

**PHYSICO-CHEMICAL EVALUATION OF  
PECTIN EXTRACTED FROM SOYBEAN HULLS  
AND SUNFLOWER HEADS BY ULTRASONIC  
AND MICROWAVE HEATING**

**By**

**ALI ABD-ELLAH ALI BEKHEIT**

**B.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Assiut Univ., Egypt, 1992**

**M.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Cairo Univ., Egypt, 2002**

**THESIS**

**Submitted in Partial Fulfillment of the  
Requirements for the Degree of**

**DOCTOR OF PHILOSOPHY**

**In**

**Agricultural Sciences  
(Food Technology)**

**Department of Food Technology  
Faculty of Agriculture  
Cairo University  
EGYPT**

**2008**

**SUPERVISION SHEET**

**PHYSICO-CHEMICAL EVALUATION OF PECTIN  
EXTRACTED FROM SOYBEAN HULLS AND  
SUNFLOWER HEADS BY ULTRASONIC AND  
MICROWAVE HEATING**

**Ph.D. Thesis**

**By**

**ALI ABD-ELLAH ALI BEKHEIT**

**B.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Assiut Univ., Egypt, 1992  
M.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Cairo Univ., Egypt, 2002**

**SUPERVISION COMMITTEE**

**Dr. SAMY MOHAMED GALAL**

**Associate Professor of Food Science and Technology,  
Fac. Agric., Cairo University**

**Dr. MOHAMED HASAN ALI**

**Associate Professor of Food Science and Technology,  
Fac. Agric., Cairo University**

**Dr. FATHI ABD EL-RAZIK ALI**

**Professor of Food Science Agri-Industrialization Unit,  
Desert Research Center**

**APPROVAL SHEET**

**PHYSICO-CHEMICAL EVALUATION OF PECTIN  
EXTRACTED FROM SOYBEAN HULLS AND  
SUNFLOWER HEADS BY ULTRASONIC AND  
MICROWAVE HEATING**

**Ph.D. Thesis**

**By**

**ALI ABD-ELLAH ALI BEKHEIT**

**B.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Assiut Univ., Egypt, 1992  
M.Sc. Agric. Sci. (Food Tech.), Fac. Agric., Cairo Univ., Egypt, 2002**

Approved by:

**Dr. MOHAMED KAMAL YOUSSEF** .....  
Professor of Food Sci. and Tech., Fac. Agric., Assiut Univ.

**Dr. MOHAMED KHAIRY EL-SAYED** .....  
Professor of Food Sci. and Tech., Fac. Agric., Cairo Univ.

**Dr. SAMY MOHAMED GALAL** .....  
Associate Professor of Food Sci. and Tech., Fac. Agric., Cairo Univ.

**Dr. MOHAMED HASAN ALI** .....  
Associate Professor of Food Sci. and Tech., Fac. Agric., Cairo Univ.

**Date:** / /

## *ACKNOWLEDGMENT*

*Firstly, all praises are due to Allah, Who blessed me with kind professors and colleagues, and gave me the support to produce this Thesis.*

*I wish to express my sincere thanks, deepest gratitude and appreciation to Dr. Samy Mohamed Galal Associate Professor of Food Science and Technology Dept., Faculty of Agriculture, Cairo University, for his patience, supervision, guidance and valuable advices, throughout this work.*

*The auther feels most deeply indebted to Dr. Mohamed Hasan Ali Associate Professor of Food Science and Technology Dept., Faculty of Agriculture, Cairo University, for his supervision, constructive criticism and advice during the whole period of this investigation.*

*Grateful appreciation is also extended to Dr. Fathy Abd El-Razik Ali, Professor of Food Science Agri-Industrialization Unit, Desert Research Center, for his helpful suggestion, kind supervision and valuable guidance throughout this work.*

## DEDICATION

*I dedicate this work to whom my heart felt thanks:  
To my wife Rasha and my daughter Bassant for their  
patience and help, as well as to my parents and  
brothers for all the support they lovely offered along  
the period of my post graduation.*

**Name of Candidate:** Ali Abd-Ellah Ali Bekheit      **Degree:** Ph.D  
**Title of Thesis:** Physico-chemical evaluation of pectin extracted from soybean hulls and sunflower heads by ultrasonic and microwave heating  
**Supervisors:** Dr. Sami Mohamed Galal, Dr. Mohamed Hasan Ali and Dr. Fathi Abd El-Razik Ali  
**Department:** Food Technology  
**Branch:** Food Technology      **Approval:**

#### ABSTRACT

This study was conducted to evaluate the effect of ultrasonic or microwave intensities, extraction temperature and extraction time on the yield and quality of pectin extracted from soybean hulls and sunflower head residues. Ultrasonic and microwave were used during extraction of pectin from alcohol insoluble solids of these materials under different conditions. The obtained pectin samples and commercial citrus low methoxyl pectin were physically, chemically and organoleptically evaluated.

The highest yield and recovery of pectin extracted from soybean hulls were obtained by using 50% microwave intensity of the maximal power for 20 min. Anhydrogalacturonic acid, methoxyl content and degree of esterification of the resulted soybean hulls pectin were 85, 5.65 and 36.77%, respectively. The highest yield and recovery of pectin extracted from sunflower head residues were obtained by using ultrasonic intensity at 60% of the maximal power at 80°C for 15 min. Anhydrogalacturonic acid, methoxyl content and degree of esterification of obtained pectin were 88.48, 4.55 and 26.06%, respectively. Extraction conditions (Ultrasonic, microwave, temperature and time) markedly affected the viscosity of solutions containing the extracted pectin.

Results indicated that gel strength, setting time and sensory properties of solutions containing soybean hulls pectin at 1.75 % and sunflower head pectin at 1.50% were almost like those of the solution contains commercial citrus low methoxyl pectin at 1%.

From the results presented in this study it could be concluded that extraction of pectin from soybean hulls with the assistance of microwave was more proper than extraction with ultrasonic. On the other hand, extraction of sunflower heads pectin with ultrasonic waves was more suitable than all other investigated methods.

**Key words:** Pectin, Soybean hulls, Sunflower heads, Ultrasonic, Microwave

# CONTENTS

|   |           |
|---|-----------|
| <b>INTRODUCTION</b> .....                                     | <b>1</b>  |
| <b>REVIEW OF LITERATURE</b> .....                             | <b>3</b>  |
| 1. Sources of pectin .....                                    | 4         |
| 2. Chemical composition of soy hulls and sunflower heads..... | 5         |
| 3. Production of pectin.....                                  | 6         |
| a. Washing process of raw materials.....                      | 6         |
| b. Extraction process of pectin.....                          | 9         |
| 1. Traditional extraction.....                                | 9         |
| 2. Microwave extraction.....                                  | 14        |
| 3. Ultrasonic extraction .....                                | 17        |
| 4. Evaluation of pectin.....                                  | 19        |
| a. Chemical properties of pectin.....                         | 19        |
| b. Physical properties of pectin.....                         | 22        |
| 5. Application of pectin.....                                 | 28        |
| <b>MATERIALS AND METHODS</b> .....                            | <b>30</b> |
| <b>1. MATERIALS</b> .....                                     | <b>30</b> |
| a. Sunflower head residues .....                              | 30        |
| b. Soybean hulls .....  | 30        |
| c. Commercial citrus low methoxyl pectin.....                 | 30        |
| d. D (+) galacturonic acid monohydrate.....                   | 30        |
| e. Cotchnel color and strawberry flavor.....                  | 30        |
| <b>2. METHODS</b> .....                                       | <b>30</b> |
| a. Production of pectin.....                                  | 30        |
| 1. Washing process .....                                      | 30        |
| 2. Extraction process .....                                   | 31        |
| a. Traditional method .....                                   | 31        |
| b. Ultrasonic method .....                                    | 31        |
| c. Microwave method .....                                     | 32        |
| 3. Precipitation and purification of extracted pectin .....   | 32        |
| 4. Pectin yield and recovery.....                             | 35        |
| b. Analytical methods .....                                   | 35        |
| 1. Chemical analysis of raw materials.....                    | 35        |
| a. Moisture content.....                                      | 35        |
| b. Ash content .....  | 35        |
| c. Protein content .....                                      | 35        |
| d. Crude fat .....  | 35        |

|   |           |
|---|-----------|
| e. Total sugars.....  | 36        |
| f. Absorbance.....  | 36        |
| g. Total pectin content.....  | 36        |
| 2. Chemical analysis of produced pectin.....  | 37        |
| a. Anhydrogalacturonic acid .....   | 37        |
| b. Methoxyl content.....  | 38        |
| c. Degree of esterification.....  | 39        |
| 3. Physical analysis of produced pectin.....  | 39        |
| a. Viscosity.....   | 39        |
| b. Turbidity.....   | 40        |
| c. Gel strength.....  | 40        |
| d. Setting time and setting temperature.....  | 41        |
| e. Jelly grade.....   | 41        |
| c. Application of low methoxyl pectin.....  | 42        |
| d. Organolyptic evaluation.....   | 42        |
| e. Statistical analysis.....  | 42        |
| <b>RESULTS AND DISCUSSION.....</b>  | <b>43</b> |
| 1. Chemical composition of raw materials.....   | 43        |
| 2. Effect of washing process on chemical composition of raw materials.....  | 46        |
| 3. Effect of thermosonic intensity and extraction time on % yield and % recovery of extracted soybean hulls pectin.....   | 48        |
| 4. Effect of thermosonic intensity and extraction time on % yield and % recovery of extracted sunflower head pectin.....  | 52        |
| 5. Effect of thermosonic intensity and extraction time on the viscosity of extracted soybean hulls pectin solution.....   | 57        |
| 6. Effect of thermosonic intensity and extraction time on the viscosity of extracted sunflower heads pectin solution..... | 59        |
| 7. Effect of microwave intensity and extraction time on % yield and % recovery of extracted soybean hulls pectin.....     | 62        |
| 8. Effect of microwave intensity and extraction time on % yield and % recovery of extracted sunflower heads pectin.....   | 65        |
| 9. Effect of microwave intensity and extraction time on the viscosity of extracted soybean hulls pectin solution.....     | 68        |
| 10. Effect of microwave intensity and extraction time on the viscosity of extracted sunflower heads pectin solution.....  | 70        |

|  |            |
|--|------------|
| 11. Chemical and physical properties of extracted soybean hulls pectin.....                                    | 73         |
| 12. Chemical and physical properties of extracted sunflower heads pectin .....                                 | 78         |
| 13. Color measurement of soybean hulls and sunflower heads pectin powders.....                                 | 83         |
| 14. Effect of extracted pectin concentration on physical properties of its solutions .....                     | 84         |
| 15. Organoleptic evaluation of jellies prepared from extracted soybean hulls and sunflower heads pectins ..... | 88         |
| <b>SUMMARY</b> .....   | <b>91</b>  |
| <b>REFERENCES</b> .....  | <b>100</b> |
| <b>ARABIC SUMMARY</b> .....  |            |

## LIST OF TABLES

| No  | Title   | Page |
|-----|---|------|
| 1.  | Chemical composition of raw materials.....  | 44   |
| 2.  | Effect of washing process on chemical composition of raw materials.....   | 47   |
| 3.  | Effect of thermosonic intensity and extraction time on % yield and % recovery of extracted soybean hulls pectin .....   | 49   |
| 4.  | Effect of thermosonic intensity and extraction time on % yield and % recovery of extracted sunflower heads pectin ..... | 53   |
| 5.  | Effect of thermosonic intensity and extraction time on the viscosity of extracted soybean hulls pectin solution.....    | 58   |
| 6.  | Effect of thermosonic intensity and extraction time on the viscosity of extracted sunflower heads pectin solution.....  | 60   |
| 7.  | Effect of microwave intensity and extraction time on % yield and % recovery of extracted soybean hulls pectin.....      | 63   |
| 8.  | Effect of microwave intensity and extraction time on % yield and % recovery of extracted sunflower heads pectin.....    | 66   |
| 9.  | Effect of microwave intensity and extraction time on the viscosity of extracted soybean hulls pectin solution.....      | 69   |
| 10. | Effect of microwave intensity and extraction time on the viscosity of extracted sunflower heads pectin solution.....    | 70   |
| 11. | Chemical and physical properties of extracted soybean hulls pectin.....   | 74   |
| 12. | Chemical and physical properties of extracted sunflower heads pectin.....   | 79   |
| 13. | Color measurement of soybean hulls and sunflower heads pectin powders.....  | 83   |
| 14. | Effect of extracted pectin concentration on physical properties of its solutions .....                                  | 84   |
| 15. | Organoleptic evaluation of jellies prepared from extracted soybean hulls and sunflower heads pectins .....              | 89   |

## LIST OF FIGURES

| <b>No</b> | <b>Title</b>   | <b>Page</b> |
|-----------|--|-------------|
| 1.        | Structure of pectin.....   | 3           |
| 2.        | Extraction of pectin from soybean hulls.....   | 33          |
| 3.        | Extraction of pectin from sunflower heads.....   | 34          |
| 4.        | Effect of thermosonic intensity and extraction time on % yield of soybean hulls pectin.....                    | 50          |
| 5.        | Effect of thermosonic intensity and extraction time on % recovery of soybean hulls pectin.....                 | 50          |
| 6.        | Effect of thermosonic intensity and extraction time on % yield of sunflower heads pectin .....                 | 54          |
| 7.        | Effect of thermosonic intensity and extraction time on % recovery of sunflower heads pectin .....              | 54          |
| 8.        | Effect of thermosonic intensity and extraction time on the viscosity of soybean hulls pectin solution.....     | 61          |
| 9.        | Effect of thermosonic intensity and extraction time on the viscosity of sunflower heads pectin solution.....   | 61          |
| 10.       | Effect of microwave intensity and extraction time on % yield of soybean hulls pectin.....                      | 64          |
| 11.       | Effect of microwave intensity and extraction time on % recovery of extracted soybean hulls pectin.....         | 64          |
| 12.       | Effect of microwave intensity and extraction time on % yield of sunflower heads pectin.....                    | 67          |
| 13.       | Effect of microwave intensity and extraction time on % recovery of sunflower heads pectin.....                 | 67          |
| 14.       | Effect of microwave intensity and extraction time on the viscosity of soybean hulls pectin solution.....       | 71          |
| 15.       | Effect of microwave intensity and extraction time on the viscosity of sunflower heads pectin solution.....     | 71          |
| 16.       | Effect of pectin concentration on viscosity of extracted pectin and commercial low methoxyl pectin.....        | 86          |
| 17.       | Effect of pectin concentration on gel strength of extracted pectin gel and commercial low methoxyl pectin..... | 86          |



## INTRODUCTION

Wastes of farms and industries play an important role in environment contamination. Sunflower (*Helianthus annuus* L.), the fourth source of oil-seeds world wide recorded a production of 31332 thousand tons of seeds in 2006. Also, the total world's production of soybean (*Glycine max* (L) Marr. ) reached 221500 thousand tons in 2006. The annual production of sunflower and soybean in Egypt was 43 and 26.5 thousand tons in 2006, respectively (FAO, 2008).

Sunflower heads which remain after seeds are removed representing a naturally rich source of low methoxyl pectin (Shi *et al.* 1996b). Also, soybean hulls are considered a by-product of soy processing. On a dry weight basis, hulls constitute about 8% of the total seed, depending on variety and seed size (Liu, 1997).

There are two types of fibers, insoluble fiber and soluble fiber. The insoluble fiber includes hemicellulose, cellulose and lignin and they were found in beans and fruits with edible seeds. The soluble fiber includes gums and pectin (Liu *et al.* 2006). Pectin represents an important component of the waste of the two commercially important fruit types, citrus and apple (Cohn and Cohn 1996). Pectin is important in food processing as food additives. Pectins are used as gelling agents in jams, jellies and fruit preparations and as stabilizers in confections, dairy products, bakery fillings and icings (Monsoor and Proctor, 2001).

A variety methods of production of pectin have already been reported by many researchers. The process of the traditional method includes degradation by acid and deposition. The process lasts more than one hour using acid solution at pH 2 and a temperature of 80 –

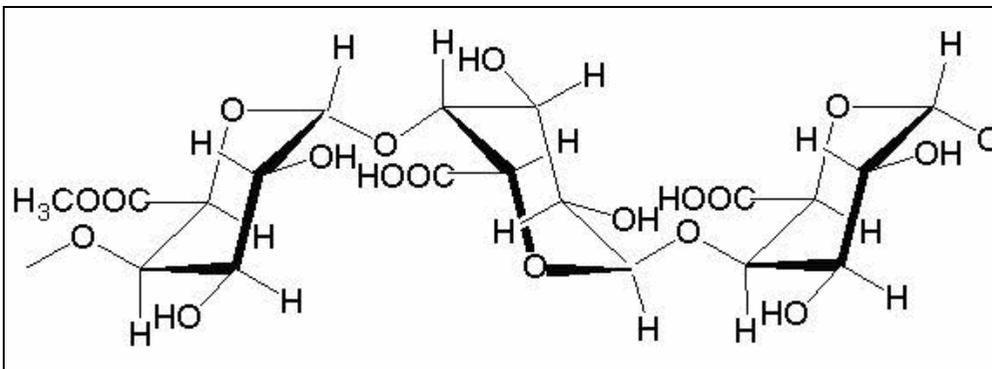
100° C. These conditions lead to pectin degradation, so this traditional method is not good for either quantity or quality of the extracted pectin. Therefore, it is necessary to establish new methods by which the pectin could be extracted in a shorter time and yields a product with better quality (Zhongdong *et al.* 2006).

New alternative processing techniques like ultrasonication have proven to increase yield and quality of pectin (Panchev *et al.* 1988). Ultrasound enhances the effect of ultrasonic waves on the vegetal material causing destruction of the cells and released the cells' contents into the extraction medium. This was documented by the increased extractability of water-soluble polysaccharides from the ethanol-insoluble plant residue obtained from the ultrasound-assisted process in comparison to that from the classical extraction (Li *et al.* 2007). Also, Manabe *et al.* (1988) used the microwave energy to extract 5% more pectin in 15 min than could be extracted by conventional methods in 60 min from mandarin orange pulp.

The aim of this study was to utilize wastes of sunflower head residues and soybean hulls to produce pectin using ultrasonic and microwave methods during the extraction compared with traditional extraction in order to maximize its yield and quality.

## REVIEW OF LITERATURE

Pectic substances are those complex, colloidal carbohydrate derivatives which occur in, or are prepared from plants and contain a large proportion of anhydrogalacturonic acid units which are thought to exist in a chain-linked combination each unit connected through the  $\alpha$  1,4 glycosidic linkages forming a polygalacturonic acids. . The carboxyl groups of poly galacturonic acid may be partly esterified by methyl groups and partly or completely neutralized by one or more bases (Pilnik and Voragen, 1970 and Rouse, 1977).



**Fig.1: Structure of pectin** (Anonymous 2008)

Pectin represents an important component of the wastes of two commercially important fruit types, citrus and apple. Both citrus peels and apple pomace are high in moisture and are typically dried within a few hours after the juicing operation to prevent attack by molds and native pectic enzymes. There is an accompanying loss in pectin yield with drying. Dry starting materials, like sunflower head residues and soybean hulls would be stable for long periods of time and not have the expense of needing to be dried, a distinct advantage (Crandall *et al.* 2004).