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**Addis Ababa University**  
**Addis Ababa Institute of Technology**  
**School of Chemical and Bio Engineering**



**Evaluation of the Effect of the Operational Parameter on Unripe  
Banana (*Musa Cavendishii*) Flour Production Process**

**By**

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**A Thesis Submitted To the School of Chemical and Bio Engineering,  
Addis Ababa Institute of Technology in Partial Fulfillment of the Requirements for the  
Degrees of Masters of Science in Chemical Engineering (Process Engineering Stream)**

**Addis Ababa University  
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Addis Ababa University  
Addis Ababa Institute of Technology  
School of Chemical and Bio Engineering

This is to certify that the thesis prepared by Firew Deneke, entitled “*Evaluation of the effect of the operational parameter on Unripe Banana (Musa cavendishii) Flour Production Process*”. submitted in partial fulfillment of the requirements for the degree of Masters of Science (Chemical and Bio Engineering, Process stream) complies with the regulation of the university and meets the accepted standards with respect to originality and quality.

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## DECLARATION

I declare that this thesis entitled “*Evaluation of the effect of the operational parameter on Unripe Banana (Musa cavendishii) Flour Production Process*” has not been submitted in any form for another degree, diploma or an award at any university or other institution of the tertiary education. Whenever contributions of others are involved, every effort is made to indicate this clearly, with due reference to the literature and discussions. Information taken from published and unpublished work of others has been acknowledged in the text and a list of references is given.

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## ABSTRACT

*Unripe Banana flour production is an excellent alternative food supply and to minimize postharvest losses and to retain the nutritive value of fresh bananas and stable markets. Unripe banana flour is rich in resistant starch, dietary fiber, and aids in colon health and contains a high amount of iron, calcium, potassium, and reducing sugars, which helps in better blood circulation. The main objective of the research is to examine effect of the three types of pretreatment techniques in production flour from unripe banana and analyze quality characteristics of the flour. The drying process was carried out for unripe banana after giving different pre-treatments such as optimal osmotic solution variables, sodium metabisulfite (SMS), and hot water (at 100 °C). Untreated samples served as control. Treated and untreated samples were oven dried with 45, 60, and 75 °C a period for 24hr. The dried slice banana was milled into a powder. For the study of optimum osmotic parameters, used design-Expert 7.0.0 three-level-full factorial RSM was applied for experimental design and statistical analysis of results. The optimum conditions for maximum water loss and minimum solid gain percentage were obtained at 30 °Brix sugar concentration, 60 °C soaking temperature and 90 min soaking time, which resulted give 29.16% water loss and 5.66% solid gain.*

**Keywords:** *Unripe banana flour; Water loss; Solid gain; Osmotic dehydration*

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## TABLE OF CONTENT

|   |      |
|---|------|
| ACKNOWLEDGEMENTS.....   | i    |
| ABSTRACT.....   | ii   |
| LIST OF TABLES.....   | vii  |
| LIST OF FIGURE.....   | viii |
| ACRONYMS.....   | x    |
| CHAPTER ONE.....  | 1    |
| 1. INTRODUCTION.....  | 1    |
| 1.2 Background.....   | 1    |
| 1.1 Statement of the problem.....   | 3    |
| 1.2 Objective of the study.....   | 4    |
| 1.2.1 General objective.....  | 4    |
| 1.2.2 Specific Objective.....   | 4    |
| 1.3 Significance of the Study.....  | 4    |
| 1.4 Scope of the Study.....   | 5    |
| CHAPTER TWO.....  | 6    |
| 2. LITERATURE REVIEW.....   | 6    |
| 2.1 Banana ( <i>Musa cavendish</i> ) in Ethiopia.....                           | 6    |
| 2.2 Banana ( <i>Musa cavendishii</i> ) Plantation and post harvesting loss..... | 7    |
| 2.3 Fresh fruits and vegetables handling considerations.....                    | 9    |
| 2.4 Pretreatments method for fruits and vegetables before drying.....           | 9    |
| 2.5 General considerations for preservation of fruits and vegetables.....       | 13   |
| 2.5.1 Water Activity ( $a_w$ ) concept and its role in food preservation.....   | 13   |
| 2.5.2 Recommended substances to reduce water activity $a_w$ in fruits.....      | 15   |

---

|                    |  |    |
|--------------------|--|----|
| 2.5.3              | Recommended substances to reduce pH.....                                     | 15 |
| 1.5.4              | Recommended chemicals to prevent browning.....                               | 16 |
| 2.6                | Drying Technology Prolong Shelf Life Fruit and Vegetable.....                | 17 |
| 2.7                | Processing Technology Production of Unripe Banana Flour.....                 | 18 |
| 2.8                | Osmosis dehydration.....   | 19 |
| 2.8.1              | Factors affecting osmotic dehydration process.....                           | 20 |
| 2.9                | Use of banana flour.....   | 22 |
| CHAPTER THREE..... |  | 24 |
| 3.                 | MATERIALS AND METHODS.....   | 24 |
| 3.1                | Reagents and Equipment.....  | 24 |
| 3.1.1              | List of Reagent and chemicals.....   | 24 |
| 3.1.2              | List of instrument and apparatus.....  | 24 |
| 3.2                | Raw Material Collection, Transportation, Sample Preparation and Storage..... | 25 |
| 3.2.1              | Raw material collection.....   | 25 |
| 3.1.1              | Preparation of green banana flour by different pretreatments.....            | 26 |
| 3.1.2              | Preparation of Osmotic dehydrated banana flour.....                          | 27 |
| 3.1.3              | Sodium metabisulfite and hot water pre treatment.....                        | 28 |
| 3.3                | Experimental Procedures.....   | 29 |
| 3.3.1              | Determination of moisture content.....                                       | 29 |
| 3.3.2              | Determination of ash content.....  | 30 |
| 3.3.3              | Determination of crude fat content Fat.....                                  | 30 |
| 3.3.4              | Determination of crude Protein.....  | 31 |
| 3.3.5              | Determination of Crude fiber determination.....                              | 32 |
| 3.3.6              | Determination of carbohydrates.....  | 32 |
| 3.3.7              | Determination of gross energy.....   | 33 |

---

|                   |   |    |
|-------------------|---|----|
| 3.4               | Physicochemical Analysis.....   | 33 |
| 3.4.1             | Water holding capacity (WHC) and oil holding capacity (OHC).....  | 33 |
| 3.1.4             | Non-enzymatic browning (NEB).....   | 34 |
| 3.4.2             | Total soluble solids (TSS).....   | 34 |
| 3.4.3             | pH of sample.....   | 34 |
| 3.4.4             | Determination of minerals .....   | 34 |
| 3.4.5             | Water loss (WL) and Solid gain (SG).....  | 36 |
| 3.5               | Experimental design and Statistical Analysis .....  | 37 |
| CHAPTER FOUR..... |   | 39 |
| 4.                | RESULTS AND DISCUSSION.....   | 39 |
| 4.1               | Analysis of response surface methodology on osmotic dehydration of unripe banana sliced pulp .....                    | 39 |
| 4.1.1             | Model Adequacy.....   | 42 |
| 4.1.2             | Residual plots for water loss.....  | 45 |
| 4.1.3             | Residual plots for solid gain .....   | 46 |
| 4.1.4             | Effect of Single Factor Variables on osmotic dehydration banana slice .....   | 47 |
| 4.1.5             | Effect of Interaction between Process Variables .....   | 47 |
| 4.1.6             | Development of Regression Model Equation.....   | 52 |
| 4.1.7             | Optimization of process variables.....  | 53 |
| 4.2               | Effects of pre treatment techniques and conventional oven drying temperature on characterization of banana flour..... | 53 |
| 4.2.1             | Effect of treatments and drying temperature on banana flour quality .....   | 54 |
| 4.2.3             | Effect of pre-treating and drying temperature on total dissolving solid (TDS) & pH of banana flour.....               | 57 |
| 4.2.4             | Effect of pre-treating and drying temperature on non enzymatic browning in banana flour .....                         | 58 |

---

|             |  |    |
|-------------|--|----|
| 4.2.5       | Composition of minerals in pretreated and untreated banana flour ..... | 58 |
| CHAPTER 5   | .....  | 61 |
| 5.          | CONCLUSIONS AND RECOMMENDATIONS .....                                  | 61 |
| 5.1         | Conclusions .....  | 61 |
| 5.2         | Recommendation.....  | 62 |
| REFERENCE   | .....  | 63 |
| APPENDIX A: | Anova results from design- expert .....                                | 70 |
| APPENDIX B: | Single factor effect .....   | 72 |
| APPENDIX C: | P-Value (ANOVA) for proximate value ANOVA .....                        | 75 |
| APPENDIX D: | P-Value (ANOVA) for Physico-Chemical test .....                        | 76 |
| APPENDIX E: | Active metals laboratory result.....                                   | 77 |
| APPENDIX F: | Pictures while conducting product preparation.....                     | 77 |

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## LIST OF TABLES

|   |    |
|---|----|
| Table 2.1: Banana varieties, consumption category and yield.....  | 6  |
| Table 3.1: List of equipment and instruments .....  | 24 |
| Table 3.2 Coded and un-coded values of variables and their level .....                                    | 38 |
| Table 4.1: Designed experiments according to full factorial design and measured .....                     | 39 |
| Table 4.2A: Design summary for factors.....   | 40 |
| Table 4.3: Water loss Model Summary Statistics.....   | 41 |
| Table 4.4: Solid Gain Model Summary Statistics.....   | 41 |
| Table 4.5: Analysis of variance (ANOVA) on Response 1(water loss) .....                                   | 42 |
| Table 4.6: Analysis' of variance (ANOVA) on response 2 (Solid Gain) .....                                 | 43 |
| Table 4.7: Verification code .....  | 44 |
| Table 4.8: Effect of pre-treatment and drying temperature on chemical composition of banana flour.....    | 54 |
| Table 4.9: Effect of pretreatments the concentration of Zn, Fe, Cd, Pd and Cr in unripe banana flour..... | 60 |

---

## LIST OF FIGURE

|  |    |
|--|----|
| Figure 2.1: Major banana production site and market .....  | 7  |
| Figure 2.2: Ethiopia Fruit Production From 1997-2005 .....   | 8  |
| Figure 2.3: Pretreatment methods of fruits or vegetables prior to thermal drying. ....   | 10 |
| Figure 2.4: Osmotic dehydration process advantage and disadvantage .....   | 13 |
| Figure 2.5: Types of drying method .....   | 18 |
| Figure 2.6: Unripe banana flour production process .....   | 19 |
| Figure 2.7: Osmotic dehydration process .....  | 20 |
| Figure 3.1: Fresh Unripe banana a) Stage of banana ripping b) Banana with bunch and c) Banana after bunch removed .....  | 25 |
| Figure 3.2: Framework of experiment set up.....  | 27 |
| Figure 3.3: SMBS & Hot water pretreated banana slice and flour . a) hot water pretreated banana slices, b) hot water pretreated banana flour, c) SMBS pretreated banana slices and d) SMBS pretreated banana flour ..... | 29 |
| Figure 3.4: Crude fat laboratory extraction a) Soxhlet-extraction and b) Rotary evaporator.....  | 31 |
| Figure 3.5: Uv- Spectroscopy NEB absorbance reading .....  | 34 |
| Figure 3.6: Unripe banana flour mineral composition reading a) GFASS and b) AAS .....  | 36 |
| Figure 4.1: Diagnostic plot for water loss, a) normal plot of residues, (b) Residual Vs Predicted, c) Residual Vs run.....   | 46 |
| Figure 4.2: Diagnostic plot for solid gain response, a) normal plot of residues, (c) residual Vs predicted, c) residual Vs run .....   | 47 |
| Figure 4.3: Interaction effect soaking temperature and sugar concentration versus WL when the soaking time at 60 min. a) interaction plot & b) contour plot.....   | 48 |
| Figure 4.4 : interaction effect soaking temperature and sugar concentration versus solid gain when the soaking time at 60 min. a) interaction plot & b) Contour plot .....   | 49 |
| Figure 4.5: Contour plot of the interaction effect soaking temperature and time versus WL when the sugar concentration at 40 Brix .....  | 49 |
| Figure 4.6 Contour plot of the interaction effect soaking temperature and time versus solid gain when the sugar concentration at 40 <sup>0</sup> Brix. a) Interaction plot & b) contour plot.....                        | 50 |

---

|  |    |
|--|----|
| Figure 4.7: interaction plot of the interaction effect sugar concentration and soaking time versus WL when the sugar temperature at 40 Brix. a) Interaction plot & b) contour plot ..... | 51 |
| Figure 4.8 3D-surface of the interaction effect of sugar concentration and soaking time versus WL when the soaking temperature at 50 <sup>0</sup> C .....                                | 51 |
| Figure 4.9: 3D-surface of the interaction effect of sugar concentration and soaking time versus SG when the soaking temperature at 50 <sup>0</sup> C .....                               | 52 |
| Figure 4.10: Energy value pretreated and untreated banana flour at different temperature .....   | 55 |
| Figure 4.11: Water and oil holding capacity pretreated and untreated unripe banana flour.....  | 56 |
| Figure 4.12: Effect of pretreatment on TDS and pH unripe banana flour .....  | 57 |
| Figure 4.13: Effect pretreatment on non-enzymatic browning unripe banana flour.....  | 58 |
| Figure 4.14: Effect of pretreatments the concentration of Ca, Mg, Na and K in unripe banana flour.....   | 59 |

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## ACRONYMS

|       |   |
|-------|---|
| AAS   | Atomic Absorptive Spectrometry                  |
| ANOVA | Analysis of variance                            |
| CSA   | Central Statistical authority                   |
| CFC   | Common Fund for Commodities                     |
| FAO   | Food and agricultural organization              |
| FDA   | Federal drug administration                     |
| GBF   | Green banana flour                              |
| GFAAS | Graphite furnace Atomic Absorptive Spectrometry |
| HHB   | Hot water bath                                  |
| NEB   | Non-enzymatic browning                          |
| OD    | Osmotic dehydration                             |
| OHC   | Oil holding capacity                            |
| OS    | Osmotic solution                                |
| RSM   | Response surface methodology                    |
| SG    | Solid gain                                      |
| SMS   | Sodium Meta bisulphate                          |
| TSS   | Total soluble solid                             |
| UBF   | Unripe banana flour                             |
| WHC   | Water holding capacity                          |
| WL    | Water loss                                      |

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## CHAPTER ONE

### 1. INTRODUCTION

#### 1.2 Background

Banana is one of the commodities that have a very high chance for diversification, secure food security and agro processing industry in Ethiopia. Because of banana potential source of carbohydrates, minerals and fiber content are highly qualified as food commodities. According to Choo & Aziz (2010) studies, mature green bananas are very rich in starch, which is resistant to amylase and glycol-amylase due to its high degree of intrinsic crystalline structure.

As stated by FAO (2008), the world annual banana production reached 100 million metric tons and increasing every year. As fruit market analysis, banana production takes place the first position in the world. Banana in Ethiopia covers about 59.64% (53,956.16 hectares) of the total fruit area, about 68.00% (478,251.04 tones) of the total fruits produced, and about 38.30%(2,574,035) of the total fruit producing farmers ( CSA, 2015). Gamo-Gofa, Bench-Maji and Sheka zones are among the major banana production zones of the SNNPRS, of which Gamo-Gofa zone alone covers over 70% of the total banana marketed across the major market outlets in Ethiopia (CFC, 2004 & Zenebe et al., 2015).

According to FAO (2008) report, postharvest loss of perishable commodities in the world were reached 50% and this loss in Ethiopia reported from 26.5% (Muluaem et al., 2015) to 30-40% (Dawit Alemu et al., 2008). Post harvest loss tends to face against the production and the business of banana in the country.

Those loss including

- ✓ Poor postharvest handling,
- ✓ Its physical and chemical characteristics,
- ✓ Middle man or brokers,
- ✓ Price fluctuation due to seasonal high and low production,

Recently, Ethiopian struggle to transform from the agriculture sector to the manufacturing sector (agro processing) and secure their people food stability. So, secure food security and enhance the agro processing sector banana fruit has a high potential to process into banana flour, banana juice, banana wine, banana chips, banana sauce, and banana jam and other value-added products because of their high resource amount and nutrient values. On other hand, most of the people in

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Ethiopia consumed banana as additional or dessert food but it is possible to use banana as primary food and enhance staple food supply alternative. One of the aims of this research change this scenario to transfer banana as primary foods or staple foods like maize, teff, wheat etc..

The diversification of food were considered the most rational way to solve the problem of meeting the needs of food especially carbohydrates. Banana flour is used as best ingredient for the food industry as an alternative to enhance fiber content and minimize banana wastage. The UBF can be used as an ingredient in other food formulations such as porridge, soups, for milk enrichment, in ice creams and other foods. It can also be used as in bread making, where it is mixed in the proportion of 1/3 of banana flour to 2/3 of wheat flour (Cabrerapadilla, 2003).

Banana processing is a value-adding step that fills the gap between farm production and marketing. The objective of processing is to convert highly perishable to more stable forms.

This research work gives special concern on how banana goes to more value-added product such as banana flour. Banana flour is generally produced with green bananas that are peeled, sliced, dried, and then ground into flour (Ovando-Martinez et al., 2009). UBF production has long history, people in different countries use traditionally banana flour as an alternative to high priced wheat flour in various parts of Africa (Uganda, Kenya etc..) and Jamaica (Coglan, Lea, 2014).

Banana has non-digestible carbohydrates, high starch (resistant starch), dietary fiber and polyphenol content in its unripe form makes the fruit a suitable material for the production of flour with great application in glycemic index reduction, diabetes and colon cancer prevention (Anyasi, Jideani, & Mchau, 2013). Various methods have been employed in the development of unripe banana flour. One of such methods is the use of organic acid, sodium metabisulphite, hot/boiling water and osmotic dehydration as pretreatment in the prevention of enzymatic browning, biochemical and microbial activities that occur during flour production. This research used sodium metabisulphite, boiling water and osmotic dehydration as pretreatments prior to drying.

The objective of this study therefore was to determine the effect of sodium metabisulphite (0.2w/v), boiling water and osmotic solution (sugar solute) pretreatment on the physical and proximate properties of unripe banana flour and also determine the effect of conventional oven drying temperature on quality of banana flour.

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## 1.1 Statement of the problem

Fruits, vegetables and their products in the dried form are good sources of energy, minerals and vitamins. However, during the process of dehydration, there are changes in nutritional quality (Sablani, 2006). Banana is a fast growing and high biomass-yielding crop in the tropical and sub-tropical regions in the worlds. Banana plays an important role for human's diet. The nutrient, which makes it rich, is fibers, carbohydrates, vitamins, minerals, and antioxidant. For their use throughout the year which is beneficial to health. Due to seasonality, poor harvest techniques and high perish ability makes their use in fresh can be possible only after a demanding and expensive storage.

Postharvest losses of fruits and vegetables are estimated 5-20% in developed countries and 20-50 % in developing countries (Mashav, 2010). In Ethiopian banana postharvest loss are estimated 26.5% (Mulualem et al., 2015). This loss due to the physical and the chemical nature of banana such as, perish ability nature, long distance overloaded transportation and traditional kerosene gas smoking ripping techniques. Therefore, in order to prolong the shelf life of the postharvest product, processing is necessary. To minimize the losses and to provide sustainable benefits to the growers and the community, the production of unripe banana flour is an excellent option.

One of the major preservation methods for any fruit and vegetable is drying, which is accomplished either traditional sun drying, industrial dryers or mechanical dehydrators. However, various pretreatments needed prior to drying, like blanching (boiling water), chemical treatments; sodium metabisulphite, citric acid, calcium chloride, ascorbic acid, and osmotic dehydration for obtaining better quality characteristics. Recently, various pretreatment methods have been employing in the development of unripe banana flour. One of such methods is the use of organic acid, sodium metabisulphit, boiling water and osmotic dehydration before drying as pretreatment in the prevention of enzymatic browning, biochemical and microbial activities that occur during flour production. In sum, proper pretreatments can reduce the initial water content, or modify the properties of tissue in some extent, thereby increasing the drying rate, improving the quality of material; and inhibit the bio-enzymes, then minimize possible deterioration reactions during drying and subsequent storage.

Therefore, it is required to determine the effect of operational parameter such as pre-treatments type, pre-treatments concentration, pre-treatments contact time, and drying temperature on

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banana flour production. The present studies focus to solve the problem, selection of appropriate pre-treatments and drying temperature to determine quality flour from unripe banana.

## **1.2 Objective of the study**

### **1.2.1 General objective**

The general objective of the project is to study the effect of process conditions on unripe Banana (**Musa cavendishii**) flour production process

### **1.2.2 Specific Objective**

Specific objectives of the study are:

- To study and find the optimal processing osmotic dehydration variables (osmotic solute concentration, soaking temperature and soaking time )
- To study the effect of hot water bathing and Sodium metabisulfite pretreatments
- To investigate drying (oven drying) temperature range on the physicochemical properties of banana flour
- To analyze the physicochemical and proximate characteristics of unripe banana flour

## **1.3 Significance of the Study**

The water content of most of fruits and vegetables is higher than 60%, which limits their shelf life and makes them more susceptible to storage and transport conditions.

Hence, this study should be significance in the sense that it will:

- Provide technically and economically feasible option for banana flour production
- Introduce alternative food supply and market
- Add knowledge and understanding selection of appropriate pre-treatments method and drying temperature during production of unripe banana flour
- Create an understanding of the process that is simple to practice and socially acceptable
- Reduced post-harvesting loss
- Simplify banana peel waste collection

Additional, studies give the consumers a product which is affordable with health benefits; typically gluten-free alternative to wheat-based flours for those suffering from celiac disease and those who choose a gluten-free diet ( Crofts. N & Coghlan. L, 2014).

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#### **1.4 Scope of the Study**

As the title of the proposals, the project gives special emphasis to develop unripe banana flour production processes using different pretreatments. Through this study, optimize osmotic dehydration operational variables: soaking temperature, soaking time and osmotic solute concentration. The effect of optimize osmotic solution variable, sodium metabisulfite solution and hot water pretreatments on banana flour was studied. The characterization of the untreated and pretreated unripe banana flour proximate, mineral composition and functional values was analyzed.

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## CHAPTER TWO

### 2. LITERATURE REVIEW

#### 2.1 Banana ( *Musa cavendish* ) in Ethiopia

African banana are grouped into three categories, including East African (mainly dessert) bananas, the African plantain banana grown mainly in Central and West Africa, and the East African Highland banana, used for cooking and beer preparation (De Vries, 2001). According to FAO (2002), total fruit production in Ethiopia was estimated at about 320,000 tonnes. Banana export from Ethiopia (including Eritrea) started at less than 5,000 tonnes in 1961 but jumped to 60,000 tonnes in 1972 and was exported to many countries in Europe, Asia and Africa (Taye, 1975).

In Ethiopia, even though both dessert and cooking types/varieties of banana are released by the research system, the types of varieties that are under production are dessert type that has been under production since the early 1970s (Dawit.A&Asmare. D, 2008). Ducasse hybrid is starchy type and is not preferred as dessert, but other African countries use it for brewing ( Dawit. A & Asmare. D, 2008). Showed Table 2.1 summarizes the banana varieties that have been released by the Ethiopian Institute of Agricultural Research (EIAR).

Table 2.1: Banana varieties, consumption category and yield

| Name of variety | Consumption category (type) | Potential yield(q/ha) | Average height(m) | Year of release |
|-----------------|-----------------------------|-----------------------|-------------------|-----------------|
| Williams-1      | Dessert                     | 556                   | 2.90              | 2006            |
| Grand Nain      | Dessert                     | 436                   | 3.00              | 2006            |
| Robusta         | Dessert                     | 395                   | 3.60              | 2006            |
| Butuzua         | Dessert                     | 391                   | 3.60              | 2006            |
| Cardaba         | Cooking                     | 480                   | 4.09              | 2006            |
| Kitawira        | Cooking                     | 463                   | 4.00              | 2006            |
| Nijiru          | Cooking                     | 482                   | 3.31              | 2006            |
| Matoke          | Cooking                     | 421                   | 3.26              | 2006            |
| Poya            | Dessert                     | 482                   | 3.47              | 1970            |

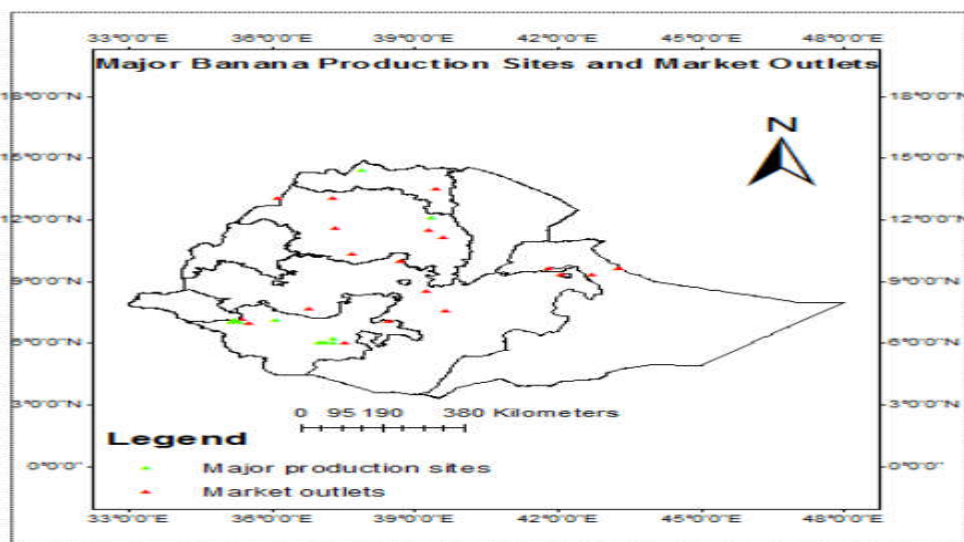
|                 |         |     |      |      |
|-----------------|---------|-----|------|------|
| Giant Cavendish | Dessert | 372 | 3.40 | 1970 |
| Dwarf Cavendish | Dessert | 531 | 2.21 | 1970 |
| Ducasse Hybrid  | Dessert | 261 | 4.60 | 1970 |

Source: MoARD, 2006

## 2.2 Banana (*Musa cavendishii*) Plantation and post harvesting loss

In 2010, world commerce in banana was valued at US 8.05 billion and the total world production of banana is about 106,541,709.00 tons (FAOSTAT, 2012). For many African, Asian and Latin American countries, banana is as well one of the most important crops for foreign exchange earnings. Banana in Ethiopia covers about 59.64% (53,956.16 hectares) of the total fruit area, produced 68.00% (478,251.04 tones) of the total fruit, and about 38.30 % ( 2,574,035) of the total fruit producing farmers (CSA, 2014). On the other hand, about 68.72% (37,076.85 hectares) hectares of land covered by banana, about 77.53% (370,784.17 tones) of the banana produced and 22.38% (1,504,207) of the banana producers in Ethiopia are found in the Southern Nations Nationalities and Peoples' National Regional state ( SNNPR) (CSA, 2014).

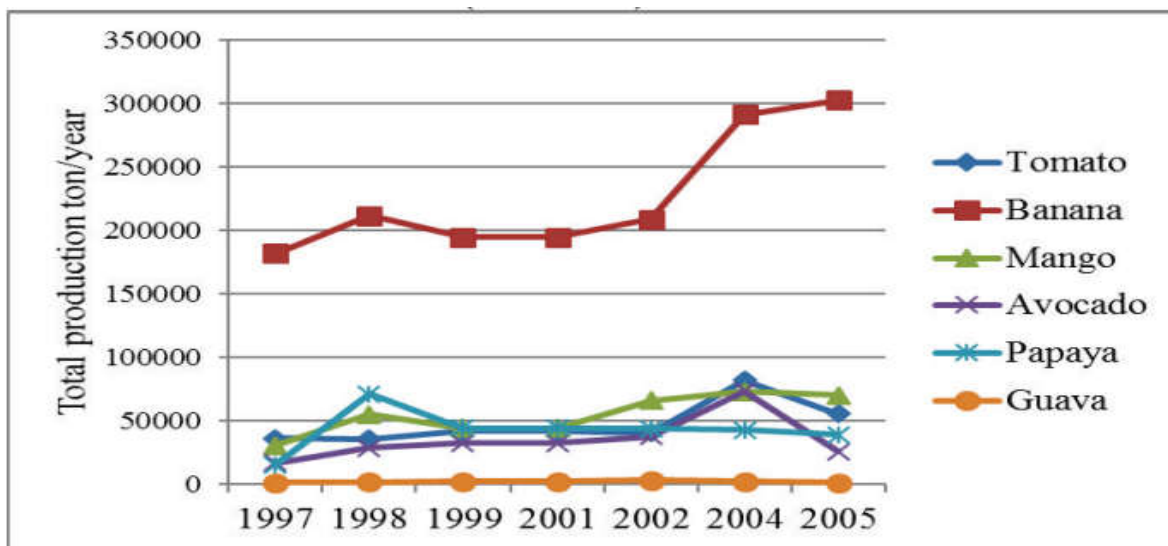
Gamo-Gofa, Bench-Maji and Sheka zones are among the major banana producing zones of the SNNPRS, of which Gamo-Gofa zone alone covers over 70% of the total banana marketed across the major market outlets in Ethiopia (CFC, 2004). Show figure below to Summarizes the Major banana production areas and market outlets.



Source (Zeneb et al., 2015).

Figure 2.1: Major banana production site and market

Postharvest losses of fruits and vegetables are estimated 5-20% in developed countries and 20-50 % in developing countries (Mashav, 2010). The country's agricultural potential for food production is known to be immense and over 90 percent of its export earnings come from this sector. Coffee, oil seeds, spices, fresh fruit and vegetables contribute the largest portion of the export earnings. From 39.7 million tonnes of total crops produced in Ethiopia, 23.1 million tonnes are not highly perishable whereas 6.6 million tonnes are highly perishable. From the highly perishable 0.5 million tonness are tropical fruits including Tomato, Banana, Mango, Papaya, Avocado, Guava and Pineapple ( CSA, 2012). Show in the Figure below to summery tropical fruit production post (Tomato, Banana, Mango, Avocado, Papaya and Guava) in Ethiopia



Source ( muluken et al,2017)

Figure 2.2: Ethiopia Fruit Production From 1997-2005

Postharvest loss of fresh fruits and vegetables in Ethiopia and developing counties is a major challenge in the postharvest sector. Fruits are the most perishable agricultural produces facing a extremely loss from harvest to consumption. The total postharvest loss of banana was estimated to be 26.5% where 56% of the loss occurred at the retail level, while 27% and 17% of the losses occurred at wholesale and farm levels, respectively in Ethiopia ( Mulualem et al.,2015). Mechanical damage was identified as the main cause for postharvest banana loss at farm and

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wholesale levels while rotting was the main cause at retail level. Poor postharvest handling practices from farm to the retail were the major factors influencing banana loss in the supply chain ( Mulualem et al.,2015).

## **2.3 Fresh fruits and vegetables handling considerations**

### **2.3.1 Maturity index for fruits and vegetables**

The principles dictating at which stage of maturity a fruit or vegetable should be harvested are crucial to its subsequent storage and marketable life and quality. Post-harvest physiologists distinguish three stages in the life span of fruits and vegetables: maturation, ripening, and senescence (FAO, 2003). Maturation is indicative of the fruit being ready for harvest. At this point, the edible part of the fruit or vegetable is fully developed in size, although it may not be ready for immediate consumption. Ripening follows or overlaps maturation, rendering the produce edible, as indicated by taste. Senescence is the last stage, characterized by natural degradation of the fruit or vegetable, as in loss of texture, flavor, etc. (senescence ends at the death of the tissue of the fruit). Some typical maturity indexes are:

- Skin color
- Optical methods
- Shape
- Size
- Aroma
- Fruit opening
- Leaf changes
- Firmness
- Juice content
- Oil content and dry matter percentage
- Moisture content
- Sugars
- Starch content
- Acidity
- Specific gravity

## **2.4 Pretreatments method for fruits and vegetables before drying**

Fruits, vegetables and their products in the dried form are good sources of energy, minerals and vitamins. However, during the process of dehydration, there are changes in nutritional quality (Sablani, 2006). Product quality is becoming more and more important for dehydrated fruits and

vegetables, which must retain quality attributes (color, texture) and nutritional quality after rehydration. Improvement of such qualities can be achieved by pretreatments before drying. Suitable pretreatments can improve the drying process by reducing the drying time, yielding higher-quality products, and energy savings. Steam blanching retains higher amounts of vitamin C in spinach compared with hot-water blanching (Ramesh et al., 2001). Blanching in sulphite solution can retain more ascorbic acid in okra (Inyang and Ike, 1998).

Pretreatments are necessary step in drying process to stop the enzymatic & non-enzymatic browning, microbial growth and effect. Fruits and vegetables are usually subjected to physical or chemical pretreatment before drying to shorten the drying time, reduce the energy consumption and preserve the quality of products (Yu et al., 2017). As show in figure 2.3, Pretreatments before drying can be classified in to two groups: chemical and non-chemical treatments (Physical) treatment in order to ensure the increased rate of water removal and maintain natural quality during the drying process. A faster water removal rate decreases the rate of browning and important to use desirable pretreatments before drying in order to minimize adverse changes occurring during drying and subsequent storage. Pre-treating helps to keep light colored fruits from darkening during drying and storage and it speeds up the drying of fruits with tough skins (wang et., 1996). In addition to that they are improves nutritional, sensorial and functional properties of the dehydrated food without changing its integrity. It also improves the texture as well as stability of the pigment during dehydration and the storage of dehydrated products.

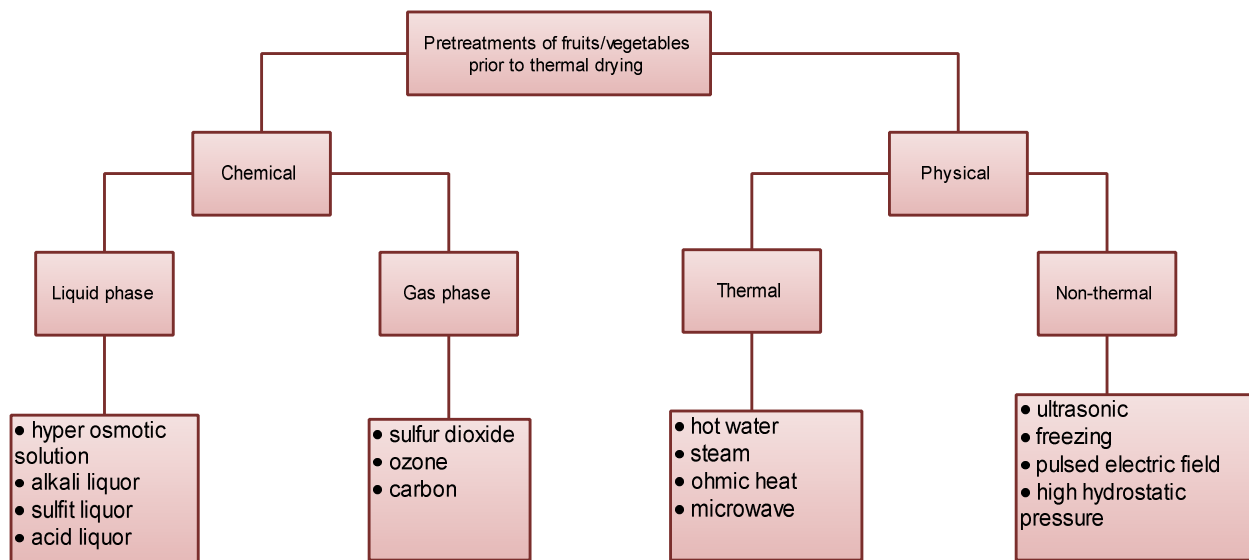


Figure 2.3: Pretreatment methods of fruits or vegetables prior to thermal drying.

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Fruits and vegetables can be pre-processed by blanching, organic acid and sulphiting to eliminate enzymes and microorganisms. Fermentation of fruits and vegetables is a preservation method used in rural areas, and due to the simplicity of the process, there is no need for sophisticated equipment. Sun, solar, tray, spray and integrated drying and osmotic dehydration of fruit and vegetables another method of preservation used in process industries.

#### ❖ **Blanching in hot water**

Thermal blanching is widely used prior to drying, its primary goal is to inactivate the enzymes involved in the spoilage of fresh agro-products, in addition to reduce the microbial load of products so as to improve its conservation, to soften tissues for facilitating drying process, and to eliminate intracellular air to prevent oxidation. Blanching also helps with color retention and modification of product texture (Mate et al., 1999; Ahmed et al., 2001). Moreover, blanching can help increase the drying rate, hence reducing the drying time (Severini et al., 2005).

Fruits, fresh vegetables and root vegetable pieces are immersed in a bath containing hot water (or boiling water) for 1-10 minutes at 91-99°C, to reduce microbial levels, and partially reduce peroxidase and polyphenoloxidase (PPO) activity. The heating time will depend on the type of vegetable product processed Boiling water has been used to provide thermal inactivation of *L. monocytogenes* on celery leaves (Wiley, 1997).

Currently, conventional hot water blanching is the most popular and commercially adopted method, because of its simple equipment and easy operation. It has been widely applied on pretreatment of agro-products to enhance the drying rate and improve the product quality.

#### ❖ **Sulfit solution**

During this operation, the fruit or vegetable pieces (or slices) are immersed in a solution of sodiumbisulphite (200 ppm) to prevent undesirable changes in colour and any additional microbial and enzyme activity, and to retain a residual concentration of 100 ppm in the final product. Sulfitation or sulfuring has been widely used in the food industry to reduce darkening during drying and prevent quality loss during process and storage of foods (Miranda et al., 2009). It is usually performed using sulfur dioxide gas or water-soluble sulfide salts such as potassium metabisulfite ( $K_2S_2O_5$ ), sodium metabisulfite ( $Na_2S_2O_5$ ) and sodium hydrogen sulfite ( $NaHSO_3$ ). When  $SO_2$  is absorbed into the fruit, it is converted mainly to the bisulphate ion. Both

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enzymatic and non-enzymatic browning and microbial activity are prevented by using sulfites at low concentration (Joslyn & Braverman, 1954).

It also acts as an antioxidant in preventing loss of ascorbic acid and protecting lipids, essential oils, and carotenoids against oxidative deterioration during processing, and it has the advantages of maintaining color, preventing spoilage, and preserving certain nutritive attributes (Mujumdar, 2006).

#### ❖ **Osmotic dehydration**

Osmotic dehydration is one of the most widely practiced pretreatments prior to drying to reduce energy consumption and improve food quality (Torreggiani, 1993). It involves the immersion of material in hypertonic solution (mainly sugar or salt) for several hours.

In osmotic dehydration and crystallization, the fruit is preserved by heating the product in sugar syrup or salt, followed by washing and drying to reduce the sugar concentration at the fruit surface. During osmotic pretreatment, plant cellular structure acts as a semi-permeable membrane, countercurrent transfer of mass occurs: the solute flows into the products, while moisture is transferred from the interior to the hypertonic solution (Ciurzynska et al., 2016), as shown in Fig. 2. The driving force of water removal from food material to the osmotic solution is the osmotic pressure difference between food material and the hypertonic solution (Corzo & Gomez, 2004). The advantage of this method is the prevention of discoloration and browning of fruit produced by enzymatic reactions. Thus, the high concentration of sugar in the fruit produces a dehydrated product with good coloring, without the need of chemical preservatives such as sulphur dioxide.

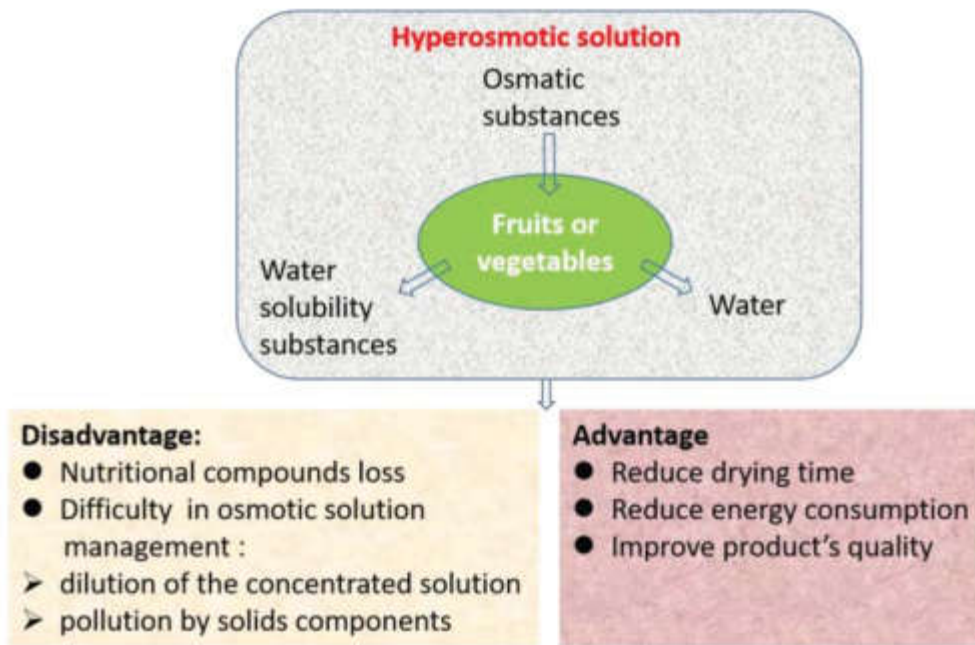


Figure 2.4: Osmotic dehydration process advantage and disadvantage

## ❖ Fermentation

This is another useful preservation process for fruit and vegetable products. For vegetables, the product is immersed into a sodium chloride solution, as in the case of cucumbers, green tomatoes, cauliflower, onions, and cabbage (sauerkraut). Composition of the salt (sodium chloride) is maintained at about 12% by weight so that active organisms during fermentation, such as Lactic acid bacteria, and the *Aerobacter* group, produce sufficient acid to prevent any food poisoning organisms from germinating (Holdsworth, 1983). Fruits, on the other hand, can be preserved by fermenting the fruit pulp into wine, by preparing a solution of sugar and water and then inoculating it with a strain of *Saccharomyces cerevisiae*.

## 2.5 General considerations for preservation of fruits and vegetables

### 2.5.1 Water Activity ( $a_w$ ) concept and its role in food preservation

The concept of  $a_w$  has been very useful in food preservation and on that basis many processes could be successfully adapted and new products designed. Water has been called the universal solvent as it is a requirement for growth, metabolism, and support of many chemical reactions occurring in food products. The ratio of the equilibrium vapor pressure to the saturation vapor

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pressure is known as the equilibrium relative humidity (ERH), or water activity. Water activity  $\alpha_w$ , is of great importance for food preservation as it is a measure and a criterion of microorganism growth and probably toxin release, of enzymatic and non-enzymatic browning development.

$$a_w = \left( \frac{P_w}{P_0} \right)$$

For every food or agricultural product there exists an activity limit below which microorganisms stop growing. Beuchat (1981) refers that the vast majority of bacteria will grow at about  $\alpha_w = 0.85$ , mold and yeast about  $\alpha_w = 0.61$ , fungi at  $\alpha_w < 0.70$ , etc. Comparing this vapour pressure with that of pure water (at the same temperature) results in a ratio called water activity ( $a_w$ ). Pure water  $a_w$  equal to one. According to Rahman and Labuza (1999), enzyme-catalyzed reactions can occur in foods with relatively low water contents. The authors summarized two features of these results as follows:

- I.** The rate of hydrolysis increases with increased water activity but is extremely slow with very low activity.
- II.** For each instance of water activity there appears to be a maximum amount of hydrolysis, which also increases with water content

The relationship between water content and water activity is complex. An increase in  $a_w$  is usually accompanied by an increase in water content, but in a non-linear fashion. This relationship between water activity and moisture content at a given temperature is called the moisture sorption isotherm. These curves are determined experimentally and constitute the fingerprint of a food system many methods and instruments are available for laboratory measurement of water activity in foods. Methods are based on the colligative properties of solutions. Water activity can be estimated by measuring the following:

- Vapour pressure
- Osmotic pressure
- Freezing point depression of a liquid
- Equilibrium relative humidity of a liquid or solid
- Boiling point elevation
- Dew point and wet bulb depression
- Suction potential, or by using the isopiestic method

- 
- Bithermal equilibrium
  - Electric hygrometers
  - Hair hygrometers

## 2.5.2 Recommended substances to reduce water activity $a_w$ in fruits

### ❖ Glucose

Glucose is not very good humectants due to the lower water holding capacity (WHC), which makes it difficult to obtain the isotherm curve at low  $a_w$ .

### ❖ Fructose

Fructose has a higher water activity reduction capacity and therefore is more desirable as a humectants in stabilizing food products.

### ❖ Sucrose

Sucrose is one of the most studied sugars and is widely used in food systems, in the confectionary industry.

## 2.5.3 Recommended substances to reduce pH

### a. Organic acid

Organic acids, whether naturally present in foods due to fermentation or intentionally added during processing, have been used for many years in food preservation. Some organic acids behave primarily as fungicides or fungistats, while others tend to be more effective at inhibiting bacterial growth. The mode of action of organic acids is related to the pH reduction of the substrate, acidification of internal components of cell membranes by ionization of the undissociated acid molecule, or disruption of substrate transport by alteration of cell membrane permeability. The most commonly used organic acids in food preservation include: citric, succinic, malic, tartaric, benzoic, lactic, and propionic acids (FAO,2003).

- ❖ **Citric acid** is present in citrus fruits. It has been demonstrated that citric acid is more effective than acetic and lactic acids for inhibiting growth of thermophilic bacteria. Also, combinations of citric and ascorbic acids inhibit growth and toxin production of *C. botulinum* type B in vacuum-packed cooked potatoes.

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- ❖ **Malic acid** is widely found in fruits and vegetables. It inhibits the growth of yeasts and some bacteria due to a decrease in pH.
  - ❖ **Tartaric acid** is present in fruits such as grapes and pineapples. The antimicrobial activity of this acid is attributed to pH reduction.
  - ❖ **Benzoic acid** is the oldest and most commonly used preservative. It occurs naturally in cranberries, raspberries, plums, prunes, cinnamon, and cloves. As an additive, sodium salt in benzoic acid is suitable for foods and beverages with pH below 4.5. Benzoic acid is primarily used as an antifungal agent in fruit-based and fruit beverages, fruit products, bakery products, and margarine.
  - ❖ **Lactic acid** is not naturally present in foods; it is formed during fermentation of foods such as sauerkraut, pickles, olives, and some meats and cheeses by lactic acid bacteria. It has been reported that lactic acid inhibits the growth of spore forming bacteria at pH 5.0 but does not affect the growth of yeast and moulds.
  - ❖ **Propionic acid** occurs in foods by natural processing. It is found in Swiss cheese at concentrations up to 1%, produced by *Propionibacterium shermanii*. The antimicrobial activity of propionic acid is primarily against moulds and bacteria

#### **b. Inorganic acids**

Inorganic acids include hydrochloric, sulphuric, and phosphoric, the latter being the principal acid used in fruit and vegetable processing). They are mainly used as buffering agents, neutralizers, and cleaners

#### **1.5.4 Recommended chemicals to prevent browning**

##### **❖ Sulphites, bisulphites, and metabisulphites**

Sodium bisulphite is a potential browning inhibitor in fruit and vegetable products (e.g., peeled potatoes, bananas and apples). This preservative when used in food production can delay or prevent undesirable changes in the colour, flavour, and texture of fresh fruits and vegetables, potatoes, drinks, wine, etc. Potassium bisulphite is used in a similar way to sodium bisulphite, and is used in the food industry to prevent browning reactions in fruit and vegetable products.

Sulphites are highly effective in controlling browning in fruits and vegetables, but are subject to regulatory restrictions because of adverse effects on health. Sulphites inhibit non-enzymatic browning by reacting with carbonyl intermediates, thereby preventing further reaction. The

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maximum sulphur dioxide levels in fruit juices, dehydrated potatoes, and dried fruits permitted by the Food and Drug Administration (FDA) are 300, 500, and 2000 ppm, respectively.

## **2.6 Drying Technology Prolong Shelf Life Fruit and Vegetable**

Hot air driers commonly use for drying different food products. In hot air driers, the food is in contact with a moving stream of hot air. Kiln driers, cabinet tray or compartment driers, tunnel driers, spray driers, conveyer driers, bin dryers, fluidized bed driers, pneumatic driers and rotary driers use hot air for drying solid foods. By definition, food dehydration is the process of removing water from food by circulating hot air through it, which prohibits the growth of enzymes and bacteria (Naseer et al.,2013). The prime objective of drying apart from extended storage life can also be quality enhancement, ease of handling, further processing and sanitation and is probably the oldest method of food preservation practiced by humankind (Mujumdar, 2007). It brings about substantial reduction in weight and volume, minimizing packing, storage, and transportation costs and enables storability of the product under ambient temperatures. These features are especially important for developing countries, in military feeding and space food formulations (vanArsdel, 1965).

Different drying methods are available for drying of the agricultural crops. Rotary drums, widely used dryer in the drying of by-products of vegetable origin requires high temperature of the drying gas (about 600 °C), solid agglomeration is frequently produced, large installation size and great size cyclones for the cleaning of the exit gases necessary and the process control is difficult (Aragon et al., 2005). fluidized bed dryers present some advantage over other type of dryer as gas-solid contact is very good and the operation control and monitorization are easy ( Chua et al.,2003). Showed figure 2.3 to summarize types of drying type and method

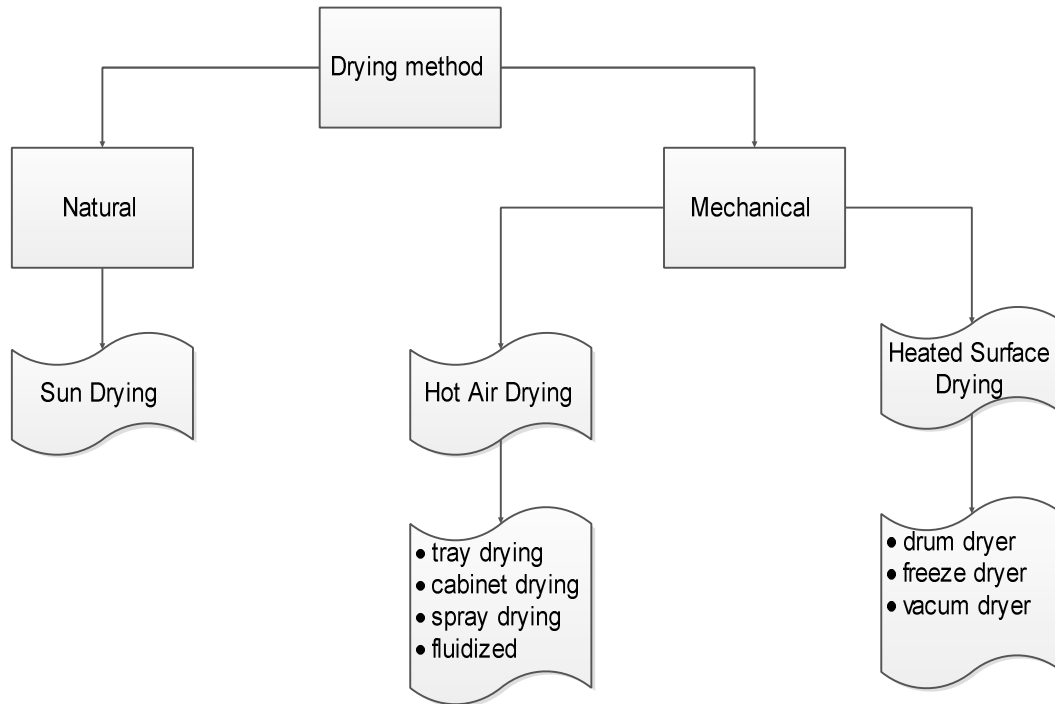


Figure 2.5: Types of drying method

Drying using solar radiation, i.e. drying under direct sunlight, is one of the oldest techniques used by humankind to preserve agriculture based food and non-food products. Preservation of fruits, vegetables, and food are essential for keeping them for a long time without further deterioration in the quality of the product. Several process technologies have been employed on an industrial scale to preserve food products; the major ones are canning, freezing, and dehydration. Among these, drying is especially suited for developing countries with poorly established low-temperature and thermal processing facilities (G.L Visavale, 2012).

## 2.7 Processing Technology Production of Unripe Banana Flour

Fruit and vegetable can be process into powder or flour in several ways. But, the general production process are the same such as raw material collecting, sorting, washing, peeling, slicing/chopping, pre-dehydration( mechanically or chemically), drying and finally milling. For banana flour production the following process are the major one such as raw material collecting and handling, peeling, hot water bathing, slicing/chopping, pretreatments (osmosis dehydration, SMB socking etc) and drying.

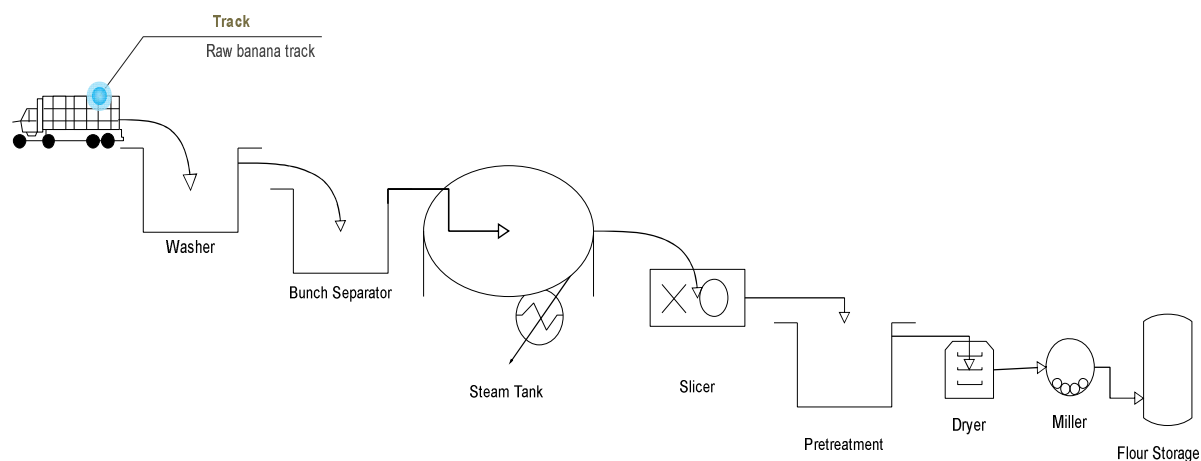


Figure 2.6: Unripe banana flour production process

## 2.8 Osmosis dehydration

Pre-dehydration process can be divided into two mechanically and chemically. In mechanically use squeezing, filtering or mechanical pressing to remove amount of water before drying and in chemical treatment use solution of sugar or salt solution to remove water from sample. However, for food processing OD is the efficient for a pre-dehydration process, and describes YV Shete et al, (2018) it is an operation used for the partial removal of water from plant tissues by immersion in a hypertonic solution, sugar and/or salt solution, to reduce the moisture content of foods before actual drying process.

Main disadvantages of direct convective drying are long drying duration, damage to sensory characteristics and nutritional properties of foods and solute migration from interior of the food to the surface causing case hardening (Sharma and Prasad, 2005). Some important properties of the products have changed such as loss of colour, change of texture, chemical changes affecting flavour and nutrients and shrinkage. The high temperature of the drying process is an important cause for loss of quality. Lowering the process temperature has great potential for improving the quality of dried products (Kumar and Sagar, 2009).

As show in figure 2.7, The osmotic dehydration (OD) process consists of the removal of water from fresh material (i.e. fruits and vegetables) where in the solutes from the osmotic solution are transported to the plant material ( lenart and lewick, 2006). OD is a pre-treatment process, which has many advantages such as improving the physical and chemical food properties including

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colour, flavour, aroma, and texture, as well as possibly improves the biological activity of the products (sareba et al.,2016).

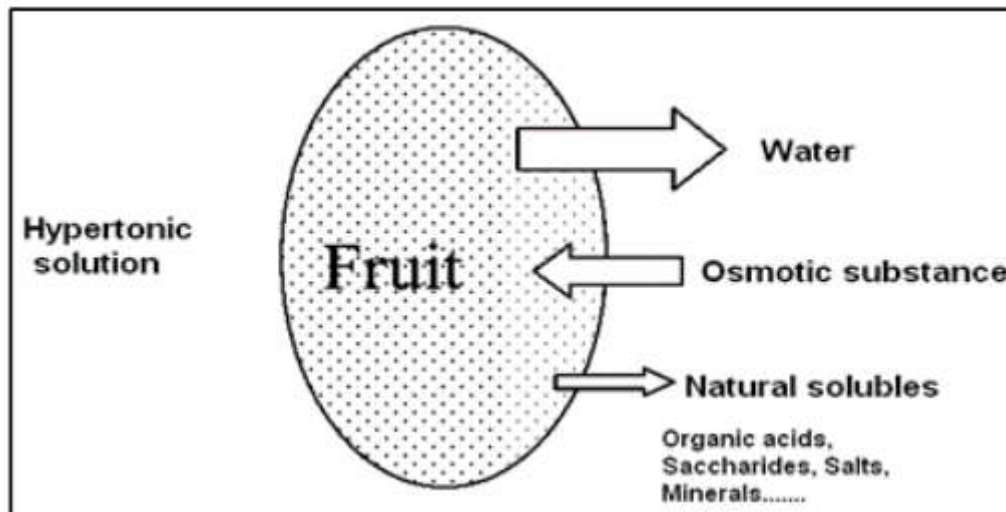


Figure 2.7: Osmotic dehydration process

Osmotic dehydration has received greater attention in recent years as an effective method for preservation of fruits and vegetables. Being a simple process, it facilitates processing of tropical fruits and vegetables such as banana, sapota, pineapple, mango, and leafy vegetables etc. with retention of initial fruit and vegetables characteristics namely, color, aroma and nutritional compounds (Pokharkar & Prasad, 1998).

### 2.8.1 Factors affecting osmotic dehydration process

The selection of process parameters also depends on then application. Choosing the optimal parameters of the process prevents adverse changes that may occur in the case of certain raw materials, especially those with a delicate structure (Ciurzynska *et al.*, 2016). Variables like maturity, variety, pretreatments, temperature, immersion time, nature and concentration of osmotic agent, agitation, geometry of the material, fruit pieces to osmotic solution ratio, physico-chemical properties, additives, structure and pressure affecting the osmotic dehydration process.

#### ❖ Shape, size and thickness of the fruit pieces

Water loss increases with increase in the surface area of fruit pieces. Panagiotou *et al.* (1998) observed that the size of fruit samples had a negative effect on water loss during osmotic treatment. Rahman (1992) observed that the distribution coefficient of water decreased with

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increasing temperature and surface area and it increased with the increase in syrup concentration and thickness of minimum geometric dimension.

#### ❖ **Pretreatments**

Dipping in 1 percent, citric acid solution prior to drying or osmotic dehydration was used to prevent enzymatic browning of fruits. Immersion of product in alkaline or acid solutions of oleate esters prior to drying of fruits affected the prevention of discoloration (Hussain *et al.*, 2004; Sunkja & Ragharan, 2004). Torreggiani, (1993) reported that pretreatment with chemicals (SO<sub>2</sub>), or blanching prior to dehydration of fruits and vegetables effected the prevention of discoloration.

#### ❖ **Immersion time**

The increase in immersion time leads to higher loss of moisture during osmotic dehydration (Ispir and Toğrul, 2009; Mundada *et al.*, 2011). In general, as the time of treatment increases, the weight loss increases, but the rate at which it occurs decreases. The treatment time can be selected in such a way that the amount of water removal is maximum with no appreciable uptake of solids.

#### ❖ **Temperature of osmotic solution**

The temperature of osmotic solution markedly affected the rate of osmosis. Although the rate increased with temperature, it was limited up to 60 °C as higher temperature destroyed the cell membranes. Pokharkar and Prasad (1998) developed kinetic model for osmotic dehydration of banana slices and reported that the temperature of the osmotic solution affected the parameters like water and sugar gain of osmosis process.

#### ❖ **Osmotic agents**

The concentration of osmotic agent plays an important role in osmotic dehydration. Increased solution concentration resulted in the increase in the osmotic pressure gradients and higher water loss (Phisut, 2012). During extended osmotic treatment, the increase of solute concentrations results in the increase in water loss and solid gain rates (Phisut, 2012). Less concentrated sucrose solution leads to minimal loss of water and solid gain ratios (Tortoe, 2010).

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### ❖ Fruit pieces to osmotic solution ratio

With an increase in solution to sample ratio, the rate of osmosis increases up to a certain extent. However, it is essential to use an optimum ratio since large ratios offer practical difficulties in handling the syrup fruit mixture for processing.

## 2.9 Use of banana flour

Banana is the fourth most important crop after rice, wheat and corn. It contains appreciable amounts of vitamins B and C, as well as minerals like potassium and calcium. In its green stage it has high levels of starch, mainly in the form of resistant type 2 (Cano et al., 1997; Gutierrez et al., 2008).

Banana is considered one of the most important fruits worldwide because of its high content of vitamins (B, C and K) and minerals (iron, phosphorus and calcium). Banana is sensitive to chemical and microbial deterioration during postharvest storage and handling; therefore, it has a limited shelf life in a fresh form, causing economic losses.

Starch is the most important component of green banana fruit, representing a mass fraction of 70-80g/100 g db. According to Juarez-Garcia et al. (2006) green banana flour (GBF) produced under specific conditions is composed as follows: 73.4 g/100 g of total starch, 17.5 g/100 g of RS and 14.5 g/100 g of dietary fiber (DF). Moreover, great part of the RS found in GBF is the type 2 (RS2) which crystallinity makes it scarcely susceptible to hydrolysis and therefore offers positive effects in the human colon and implications for health (Langkilde, Champ, & Anderson, 2002), in view of the reduction of postprandial glycemic and insulinemic levels when ingested.

According to Ovando Martinez *et al* (2009) green bananas are an excellent source of carbohydrates as well as nutritionally important bioactive compounds. Moreover, unripe banana is a source of antioxidant polyphenols which show strong protective effects against certain diseases, such as cancer, rheumatoid arthritis and cardiovascular disease (Chong and Noor Aziah, 2010; Ovando-Martinez *et al.*, 2009).

Additionally, studies give the consumers a product which is affordable with health benefits; typically a gluten-free alternative to wheat-based flours for those suffering from celiac disease and those who choose a gluten-free diet (Crofts, N & Coghlan, L, 2014). Moreover, according to Langkilde et al (2002) green banana flour has an excellent and useful source of resistant starch.

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As stated Iowa state University (2014) research has shown that increased resistant starch intake may reduce the risk of obesity, diabetes, and colon cancer. Bananas are significant sources of potassium and have a beneficial effect on blood pressure. Many epidemiological and intervention studies have shown that potassium intake is associated with a blood pressure lowering effect. Additional development of a shelf-stable banana flour product from fresh banana is an important consideration to reduce post harvest-losses, healthy nutrition and food security.

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## CHAPTER THREE

### 3. MATERIALS AND METHODS

#### 3.1 Reagents and Equipment

##### 3.1.1 List of Reagent and chemicals

Sodium metabisulfite, sulfuric acid, glycerol, hexane, acetone, citric acid, sodium hydroxyde, HCL, nitric acid, sodium carbonate, sodium phosphate, iron(III) chloride, potassium oxalate, hydrogen peroxide, ethanol ,distilled water, and methanol

##### 3.1.2 List of instrument and apparatus

The following equipment were used for the study

Table 3.1: List of equipment and instruments

| Name of instrument and apparatus                        | Model                      | Use                                    |
|---|----------------------------|--|
| AAS( atomic absorption spectroscopy)                    | NOV A400P                  | To determine of chemical elements      |
| UV- Spectrophotometer                                   | ANALAYTIC GENA 233H160C    | To determine non-enzymatic reaction    |
| Kjeldah digester  |                            | To determine crude protein             |
| Muffle furnace  | Carbolite CSF 1200 Germany | To determine ash content               |
| Electronic oven dryer                                   | Model 30-160               | To dry sample                          |
| Electronic Balance                                      | ABS 220-4N                 | To weigh sample                        |
| GFAAS (Graphite furnace atomic absorption spectroscopy) | NOVA-400P-Germany          | To determine trace metals              |
| Portable Digital Refractometer                          | WZB 45                     | To determine <sup>0</sup> Brix and TDS |

|                              |                    |   |
|------------------------------|--------------------|---|
| Vortex mixer                 | VM-300 Germany     | To mix sample with water and oil        |
| P <sup>H</sup> meter         | HI8314Membrane     | To measure pH                           |
| Miller                       | PW100              | To mill sample                          |
| Digital reading pipettes     | CHK-C210 & CHC-C20 | To take sample in small scale           |
| Digital infraned thermometer | IR 1600            | To measure temperature                  |
| Water bath                   | HH-50              | To bath sample at different temperature |
| Filter paper                 | Watt man           | To filter samples                       |
| Microwave digester           | BOV 80001 Germany  | To digest sample before AAS and GFAAS   |
| Soxhlet extractor            |                    | To extract fat                          |
| Rotary evaporator            | RVO 400 Germany    | To remove water                         |

### 3.2 Raw Material Collection, Transportation, Sample Preparation and Storage

#### 3.2.1 Raw material collection

Preparation of unripe banana flour: Bananas were collected from Shalle, Arba Minch Zuria Werda Gamo Gofa SNNPR, Ethiopia. A total of about 20-30kg bananas purchased birr five per kg, more green than yellow in the ripeness stage 2-3(show in figure 3.1a below) from farm site. After that, washing with tap water and stored in a refrigerator at 4 °C prior to the experiment.

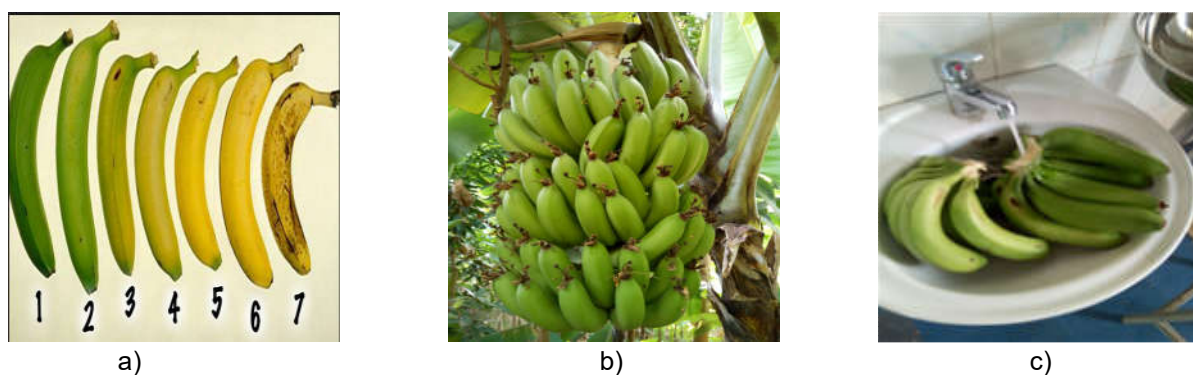


Figure 3.1: Fresh Unripe banana a) Stage of banana ripping b) Banana with bunch and c) Banana after bunch removed

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### 3.1.1 Preparation of green banana flour by different pretreatments

Fresh green bananas washed with water, peeled and slices into pieces for  $3\pm 1$ mm thickness and followed by pre-treated with 0.2% SMS and osmotic solution dehydration different concentrations (30 °Brix, 40 °Brix and 50 °Brix), soaking temperature (40, 50 and 60 °C) and soaking time (30, 60 and 90 min). However, hot water pre treatment were accomplished before peeled the fresh banana, that fresh banana treated with hot water at 100 °C for 10 min in water bath, then peeled and slices for  $3\pm$ mm thickness.

Slices were dried for 24 hours at different temperature (45°C, 60°C and 75°C) in oven dryer. After drying ground all of them into flour using grinder. Banana flour was sieved using 500- $\mu$ m sieve, packed into different zip lock pouches, and then stored at room temperature separately for further analysis. More Show figures 3.2 experiment framework.

### 3.1.1.1 Experiment framework setup

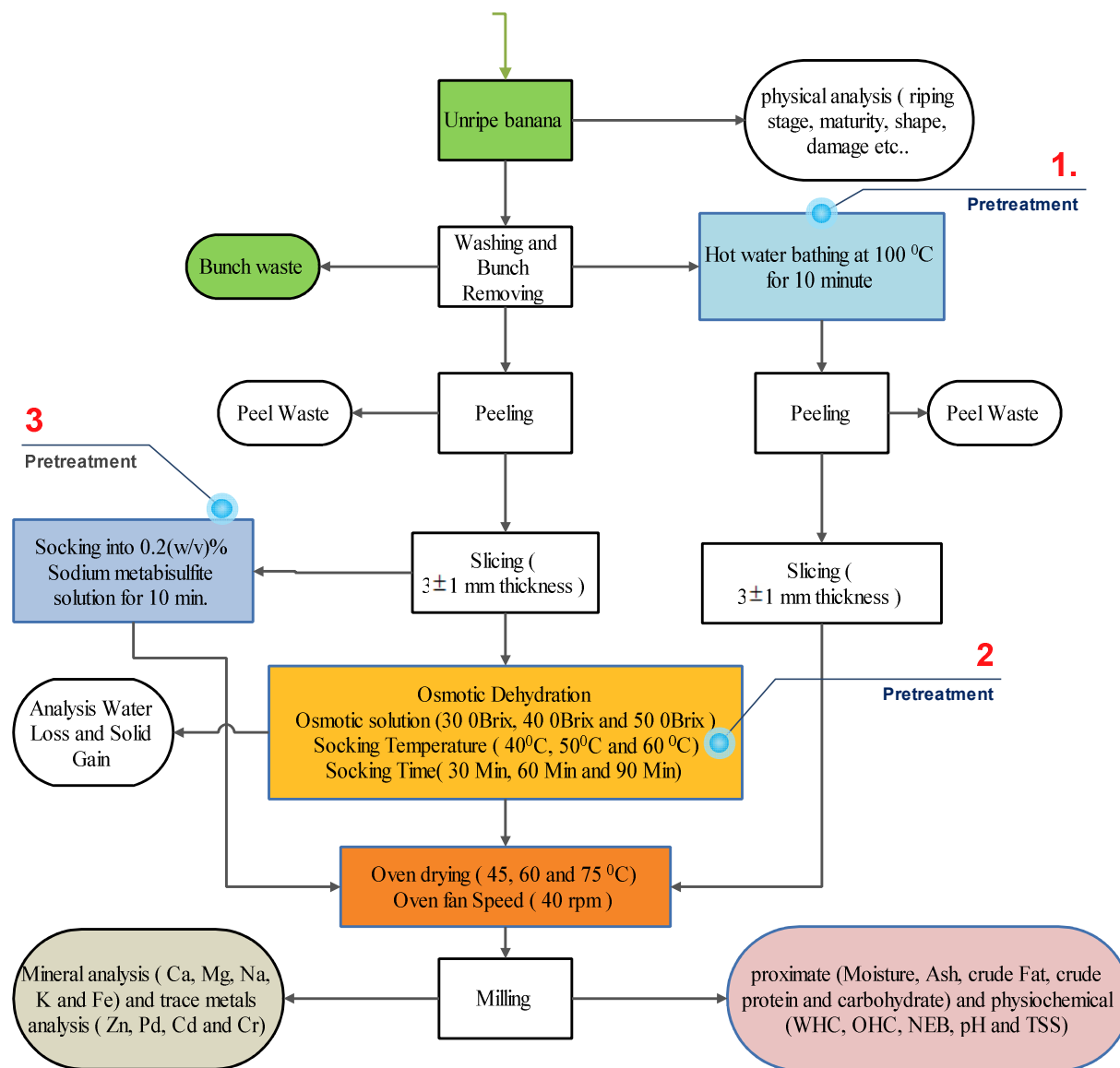


Figure 3.2: Framework of experiment set up

### 3.1.2 Preparation of Osmotic dehydrated banana flour

This experiment had three pre-treatment parts before conventional oven drying such as:

- ✓ Osmotic dehydration,

- 
- ✓ Sodium metabisulfite solution soaking and
  - ✓ Hot water bathing

For the first part experiment raw banana washed by water and peeled, then cut the weight of the pulp 100g into transverse slices about the  $3\pm 1$ mm thickness for every experiment. The prepared slice pulp then treated with osmotic sucrose concentration of (30<sup>0</sup>Brix, 40<sup>0</sup>Brix & 50<sup>0</sup>Brix), soaking temperature (40<sup>0</sup>C, 50<sup>0</sup>C, and 60<sup>0</sup>C) and soaking time (30 min, 60 min, and 90 min). The osmotic dehydration experiment was performed in a beaker placed in a water bath, which was equipped with temperature regulator and timer.

The osmotic solutions was prepared by purchasing commercial grade sugar and then prepare osmotic solutions for different concentration (30<sup>0</sup>Brix , 40<sup>0</sup>Brix and 50<sup>0</sup>Brix) by dissolved in the required amount of distilled water and maintain sample to solution ratio 1:3 (w/v). The concentration of the solution was measured using hand refractometer at room temperature.

Procedure repeated for 40°C, 50°C and 60 °C temperature. The temperature was maintained constant using hot water temperature bath. Then the solution was drained away and slices were taken out. The percentage of water loss and solid gains was calculated for each observation and finally obtains optimal parameter among that gives maximum water loss and minimum solid gains. The optimal obtained banana slices after treatment dried in an oven dryer for a period of 24 hours at 45 °C, 60 °C and 75 °C. After dried milled all of them into flour using grinder and sieved using 500-µm sieve. Finally packed into different zip lock pouches, and then stored at room temperature separately for further analysis.

### **3.1.3 Sodium metabisulfite and hot water pre treatment**

The second and third part pre-treatment socked banana slice into sodium metabisulfite (0.2% w/v) solution for 10 minutes. However, during hot water bath treatment socked fresh banana into hot bath at 100<sup>0</sup>C for 10 minutes before peeling. After pre-drying, sodium metabisulfite and hot water bath, dried in on oven at 45, 60 & 75<sup>0</sup>C. Finally, the dried samples were milled and packed in zip-lip low-density polyethylene (LDPE) bags, sealed properly and keep at ambient temperature or desiccators for further quality analysis.



Figure 3.3: SMBS & Hot water pretreated banana slice and flour . a) hot water pretreated banana slices, b) hot water pretreated banana flour, c) SMBS pretreated banana slices and d) SMBS pretreated banana flour

### 3.3 Experimental Procedures

The proximate, mineral composition and physiochemical analysis was conducted at Arba Minch University, Addis Ababa University and Bless Agri-food laboratory, those are moisture content, ash content, protein, fat, crude fiber and carbohydrate determines according to AOAC methods. In addition, mineral composition and functional property unripe banana flour analyzed.

#### 3.3.1 Determination of moisture content

The amount of water present in the green banana flour sample considered equal to the loss of weight after drying. The moisture content of the flour, samples were determined by dry air oven method according to (AOAC, 2005). The Petri dish was dry at  $130 \pm 3^\circ\text{C}$  for 1 hour and placed in desiccators and weighed after cooling. About 3g of flour were weighed by analytical balance and put on a dry dish. Dry dish and its contents put in the oven maintained at  $135^\circ\text{C} \pm 3^\circ\text{C}$  for 1hour. The sample-containing dish was transferred to a desiccator and weighed soon after reaching room temperature. Then, the moisture content was estimated by the formula:-

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$$\text{Moisture (\%)} = \frac{(W_2 - W_3)}{(W_2 - W_1)} \times 100 \dots \dots \dots (3.1)$$

Where:

W1 = Weight of the drying Petri dish,

W2= Mass of the drying Petri dish and the sample before drying,

W3 = Mass of the drying Petri dish and the sample after drying

### 3.3.2 Determination of ash content

Ash contents was determined by the method of the according to AOAC (2000). Clean porcelain crucible, dried at 120°C in an oven was cooled in desiccators and weighed (M1). Then weight 2.5 g of samples in a porcelain crucible ( M2). The crucible with the contents was placed in a Muffle furnace, at 550°C for five hours to ignite until ash were completed. After this period, the crucible with its content removed and cooled in desiccators. The crucible with the residue weighted (M3). The weights of the ash were expressed as a percentage of the initial weight of the samples. The total ashes were expressed as percentages on a dry matter basis as follows.

$$\text{Ash content (\%)} = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 100 \dots \dots \dots 3.2$$

Where:

M3 =Final mass of sample with crucible (g)

M2=Sampled mass with crucible (g) and

M1=Mass of crucible (g)

### 3.3.3 Determination of crude fat content Fat

Crude fat UB flour was determined by Soxhlet-extraction according to (AOAC, 2005). About 2 g of the sample were weight and put into a thimble. The thimble and its extraction flask were transferred into an extraction unit. The sample contained in the thimble extracted with the solvent petroleum ether on Soxhlet extraction apparatus for 6 hours. After the extraction completed, the extraction thimble was dried in the oven for 30 minutes at 102°C ± 2°C to remove moisture. Then, it removed from the oven and cooled in desiccators. The cup and its contents were weight. The crude fat was determined by the following formula:-

$$\text{Crude fat (\%)} = \frac{W_2 - W_1}{\text{weight of sample}} \times 100 \dots \dots \dots (3.3)$$

Where:

W1= Weight of extraction flask before extraction

W2 = Weight of extraction flask after extraction



Figure 3.4: Crude fat laboratory extraction a) Soxhlet-extraction and b) Rotary evaporator

### 3.3.4 Determination of crude Protein

The crude protein content of UBF was analyzed by the Kjeldahl method of nitrogen analysis according to AOAC (2005). About 0.3 gm of the sample, 1 g of catalyst mixture of  $K_2SO_4$  and  $CuSO_4$  and 5 ml of sulfuric acid were added to digestion flask at about  $370^\circ C$  digested until the solution became clear. Then, distillation took place by adding 25 ml of 40% NaOH and using 25 ml of boric acid with 10 drops of indicator solution. As the distillation was going on, the pink color solution of the receiver flask turned green indicating the presence of ammonia. The green color solution was then titrated with 0.1N HCl solutions. At the endpoint, the green color turned to red-pink color, which indicated that all the nitrogen trapped as ammonium borates had been removed as ammonium chloride. The distillates were titrated with standardized 0.1N sulfuric acid to a reddish color. Finally, the percentage of nitrogen contents was estimated using the following formula:

$$\text{Total nitrogen (\%)} = \frac{(T-B) \times N \times 14.007}{\text{weight of sample}} \times 100 \dots \dots \dots (3.4)$$

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$$\text{Crude protein} = 6.25 \times \text{Total Nitrogen} \dots \dots \dots (3.5)$$

Where:

T = Volume in ml of the standard acid solution used in the titration for the test material

B = Volume in ml of the standard acid solution used in the titration for the blank determination

N = Normality of standard sulfuric acid

### 3.3.5 Determination of Crude fiber determination

The crude fiber was determined by the non-enzymatic gravimetric method of (AOAC, 2005). About 1.5 g UBF was placed into a 600mL beaker. A 200 ml of 1.25% H<sub>2</sub>SO<sub>4</sub> solution were added to each beaker and allowed boiling for 30 min by rotating and stirring. During boiling, the level was kept constant by addition of hot distilled water. After 30 min, 20 ml of 28% potassium hydroxide solution added into each beaker and again allowed to boil for another 30 min. The level was kept constant by addition of hot distilled water. The solution in each beaker was filtered through filter paper. During filtration, the sample will be washed with hot distilled water. The final residue was washed with 1% H<sub>2</sub>SO<sub>4</sub> solution, hot distilled water, 1% NaOH solution, 1% H<sub>2</sub>SO<sub>4</sub>, hot distilled water and finally with acetone. It was allowed to dry and scrapped into a crucible, each of the crucibles with their contents will be dried for 2 hours at 130°C in the oven cooled in desiccators and weighed (M<sub>1</sub>). Then again, it was ashed for 30 min at 550°C in the furnace and cooled in desiccators. Finally, the mass of each crucible was weighed (M<sub>2</sub>) to subtract ash from the fiber. The crude fiber calculated from equation.

$$\text{Crude fiber}(\%) = \frac{(M_2 - M_1)}{W_0} \times 100 \dots \dots \dots (3.6)$$

Where:

M<sub>1</sub> = Mass of the crucible and sample after ashed

M<sub>2</sub> = Mass of crucible and sample after drying in an oven

W<sub>0</sub> = Sample weight

### 3.3.6 Determination of carbohydrates

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The carbohydrate contents was determined by subtracting the summed up percentage compositions of moisture, protein, lipid, fiber, and ash contents from 100 g of the sample (AOAC, 2000)

$$\text{Carbohydrate (\%)} = 100\% - (\text{moisture\%} + \text{protein \%} + \text{fat \%} + \text{ash\%} + \text{fiber \%}) \dots (3.7)$$

### 3.3.7 Determination of gross energy

The sample calorific value was estimated (in kcal/g) by multiplying the percentages of crude protein, crude lipid and carbohydrate with the recommended factors (4, 9, and 4 respectively) (Nguyet et al., 2007). The amounts of gross energy were determined by the given equation.

$$\text{Gorse Energy (Kcal/100gm)} = (4x \text{ g protein} + 4 x \text{ g Carbohydrate} + 9 x \text{ g fat}) \dots \dots (3.8)$$

## 3.4 Physicochemical Analysis

### 3.4.1 Water holding capacity (WHC) and oil holding capacity (OHC)

The WHC and OHC of the samples were determined by using the methods suggested by Beuchat (1977). The flour (1 g) was vortexes with distilled water (10 mL) in pre-weighed centrifuge tube for 30 minutes. After standing at room temperature for 30 minutes, the sample was centrifuged for 25 min at 3000 ×g. The sediments were weighed after complete removal of the supernatant. For the determination of OHC, the flour (0.5 g) was homogenized with canola oil (5mL) in pre-weighed centrifuge tube and proceeded further as described for WHC.

The WHC and OHC (%) were calculated as:

$$\text{WHC or OHC (\%)} = \frac{W_2 - W_1}{W_0} \times 100 \dots \dots \dots (3.9)$$

Where

W0 = Weight of the sample,

W1 = Weight of centrifuge tube plus sample and

W2 = Weight of the centrifuge tube plus sediments

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### 3.1.4 Non-enzymatic browning (NEB)

For estimation of browning reaction, 5 g of the sample was mix with 100 ml of 60 ml/100 ml absolute alcohol in a glass flask. The mixture was shaking thoroughly, kept for 12 h and then filtered through Whatman No. 4 filter paper. The optical density (OD) of the filtrate was measured at 420nm in a spectrophotometer(SEECORD/50) Browning index was expressed as absorbance value at 420 nm ( Sharma et al.,1993).



Figure 3.5: Uv- Spectroscopy NEB absorbance reading

### 3.4.2 Total soluble solids (TSS)

Total soluble solid (TSS) in sample were measured using digital refractometers at 20 °C. A known (5g) quantity of sample was crushed and dissolve with a 5ml of distilled water at 1:1 ratio after 30 minutes the TSS were measured by placing one to two drops of clear juice. TSS of juice obtained was read in a digital refractor meters 0-45% in Brix ranges ( Guine et al.,2014).

### 3.4.3 pH of sample

The pH of the sample where measured with pH meter (HI8314). Prior to pH measurement, the instruments were calibrated with buffer. The pH of the samples will be estimated directly according to Velic et al (2007).

### 3.4.4 Determination of minerals

Elements such as sodium (Na), potassium (K), magnesium (Mg), calcium (Ca) and iron (Fe) was determined by instrument Atomic Absorption spectroscopy(AAS) and copper (Cu), Zinc (Zn),

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Lead(Pd) and cadmium(Cd) graphite furnace atomic absorption spectroscopy (GFAAS) using microwave acid digestion process method AOAC( Fung W., 2016).

Wet digestion method was use for digestion of the both dried and fresh samples. 0.5 g of each of the sun-dried and oven dried, samples was accurately weighted into a digestion tube. The ratio of 3:2:1 the ratio of Nitric, per chloric acid and hydrogen peroxide were measured and added into the digestive tube and swirled gently to mix the sample properly. The digestion vessel were putted in microwave oven for 30 min on power (90%). At the end of the digestion cycle, the turntable rotation will be stopped and Leave the digestion vessels in the microwave oven for about 5 minutes to exhaust fumes. The digestion vessels Removed from the microwave oven, remove the cap (in a fume hood), and rinse with water. Rinse down sides of container. Sample solutions were Filtered using Whatman No. 42 filter paper into 100-mL volumetric flasks. After thorough mixing, transfer an aliquot into a 60-mL Nalgene bottle for determination of calcium (Ca), magnesium (Mg), potassium (K), sodium (Na), iron (Fe) and Zinc (Zn)( Isaac *et al*, 1998).

For iron, content determination absorbance were measured at 248.4 nm. For zinc concentration determination, absorbance will measure at 213.9 nm. Calcium content determination, absorbance were measured at 422.7 nm, magnesium content determination; absorbance were measured at 285.2 nm. Mineral content will calculate using the following formula: -

$$\text{Mineral content (mg/100g)} = \frac{[(A-B)*V]}{10W} \times 100\%$$

Where:

W= Weight (kg) of samples

V= Volume (liter) of extract

A= Concentration ( $\mu\text{g/L}$ ) of sample solution

B = Concentration ( $\mu\text{g/ml}$ ) of blank solution

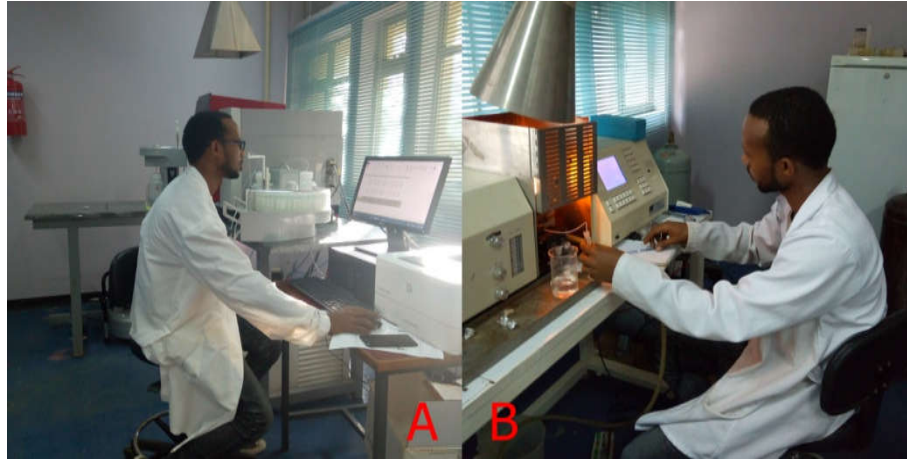


Figure 3.6: Unripe banana flour mineral composition reading a) GFASS and b) AAS

### 3.4.5 Water loss (WL) and Solid gain (SG)

The weight and moisture content of samples before and after the osmotic process determine and used to calculate the variables water loss (WL) and solid gain (SG) of processed banana. Solid gain is the amount of solid added from the osmotic solution to the sample during OD. Solid gain (g/100g fresh sample), and water loss (WL, g/100g fresh sample) was calculated as follows: Based on review of earlier works (Lenart and Flink, 1984), the Water losses (ML) were characterized using the following equation:

$$WL = \frac{(X_{0w} * M_o - X_{tw} * M_t)}{M_o} \times 100 \dots \dots \dots (3.10)$$

The solid gain (SG) was measure by the following equation as suggested by Lenart and Flink(1984).

$$SG = \frac{X_{tts} * M_t - X_{ots} * M_o}{M_o} \dots \dots \dots (3.11)$$

Where:

$M_o$  = Initial mass of the sample

$X_{tts}$  = Total solid concentration of sample at time t

$X_{ots}$  = Initial solid concentration of sample

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$X_{ow}$  = Initial water concentration of sample

$X_{tw}$  = Water content of sample at time t

### 3.5 Experimental design and Statistical Analysis

Response surface methodology (RSM) is used to study the main effects of the process variables on mass transfer during OD pre-drying process and to find the optimum operation conditions. The experimental design adopted were a full factorial design with three factors and three levels. The actual factor values, chosen from preliminary tests, and the corresponding coded values (-1, 0, 1) are given in Table 3.2. Weight loss and solids gain of the dehydrated samples is the dependent variables. The complete design consists of 32 experimental runs with five replications of the center point. Experimental runs will be carried out in random order.

All experiments were conducted in duplicate and the data subject to analysis of variance. A significance level of 0.05 was used in all cases. The design of experiment was done by full factorial design and using statistical software design expert 7.0.0. Response surface methodology RSM was employed using general factorial design for three numeric factors. The experiments for OD of slice banana using three factors: sugar concentration (30 to 50 °Brix), soaking time (30 to 90 minutes) and temperature (45 to 75 °C).

The variables were process temperature, solute concentration and duration of OD process. Three different levels for each experiment in coded form were -1, 0 and +1. The levels of input variables coded and uncoded given at Table 3.2. Further, three functions exist between each response and the input factors. Kumar et al. (2008) stated that when regression coefficient has a positive sign, the increase of the associated factor causes an increase in response and a negative sign would cause a decrease in the optimization parameter. Second order polynomial equations were fitted to the experimental data of each independent variable as given below.

$$Y_k = B_{ko} + \sum_{i=1}^3 B_{ki}x_i + \sum_{i=1}^3 B_{kii}x_i^2 + \sum_{i=1}^3 \sum_{j=1}^3 B_{kij}x_i x_j + e_k \dots \dots \dots (3.12)$$

Where

$Y_k$  = Response variable (Y1 = water loss; Y2 = solid gain) and

$x_i$  = Coded independent variables ( $i = 1, 2, 3 = A, B, C$  respectively, where, A = process temperature; B = solute concentration; C = process duration),  
 $B_{k0}$  = Value of the fitted response at the center point of the design,  
 $B_{ki}$ ,  $B_{kii}$  and  $B_{kij}$  = Linear, quadratic and interaction regression coefficients, respectively.

Table 3.2 Coded and un-coded values of variables and their level

| Independent variable                         | Code value | -1 | 0  | +1 |
|--|------------|----|----|----|
| Temperature (process temperature, $C^0$ )    | A          | 45 | 60 | 75 |
| Osmotic solution concentration ( $^0$ Brix ) | B          | 30 | 40 | 50 |
| Soaking time (minutes)                       | C          | 30 | 60 | 90 |

Regression analysis and analysis of variance was carried out for fitting the model the experimental data and to examine the statistical significance of the model. Three dimensional response models were generating. The response surface plots generate for different variables, while holding the values of third variable as constant (at the central value). Such response surface plots give accurate geometrical representation and provide useful information about the behavioral system with experimental design. The models obtained are use to interpret the effect of various variables on the responses i.e. WL and SG.

The optimization of the OD process was aimed at finding the levels of three independent factors (sugar concentration, time and temperature) which would give maximum possible WL and minimum SG. After the OD optimal variable determined, followed by oven dryer at different temperature ( $45^0C$ ,  $60^0C$ , and  $75^0C$ ). Results were express as mean values  $\pm$  standard deviations.

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## CHAPTER FOUR

### 4. RESULTS AND DISCUSSION

#### 4.1 Analysis of response surface methodology on osmotic dehydration of unripe banana sliced pulp

Osmotic dehydration of banana response water loss and solid gain slice were affected by different treating parameters including soaking temperature, osmotic solute and soaking time. The experimental design selected for this study was response surface method and the response measured were the water loss and solid gain. The experimental design selected for this study was Response Surface Methodology (RSM) and the response variable measured was percentage water loss and solid gain. RSM provides an estimate for the value of responses for every possible combination of the factors by varying the values of all factors in parallel, making it possible to comprehend a multi-dimensional surface with non-linear shapes (Jerry Fireman et. al., 2011).

In this section, the results obtained from the pretreatment of unripe banana in osmotic solution presented in table 4.1. The influence of operation variables on water loss and solid gain were discuss as follow:

Table 4.1: Designed experiments according to full factorial design and measured

| Std Order | Factor 1<br>A: <i>Temperature</i><br>( <sup>o</sup> C) | Factor 2<br>B: Concentration<br>(Brix) | Factor 3<br>C:Time(Min) | Response 1<br><b>Water loss(%)</b> | Response 2<br><b>Solid gain(%)</b> |
|-----------|--|--|-------------------------|------------------------------------|------------------------------------|
| 1         | 40   | 30                                     | 30                      | 20.858                             | 3.21                               |
| 2         | 50   | 30                                     | 30                      | 22.958                             | 3.6                                |
| 3         | 60   | 30                                     | 30                      | 24.358                             | 4.2                                |
| 4         | 40   | 40                                     | 30                      | 26.0798                            | 4.8                                |
| 5         | 50   | 40                                     | 30                      | 24.778                             | 4.712                              |
| 6         | 60   | 40                                     | 30                      | 27.184                             | 5.6                                |
| 7         | 40   | 50                                     | 30                      | 27.389                             | 4.34                               |
| 8         | 50   | 50                                     | 30                      | 27.723                             | 4.8                                |
| 9         | 60   | 50                                     | 30                      | 28.114                             | 6.2                                |
| 10        | 40   | 30                                     | 60                      | 25.479                             | 4.7                                |
| 11        | 50   | 30                                     | 60                      | 22.951                             | 5.436                              |

|    |    |    |    |         |        |
|----|----|----|----|---------|--------|
| 12 | 60 | 30 | 60 | 29.674  | 6.78   |
| 13 | 40 | 40 | 60 | 29.541  | 7.82   |
| 14 | 50 | 40 | 60 | 27.702  | 9.037  |
| 15 | 60 | 40 | 60 | 33.334  | 10.6   |
| 16 | 40 | 50 | 60 | 34.755  | 10.763 |
| 17 | 50 | 50 | 60 | 32.715  | 13.12  |
| 18 | 60 | 50 | 60 | 36.755  | 13.99  |
| 19 | 40 | 30 | 90 | 20.106  | 3.12   |
| 20 | 50 | 30 | 90 | 21.191  | 3.65   |
| 21 | 60 | 30 | 90 | 29.14   | 4.31   |
| 22 | 40 | 40 | 90 | 30.842  | 9.2    |
| 23 | 50 | 40 | 90 | 30.665  | 9.946  |
| 24 | 60 | 40 | 90 | 37.909  | 12.43  |
| 25 | 40 | 50 | 90 | 38.569  | 13.2   |
| 26 | 50 | 50 | 90 | 38.4084 | 14.6   |
| 27 | 60 | 50 | 90 | 39.415  | 17.143 |
| 28 | 50 | 40 | 60 | 25.145  | 8.125  |
| 29 | 50 | 40 | 60 | 26.096  | 8.326  |
| 30 | 50 | 40 | 60 | 26.917  | 8.415  |
| 31 | 50 | 40 | 60 | 27.564  | 8.018  |
| 32 | 50 | 40 | 60 | 27.567  | 8.154  |

Table 4.2A: Design summary for factors

| Study Type Response = Response Surface |               |          |         | Runs = 32         |             |           |            |      |           |
|--|---------------|----------|---------|-------------------|-------------|-----------|------------|------|-----------|
| Initial Design= 3 Level Factorial      |               |          |         | Blocks =No Blocks |             |           |            |      |           |
| Design Model= Quadratic                |               |          |         |                   |             |           |            |      |           |
| Factor                                 | Name          | Units    | Type    | Low Actual        | High Actual | Low Coded | High Coded | Mean | Std. Dev. |
| A                                      | Temperature   | degree C | Numeric | 40                | 60          | -1        | 1          | 50   | 7.5       |
| B                                      | Concentration | Brix     | >>      | 30                | 50          | -1        | 1          | 40   | 7.5       |
| C                                      | Time          | Min      | >>      | 30                | 90          | -1        | 1          | 60   | 22.5      |

Table 4.2B: Design summary for response

| Response | Name       | Units | Obs | Analysis   | Min   | Max.   | Mean  | Std. Dev. | Ratio | Trans | Model     |
|----------|------------|-------|-----|------------|-------|--------|-------|-----------|-------|-------|-----------|
| Y1       | water loss | %     | 32  | Polynomial | 20.11 | 39.415 | 28.81 | 5.247     | 1.96  | Non   | Quadratic |
| Y2       | solid gain | %     | 32  | Polynomial | 3.12  | 17.143 | 7.88  | 3.718     | 5.49  | Non   | Quadratic |

"Model Summary Statistics" as showed table 4.3 and table 4.4 below Focus on the model maximizing the "Adjusted R-Squared" and the "Predicted R-Squared". The predicted R-squared indicates the closeness of the factors for the model, it approach to 1 means good fit for the model selection, quadratic model was fit for in this case work. The standard deviation indicates the difference between each factor from temperature, concentration, time and grouped factor difference. Therefore, from the difference took the smallest Std. Dev and maximum Adjusted R-Squared & the Predicted R-Squared in number. Therefore, in this case both WL & SG response the suggested models were quadratic model.

Table 4.3: Water loss Model Summary Statistics

| Source    | Std. Dev. | R-Squared | Adjusted R-Squared | Predicted R-Squared | PRESS   |           |
|-----------|-----------|-----------|--------------------|---------------------|---------|-----------|
| Linear    | 2.817     | 0.748     | 0.721              | 0.655               | 303.666 |           |
| 2FI       | 2.163     | 0.867     | 0.835              | 0.796               | 179.583 |           |
| Quadratic | 1.547     | 0.940     | 0.916              | 0.850               | 131.916 | Suggested |
| Cubic     | 1.607     | 0.956     | 0.909              | 0.588               | 362.651 | Aliased   |

Table 4.4: Solid Gain Model Summary Statistics

| Source           | Std. Dev.    | R-Squared    | Adjusted R-Squared | Predicted R-Squared | PRESS         |                  |
|------------------|--------------|--------------|--------------------|---------------------|---------------|------------------|
| Linear           | 1.956        | 0.757        | 0.732              | 0.649               | 155.189       |                  |
| 2FI              | 1.109        | 0.930        | 0.914              | 0.868               | 58.166        |                  |
| <u>Quadratic</u> | <u>0.573</u> | <u>0.984</u> | <u>0.977</u>       | <u>0.965</u>        | <u>15.491</u> | <u>Suggested</u> |
| Cubic            | 0.610        | 0.987        | 0.974              | 0.922               | 34.392        | Aliased          |

### 4.1.1 Model Adequacy

The adequacy of the model both WL and SG in table 4.5 and table 4.6 was further checked with analysis of variance (ANOVA) as shown in (Table 4.5), based on a 95% confidence level, F-value is a test for comparing model variance with residual (error) variance.

Table 4.5: Analysis of variance (ANOVA) on Response 1(water loss)

|                 | Sum of  |    | Mean    | F       | p-value  |                 |
|-----------------|---------|----|---------|---------|----------|-----------------|
| Source          | Squares | Df | Square  | Value   | Prob > F |                 |
| Model           | 828.406 | 9  | 92.045  | 38.469  | < 0.0001 | significant     |
| A-Temperature   | 57.832  | 1  | 57.832  | 24.170  | < 0.0001 |                 |
| B-Concentration | 421.742 | 1  | 421.742 | 176.261 | < 0.0001 |                 |
| C-Time          | 179.258 | 1  | 179.258 | 74.918  | < 0.0001 |                 |
| AB              | 14.428  | 1  | 14.428  | 6.030   | 0.0224   |                 |
| AC              | 11.248  | 1  | 11.248  | 4.701   | 0.0412   |                 |
| BC              | 79.585  | 1  | 79.585  | 33.261  | < 0.0001 |                 |
| A <sup>2</sup>  | 62.297  | 1  | 62.297  | 26.036  | < 0.0001 |                 |
| B <sup>2</sup>  | 0.304   | 1  | 0.304   | 0.127   | 0.7247   |                 |
| C <sup>2</sup>  | 7.416   | 1  | 7.416   | 3.099   | 0.0922   |                 |
| Residual        | 52.640  | 22 | 2.393   |         |          |                 |
| Lack of Fit     | 47.412  | 17 | 2.789   | 2.667   | 0.1411   | Not significant |
| Pure Error      | 5.228   | 5  | 1.046   |         |          |                 |
| Cor Total       | 881.046 | 31 |         |         |          |                 |

The Model F-value of 38.469 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise (personal error or disturbance). Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case were A, B, C, AB,

AC, BC and A<sup>2</sup> were significant model terms. B<sup>2</sup> and C<sup>2</sup> P-Values greater than 0.1000 indicate the model terms are not significant.

The "Lack of Fit F-value" of 2.667 implies the Lack of Fit is not significant relative to the pure error. There is a 14.11% chance that a "Lack of Fit F-value" this large could occur due to noise. Non- significant lack of fit is good so that the model could fit. This shows that the soaking temperature, sugar concentration, soaking time, interaction between soaking temperature and sugar concentration, interaction between soaking temperature and time, sugar concentration and soaking time affects the percentage water loss significantly.

Table 4.6: Analysis' of variance (ANOVA) on response 2 (Solid Gain)

|                 | Sum of  |    | Mean   | F      | p-value  |                 |
|-----------------|---------|----|--------|--------|----------|-----------------|
| Source          | Squares | Df | Square | Value  | Prob > F | `               |
| Model           | 435.06  | 9  | 48.34  | 147.29 | < 0.0001 | Significant     |
| A-Temperature   | 22.45   | 1  | 22.45  | 68.39  | < 0.0001 |                 |
| B-Concentration | 194.37  | 1  | 194.37 | 592.24 | < 0.0001 |                 |
| C-Time          | 118.26  | 1  | 118.26 | 360.32 | < 0.0001 |                 |
| AB              | 1.90    | 1  | 1.90   | 5.78   | 0.0251   |                 |
| AC              | 1.85    | 1  | 1.85   | 5.64   | 0.0267   |                 |
| BC              | 72.68   | 1  | 72.68  | 221.46 | < 0.0001 |                 |
| A <sup>2</sup>  | 2.08    | 1  | 2.08   | 6.34   | 0.0196   |                 |
| B <sup>2</sup>  | 0.80    | 1  | 0.80   | 2.44   | 0.1326   |                 |
| C <sup>2</sup>  | 20.30   | 1  | 20.30  | 61.87  | < 0.0001 |                 |
| Residual        | 7.22    | 22 | 0.33   |        |          |                 |
| Lack of Fit     | 6.54    | 17 | 0.38   | 2.85   | 0.1256   | Not significant |
| Pure Error      | 0.68    | 5  | 0.14   |        |          |                 |
| Cor Total       | 442.28  | 31 |        |        |          |                 |

The Model F-value of 147.29 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise (personal error or disturbance). Values of

"Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A<sup>2</sup> and C<sup>2</sup> are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

This shows that the socking temperature, sugar concentration, socking time, interaction between socking temperature and sugar concentration, interaction between socking temperature and time, sugar concentration and socking time affects the percentage of solid gain significantly during osmotic dehydration.

The "Lack of Fit F-value" of 2.85 implies the Lack of Fit is not significant relative to the pure error. There is a 12.56 % chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good so that the model could fit.

Table 4.7: Verification code

| Response   | R-Squared | Adj R-Squared | Pred R-Squared | Adeq Precision | C.V. % |
|------------|-----------|---------------|----------------|----------------|--------|
| Water loss | 0.940     | 0.916         | 0.851          | 23.832         | 5.369  |
| Solid gain | 0.983     | 0.977         | 0.9649         | 45.317         | 7.265  |

The quality of the model developed could be evaluated from their coefficients of correlation. The value of R-Squared for WL developed correlation show in Table 4.7 was 0.940. It implies that 94.0% of the total variations in the percentage water loss were attributed to the experimental variables studied.

In the same SG of R-Squared for the developed correlation was 0.983. It implies that 98.30% solid gains were attributed to the experiment variable studied. The Predicted R-Squared of 0.851 and 0.965 is in reasonable agreement with the Adjusted R-Squared of 0.916 and 0.977 water loss and solid gain respectively. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. In this case, ratio of 23.832 and 45.353 indicates an adequate signal. This model can be used to navigate the design space.

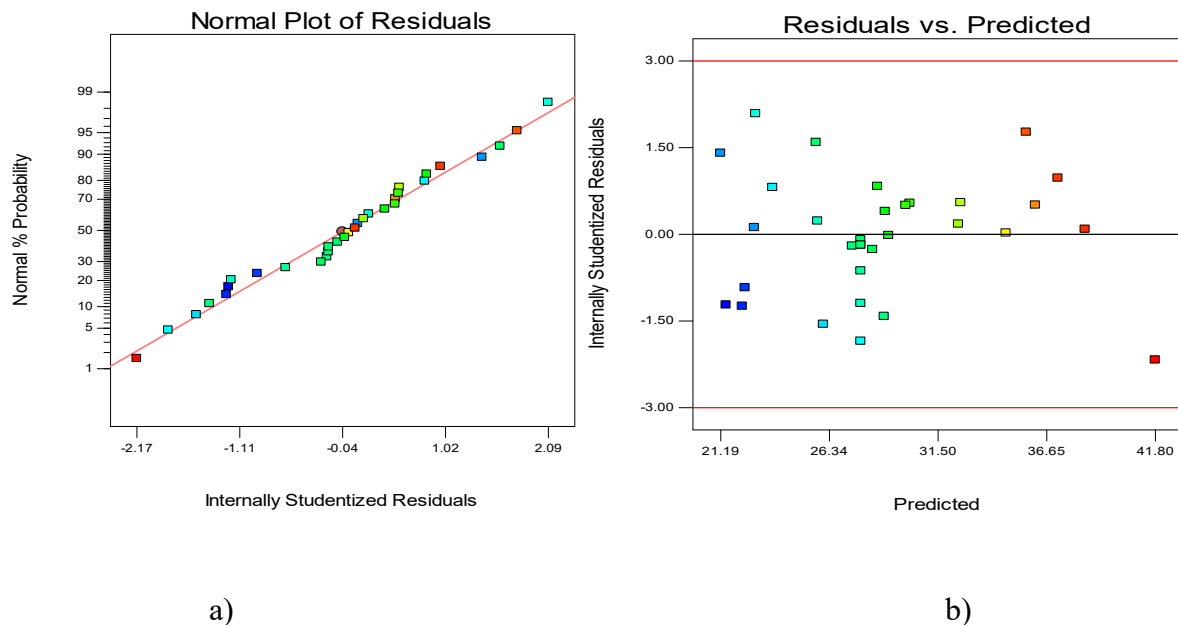
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### 4.1.2 Residual plots for water loss

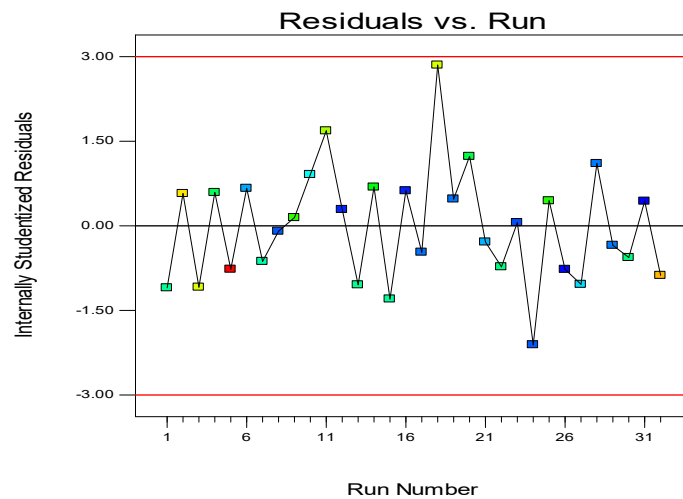
The graph of the predicted values obtained using the developed correlation predicted versus actual values is shown in Figure (4.1a & 4.2a). The results in Figure demonstrate that the regression model equation provided an acceptable description of the experimental data, in which most of the points are close to the line of perfect fit. This indicates that it was successful in capturing the correlation between the three osmotic dehydration process variables to the percentage of solid gain and water loss. As showed both the graphs 4.1 and 4.2 below, normal plot of residues, residual vs. predicted and residual vs. run tells the validity of the model with fitted data.

The residual vs. predicted plot figure 4.1b and figure 4.2b showed that no regular shape b/n the experimental data and the predicted value which shows that the data taken was collected randomly in the experiment. Residual Vs run order plot figure 4.1c and figure 4.2c is useful however only if data have been collected in randomized run order or some other order that is not increasing or decreasing in any of the predictor variables used in the model.

Therefore, residual vs run order graph of WL & SG figure below there no functional relationship between the predictor with percentage WL and SG which tells as the experimental data of yield was done in randomized order.







(c)

Figure 4.2: Diagnostic plot for solid gain response, a) normal plot of residues, (c) residual Vs predicted, c) residual Vs run

#### 4.1.4 Effect of Single Factor Variables on osmotic dehydration banana slice

Response surface methodologies were used to estimate the effect of three treating variables on osmotic dehydration the water loss and solid gain. Interaction plots were drawn by using 3-level factorial Design to investigate the effect of all the factors on the responses. Based on the analysis of variance, the single factor variables was strongly significant ( $p > 0.001$ ) for both water loss and sold gain response. Individual process variables soaking temperature, soaking time and osmotic solute that strongly affect the water loss and solid gain of slice banana significantly (Appendix B).

#### 4.1.5 Effect of Interaction between Process Variables

The most common way to summarize the results of response surface methodology (3-level full factorial) design experiment is in the form of three dimensional response surface plots and via response contour plot.

The process variables on both water loss and solid gain were found to have high significant interaction effects were observed between sugar concentration and soaking time, relatively less interaction effect were observed between soaking temperature and soaking time. Figure (4.3 & 4.4) shows the interaction effect of soaking temperature with sugar

concentration, the interaction effect of soaking temperature with soaking time( fig 4.5 & 4.6) and the interaction effect of sugar concentration with soaking time, respectively.

As shown in Figure (4.3) the maximum water loss had achieved at medium soaking temperature and higher sugar concentration, and minimum solid gain ( fig 4.4) had achieved at minimum soaking temperature & sugar concentration. In addition, it can be seen in Figure (4.5 ) that the maximum water loss had achieved at higher temperature and high soaking time, and minimum solid gain (fig 4.6) had achieved at minimum temperature and time. Figure (4.7) also shows that at higher sugar concentration and higher soaking time achieved maximum WL.

#### 4.1.5.1 Interaction Effect of Soaking Temperature and Sugar concentration on Water loss and Solid Gain

As shown in Figure 4.3 the maximum water loss had achieved at medium soaking temperature and higher sugar concentration.

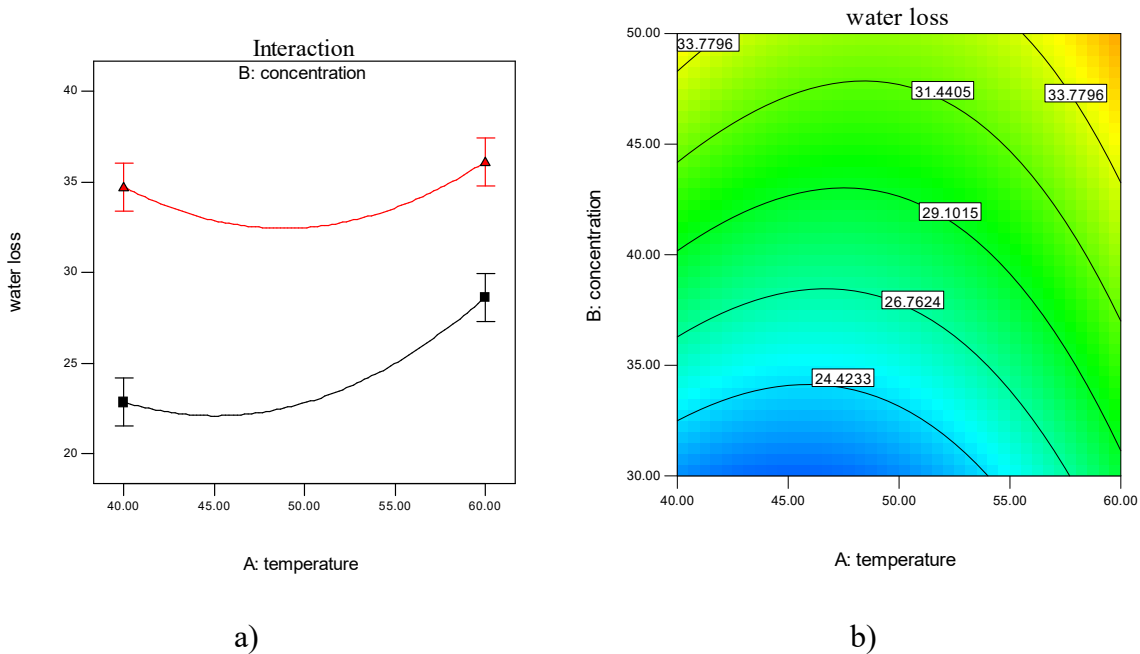


Figure 4.3: Interaction effect soaking temperature and sugar concentration versus WL when the soaking time at 60 min. a) interaction plot & b) contour plot

As shown in Figure (4.4) the minimum solid gain had achieved at minimum soaking temperature & sugar concentration.

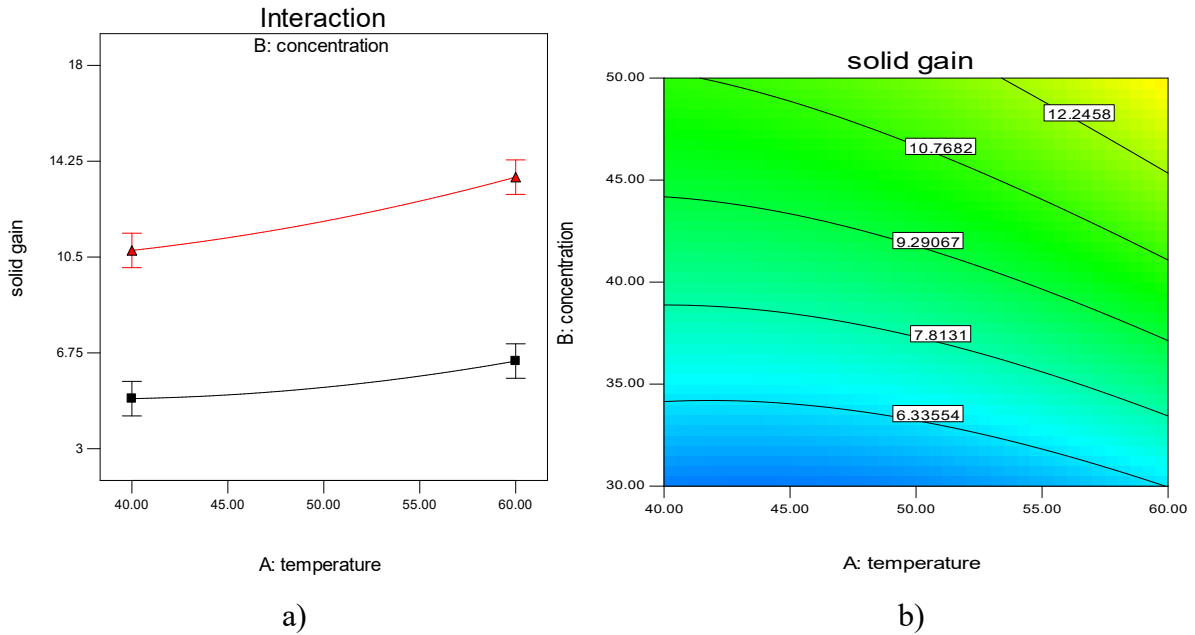


Figure 4.4 : interaction effect soaking temperature and sugar concentration versus solid gain when the soaking time at 60 min. a) interaction plot & b) Contour plot

#### 4.1.5.2 Interaction effect between soaking temperature and time on water loss and solid gain

As shown in Figure 4.5 surface interaction soaking time and temperature, the maximum water loss percentage had achieved at maximum soaking temperature and higher time.

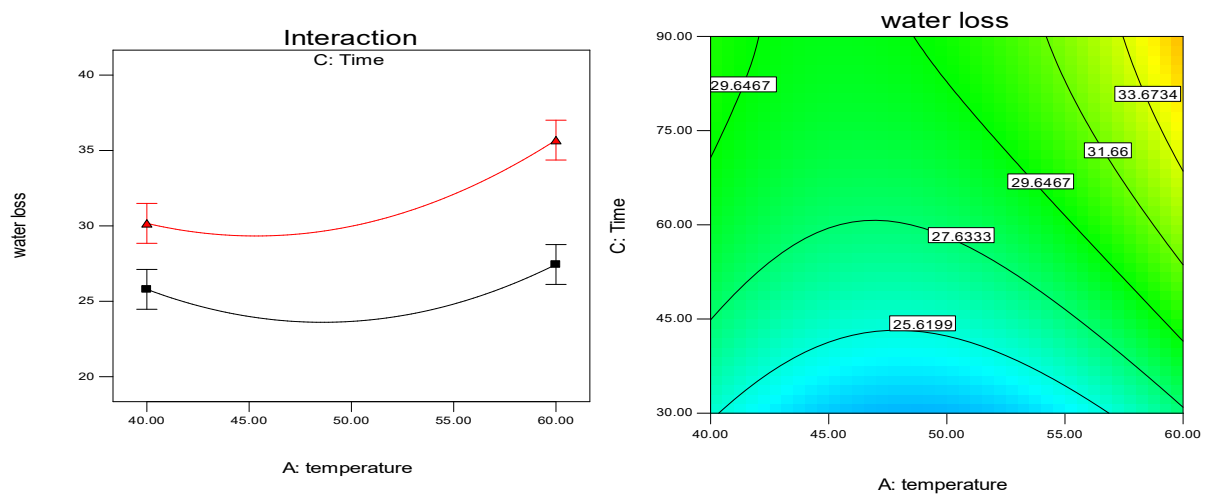


Figure 4.5: Contour plot of the interaction effect soaking temperature and time versus WL when the sugar concentration at 40 Brix

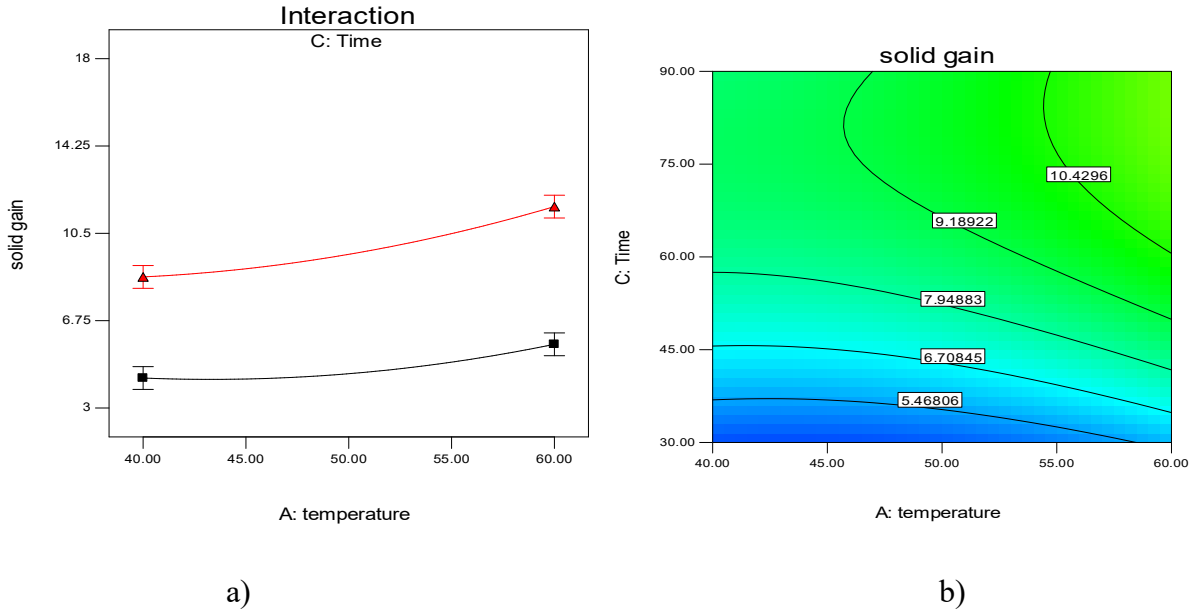
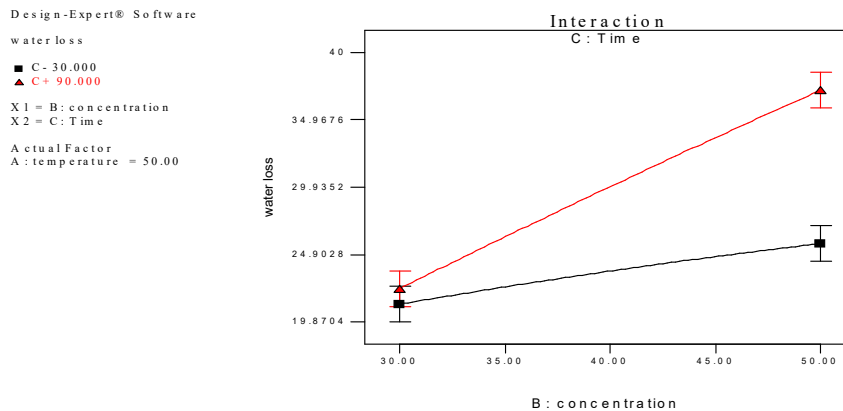


Figure 4.6 Contour plot of the interaction effect soaking temperature and time versus solid gain when the sugar concentration at 40<sup>0</sup>Brix. a) Interaction plot & b) contour plot

As shown in Figure 4.6 the minimum solid gain had achieved at lower soaking temperature and minimum time.

#### 4.1.5 .3 Interaction effect between soaking time and sugar concentration on WL and SG

The interaction effect both soaking time and concentration were strong in water loss as Figure 4.7 (a & b) the maximum water loss had achieved continuously with increase soaking time and concentration.



a) Interaction plot

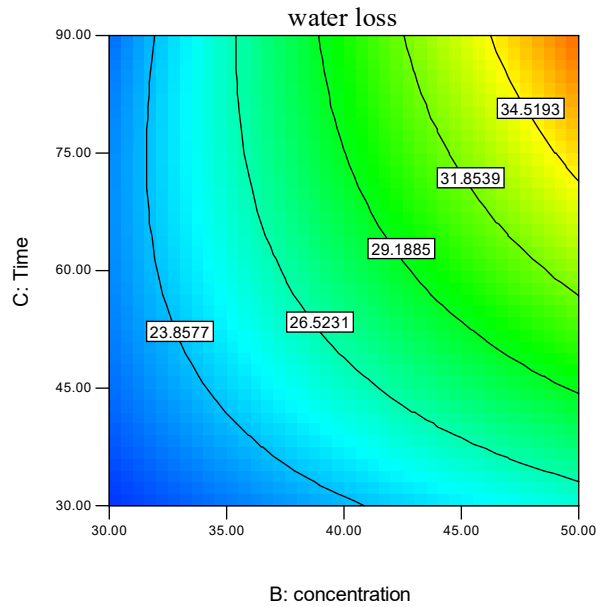
Design-Expert® Software

water loss



X1 = B: concentration  
X2 = C: Time

Actual Factor  
A: temperature = 50.00



b) Contour plot

Figure 4.7: interaction plot of the interaction effect sugar concentration and soaking time versus WL when the sugar temperature at 40 Brix. a) Interaction plot & b) contour plot

In as shown in Figure 4.8 3D surface interactions, percentage of water loss increase directly with increase soaking time and increasing concentration at constant temperature

Design-Expert® Software

water loss



X1 = B: concentration  
X2 = C: Time

Actual Factor  
A: temperature = 50.00

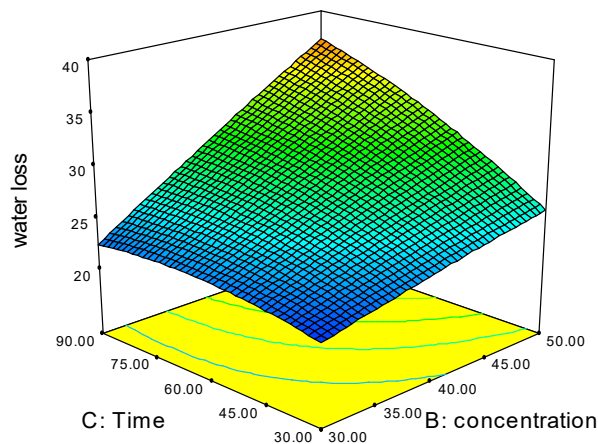


Figure 4.8 3D-surface of the interaction effect of sugar concentration and soaking time versus WL when the soaking temperature at 50°C

In as shown in Figure 4.9 3D surface interactions, percentage of solid gain reduce directly with decrease soaking time and decrease concentration at constant temperature 50 °C

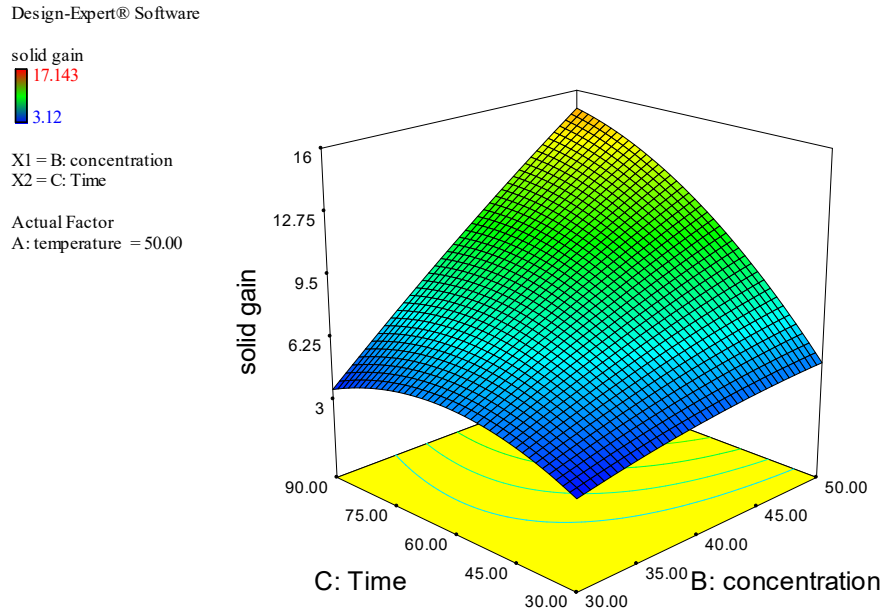


Figure 4.9: 3D-surface of the interaction effect of sugar concentration and soaking time versus SG when the soaking temperature at 50°C

#### 4.1.6 Development of Regression Model Equation

Experimental values were fitted to a second-order polynomial model Equation (4.1 and 4.2) and the model equation that correlates the water loss and solid gain response to osmotic dehydration process variables in terms of actual value after excluding the insignificant terms is given below. The predicted model for percentage of WL and SG in terms of the coded factors is given in Equation (4.1 and 4.2).

##### Final Equation in Terms of Coded Factors:

$$\text{Water loss} = +27.31 + 1.79 \times A + 4.84 \times B + 3.16 \times C - 1.1 \times A \times B + 0.97 \times A \times C + 2.58 \times B \times C + A^2 \dots \dots \dots (4.1)$$

---


$$\text{Solid gain} = +8.60 + 1.12 \times A + 3.29 \times B + 2.56 \times C + 0.4 \times A * B + 0.39 \times A \times C + 2.46 \times B \times C + 0.48 \times A^2 - 1.75 \times C^2 \dots \dots \dots (4.2)$$

Where:

A=Soaking temperature

B=Sugar concentration

C=Soaking time

#### 4.1.7 Optimization of process variables

The results above have shown that the three process variables and the interaction among the parameters affect the percentage water loss and solid gain osmotic dehydration. Therefore, the next step is optimizing the process variables in order to obtain the higher water loss and the minimum solid gain percentage using the developed model regression. In optimizing the enhancing water loss and reducing solid gain; soaking temperature and soaking time are a set of process variables held to be "in range" and sugar concentration set in "minimum" while percentage water loss and solid gain are set of responses that need to be "maximized" and "minimized" respectively.

Therefore, in order to obtain the maximum water loss and minimum solid gain, the predicted combination of parameters was as follows: soaking temperature 60 °C, sugar concentration of 30 °Brix and soaking time of 90 minutes. Under this conditions, the model predicted water loss of 29.1624% and solid gain of 5.664%.

Therefore, OD of banana slice could effectively used as a pretreatment prior to oven drying to reduce energy costs and maintain the quality & natural test of the product.

#### 4.2 Effects of pre treatment techniques and conventional oven drying temperature on characterization of banana flour

In this section discussed the effect of pre-treatment and drying temperature on quality characteristics of unripe banana flour. The pretreated banana slices were dry at three different temperatures (45, 60 and 75 °C) in conventional oven at constant 40-RPM fun speed to determine the influence of temperature and pre-treatment on nutritional quality of banana flour. The quality parameters investigated were nutritional content (moisture, ash, fat, protein, fiber, carbohydrate and energy value), physicochemical test ( TDS, pH, oil holding & water holding

capacity and non-enzymatic browning reaction ), mineral composition( all active metals and trace metals).

#### 4.2.1 Effect of treatments and drying temperature on banana flour quality

Table 4.8: Effect of pre-treatment and drying temperature on chemical composition of banana flour

| Name sample                                    | Temperature (°C) | Moisture % | Crude Protein % | Crude fat % | Ash %       | Crude Fiber % | Carbohydrate % |
|--|------------------|------------|-----------------|-------------|-------------|---------------|----------------|
| Banana flour Without any treatment             | 45               | 5.06±0.283 | 0.385±0.04      | 0.22±0.19   | 1.049±0.062 | 10.78±0.1     | 83.43±0.52     |
|  | 60               | 5.29±0.001 | 0.963±0.05      | 0.43±0.003  | 2.23±0.128  | 14.68±2.25    | 76.393±2.75    |
|  | 75               | 4.64±0.01  | 0.700±0.01      | 0.69±0.001  | 2.01±0.01   | 10.79±0.03    | 81.162±0.11    |
| Banana flour Sodium meta bisulphate pretreated | 45               | 6.38±0.01  | 0.350±0.02      | 0.32±0.001  | 2.35±0.01   | 11.1±0.01     | 79.5±0.11      |
|  | 60               | 5.1±0.01   | 0.263±0.02      | 0.46±0.01   | 1.94±0.01   | 13.89±0.12    | 78.4±0.11      |
|  | 75               | 3.3±0.01   | 0.26±0.02       | 0.664±0.01  | 1.41±0.01   | 11.71±0.02    | 82.66±0.02     |
| Banana flour Osmotically pretreated            | 45               | 4.79±0.01  | 0.31±0.06       | 0.302±0.01  | 1.80±0.13   | 10.58±0.02    | 82.21±0.11     |
|  | 60               | 2.92±0.06  | 0.53±0.02       | 0.332±0.01  | 1.26±0.06   | 12.33±0.1     | 82.03±0.2      |
|  | 75               | 2.25±0.035 | 0.44±0.04       | 0.446±0.03  | 0.71±0.02   | 10.79±0.04    | 85.24          |

Values are means of three replicates ± standard deviation.

From data in table 4.8, it could be noticed that, proximate composition was gradually increased by increasing of processing temperature. The moisture content of sample in the given parameter was in the range of 2.249-6.521%. Maximum moisture content was observed in hot water pretreated banana slice dried in oven at 45<sup>0</sup>C sample (6.521%) and minimum was in osmotically pretreated sample (2.249%) dried at 75<sup>0</sup>C. The ash content of sample in the given parameter was in the range of 0.711-2.459%. Maximum ash content was observed in hot water pretreated banana slice dried in oven at 45<sup>0</sup>C sample (2.459%) and minimum was in osmotically pretreated sample (0.711%) dried at 75<sup>0</sup>C. The fat content of sample in the given parameter was in the range of 0.222-0.695%. Maximum fat content was observed in hot water pretreated banana slice dried in oven at 75<sup>0</sup>C sample (0.695%) and minimum was in un-treated sample (0.222%) dried at 45<sup>0</sup>C. Proximate analysis of untreated and pretreated banana flour showed that value of ash, crude fiber, fat, protein carbohydrate and gross energy content had significant difference ( $p < 0.05$ ). In addition as showed in figure 4.10, Energy values in the given parameter maximum in osmotically pretreated sample (345.901kcal) dried at 75<sup>0</sup>C and minimum in hot water pretreated sample (313kcal)

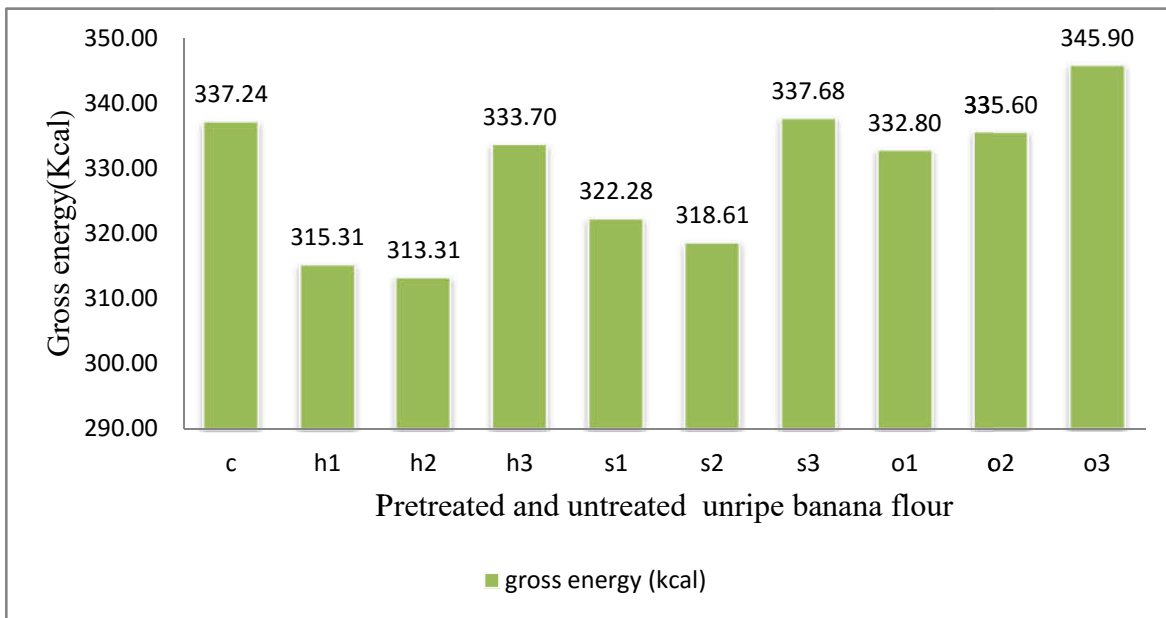


Figure 4.10: Energy value pretreated and untreated banana flour at different temperature

Symbols notations

*c* = Unripe Banana flour, without any treatment and dried at 45<sup>0</sup>C

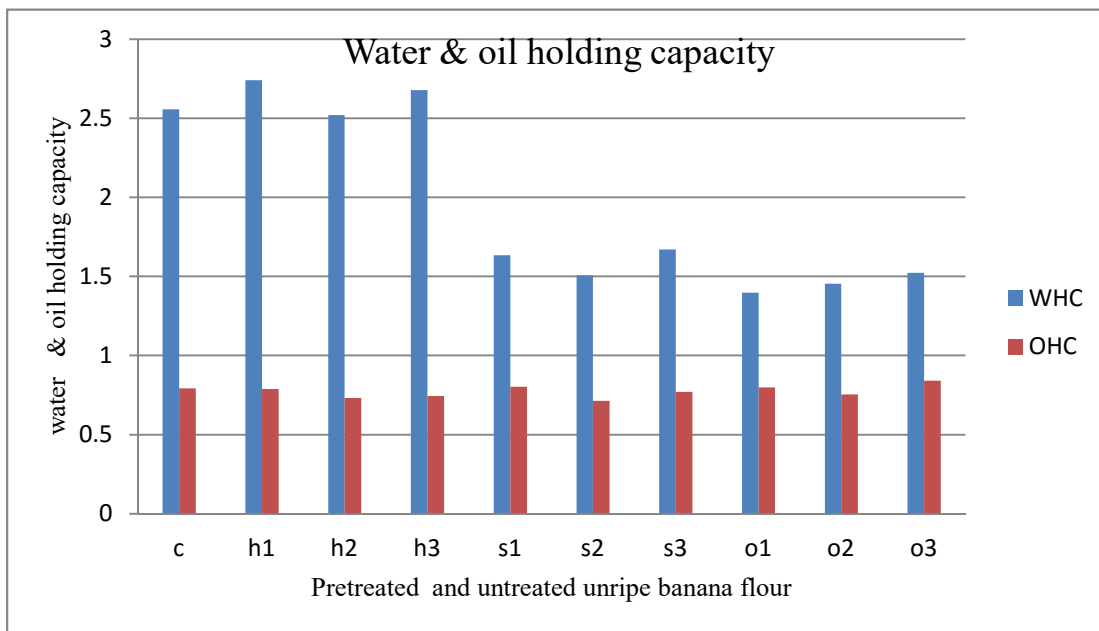
*H1, h2 & h3* = Unripe Banana flour, hot water pretreated and dried at 45, 60 & 75<sup>0</sup>C respectively.

*S1,s2 & s3 = Unripe Banana flour, sodium metabisulphit pretreatment and dried at 45, 60 & 75 °C respectively*

*O1, o2, & o3 =Unripe banana flour, osmotic dehydration pretreated and dried at 45, 60 & 75 °C respectively*

#### 4.2.2 Effect of pre-treating and drying temperature on water and oil holding capacity banana flour

Water and oil holding capacity related with the absorption of product at specific condition and it's could be related to the physical state of starch (Waliszewski *et al.*, 2003), dietary fiber and protein in the flour. Figure (4.11) represents the WHC & OHC of pretreated and untreated of unripe banana flour unripe banana flour there has slightly significant difference. The mean values of WHC & OHC prepared sample are  $2.0049 \pm 0.576$  and  $0.754 \pm 0.046$  respectively. WHC found is higher in hot water pretreated sample dried at  $45^{\circ}\text{C}$ . In addition, OHC found there is no significant difference. OHC is characterized by the hydrophilic tendency of the starches present in the flour (Rodríguez *et al.*, 2008). Higher water and oil holding capacity flour is preferable food preparation and mixing with other ingredients. According to (Anon, 1990) a range of WHC from 1.491 to 4.715 is consider as favorable for the preparation of viscous foods such as soups, gravies and bakery products.



*Water holding capacity g water / g dry sample and oil holding capacity g oil / g dry sample*

Figure 4.11: Water and oil holding capacity pretreated and untreated unripe banana flour

##### Symbols notations

*c = Unripe Banana flour, without any treatment and dried at  $45^{\circ}\text{C}$*

*h1, h2 & h3= Unripe Banana flour, hot water pretreated and dried at 45, 60 &  $75^{\circ}\text{C}$  respectively.*

*S1, s2 & s3= Unripe Banana flour, sodium metabisulphit pretreatment and dried at 45, 60 &  $75^{\circ}\text{C}$  respectively*

*O1, o2 & o3 =Unripe banana flour, osmotic dehydration pretreated and dried at 45, 60 &  $75^{\circ}\text{C}$  respectively*

### 4.2.3 Effect of pre-treating and drying temperature on total dissolving solid (TDS) & pH of banana flour

The mean pH of flours ranged between 5.14 to 6.224 and osmotic dehydrated banana flour showed the highest pH whilst sodium meta bisulphate pretreated banana flour showed the lowest, though the differences all the flours were statistically significant (figure 4.12).

The mean of TSS of banana flour ranged between 1.333 to 3.866 and osmotic dehydrated banana flour had the maximum TSS of 3.866 °Brix. While others banana except OD flour showed as figure 4.12 there is no significant TDS difference. The lower TDS of green banana flour is acceptable since it is known that amylase, glycosidase, phosphorylase, sucrose synthase and invertase can act in the degradation of starch and the formation and accumulation of soluble sugars (Emaga *et al.*, 2007; Terra, Garcia and Lajolo,1983).

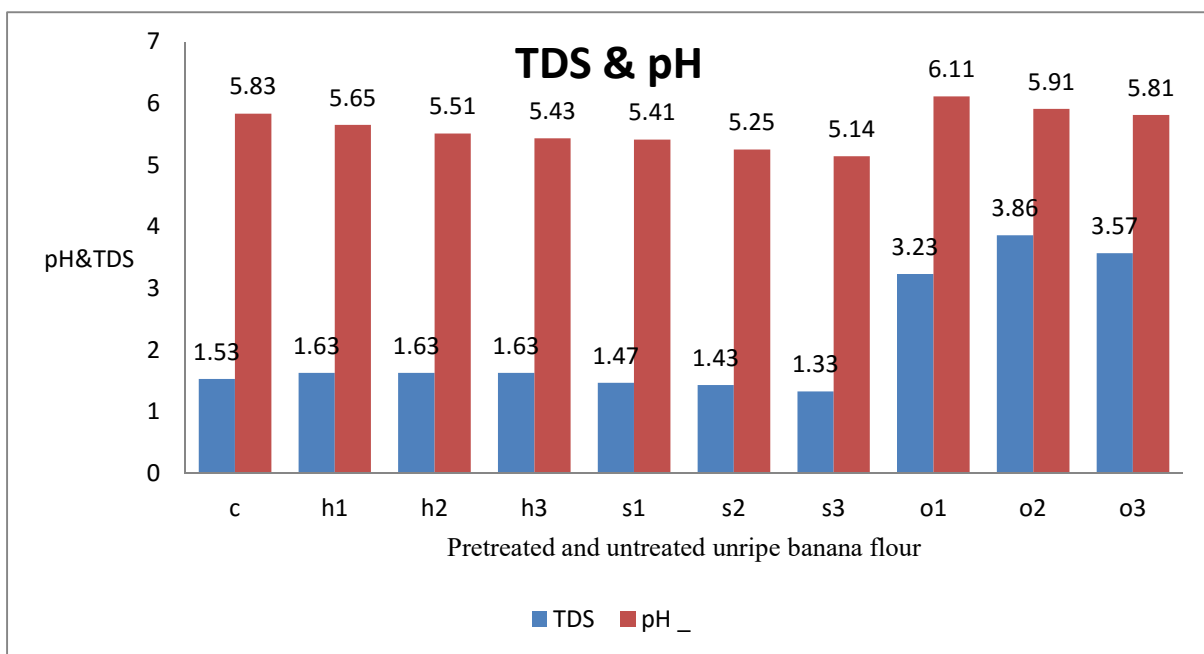


Figure 4.12: Effect of pretreatment on TDS and pH unripe banana flour

#### Symbols notations

*c* = Unripe Banana flour, without any treatment and dried at 45 °C

*h1, h2 & h3*= Unripe Banana flour, hot water pretreated and dried at 45, 60 & 75 °C respectively.

*S1, s2 & s3*= Unripe Banana flour, sodium metabisulphit pretreatment and dried at 45, 60 & 75 °C respectively

*O1, o2 & o3* =Unripe banana flour, osmotic dehydration pretreated and dried at 45, 60 & 75 °C respectively

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#### 4.2.4 Effect of pre-treating and drying temperature on non enzymatic browning in banana flour

Non-enzymatic browning (NEB) found is less in SMB pretreated sample and more in osmotic dehydrated samples. Increase NEB (Millard reaction) produces dark pigments and destroys the natural color of products (Poretta & Sandei, 1990; Nguyen & Schwartz, 1998).

As showed the present SMS pretreated samples had low NEB and, high tendency to protect color retention.

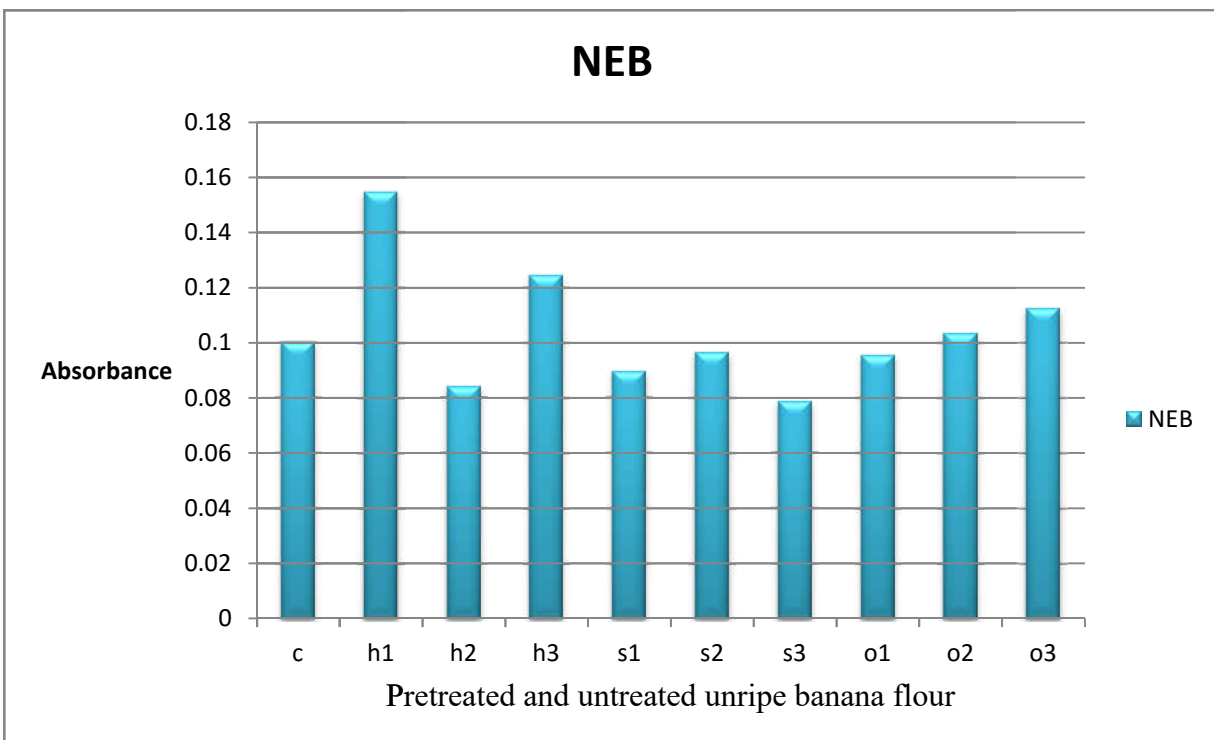


Figure 4.13: Effect pretreatment on non-enzymatic browning unripe banana flour

##### Symbols notations

*c = Unripe Banana flour, without any treatment and dried at 45 °C*

*h1, h2 & h3= Unripe Banana flour, hot water pretreated and dried at 45, 60 & 75 °C respectively.*

*S1, s2 & s3= Unripe Banana flour, sodium metabisulphit pretreatment and dried at 45, 60 & 75 °C respectively*

*O1, o2 & o3 =Unripe banana flour, osmotic dehydration pretreated and dried at 45, 60 & 75 °C respectively*

#### 4.2.5 Composition of minerals in pretreated and untreated banana flour

##### 4.2.5.1 Composition of active metals pretreated and untreated banana flour

In present study showed values of the mineral compositions with varying amounts of minerals such as potassium, sodium, magnesium and calcium. The highest mean value mineral was

potassium with 711.678mg/100g followed by magnesium 213.778mg/100g and Calcium 43.57mg/100g. The least value was recorded for sodium 22.0171 mg/100g with followed by Calcium 43.57mg/100g. Potassium, calcium, magnesium and iron values was highest in hot water pretreated dried at 75<sup>0</sup>C flour sample. In addition, lowest composition (Ca, Na, K and Mg and Fe) showed in osmotic dehydrated pretreated and dried at 45 <sup>0</sup>C unripe banana flours.

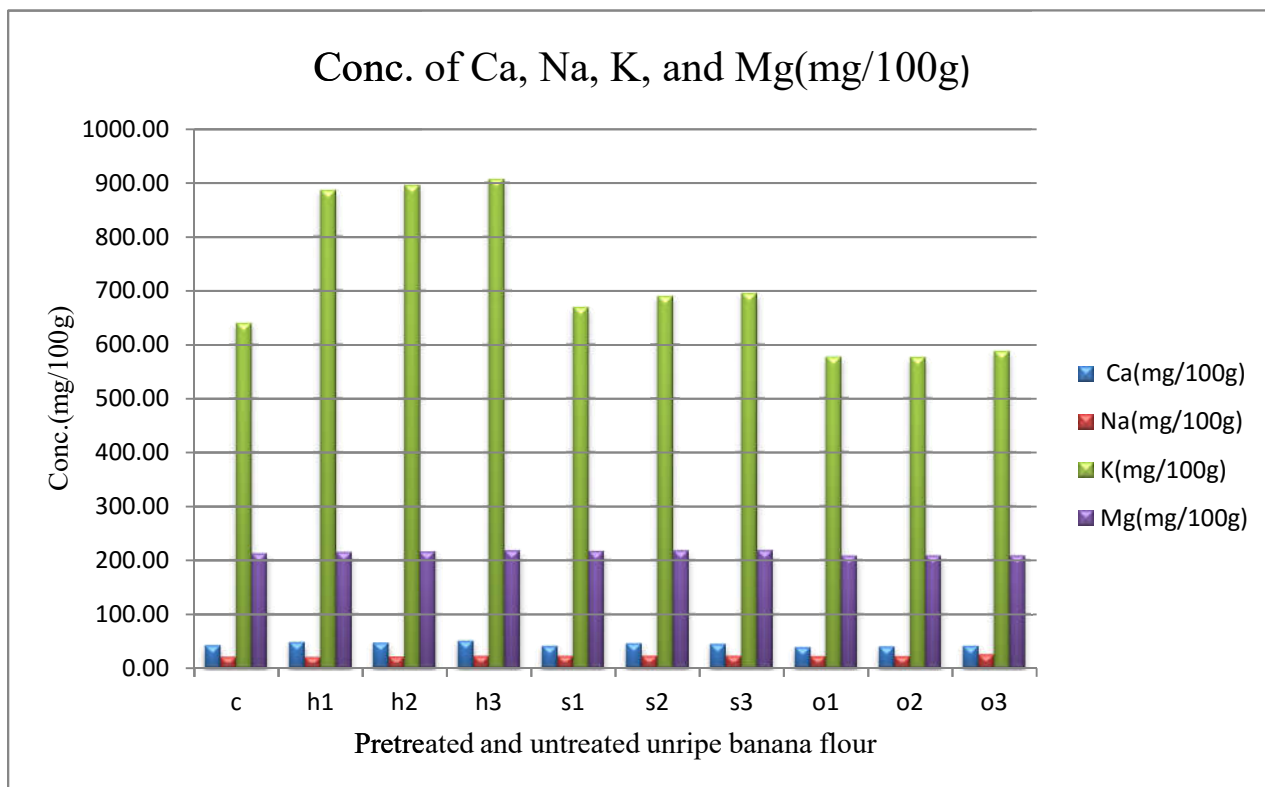


Figure 4.14: Effect of pretreatments the concentration of Ca, Mg, Na and K in unripe banana flour

Symbols notations

*c* = Unripe Banana slice without any treatment and dried at 45 <sup>0</sup>C

*h1, h2 & h3*= Unripe Banana hot water pretreated and dried at 45, 60 & 75 <sup>0</sup>C respectively.

*s1, s2 & s3*= Unripe Banana slice sodium metabisulphit pretreatment and dried at 45, 60 & 75 <sup>0</sup>C respectively

*o1, o2 & o3* =Unripe banana slice osmotic dehydration pretreated and dried at 45, 60 & 75 <sup>0</sup>C respectively

**4.2.5.2 Composition of trace metals pretreated and untreated banana flour**

In present study showed values of the trace metals composition with varying amounts such as Zink, Iron, Cadmium, lead and chromium. In these, study toxic metals (Cd, Pd and Cr) not detected both pretreated and untreated unripe banana flour excluded SMS pretreated flour in

such small amount Cd, Pd, and Cr detected. Amounts of Zn and Fe were highest in osmotically pretreated sample (2.19mg/100g) and hot water pretreated sample (2.101 mg/100g) respectively.

Table 4.9: Effect of pretreatments the concentration of Zn, Fe, Cd, Pd and Cr in unripe banana flour

| Name of sample | Zn(mg/100g) | Fe(mg/100g) | Cd(mg/100g) | Pd(mg/100g) | Cr(mg/100g) |
|----------------|-------------|-------------|-------------|-------------|-------------|
| c              | 0.745       | 1.394       | ND          | ND          | ND          |
| h1             | 0.2988      | 1.731       | ND          | ND          | ND          |
| h2             | 0.294       | 2.096       | ND          | ND          | ND          |
| h3             | 0.924       | 2.101       | ND          | ND          | ND          |
| S1             | 1.677       | 1.470       | 0.024       | ND          | ND          |
| S2             | 1.701       | 1.745       | 0.0345      | ND          | ND          |
| S3             | 1.983       | 1.606       | 0.046       | ND          | ND          |
| O1             | 2.124       | 1.110       | ND          | ND          | ND          |
| O2             | 2.198       | 1.522       | ND          | ND          | ND          |
| O3             | 2.1882      | 1.683       | ND          | ND          | ND          |

Symbols notations

*c = Unripe Banana slice without any treatment and dried at 45 °C*

*h1, h2 & h3= Unripe Banana hot water pretreated and dried at 45, 60 & 75 °C respectively.*

*S1, s2 & s3= Unripe Banana slice sodium metabisulphit pretreatment and dried at 45, 60 & 75 °C respectively*

*O1, o2 & o3 =Unripe banana slice osmotic dehydration pretreated and dried at 45, 60 & 75 °C respectively*

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## CHAPTER 5

### 5. CONCLUSIONS AND RECOMMENDATIONS

#### 5.1 Conclusions

Banana has a limited shelf life at ambient conditions and is highly perishable. Its physicochemical properties cause a high post-harvest loss during production season and become scarce during the off-season. Short shelf life coupled with inadequate processing facilities and backward fruit handling results in high-income loss to the country.

The objective of this research was to determine the effect of pretreatments and drying temperature during preparation of unripe banana flour. The drying process was followed after giving three different pre-treatments such as osmotic dehydration, sodium metabisulfite (SMS), and hot water (at 100 °C). The optimum osmotic dehydration conditions for maximum water loss and minimum solid gain were obtained at 30 °Brix sugar concentration, 60 °C soaking temperature and 90 min soaking time, which resulted in 29.16% water loss and 5.66% solid gain. In the present work, it was demonstrated that pretreatments and drying process significantly changed the nutritional and physicochemical value of the unripe banana flour. The average proximate composition shows that the crude protein (0.641%), crude fiber (12.80%), crude fat (0.57%), ash (2.23%) and moisture (5.48%) contents of hot water pretreated banana flour were more than sodium metabisulfite and osmotic dehydration pretreatments. However, carbohydrate (83.16%) and energy (338.09 kcal) values of osmotically pretreated banana flour were higher than hot water and sodium metabisulfite pretreatments.

On the other hand, hot water pretreated unripe banana flour shows higher average water holding capacity and not significant change in oil holding capacity than other pretreatments. In addition, osmotically pretreated flour shows higher average TDS (3.55) and pH (5.94) values. Moreover, Non-enzymatic browning (NEB) found was less in SMS pretreated sample and more in osmotic dehydrated samples. The mean potassium (895.768mg/100g), calcium (48.366mg/100g), magnesium (216.49mg/100g), and iron values were highest in hot water pretreated flour sample. In addition, study toxic metals (Cd, Pb and Cr) were not detected in both pretreated and untreated unripe banana flour. However, SMS pretreated flour had such small amounts of Cd, Pb, and Cr detected. Amounts of Zn and Fe were highest in osmotically pretreated sample (2.19mg/100g) and hot water pretreated sample (2.101 mg/100g) respectively. This concludes that pretreatments and

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drying process affect the quality characteristics of unripe banana flour and could increase: the quality of the flour, shelf life, market stability, staple food option and would increase other alternative utilization. Moreover, it will help the food industry to select the appropriate pretreatment and drying processes for produce unripe banana flour products.

## **5.2 Recommendation**

- ✓ Combining pretreatments effect between osmotic solution with sodium meta bisulphit , sodium meta bisulphit with boiled water and boiled water with osmotic solution needs further investigation
- ✓ It is recommended that further research using other pretreatments chemicals and drying methods to produce unripe banana flour
- ✓ To recommended that further research in osmotic solution processing variables such as concentration, socking time and socking time.
- ✓ It is suggested to establish recycle methods to recover osmotic solute (sugar )
- ✓ To perform comprehensive study on shelf life stability and microbial analysis tests in order to determine quality of banana flour
- ✓ The economic feasibility and healthy benefit of this treatment method needs further investigation to confirm its advantages compared to other treatments
- ✓ Blending unripe banana flour other flours needs further research.
- ✓ Further researches is required to produce other value added product from banana peels and bunch waste like bio ethanol, starch, animals feed and activated carbon

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**APPENDIX A: Anova Results From Design- Expert**

**Table A1:** Diagnostics Case Statistics. Response one ( water loss )

| Stand<br>ard<br>Order | Actu<br>al<br>Valu<br>e | Predict<br>ed<br>Value | Resid<br>ual | Levera<br>ge | Internall<br>y<br>Studenti<br>zed<br>Residual | Externall<br>y<br>Studenti<br>zed<br>Residual | Influence on<br>Fitted<br>Value<br>DFFITS | Cook'<br>s<br>Distan<br>ce | Run<br>Ord<br>er |
|-----------------------|-------------------------|------------------------|--------------|--------------|---|---|---|----------------------------|------------------|
| 1                     | 20.86                   | 22.22                  | -1.36        | 0.50         | -1.24   | -1.26   | -1.25                                     | 0.15                       | 26               |
| 2                     | 22.96                   | 21.19                  | 1.77         | 0.34         | 1.40  | 1.44  | 1.03                                      | 0.10                       | 16               |
| 3                     | 24.36                   | 26.06                  | -1.71        | 0.50         | -1.56   | -1.61   | -1.60                                     | 0.24                       | 23               |
| 4                     | 26.08                   | 25.79                  | 0.29         | 0.34         | 0.23  | 0.23  | 0.16                                      | 0.00                       | 28               |
| 5                     | 24.78                   | 23.66                  | 1.11         | 0.21         | 0.81  | 0.80  | 0.42                                      | 0.02                       | 19               |
| 6                     | 27.18                   | 27.44                  | -0.26        | 0.34         | -0.20   | -0.20   | -0.14                                     | 0.00                       | 21               |
| 7                     | 27.39                   | 28.95                  | -1.56        | 0.50         | -1.42   | -1.46   | -1.45                                     | 0.20                       | 8                |
| 8                     | 27.72                   | 25.72                  | 2.00         | 0.34         | 1.59  | 1.65  | 1.19                                      | 0.13                       | 29               |
| 9                     | 28.11                   | 28.40                  | -0.29        | 0.50         | -0.26   | -0.26   | -0.26                                     | 0.01                       | 27               |
| 10                    | 25.48                   | 22.85                  | 2.63         | 0.34         | 2.09  | 2.28  | 1.63                                      | 0.22                       | 17               |
| 11                    | 22.95                   | 22.79                  | 0.16         | 0.21         | 0.12  | 0.11  | 0.06                                      | 0.00                       | 6                |
| 12                    | 29.67                   | 28.63                  | 1.04         | 0.34         | 0.83  | 0.82  | 0.59                                      | 0.04                       | 10               |
| 13                    | 29.54                   | 29.00                  | 0.54         | 0.21         | 0.40  | 0.39  | 0.20                                      | 0.00                       | 7                |
| 14                    | 27.70                   | 27.84                  | -0.14        | 0.11         | -0.09   | -0.09   | -0.03                                     | 0.00                       | 4                |
| 15                    | 33.33                   | 32.58                  | 0.75         | 0.21         | 0.55  | 0.54  | 0.28                                      | 0.01                       | 25               |
| 16                    | 34.76                   | 34.73                  | 0.03         | 0.34         | 0.02  | 0.02  | 0.02                                      | 0.00                       | 9                |
| 17                    | 32.72                   | 32.47                  | 0.24         | 0.21         | 0.18  | 0.17  | 0.09                                      | 0.00                       | 18               |
| 18                    | 36.76                   | 36.12                  | 0.64         | 0.34         | 0.51  | 0.50  | 0.36                                      | 0.01                       | 2                |
| 19                    | 20.11                   | 21.45                  | -1.34        | 0.50         | -1.22   | -1.24   | -1.23                                     | 0.15                       | 31               |
| 20                    | 21.19                   | 22.35                  | -1.16        | 0.34         | -0.92   | -0.92   | -0.66                                     | 0.04                       | 12               |
| 21                    | 29.14                   | 29.16                  | -0.02        | 0.50         | -0.02   | -0.02   | -0.02                                     | 0.00                       | 24               |
| 22                    | 30.84                   | 30.17                  | 0.68         | 0.34         | 0.54  | 0.53  | 0.38                                      | 0.01                       | 20               |
| 23                    | 30.67                   | 29.98                  | 0.69         | 0.21         | 0.50  | 0.49  | 0.26                                      | 0.01                       | 14               |
| 24                    | 37.91                   | 35.69                  | 2.22         | 0.34         | 1.77  | 1.86  | 1.34                                      | 0.16                       | 11               |
| 25                    | 38.57                   | 38.47                  | 0.10         | 0.50         | 0.09  | 0.09  | 0.09                                      | 0.00                       | 3                |
| 26                    | 38.41                   | 37.18                  | 1.22         | 0.34         | 0.97  | 0.97  | 0.70                                      | 0.05                       | 32               |
| 27                    | 39.42                   | 41.80                  | -2.38        | 0.50         | -2.17   | -2.40   | * -2.38                                   | 0.47                       | 5                |
| 28                    | 25.15                   | 27.84                  | -2.69        | 0.11         | -1.85   | -1.96   | -0.70                                     | 0.04                       | 1                |
| 29                    | 26.10                   | 27.84                  | -1.74        | 0.11         | -1.20   | -1.21   | -0.43                                     | 0.02                       | 22               |
| 30                    | 26.92                   | 27.84                  | -0.92        | 0.11         | -0.63   | -0.62   | -0.22                                     | 0.01                       | 30               |
| 31                    | 27.56                   | 27.84                  | -0.27        | 0.11         | -0.19   | -0.18   | -0.07                                     | 0.00                       | 15               |
| 32                    | 27.57                   | 27.84                  | -0.27        | 0.11         | -0.19   | -0.18   | -0.06                                     | 0.00                       | 13               |

**Table A2: Diagnostics Case Statistics. Response two (Solid gain)**

| Standar<br>Order | Actual<br>Value | Predicted<br>Value | Residual | Leverage | Internall<br>Studentized<br>Residual | Externall<br>Studentized<br>Residual | Influence<br>on<br>Fitted<br>Value<br>DFFITS | Cook's<br>Distan<br>ce | Run<br>Order |
|------------------|-----------------|--------------------|----------|----------|--------------------------------------|--------------------------------------|--|------------------------|--------------|
| 1                | 3.21            | 3.524              | -0.314   | 0.497    | -0.772                               | -0.765                               | -0.761                                       | 0.059                  | 26           |
| 2                | 3.60            | 3.311              | 0.289    | 0.340    | 0.621                                | 0.612                                | 0.439  | 0.020                  | 16           |
| 3                | 4.20            | 4.177              | 0.023    | 0.497    | 0.058                                | 0.056                                | 0.056  | 0.000                  | 23           |
| 4                | 4.80            | 4.286              | 0.514    | 0.340    | 1.104                                | 1.110                                | 0.796  | 0.063                  | 28           |
| 5                | 4.71            | 4.470              | 0.242    | 0.211    | 0.475                                | 0.467                                | 0.242  | 0.006                  | 19           |
| 6                | 5.60            | 5.734              | -0.134   | 0.340    | -0.287                               | -0.281                               | -0.202                                       | 0.004                  | 21           |
| 7                | 4.34            | 4.379              | -0.039   | 0.497    | -0.096                               | -0.093                               | -0.093                                       | 0.001                  | 8            |
| 8                | 4.80            | 4.961              | -0.161   | 0.340    | -0.345                               | -0.338                               | -0.243                                       | 0.006                  | 29           |
| 9                | 6.20            | 6.622              | -0.422   | 0.497    | -1.038                               | -1.040                               | -1.034                                       | 0.107                  | 27           |
| 10               | 4.70            | 4.918              | -0.218   | 0.340    | -0.468                               | -0.459                               | -0.329                                       | 0.011                  | 17           |
| 11               | 5.43            | 5.098              | 0.338    | 0.211    | 0.665                                | 0.657                                | 0.340  | 0.012                  | 6            |
| 12               | 6.78            | 6.356              | 0.424    | 0.340    | 0.910                                | 0.907                                | 0.650  | 0.043                  | 10           |
| 13               | 7.82            | 8.141              | -0.321   | 0.211    | -0.631                               | -0.622                               | -0.322                                       | 0.011                  | 7            |
| 14               | 9.03            | 8.718              | 0.319    | 0.113    | 0.591                                | 0.582                                | 0.208  | 0.004                  | 4            |
| 15               | 10.6            | 10.374             | 0.226    | 0.211    | 0.444                                | 0.435                                | 0.225  | 0.005                  | 25           |
| 16               | 10.7            | 10.695             | 0.068    | 0.340    | 0.146                                | 0.143                                | 0.102  | 0.001                  | 9            |
| 17               | 13.1            | 11.670             | 1.450    | 0.211    | 2.851                                | 3.507                                | 1.816  | 0.218                  | 18           |
| 18               | 13.9            | 13.723             | 0.267    | 0.340    | 0.573                                | 0.564                                | 0.404  | 0.017                  | 2            |
| 19               | 3.12            | 2.942              | 0.178    | 0.497    | 0.437                                | 0.429                                | 0.427  | 0.019                  | 31           |
| 20               | 3.65            | 3.515              | 0.135    | 0.340    | 0.290                                | 0.284                                | 0.204  | 0.004                  | 12           |
| 21               | 4.31            | 5.166              | -0.856   | 0.497    | -2.108                               | -2.306                               | * -2.29                                      | 0.440                  | 24           |
| 22               | 9.20            | 8.627              | 0.573    | 0.340    | 1.232                                | 1.247                                | 0.894  | 0.078                  | 20           |
| 23               | 9.94            | 9.597              | 0.349    | 0.211    | 0.687                                | 0.678                                | 0.351  | 0.013                  | 14           |
| 24               | 12.4            | 11.645             | 0.785    | 0.340    | 1.685                                | 1.764                                | 1.265  | 0.146                  | 11           |
| 25               | 13.2            | 13.642             | -0.442   | 0.497    | -1.088                               | -1.092                               | -1.087                                       | 0.117                  | 3            |
| 26               | 14.6            | 15.009             | -0.409   | 0.340    | -0.879                               | -0.874                               | -0.627                                       | 0.040                  | 32           |
| 27               | 17.3            | 17.456             | -0.313   | 0.497    | -0.770                               | -0.762                               | -0.758                                       | 0.059                  | 5            |
| 28               | 8.13            | 8.718              | -0.593   | 0.113    | -1.099                               | -1.105                               | -0.394                                       | 0.015                  | 1            |
| 29               | 8.36            | 8.71               | -0.392   | 0.113    | -0.727                               | -0.719                               | -0.256                                       | 0.007                  | 22           |
| 30               | 8.41            | 8.718              | -0.303   | 0.113    | -0.562                               | -0.553                               | -0.197                                       | 0.004                  | 30           |
| 31               | 8.02            | 8.718              | -0.700   | 0.113    | -1.298                               | -1.319                               | -0.471                                       | 0.021                  | 15           |
| 32               | 8.15            | 8.718              | -0.564   | 0.113    | -1.046                               | -1.048                               | -0.374                                       | 0.014                  | 13           |

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## APPENDIX B: Single factor effect

### B1) Effect of Temperature on Water loss & solid gain in osmotic dehydration

As shown in Figure (B1 & B2) the percentage WL and SG removal were significantly affected by soaking temperature respectively. An increase in percentage WL & SG with an increase in temperature. However, SG increases more linearly than WL.

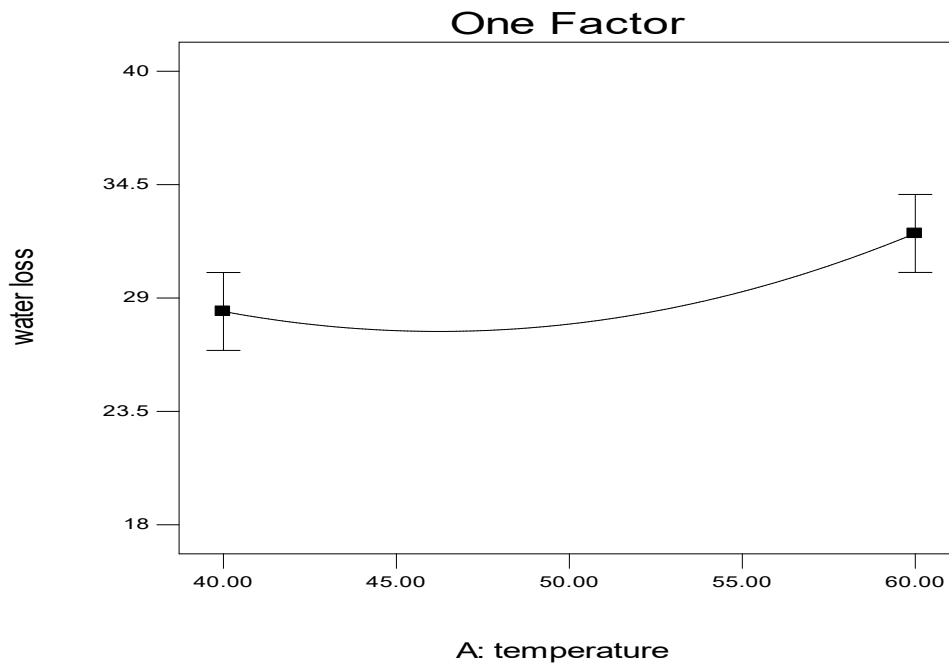


Figure B1.1: Effect of temperature on water loss in osmotic dehydration banana slice

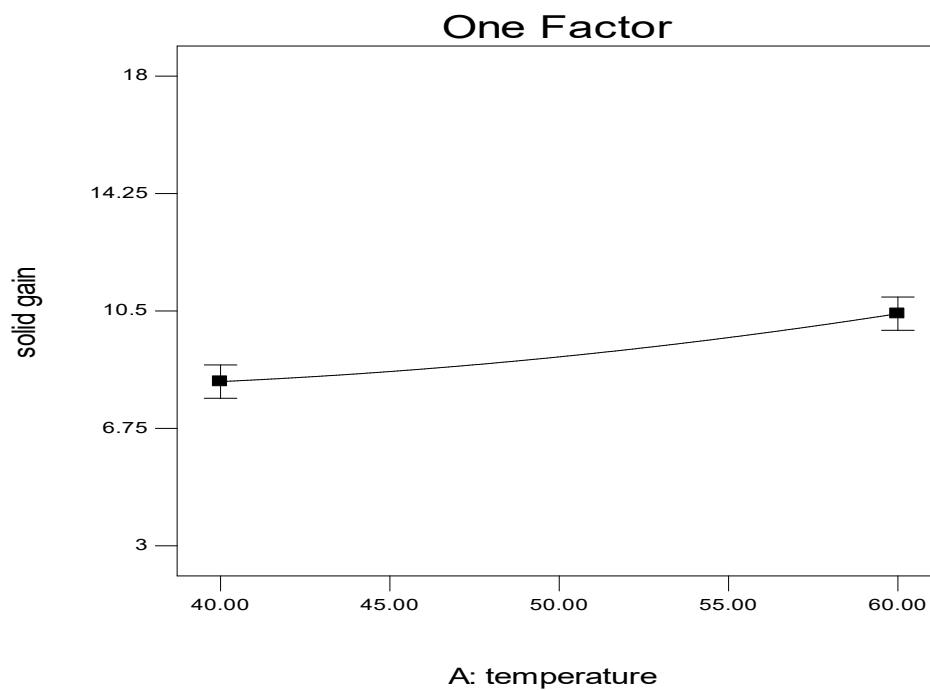


Figure B1.2: Effect of temperature on solid gain in osmotic dehydration banana slice

**B2) Effect of concentration on Water loss & solid in osmotic dehydration**

As shown in Figure (B2.1 & B2.2) the percentage WL and SG removal were significantly affected by osmotic solute concentration. Both WL and SG increase linearly with increase concentration .

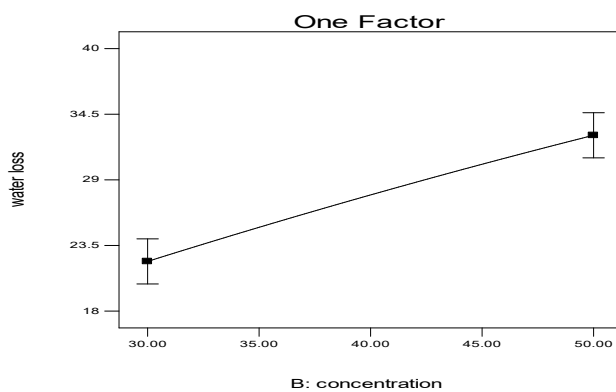


Figure B2.1: Effect of concentration on Water loss in osmotic dehydration banana slice

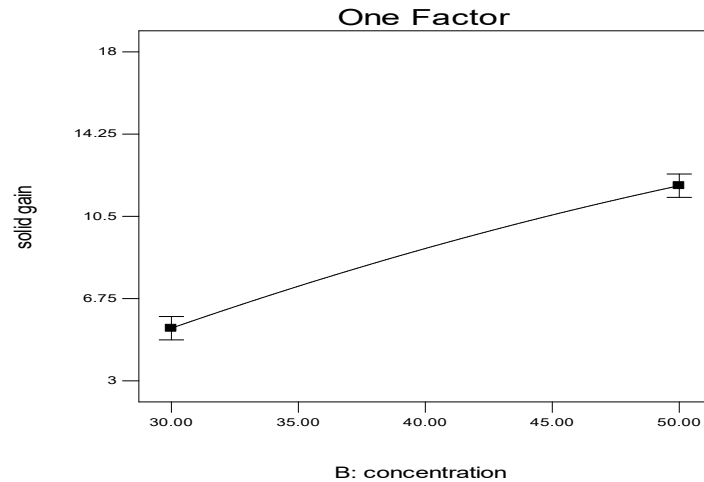


Figure B2.2: Effect of concentration on solid in osmotic dehydration banana slice

### B3) Effect of time on Water loss & solid gain in osmotic dehydration

As shown in Figure (B3.1 & B3.2 ) the percentage WL and SG removal were significantly affected by soaking temperature. An increase in percentage WL & SG with an increase in temperature and attained constant after 60 min.

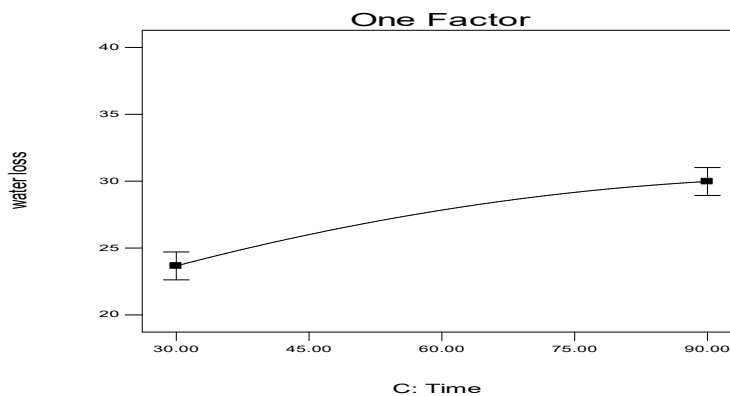


Figure B3.1: Effect of time on Water loss in osmotic dehydration banana slice

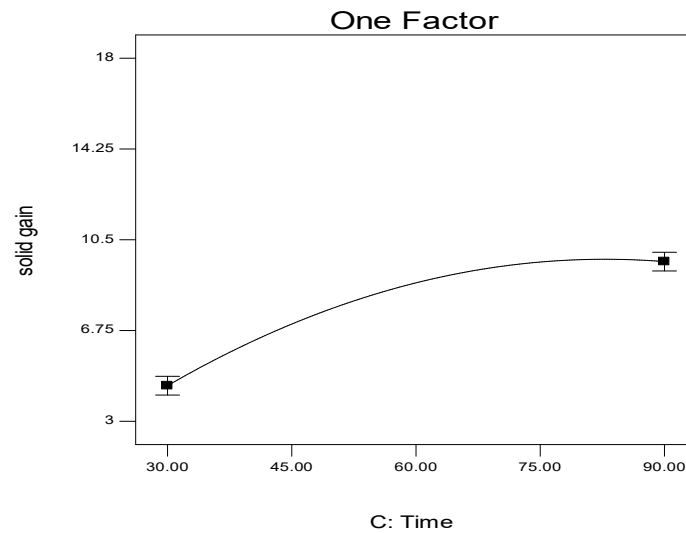


Figure B3.1: Effect of time on solid gain in osmotic dehydration banana slice

#### APPENDIX C. P-Value (ANOVA) for proximate value ANOVA

|          |                | Sum of Squares | Df | Mean Square | F        | Sig. |
|----------|----------------|----------------|----|-------------|----------|------|
| Moisture | Between Groups | 53.528         | 9  | 5.948       | 695.291  | .000 |
|          | Within Groups  | .171           | 20 | .009        |          |      |
|          | Total          | 53.699         | 29 |             |          |      |
| Protein  | Between Groups | 1.436          | 9  | .160        | 2454.550 | .000 |
|          | Within Groups  | .001           | 20 | .000        |          |      |
|          | Total          | 1.437          | 29 |             |          |      |
| Fat      | Between Groups | .661           | 9  | .073        | 19.509   | .000 |
|          | Within Groups  | .075           | 20 | .004        |          |      |
|          | Total          | .736           | 29 |             |          |      |
| Ash      | Between Groups | 8.800          | 9  | .978        | 468.736  | .000 |
|          | Within Groups  | .042           | 20 | .002        |          |      |
|          | Total          | 8.842          | 29 |             |          |      |

|                   |                |         |    |        |        |      |
|-------------------|----------------|---------|----|--------|--------|------|
| Fiber             | Between Groups | 40.049  | 9  | 4.450  | 8.725  | .000 |
|                   | Within Groups  | 10.200  | 20 | .510   |        |      |
|                   | Total          | 50.248  | 29 |        |        |      |
| Carbohydra<br>tes | Between Groups | 174.119 | 9  | 19.347 | 24.301 | .000 |
|                   | Within Groups  | 15.922  | 20 | .796   |        |      |
|                   | Total          | 190.041 | 29 |        |        |      |

**APPENDIX D. P-Value (ANOVA) for Physico-Chemical test**

**ANOVA**

|     |                | Sum of Squares | Df | Mean Square | F       | Sig. |
|-----|----------------|----------------|----|-------------|---------|------|
| TDS | Between Groups | 24.440         | 9  | 2.716       | 194.484 | .000 |
|     | Within Groups  | .279           | 20 | .014        |         |      |
|     | Total          | 24.719         | 29 |             |         |      |
| pH  | Between Groups | 3.071          | 9  | .341        | 17.237  | .000 |
|     | Within Groups  | .396           | 20 | .020        |         |      |
|     | Total          | 3.466          | 29 |             |         |      |
| NEB | Between Groups | .087           | 9  | .010        | 2.435   | .047 |
|     | Within Groups  | .080           | 20 | .004        |         |      |
|     | Total          | .167           | 29 |             |         |      |
| WHC | Between Groups | 8.461          | 9  | .940        | 393.454 | .000 |
|     | Within Groups  | .048           | 20 | .002        |         |      |
|     | Total          | 8.509          | 29 |             |         |      |
| OHC | Between Groups | .041           | 9  | .005        | 4.225   | .003 |
|     | Within Groups  | .022           | 20 | .001        |         |      |
|     | Total          | .063           | 29 |             |         |      |

## APPENDIX E. Active metals laboratory result

| No. sample  | Drying Temp.( <sup>o</sup> C) | Ca(mg/100g) | Na(mg/100g) | K(mg/100g) | Mg(mg/100g) |
|---|-------------------------------|-------------|-------------|------------|-------------|
| Untreated banana flour                                      | 45                            | 41.83       | 20.751      | 639.373    | 212.227     |
| Hot water pretreated unripe banana flour                    | 45                            | 47.733      | 20.096      | 885.337    | 214.85      |
|   | 60                            | 46.689      | 21.085      | 895.47     | 216.192     |
|   | 75                            | 50.584      | 22.178      | 906.504    | 218.428     |
| Sodium meta bisulphate(0.2%) pretreated unripe banana flour | 45                            | 40.590      | 22.315      | 668.409    | 216.366     |
|   | 60                            | 45.496      | 22.631      | 688.734    | 217.633     |
|   | 75                            | 44.080      | 22.51       | 694.541    | 218.279     |
| Osmotic dehydrated unripe banana flour                      | 45                            | 38.340      | 21.673      | 575.494    | 207.626     |
|   | 60                            | 39.384      | 21.858      | 575.784    | 208.018     |
|   | 75                            | 40.982      | 25.074      | 587.137    | 208.168     |

## APPENDIX F. Pictures while conducting product preparation





**Washing**

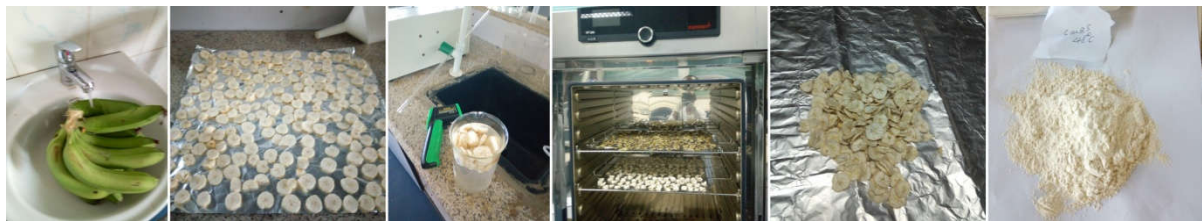
**oven drying**

**after drying**

**milling**

Figure F1. During osmotic pretreated product preparation

**APPENDIX F2. During SMB pretreated unripe banana flour preparation**



**Washing**

**Sliced**

**Dipping**

**Drying**

**After dried**

**UBF**

**APPENDIX F3. During hot water pretreated unripe banana flour preparation**



**Washing**

**Hot water bathing**

**After peeling**

**Sliced**



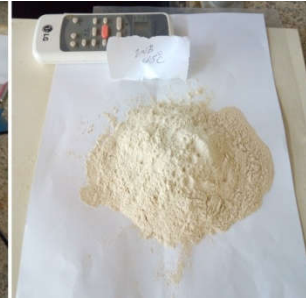
**Oven drying**



**After dry**



**Milling**



**Final flour**

**APPENDIX I. Picture during product/sample characterization**



**Ash test**



**Fat test**



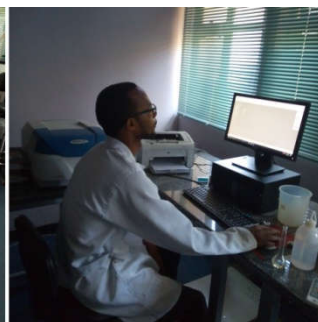
**Fiber test**



**Protein test**



**TDS test**



**NEB test**



**Active metals test**



**Trace metals**