 0.05 %. Extract from coffee, tea and other plants can also be optimally concentrated in evaporation plants.

In the concentration of many fruit juices and other heat sensitive materials, single pass evaporators are preferred, because the product quality is not damaged appreciably by the short time exposure to heat. Single pass evaporators include the tubular rising film, falling film, combination of these, and the plate and centrifugal types (Saravacos et al.,1970).

The basic components of this process consist of a heat exchanger, vacuum, vapour seperator, condenser.The heat exchanger is enclosed in a large chamber and transfers heat from the heating medium, usually low pressure steam, to the product usually via indirect contact surfaces. The vacuum keeps the product temperature low and the difference in temperatures high. The vapour separator removes entrained solids from the vapours, channelling solids back to the heat exchanger and the vapours out to the condenser. The condenser condenses the vapours from inside the heat exchanger and may act as the vacuum source (http://www.foodsci.uoguelph.ca).

Rising Film Evaporator: The concept of film evaporation was first practically applied in rising film evaporation which was pioneered by Paul Kestner in the early 1900's. The rising film concept overcame the problem of liquor distribution among

tubes by careful design of the bottom of the tube and an appropriate pre- heating of the liquor. However the rising film is only established by heating the incoming liquid to above the equivalent boiling temperature of the liquid at the top of the tube, this superheating being imposed by hydrostatic and velocity pressures present during the passage of the liquid up the tube (Addison, 1981).

Evaporators are used in a wide range of processes, including pharmaceuticals, foods and beverages, pulp and paper, chemicals, polymers and resins, inorganic salts, acids, bases, and a variety of other materials. There are many types and variations of evaporators, and the best for a particular application depends on the product characteristics and desired results. Either the vapor or the concentrate stream, or both, may be the desired product Therefore, the evaporator should be designed to provide a clean separation of the vapors from the condensate and the feed.

Schematic diagram of the used rising film evaporator in this study is shown in Figure 1.3. The liquid feed is processed by gravity from a stainless stell feed tank through a flow meter and manually operated control valve. Vapor, produced by boiling in the tube, rises and carries a 'film' of more concentrated liquid up the tube and into a cyclone separator where the vapor and liquid are separated. The vapor enters water cooled condenser where it condenses and flows out into a condensate collecting tank. The liquid concentrate can be recycled back through evaporator tube or collected as product in the product collecting tank. The evaporating ducting, cyclone and condenser are all constructed from stainless steel, and a glass 'elbow' at the top of the evaporator allows the vapor/liquid film mechanism to be seen before and condensate collecting tanks a re borosilicate glass to allow the condition and quantity of the products to be observed (http://www.armfield.co.uk).

1.3 Drying

Drying is the process of removing liquid from solids by evaporation. The drying process has been used for thousands of years to reduce transport weight and increase the storage life of numerous products and materials. For centuries, drying meant spreading a product out in the open air and letting the sun provide the energy for water evaporation. With the dawn of the industrial age, many different drying

processes have been developed to increase drying speed and improve product quality and uniformity.

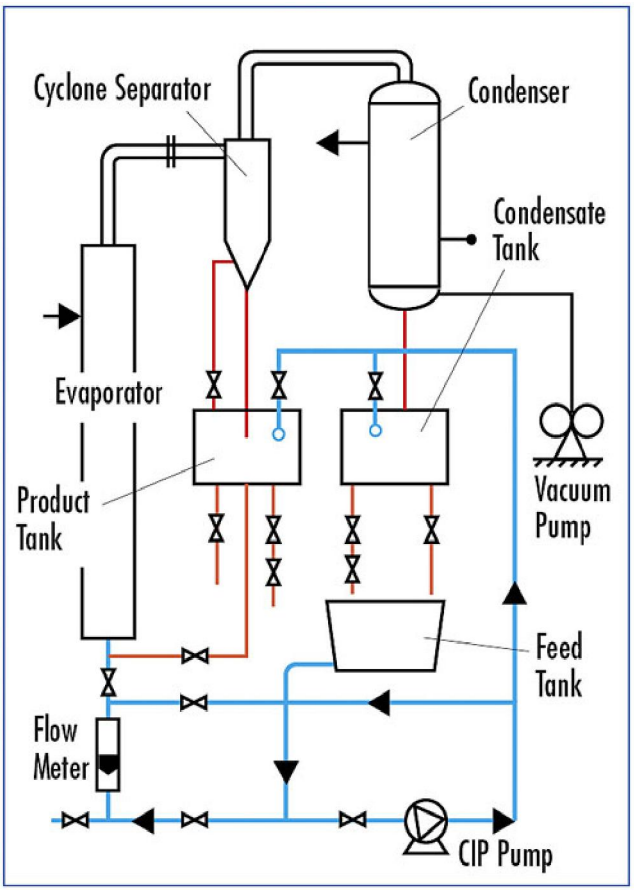


Figure 1.3. Schematic diagram of the rising film evaporator

It is the simplest and most natural of all food processing technologies, and preserves food by removing most of its free water. Schematic diagram drying process is shown in Figure 1.4. The lower water content slows the rate of respiration, enzymatic action and overall deterioration rate, makes products less susceptible to decay and much easier and less expensive to transport. Because drying removes moisture, the food shrinks and decreases in size and weight, thus requiring less space for storage (Lecorvaisier et al., 2010).

Drying removes the moisture from the food so bacteria, yeast and mold cannot grow and spoil the food. Drying also slows down the action of enzymes (naturally occurring substances which cause foods to ripen), but does not inactivate them. Because drying removes moisture, the food becomes smaller and lighter in weight.

When the food is ready for use, the water is added back, and the food returns to its original shape. Foods can be dried in the sun, in an oven or in a food dehydrator by using the right combination of warm temperatures, low humidity and air current.

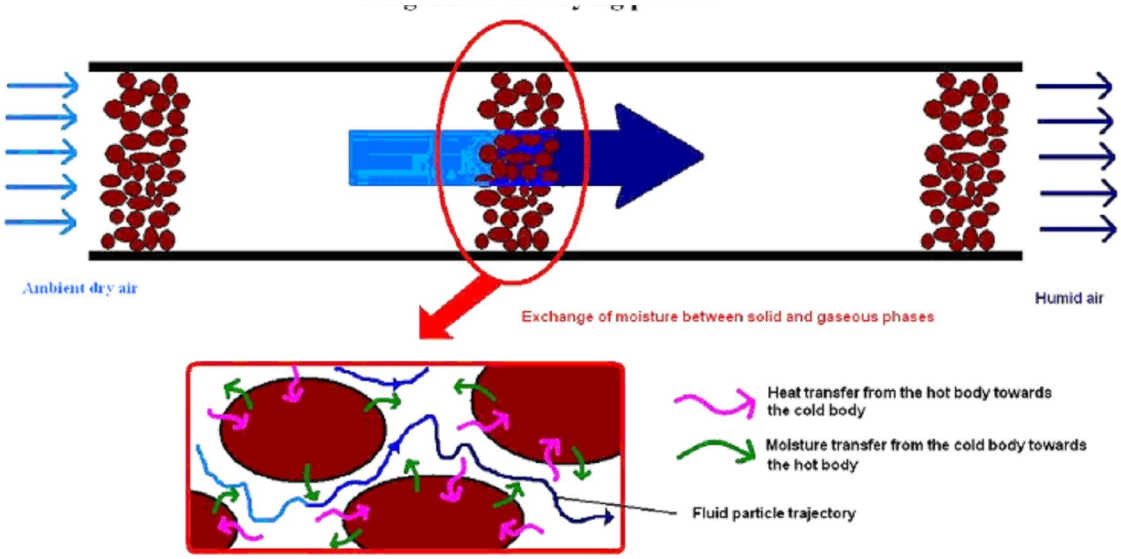


Figure 1.4 Schematic diagram of drying process

Dried foods are tasty, nutritious, lightweight, easy-to- prepare, and easy-to-store and use. The energy input is less than what is needed to freeze or can, and the storage space is minimal compared with that needed for canning jars and freezer containers. The nutritional value of food is only minimally affected by drying. Dried fruits and vegetables are high in fiber and carbohydrates and low in fat, making them healthy food choices. Dried fruit has a higher concentration of carbohydrate than fresh fruit and therefore serving sizes tend to be smaller (Smith, 2009).

Spray Drying: Food powders have many benefits and economic potentials over their liquid counterparts such as reduced volume or weight, reduced packaging, easier handling and transportation, and much longer shelf life. Besides, their physical state provides a stable, natural, and easily dosable ingredient, which generally finds usage in many foods and pharmaceutical products such as flavoring and coloring agents (Goula and Adamapoulos, 2010).

Spray drying is widely used for commercial production of dried fruits and vegetables. It is a well-established method for transforming a wide range of liquid

food products into powder form. Spray-dried powders are suitable for transport and storage. Furthermore, it is a highly appropriate process for heat sensitive components such as carotenoids. Spray drying has many applications, particularly in the food, pharmaceutical, and agrochemical industries.

The process results in powders with good quality, low water activity and easier transport and storage. The physicochemical properties of powders produced by spray drying depend on some process variables, such as the characteristics of the liquid feed (viscosity, particles size, flow rate) and of the drying air (temperature), as well as the type of atomizer. Therefore, it is important to optimize the drying process, in order to obtain products with better sensory and nutritional characteristics and better process yield (Tonon et al., 2008).

Schematic representation of the spray dryer is shown in Figure 1.5. Spray drier contains three main parts; heater, drying chamber, product collection area.In spray drier, the heat and mass transfers occur between the air and vapor films surrounding the feed particals. This vapor film surrounding the droplet keep the partical at saturation temperature.

In spray drier, inlet air passes from filter (before heating section) to be refined from dust and other things. The nozzle (atomizer type) has a very small hole; therefore supplies a very high pressure.

One prevalent problem in spray drying is the stickiness that occurs while spray drying sugar-rich foods such as fruit juices, honey, and fruit and vegetable extracts. The stickiness results in the deposition of the powder particles onto the internal dryer wall, ultimately leading to poor yields. To minimize the stickiness problem, both process-based and material science-based approaches are in place. Process-based approaches include the mechanical scraping of the chamber wall, the introduction of cold air at the bottom, and the use of low temperature/low humidity air. An example of the material science-based approach involves the addition of drying agents to reduce the stickiness of the powders.Process-based modifications are not easy and, in many instances, economically nonviable.

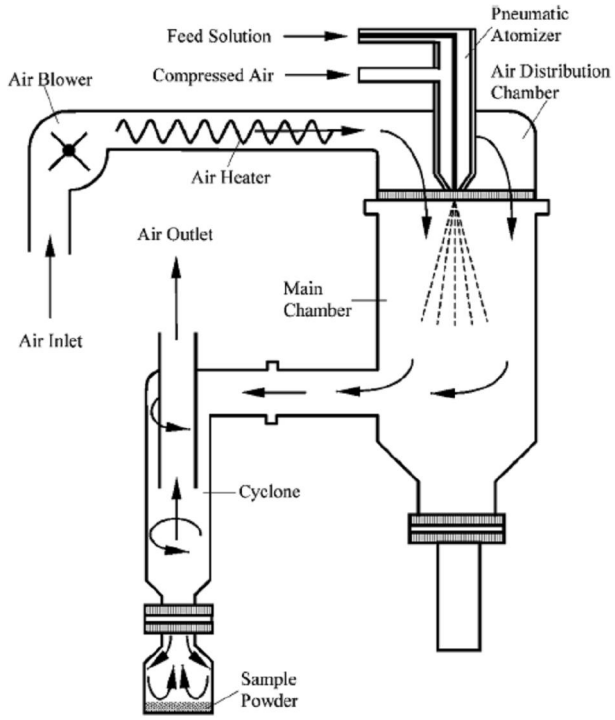


Figure 1.5. Schematic representation of the spray dryer

Fruit juice powders obtained by spray drying may have problems in their properties, such as stickiness, hygroscopicity and solubility, due to the presence of low molecular weight sugars and acids, which have low glass transition temperature. Thus they can stick on the dryer chamber wall during drying, leading to low product yield and operational problems (Tonon et al., 2008).

Various methods capable of producing a free-flowing fruit juice powder have been proposed: addition of drying aids (maltodextrins, glucose, soybean protein, sodium chloride, and skim milk powder), scrapping of dryer surfaces, cooling of the drying chamber walls , and admission of atmospheric air near the chamber bottom, allowing transport of the powder to a collector having a low humidity atmosphere (Goula and Adamapoulos, 2010). Besides reducing powder hygroscopicity, such agents, normally used for microencapsulation, can protect sensitive food components against unfavorable ambient conditions, mask or preserve flavors and aromas, reduce the volatility and reactivity and provide additional attractiveness for the merchandising of food products (Tonon et al., 2008).

Maltodextrin is one of the common drying aids for spray drying owing to its beneficial role as a carrier or an encapsulating agent in increasing the stability of carotenoids, reasonably cheap and commercially available.

Maltodextrins (added prior to drying or mixed in the bulk powder) function in four different ways: (i) compete with host powders for moisture, (ii) act as physical barrier between particles, (iii) act as moisture-protective barrier, and (iv) increase glass transition temperature thus sticky temperature (Sablani et al., 2008).

1.4. Quality Parameters of Liquorice Powders

1.4.1 Moisture Content

Determination of moisture content in foodstuffs is very important as the moisture influences the shelf life of food products. Depending upon the water content, storage capacity of a food item varies. In addition, it influences the cost of transportation. Above all, moisture content determines how nutritive and tasty the food is. Hence, determination of moisture content becomes inevitable in food. Accuracy of moisture determination varies with the method employed. So, moisture analysis is still in a progress stage with various problems to be solved (Nagarajan, 2006).

The determination of moisture in a food is done for many purposes such as assessment of quality, quality control, quality assurance, detection and estimation of adulteration, conformity with food standards and other statutory requirement, calculation of total food solids content, assessment of stability, shelf life and storage life.

Water in a food item can be present in 2 ways:

- Free water (which is physically linked to the food matrix and easily lost by evaporation or drying as a separate constituent)
- Bound water (include water molecules chemically bonded to ionic and polar groups or water of crystallization or hydrates which is difficult to remove

In the food industry, the determination of the moisture content in dehydrated foods, and especially in food powders is usually performed by the classical oven

method. Depending on the product composition and sensitivity, the drying temperature is typically ranged between 70 and 135 °C and the drying time between 2 and 24 h. With oven drying, the sample is heated under specified conditions, and the loss of weight is used to calculate the moisture content of the sample.

Moisture can be problematic. Incorrect and out-of-date information on moisture can result in poor plant operating decisions costing invaluable time and money. Typical problems experienced by food manufacturers due to poor moisture control include clogging of machinery, incorrect dosing or dispersing of moisture, plant down-time, increased reject material, product non-conformance, down-graded product, product payment penalties, decreased yield and volume production anomalies (www.callidan.com).

1.4.2 Bulk density

Bulk density is a measure describing the weight of an ingredient per unit volume. It is an important factor to consider when determining the storage volume of transport vehicles, vessels, containers, totes, and bags. Bulk density affects transport and storage costs (low bulk density ingredients have higher cost per unit of weight). It also affects the amount of ingredient segregation that may occur during handling of complete feeds (high bulk density particles setle to the bottom of a load during transport, whereas low bulk density particles rise to the top of a load).

Bulk density control is a major objective of many food processes, especially when spray drying and grinding (Goula and Adamapoulos, 2005).

Bulk density is affected by particle size and density, occluded and interstitial air content, which are related tothe feed properties, drying air temperature, drying time and powder handling procedures e.g. crushing and grinding. Generally, the effect of the drying conditions on bulk density is highly product dependent However, in spray drying there are some general trends. Higher atomizer wheel speeds and nozzle pressures decrease droplet and therefore particle size, thus increasing bulk density .Spherical shaped particles can result in a low degree of interstitial air, as the small particles in the size distribution fill the void spaces between the large particles. Irregular shaped particles and agglomerates can lead to a lower bulk density Bulk

density tends to increase with a decrease in air outlet temperature, and with an increase in feed solids (www.bete.com).

Factors affecting bulk density;

- Increasing feed rate increases bulk density if the residual moisture increases
- If increasing feed temperature leads to the production of spherical droplets instead of 'threads', bulk density increases.
- For easily atomized feeds, increased temperature can lower bulk density.
- Bulk densities often increase on powder cooling.
- A coarse homogenous powder has a lower bulk density than a fine homogenous powder.
- A powder with a wide distribution of particle sizes will have a higher bulk density than a powder with a narrower distribution of particle sizes.
- Feed aeration decreases bulk density.
- Feed suspensions give higher bulk densities than feed solutions.
- Increasing residual moisture content increases bulk density.
- Increasing inlet air temperature decreases bulk density.
- Reducing the outlet air temperature increases residual moisture and therefore increases bulk density.
- Co-current dryers produce powders with lower bulk densities than counter-current dryers.

1.4.3 Hygroscopicity

The term, hygroscopicity, is widely used to describe the water vapor uptake behavior of solids. However, there is no universally accepted definition of hygroscopicity in the literature. The term 'hygroscopicity' describes the ability of a solid to take up and retain water. Hygroscopicity could be defined as the rate and extent of water vapor uptake by a solid at certain RH values and temperatures.

A system can be classified according to its water binding characteristics, such as non-hygroscopic or hygroscopic. A material that is non-hygroscopic, such as glass wool or ceramic, does not contain bound water where the partial pressure of the water in the material is equal to the vapor pressure of free water.A material that is

hygroscopic contains bound water, and the vapor pressure of the material is less than that of free water. Foods, in general, can be considered hygroscopic (Srikiatden and Roberts, 2007).

Hygroscopicity is an important tools for predicting interactions between the water and the food components. Knowledge of hygroscopicity is important in various food processes, such as drying, storage and packaging, since it is used to estimate drying time, ingredient behavior on mixing, packaging selection and modeling moisture changes that occur during storage (Kuruzowa et al., 2009).

1.4.4 Color

Color is essentially a beam of light composed of irregularly distributed energy emitted at different wavelengths. Depending on the type of illumination, the same material can show different light qualities and produce different sensations. Foods, along with other materials, have color properties, which depend exclusively on their composition and structure (Harold, 2001).

The color perceived when the eye views a food is related to the following three factors: the spectral composition of the light source, the chemical and physical characteristics of the food, and the spectral sensitivity properties of the eye. To evaluate the colorimetric properties of a food, two of these factors must be standardized. Although the human eye can give fairly uniform results, it can be replaced by some instrumental sensor or photocell to provide even more consistent determinations. Visual colorimeters facilitate visual comparisons and eliminate differences in interpretation between operators (Harold, 2001).

Description of color for purchase specifications of food commodities or packaging materials involves color tolerances, which are defined in one, two, or three dimensions in color space to avoid variability of the human eye. Several systems of color analysis have been created. The most used are the CIE, Munsell, Hunter, and Lovibond systems.

The Hunter *L, a, b* color scale evolved during the 1950s and 1960s. At that time, many of the scientists involved with color measurement were working on uniform

color scales. The uniform color scales being investigated gave better indications of the color of a sample based solely on the numbers. There were several permutations of the Hunter *L, a, b* color scale before the current formulas were released in 1966 (www.hunterlab.com).

The diagram of the Hunter *L, a, b* color space is shown in Figure 1.6. The *L* axis runs from top to bottom. The maximum for *L* is 100, which would be a perfect reflecting diffuser. The minimum for *L* would be zero, which would be black. The Hubter *a* and *b* axes have no specific numerical limits. Positive *a* is red. Negative a is green. Positive *b* is yellow. Negative *b* is blue (www.hunterlab.com).

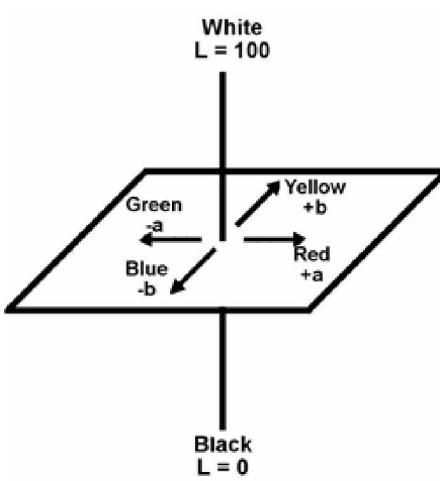


Figure 1.6. Diagram of the Hunter L, a, b color space

1.4.5 Acidity

Titratable acidity is used as a guide to determine how acidic the product will taste. This determination measures the concentration of all available hydrogen ions present in the sample.

Acidity affects taste, thus this parameter is tested to determine the quality of the product. Acidity is determined by an end-point titration using sodium hydroxide (a base) and is defined as the consumption of base necessary to shift the Ph value (www.hannaist.com).

The acid strength of a solid surface is defined as the ability of the surface to convert an adsorbed neutral base into its conjugate acid.

Neutralization reaction refers to acid and base reaction producing salt and water. The equivalent amounts of acid react with base to form equivalent amounts of salt and water. When the reaction is complete, acid and base are said to neutralize each other. Since water is produced, neutralization reaction is also referred as "water forming reaction".

$$\underset{\text{Glycyrrhizic acid}}{C_{42}H_{62}O_{16}} + 2NaOH \rightarrow \underset{\text{Disodium glycyrrhizin}}{C_{42}H_{60}Na_2O_{16}} + 2H_2O \qquad (1.1)$$

Phenolphthalein is an acid-base indicator which changes from colorless to a pink (magenta) at a pH of about 8.3. Generally, acidity is measured by titration to pH 8.3 with NaOH titrant. Metacresol purple also changes color at pH 8.3, but gives a sharper color change than phenolphthalein.

1.4.6 Solubility

The solubility of a solute is the maximum quantity of solute that can dissolve in a certain quantity of solvent or quantity of solution at a specified temperature.

In process of dissolving, molecules of the solute are inserted into a solvent and surrounded by its molecules. In order for the process to tak place, molecular bonds between molecules of solute (ie. sugar) have to be broken and molecular bonds of the solvent also have to be disrupted. Both of these require energy.

1.5 The aim of this thesis

Powders have many benefits and economic potentials over their liquid counterparts such as reduced volume or weight, reduced packaging, easier handling and transportation, and much longer shelf life. Besides, their physical state provides a stable, natural, and easily dosable ingredient, which generally finds usage in many foods and pharmaceutical products such as flavoring and coloring agents (Goula and Adamapoulos, 2010).

Relatively high temperatures are encountered in spray drying, even taking into account the evaporative cooling which occurs. On the other hand, the drying times are much shorter than those required by other methods (freeze drying, vacuum drying) and so heat damage can be controlled provided the residence time of the dried particles in the hot zone of the drier is controlled (Brennan et al., 1971).

The aim of this thesis was to investigate the effect of different air inlet temperatures and addition of different DE maltodextrin, and addition of maltodextrin with different concentrations on the spray drying of liquorice extract. Also the powder properties (moisture content, bulk density, hygroscopicity, color, and acidity& pH, solubility) and spray drying performance (yield) was studied as a parameters for investigation.

CHAPTER II
MATERIALS AND METHODS

2.1 Materials

The liquorice roots used were provided from Gold Üçyıldız Biber Baharat Gıda Sanayi Tic. Paz. (Kahramanmaraş). 12 DE, 18 DE and 19 DE maltrodexins were used and provided from Veser Chemical Co. and Cargil Co. PCl_3, NaOH, phenolphthalein used were supplied from Fluka, Merck, and Merck, respectively.

2.2 Solid Liquid Extraction (Leaching)

Preliminary expriments were carried out to establish the optimum extraction procedure. Three temperatures and liquorice/water ratios (Table 2.1) were selected for leaching. 100 grams of liquorice were used for trials. Extraction at 25 ˚C by using the 1/3 as the ratio of liquorice/water was recorded as the optimum extraction procedure. So 10 kilograms of liquorice was placed in a clean bin. Each bin was filled with 30 kilograms of water and was kept at 25 ˚C for 24 h. The extract was then filtered trough a gauze and transfered to a bottle. Brix measurements were carried out by a digital refractometer and noted. The extracts obtained were stored at refrigerator at 4 ˚C for one day.

Table 2.1. Exprimental design for solid liquid extraction (leaching)

Run No	Temperature (°C)	Liquorice/water ratio
1	4	1/5
2	4	1/4
3	4	1/3
4	25	1/5
5	25	1/4
6	25	1/3
7	37	1/5
8	37	1/4
9	37	1/3

2.3 Evaporation

Filtered extracts was then concentrated in a rising film evaporator (Armfield FT 22,UK).

Table 2.2. Evaporation operation conditions

Feed rate	267 ml/h
Vaccum pressure	0.6 bar
Concentrate temperature	53.6 ˚C

Brix measurements were carried out by a digital refractometer (PTR 46X, Index Instruments) and noted. The concentrate obtained were stored at refrigerator at 4 ˚C.

2.4 Spray Drying

Maltodextrin was added accordingly to the liquorice extract. The mixture is then stirred till all maltodextrin was dissolved and ready to spray dry. A laboratory scale (LabPlant SD-04, Huddersfield, England) spray dryer was employed for the spray drying process. The unit was self contained and supplied complete and ready for immediate operation. All major components were housed within a stainless steel cabinet. Three inlet air temperatures (110 ˚C, 120 ˚C, and 130 ˚C) were studied. Maltodextrin (%20, % 15, and % 10) was added according to the liquorice extract (20 °Bx). 12 DE, 18 DE and 19 DE maltodextrins were used. The dryer was washed with water at desired parameter settings for 10-15 minutes before spray drying process. Full factorial design was studied. The experimental design is shown in Table 2.3. The powders produced were collected in a container, sealed, weighed and stored in dark.

2.5 Analysis

2.5.1 Yield

Yield was evaluated through determination of recovered product after spray drying, given by the ratio between the total recovered product mass and the mass of extract initially fed into the system. Process yield was calculated as the relationship between total solids content in the resulting powder and total solids content in the feed mixture (Tonon et al.,2008).

Table 2.3. Experimental design for spray drying

Run No	DE*	Inlet Air Temperature (°C)	Maltodextrin Concentration (%)	Outlet Air Temperature (°C)	Feed rate ml/min
1	18	110	20	65-72	5
2	18	110	15	65-72	5
3	18	110	10	65-72	5
4	18	120	20	70-78	5
5	18	120	15	70-78	5
6	18	120	10	70-78	5
7	18	130	20	82-86	5
8	18	130	15	82-86	5
9	18	130	10	82-86	5
10	19	110	20	65-72	5
11	19	110	15	65-72	5
12	19	110	10	65-72	5
13	19	120	20	70-78	5
14	19	120	15	70-78	5
15	19	120	10	70-78	5
16	19	130	20	82-86	5
17	19	130	15	82-86	5
18	19	130	10	82-86	5
19	12	110	20	65-72	5
20	12	110	15	65-72	5
21	12	110	10	65-72	5
22	12	120	20	70-78	5
23	12	120	15	70-78	5
24	12	120	10	70-78	5
25	12	130	20	82-86	5
26	12	130	15	82-86	5
27	12	130	10	82-86	5

** Maltodextrin dextrose equivalent*

2.5.2 Moisture Content

Each samples of liquorice powder (1 g each) were weighed and then dried in a drying oven at 80 ˚C for 3 hours. The samples were taken out from the oven , cooled in a dessicator for 1 hour and weighed. The drying and weighing processes were repeated until constant weights were obtained. The moisture content was expressed in terms of the percent wet basis (% w.b.) as 100×kg water/kg wet material (Rattes and Oliveira, 2007). Dublicate samples were analysed and the mean reading was recorded.

2.5.3 Bulk Density

The bulk density of the powder was measured by weighing out 1 g of the sample and placing it into a 10mL graduated cylinder. This was tapped 10 times onto a cloth from a height of 10 cm. The volume was then recorded and used to calculate bulk density as g/mL (León-Martínez, 2010). The bulk density was calculated by dividing the mass of the powder by the volume occupied in the cylinder. Dublicate samples were analysed and the mean reading was recorded.

2.5.4 Hygroscopicity

Samples (1 g) of each powder were placed in small glass covers, weighed and equilibrated over a saturated salt solution (PCl_3 solution; relative humidity of 90 %) in desiccators at 25 °C. After seven days, the samples were weighed and the hygroscopicity is expressed as g moisture/100 g solids (Kuruzowa et al., 2009). Dublicate samples were analysed and the mean reading was recorded.

2.5.5 Color

The color of liquorice powder samples (L *, a*, and b* values) was measured with a HunterLab Colorimeter (Colorflex, USA) and results were expressed in accordance with the CIE Lab system (Ersus and Yurdagel, 2007). Triplicate samples were analysed and the mean reading was recorded.

2.5.6 Acidity & pH

The pH of reconstituted powder was measured by a pH meter. 10 milliliters of distelled water was added to 2 grams of powder. The titratable acidity was determined as glycyrrhizic acid % (w/w) by titration with 0.1N NaOH to a

phenolphthalein end point at room temperature (Yasar, 2008). Dublicate samples were analysed and the mean reading was recorded.

2.5.7 Solubility

50 mL water was added to 2 grams of powder. Then the mixture was stirred on the magnetic mixer. Solubility was determined as the time (second) that all of the powder was comletely dissolved.

2.6 Statistical Analysis

Analysis of Variance (ANOVA) and LSD Test were carried out using SPSS 15.0. ANOVA was done to see the significance of the parameters on analysis and LSD Test was provided to make multiple comparision.

CHAPTER III
RESULTS AND DISCUSSION

3.1. Solid Liquid Extraction (Leaching)

Brix is used to describe the soluble solid content in a liquid. It is a measurement of the concentration of dissolved solid to water and often shown as °Bx. Figure 3.1 shows us the variation of °Bx of the solutions for 1/5, 1/4, and 1/3 liquorice /water ratios at 4˚C, 25˚C, and 37˚C. ˚Bx increased with increasing temperature. The highest °Bx was obtained at 37 ˚C when the liquorice/water ratio was used as 1/3. But it was seen that leaching at 37 ˚C was not as effective as 25 ˚C in terms of the yield. Although ˚Bx measurements at 25 ˚C and at 37 ˚C was close to each other, the yield (%) of the liquorice extract obtained at 25 ˚C was higher than that of at 37 ˚C . As a result, it was more reasonable to do leaching process at 25 ˚C , also in terms of economical reasons and time.

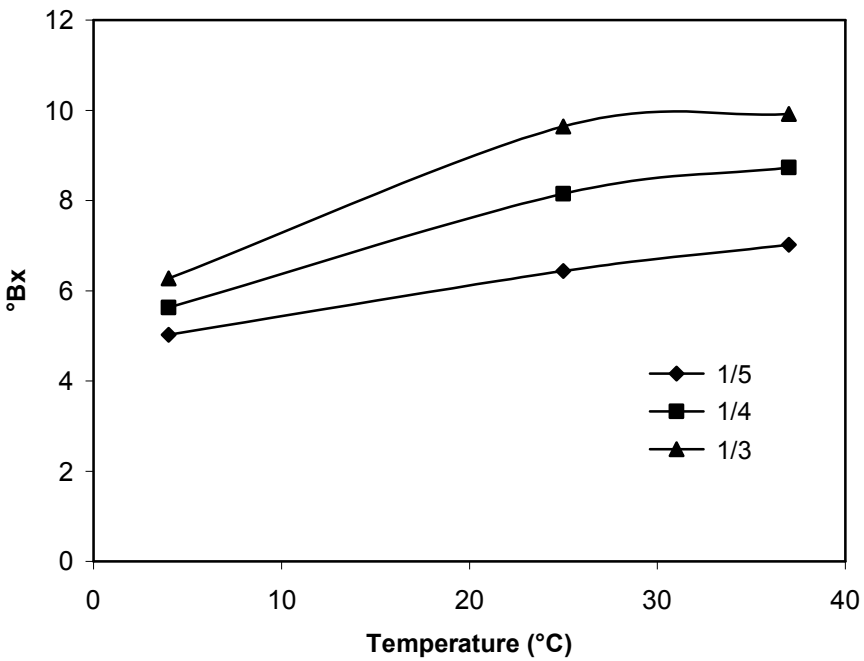


Figure 3.1. Graph of brix (˚Bx) measurements at 4˚C, 25˚C, and 37˚C

After deciding the temperature as 25˚C, the effect of leaching time on ˚Bx was evaluated (Table 3.1). 24 hour (1440 min.) was observed as the optimum leaching time. Figure 3.2 shows us the variation of °Bx of the solutions for 1/5, 1/4, and 1/3 liquorice / water ratios at 25˚C.

Table 3.1. Brix measurements at different time intervals for different liquorice/water ratios at 25˚C

		Liquorice/water ratio		
	Time (min)	1/5	1/4	1/3
Brix measurements	30	2,24	3,12	4,48
	60	2,56	3,46	5,36
	90	3,38	4,33	6,50
	120	4,23	5,24	7,21
	150	5,02	6,06	7,46
	180	5,45	6,52	8,32
	1440	6,44	8,15	9,64

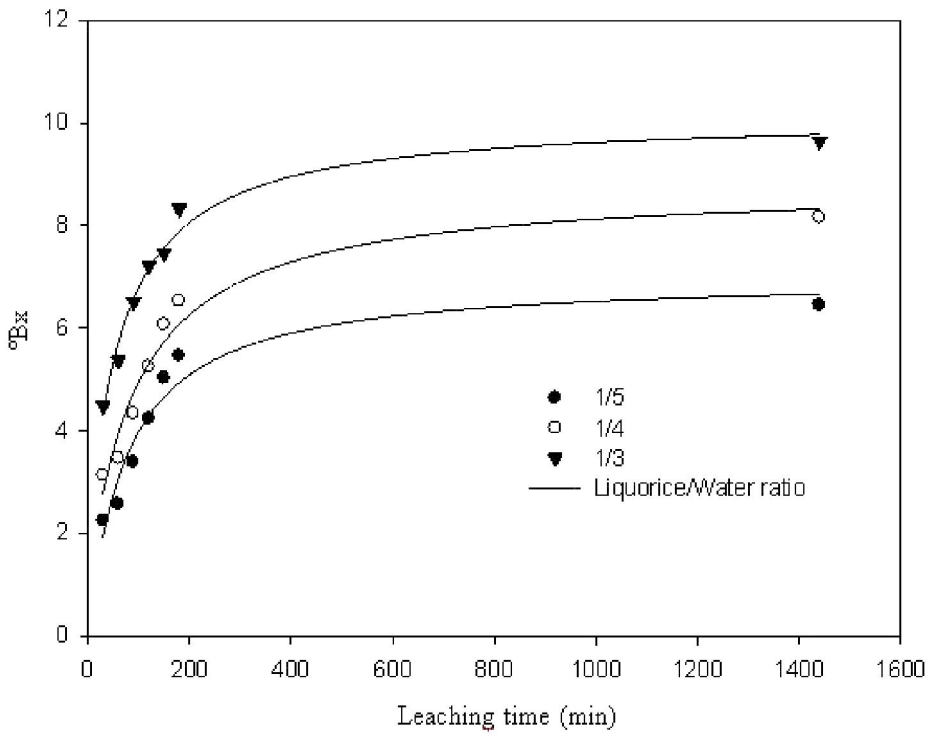


Figure 3.2. Leaching curve of liquorice roots at 25˚C

3.2 Spray Drying

The results of the yield and final product analysis are shown in Table 3.2.

Table 3.2 Results of the spray drying yield and final product analysis.

Run No	Yield (%) (d.b.)	Moisture Content (% w.b.)	Bulk density (g/ mL)	Hygroscopicity (g/100 g) (d.b.)	Solubility (s)
1	42	4,62 ± 0,03	0,231 ± 0,007	0,41 ± 0,02	37 ± 2
2	39	4,84 ± 0,05	0,265 ± 0,001	0,47 ± 0,01	35 ± 1
3	33	5,02 ± 0,03	0,281 ± 0,009	0,54 ± 0,05	33 ± 2
4	47	4,51 ± 0,07	0,25 ± 0,010	0,38 ± 0,07	34
5	45	4,46 ± 0,05	0,258 ± 0,002	0,44 ± 0,15	32 ± 1
6	38	4,55 ± 0,03	0,255	0,58 ± 0,05	31
7	53	3,74 ± 0,09	0,224 ± 0,001	0,33 ± 0,08	28
8	48	3,85 ± 0,01	0,251 ± 0,001	0,37 ± 0,02	24 ± 3
9	45	4,02 ± 0,15	0,24 ± 0,020	0,40 ± 0,04	23 ± 2
10	36	5,23 ± 0,05	0,242 ± 0,003	0,72 ± 0,16	42 ± 4
11	38	5,54 ± 0,05	0,265 ± 0,007	0,76 ± 0,02	40 ± 1
12	33	5,63 ± 0,03	0,323 ± 0,003	0,82 ± 0,01	39
13	42	4,81	0,255 ± 0,001	0,68 ± 0,02	41 ± 2
14	38	4,96 ± 0,02	0,288 ± 0,001	0,65 ± 0,11	38 ± 1
15	35	5,18 ± 0,01	0,308 ± 0,005	0,70 ± 0,05	37 ± 3
16	44	4,55 ± 0,08	0,240 ± 0,010	0,59 ± 0,03	35
17	40	4,62 ± 0,03	0,253 ± 0,005	0,64 ± 0,18	36 ± 2
18	39	5,00 ± 0,01	0,270 ± 0,005	0,72 ± 0,20	34 ± 1
19	51	3,52 ± 0,02	0,229 ± 0,013	0,34 ± 0,20	30 ± 2
20	44	3,65 ± 0,22	0,230 ± 0,005	0,37 ± 0,50	27
21	48	3,82 ± 0,03	0,242 ± 0,003	0,42	25 ± 1
22	53	3,16 ± 0,06	0,226 ± 0,001	0,22 ± 0,01	23 ± 3
23	47	3,38 ± 0,07	0,229 ± 0,002	0,26 ± 0,01	22 ± 2
24	42	3,50 ± 0,30	0,238 ± 0,003	0,30 ± 0,05	24 ± 1
25	66	2,56 ± 0,02	0,180 ± 0,030	0,2 ± 0,03	21 ± 3
26	62	3,29 ± 0,05	0,211 ± 0,021	0,23 ± 0,14	20 ± 1
27	54	3,45 ± 0,17	0,224 ± 0,003	0,25 ± 0,23	18 ± 2

Table 3.2 (Continuous)

Run No	Color			pH	Acidity (%)
	L*	a*	b*		
1	60,82 ± 0,55	2,85 ± 0,07	29,43 ± 0,04	6,06 ± 0,03	2,42 ± 0,02
2	57,24 ± 0,25	3,59 ± 0,01	29,07 ± 0,17	6,12 ± 0,01	2,14 ± 0,10
3	60,45 ± 0,37	2,96 ± 0,06	28,92 ± 0,12	6,2 ± 0,02	1,77 ± 0,1
4	61,03 ± 0,34	2,93 ± 0,09	29,42 ± 0,12	6,15 ± 0,03	1,60 ± 0,06
5	59,4 ± 0,55	3,31 ± 0,09	29,78 ± 0,06	5,97 ± 0,01	1,89 ± 0,03
6	54,95 ± 0,33	3,41 ± 0,03	28,05 ± 0,44	6,22 ± 0,05	1,69 ± 0,11
7	62,43 ± 0,81	2,51 ± 0,14	28,73 ± 0,10	6,36 ± 0,02	1,48 ± 0,05
8	58,72 ± 0,01	3,14 ± 0,02	28,56 ± 0,03	6,41 ± 0,01	1,32
9	56,59 ± 0,52	3,12 ± 0,15	27,54 ± 0,23	6,52 ± 0,01	1,56 ± 0,01
10	59,36 ± 0,10	2,97 ± 0,08	28,71 ± 0,03	6,17 ± 0,05	2,10 ± 0,03
11	59,48 ± 0,11	2,83 ± 0,03	27,8 ± 0,03	6,02	1,69 ± 0,03
12	55,37 ± 0,11	3,46 ± 0,02	27,86 ± 0,05	6,34 ± 0,04	0,95 ± 0,05
13	59,1 ± 0,30	3,17 ± 0,10	28,64 ± 0,08	6,15 ± 0,02	1,32 ± 0,02
14	57,69 ± 0,48	3,22 ± 0,07	28,24 ± 0,04	6,37 ± 0,01	1,85 ± 0,06
15	56,68 ± 2,60	3,35 ± 0,26	27,9 ± 0,66	6,34 ± 0,03	0,82
16	56,54 ± 0,19	3,23 ± 0,03	28,05 ± 0,05	6,44 ± 0,02	1,19 ± 0,04
17	56,55 ± 0,04	3,65 ± 0,03	29,26 ± 0,14	6,53 ± 0,07	1,07 ± 0,09
18	56,67 ± 0,14	3,23 ± 0,02	27,92 ± 0,05	6,56 ± 0,02	0,7 ± 0,01
19	58,68 ± 1,94	3,03 ± 0,20	28,41 ± 0,46	5,64 ± 0,06	3,21 ± 0,12
20	58,34 ± 0,08	3,8 ± 0,10	29,35 ± 0,10	5,92 ± 0,01	2,79 ± 0,03
21	55,1 ± 0,26	3,65 ± 0,05	28,33 ± 0,07	5,97	2,42 ± 0,01
22	60,93 ± 0,68	2,58 ± 0,08	27,86 ± 0,07	5,93	2,26
23	55,78 ± 0,26	3,5 ± 0,04	28,49 ± 0,12	6,02 ± 0,04	2,67 ± 0,04
24	54,92 ± 0,52	3,59 ± 0,05	28,57 ± 0,06	6,17 ± 0,08	2,38 ± 0,10
25	61,75 ± 0,24	2,31 ± 0,06	27,68 ± 0,11	6,06 ± 0,02	2,63 ± 0,03
26	58,07 ± 0,63	3,18 ± 0,01	28,33 ± 0,06	6,18 ± 0,01	2,51 ± 0,02
27	56,07 ±0,40	3,45 ± 0,07	28,23 ± 0,04	6,24 ± 0,03	2,10 ± 0,05

3.2.1 Effect of Parameters on Yields

It was first attempted to spray dry liquorice extract at 20 % w/w solids with inlet air temperatures ranging from 110 ° C to 130 ° C .

The operating variables independently controlled in the experiments were:

- the DE maltodextrin
- the maltodextrin concentration
- inlet air temperature

The results of the yields are as shown in Table 3.2. Increasing temperatures led to higher process yield (Figure 3.3), which can be attributed to the greater efficiency of heat and mass transfer processes occurring when higher inlet air temperatures are used. This is in agreement with the results published by Tonon et al. (2008), working with spray drying of acai (*Euterpe oleraceae Mart.*) powder.

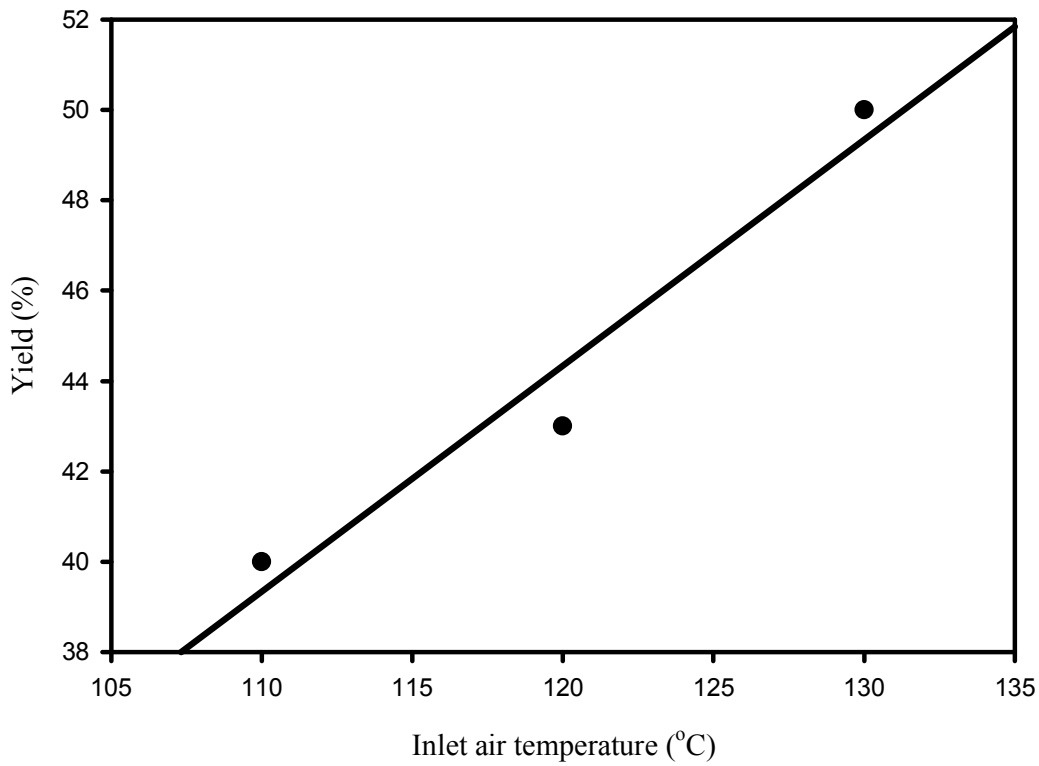


Figure 3.3 Graph of yield (%) for different inlet air temperatures (°C)

Figure 3.4 shows the influence of maltodextrin concentration on yield. Increases in maltodextrin concentration resulted in increases in the yield. These finding can be explained by the fact that maltodextrin helped the powder to be less sticky and less deposited to the chamber wall. A similar result was also reported by Papadakis et al. (2006) who carried out tests on raisin juice powder.

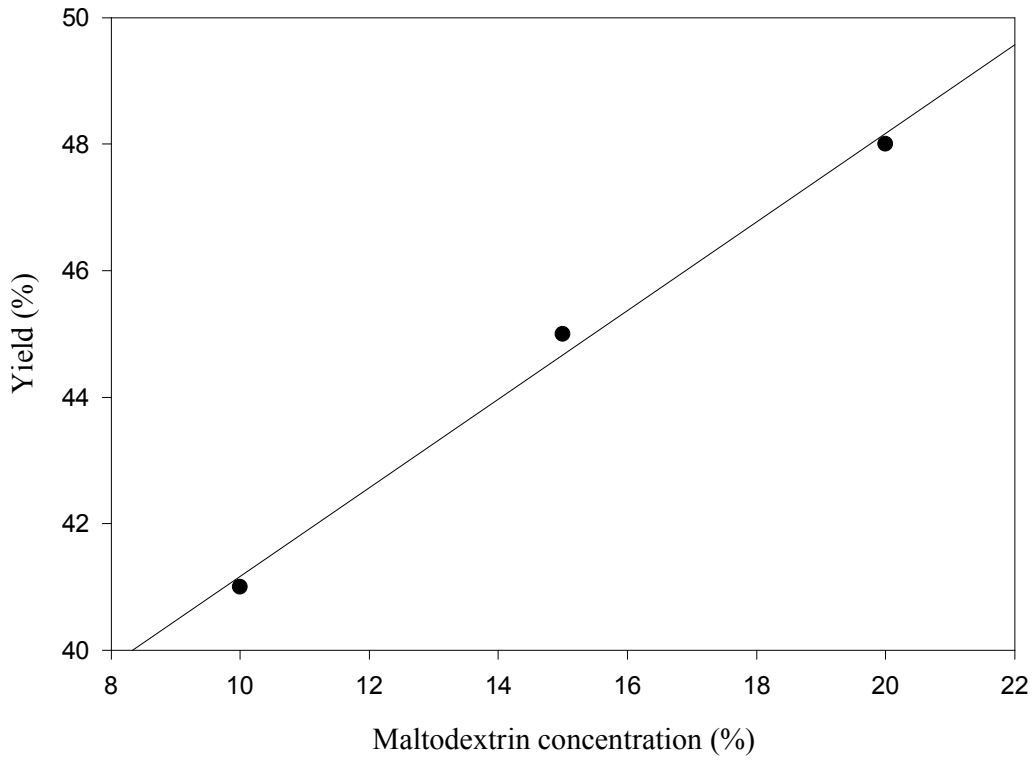


Figure 3.4. Graph of yield (%) for different maltodextrin concentrations (%)

In addition, higher maltodextrin dextrose equivalent caused a decrease in yield. This is related with the fact that the lower molecular weight implies shorter chains and more stickiness. This observation is similar to that reported by other researcher (Yasar, 2008).

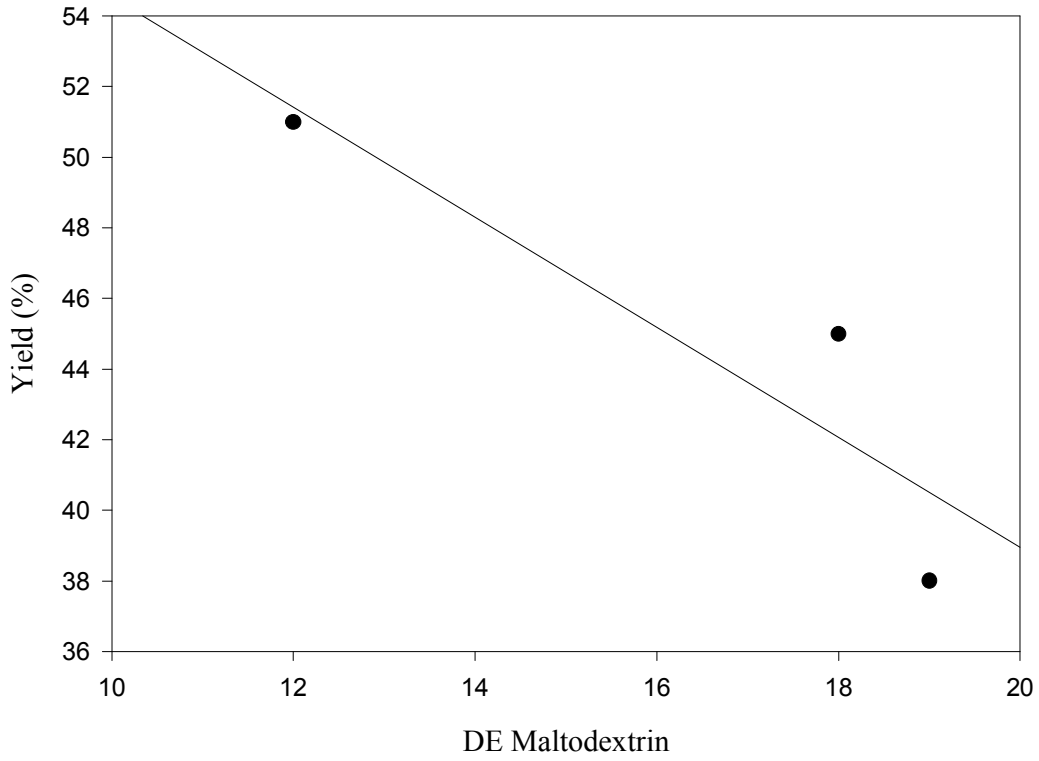


Figure 3.5 Graph of yield (%) for different maltodextrin dextrose equivalents (DE)

3.2.2 Effect of Parameters on Final Product Moisture Content

Moisture content represents the water composition in a food system. Analyzing Table 3.2, the powder moisture contents varied from 2.56 % to 5.63 % (wet basis).

Figure 3.6 shows the influence of inlet temperature on powders moisture content. Increases in the inlet temperature resulted in decreases in the powder moisture content. This is because at higher inlet temperature, the heat transfer between particles is greater which will give a greater driving force for moisture evaporation. Thus, powders with less moisture content were formed. Osman and Endut (2009), Quek et al. (2007), Rattes and Oliveira (2007), Goula and Adamapoulos (2010), Ersus and Yurdagel (2007), and Tonon et al. (2008) also observed a reduction of powders moisture content with increasing air temperatures, studying the spray drying of roselle-pineapple juice, watermelon juice, sodium diclofenac, orange juice concentrate, black carrot and açai, respectively.

Figure 3.7 shows the influence of maltodextrin concentration on powders moisture content. An increase in solids in the feed reduces amount of free water for evaporation. So that, a decrease in the moisture content of liquorice powder was obtained when the maltodextrin concentration increased. Similarly, Abadio et al. (2004) found that an increased concentration of maltodextrin, reduced the moisture content of resultant pineapple juice powders. A similar result was also reported by Osman and Endut (2009) who carried out tests on roselle-pineapple juice powders.These findings could be explained by the fact that additional concentrations of maltodextrin resulted in an increase in feed solids and a reduction in total moisture for evaporation (Kha et al., 2010).

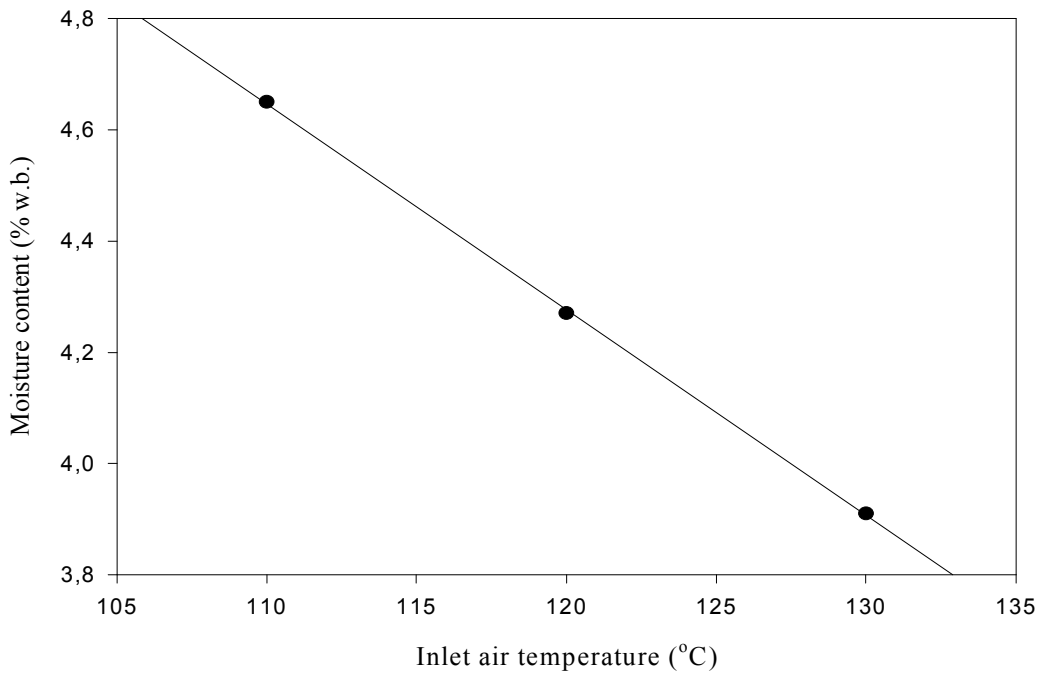


Figure 3.6 Graph of moisture content (% wet basis) for different inlet air temperatures (°C)

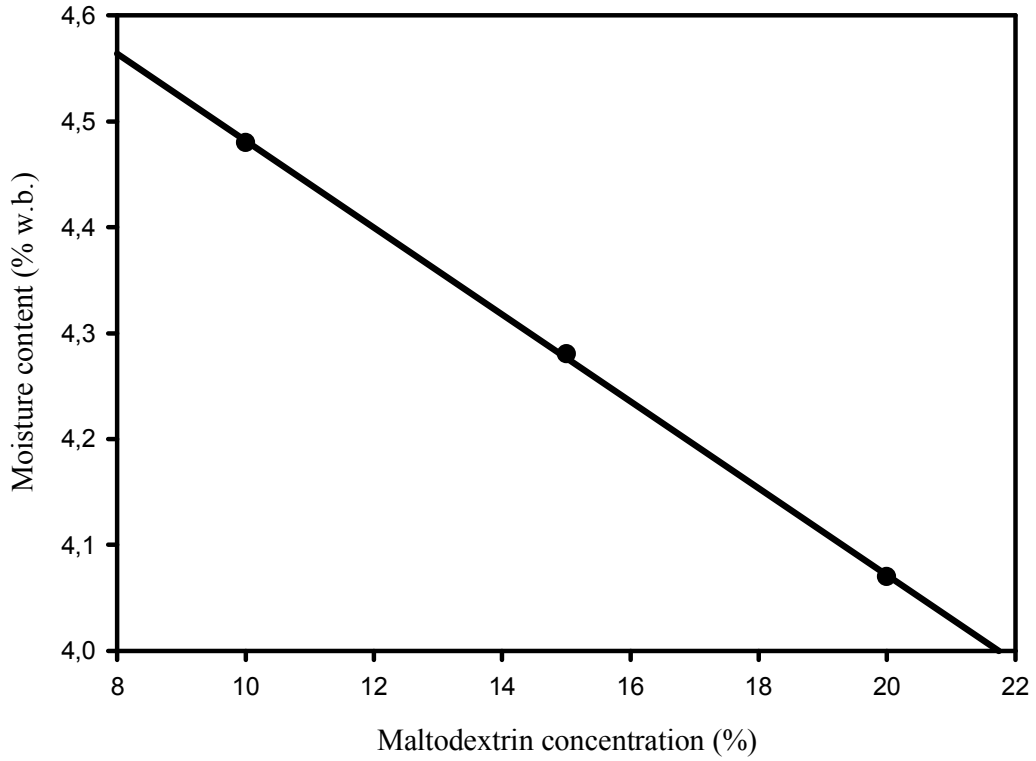


Figure 3.7 Graph of moisture content (% wet basis) for different maltodextrin concentrations (%)

In addition, higher maltodextrin dextrose equivalent caused an increase in powder moisture content (Figure 3.8). Goula and Adamopoulos (2010) mentioned that this may be due to the fact that high-DE maltodextrins develop stickiness slower and reach a state of non-adhesion slower than low- DE maltodextrins. The stickier a material is, the lower its drying rate.

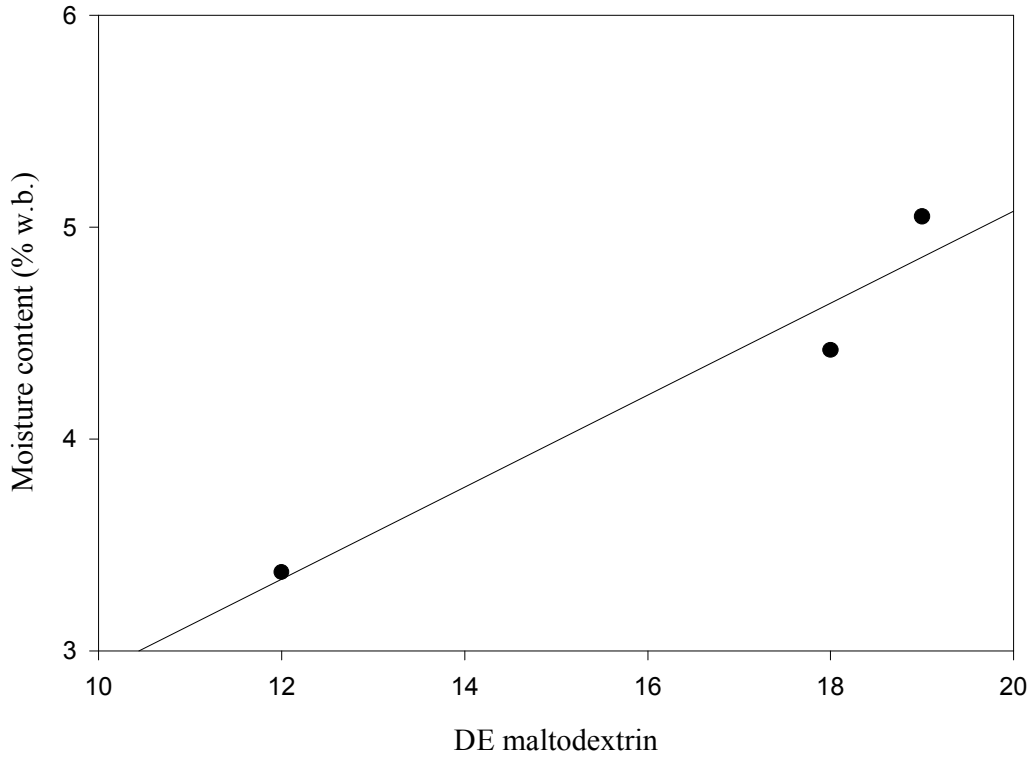


Figure 3.8. Graph of moisture content (% wet basis) for different maltodextrin dextrose equivalents (DE)

The effects of inlet air temprature and DE maltodextrin on moisture content were statistically significant ($p<0,05$). But the effect of maltodextrin concentration on moisture content was not significantly different. The final products which has maximum moisture content is 12 th., minimum moisture content is 25 th.

3.2.3 Effect of Parameters on Final Product Bulk Density

The bulk density of liquorice powders varied from 0.18 to 0.323 g/mL (Table 3.2). Decreases in bulk density was observed with increase in inlet air temperature (Figure 3.9).Walton and Mumford (1999) observed similar trends for the bulk density. Kha et al. (2010); Goula and Adamapoulos (2010) also observed a reduction of powders bulk density with increasing air temperatures, studying the spray drying of Gac (Momordica cochinchinensis) fruit aril powder and orange juice powders , respectively. Goula and Adamapoulos (2010) reported that evaporation rates are faster and products dry to a more porous or fragmented structure, there was a greater

tendency for the particles to be hollow. The higher drying rate obtained at higher drying temperatures that produce a higher ratio of surface to volume for the spray dried capsules, thus causing lower bulk density of the powders. This leads to the formation of vapor impermeable films on the droplet surface, followed by the formation of vapor bubbles and, consequently, droplet expansion (León-Martínez, 2010).

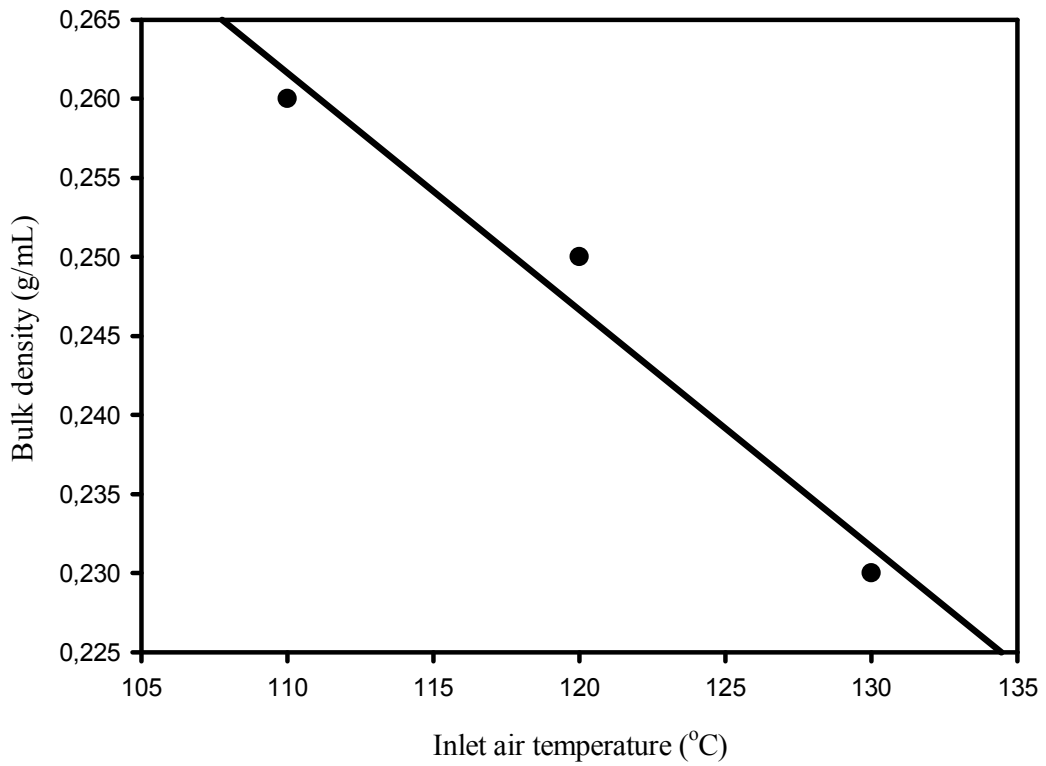


Figure 3.9. Graph of bulk density (g/mL) for different inlet air temperatures (°C)

Bulk density showed a decrease with an increase in maltodextrin concentration (Figure 3.10). This effect may be attributed to the fact that maltodextrin addition minimizes thermoplastic particles from sticking. In addition, an increase in maltodextrin concentration may cause an increase in the volume of air trapped in the particles, as maltodextrin is a skin-forming material. Generally, an increase in the volume of trapped air causes a decrease in the apparent density of the particles and this apparent density primarily determines the powder bulk density (Goula and Adamapoulos, 2010). Similar results were also reported by Yasar (2008) who carried out tests on pomegranate juice powder.

An increase in maltodextrin dextrose equivalent resulted in an increase in bulk density (Figure 3.11). This can be attributed to the fact that the higher the maltodextrin DE, the lower its glass transition temperature and, as a consequence, the lower the elevation of the Tg of the liquorice extract–maltodextrin mixture is and the more stickier the mixture is (Goula and Adamapoulos, 2010). Similar results were also reported by Yasar (2008) who carried out tests on pomegranate juice powder.

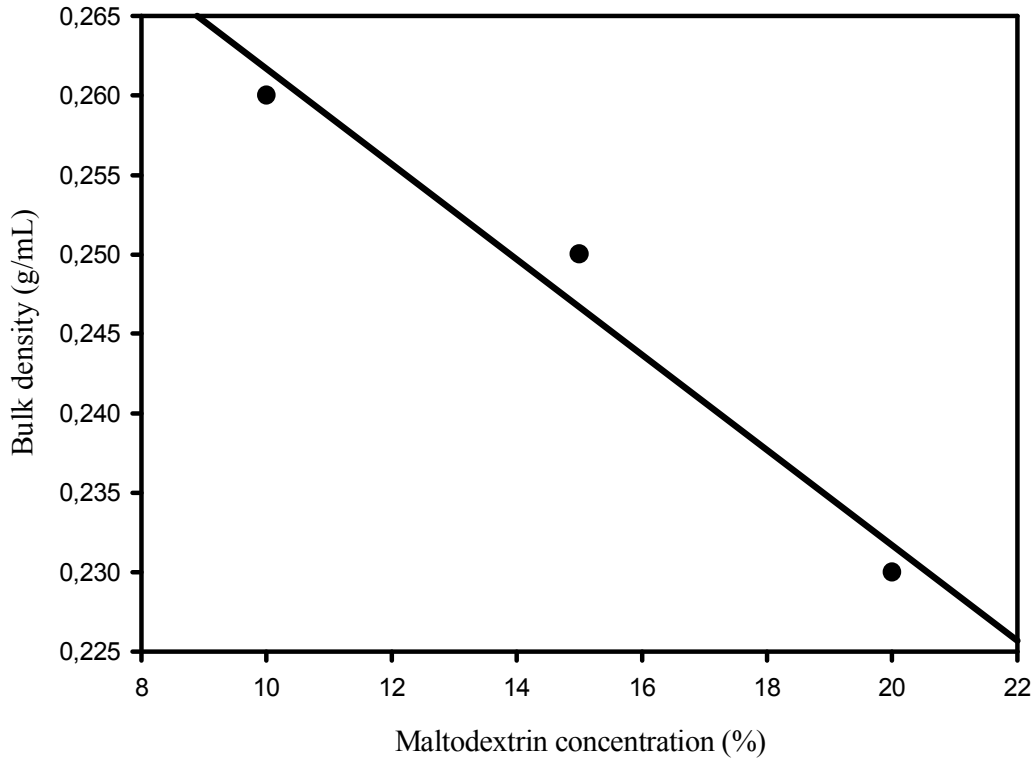


Figure 3.10 Graph of bulk density (g/mL) for different maltodextrin concentrations (%)

The effects of inlet air temprature, maltodextrin concentration, and DE maltodextrin on bulk density were statistically significant ($p<0,05$). Although the effects of 18 DE and 19 DE were not significantly different, that of 12 DE and 18 DE; 12 DE and 19 DE were significantly different. Although the effects of 110 °C and 120°C were not significantly different, that of 120 °C and 130 °C, 110 °C and 130°C were significant. The final products which has maximum bulk density is 12 th., minimum bulk density is 25 th.

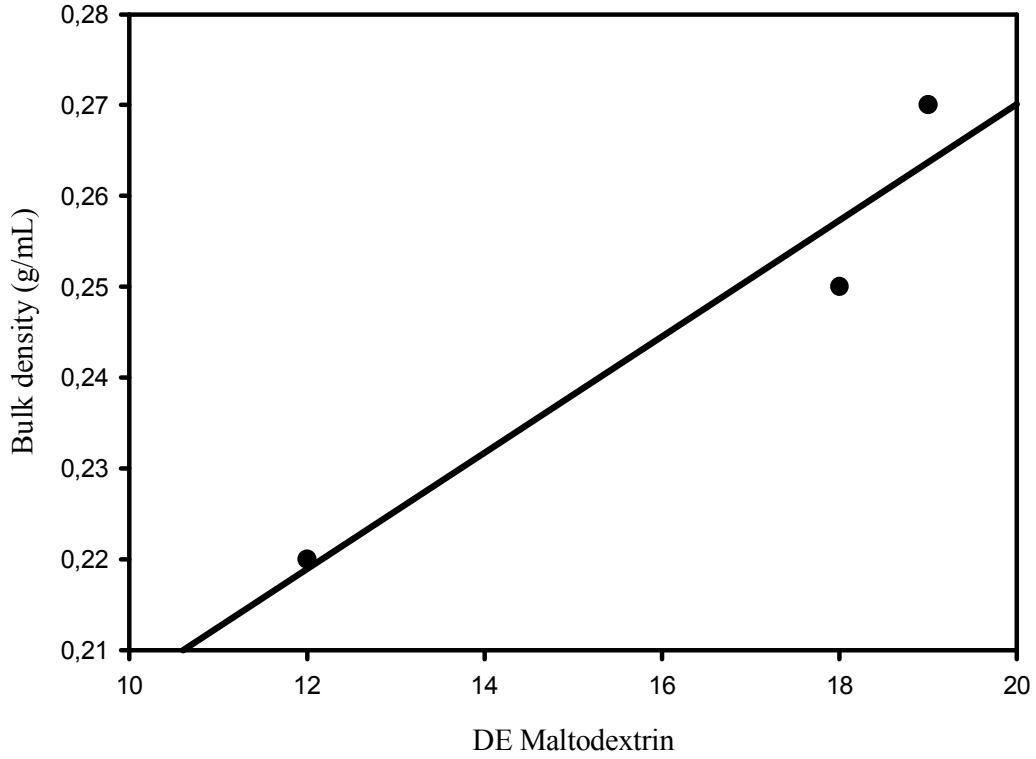


Figure 3.11. Graph of bulk density (g/mL) for different maltodextrin dextrose equivalents (DE)

3.2.4 Effect of Parameters on Final Product Hygroscopicity

Liquorice powder is evidently hygroscopic. Spray dried particles can easily absorb moisture from the surrounding air and, unless necessary precautions are taken, the surface of the powder becomes sticky and powder caking occurs (Goula and Adamapoulos, 2010). Moisture adsorption of the spray dried powders at 25 °C and 90% relative humidity after one week is shown in Table 3.2. Increases in inlet air temperature lead to higher powder Tg and, as a result, to lower hygroscopicity (Figure 3.12). Osman and Endut (2009) reported that the higher hygroscopicity of the powders produced at lower temperatures seems to be related to their higher moisture content. A major factor affecting powder stability is moisture content, since a small amount of water is able to depress the temperature enough to increase the mobility of the matrix during storage. Jaya and Das (2004) obtained similar results, working with the production of mango powder.

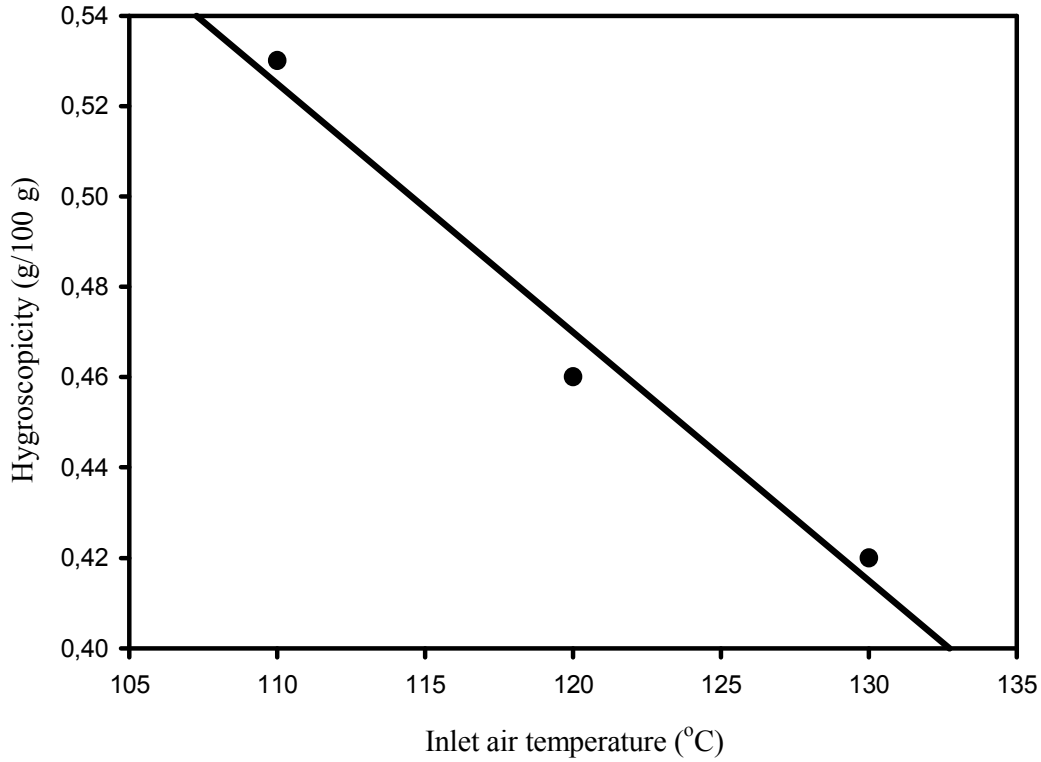


Figure 3.12. Graph of hygroscopicity (g/100 g) for different inlet air temperatures (°C)

Figure 3.13 shows the influence of maltodextrin concentration on hygroscopicity. Increases in the maltodextrin concentration lead to higher powder Tg (Table 3.2) and, as a result, to lower hygroscopicity. According to Kuruzowa et al. (2009), since the glass transition temperature increases with increase in molecular weight, the addition of maltodextrin to the feed solution contributed significantly to powder stability, increasing the Tg of the powder, and consequently reducing hygroscopicity and the stickiness. Maltodextrin is a material with low hygroscopicity, and confirms its efficiency as a carrier agent. This observation is similar to that reported by other researchers (Tonon et al., 2008; Goula and Adamapoulos, 2010).

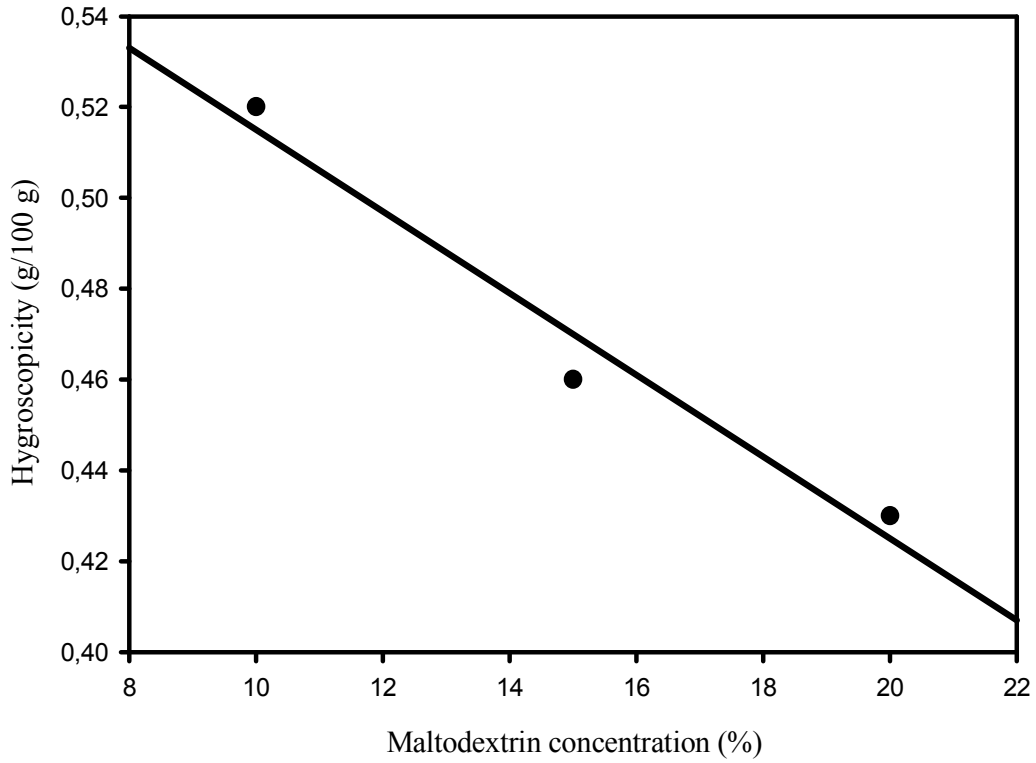


Figure 3.13. Graph of hygroscopicity (g/100 g) for different maltodextrin concentrations (%)

In addition, higher maltodextrin dextrose equivalent caused an increase in hygroscopicity (Figure 3.14). Tg is related to chain stiffness and polymer chain structure; it increases as cross-link density increases. A lower Tg causes higher hygroscopicity in the spray dried powder, as lower molecular weight implies shorter chains and more hydrophilic groups. Goula and Adamapoulos (2010), León-Martínez (2010) and Kuruzowa et al. (2009), and also observed similar results, studying the spray drying of orange juice concentrate, nopal mucilage (*Opuntia ficus-indica*) and, chicken meat protein hydrolysate , respectively.

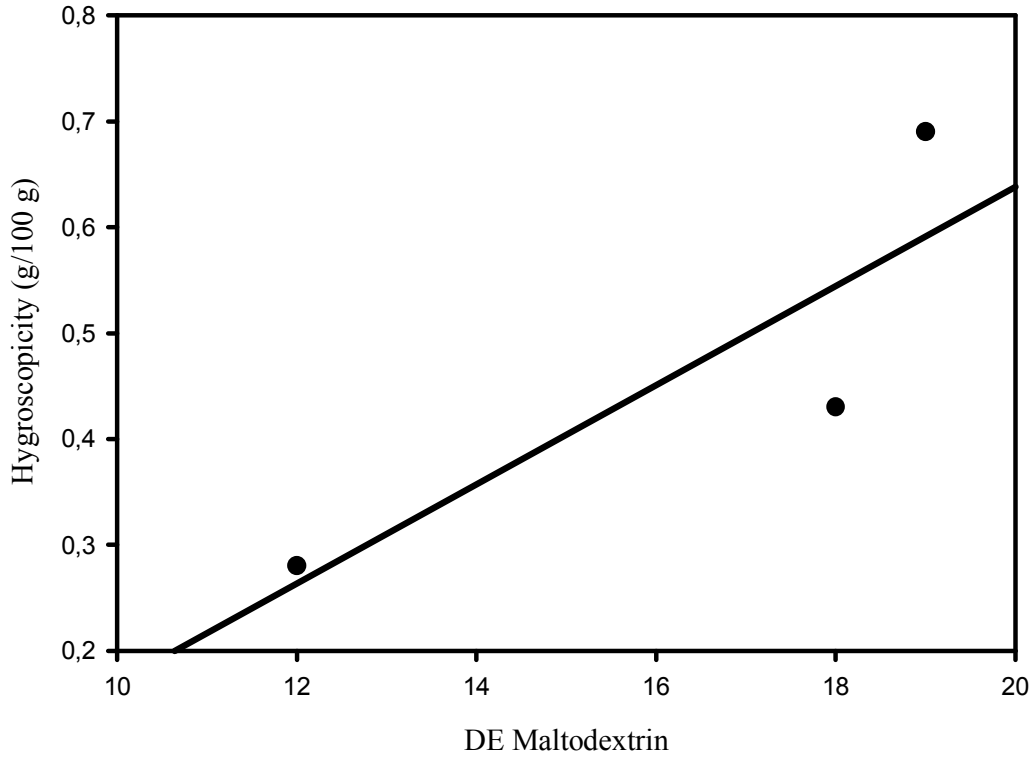


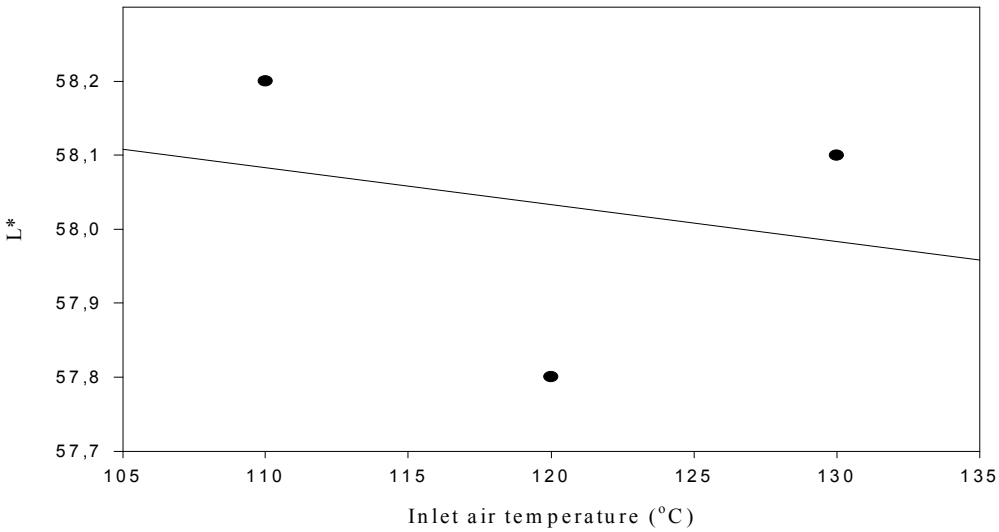
Figure 3.14. Graph of hygroscopicity (g/100 g) for different maltodextrin dextrose equivalents (DE)

The effects of inlet air temprature, maltodextrin concentration, and DE maltodextrin on hygroscopicity were statistically significant ($p<0,05$). Although the effects of 120°C and 130°C were not significantly different, that of 110°C and 120°C; 110°C and 130°C were significantly different. For maltodextrin concentration , the effects of %15 and % 20 maltodextrin concentrations were not significantly different. The final products which is the most hygroscopic is 12 th., least hygroscopic is 25 th.

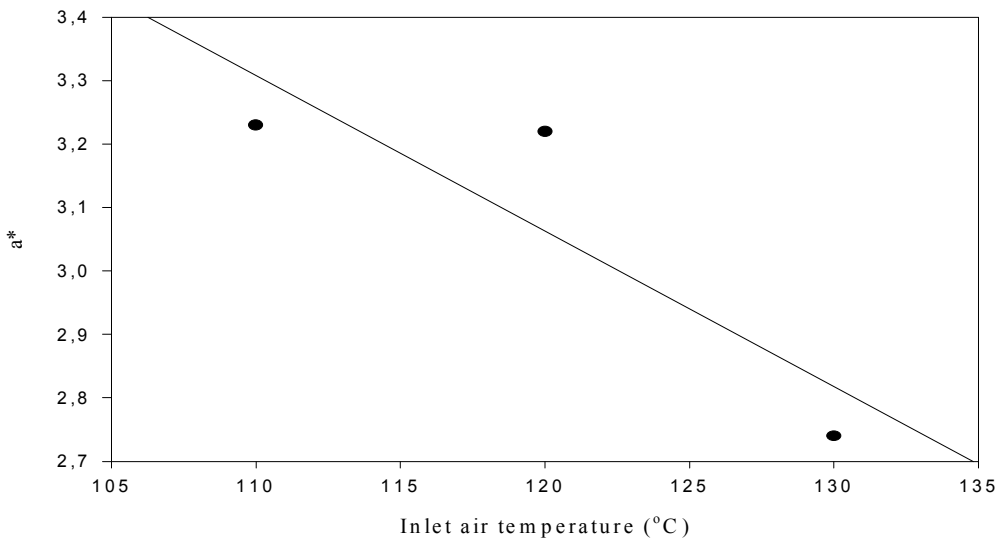
3.2.5 Effect of Parameters on Final Product Color

It is well known that high temperature can significantly decrease the quality of dried materials; e.g., by change of color. The results of the colour measurement for powders are shown in Table 3.2. L* value measures the lightness of the sample, +*a** measures the redness while +b* measures the yellowness. It was found that when inlet temperature increased, both the +b* values and the +*a** values decreased (Figure 3.15). The possible explanation for this phenomenon is that carrying out the

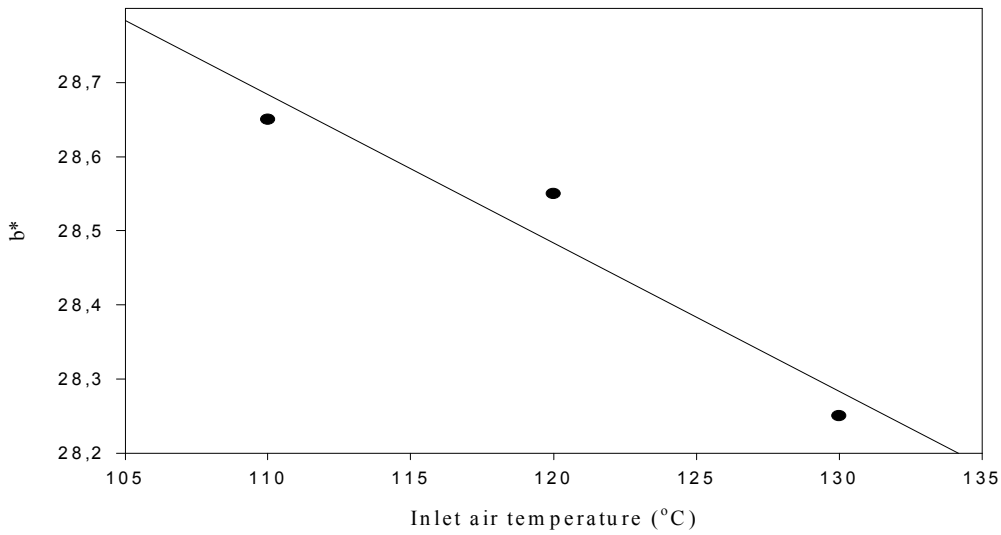
spray-drying process caused rapid pigment oxidation, non-enzymatic browning. Therefore, the spray drying conditions at high temperature resulted in a high loss of red colour due to thermal degradation of pigments (Kha et.al.2010). Overall, the lightness of the powders reduced. This means that the colour of the powders has become darker at higher inlet temperature.This is related with the sugars liquorice contain. Sugars could contribute to browning of the powders at higher inlet temperature. (Quek et al., 2007) and (Shi et al., 1999) also observed similar results, studying the spray drying of watermelon juice and tomato products, respectively.



(a)



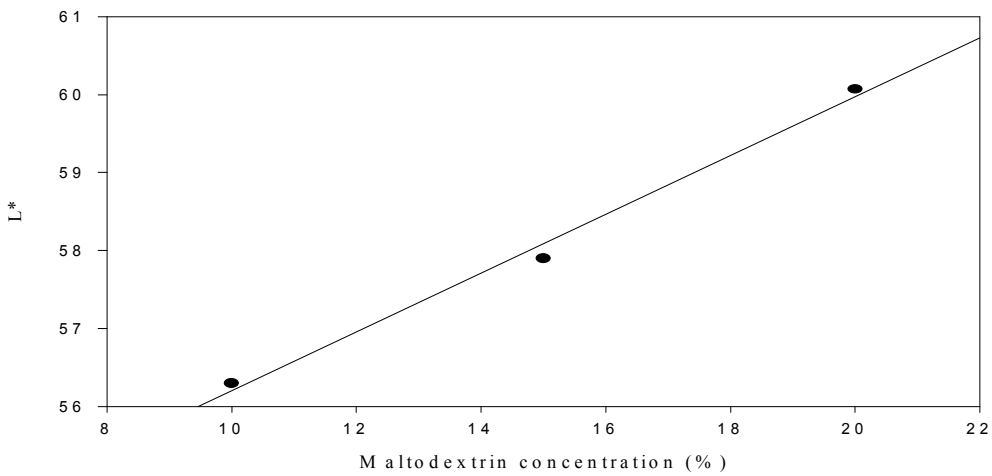
(b)



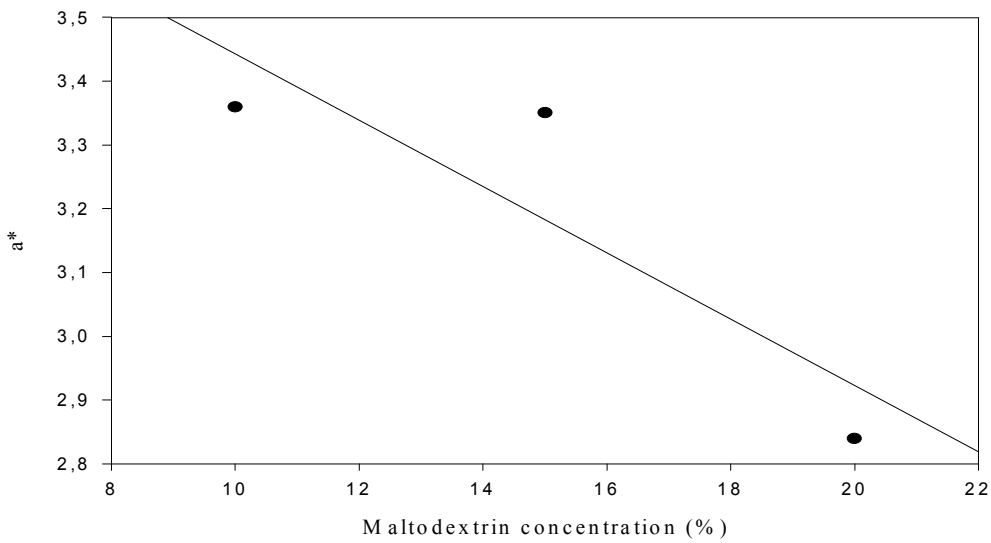
(c)

Figure 3.15 Graph of color change for different inlet air temperatures (°C) (a) L* values (b) a* values (c) b* values

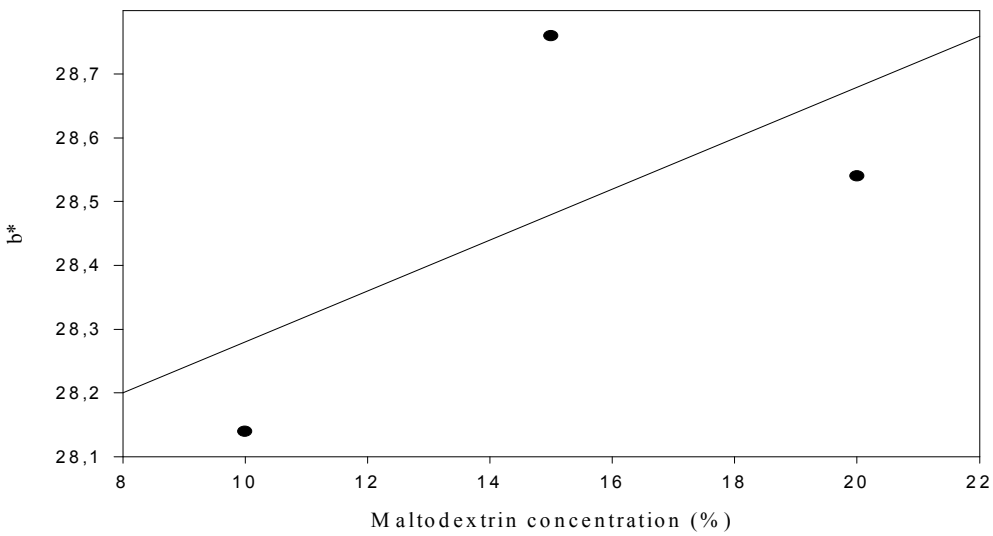
Generally, an increase in the lightness value of the powders was observed with an increased maltodextrin concentration due to the effect of the maltodextrin. Because of white colour of maltodextrin, a greater lightness of powders, represented by a higher L* value, was obtained at higher concentrations of maltodextrin. Lower values of a* were observed as a result of increasing maltodextrin concentration. These results indicate that the loss of redness of powder products increased in these spray drying conditions. Higher values of b* were observed as a result of increasing maltodextrin concentration. Similar results were also found in spray-dried gac powder (Kha et al., 2010) and in pineapple juice powders (Abadio et al., 2004).



(a)



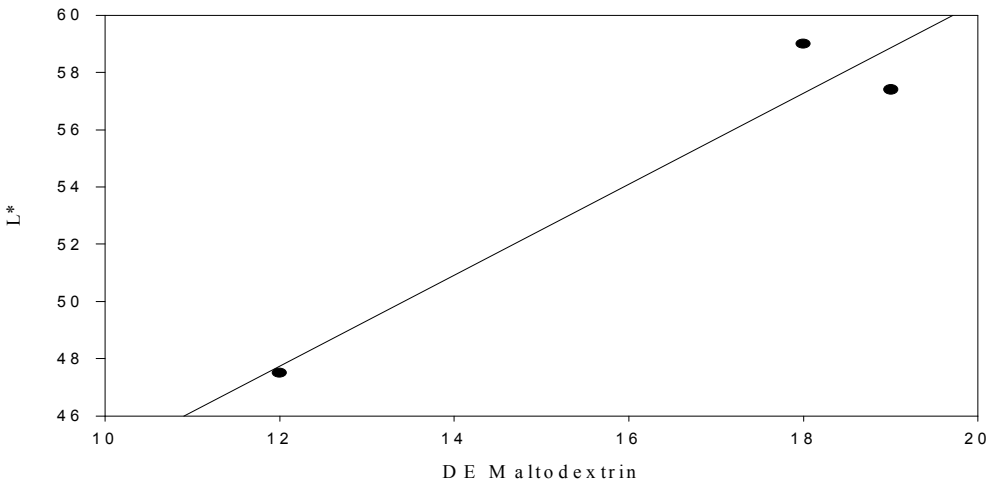
(b)



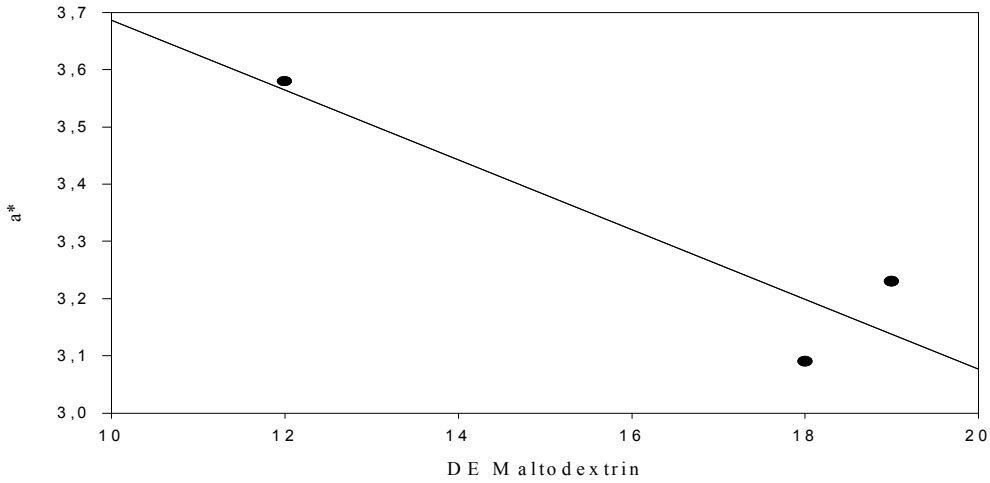
(c)

Figure 3.16. Graph of color change for different maltodextrin concentrations (%) (a) L* values (b) a* values (c) b* values

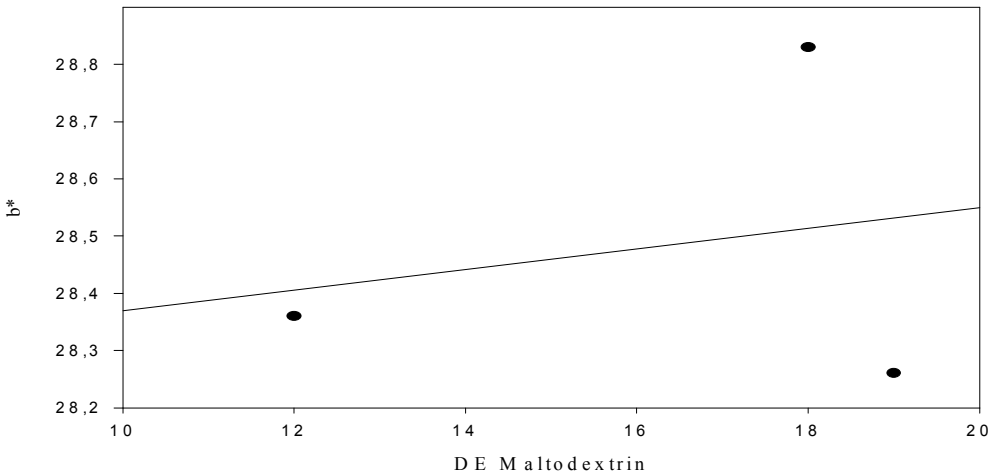
Increase in DE maltodextrins were resulted in increase in L* and b* values, decrase in a* values. Ersus and Yurdagel (2007); Kha et al. (2010) was observed different results for the tests.When the study of Yasar (2008) investigated, it was seen that some of results of the study is similar with that of this study and some of them are diffrent.This can be related with the experimental conditions.



(a)



(b)



(c)

Figure 3.17. Graph of color change for different maltodextrin dextrose equivalents (DE)a) L* values (b) a* values (c) b* values

The effects of all parameters studied were observed statistically significant ($p<0,05$).

3.2.6 Effect of Parameters on Final Product Acidity & pH

The results of the acidity & pH measurements for powders are shown in Table 3.2. A decrease in the acidity was observed with an increased inlet air temperature (°C), an increased DE maltodextrins, and decreased maltodextrin concentration (Figure 3.18, Figure 3.19, and Figure 3.20).

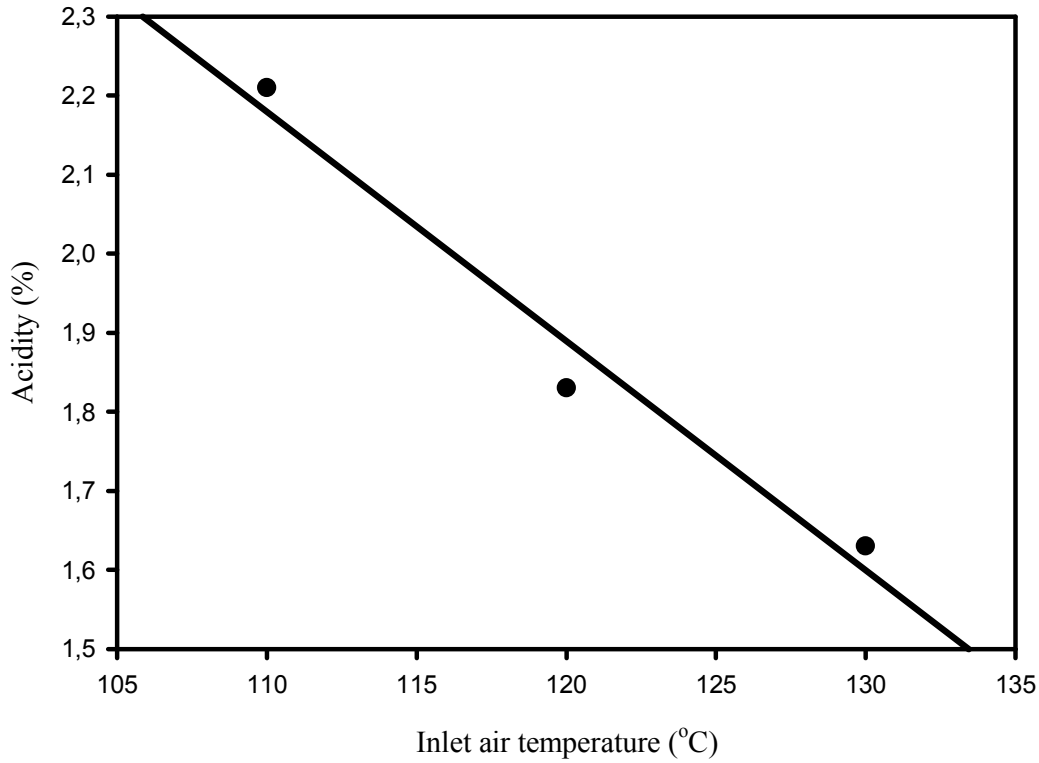


Figure 3.18. Graph of acidity (%) for different inlet air temperatures (°C)

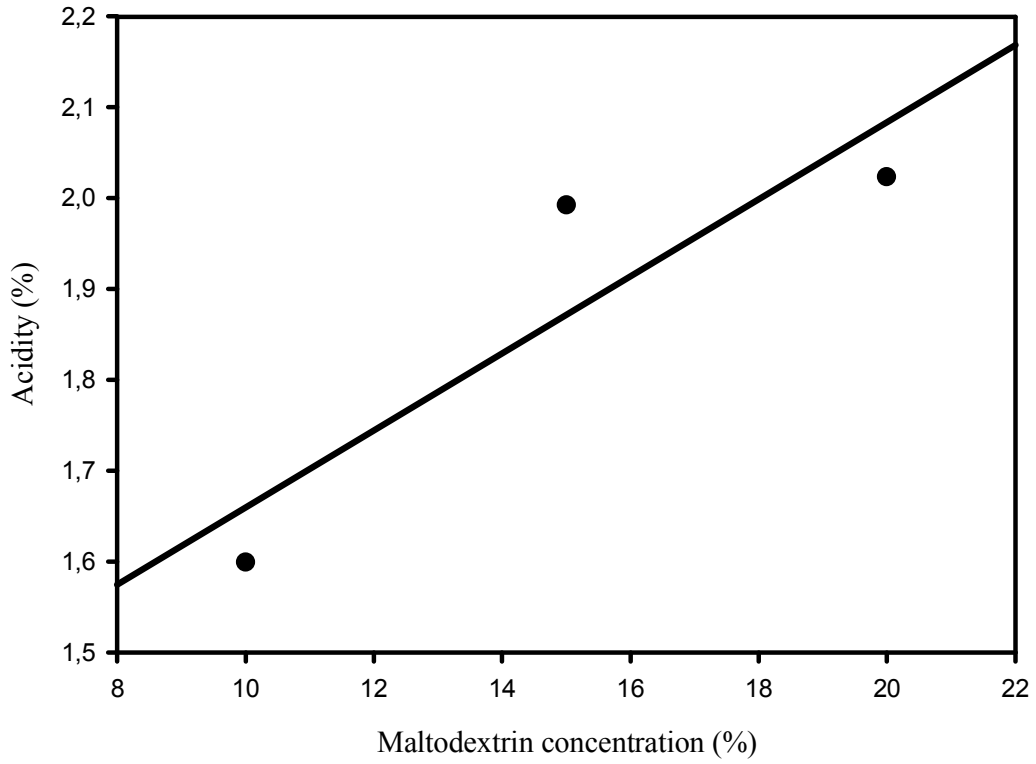


Figure 3.19. Graph of acidity (%) for different maltodextrin concentrations (%)

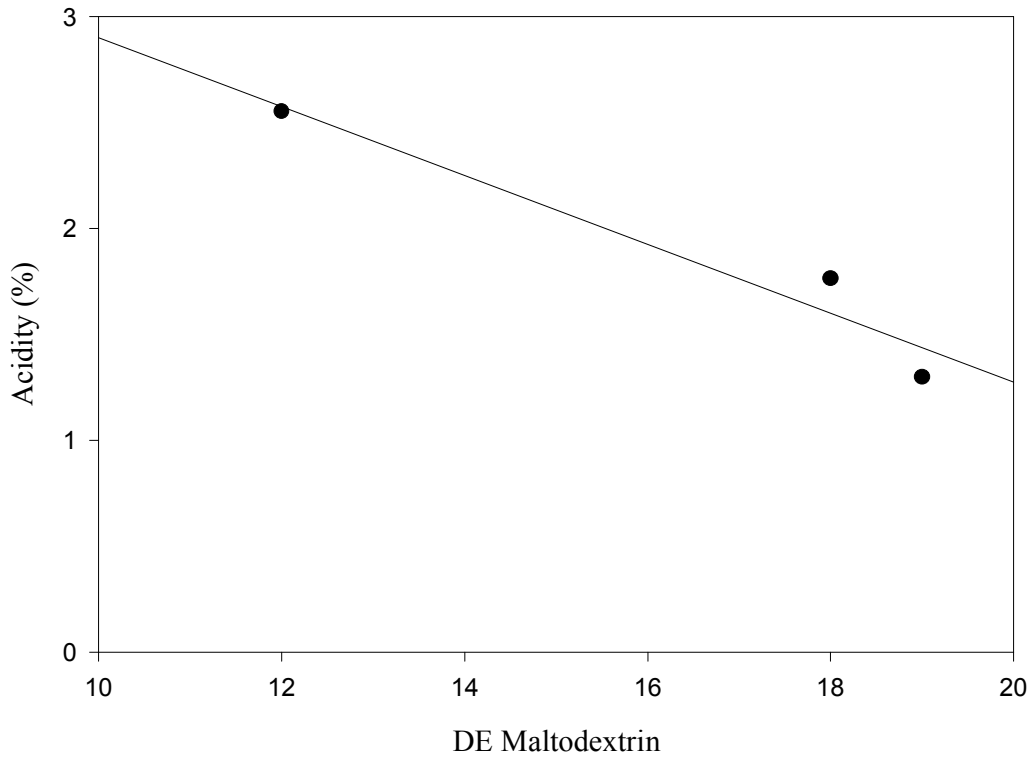


Figure 3.20. Graph of acidity (%) for different DE maltodextrins

Also an increase was observed in the pH with an decreased inlet air temperature (°C), an decreased DE maltodextrins, and increased maltodextrin concentration (Figure 3.21, Figure 3.22, and Figure 3.23). Kha et al. (2010) and Koc et al. (2010) also observed similar results, studying the spray drying of Gac (*Momordica cochinchinensis*) fruit aril powder and yogurt, respectively.

The effects of maltodextrin concentration and DE maltodextrin on acidity & pH were statistically significant ($p<0,05$). But the effect of inlet air temperature on moisture content was not significant. The final products which is the most acidic is 19 th., least acidic is 18 th.

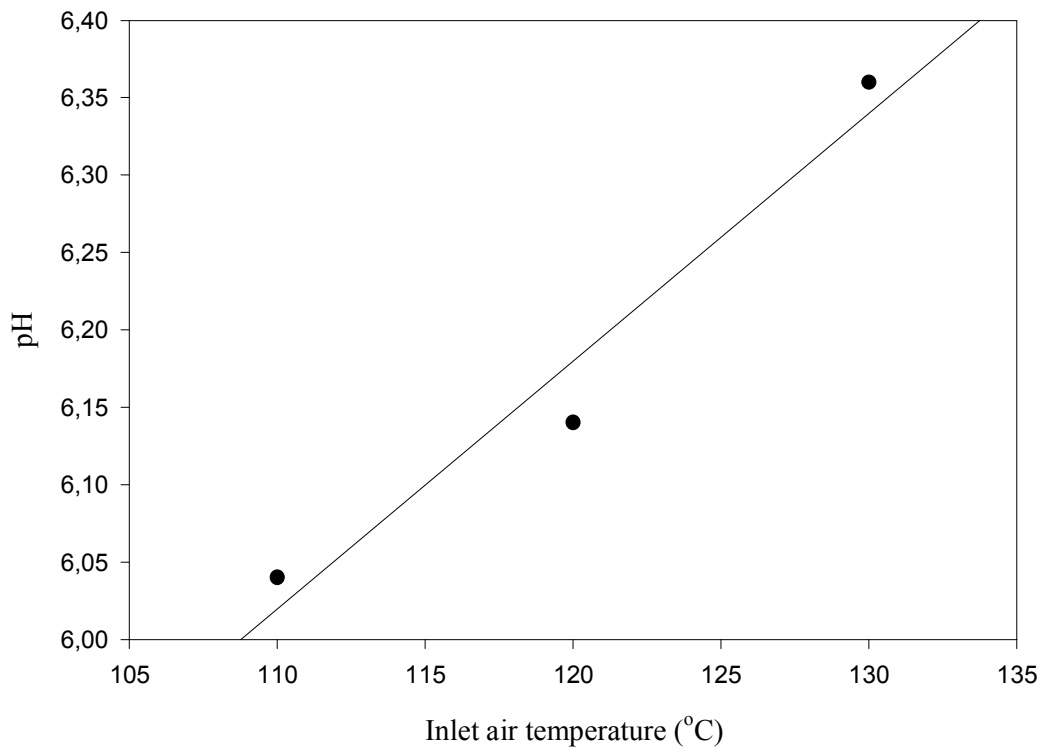


Figure 3.21. Graph of pH for different inlet air temperatures (°C)

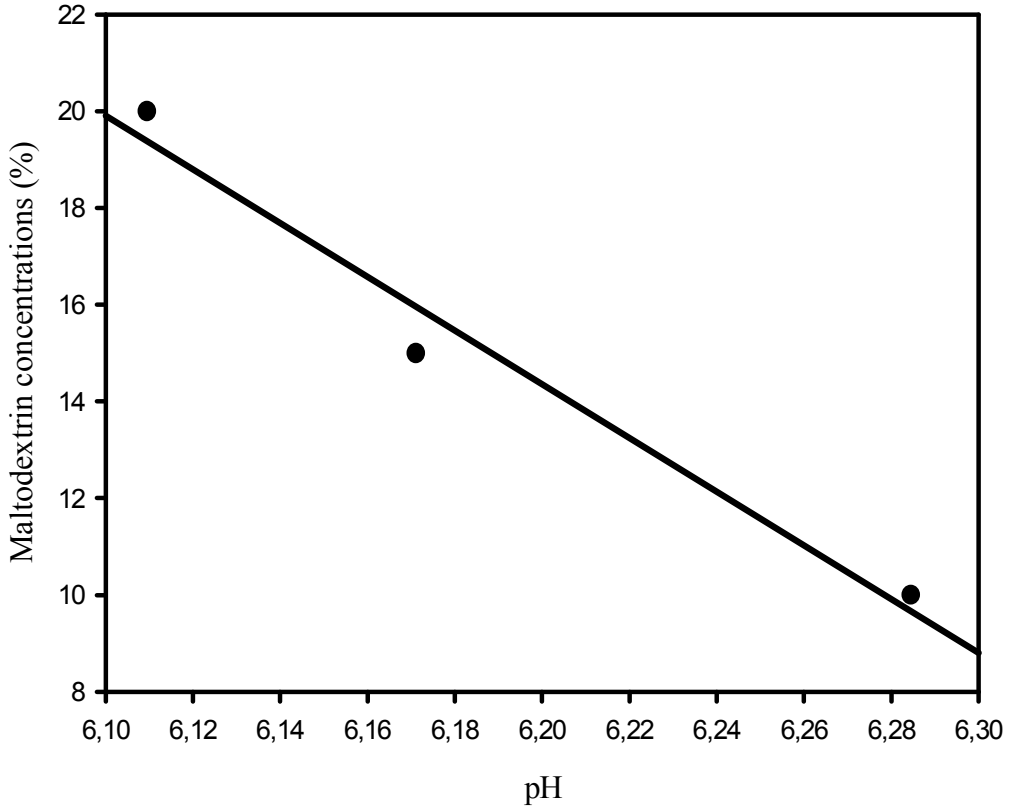


Figure 3.22. Graph of pH for different maltodextrin concentrations (%)

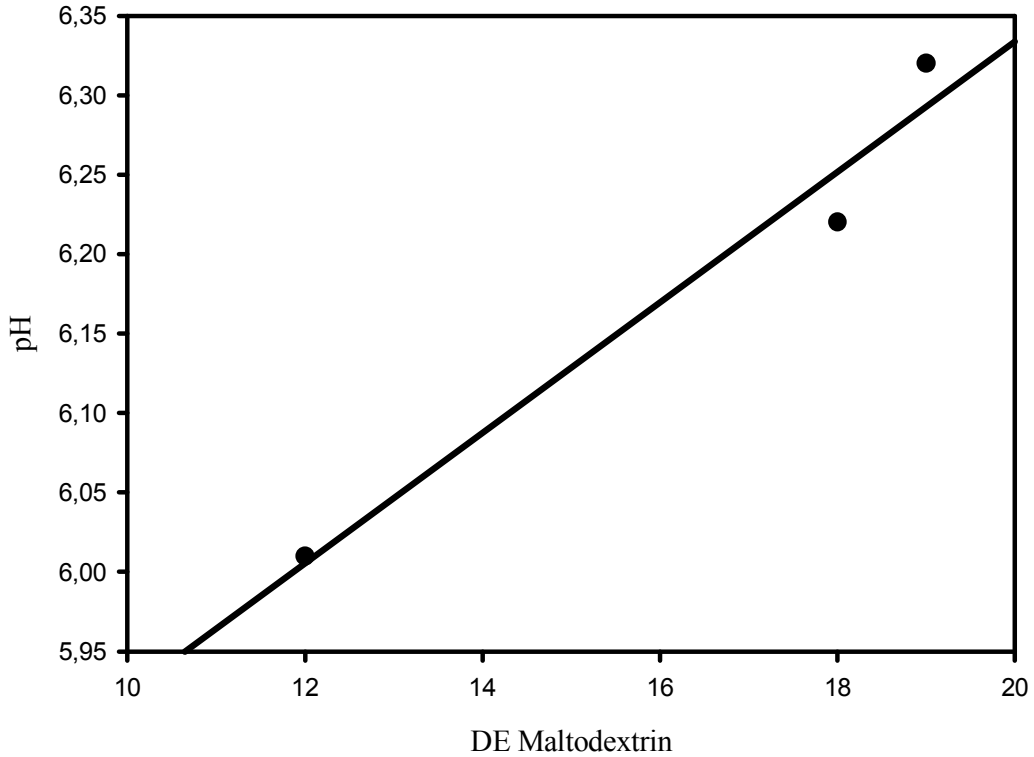


Figure 3.23. Graph of pH for different maltodextrin dextrose equivalents (DE)

3.2.7 Effect of Parameters on Final Product Solubility

The results of the solubility measurements for powders are shown in Table 3.2. A decrease in the solubility was observed with decreased inlet air temperature (°C), an increased DE maltodextrins, and increased maltodextrin concentration (Figure 3.24, Figure 3.25, and Figure 3.26). The higher the temperature of the solvent, the faster its rate of dissolving and the greater its solubility. Abadio et al.(2004) also observed similar results.

The effects of inlet air temperature and DE maltodextrin on solubility were statistically significant ($p<0,05$). But the effect of maltodextrin concentration on solubility was not significant. The final products which are dissolved earliest is 27 th., latest is 10 th.

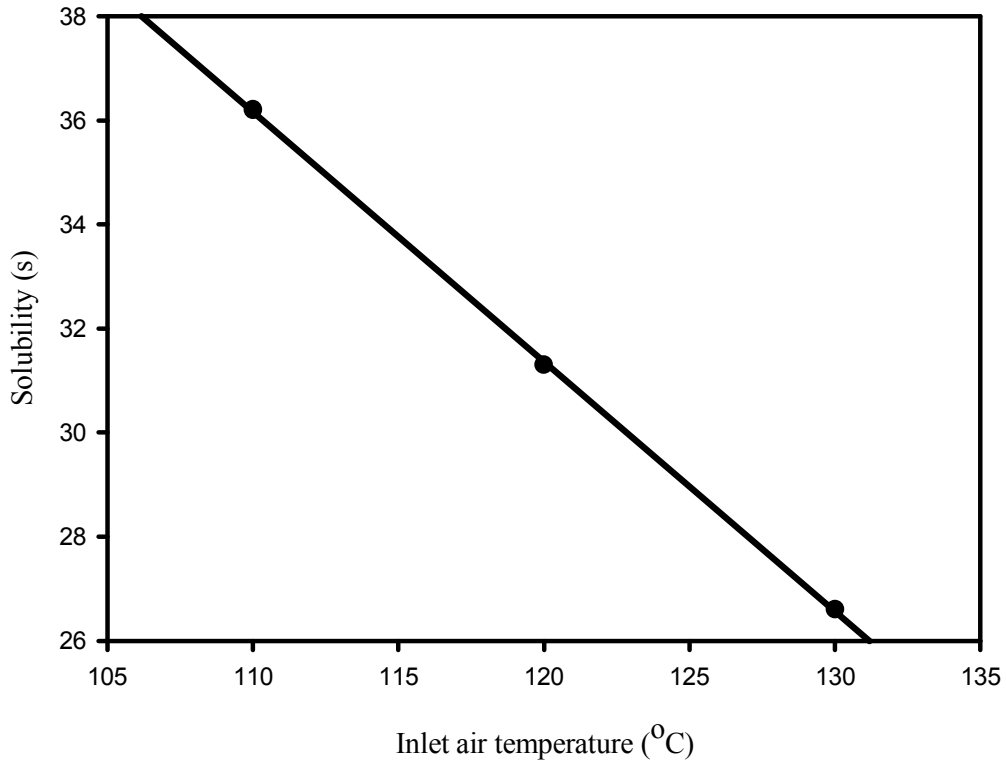


Figure 3.24. Graph of solubility (s) for different inlet air temperatures (°C)

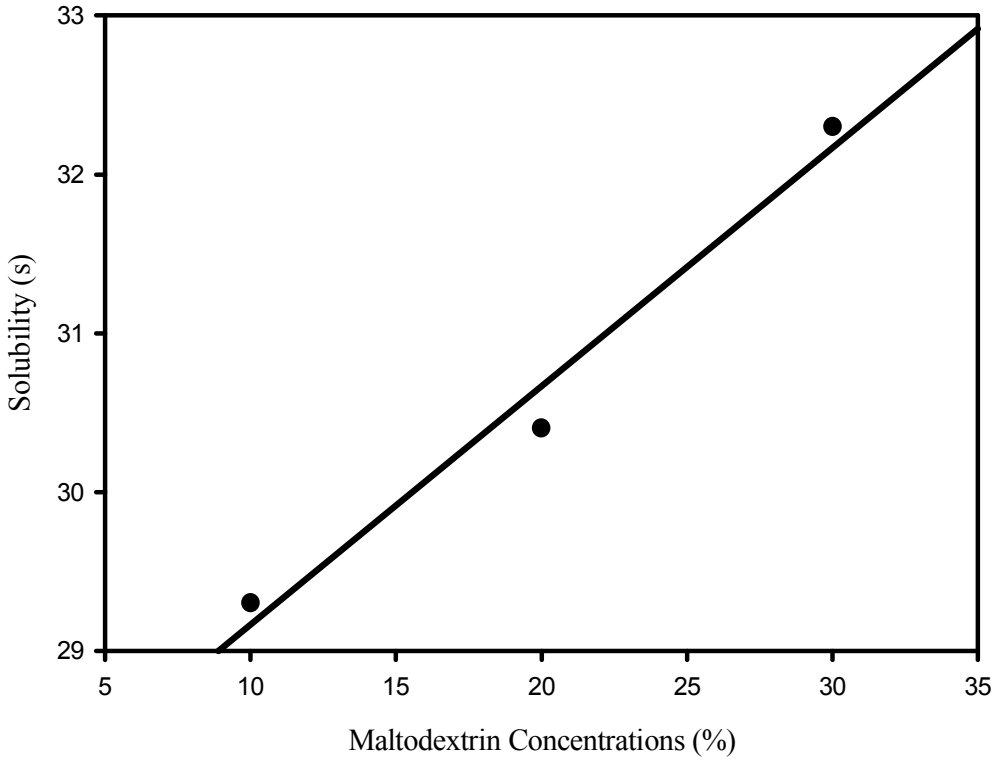


Figure 3.25. Graph of solubility (s) for different maltodextrin concentrations (%)

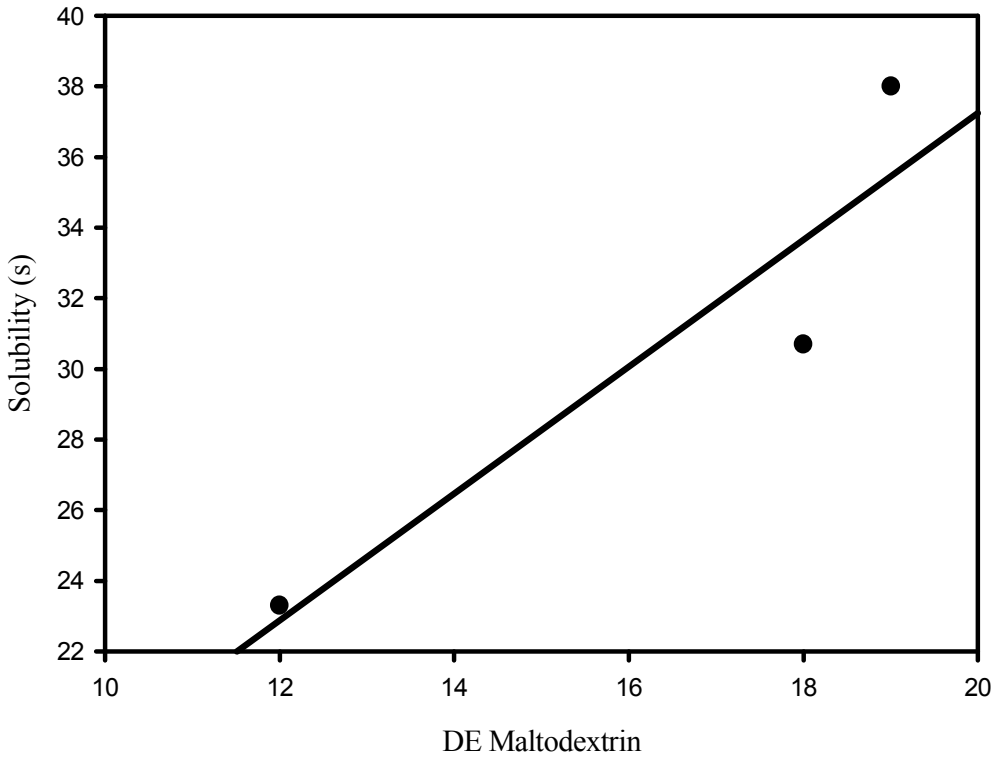


Figure 3.26. Graph of solubility (s) for different maltodextrin dextrose equivalents

CONCLUSIONS

Spray drying of liquorice extract using maltodextrin as drying agent was studied. The effect of maltodextrin concentration, inlet air temperature and the maltodextrin dextrose equivalent on the yield and powder properties was observed.

It was found that:

− Yield increases with an increase in maltodextrin concentration, inlet air temperature and a decrease in maltodextrin dextrose equivalent.

− Moisture content decreases with an increase in inlet air temperature and increase in maltodextrin concentration and dextrose equivalent.

− Bulk density increases with an increase in dextrose equivalent, a decrease in inlet air temperature and maltodextrin concentration.

- Hygroscopicity decreases with a decrease in dextrose equivalent, an increase in inlet air temperature and maltodextrin concentration.

- L* value increases with a decrease in inlet air temperature, an increase in maltodextrin concentration and maltodextrin dextrose equivalent. Increase in temperature, maltodextrin concentration and dextrose equivalent cause a decrease in a* value.An increase in inlet air temperature, a decrease in maltodextrin concentration and maltodextrin dextrose equivalent cause a decrease in a* value.

-Acidity decreases with a decrease in maltodextrin concentration, an increase in inlet air temperature and maltodextrin dextrose equivalent. pH increase with a decrease in maltodextrin concentration, an increase in inlet air temperature and maltodextrin dextrose equivalent

- A decrease in the solubility was observed with decreased inlet air temperature (°C), an increased DE maltodextrins, and increased maltodextrin concentration.

Addition of maltodextrin is an effective way of spray drying. It reduces stickiness at the dryer chamber and gave the better physical properties of the produced powders. Best product with minimum moisture content, bulk density, hygroscopicity, color quality loss and maximum solubility and yield.

REFERENCES

Ariño, A., Herrera, M., Estopañan, G., Juan, T. (2007). High levels of ochratoxin A in licorice and derived products. *International Journal of Food Microbiology* , 114, 366–369

Abadio, F.D.B., Domingues, A.M., Borges, S.V., Oliveira, V.M. (2004). Physical properties of powdered pineapple (Ananas comosus) juice—effect of maltodextrin concentration and atomization speed. *Journal of Food Engineering*, 64, 285–287

Brennan, J. G., Herrera, J. G., Jowitt, R. (1971). A study of some of the factors affecting the spray drying of concentrated orange juice, on a laboratory scale. *Journal of Food Technology*, 6, 295-307.

Ersus, S., Yurdagel, Ü. (2007). Microencapsulation of anthocyanin pigments of black carrot (Daucuscarota L.) by spray drier. *Journal of Food Engineering*, 80, 805–812

Fiore, C., Eisenhut, M., Ragazzi, E., Zanchin, G., Armanini, D. (2005). A history of the therapeutic use of liquorice in Europe. *Journal of Enthopharmacology*, 99, 317-324

Goula, A.M., Adamopoulos, K.G. (2010). A new technique for spray drying orange juice concentrate. *Innovative Food Science and Emerging Technologies*, 11, 342–351

Goula, A. M., Adamopoulos, K.G. (2005). Stability of lycopene during spray drying of tomato pulp. *LWT*, 38, 479–487

Harold, R. W. (2001). An Introduction to Appearance Analysis. *Second Sight, Graphic Arts Technical Foundation.* Number 84.

http://www.armfield.co.uk(20.12.2010)

http:// www.bete.com, (20.12.2010)

http:// www.callidan.com, (20.12.2010)

http://www.foodsci.uoguelph.ca, (20.12.2010)

http://www.hunterlab.com, (20.12.2010)

Isbrucker, R.A., Burdock, G.A. (2006). Risk and safety assessment on the consumption of Licorice root (Glycyrrhiza sp.), its extract and powder as a food ingredient, with emphasis on the pharmacology and toxicology of glycyrrhizin. *Regulatory Toxicology and Pharmacology,* 46, 167–192

Jaya, S., Das, H. (2004). Effect of maltodextrin, glycerol monostearate and tricalcium phosphate on vacuum dried mango powder properties. *Journal of Food Engineering*, 63, 125–134

John Shi, J., Maguer, M.L., Kakuda, Y., Liptay, A., Niekamp, F. (1999). Lycopene degradation and isomerization in tomato dehydration. *Food Research International*, 32, 15-21

Kha, T. C., Nguyen, M. H., Roach, P. D. (2010). Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (Momordica cochinchinensis) fruit aril powder. *Journal of Food Engineering*, 98, 385–392

Koc, B., YIlmazer, M. S., Balkır, P., Ertekin, F. K. (2010). Spray Drying of Yogurt: Optimization of Process Conditions for Improving Viability and Other Quality Attributes. *Drying Technology,* 28: 4, 495 — 507

Kurozawa, L.E., Park, K. J., Hubinger, M.D. (2009). Effect of carrier agents on the physicochemical properties of a spray dried chicken meat protein hydrolysate. *Journal of Food Engineering,* 94, 326–333

Lee, Y.C. and Lee, S.W. (1999). Quality changes during storage in Korean clear pear juices concentrated by three methods. *Journal of Food Quality*. 22, 565-571

León-Martínez, F.M., Méndez-Lagunas, L.L., Rodríguez-Ramírez, J. (2010). Spray drying of nopal mucilage (Opuntia ficus-indica): Effects on powder properties and characterization. *Carbohydrate Polymers*, 81, 864–870

Lecorvaisier, E., Darche, S., Silva, Z. E., Silva, C. K. F. (2010) . Theoretical model of a drying system including turbulence aspects. *Journal of Food Engineering*, 96, 365–373

Mukhopadhyay, M., Panja, P. (2008). A novel process for extraction of natural sweetener from licorice (Glycyrrhiza glabra) roots. *Separation and Purification Technology*, 63, 539–545

Nagarajan, R., Singh, P., Mehrotra, R. (2006). Direct Determination of Moisture in Powder Milk Using Near Infrared Spectroscopy. *Journal of Automated Methods and Management in Chemistry,* Volume 2006, Pages 1–4

Naviglio, D., Pizzolongo, F., Romano, R., Ferrara, L., Naviglio, B., Santini, A. (2007). An innovative solid-liquid extraction technology: use of the naviglio extractor for the production of lemon liquor. *African Journal of Food Science*, Vol 1 pp., 042-050

Nestlé Research Center. (2009). *TG–DTA approach for designing reference methods for moisture content determination in food powders*. Switzerland: Vuataz, G., Meunier, V., Andrieux, J. C.

Pan, X., Liu , H., Jia, G., Shu, Y. Y. (2000). Microwave-assisted extraction of glycyrrhizic acid from licorice root. *Biochemical Engineering Journal,* 5, 173–177

Papadakis, S. E., Gardeli, C., Tzia, C. (2006). Spray Drying of Rising Juice Concentrate. *Drying Technology*, 24, 173-180

Proceedings of The South Afvican Sugar Technologists' Association. (1981). Large Fallig Film Evaporators and Vapour Compression. Addison, W.

Quek, S.Y., Chok, N. K., Swedlund, P. (2007). The physicochemical properties of spray-dried watermelon powders. *Chemical Engineering and Processing*, 46, 386–392

Rattes, A. L. R., Oliveira, W. P. (2007). Spray drying conditions and encapsulating composition effects on formation and properties of sodium diclofenac microparticles. *Powder Technology*, 171, 7–14

Riberio Jr., C.P., Borges, C.P., Lage, P.L.C., (2007). Sparger effects during the concentration of fruit juices by direct-contact evaporation. *Journal of Food Engineering*. 79, 979-988

Sablani, S. S., Shrestha, A. K., Bhandari, B. R. (2008), A new method of producing date powder granules: Physicochemical characteristics of powder. *Journal of Food Engineering*, 87, 416–421

Saravacos, G.D., Mayer, J.C., Wooster, G.D.(1970). Concentration of Liquid Foods in a Pilot-Scale Falling Film Evaporator.*New York's* F*ood Science and Technology*. No:4

Second International Conference on Environmental and Computer Science. (2009). Spray Drying of Roselle-Pineapple Juice. Effects of Inlet Temperature and Maltodextrin on the Physical Properties. Malaysia: Osman, A.F.A., Endut. N.

Srikiatden, J., Roberts, J. S. (2007). Moisture Transfer In Solid Food Materials: A Review of Mechanisms, Models, and Measurements. *International Journal of Food Properties*, 10, 739–777

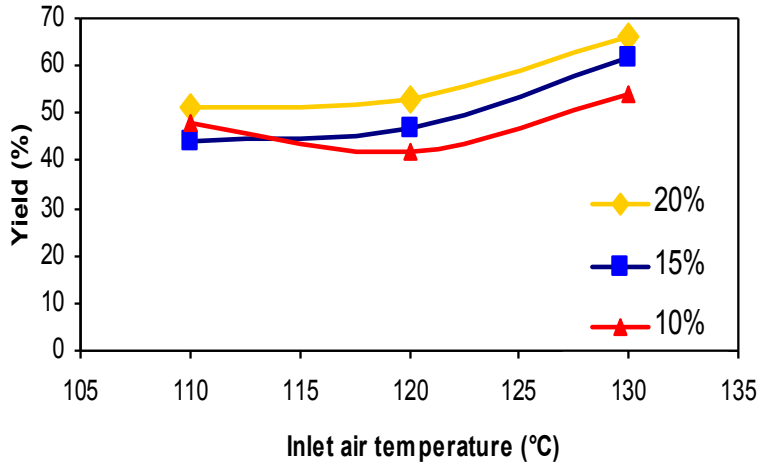
The Ohio Satate University. (2009).Drying Fruits and Vegetables. Ohio. Smith, L. K., Ph.D.

Tonon, R.V., Brabet, C., Hubinger, M.D. (2008). Influence of process conditions on the physicochemical properties of acai (Euterpe oleraceae Mart.) powder produced by spray drying. *Journal of Food Engineering*, 88, 411–418

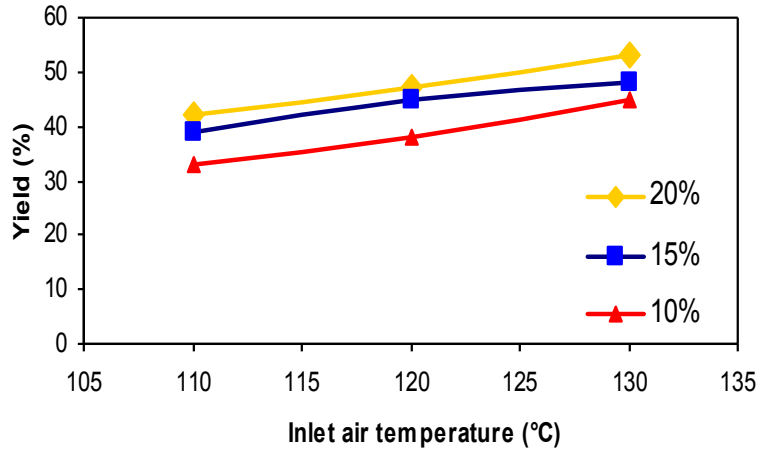
Yasar, M. (2000). Nar Suyuna Farklı Oranlarda Maltodextrin Eklenerek Püskürtmeli Kurutucu ile Nar Suyu Tozu Elde Edilmesi Üzerine Bir Çalışma. *A thesis submitted in conformity with the requirements for the degree of Master of Science in the Food Engineering Department, University of Harran.*

Yurdakoç, M. , Akçay, M., Tonbul, Y., Yurdakoç, K. (1999). Acidity of Silica-Alumina Catalysts By Amine Titration Using Hammett Indicators and FT-IR Study of Pyridine Adsorption. *Turk J Chem,* 23, 319-327.

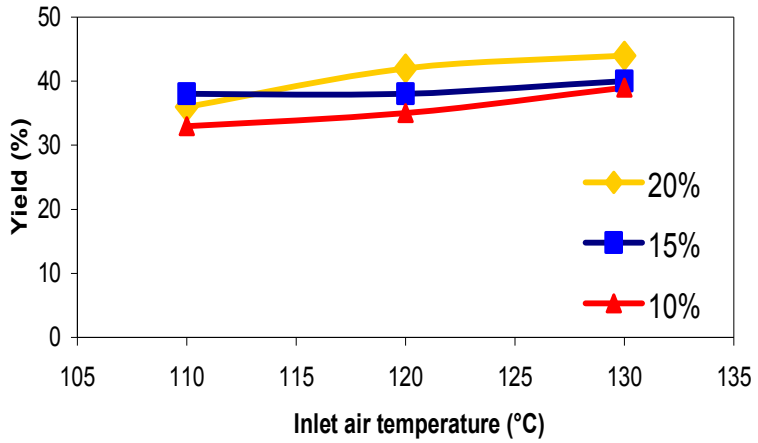
APPENDICES A



(a)

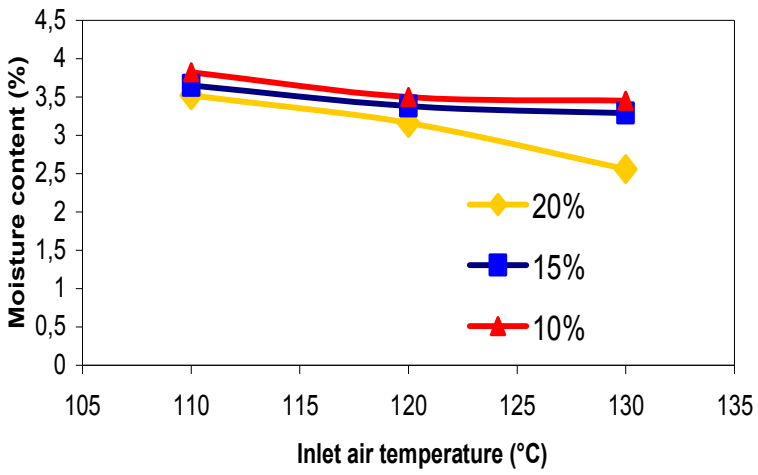


(b)

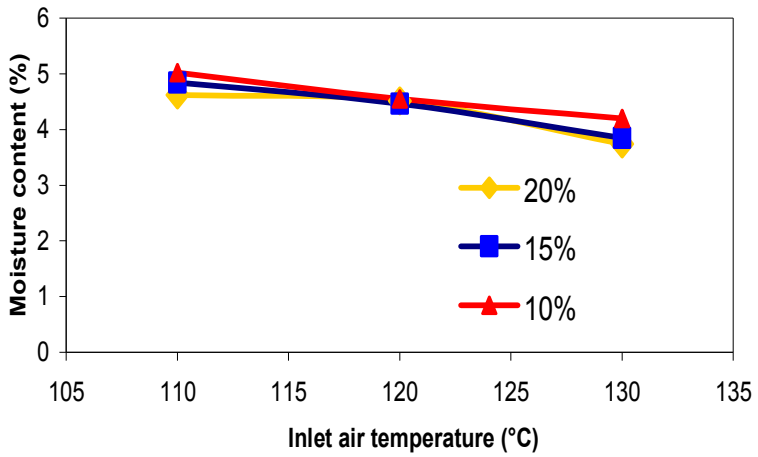


(c)

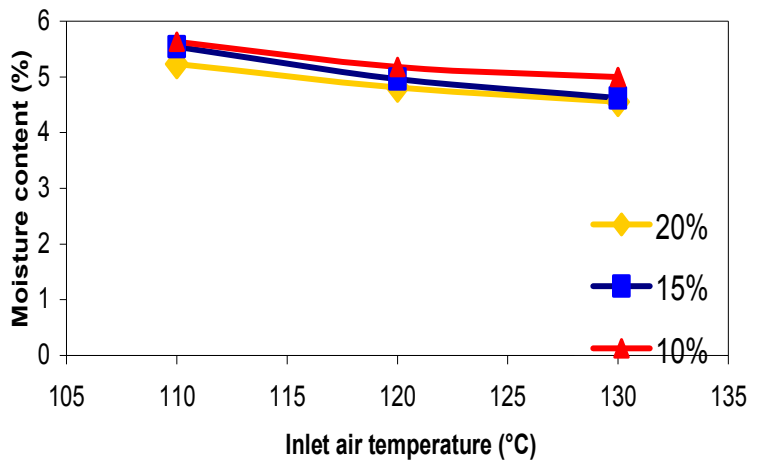
Figure A.1. Graphs of yield (%) at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

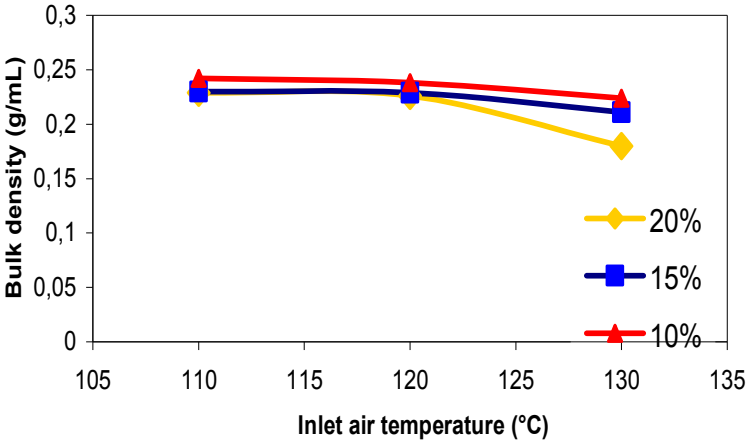


(b)

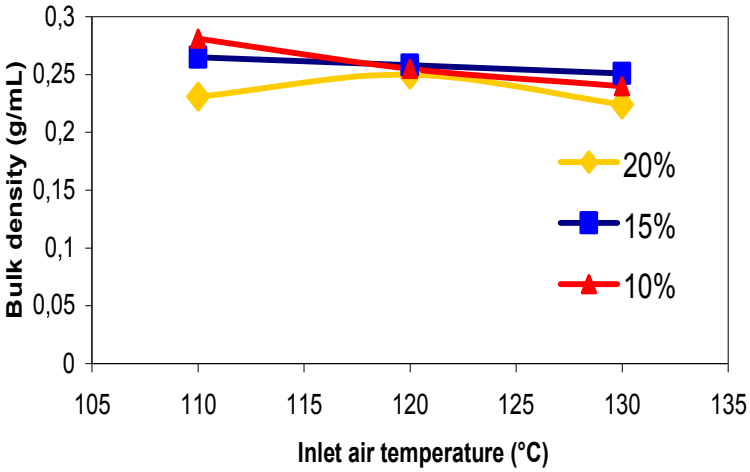


(c)

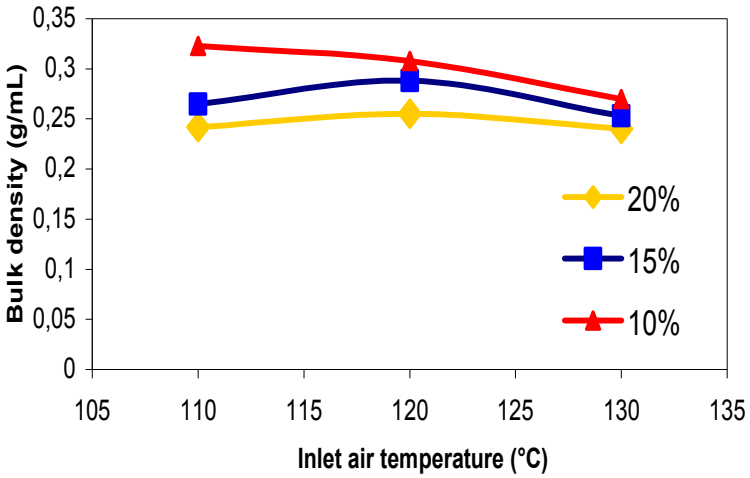
Figure A.2. Graphs of moisture content (%) at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

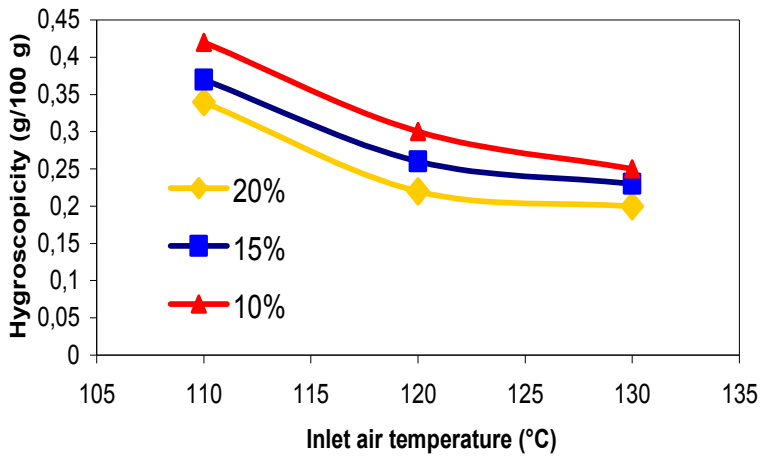


(b)

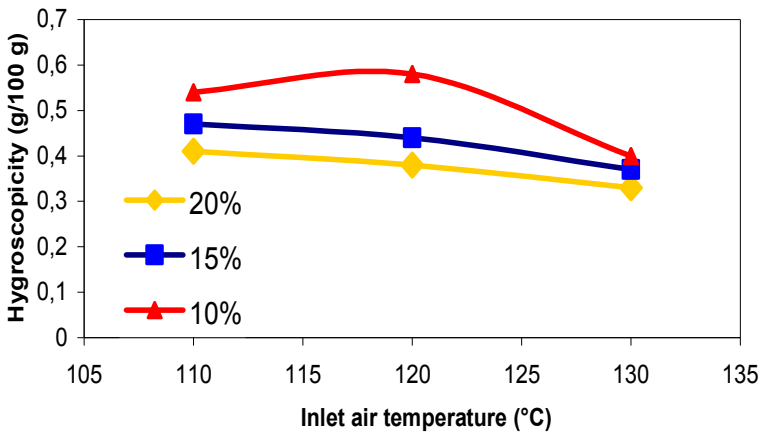


(c)

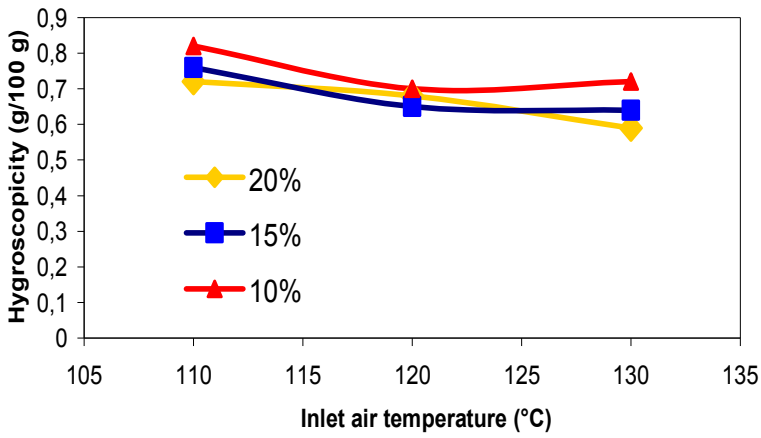
Figure A.3. Graphs of bulk density (g/mL) at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

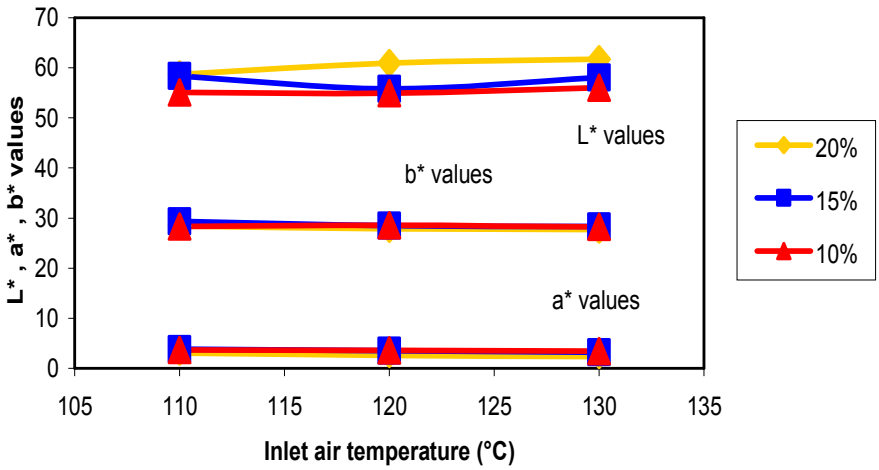


(b)

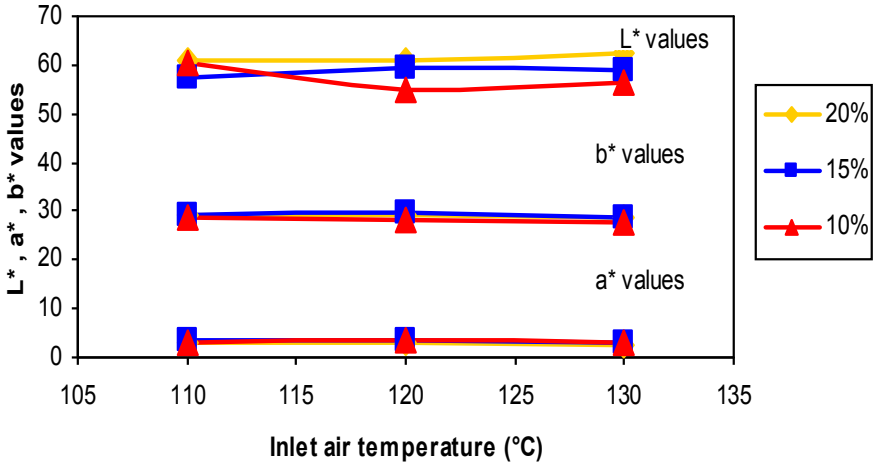


(c)

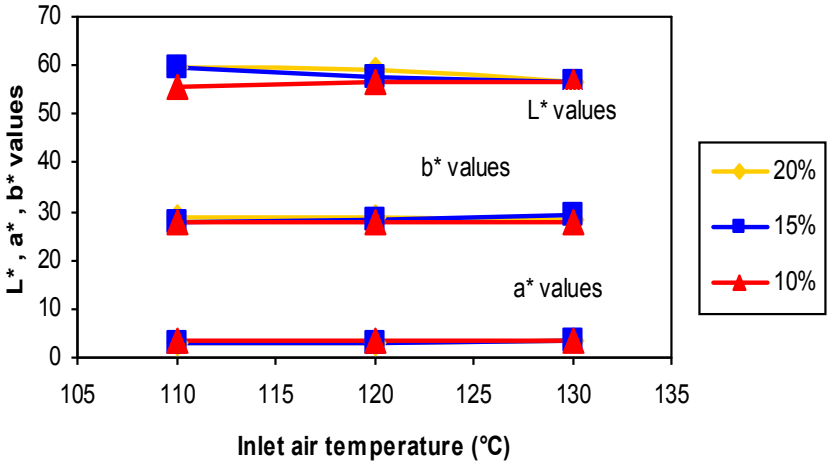
Figure A.4. Graphs of hygroscopicity (g/100g) at different inlet air temperatures (°C), maltodextrin concentrations (%), DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

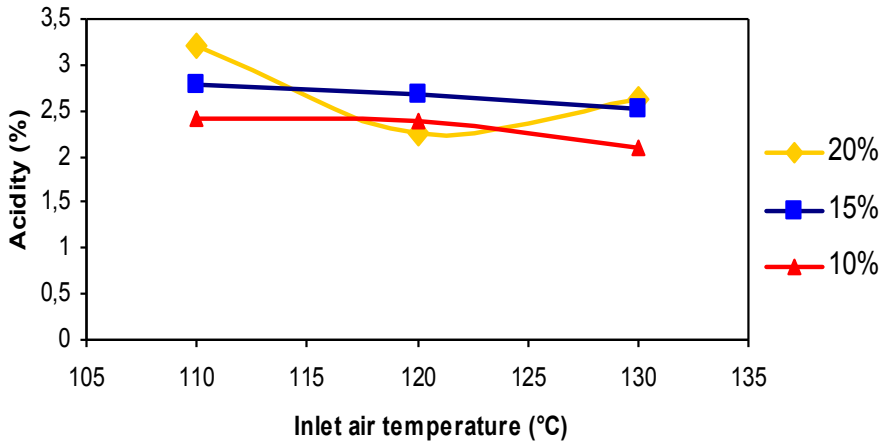


(b)

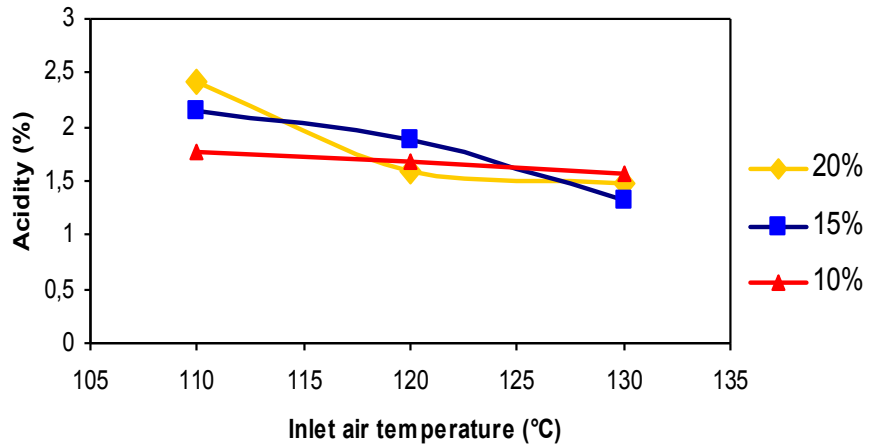


(c)

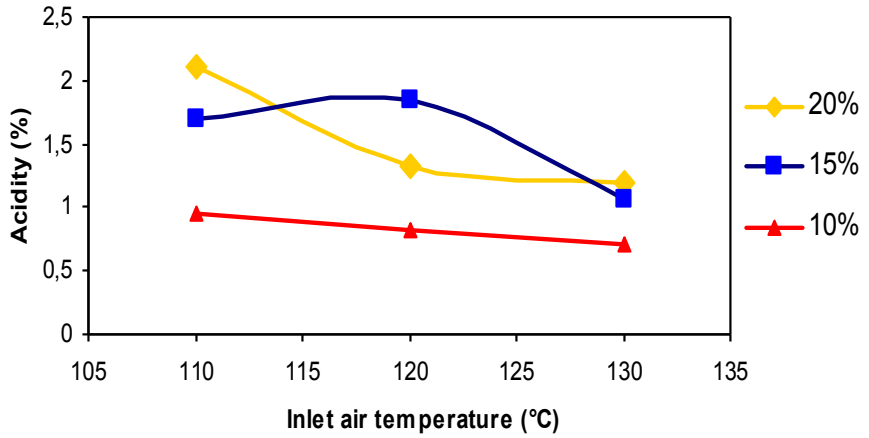
Figure A.5. Graphs of color change at different inlet air temperatures (°C), maltodextrin concentrations (%), DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

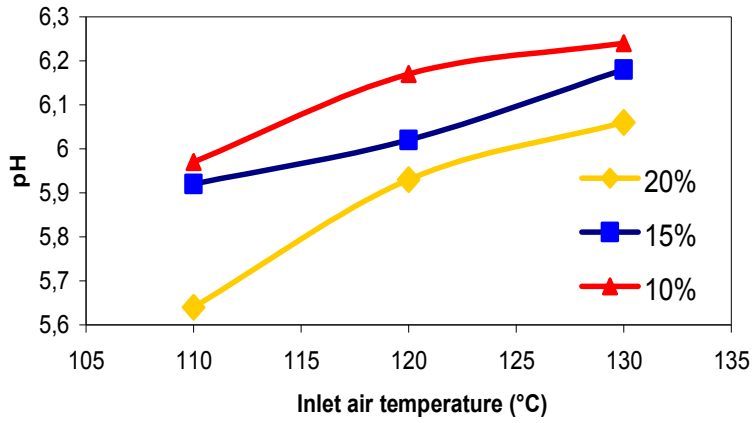


(b)

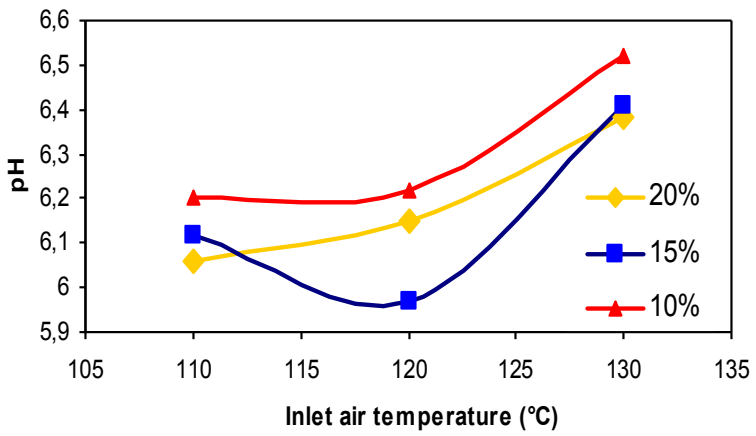


(c)

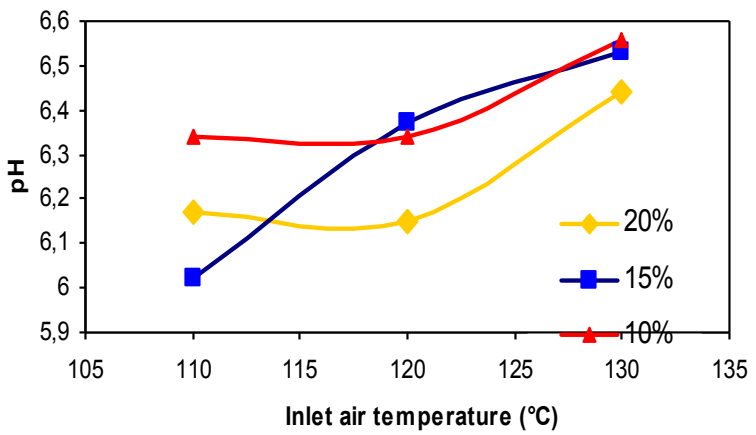
Figure A.6. Graphs of acidity (%) at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)

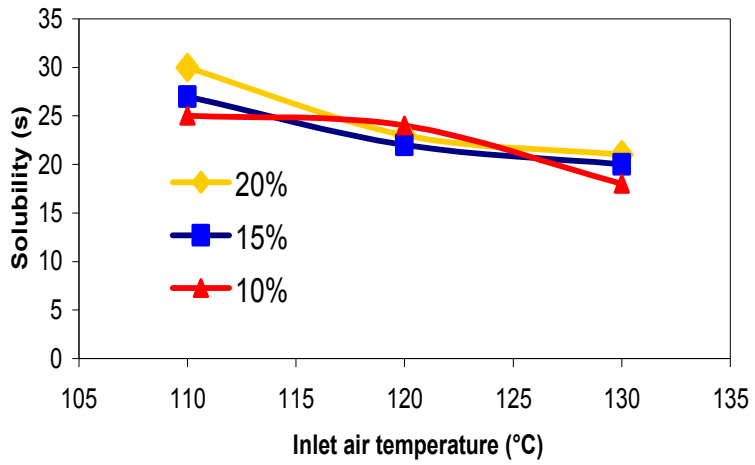


(b)

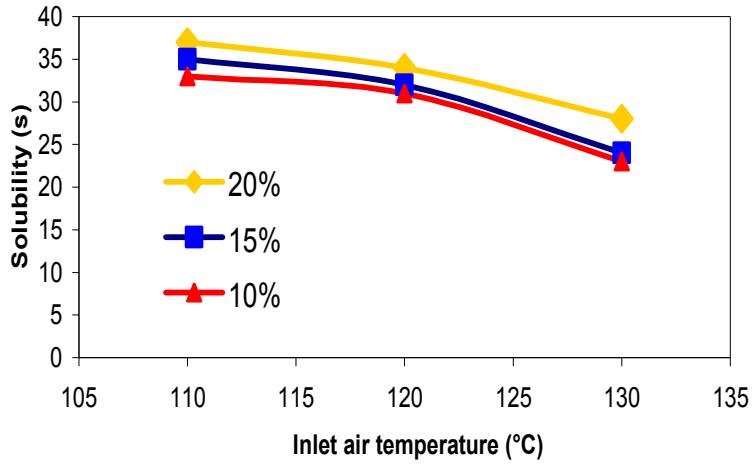


(c)

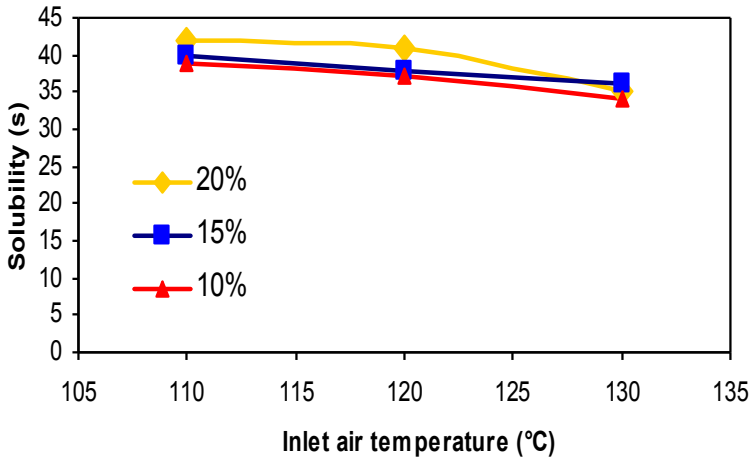
Figure A.7. Graphs of pH at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19



(a)



(b)



(c)

Figure A.8. Graphs of solubility at different inlet air temperatures (°C), maltodextrin concentrations (%) , DE Maltodextrins (a) DE 12 (b) DE 18 (c) DE 19

APPENDICES B

Table B.1. One-way ANOVA and LSD Test for yield analysis

Tests of Between-Subjects Effects

Dependent Variable: Yield

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1406,074[a]	8	175,759	9,568	,000
Intercept	53511,259	1	53511,259	2912,911	,000
DE	845,852	2	422,926	23,022	,000
Inlet_air_temperature	451,630	2	225,815	12,292	,000
DE * Inlet_air_ temperature	108,593	4	27,148	1,478	,250
Error	330,667	18	18,370		
Total	55248,000	27			
Corrected Total	1736,741	26			

Multiple Comparisons

Dependent Variable: Yield

	(I) Inlet_air_temperature	(J) Inlet_air_temperature	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	110	120	-2,56	3,450	,742	-11,17	6,06
		130	-9,67*	3,450	,026	-18,28	-1,05
	120	110	2,56	3,450	,742	-6,06	11,17
		130	-7,11	3,450	,119	-15,73	1,50
	130	110	9,67*	3,450	,026	1,05	18,28
		120	7,11	3,450	,119	-1,50	15,73
LSD	110	120	-2,56	3,450	,466	-9,68	4,56
		130	-9,67*	3,450	,010	-16,79	-2,55
	120	110	2,56	3,450	,466	-4,56	9,68
		130	-7,11	3,450	,050	-14,23	,01
	130	110	9,67*	3,450	,010	2,55	16,79
		120	7,11	3,450	,050	-,01	14,23

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Yield

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1114,741[a]	8	139,343	4,032	,007
Intercept	53511,259	1	53511,259	1548,557	,000
Maltodextrin_ concentration	249,407	2	124,704	3,609	,048
DE	845,852	2	422,926	12,239	,000
Maltodextrin_ concentration * DE	19,481	4	4,870	,141	,965
Error	622,000	18	34,556		
Total	55248,000	27			
Corrected Total	1736,741	26			

Multiple Comparisons

Dependent Variable: Yield

	(I) Maltodextrin_ concentration	(J) Maltodextrin_ concentration	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Tukey HSD	10	15	-3,78	2,771	,380	-10,85	3,29
		20	-7,44*	2,771	,038	-14,52	-,37
	15	10	3,78	2,771	,380	-3,29	10,85
		20	-3,67	2,771	,401	-10,74	3,41
	20	10	7,44*	2,771	,038	,37	14,52
		15	3,67	2,771	,401	-3,41	10,74
LSD	10	15	-3,78	2,771	,190	-9,60	2,04
		20	-7,44*	2,771	,015	-13,27	-1,62
	15	10	3,78	2,771	,190	-2,04	9,60
		20	-3,67	2,771	,202	-9,49	2,16
	20	10	7,44*	2,771	,015	1,62	13,27
		15	3,67	2,771	,202	-2,16	9,49

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Yield

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1406,074[a]	8	175,759	9,568	,000
Intercept	53511,259	1	53511,259	2912,911	,000
DE	845,852	2	422,926	23,022	,000
Inlet_air_temperature	451,630	2	225,815	12,292	,000
DE * Inlet_air_ temperature	108,593	4	27,148	1,478	,250
Error	330,667	18	18,370		
Total	55248,000	27			
Corrected Total	1736,741	26			

Multiple Comparisons

Dependent Variable: Yield

	(I) DE	(J) DE	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Tukey HSD	12	18	8,56*	2,872	,017	1,38	15,73
		19	13,56*	2,872	,000	6,38	20,73
	18	12	-8,56*	2,872	,017	-15,73	-1,38
		19	5,00	2,872	,211	-2,17	12,17
	19	12	-13,56*	2,872	,000	-20,73	-6,38
		18	-5,00	2,872	,211	-12,17	2,17
LSD	12	18	8,56*	2,872	,007	2,63	14,48
		19	13,56*	2,872	,000	7,63	19,48
	18	12	-8,56*	2,872	,007	-14,48	-2,63
		19	5,00	2,872	,095	-,93	10,93
	19	12	-13,56*	2,872	,000	-19,48	-7,63
		18	-5,00	2,872	,095	-10,93	,93

Based on observed means.

*. The mean difference is significant at the ,05 level.

Table B.2. One-way ANOVA and LSD Test for moisture content analysis

Tests of Between-Subjects Effects

Dependent Variable: Moisture_content

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	15,647[a]	8	1,956	34,206	,000
Intercept	495,282	1	495,282	8661,578	,000
Inlet_air_temperature	2,428	2	1,214	21,227	,000
DE	13,076	2	6,538	114,341	,000
Inlet_air_temperature * DE	,144	4	,036	,628	,649
Error	1,029	18	,057		
Total	511,959	27			
Corrected Total	16,677	26			

Tests of Between-Subjects Effects

Dependent Variable: Moisture_content

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	13,879[a]	8	1,735	11,164	,000
Intercept	495,282	1	495,282	3186,990	,000
DE	13,076	2	6,538	42,071	,000
Maltodextrin_ concentration	,740	2	,370	2,382	,121
DE * Maltodextrin_ concentration	,063	4	,016	,101	,981
Error	2,797	18	,155		
Total	511,959	27			
Corrected Total	16,677	26			

Tests of Between-Subjects Effects

Dependent Variable: Moisture_content

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	15,647[a]	8	1,956	34,206	,000
Intercept	495,282	1	495,282	8661,578	,000
Inlet_air_temperature	2,428	2	1,214	21,227	,000
DE	13,076	2	6,538	114,341	,000
Inlet_air_temperature * DE	,144	4	,036	,628	,649
Error	1,029	18	,057		
Total	511,959	27			
Corrected Total	16,677	26			

Table B.3. One-way ANOVA and LSD Test for bulk density analysis

Tests of Between-Subjects Effects

Dependent Variable: Bulk_density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,014[a]	8	,002	3,856	,008
Intercept	1,667	1	1,667	3622,968	,000
Inlet_air_temperature	,003	2	,002	3,705	,045
DE	,011	2	,005	11,492	,001
Inlet_air_temperature * DE	,000	4	5,24E-005	,114	,976
Error	,008	18	,000		
Total	1,689	27			
Corrected Total	,022	26			

Multiple Comparisons

Dependent Variable: Bulk_density

	(I) Inlet_air_temperature	(J) Inlet_air_temperature	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Tukey HSD	110	120	,0001	,01011	1,000	-,0257	,0259
		130	,0239	,01011	,072	-,0019	,0497
	120	110	-,0001	,01011	1,000	-,0259	,0257
		130	,0238	,01011	,074	-,0020	,0496
	130	110	-,0239	,01011	,072	-,0497	,0019
		120	-,0238	,01011	,074	-,0496	,0020
LSD	110	120	,0001	,01011	,991	-,0211	,0214
		130	,0239*	,01011	,030	,0026	,0451
	120	110	-,0001	,01011	,991	-,0214	,0211
		130	,0238*	,01011	,030	,0025	,0450
	130	110	-,0239*	,01011	,030	-,0451	-,0026
		120	-,0238*	,01011	,030	-,0450	-,0025

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Bulk_density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,017[a]	8	,002	6,951	,000
Intercept	1,667	1	1,667	5459,508	,000
DE	,011	2	,005	17,318	,000
Maltodextrin_ concentration	,005	2	,003	8,463	,003
DE * Maltodextrin_ concentration	,001	4	,000	1,013	,427
Error	,005	18	,000		
Total	1,689	27			
Corrected Total	,022	26			

Tests of Between-Subjects Effects

Dependent Variable: Bulk_density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,017[a]	8	,002	6,951	,000
Intercept	1,667	1	1,667	5459,508	,000
DE	,011	2	,005	17,318	,000
Maltodextrin_ concentration	,005	2	,003	8,463	,003
DE * Maltodextrin_ concentration	,001	4	,000	1,013	,427
Error	,005	18	,000		
Total	1,689	27			
Corrected Total	,022	26			

Multiple Comparisons

Dependent Variable: Bulk_density

	(I) DE	(J) DE	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	12	18	-,0273*	,01011	,037	-,0531	-,0015
		19	-,0483*	,01011	,000	-,0741	-,0225
	18	12	,0273*	,01011	,037	,0015	,0531
		19	-,0210	,01011	,123	-,0468	,0048
	19	12	,0483*	,01011	,000	,0225	,0741
		18	,0210	,01011	,123	-,0048	,0468
LSD	12	18	-,0273*	,01011	,015	-,0486	-,0061
		19	-,0483*	,01011	,000	-,0696	-,0271
	18	12	,0273*	,01011	,015	,0061	,0486
		19	-,0210	,01011	,052	-,0422	,0002
	19	12	,0483*	,01011	,000	,0271	,0696
		18	,0210	,01011	,052	-,0002	,0422

Based on observed means.

*. The mean difference is significant at the ,05 level.

Table B.4. One-way ANOVA and LSD Test for hygroscopicity analysis

Tests of Between-Subjects Effects

Dependent Variable: Hyroscopicity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,857[a]	8	,107	35,280	,000
Intercept	6,059	1	6,059	1994,928	,000
Inlet_air_temperature	,070	2	,035	11,551	,001
DE	,776	2	,388	127,772	,000
Inlet_air_temperature * DE	,011	4	,003	,898	,486
Error	,055	18	,003		
Total	6,970	27			
Corrected Total	,912	26			

Multiple Comparisons

Dependent Variable: Hyroscopicity

	(I) Inlet_air_temperature	(J) Inlet_air_temperature	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	110	120	,0711*	,02598	,034	,0048	,1374
		130	,1244*	,02598	,000	,0581	,1907
	120	110	-,0711*	,02598	,034	-,1374	-,0048
		130	,0533	,02598	,128	-,0130	,1196
	130	110	-,1244*	,02598	,000	-,1907	-,0581
		120	-,0533	,02598	,128	-,1196	,0130
LSD	110	120	,0711*	,02598	,014	,0165	,1257
		130	,1244*	,02598	,000	,0699	,1790
	120	110	-,0711*	,02598	,014	-,1257	-,0165
		130	,0533	,02598	,055	-,0012	,1079
	130	110	-,1244*	,02598	,000	-,1790	-,0699
		120	-,0533	,02598	,055	-,1079	,0012

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Hyroscopicity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,822[a]	8	,103	20,546	,000
Intercept	6,059	1	6,059	1211,734	,000
Maltodextrin_ concentration	,042	2	,021	4,199	,032
DE	,776	2	,388	77,610	,000
Maltodextrin_ concentration * DE	,004	4	,001	,187	,942
Error	,090	18	,005		
Total	6,970	27			
Corrected Total	,912	26			

Multiple Comparisons

Dependent Variable: Hyroscopicity

	(I) Maltodextrin_ concentration	(J) Maltodextrin_ concentration	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	10	15	,0600	,03333	,198	-,0251	,1451
		20	,0956*	,03333	,026	,0105	,1806
	15	10	-,0600	,03333	,198	-,1451	,0251
		20	,0356	,03333	,546	-,0495	,1206
	20	10	-,0956*	,03333	,026	-,1806	-,0105
		15	-,0356	,03333	,546	-,1206	,0495
LSD	10	15	,0600	,03333	,089	-,0100	,1300
		20	,0956*	,03333	,010	,0255	,1656
	15	10	-,0600	,03333	,089	-,1300	,0100
		20	,0356	,03333	,300	-,0345	,1056
	20	10	-,0956*	,03333	,010	-,1656	-,0255
		15	-,0356	,03333	,300	-,1056	,0345

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Hyroscopicity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,857[a]	8	,107	35,280	,000
Intercept	6,059	1	6,059	1994,928	,000
Inlet_air_temperature	,070	2	,035	11,551	,001
DE	,776	2	,388	127,772	,000
Inlet_air_temperature * DE	,011	4	,003	,898	,486
Error	,055	18	,003		
Total	6,970	27			
Corrected Total	,912	26			

Table B.5. One-way ANOVA and LSD Test for acidity analysis

Tests of Between-Subjects Effects

Dependent Variable: Acidity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1,372[a]	2	,686	1,731	,199
Intercept	94,566	1	94,566	238,535	,000
Inlet_air_temperature	1,372	2	,686	1,731	,199
Error	9,515	24	,396		
Total	105,453	27			
Corrected Total	10,887	26			

Tests of Between-Subjects Effects

Dependent Variable: Acidity

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	,242[a]	8	,030	8,217	,000
Intercept	2,670	1	2,670	726,614	,000
DE	,205	2	,102	27,874	,000
Maltodextrin_ concentration	,028	2	,014	3,832	,041
DE * Maltodextrin_ concentration	,009	4	,002	,582	,679
Error	,066	18	,004		
Total	2,977	27			
Corrected Total	,308	26			

Table B.6. One-way ANOVA and LSD Test for solubility analysis

Tests of Between-Subjects Effects

Dependent Variable: Solubility

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1262,296[a]	8	157,787	46,307	,000
Intercept	25453,370	1	25453,370	7470,011	,000
DE	968,074	2	484,037	142,054	,000
Inlet_air_temprature	269,852	2	134,926	39,598	,000
DE * Inlet_air_temprature	24,370	4	6,093	1,788	,175
Error	61,333	18	3,407		
Total	26777,000	27			
Corrected Total	1323,630	26			

Multiple Comparisons

Dependent Variable: Solubility

	(I) DE	(J) DE	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	12	18	-7,44*	,870	,000	-9,67	-5,22
		19	-14,67*	,870	,000	-16,89	-12,45
	18	12	7,44*	,870	,000	5,22	9,67
		19	-7,22*	,870	,000	-9,44	-5,00
	19	12	14,67*	,870	,000	12,45	16,89
		18	7,22*	,870	,000	5,00	9,44
LSD	12	18	-7,44*	,870	,000	-9,27	-5,62
		19	-14,67*	,870	,000	-16,49	-12,84
	18	12	7,44*	,870	,000	5,62	9,27
		19	-7,22*	,870	,000	-9,05	-5,39
	19	12	14,67*	,870	,000	12,84	16,49
		18	7,22*	,870	,000	5,39	9,05

Based on observed means.

*. The mean difference is significant at the ,05 level.

Tests of Between-Subjects Effects

Dependent Variable: Solubility

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1012,296[a]	8	126,537	7,316	,000
Intercept	25453,370	1	25453,370	1471,608	,000
Maltodextrin_ concentration	41,407	2	20,704	1,197	,325
DE	968,074	2	484,037	27,985	,000
Maltodextrin_ concentration * DE	2,815	4	,704	,041	,997
Error	311,333	18	17,296		
Total	26777,000	27			
Corrected Total	1323,630	26			