"DETERMINATION OF THE CORRELATION BETWEEN AMYLOSE AND PHOSPHORUS CONTENT AND GELATINIZATION PROFILE OF STARCHES AND FLOURS OBTAINED FROM EDIBLE TROPICAL TUBERS USING DIFFERENTIAL SCANNING CALORIMETRY AND ATOMIC ABSORPTION SPECTROSCOPY "

By

Elevina E. Pérez Sira

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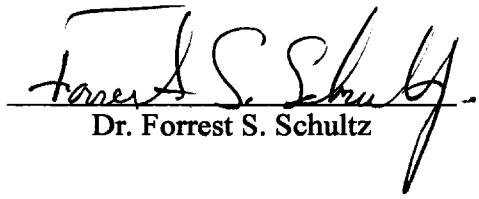
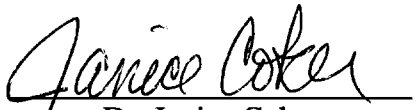
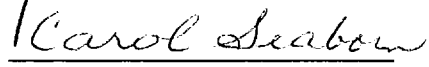


Dr. Forrest S. Schultz

Thesis Committee Members



Dr. Janice Coker



Dr. Carol Seaborn

The Graduate College.

University of Wisconsin-Stout

November, 2000

The Graduate College

University of Wisconsin-Stout

Menomonie, WI 54751

ABSTRACT

Pérez (Last name) Elevina (First) E (Initial).

Determination of the correlation between amylose and phosphorus content and gelatinization profile of starches and flours obtained from edible tropical tubers using Differential Scanning Calorimetry and Atomic Absorption Spectroscopy
(Title)

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Katel Turabian, 1987.
(Name of style Manual Used in this Study)

Xanthosoma sagittifolium, Colocassia esculenta, and *Ipomoea batata* plants produce underground storage organs that contain mainly starch and fiber. These staple food items have been misused for many years and in many instances they exhibit a high percentage of loss because of spoilage. However, the availability of tropical and subtropical crops such as *Xanthosoma sagittifolium, Colocasia esculena*, and *Ipomoea batata* in the temperate zones of the world has increased in recent years because research has improved varieties of these crops by agronomic and genetic techniques. With the excellent varieties available today, they could be grown more extensively and constitute farinaceous foods of high nutritive and economical value. Before they are more widely used, the functional properties of these tubers must be evaluated. One of the approaches to characterize functional properties of the starches or flours obtained from storage

organs of these plants is through gelatinization profiles. The gelatinization profile can be determined using several techniques of which differential scanning calorimetry (DSC) is the most common. It is a general consensus that the gelatinization profile is a function of the amylose and phosphorous contents of starches. In this study the gelatinization profiles of starches isolated from *Colocasia esculenta, Xanthosoma sagitifolium*, and *Ipomea batata* storage organs were evaluated using changes in the heat flow or enthalpy during the gelatinization process by DSC methodology. The amylose content was also evaluated using the DSC technique and a colorimetric method. The phosphorous content was analyzed by colorimetry using the method described in AOAC, 1993. The results show that starch isolated from *Ipomoea batata* has a similar amylose content as starch isolated from *Xanthosoma sagittifolium*. Both show more starch content than *Colocasia esculenta*. The phosphorous content was higher in *Ipomoea batata* than *Xanthosoma sagittifolium* or *Colocasia esculenta* starches. The gelatinization profile range is wider in *Ipomoea batata* than the other two starches. Differences in these parameters may affect the functional properties of the products formulated with these starches.

Lope de Vega:

-

"A mis soledades voy,
de mis soledades vengo,
porque para andar conmigo
me bastan mis pensamientos"

" To my solitude I go,
From my solitude I come,
But, to walk by myself with my thoughts is enough"

-

Campoamor:

-

"pero es más espantosa todavía
la soledad de dos en compañía".

-

"But it is quite horrifying furthermore
The loneness of two together"

ACKNOWLEDGEMENTS

I would like to express my sincere appreciation to my thesis adviser Dr. Forrest Schultz for his patience, constructive suggestions, and invaluable assistance. I am also grateful for the helpful comments and professional suggestions given by the thesis committee members: Dr. Janice Coker, and Dr. Carol Seaborn. I also am grateful to the UW-Stout Chemistry Department for their generosity, patience, and support.

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CHAPTER I. INTRODUCTION

Most tropical plants produce underground storage organs that can be classified as roots (e.g., root of *Manihot esculenta*, commonly named as cassava or tapioca roots) or modified stems. Modified stems that grow underground are named rhizomes, corms, or tubers. Examples of modified stems are *Xanthosoma sagittifolium*, *Colocassia esculenta*, and *Ipomoea batata*. The term aroid is also used to name the storage organs from plants belonging to the family Araceae. *Xanthosoma sagittifolium* and *Colocassia esculenta* are members of the Araceae family. Like potatoes, these roots and modified stems contain moistly starch and fiber (10,32). The term tuber will be used to identify the underground storage organs examined in this thesis.

In the tropical areas of South America, *Xanthosoma sagittifolium*, *Colocassia esculenta*, and *Ipomoea batata* have been grown in artisan culture for centuries, where they have been consumed domestically, either boiled in soups or mashed (26). They are not grown extensively and much of the production never enters commercial channels. For instance, they are frequently sold in markets as raw vegetables. However, the availability of tropical and subtropical tubers in the temperate zones of the world has increased in recent years because research has improved crop varieties by agronomic and genetic techniques (6, 11, 21, 26, 31, 39, 41, 46, 47, 50, 51, 53, 58, 60). With the excellent varieties available today, they could be grown much more extensively and constitute foods of high nutritive value and economical value.

Tubers of these plants are potential sources of flour and industrial starch that have not yet been utilized. On the other hand, these tubers are perishable because they have

both high moisture content and a high metabolic activity after harvesting. They are subject to post-harvest losses owing to their continuing metabolism and damage during harvest and storage (15). It has been estimated that there is an average of 30% loss during storage of these tubers, and that this portion could resolve starvation problems in non-developed countries (2,16,28,53,56). For example, according to a Food and Agriculture Organization of the United Nations (FAO) report in 1977, losses of potatoes on farms in developed countries were to the extent of 20-40% (16). Developed country's losses have been decreased for potatoes with improved storage systems that control temperature and humidity. In tropical areas, the temperature and humidity are naturally high. Losses of all types of roots and tubers are prevalent today and have been estimated to vary depending of the type of tuber from 5-40% (15). In order to minimize tuber losses, they must be converted from perishable to non-perishable through food processing operations. Since the transformation into starch or flour will decrease losses after the tubers have been harvested, value added processes such as drum drying and wet milling may be useful in order to obtain flours and starches from these tubers. It is, therefore, clear that a significant amount of work remains to be done on the functional characteristics of native, as well as, modified tropical starches if they are ever to become competitive with commercial starches such as corn, wheat, and potato. In Venezuela, these tubers are consumed domestically as a perishable food because they have a short shelf life. Converting tubers into flours and starches may be an important process that could contribute to minimizing losses and to allow the commercial food industry to store the tuber throughout the year. In addition, the starch production in Venezuela is confined to two or three factories that are processing corn and tapioca starches. Most of the

modified starches that have been used in the Venezuelan food industry are imported (29), where wheat and rice are a non-native botanical source for flours. The wheat that is used in Venezuela is imported from developed countries (13,57).

Before consideration is given to tubers as potential sources of flour and starch to produce foods, it is necessary to characterize their chemical composition, physical, physicochemical, and functional properties. The chemical composition of flours and starches exhibits differences especially in amylose and phosphorous content, as a function of the botanical origin. It is significant because of the influence of amylose and phosphorous content in the functional properties of flours and starches. It is a general consensus that the influence of both amylose and phosphorous content affects the gelatinization and pasting behavior of starches and flours (20,30,59). These two parameters determine the functional properties of flours and starches such as: texture, consistency, binding, coating, adhesiveness, cohesiveness, thickening, viscosity, and palatability. A number of methodologies have been used to characterize these properties. One of the main techniques utilizes the differential scanning calorimeter. Differential Scanning Calorimetry (DSC) is widely applied in the food industry to interpret water, starch, protein, and lipid and carbohydrate interactions. In 1971, Stevens and Elton, appear to have been the first to apply DSC to the study of starch gelatinization (48). Since then, many investigators have used DSC to study starch and starch systems (5,8,34,35,48). However, these investigations have been performed on conventional starches such as corn, wheat, and potato starches (5, 48). DSC analysis has been performed much less commonly on the starch systems obtained from tropical tubers (34,35).

OBJECTIVES

The aim of this study will be to obtain theoretical and practical knowledge about the functional properties of starches and flours isolated from non-conventional sources of starches and flours such as *Xanthosoma sagittifolium*, *Colocassia esculenta,* and *Ipomoea batata* tubers and then to correlate them with amylose and phosphorous content. This study will also ascertain the pertinence of thermal and spectrophotometric methods such as differential scanning calorimetry (DSC) and atomic absorption spectroscopy (AA) on the characterization of the properties and composition of non-conventional starches and flours. Therefore, the specific objectives are as follows:

1. Determination of amylose content of starches and raw and modified flours obtained from tubers of one variety of *Xanthosoma sagittifolium*, *Colocassia esculenta,* and *Ipomoea batata* using DSC and colorimetric methodologies.
2. Determination of phosphorus and calcium content of starches and raw and modified flours obtained from tubers of one variety of *Xanthosoma sagittifolium*, *Colocassia, esculenta* and *Ipomoea batata* using AA.
3. Characterization of gelatinization profile of starches and raw and modified flours obtained from tubers of one variety of *Xanthosoma sagittifolium*, *Colocassia esculenta,* and *Ipomoea batata* using DSC.
4. Correlation among the gelatinization profiles and the amylose and phosphorous contents of *Xanthosoma sagittifolium*, *Colocassia esculenta*, and *Ipomoea batata* in order to define their behavior in food systems.

STATEMENT OF THE PROBLEM

There are tropical plants not cultivated extensively in the South American tropical areas such as *Xanthosoma sagittifolium*, *Colocassia esculenta,* and *Ipomoea batata* that develop underground storage organs. The underground storage organs are rich in moisture and starch. Because of their high moisture content, they are perishable with an estimated annual loss of 30%. In contrast, the fact that they have high starch content makes them attractive to the food industry. The general purpose of this study is to convert perishable underground storage organs into non-perishable starches and flours with adequate functional properties using value added processes. Since functional properties of starches and flours are dependent on botanical source, composition of amylose and amylopectin, and phosphorous content, these properties must be studied and characterized to make these tubers attractive to the food industry.

The specific purpose of this study is to determine the gelatinization profiles as measured by enthalpic changes (ΔH) using DSC on starches and flours isolated from *Xanthosoma sagittifolium*, *Colocassia esculenta*, and *Ipomoea batata*, which may differ in the amount of amylose and phosphorous content as measured in percentage (w/w) by DSC and colorimetric methodologies.

HYPOTHESIS

General

A review of the literature shows that non-conventional tropical tubers such as *Xanthosoma saggitifolium*, *Colocassia esculenta,* and *Ipomoea batata* have been less cultivated as food staples than potato and tapioca. However, they could be grown

extensively in the tropics by their enhancement using agronomic and modern genetic techniques. They are perishables because of their high moisture content. The risk of high loss persists during harvesting and storage. Studies have shown that using value added processes such as dehydration to obtain flours and isolation of starches from conventional tubers, such as potatoes, could reduce such losses. These value added processes may allow for a commercial distribution system of the staple food during non-harvesting season. Before these tubers are introduced as new food staples into commercial systems, it must be demonstrated that it is feasible to use these tubers by studying their functional properties. Therefore, the research hypothesis for this study is that the perishable tubers from *Xanthosoma saggitifolium*, *Colocassia esculenta,* and *Ipomoea batata* can be converted into non-perishables products with adequate functional properties to be used as food staples and ingredients for new product development using the value added processes.

Specific

A review of the literature shows that starches and flours isolated from different sources have different functional properties that can be measured by gelatinization profiles. Studies also show that phosphorous and amylose contents modify the gelatinization profiles of starches. Therefore, the research hypothesis for this study is that flours and starches isolated from tubers of *Xanthosoma saggitifolium, Colocassia esculenta*, and *Ipomoea batata* have functional properties that are dependent on their content of amylose and phosphorous.

VARIABLES

Moisture of starches and flours expressed as percentage (w/w)

Ash of starches and flours expressed as percentage (w/w)

Gelatinization profiles of starches and flours expressed as enthalpy changes (ΔH)

Gelatinization temperatures of starches and flours expressed as degree Celsius (°C)

Amylose content of starches and flours expressed as percentage (w/w)

Phosphorous content of starches and flours expressed as mg/100g sample

Calcium content of starches and flours expressed as percentage (w/w)

Aerobic microorganisms of starches and flours expressed as Colony Forming Units (CFU)

Molds and yeasts of starches and flours expressed as Colony Forming Units (CFU)

NEEDS FOR STUDY

Xanthosoma sagittifolium, *Colocasia esculenta*, and *Ipomoea batata* tubers store a high starch concentration that ranges between 22 to 40% (9,13,25,26,39,55,56) and for this reason, they are considered carbohydrate foods (44). They are not extensively commercialized at the present time, but are mostly grown in domestic gardens or "conucos". Improvements in agronomic techniques and utilization of modern genetic techniques may allow these tubers to be cultured and commercialized extensively. In addition, these tubers are widely consumed in tropical areas and may resolve starvation problems elsewhere. However, they have a short shelf life because of their high moisture

content. One of the best ways to preserve them may be by processing them to obtain flour and/or starches. The study of the flours and starches obtained from these tubers and the characterization of their functional properties will be especially relevant to the Venezuelan food industry. Once the starches or flours are obtained from the tubers, it is necessary to understand their functional properties in order to use them in the food industry. The functional properties of starches and flours and their relationships with amylose and phosphorus contents may be elucidated using methods such as DSC and AA Spectroscopy. Once the functional properties of starches and flours are better characterized, the food processor will have the opportunity to use these starches and flours. The availability of a new source of starches and flour are welcome to Centro- and South-American countries, because almost all of the starches used in the food industry in these areas are imported from industrialized countries. The imported starches and flours are derived from corn, tapioca, and wheat.

LIMITATIONS

Limitations for the use of this research are dependent upon agricultural developments of these crops. There are numerous factors that are related to these limitations such as: lack of interest in these cultures, especially crops of *Xanthosoma sagittifolium* and *Colocasia esculenta*, the climate and growing condition requirements of these crops, and unavailable information related to these crops.

DEFINITION OF TERMS

Tubers: Tubers are also named corms and rhizomes. Generally, tubers are defined as short thickened fleshy stems or terminal portions of stems that are usually formed underground.

***Xanthosoma sagittifolium* tubers:** Tropical corms or aroids belonging to tuberous plants of the Araceae family. It is the edible portion of the *Xanthosoma sagittifolium (L) Schott* plant. The aroids are named ocumo, cocoyam, yautia, and the plant is called malanga.

***Colocasia esculenta* tubers**: Tropical corms or aroids belonging to tuberous plants of the Araceae family. It is the edible portion of the *Colocasia esculenta* plants. The aroids are called ocumo chino, or taro.

***Ipomoea batata* tubers**: Tropical tubers belonging to tuberous plants of the Convolvulaceae family. It is the edible portion of the *Ipomoea batata* plants. The aroids are called batata, or sweet potato.

DSC-thermograms: These are curves that monitor changes in physical or chemical properties of a material as a function of temperature by detecting the heat or enthalpic changes associated with such processes.

Gelatinization profiles: Gelatinization profiles define the gelatinization and pasting behavior of starchy systems. They indicate that the intermolecular starch bonds are breaking with increasing temperature, and aqueous condition; therefore, H-bonding sites engage more water. It produces an increased randomness in structure, decreased crystallization regions, and loss of birefringence. In this thesis, gelatinization profiles of starches and flours are measured during the enthalpic changes (ΔH) and are expressed as

the initial (ITG), middle (MTG), and end (ETG) gelatinization temperature in degrees Celsius.

Starch: Starch is the storage form of sugar in plants. Starch is composed entirely of glucose units bonded by α -1,4 and α-1,6 glucosidic links. Starch is structurally formed by amylose and amylopectin. Both are packed in small discrete semicrystalline structures called starch granules. The specific bonding pattern (or linkage) between adjacent glucose units causes chains of glucose to coil. This coiling orients the linkages in such a way that they are accessible to enzymes that break down starch.

Flour: Flour is produced by every grinding machine in the break, scratch, and reduction system of the normal mill flow from raw material. To obtain flours from tubers, they have to be dehydrated before the grinding and reduction processes.

Phosphorous: Phosphorous can occur inside of the amylose or amylopectin structures and therefore modify their functional properties.

Calcium: Calcium can occur in the edible tissue of aroids as calcium oxalate. Some hand allergies are associated with calcium oxalate crystal.

CHAPTER II. REVIEW OF LITERATURE

Tropical storage organs

Nature has produced an estimated of 300,000 different plant species, of which only a few hundred are used in organized agriculture (11). The tropical root and tuber crops are comprised of crops covering several genera. The most economically significant root crops globally include potato (*Solanum tuberosum* L.), sweet potato (*Ipomoea batatas* (L) Lam.), cassava (*Manihot esculenta* Crantz), yams (*Dioscorea* spp.), and aroids (principally *Colocasia esculenta* (L.) Schott and *Xanthosoma* spp.) (6,10). Although consumers, governmental organizations, and researchers have traditionally considered root vegetables of low status and generally unimportant crops, they account for three of the seven most important food crops in the world (11).

Tropical roots and tubers are staple foods in many parts of the tropics, accounting for the main source of the daily carbohydrate intake for large populations. These carbohydrates are mostly starches found in storage organs, which may be enlarged roots or tubers. Roots such as cassava and tubers such as potatoes, sweet potatoes and yams are, together with plantains, the staple foods of over 1.5 billion of the world's population (53). Some 650 million tons of these foods are produced each year, and 70% originate in tropical countries. Their cultivation is an issue of such importance that a network for the promotion of tropical starch-producing plants (PROAMYL-CIO) has been set up, which links a hundred or so full-time researchers from CIRAD, France's International Center for Co-operation in Agricultural Research for Development, INRA, the French National Institute for Agricultural Research, and ORSTOM, the French Institute of Scientific

Research Developed in Co-operation (11). There is a tremendous potential for the profitable commercial use of tropical starches, but considerable research and product development of new types is necessary to properly utilize these materials. The model for product quality and reliability has already been set by the international starch industry (44).

Ipomoea batata plants (Figure 1) belong to the convolvulaceae family, (they are commonly misnamed yams, especially at thanksgiving time, when it is usually the sweet potato that is eaten). Many varieties of sweet potatoes (Ipomoea) are grown, with a purple or tan outside and with a white, orange, or purple meat (37). Nutritionists favor the orange meat variety (Figure 2) because of its higher carotene content (45). Originating in South America, its production has spread throughout the tropics and it is a staple food in countries of Africa and the South Pacific (28, 49, 50, 54, 55). Sweet potatoes are a critical source of carbohydrates in much of the tropics and subtropics, especially in Africa and Latin America (11). The sweet potato tuber is eaten raw, boiled, steamed, baked, fried, mashed, dried, or fermented. In addition, it is used as a source of starch (10). Only potato and sweet potato are grown to any extent in the United States, and of these two, sweet potato has the greatest potential for increased usage and consumption (6).

Manihot esculenta Crantz (Figure 3) belongs to the Euphorbiaceae family and is a probable native of Brazil, which is now widespread in the tropics and subtropics. Cassava, yuca, tapioca, manioca are some of the common names for *Manihot esculenta* Crantz (42). Tapioca pearls are a form of starch that is extracted from the roots of *Manihot esculenta Crantz* (Figure 4 and 5). The starch is used in many different

Figure 1. Ipomea batata plants. Source: Carr, G. Botany Department of the University of Hawaii at Manoa, 2000.
http://www.botany.hawaii.edu/faculty/carr/convolvul.htm

Figure 2. Ipomea batata tubers. Source: Kaplan, M. MK Salad, 2000.
http://www.sonic.net/~melissk/mksalad.html

industries as an ingredient or additive. In powdered form, it is called tapioca flour or "goma de mandioca" and is employed as a thickening agent for soups, sauces and crepes (10). The roots of the sweet varieties contain less cyanide than of the bitter types (45).

Figure 3. *Manihot esculenta* C. plants. Source: Armstrong, W.P. Wayne's World. Vegetable from underground, Economic plant photographs: Vegetables # 1, 2000. http://daphne.palomar.edu/wayne/vege1.htm

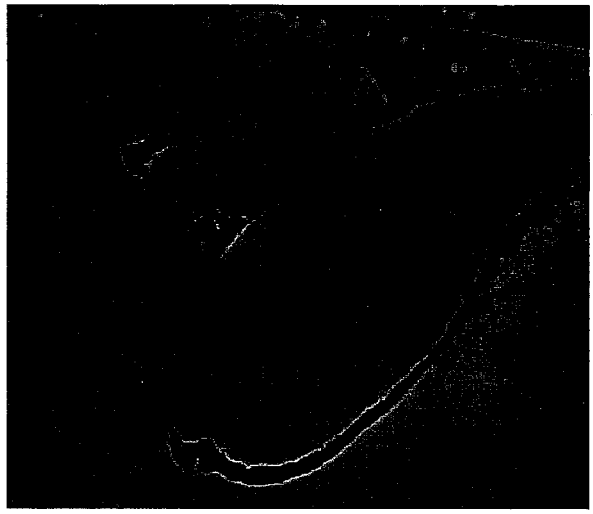


Figure 4. *Manihot esculenta Crantz*. Roots. Source: Slimak, K. Special Food Company, 2000.
http://www.specialfoods.com/cassava.html

Figure 5. *Manihot esculenta C.* tubers and culture. Source: Armstrong, W.P. Wayne's World. Vegetable from underground. Economic plant photographs: Vegetables # 1, 2000.
http://daphne.palomar.edu/wayne/vege1.htm

Colocasia esculenta Schott grows in the tropical and subtropical areas, and produces a large central corm or tuber and many side cormels, generally known as cocoyam, taro, or dasheen (19, 43,45). Numerous cultivars of varieties occur in the areas where they are cultivated. Most are acrid and cannot be eaten raw; however, many cultivars appear to have been selected for low acridity and one is reported to be completely palatable uncooked (43). The sharp and harsh irritation of the throat and mouth (acridity) with the ingestion of uncooked material has long been recognized as a characteristic of the Araceae family. It is produced by raphides (43). Raphides are structures formed as needle-shaped crystals of calcium oxalate in plant cell vacuoles. Acridity may be removed by cooking using an acid treatment, or drying at high temperature (22,43).

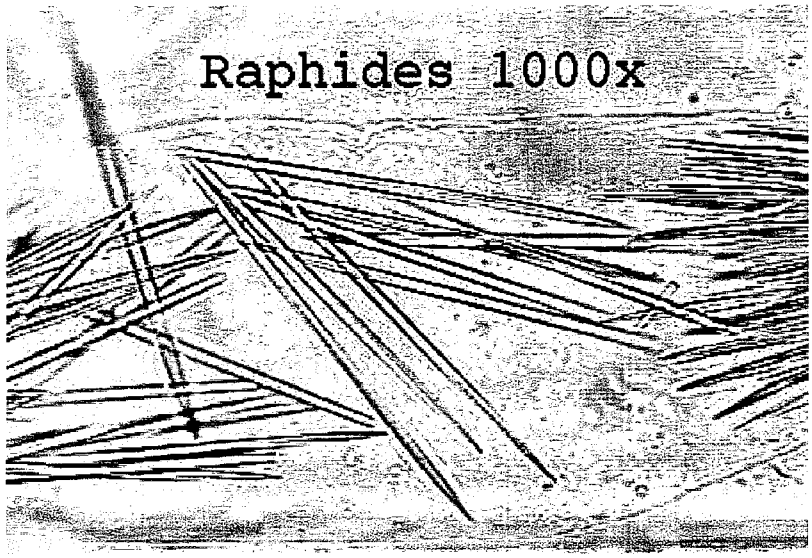


Figure 6. Raphides. Source: Merryl, R. Botany 130. General Botany Home Page. of the University of Wisconsin-Madison, 2000. http://www.wisc.edu/botit/Botany_130/Eukaryotic_Cell/Raphides.html

Chinese writings of 2000 years ago mention the cultivation of taro, *Colocasia esculenta*. Taro was one of the plants the first Polynesian settlers brought to Hawaii about 1500 years ago. Before 1778, about 300 varieties of taro were grown in Hawaii. Taro is usually cultivated in a complex terraced system of *lo'i* (field ponds) fed by *'auwai* (ditches). Wetland taro grows under a slow-moving layer of water throughout its life. At one time, taro fields covered the fertile floors of the windward valleys of many major islands (45). Today, because of the scarcity of water and agricultural land, only a few such areas remain. Except for a few large commercial ventures, most farmers cultivate taro part-time. Taro fields are quickly disappearing from the rural landscape. Poi, a pounded, paste-like food made from boiled taro and was once the most important staple in the Hawaiian diet and is today considered a rare delicacy (45). In this family, many plants have arrowhead shaped leaves (Figure 7). Taro is widely cultivated in the Pacific and especially in Polynesia. The part of the taro that is eaten is not a true root but rather a corm or tuber (Figure 8). A corm is an underground stem, like a rhizome, usually monocot storage rhizomes are called corms or tubers rather than rhizomes. The leaves of some varieties of taro can also be eaten and are sometimes called "callaloo." But it is important not to eat them if you are not certain, since some varieties can contain oxalic acid in their leaves (45).

Figure 7. *Colocasia esculenta* plants. Source: Kinnear, E. How to plant a native hawaiian garden. Hawaiian Plants Conservation Center, 2000. http://www.hawaii.gov/health/oeqc/garden/eioegkal.htm

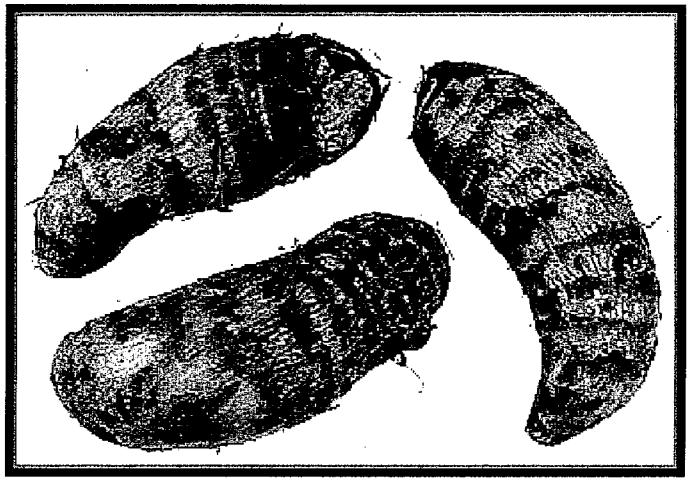


Figure 8. *Colocasia esculenta* tubers. Source: Armstrong, W.P. Wayne's World. Vegetable from underground. Economic plant photographs: Vegetables # 1, 2000. http://daphne.palomar.edu/wayne/vege1.htm

Xanthosoma sagittifolium's common names for its tubers are cocoyam, tannia, tanier, or yautia. Compared to *Colocasia esculenta*, tubers of *Xanthosoma sagittifolium* plants are the most resistant to diseases and require the least amount of moisture to grow (45). The cormels or tubers of *Xanthosoma sagittifolium* are harvested for food since the main corms are too acrid (45). *Xanthosoma sagittifolium* is a close relative of taro and is often confused with taro. They are, of course, in the same family, Araceae. Malanga is the name for *Xanthosoma sagittifolium* plants (Figure 9) in some areas, and sometimes they are called yautia, tannia, elephant ear, and even cocoyam (although cocoyam is supposed to refer to taro). Malangas are sold in many garden markets in the tropical areas and can also be found in Florida at hispanic markets. As with taro, the part of malanga that is eaten is the tuber (Figure 10).

Figure 9. *Xanthosoma saggitifolium* plants. Source: Duke, J.A. Legumes and starchy staples. BSC124. Lecture 26. College of Life Science of the University of Maryland, 2000. http://www.life.umd.edu/classroom/BSCI124/lec26.html

The leaves of malanga contain oxalic acid and should not be eaten (22) because the calcium is poorly absorbed (14). In tubers, calcium occurs principally in raphides (43) as was mentioned earlier.

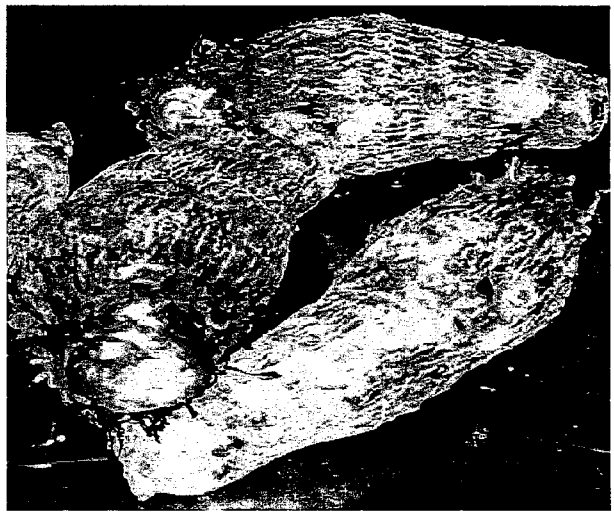


Figure. 10 *Xanthosoma saggitifolium* tubers.
Source: Slimak, K. Special Food Company, 2000.
http://www.specialfoods.com/malanga.html

Development of New Value-added Products

Xanthosoma saggitifolium, *Colocassia esculenta* and *Ipomoea batata* are tropical tubers that can be potentially transformed into flour or starch because they store a high starch content. *Xanthosoma saggitifolium*, *Colocassia esculenta,* and *Ipomoea batata* have starch contents between 23.8 to 30.0%; 22.0 to 40.3%, and 22 to 28%, respectively

on a wet basis (13, 22, 25, 26, 39, 47, 55). They have a short shelf life because of their high moisture content (13, 25) and high metabolic activity after harvesting (1, 2, 31, 41).

In 1998, the quarterly bulletin issued by the Food and Agriculture Organization of the United Nations (FAO) shows charts of root crops and tuber production, areas harvested, and yields as of February 28, 1998 for all countries belonging to this organization. The charts include roots and tubers, such as potatoes, sweet potatoes, cassava, yams, and taro (11).

The range of variability in sweet potatoes is so great that many different phenotypes can be made available for special product development depending on the characteristics needed. Often it is difficult to determine, even through trial and error, what the best characteristics are for particular products. Value-added products such as french fries, chips, and flakes have been developed from sweet potatoes, but none has been successfully marketed for any length of time (6). Much effort has been devoted to sweet potato fries. However, consumer comments often refer to the sweetness, texture, and oil content as problems. The products have always been developed from the existing single phenotype grown currently in the United States that may be a major reason for the disappointing results with these products (6).

Conversion of tubers into flour and starch is technologically feasible. There exist methods such as wet and dry milling and conventional dehydration techniques (37, 38, 44, 55) that have already been used to produce starches and flours from other substrates. Examples of these are wheat, corn, potato, unripe plantain, among others, that could be used to produce starch and flour from the tubers.

Physical and Chemical properties of starches

Amylose and amylopectin do not exist free in nature, but as components of discrete, semicrystalline aggregates called starch granules (Figure 11). The size, shape, and structure of these granules vary substantially among botanical sources (Figure 12) (54). Starch, from any source, exits in the form of white granules of varied size and form; these granules are organized structures (Figure 13), although their existence in relation to that of the cell is transitory (54). They are the first formed products of assimilation, insoluble in the ordinary cell-sap of the plants containing them, through a process of organization analogous to that by which the development of the cell itself is effected. When these minute granules acquire appreciable dimensions, concentric lines may be observed, more or less distinctly in different cases; a relevant example is the granules of the potato-starch. These lines increase in number with an increase in size, and in many cases, become eccentrical from the preponderating growth of one side of the granule (9,49,54,58). In freshly extracted granules, the original center generally appears solid, or with a minute black point; but if the starch is dry, the center appears hollow, sometimes it is even occupied by air with some starch grains containing a large cavity. If alcohol is applied to the fresh grains, the extraction of water, likewise, produces a hollow in the central point of growth, and in all these cases, cracks typically run out toward the surface. The lines in the starch granules are the boundaries of concentric superimposed layers. Sometimes these lines are very distinct and faint. Quite often, more distinct lines appear at intervals in the series of the same granule, and in these cases, a thin vacancy, or in the dried granules a stratum of air seems to exist between the layers. The specific

gravity of starch is 1.53, and its chemical composition is $C_6H_{10}O_5$, or a multiple of this formula (44,49,54).

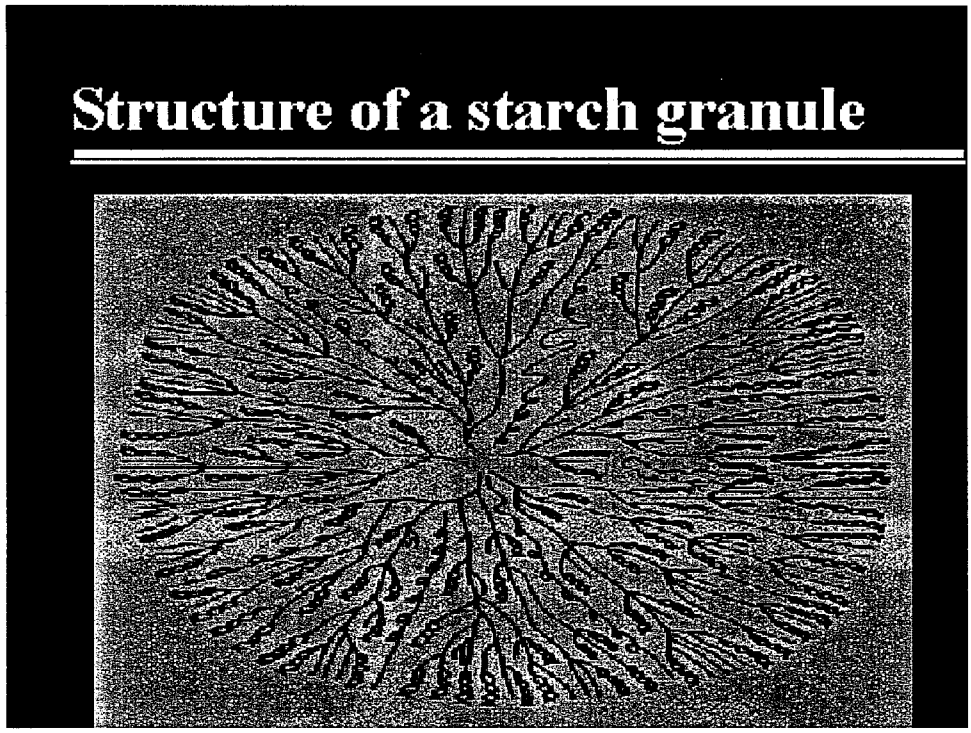


Figure 11. Structure of starch granule. Source: Price, R.L. History of sauces. College of Agriculture and Life Science of the University of Arizona, 1998. http://ag.arizona.edu/nsc/courses/251nsc/sauces/sld009.htm

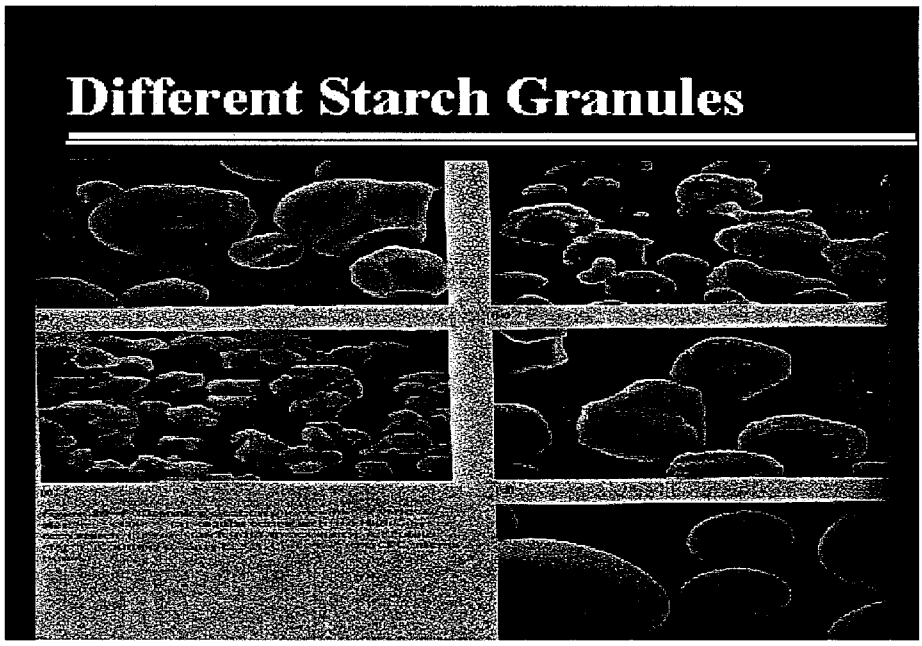


Figure 12. Different starch granules. Source: Price, R.L. History of sauces. College of Agriculture and Life Science of the University of Arizona, 1998. http://ag.arizona.edu/nsc/courses/251nsc/sauces/sld009.htm

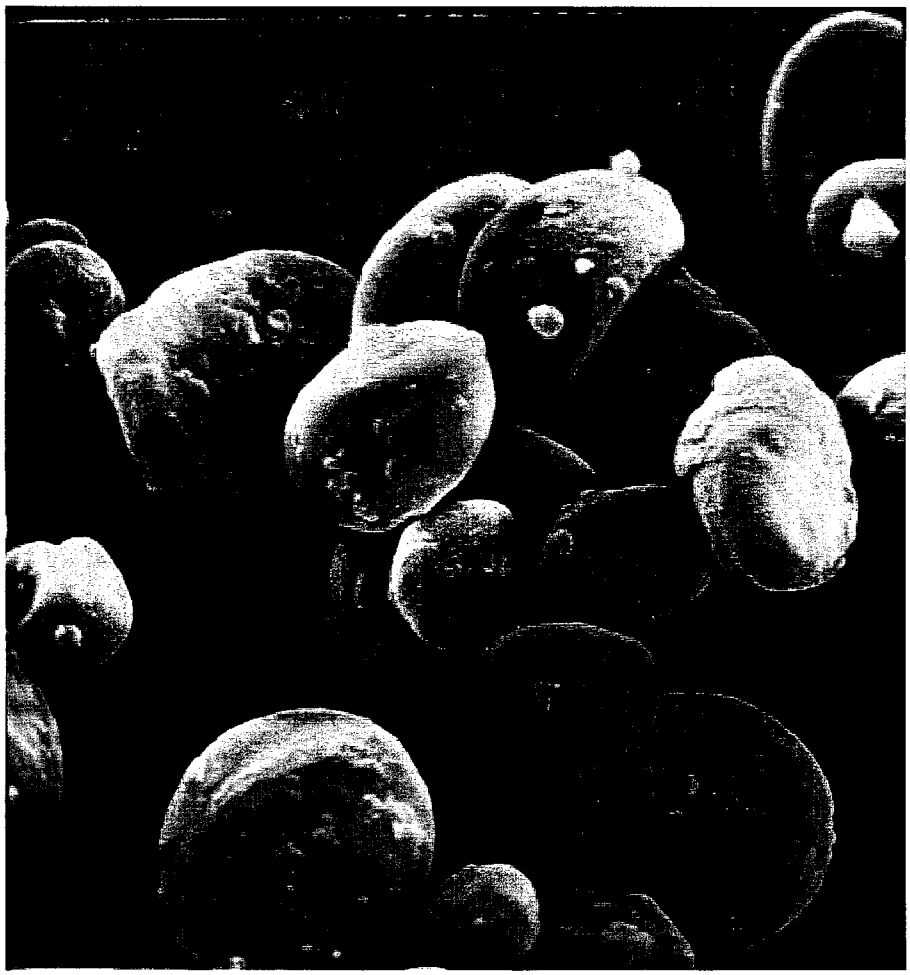
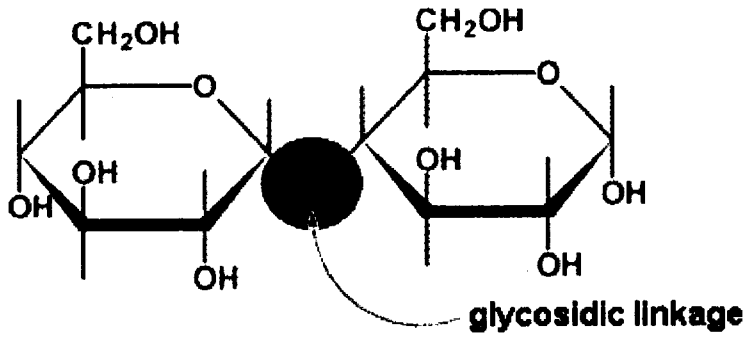


Figure 13. Wheat starch. Source: Morton, S. Functional properties of starches. Food and Agriculture Organization of the United Nations (FAO), 1999. http://www.fao.org/WAICENT/FAOINFO/AGRICULT/ags/agsi/starch41.htm

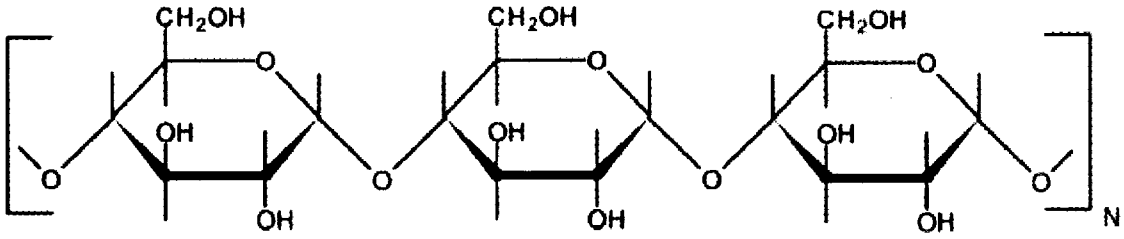
Because of their composition, starch and products derived from starch are used to modify the physical properties of many foods. As mentioned above, starch consists primarily of D-glucopyranose polymers linked together by α-1,4, and α-1,6 glycosidic bonds. Glucose polymerization in starch results in two types of polymers: amylose and amylopectin. Amylose is considered to be an essentially linear polymer composed almost entirely of a α-1,4-linked D-glucopyranose or maltose units (Figure 14 and 15). Recent evidence has suggested that some branches are present on the amylose polymer;

whereas, the amylopectin molecule is much larger and more branched (Figure 16 and 17). The structural differences between these two polymers contribute to significant differences in the starch properties and functionality (37, 38, 44, 51, 54, 59).



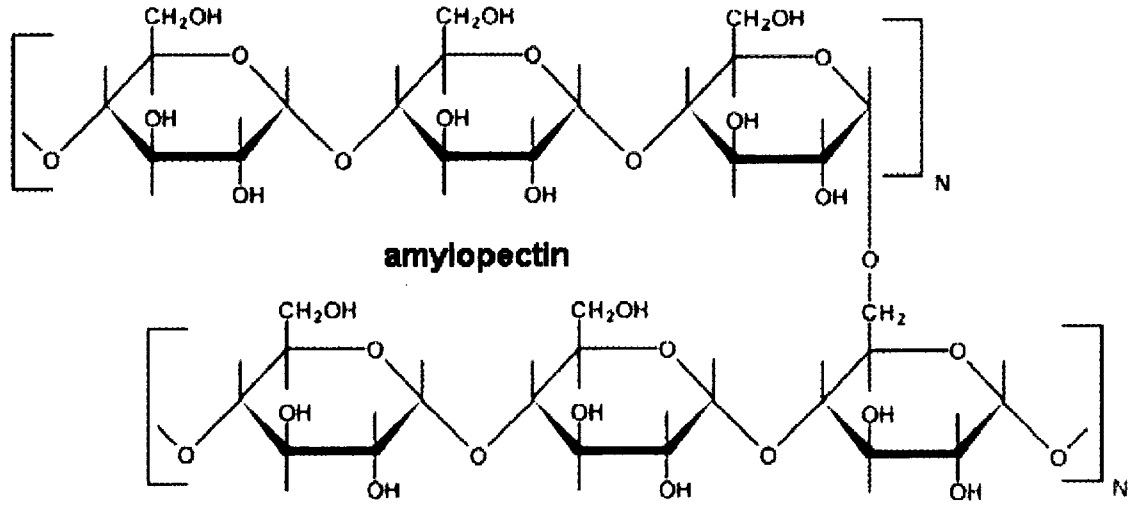
maltose (glucose - glucose)

Figure 14. Maltose structure. Source: Chemical Technology, 2000. http://www.chemtech.org/cn/cn2325/2325-12.htm#1



amylose
(n = several thousand)

Figure 15. Amylose structure. Source: Chemical Technology, 2000.
http://www.chemtech.org/cn/cn2325/2325-12.htm#1



amylopectin

Figure 16. Amylopectin structure. Source: Chemical Technology, 2000.
http://www.chemtech.org/cn/cn2325/2325-12.htm#1

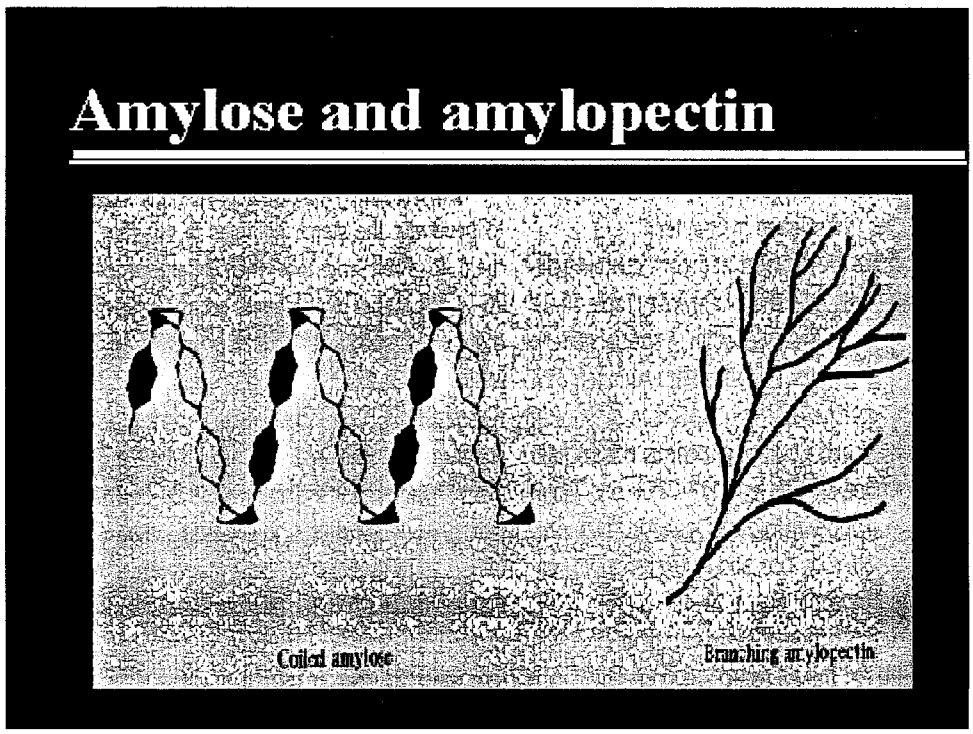


Figure 17. Amylose and amylopectin structures. Source: Price, L. History of sauces. College of Agriculture and Life Science of the University of Arizona, 1998. http://ag.arizona.edu/nsc/courses/251nsc/sauces/sld009.htm

Starch granules usually contain 10-20% (w/w) moisture and small amounts of proteins, fatty materials, phosphorous, and traces of mineral elements and inorganic salts (49). For example, tuber starches contain covalently bound phosphates (54). The phosphorous in the cereal starches is mainly present as phospholipids. The root starches (e.g., tapioca) contain a very low amount of phosphorous compounds. Potato starch is the only commercial starch that contains an appreciable amount of bound phosphate ester groups. The ester phosphate groups are bound to the C-6 position of glucose units of amylopectin molecules in potato starch. The number of phosphate groups in potato starch ranges from 1 phosphate group per 200 to 400 glucose units (37,49,51). The phosphate substituent confers on potato starch amylopectin the properties of a polyelectrolyte when dispersed into aqueous solutions. The mutual repulsion of the

charged group forces the molecule to expand. The phosphate group can be considered an ion-exchanging group (37,49). Generally, potato starch shows a higher paste viscosity than other starches when compared at similar conditions. This may be explained by the influence of the phosphate groups in potato starch. A higher phosphate content in potato starch results in a higher viscosity. The root and waxy starches also tend to have a higher paste viscosity (49).

All commercial starches contain minor or trace quantities of inorganic materials. Collectively, these minerals and salts are referred to as ash. The approximate concentration of these materials is determined as a residue after complete combustion at a specified temperature. This residue is reported as percent of ash. The ash content of the commercial starches contains mainly sodium, potassium, magnesium and calcium as metal compounds (49). As was mentioned earlier, tuber starches contain covalently bound phosphate groups (54). Ash content can vary depending upon the source of the raw material, agronomic practices, milling procedures, and types of chemical modifications that may be done to the starch. The ash content of starch is typically less than 0.5% of the dry matter (54).

Functional properties and analysis of starches and flours

The characterization of the functional properties of these non-conventional starches and flours is necessary in order to use these staples in a commercial environment. Characterization of functional properties can be performed through the gelatinization profiles. These profiles are defined by the phenomenon described below:

1. Gelatinization means intermolecular bonds break with increasing temperature, and H-bonding sites absorb more water, therefore increasing randomness in the structure, decreasing crystallization regions, and loss of birefringence is observed. High amylose content starches are difficult to gelatinize over 100°C and can form films and fibers with greater solubility and swelling under alkaline conditions. Their helical structure may entrap fatty acids and retard granule swelling.

2. Pasting is the phenomenon following the gelatinization process in the dissolution of starch. It involves granular swelling, exudation of molecular components from the granule, and eventual total disruption of the granules.

3. Retrogradation is related to the amount of branching. H-bonding between OH groups in amylose in gelatinized starches during cooling produces retrogradation. The water is forced out of the gel structure and the starch is insolubilized. High amylopectin starches (e.g., waxy maize) do not produce retrogradation when frozen (5, 20, 30, 59).

The gelatinization process is of primary importance in the food industry because of its impact on the texture of starch-based food. A number of methods that follow the gelatinization process have been devised based on turbidity, swelling, solubility, absorption of dyes, X-ray diffraction, birefringence, enzymatic digestibility, NMR, light scattering and DSC. DSC is particularly well suited to investigate the heat-induced phase transition of starch/water systems because it is capable of studying these processes over a wide range of temperatures and moisture content (5, 8). DSC is an instrument that raises the temperature of a sample at a fixed rate and over a fixed range and records the heat used to raise the temperature in a thermogram (5). When the heat used is plotted as a

function of temperature, a straight line will result if no thermodynamic event happens to the sample during the heating process. If heat-absorbing events occur, such as melting or heating through a glass transition, a peak will appear at the temperature at which the event occurred. If heat-evolving events occur, such as freezing or cooling through a glass transition, an inverted peak will occur (8).

CHAPTER III. MATERIALS AND METHODS

Materials.

Commercial *Manihot esculenta C.* starch was obtained from Alfonzo Riva C.A, Cagua, Venezuela. Starches of *Xanthosoma sagittifolium, Colocasia esculenta,* and *Ipomoea batata* were obtained following the method described by Pérez et al, 1998 (33), with some modification. The cleaned tubers were peeled, weighed, sliced and ground for 2 minutes at high speed in a Waring blender with small volumes of distilled water. The homogenate was passed through an 80-mesh sieve. This grinding and screening operation was repeated 4 more times. The resulting slurry was passed consecutively through a 200-mesh and a 270-mesh sieve and centrifuged at 1500 rpm for 20 min. After removing the mucilaginous layer, the sediment was washed several times by suspension in distilled water and centrifuging until it appeared to be free of non-starch material. The sediment was then dried in an oven at 45 °C. The *Xanthosoma sagittifolium, Colocasia esculenta,* and *Ipomoea batata* dried starches were blended, passed through an 80-mesh sieve, and stored at room temperature in sealed plastic bags.

Flours were obtained from *Xanthosoma sagittifolium, Colocasia esculenta,* and *Ipomoea batata* tubers by conventional dehydration techniques (18). Fifteen pounds of each tuber was obtained directly from the Venezuelan farmers (to be sure they were not mixed with other varieties), then cleaned, peeled, and sliced into 2 inch diameter slices. Slices of the edible portions were dehydrated at 45°C for 24 h in a dehydrator (Mitchell, Mod. 645159). The relation of temperature/time used for dehydration was lower than the expected initial gelatinization temperature for each starch.

Methods.

Starches and flours of each tuber were analyzed for moisture (air oven method), and ash (direct method) content, as a percentage (w/w), following methods N° 14.004, and 14.006 respectively as described in AOAC, 1980 (3). Phosphorous content, as a percentage (w/w), was determined following the photometric method N° 7123 as described by AOAC, 1980 (3) (see Appendix 1). Calcium content, as a percentage (w/w), was analyzed using an atomic absorption (AA) spectrophotometer following method described by AOAC, 1980 (3) N° 49.001 and Perkin Elmer AA Manual, 1968 (36). The standard curve was performed using $CaCl_2$ as a stock solution (See Appendix 2). Amylose content, as a percentage (w/w), was analyzed using a DSC method described by Mestre, 1996 (24). Calorimetric determination of amylose was performed using a Perkin Elmer DSC instrument (DSC 4; Perkin Elmer, Norwalk, USA). Samples (approximately 11 mg starchy material and approximately 5 mg of pure potato amylose: A0512 Sigma Type III) were weighed accurately in a medium pressure pan (70 µl) using a 0.01 mg precision balance. Then 50 µl of 2% solution of L-α-lysophosphatidylcholine: L4129 Sigma Type I from egg yolk (LPC) was directly added and the pan was hermetically sealed. The pan was stored for one hour before the analysis was performed. The sample pan was placed in the sample cell and a pan filled with 50 µl of water was placed in the reference cell. The temperature was raised from 25 to 160°C at a rate of 15°C/min and kept at this temperature for 2 min. The temperature was then decreased from 160 to 25°C at a rate of 5°C /min. Enthalpic data were collected during the cycle. The exotherm of

gelatinization for pure potato amylose was performed in duplicate. The amylose content, as a percentage was calculated using the equation below:

$$\% \text{ amylose} = \frac{100 \times \text{amylose weight} \times \Delta H_1}{\Delta H_2 \times \text{sample weight}}$$

ΔH_1 : Enthalpy change of the sample

ΔH_2 : Enthalpy change of the amylose

The amylose content was also determined by a colorimetric method described by McGrance, et al.,1998 (23), Whistler, 1964 (58), and Whistler and Paschall, 1967 (59). The standard curve was performed using pure potato amylose: A0512 Sigma Type III (see Appendix 3). The gelatinization profiles reported in degrees Celsius were performed following methodologies described by Davis, 1994, (8) and Pérez, et al., 1998 a, b (34,35). The gelatinization profile describes the change of enthalpy for the sample for the first, middle, and end points of the peak over the isotherm region. A microbiological test for aerobic count and yeast and mold count were performed following the pour-plate method described in Bacteriological Analytical Methods (4) and Food Microbiology UW-Stout 308-506 Laboratory Methods (12). Plate count agar and potato dextrose agar/tartaric acid were used to plate aerobic and yeast and mold, respectively. A correlational study for parameters such as phosphorous, calcium, gelatinization profile, and enthalpy changes (ΔH) associated with ash and amylose content of *Xanthosoma sagittifolium*, *Colocasia esculenta, Ipomoea batata,* and *Manihot esculenta* starches and flours was performed following methods described by Crowl, 1993 (7). The correlation coefficient of each two set of parameter was determined. The correlation coefficient is a statistical measure of the degree of relationship between two quantitative variables. The statistical symbol used for the correlation coefficient is ***r***. Also was determined the regression line of each set of variables in order to use value of one variable to predict

values of the other variable (Crowl, 1993). The manuscript was prepared following guide described by Turubian, 1987.

CHAPTER IV. RESULTS AND DISCUSSION

Moisture and ash content of flours and starches obtained from each tuber

Table 1 shows the moisture and ash content of flours and starches obtained from each tuber. The moisture of these starches and flours are among the moisture range generally accepted for dry products in order to obtain a desirable shelf life (49). The moisture content of flours and starches depends on the relative humidity (RH) of the atmosphere in which they have been stored. If the RH decreases, the starches give up moisture. If the RH increases, they absorb humidity (49). The equilibrium moisture content of starch is also dependent on the type of starch products. Under normal atmospheric conditions, most commercial native starches contain 10-20% (w/w) moisture (e.g., corn, sweet potato, and tapioca starches contain ca. 13% moisture, and potato starch contains 19% moisture)(49). Similarly, wheat flour has a moisture content at the same conditions of 14% or less (62).

All commercial flours and starches contain minor or trace quantities of inorganic materials. The approximate concentration of these materials is expressed as a percentage (w/w) of ash content. Usually the ash of commercial starches contains mainly sodium, potassium, magnesium, and calcium as metal compounds. But the ash content of potato and cereal starches is correlated with the amount of phosphate groups and partly with the amount of phospholipids (49). The range of ash for wheat flour is 2.0-2.7% (58) and for wheat starch is 0.07-0.5 % (49). As is shown in Table 1, the ash content of the tropical tuber starches fall in the range found in the literature for commercial starches (49). In contrast, the tropical tuber flours exhibited a higher ash content (2.48-4.10%) than those

found in the literature for wheat flours (2.0-2.7%)(62). Because of how the flours were obtained, the mineral content of flours is only dependent on the botanical source. In contrast, due to the isolation methods to obtain starches, their mineral content is dependent not only on botanical source, but also is dependent on the extraction methods. The ash content of the isolated starches falls in the range of the commercial starch ash content.

Table 1. Moisture and ash percent (w/w; dry basis) composition of starches isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomoea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	**% Moisture**		**% Ash**	
	Flour	**Starch**	**Flour**	**Starch**
Xanthosoma sagittifolium	10.83 ± 0.32	13.43 ± 0.01	4.10 ± 0.03	0.20 ± 0.04
Colocasia esculenta	10.66 ± 0.42	14.01 ± 0.05	2.62 ± 0.03	0.31 ± 0.01
Ipomoea batata	8.35 ± 0.43	13.29 ± 0.08	2.48 ± 0.22	0.15 ± 0.03
Manihot esculenta C. (commercial product)	NA	13.63 ± 0.12	NA	0.12 ± 0.02

NA: Not Available

Microbiological population of flours and starches obtained from each tropical tuber

Aerobic mesophilic bacteria are used as indicators of unsanitary practice. High viable counts often indicate contaminated raw materials, unsatisfactory sanitation, unsuitable time/temperature conditions during production or storage, or a combination of these (52). However, Mountney and Gould, 1988 (27), pointed out that the bacterial

count of flours might range from 20,000 to 500,000/g. An extremely heterogeneous flora is present, which may include many secondary invaders as well as the epiphytic flora of the botanical source. Cereal flours may also contain an appreciable number of mold spores that must be destroyed during baking (27). In a detailed study of the microbiology of dehydrated food products, Jay, 1996 (17), shows that dehydrated food products have an aerobic plate count (APC) of < 10,000/g, and dehydrated soup has a APC of less than 100,000/g. Dried desiccated or low-moisture (LM) foods are those that generally do not contain more than 25% moisture and have an a_w (water activity) between 0.00 and 0.60. These are the traditional dried foods and they are shelf-stable foods (17). As is shown in Table 2, except for *Ipomoea batata* flour, the APC values are less than 10,000/g.

Molds and yeasts play an important role in foods. Some molds and yeasts are desirable, but in dried foods, such as flour and starches, they are undesirable, because they can produce toxins and become a significant public health risk. In this study, except for *Colocasia esculenta* starch (that was relatively low), no population of molds and yeasts was detected in starches and flours as shown in Table 2. The relative high APC shown for *Ipomoea batata* flour is probably due to its high moisture and sugar content (61). Table 2 shows that both the starches and flours were well manufactured because of a low viable count. In conclusion, we can expect that these aroid flours and starches would have a stable shelf life.

Table 2. Population of aerobic plate count (A.P.C), yeast and molds in starches* isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomoea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	A.P.C. (CFU/g)		Yeast and Mold (CFU/g)	
	Flour	Starch	Flour	Starch
Xanthosoma sagittifolium	4 ,000	4,000	0	0
Colocasia esculenta	1,000	7,900	0	2,000
Ipomoea batata	40,000	5,000	0	0
Manihot esculenta C. (commercial product)	NA	2,000	NA	0

* Expressed as CFU: Colonies forming units/g
NA: Not Available.

Phosphorous content in starches and flour obtained from each tuber.

Phosphorous content is an important parameter used to define the functional properties of starches and flours. Potato starch usually shows a higher paste viscosity than the other starches. A higher phosphate content in potato starch results in the higher observed viscosity. The root (e.g., tapioca starch) and waxy starches tend also to have a higher paste viscosity (49). As is shown in Table 3, *Ipomoea batata* starch has higher phosphorous content than those shown for the other aroid flours and starches. The content of phosphorous in tuber starches is typically less than 500 mg /100 g and is usually referred to as ash (54). It has been reported that the *Ipomoea batata* phosphorus content varies from 9 – 22 mg/100g among starches from different varieties (61). It has also been reported that one phosphate group typically exits per 200 - 400 glucose units in a starch molecule (49). As a result of this, a higher viscosity may be expected in *Ipomoea batata*

starch when compared to those of the other tuber flours and starches. In order to establish a correlation between ash and mineral content, an analysis of correlation was performed using the data obtained by determination of the ash (Table 1) and phosphorous content (Table 3) of the aroid flours and starches (Table 9, Appendix 4 and 5). A relatively high positive linear correlation ($r = + 0.9401$; $R^2 = 0.8852$) was found between phosphorous and ash content of the flours (Appendix 4). The starches do not show the same tendency ($r = - 0.4703$; $R^2 = 0.2238$; Appendix 5). In regards to the negative correlation of the phosphorous/ash content, it is possible that the starch isolation methods can influence the phosphorous/ash relationship. The relationship is most likely related to the kind of phosphorous linkages that occur in the molecular structure of the edible portion of the tubers. A positive correlation may be expected when using data from several varieties as well as from numerous samples of the same species, but it may not be expected when using data from different families.

Table 3. Phosphorous content (mg/100 g sample; dry basis) isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomoea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	**Phosphorous content (mg/100g)**	
	Flour	**Starch**
Xanthosoma sagittifolium	**3.68 ± 0.43**	**0.07 ± 0.001**
Colocasia esculenta	**2.29 ± 0.58**	**0.01 ± 0.01**
Ipomoea batata	**2.67 ± 0.32**	**0.32 ± 0.01**
Manihot esculenta C. (commercial product)	**NA**	**0.05 ± 0.01**

NA: Not Available

Calcium content (Ca^{++}) of flours isolated from each tuber

The sharp and harsh irritation of the throat and mouth produced by acridity with the ingestion of uncooked material has long been recognized as a characteristic of the monocot family Araceae; however, *Colacasia* and *Xanthososma* have varieties that are less acrid. For long-term storage, *Colocasia* is usually processed into flour that requires cooking, acid treatment, or high temperature drying to remove the acridity (43). However, it is necessary to evaluate the calcium concentration in flours in order to properly remove the acridity of the flours. Because the calcium content of starches is not relevant, Table 4 shows only the calcium content of the aroid flours. As expected, because *Colocasia esculenta* is a member of Araceae, its flour has a relatively higher calcium content than the other two. However, *Xanthosoma sagittifolium*, even though it belongs to Aracea family, has the lowest value. Contrary to what was expected, *Ipomoea batata* flour, even though it belongs to another family not commonly associated with acridity, shows an intermediate value. We believe that because of the relatively high drying temperature, the acridity was removed from the flours, despite the fact that raphides could not be altered. Some investigations have shown that acridity can be removed without affecting the raphides. When correlation was calculated between ash and calcium content in tuber flours, there was a relative low negative correlation ($r = -0.6905$; $R^2=0.4541$) (Table 9; Appendix 6).

Table 4. Calcium content (Ca^{++}) as percent (w/w; dry basis) composition of flours obtained by dehydration of edible pulp of *Xanthosoma sagittifolium, Colocasia esculenta,* and *Ipomoea batata.*

Tubers Specie	mg Ca^{++}/100g	% Ca^{++}
Xanthosom sagittifolium	**280 ± 0.02**	**0.28 ± 0.02**
Colocasia esculenta	**720 ± 0.02**	**0.72 ± 0.02**
Ipomoea batata	**420 ± 0.02**	**0.42 ± 0.02**

The amylose content in starches and flours obtained from each tropical tuber

The amylose content in both starches and starchy flours has an important effect on their functional properties. Therefore, it is quite important that the amylose content be quantified for food processing and quality. However, the literature has pointed out a controversy related to amylose determination. The main points of the controversy are lipids and proteins concentration, extraction methods, and occurrence of intermediate material, long of the amylose molecule, and starch solubilisation. In the colorimetric method was determined that coloration is influenced also for temperature, time of reaction, pH, and electrolyte concentration. In regard other procedures they are time consuming and the result are not clearly related to those obtained by iodine complexation methodologies. Therefore, many methods have been developed for measuring amylose content of various raw materials. In order to determine amylose, two methods are considered in this study. The methods considered were the colorimetric method (23) and the calorimetric method (24). The colorimetric method is the most widely used technique for amylose determination and is a colorimetric assay in which

iodine binds with amylose to produce a blue colored complex. The ability of amylose to form this complex is due to its size and particular conformation: the polymer has a three-dimensional helical structure. When the helical chains pack two by two to form a double helix, a central hydrophobic cavity, where molecules such as iodine can be fixed, is formed. This concept is quite true for a linear polymer, which is believed for the amylose structure. Yet, a non-linear structure, such as amylopectin has a very small iodine-binding capacity. Consequently, if the amylose structure is too small or has branching, its iodine-binding capacity will also be reduced (5, 8, 21, 23, 24, 54,59).

Recently, it has been proposed that differential scanning calorimetry be used to determine the amylose content in starchy materials (5,8,23,24). The calorimetric method to determine amylose is based on the formation and melting of a amylose-lysophosphatidyl-choline complex (5, 24). The procedure is relatively simple and fast. However, some discrepancies remain for certain types of starch samples (24).

As is shown in Table 5, amylose content of *Xanthosoma sagittifolium* and *Ipomoea batata* starches, as determined by the colorimetric method, was similar among them. *Manihot esculenta* starch value was similar to those show in literature (44,49,54). And it has approximately half the amylose content of the other three starches. The amylose content of the flours is quite similar for each tuber. This might be explained as a result of the drying method. During drying, the process of dextrinization can occur with the consequent decreasing of amylose molecular size. Thus using the colorimetric method to evaluate amylose content can lead to an error in the amylose content of flours. When amylose content was correlated with phosphorous content of starches and flours, an insignificant correlation was found in flours ($r = - 0.0705$; $R^2 = 0.005$) and starches

shown a low positive correlation ($r = + 0.4547$; $R^2 = 0.2067$) (Table 9; Appendix 7 and 8).

Table 5 Amylose content (colorimetric method) as percent (w/w; dry basis) composition of starches isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	Amylose %	
	Flour	Starch
Xanthosoma sagittifolium	**11.84 ± 0.51**	**35.34 ± 0.65**
Colocasia esculenta	**11.67 ± 0.32**	**30.62 ± 0.16**
Ipomoea batata	**12.54 ± 0.11**	**35.56 ± 0.24**
Manihot esculenta C. (commercial product)	**NA**	**16.89 ± 0.09**

NA: Not Available

Table 6 shows the amylose contents measured by differential scanning calorimetry (DSC) for starches and flours. Amylose content of flours and starches measured using the calorimetric method is higher than those shown in Table 5. The data obtained for these starches and flours by this method have introduced a degree of uncertainty. Mestre et al, 1996, reported different results in their study of amylose content in cereals such as corn, rice and sorghum using the colorimetric and DSC methodologies. They found that the DSC methodology for the determination of amylose content gave results very close to those obtained by the colorimetric procedure. However, this was not true in this study, where the reported results for amylose content range over 100% for *Xanthosoma sagittifolium* and *Colocasia esculenta* starches and below 1% for

Ipomoea batata flour. The results indicate that the DSC methodology used in this study was not capable quantifying the amylose content of the tropical tubers.

Concerns also exist with the data obtained by the colorimetric method for determination of the amylose content. The standard curve for amylose determination was obtained by using potato amylose as a standard. Although this methodology is well documented in the literature, it is quite possible that the molecular size of amylose in the potato starch could be different from that of the amylose in the unknown sample. On the other hand, the degree of gelatinization is an indicator of the effect of temperature on the functional properties of the flour and starches. In order to obtain native starches and raw flours, the drying process was performed using a lower drying temperature than those reported in the literature for initial gelatinization temperature of cereal starches. There is a consensus that starches of roots and tubers have a higher initial gelatinization temperature than those possessed by cereal starches (20,34,35,59). The processes of dextrinization and gelatinization can occur while drying the tuber to obtain the flour, with the consequent decrease of amylose molecular size. During dextrinization, repolymerization and transglucosidation occurs. These two processes change the amylose structure with a consequent decrease in the formation of the blue iodine/starch complex.

Table 6 Amylose content (DSC) as percent (w/w; dry basis) composition of starches isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	Amylose %	
	Flour	Starch
Xanthosoma sagittifolium	**60.62**	**106.9**
Colocasia esculenta	**66.96**	**102.7**
*Ipomea batata**	**0.080**	**91.63**
Manihot esculenta C. (commercial product)	**NA**	**96.88**

NA: Not Available

* *Ipomea batata* flour has low value because, this flour was gelatinized during the drying procedure, despite the drying temperature was below the initial gelatinization temperature reported in literature for tuber starches (20, 34, 35, 59).

Enthalpic changes (ΔH) in cal/g of starches and flour obtained from each tuber.

The starches in Table 7 show a similar ΔH that ranges between 3.312 to 3.999 cal/g. *Ipomoea batata* flour shows an ΔH of zero, which indicates that the *Ipomoea batata* flour was gelatinized during the drying process. The gelatinization process in *Ipomoea batata* flour could have occured because of intrinsic characteristics of this flour (e.g., its composition, such as moisture content, kind of carbohydrate, lipids, etc). *Xanthosoma sagittifolium* and *Colocasia esculenta* flours show a similar ΔH.

As is shown in Table 9 and Appendix 9, the amylose content of the tropical tubers (measured by colorimetric method) and the ΔH of gelatinization exhibits a high

significant negative linear correlation (r = - 0.9975; R^2 = 0.9951). A similar correlation was not observed for the starches of the tropical tubers, which showed a low positive linear correlation (r = -0.6055; R^2 = 0.3638). Calculation of correlation between amylose content measured by DSC and ΔH of flours and starches were not performed, because of the lack of DSC data for these starches and flours.

Table 7. Enthalpic changes (ΔH expressed in cal/g) measured using the DSC technique of starches isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta* and *Ipomea batata* and their flours obtained by dehydration of edible pulp.

Tubers Specie	**Enthalpy Change (ΔH) cal/g**	
	Flour	**Starch**
Xanthosoma sagittifolium	**2.344**	**3.470**
Colocasia esculenta	**2.680**	**3.999**
Ipomoea batata	**0***	**3.999**
Manihot esculenta C. (commercial product)	**NA**	**3.312**

NA: Not Available
* This value is zero because of the gelatinization of the *Ipomoea batata* flour during the drying process.

Gelatinization profiles of starches and flour obtained from each tuber.

Table 8 shows the gelatinization profile as °C measured using the DSC technique of starches isolated from aroids of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomea batata* and its flours. The gelatinization profiles of starches show in Table 8 are quite similar to those reported in the literature (34,35). The initial, middle, and end

gelatinization temperatures of each starch (Figures 19,20,1,22,23 and 24) are higher than that of *Manihot esculenta* starch (Figure 18). *Manihot esculenta* starch was used as a control. *Colocasia esculenta* starch has a narrower gelatinization range (Figure 22) than the *Xanthosoma* and *Ipomoea* starches (Figures 20 and 24). *Ipomoea batata* flour did not show gelatinization profiles because of its totally gelatinized state, as is shown in Figure 23. This flour can be considered modified flour. *Colocasia esculenta* flour (Figure 21) shows a narrower gelatinization range (70.5-94.3 °C) than *Xanthosoma saggitifolium* flours (Figure 19). A significant correlation between amylose content (measured by colorimetric method) and gelatinization profiles (ITG: {r = + 08146; R^2 = 0.6585}, MTG: {r = + 08821; R^2 = 0.7782}, ETG: {r = + 0.9761; R^2 = 0.9528}) for starches was not observed (Table 9; Appendix 10).

Table 8. Gelatinization profile (°C) measured by the DSC technique for starches and flours isolated from tubers of *Xanthosoma sagittifolium*, *Colocasia esculenta*, and *Ipomea batata*.

Tubers Specie	Flours (Temperature in °C)			Starches (Temperature in °C)		
	Initial	Middle	End	Initial	Middle	End
Xanthosoma sagittifolium	85.2	90.3	103.3	78.0	82.6	93.8
Colocasia esculenta	79.5	85.5	94.3	77.2	83.2	89.7
Ipomoea batata	0*	0*	0*	72.0	80.0	93.3
Manihot esculenta (Commercial product)	NA	NA	NA	65.2	72.0	85.2

NA: Not Available

* This value is zero because of the gelatinization of the *Ipomoea batata* flour during the drying process.

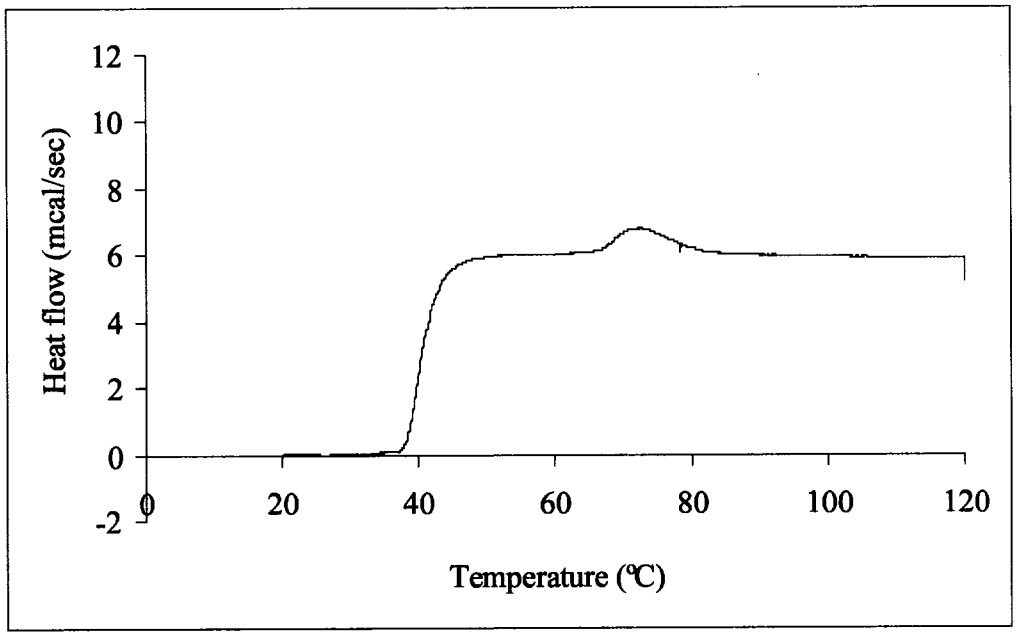


Figure 18. *Manihot esculenta* starch gelatinization profile

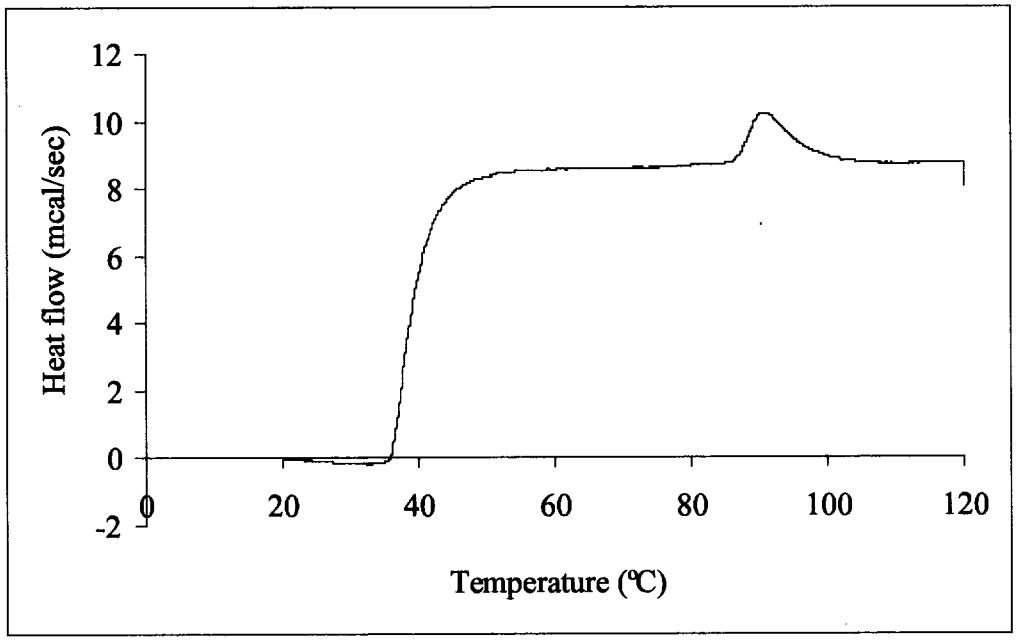


Figure 19. *Xanthosoma sagittifolium* raw flour gelatinization profile

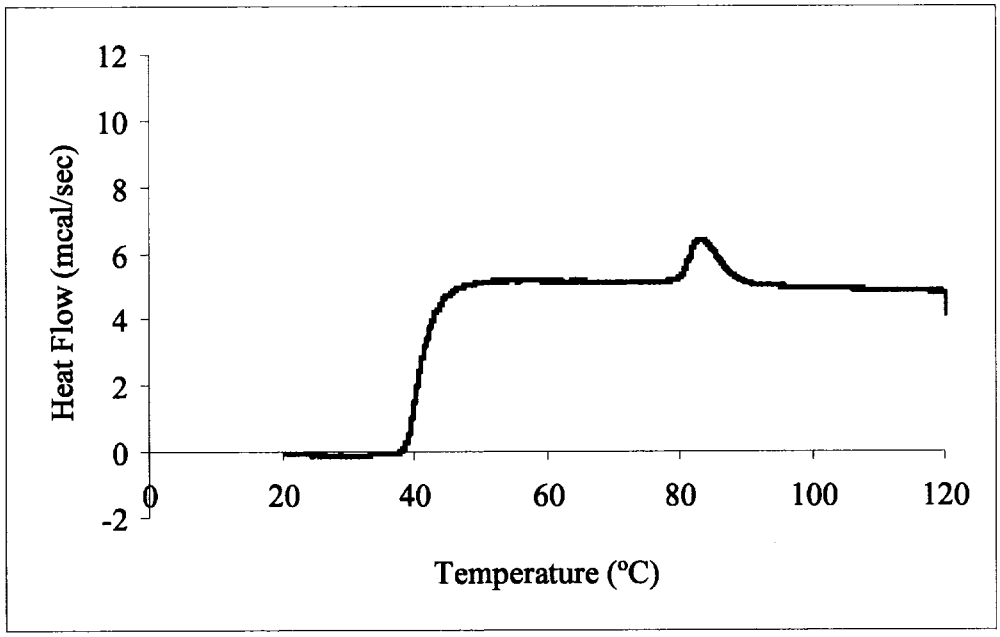


Figure 20. *Xanthosoma sagittifolium* starch gelatinization profile

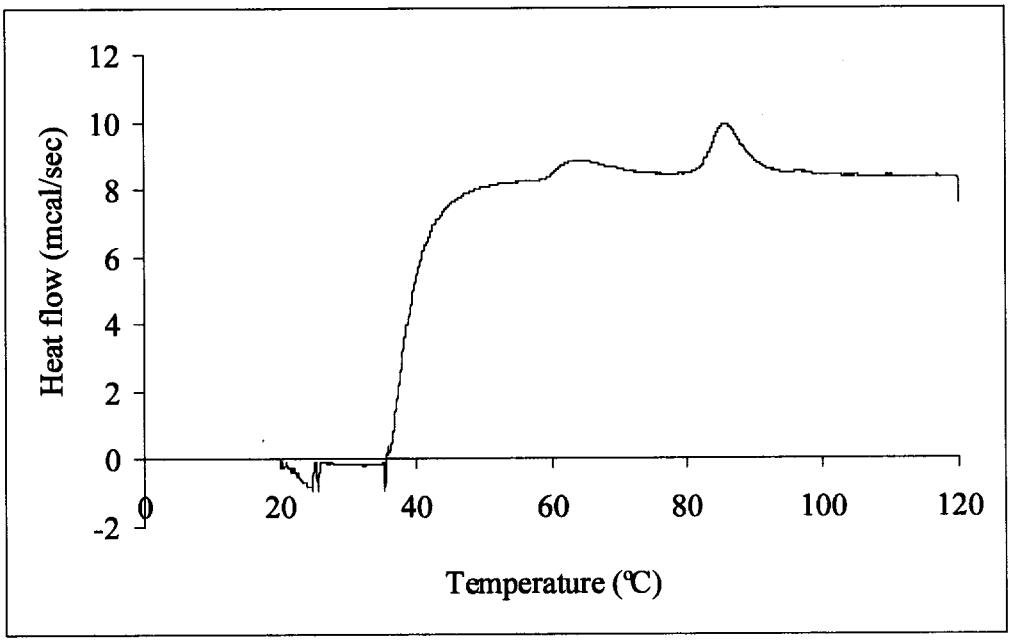


Figure 21. *Colocasia esculenta* raw flour gelatinization profile

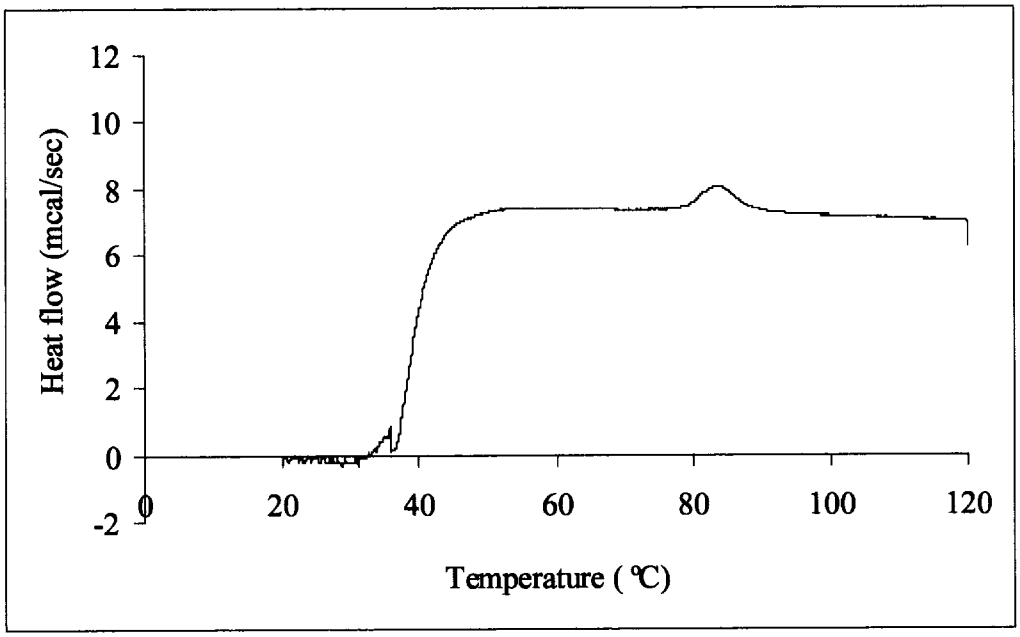


Figure 22. *Colocasia esculenta* starch gelatinization profile

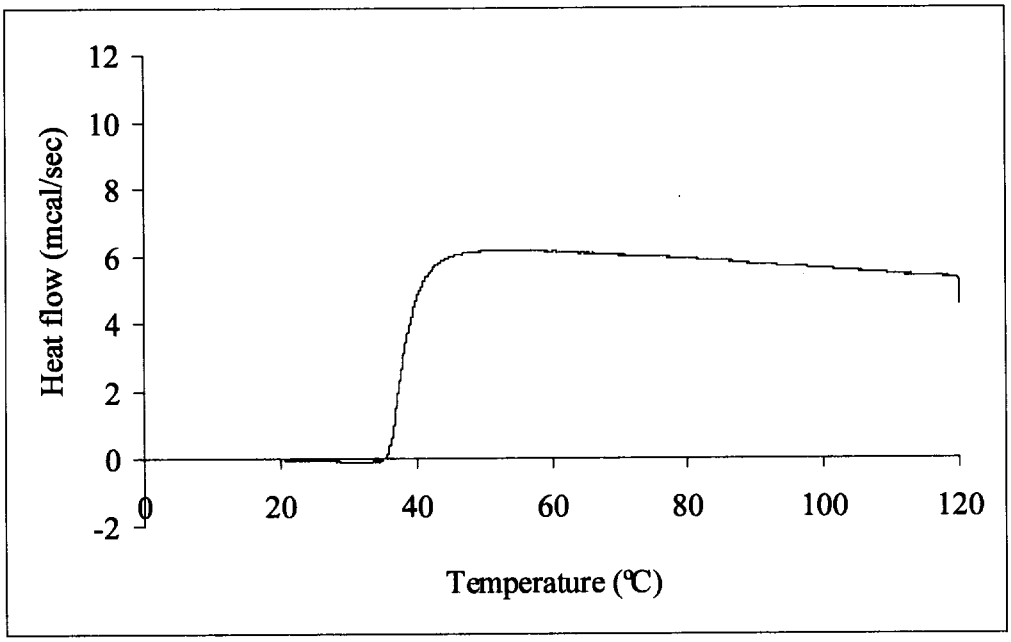


Figure 23. *Ipomoea batata* raw flour gelatinization profile

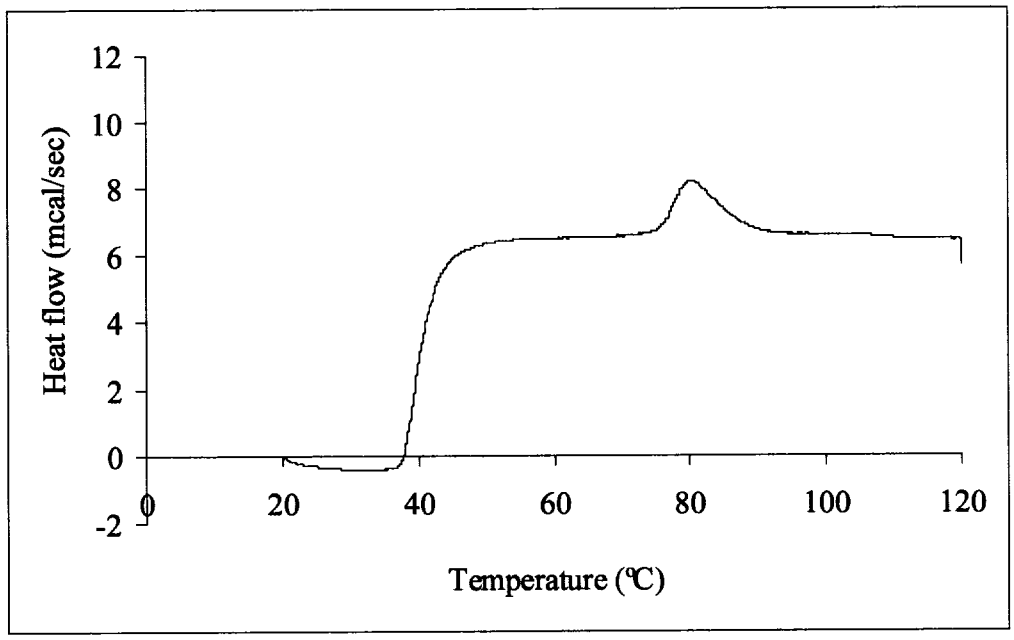


Figure 24. *Ipomoea batata* starch gelatinization profile

Table 9, shows a correlation matrix of the relevant parameters found in this study. The most significant relationships of the flour's parameters were found between the phosphorus and the ash content, and amylose content and ΔH. In regards to the starches the most significant relationship between parameters was found between amylose and gelatinization profile (ITG = initial temperature of gelatinization, MTG = middle temperature of gelatinization, and ETG = end temperature of gelatinization).

Table 9. Correlation coefficient (r) of flours and starches of *Xanthosoma sagittifolium*, *Colocasia esculenta, Ipomoea batata,* and *Manihot esculenta.*

	Flours		Starches	
Parameters	Ash	Amylose	Ash	Amylose
Phosphorous*	+ 0.9408	- 0.0705	- 0.4703	+ 0.4547
Calcium (%)	- 0.6905	-	-	-
Δ H (cal/g)	-	- 0.9975	-	+ 0.6055
I.T.G	-	-	-	+ 0.8146
M.T.G	-	-	-	+ 0.8821
E.T.G.	-	-	-	+ 0.9761

*(mg/100g)
I.T.G. = Initial temperature of gelatinization
M.T.G. = Middle temperature of gelatinization
E.T.G. = End temperature of gelatinization

CHAPTER V. CONCLUSIONS AND RECOMMENDATIONS

It can be concluded that the moisture content of starches and flours of the tropical tubers studied are similar to the moisture content generally accepted for safe storage. Except for *Xanthosoma sagittifolium* flour's ash content, the ash content of the tubers was comparable to those found in the literature for other common starches. In regards to the flours of the tropical tubers, they have higher ash content than those found in the literature for wheat flour. It also can be concluded that the mineral content of the flours is dependent on the botanical source, while the mineral content of the tuber starches is dependent on the botanical source as well as the isolation method. The population count of microorganisms in the flours and starches was determined to be safe and adequate for dehydrated food products. As expected for flours, a positive correlation between phosphorous content and ash content was found. A similar correlation was not identified with the tuber starches. *Ipomoea batata* starch has a higher phosphorous concentration than the other three. When the phosphorous content of *Xanthosoma sagittifolium* and *Colocasia esculenta* starches is compared with that of *Manihot esculenta* starch, they have similar phosphorous content values. The calcium content is higher in *Colocasia esculenta* flour than that shown by the other two aroid flours. The amylose content (colorimetric method) of the *Colocasia esculenta* starches and flour is the lowest of the three tuber species. Contrary to normal expectations, insignificant correlations were found between amylose content and ash content and amylose content and phosphorous content. Except for *Ipomoea batata* flours, the enthalpic changes were similar among the

starches and flours. Starch gelatinization profiles of common starches found in the literature were similar to those shown in this study. A relatively significant high positive correlation between amylose content and gelatinization profiles expressed as ITG, MTG and ETG of starches was observed.

Based on the results of this study, it is recommended that research continue in development of methodologies to obtain raw flours and native starches without gelatinization. It is also recommended that development of a universal test for amylose content be developed. Finally, based on the positive results of this study, it is recommended that the use of these tropical tubers be examined more thoroughly in the food industry, especially that of Venezuela.

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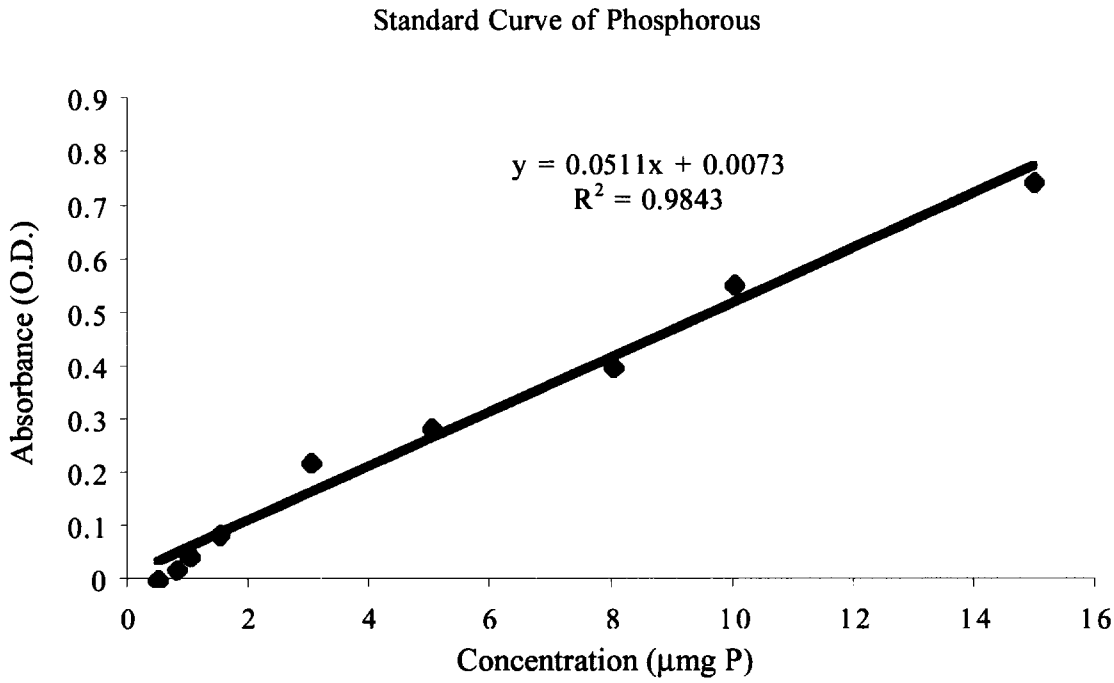
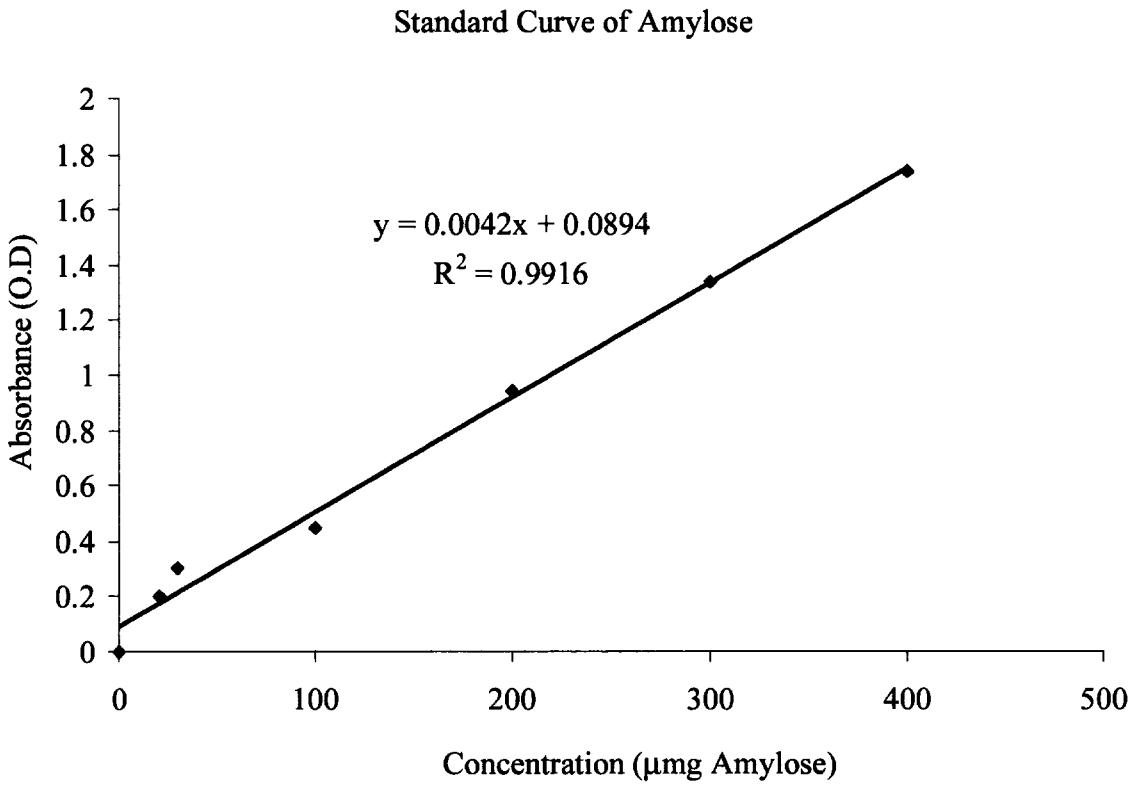
APPENDIX

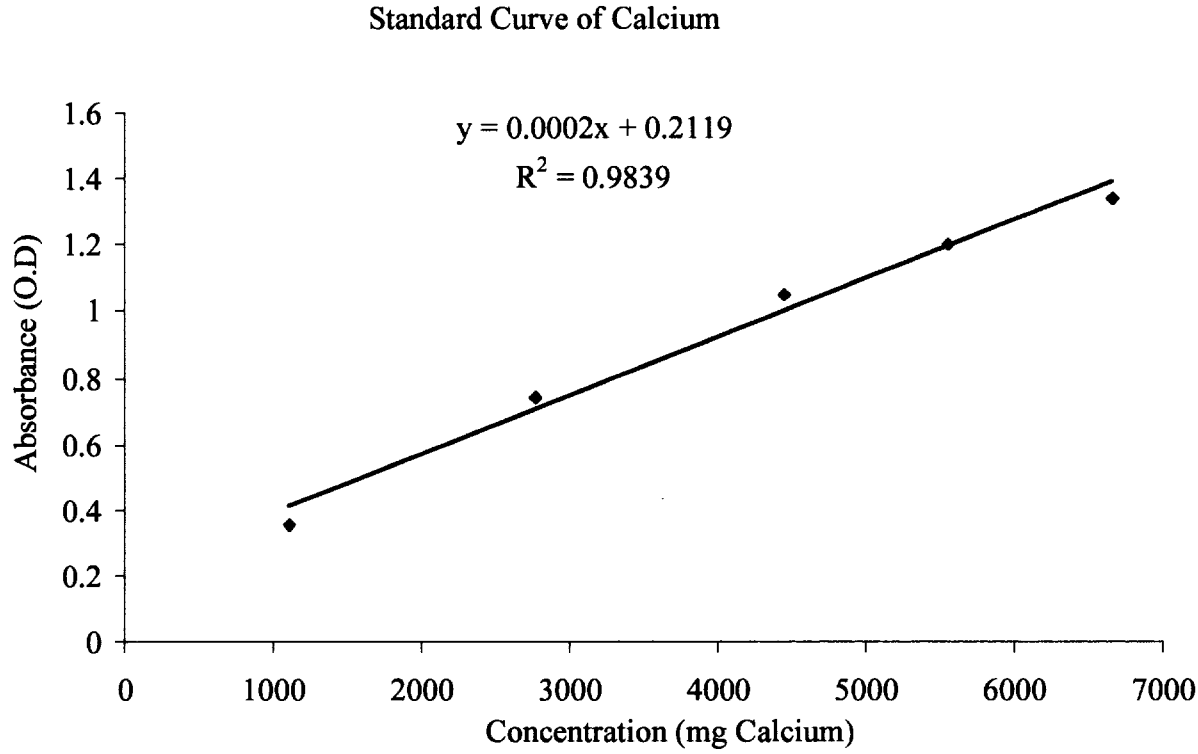
Appendix 1



Appendix 2

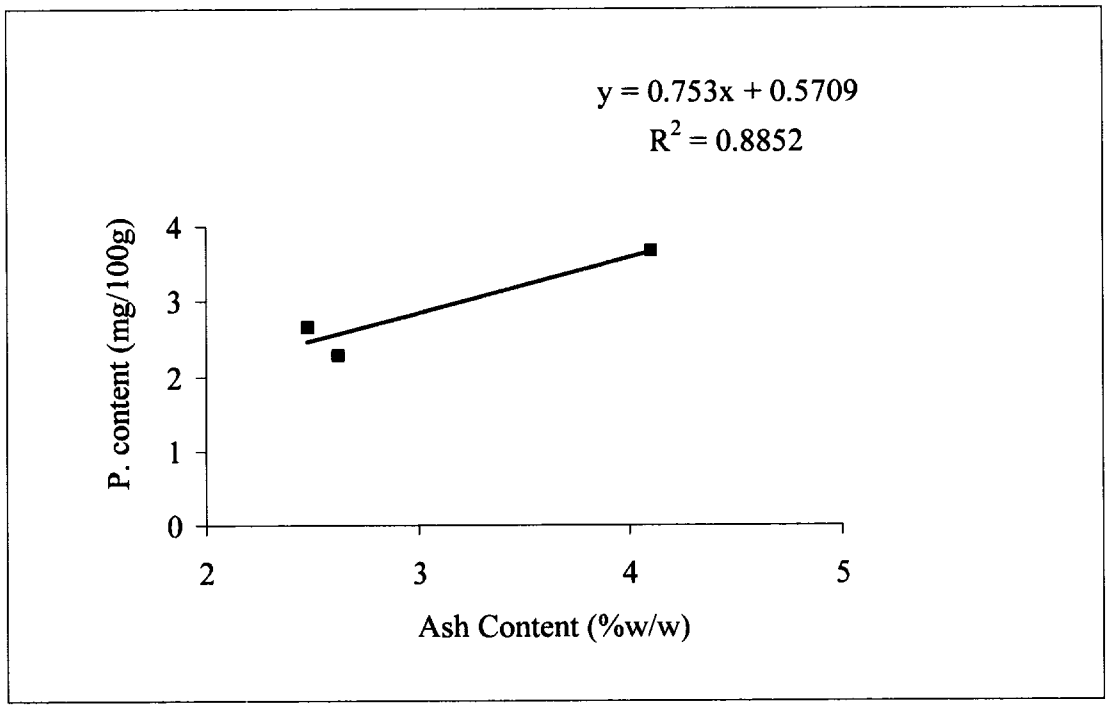

Standard Curve of Amylose
y = 0.0042x + 0.0894
R2 = 0.9916
Absorbance (O.D)
Concentration (µmg Amylose)
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
2
0
100
200
300
400
500

Appendix 3


Standard Curve of Calcium
y = 0.0002x + 0.2119
R2 = 0.9839
Absorbance (O.D)
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
1000
2000
3000
4000
5000
6000
7000
Concentration (mg Calcium)

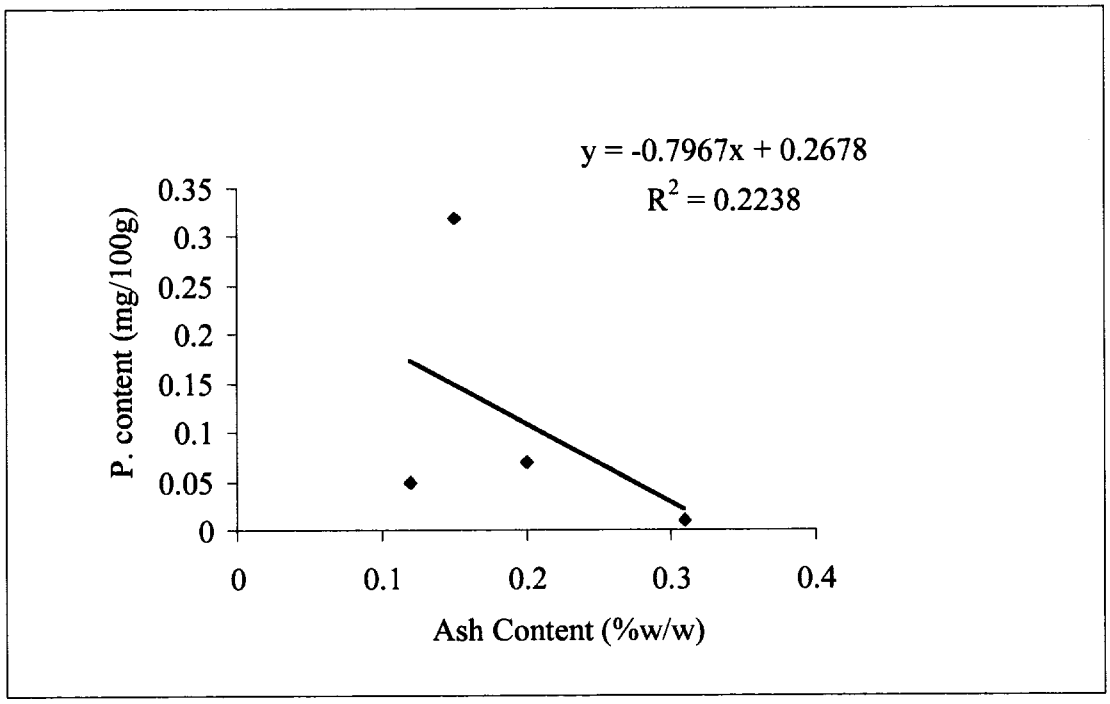
Appendix 4

Correlation between ash and phosphorous content of flour obtained from each tuber.



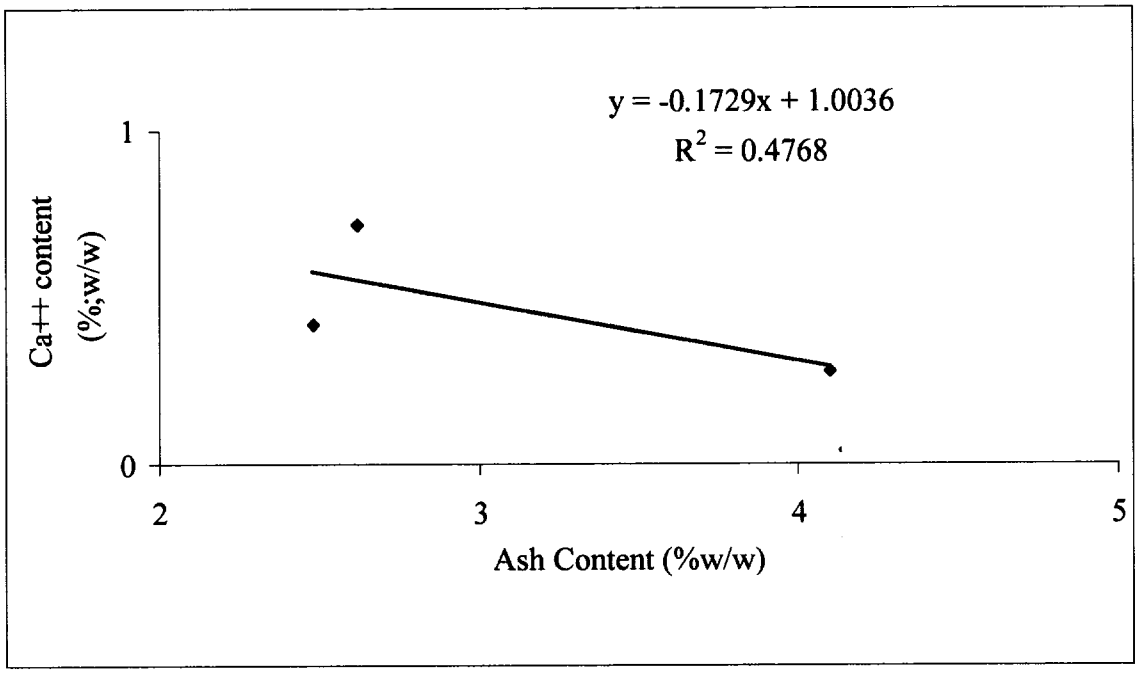
Appendix 5

Correlation between ash and phosphorous content of starches obtained from each tuber.



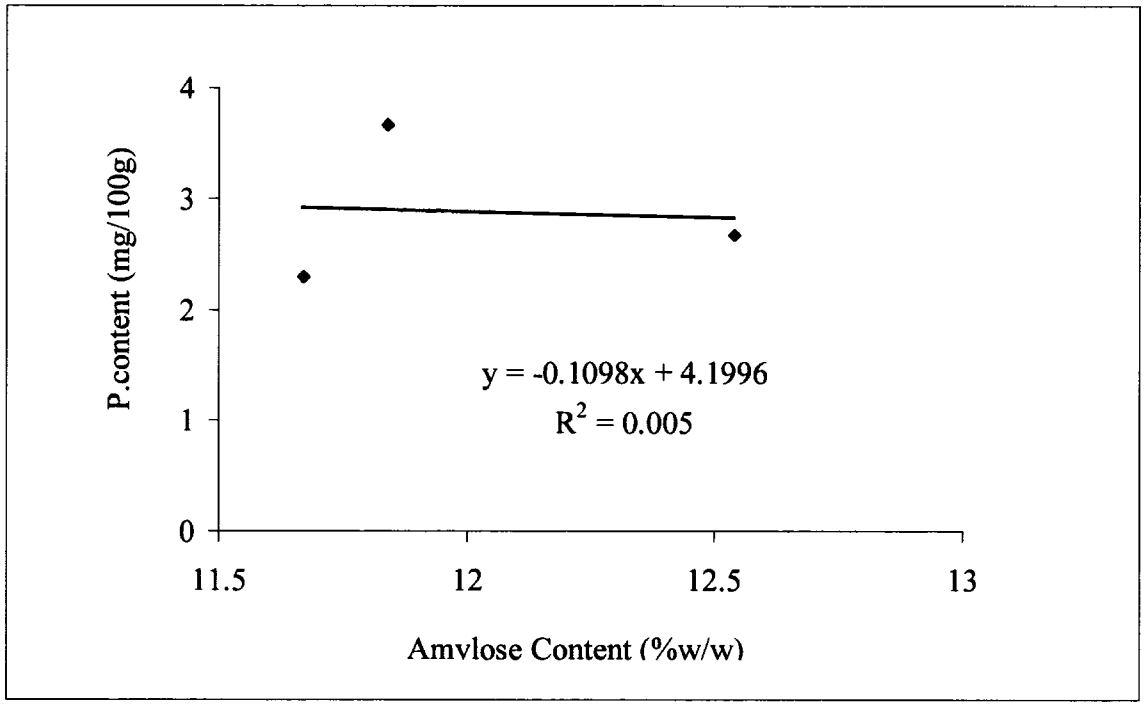
Appendix 6

Correlation between ash and calcium (Ca^{++}) content of flour obtained from each tuber.



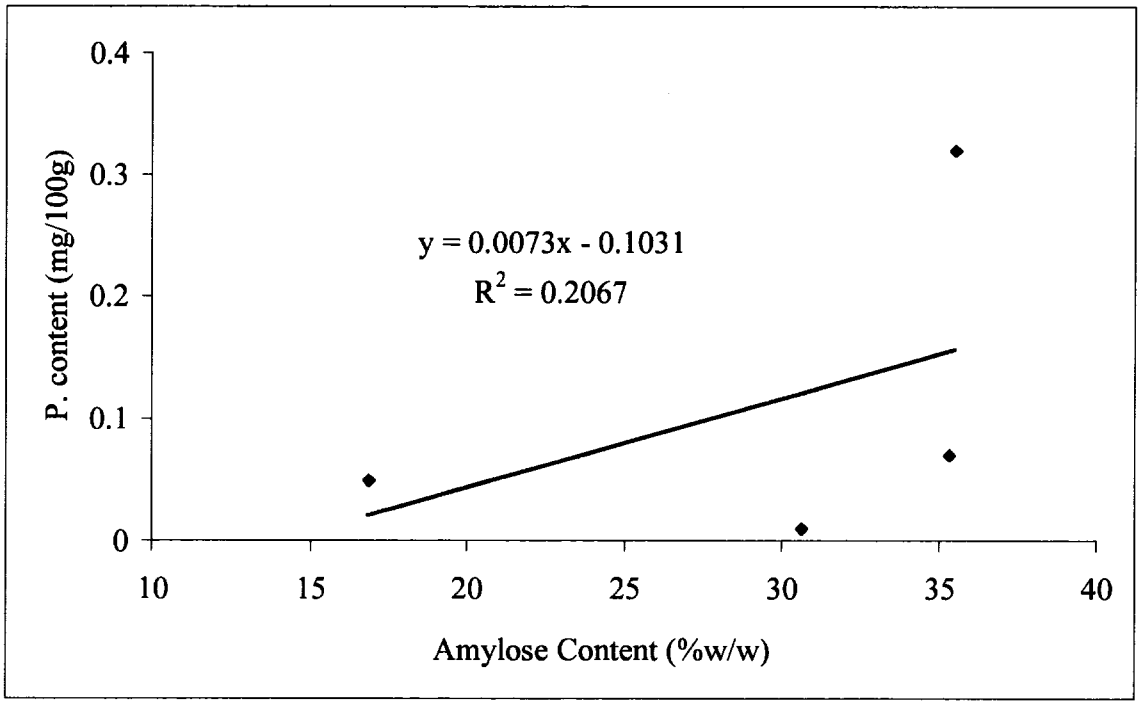
Appendix 7

Correlation between amylose and phosphorous content of flour obtained from each tuber.



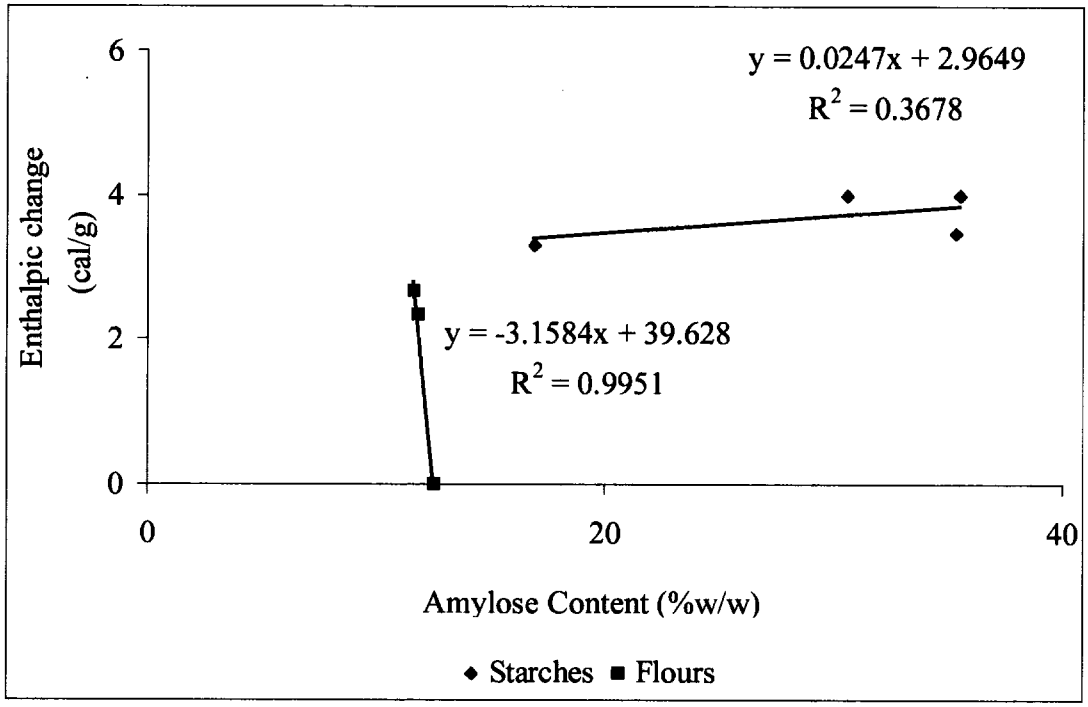
Appendix 8

Correlation between amylose and phosphorous content of starch obtained from each tuber.



Appendix 9

Correlation between enthalpic change (ΔH) and amylose content (measured by colorimetry) of starches and flour obtained from each tuber.



Appendix 10

Correlation between amylose content (measured by colorimetry) and gelatinization profile of starch and flour obtained from each tuber.