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ELEOCHARIS DULCIS				
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In the present study, the textural changes induced in Chinese waterchestnuts by physical and chemical means were of prime concern. After being subjected to chemical and heat treatments, the corms were analyzed to relate the chemical changes with the physical changes in the corms.

Chinese waterchestnuts were peeled, filled in cans with one hundred ml of various concentrations of hydrochloric acid and sodium hydroxide, and heated in the retort for one hour at 240° F (115.6°C). Distilled water was used for the control samples. Physical and chemical examinations followed, and the results of the texture measurements were compared with the chemical composition of the corms. A physical measurement of texture was obtained by using the Maryland Shear-press.

The drained weights, total solids, alcohol insoluble solids, ash, total sugars, cellulose, starch, total pectins including three pectic fractions, phosphorus, calcium and potassium were determined. Measurements of pH, degrees Brix, and color intensity were made on the processed liquid.

The results indicated the following conclusions:

1. Processing in both acid and alkali had a definite softening effect on the Chinese waterchestnut corm.

2. The highest correlation was obtained between texture (as measured by maximum force and total work) and cellulose in both the acid and alkali series.

3. The hydrolysis of cellulose and starch was more complete in the acid series.

4. A linear relationship existed between the pH of the acid series and texture.

5. The samples processed in acid showed more consistent trends than did the alkali processed samples.

6. There was a darkening in color as the concentration of both the acid and alkali increased.

7. The percentages of total solids and AIS showed a definite trend when compared with the texture measurements.

8. The relationship between firmness of the corms and the pectic substances in this study was a complex relationship due to the reactive nature of the pectic compounds.

THE EFFECT OF PROCESSING ON THE CHEMICAL COMPOSITION AND TEXTURE OF CHINESE WATERCHESTNUT, ELEOCHARIS DULCIS

by

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The author wishes to dedicate her thesis in memorial of her loving father who had been guiding her for the past 22 years but died short of seeing her in this stage.

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THE EFFECT OF PROCESSING ON THE CHEMICAL COMPOSITION AND TEXTURE OF CHINESE WATERCHESTNUT, ELEOCHARIS DULCIS

INTRODUCTION

The Chinese waterchestnut, <u>Eleocharis dulcis</u> (Burm. f.) Trin. (Synonym: <u>E. tuberosa</u>, Shult), is a perennial sedge. The plant is grass-like in general habit. The corms, or so-called tubers, are dark-colored and small, about two to four centimeters in diameter. In the southern part of China where the temperature is warm, this species is an important crop. Matai--the common Cantonese name for the Chinese waterchestnut--originated in the provinces of Kwangsi and Kwangtung. A transliteration into English of the Chinese characters for "Matai" means "horses' hoof." This name refers to the shape of the corm. The English common name, waterchestnut, may originate from the brown color of the skin, the flavor or the crisp texture of the edible corm, characteristics which resemble the nut of the genus, Castanae.

The tubers are considered as a delicacy in Chinese cookery and are boiled and eaten as a vegetable. This commodity has been imported into the United States from China since 1945. In 1947, over 2.6 million pounds were imported to the United States (11). However, since 1950, the importation from China has practically ceased. At the present time, the export markets are mostly Hong Kong, Formosa and Mexico, with very few from Cuba, Italy and Japan (28). The main ports of entry in the United States are San Francisco, New York and Los Angeles. Due to the shortage of this commodity, interest has grown in the possibility of introducing matai as a specialty crop in the United States.

In 1934, the Chinese waterchestnut was introduced into this country under P.I. No. 106274 by the Plant Introduction Section of the United States Department of Agriculture. The best cultural region has been the southern areas of the Atlantic Coast plain (11) where there is a long frost-free season. The states of Florida, Georgia and South Carolina produced approximately 50,000 pounds in 1954 (11).

This thesis is a study of the various chemical constituents of the Chinese waterchestnut corms and the relationship of these constituents to texture.

The purposes of this study were as follows:

1. To find a chemical or physical way to soften the crisp texture of the Chinese waterchestnut.

2. To correlate the textural changes with chemical changes in the corms.

Dr. E. D. Merrill, an authority on the plants of Southeast

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REVIEW OF LITERATURE

The edible portion of many vegetables such as lettuce and spinach is the leaf and stalk; for peas and lima beans it is the seed; for carrots and parsnips, the root. In the Chinese waterchestnut, an aquatic plant of economic importance in southern China, the edible portion is its dark-colored, small, round, underground tuber, or so-called corm.

The English literature on the Chinese waterchestnut is quite limited. Practically, there are few detailed analyses of the chemical composition of matai. However, comprehensive brief reports on cultural studies and related tests are available.

1. Botanical Description

The species under consideration belongs to the water-loving family of grass-like plants, Cyperaceae. The generic name, <u>Eleocharis</u>, comes from the Greek meaning "marsh delight." The species name, <u>dulcis</u>, means "sweet." Thus, we have the fitting name, "the sweet delight of the marsh," for the matai. There are more than a hundred species in the genus, <u>Eleocharis</u>, with worldwide distribution.

Dr. E. D. Merrill, an authority on the plants of Southeast Asia, wrote in 1949: "Eleocharis dulcis is of very wide, more or less natural, distribution in the Old World tropics. Man may have aided and abetted its distribution. Southeast China and Formosa to India, Madagascar, and tropical Africa, throughout Malaysia to the Philippines, New Guinea, New Caledonia, Mariannas Islands and Fiji. In most of this vast range it is never cultivated. You can safely say that the Chinese developed its cultivation. In fact, China is the only place where I definitely know it is cultivated, but I would guess also in Indo China and perhaps Japan" (8).

The unique characteristic of the plant is that, due to the lack of leaves, the photosynthetic function is carried on in the numerous, upright, tubular stems which run from three to five feet tall.

The reproduction of the plant is through the rhizomes and corms. Two kinds of stolons are observed; namely, a stolon-producing tuber, and a stolon-producing plant or culm. The first type of stolon which appears from six to eight weeks after planting has a sharp, free end and serves to multiply the number of plants during the growing season through the production of secondary plants from the mother culm. Another rhizome type appears later in the season. This type, produced both by mother and daughter plants, has a swelling, free end and is the important producer of corms which develop at the tips. It was found that several culms, numbering from three to seven or more, were connected by stolons (26).

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The corms are usually produced abundantly. In size and shape, the corms resemble the garden gladiolus, being brown-skinned, subglobose and vertically compressed. The firm white flesh inside is characteristically crisp and tastes somewhat like a Delicious apple.

The chemical composition of the edible portion of the corm is approximately 1.4 percent protein, 0.2 percent fat, 19 percent total carbohydrate, 0.8 percent fiber, 8 percent starch, 1.1 percent ash, and 4 mg, 65 mg, 0.6 mg per 100 grams of fresh corm of calcium, phosphorus and iron, respectively (20, p. 24-25).

In Table 1 are analyses of waterchestnut by Blasdale, Sherman and Wang, Adolph, Hemmi, and by Chung and Ripperton, as summarized by Winton and Winton (29, p. 123-126).

The variations in the analyses are probably due to differences in variety, stage of maturity and the length of storage (3). Other factors such as light, air, water, the pH of the soil, the quantity and quality of the nutrients and the temperature of the growing region are responsible for differences in the chemical composition of the waterchestnuts (23, p. 36).

From Table 1, it is apparent that there may be an inverse relationship between the sugar and starch contents. Starch converts into sugar as the corm ripens. The values of Hemmi's analyses

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	PERCENT									
	Protein, Sugars,									
	Water	Protein	Pure	Fat	N-f Ext.	Reducing	Sucrose	Starch	Fiber	Ash
Blasdale										
I	77.29	1.53	1.16	0.15	18.90	1.94	6.35	7.34	0.94	1.19
II	77.89	1.31	1.00	0.27	18.13	2.60	6.02	8.09	1.22	1.18
Hemmi	68.52	2. 25	1.69	0.19	26. 46 ¹	0. 24	1.06 ²	18.75	1.00	1. 58
S and W	71.19	1.63		0.13	24.63				1.24	1.18
C and R^3	79.54	5.89°		0.04	13.07			. 	0.55	0.91
$Adolph^3$	79.20	1.84		0,18	17.12				0.70	0.96

Table 1. Composition of the Chinese waterchestnuts.

¹Dextrin 0.60 percent, pentosans 0.79 percent.

²Non-reducing sugar.

³Peeled.

seems to show maturity differences of this type.

It is of interest to compare the chemical conposition of the waterchestnut to the parsnip, <u>Pastinaca sativa</u>, a vegetable in which the edible portion is the root (20, p. 24-26). When fresh, the parsnip is crisp, like a fresh raddish. After cooking, the crispness is no longer retained by the parsnip.

As seen from Table 2, the chemical compositions of the two plants are similar except for the amounts of calcium and phosphorus. The characteristically low calcium and high phosphorus contents in the waterchestnut may be factors in retaining crispness after cooking.

2. Cultivation and Fertilization

Complete details on the cultural requirements of the matai have not been determined. Limited tests have been conducted at the plant experiment stations. D. A. Bisset, Chief Scientific Aid at Savannah, Georgia (11), has given the crop considerable cultural and marketing attention. At Orlando, Florida, Mr. James Banks has carried on extensive field tests for commercial production. At Laurel, Florida, small test plantings have been carried out since 1942 (8).

In the Philippines, the Los Banos Economic Garden, Bureau of Plant Industry, received two lots of Chinese waterchestnut tubers in 1938 from Hong Kong. A brief report covering the results of the

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		Food			Carbohydrate				Phos-			
	Water <u>%</u>	Energy cal	Protein gm				Ash gm	Calcium mg	phorus mg	Iron mg	Acid mg	
Matai	78.3	78	1.4	0.2	19.0	0.8	1.1	4	6.5	0.6	4	
Parsnip	78.6	78	1.5	0.5	18.2	2. 2	1.2	57	0.7	0	18	

Table 2. Comparison of the composition of Chinese waterchestnut and parsnip. l

l 100 grams, edible portion. preliminary study on the culture of the tubers was presented by N. G. Teodoro and F. Q. Abaya of the Economic Garden (26). At the University of Maryland, Dr. B. A. Twigg has conducted studies to determine the feasibility of growing matai on the Eastern shore of Maryland and Virginia and tried to ascertain some of the cultural practices necessary for commercial production (27).

Chinese waterchestnuts may be grown as an annual crop, under controlled irrigation, provided that the region has approximately 220 days of warm, frost-free, growing season.

It is reported that the best planting time in the south is in March or April. According to D. A. Bisset and W. H. Hodge (11), planting should be made in level, fertile soil with the plot surrounded by low banks or dams, making possible the retention of four to six inches of water over the entire field. The pH of the soil should run between 6.9 and 7.3 because Chinese waterchestnuts do not grow well in acid soils. Soils containing a high amount of organic matter and neutral in pH such as muck soil and rich clay are suitable for this purpose.

Corms used for seed purposes should be selected for soundness, freedom from injury, intact terminal bud, and good form. Though small seed corms can produce a crop, superior growth and production may be expected if large corms are planted. Two methods of planting may be used depending on the latitude. In northern plantings, where the growing season is shorter, corms should be started early in well-prepared seedbeds or nursery plots maintained at approximately 60° F. The temperature should be controlled. The corms are planted about two inches deep, spaced four to six inches apart in each row, six to eight inches between rows. After planting, the bed is flooded to settle the soil, after which the water is withdrawn. The germination period is about ten days. Transplanting should be done to the permanent field after the plants attain a height of eight to twelve inches. This is a careful hand operation in which the entire young plant and its root system is moved to its final field position. Plants should be set 30 inches apart in a triangular arrangement in a flood plot and covered with four to five inches of water.

In southern plantings, the procedure described above may be used or the corms may be planted directly in permanent field plots. In this case, corms should be planted four to five inches deep and 30 inches apart in the rows. In the plantings, furrows may be opened with an opening plow or colter. The corm is placed at the bottom of the furrow and covered with loose soil with a covering plow or hiller.

After planting, the plot or field is allowed to settle and drain

naturally after flooding. When the stalks are eight to twelve inches high, the planting should again be flooded with four to five inches of water. From this stage on, the cultural procedure is similar to the northern plantings.

It is reported that under suitable condition of growth, two or three crops were harvested from the original planting due to secondary plants developing from the mother culm.

Chinese waterchestnuts require a large amount of plant food. Application of one ton of fertilizer per acre will promote maximum yields. Organic manures and high-grade complete fertilizers are suitable for this purpose. Approximately one-third or one-half of the fertilizer should be applied to the soil during the preparation for planting; another one-third or one-fourth of the total application should be made about eight to ten weeks after planting at about the time that the first of the secondary plants appear and the balance of the fertilizer should be applied prior to the initial development of the young corms. Fertilizer can be broadcast and stirred into the soil with tillage.

3. Harvesting

As mentioned by Hodge and Bisset (11), the harvesting of the crop may be started 20 or 30 days after the plants have attained maturity, or after the first killing frost. The mature corms are chestnut-brown and two to four centimeters in diameter. If the corms are dug as soon as the plants attain maturity, there will be a number of immature corms which are not as sweet and palatable as mature corms. Immature corms are usually lighter in color.

Harvesting Chinese waterchestnut is a laborious job. In small plantings, a hand tool such as a spading fork is suitable for this purpose. The soil is turned carefully and the corms are picked out by hand to avoid bruising. Another way of harvesting small plots is to use a three-quarter inch mesh screen, worked over with rubber pads or paddles. The corms that remain on the screen as the soil is worked through are picked up and dropped into a bucket filled with water to prevent bruising.

However, in larger plantings, corms may be harvested by means of a small plow to turn a furrow to a depth of about six inches. Each furrow should be raked with a potato rake, the tines of which are covered with soft rubber. The corms are picked up by hand and placed in containers. The removal of excess soil and debris is done by a suitable washing device. The corms are ready to be packed for shipment or storage.

4. Diseases and Pests

It is reported that the only difficulties in the culture of this specialty crop seem to be associated with acid soils. The plants grown

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under such conditions undergo gradual deterioration about the time of flowering and may be easily attacked by disease. A stem fungus, <u>Cylindrosporium</u>, has been reported to attack the plants growing on a soil of pH 5.5.

In Florida, Tarjan (25) reported that awl nematodes attack the stem of the plants but that a control procedure was unknown.

Rodents, especially rats and muskrats relish the corms of matai. The stems should be cut-off or burned as soon as the waterchestnut attains maturity to destroy the shelter for the rodents which may cause serious damage to the crop.

5. Yield and Marketing

According to Twigg (27), the yield of matai was larger when the soil was flooded with three to four inches of water than when the soil was thoroughly soaked once a day or when the water level was at the soil surface. Total yields of 10 to 15 tons per acre are possible on the Delmarva Peninsula, if an adequate water supply is available.

In south China, yields are reported ranging from 2700 to 5000 pounds of good-sized corms from 1/6-acre plots. Under good growing conditions, one parent plant representing the mother and all daughter plants has produced 20.75 pounds of corms in a season (11). Such a plant occupies 25 square feet at harvest time.

In general, corms 30 mm or more in diameter are

acceptable to the fresh trade. The market price ranges from 40 to 80 cents per pound. Smaller corms can be used for seeding. The damaged or bruised ones may be salvaged and canned.

6. Storage and Utilization

Before storage, the broken or damaged corms are removed and the sound corms washed in clean water to remove soil and foreign matter. The corms are then air-dried to remove surface moisture. After drying, the corms should be packed carefully in moisture-proof containers. Corms packed and stored between 30° and 40° F have been kept successfully for six months.

In regions where the winters are mild, matai may be left in the ground and harvested when needed. If the soil temperature rises above 56° F, spouting will occur.

Chinese waterchestnuts cannot be kept successfully in open storage at room temperature. The corms shrivel and become worthless in a few days.

For home use, the corms can be washed, dried, and put in the refrigerator in a plastic bag or in a mason jar, with lids on, but not sealed.

The corms of the Chinese waterchestnut have a crisp, white flesh which is both sweet and starchy like an apple. It can be eaten raw, but the crispness of its texture remains after cooking. The vegetable blends with almost any food. It can be used satisfactorily in a number of Chinese and American dishes such as soup, omelets, mixed vegetables, meat and vegetable stew and fried noodles. Upon processing, the corms retain their crisp texture, and are, therefore, a unique type of plant material.

MATERIALS AND METHODS

Source of Chinese Waterchestnuts

The Chinese waterchestnuts used in these studies were obtained from Wo Kee and Company and the Fung Chong Company, San Francisco, California. All of the corms, comprising three different lots, were imported from Hong Kong. The corm size varied between lots, with Lot A of large size, Lot B of medium size and Lot C of relatively small size. This size variation may be due to different stages of maturity and/or variety.

The corms were sorted and peeled by hand. The peeled waterchestnuts were washed several times under running tap water to remove the skin and dirt, drained on eight-mesh stainless steel trays to remove excess water, and filled into cans by hand.

Preparation and Processing

The preliminary experiments were studies of the effects of processing techniques and additives upon the texture of the Chinese waterchestnuts. The corms were separated into groups of five peeled corms and the groups submitted to eight different treatments in duplicate. The change in texture was measured by the Kramer shear-press on the drained sample. The treatments were: 1. Freeze-dried and reconstituted in tap water for 24 hours.

2. Incorporation of 10 ml, equivalent to 0.1 gram of either pectase or cellulase, into C-enamel, 307 x 200 cans each containing five peeled corms in 100 ml of H_2O . The corms had been subjected to heat treatment for one hour at 240° F (115.6° C) before the enzyme inoculation. The cans were incubated at 100° F (37.8° C) for 24 hours before determining the texture on duplicate samples.

3. The corms were frozen at 0° F (-17.8° C) and -18° F (-27.8° C) for 22 hours and thawed for 1.5 hours at room temperature.

4. Radiation treatments using Co^{60} for three groups of samples in 240 ml of H_2O for 16 hours, equivalent to a nine megarep dosage.

5. Heated at 240° F (115.6° C) in 307 x 200 cans each containing five roots in 100 ml of H_2O for 10, 20, 40 and 80 minutes.

6. One gram of Ca versene, (Calcium disodium chelate of ethylenediamine tetra acetic acid), was added in Procedure 5 and duplicate cans heated for one hour at 240° F (115.6° C).

7. One gram of EDTA, ethylenediamine tetra acetic acid tetrasodium salt, was added instead of versene in Procedure 6.

8. One gram of calgon, (sodium hexametaphosphate), was added instead of EDTA in Procedure 7.

The results show that there were some textural changes in the corms from the above treatments (Table 3). The obvious change shown in the shear-press results was the loss of the characteristic side arm peak which was found in the fresh sample work diagrams (Figure 1). The corms retained their firmness and crispness after the above treatments.

A second series of preliminary experiments showed that the waterchestnuts could be softened by processing the corms in acid and alkali solutions at 240° F (115.6° C). Based on this finding a replicated experiment was undertaken to study the chemical changes accompanying the loss of texture.

The Chinese waterchestnuts used in each replication were random corms taken from a common lot.

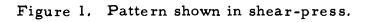
Approximately 55 grams (five corms) of peeled, cleaned, waterchestnuts were weighed and placed into 307 x 200 C-enamel cans. One hundred ml of acid (HC1) or base (NaOH) of 0.500, 0.250, 0.125, 0.063 and 0.031 N were used to fill the cans. Three replications were made of each treatment. Distilled water instead of acid and alkali served as a control. The preparation and canning procedures were all carried out at room temperature. After filling the cans, the containers were double seamed at 29 inches vacuum.

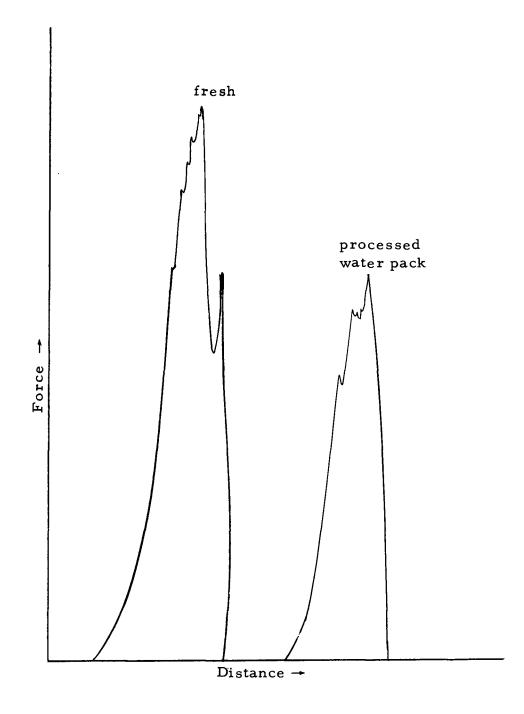
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	Texture Measurements					
	Maximum Force	Total Work				
Type of Treatment	inches ¹	inch-pounds				
Irradiated						
9 megarep	2. 5	2 63				
Freeze-dried	3.8	36 2				
Frozen and Thawed						
-18° F	4.1	494				
0° F	3.9	430				
Enzymes						
Cellulase	3.1	309				
Pectase	3.6	362				
Heat Treatment						
10 minutes	3.3	344				
20 minutes	3.1	336				
40 minutes	3.3	357				
80 minutes	3.3	33 2				
Calgon (1 g in 100 ml)	3.8	379				
Versene (1 g in 100 ml)	3.9	394				
EDTA (l g in 100 ml)	3.4	346				
Fresh Sample	4. 2	474				

Table 3. Texture changes in the Chinese waterchestnuts aftervarious processing and additive treatments.

¹Converted to pounds force by multiplying by 80.





The sealed cans were heated in a retort for one hour at 240° F (115.6° C) and cooled in running tap water for 30 minutes.

Three lots of corms were used. Each treatment was replicated three times for a total of 99 cans. The canned materials were analyzed immediately after processing for vacuum, drained weight, pH, degrees Brix, and Kramer Shear-Press texture measurements. After the texture study, the three replications of each treatment were mixed and blended in a Waring Blender for six minutes. The blended samples were frozen in glass sample bottles at 0° F (-17.8° C) and held until analysis.

Vacuum measurements were taken on the cans with a U. S. Gauge, puncture gauge at room temperature and the results reported as inches of mercury.

The pH was measured on a Beckman Zeromatic pH Meter at room temperature.

The degrees Brix was measured with a Bausch & Lomb refractometer. The sample was brought to room temperature before the measurement.

The procedure used for the determination of the drained weight was the U.S.D.A. -A.M.S. method (17, p. 16).

Resistance to shear was measured by the Maryland shearpress fitted with an X-Y recorder (Figure 2). The entire, drained



Figure 2. Kramer Shear-Press (Food Science and Technology Department, Oregon State University).

sample from each can was placed in the cup of the standard cell on the shear-press. A thousand pound ring and a transducer value of 0.01 was found to satisfactory for texture measurement of Chinese waterchestnuts. After every sample, the cup was washed with water.

The work diagrams obtained in each sample were measured for the maximum force and the total work. The area under the curve was measured with a planimeter and then converted to inchpounds of work.

The samples obtained after passing through the Shear-Press were collected, blended, sealed in bottles and held at 0° F (-17.8° C) for chemical analysis.

Analytical Procedures

All of the analytical determinations were made on composite samples of the three replications in each lot. Known quantities of distilled water were added to the composite sample in the Waring Blender. The samples were blended for six minutes.

Total Solids

Fifteen gram samples of the slurry were weighed into tared aluminum cups. The cups were then dried under 29 in of vacuum at 60° C for 16 hours, cooled in a desiccator, weighed, and calculated as the percentage total solids on the fresh weight basis.

Total Ash

The Association of Official Agricultural Chemists (A. O. A. C.) method (12, p. 262) was used for this determination.

Ten-gram samples of slurry were weighed to the nearest 0.01 gram into Vycor Crucibles which had been pre-heated at 600° C for several hours and cooled in a desiccator. The samples were dried on a steam bath until apparently dry, and then ashed at 600° C for eight hours. The crucibles were cooled in a desiccator, weighed and calculated as percentage ash on the fresh weight basis.

Alcohol Insoluble Solids (AIS)

Ten-gram samples of slurry were weighed to the nearest 0.01 gram in 50 ml beakers and transferred into 40 ml round-bottom centrifuge tubes with 35 ml of 95 percent ethanol. The tubes were heated in a water bath for ten minutes at 80° C and centrifuged for 20 minutes at 2800 rpm in an International centrifuge, Model UV. The supernatant was decanted. This procedure was repeated twice with 70 percent alcohol. The sediment was stirred each time before heating. The final residue was transferred into weighed aluminum dishes, which were dried under a vacuum of 29 inches at 60° C for 16 hours. The dishes were cooled in a desiccator and weighed. Results were reported on a fresh weight basis.

Pectin Fractions

The colorimetric carbazole method of Dietz and Rouse (5) was used for the analysis of the three pectic fractions. The three fractions of pectins were isolated by extraction procedures. The three fractions were (1) the water-soluble pectins, (2) the calgonsoluble pectins and (3) the alkali-soluble pectins. The results were calculated on a fresh weight basis.

1. Water-soluble pectins (WSP).

Ten ml of distilled water were added to each AIS residue in the aluminum dishes and the dishes heated on a water bath for one hour. The residue was quantitatively transferred to a 50 ml centrifuge tube. Thirty ml of distilled water were added and about one gram of Hyflo-Supercel was added to each tube to aid in flocculating the residue. The mixture was stirred, allowed to stand for one hour, centrifuged for 30 minutes and decanted into a 100 ml volumetric flask. A second extraction was made and the extracts were combined in the 100 ml volumetric flask. Five ml of 1 N NaOH were added to each flask and distilled water was added to volume.

2. 0.4 percent Calgon-soluble pectins (CSP).

To the residue in the tubes, 35 ml of 0.4 percent sodium hexametaphosphate (calgon) solution were added. The tubes were allowed to stand for ten minutes, centrifuged at 2800 rpm for 20 minutes and the supernatant was decanted into 100 ml volumetric flasks. A second extraction was made and the extracts were combined, 5 ml of 1 N NaOH were added to each flask and the flasks made to volume with distilled water before analyzing for the CS pectins.

3. 0.05 N NaOH-soluble pectins (ASP).

Thirty-five ml of 0.05 N NaOH were added to the residue. The tubes were allowed to stand with occasional stirring for 15 minutes before centrifuging. The extraction was repeated, the extracted materials combined and made up to a volume of 100 ml.

Determination of the Three Pectic Fractions

The standardization of the instrument and the color development of the samples have been described by Dietz and Rouse (5) except for minor modifications.

A Bausch and Lomb, Spectronic-20 spectrophotometer was used for reading the pectin content colorimetrically. The percent transmittance was measured at the 525 m μ wavelength.

One ml aliquots of the pectin solutions were pipetted into

6 x 1 inch Pyrex test tubes, and 0.5 ml of 0.1 percent alcoholic carbazole were added to the samples. Six ml of concentrated sulfuric acid were added to each tube with constant agitation. The tubes were stoppered and the color allowed to develop for ten minutes.

Exactly 15 minutes after adding the acid, the percentage transmittance was measured.

A standard curve was run with vacuum-dried, Eastman, Practical grade, D-galacturonic acid by the same procedure.

Five tenths ml of purified ethyl alcohol and one ml of distilled water were used instead of the carbazole and pectin solution in the blank tube.

The percent transmittance readings were transferred to galacturonic acid by means of a standard curve plotted on semi-log paper.

Sugar Determinations

The phenol method of M. Dubois <u>et al</u>. (6) was employed for this analysis. After the preliminary tests, the following procedure was adopted.

Five gram samples of slurry were weighed into 50 ml beakers and quantitatively transferred to 50 ml centrifuge tubes with 40 ml of distilled water. After centrifuging at 2800 rpm for 30 minutes, the supernatant was decanted into a 100 ml volumetric flask. The extraction was repeated, the supernatants mixed and made up to 100 ml with distilled water. A one ml aliquot was taken and diluted to 100 ml.

To determine the sugar, two ml of the sugar solution were pipetted into a colorimeter tube and one ml of five percent phenol was added to each tube. Five ml of concentrated sulfuric acid were added rapidly to obtain good mixing. The tubes were shaken and placed in a boiling water bath for five minutes and cooled in running tap water for five minutes. The resulting color was stable for several hours.

The tubes were read for percentage transmittance on a Spectronic-20 spectrophotometer at a wave length of 490 m μ . Blanks were prepared by substituting distilled water for the sugar solution.

A standard curve was established using dextrose.

Starch Determination

The wet residue from the sugar analysis was transferred to 250 ml Erlenmyer flasks. One hundred ml of distilled water and ten ml of concentrated HCl were added. The flasks were covered with aluminum foil and boiled for three hours in a covered water bath. The samples were filtered through folded filter paper and diluted to 200 ml. Two ml aliquots were taken and diluted to 100 ml in a volumetric flask. The same procedure described for the sugar determination was followed, using a conversion factor of 0.9 for starch.

Cellulose Determination

The method by R. A. Gallop (7, p. 58) was modified for this determination. Thirty ml of acetone were added to the residue in each tube remaining from the starch determination, heated in a water bath at 55° C for 30 minutes with occasional stirring, and centrifuged. The extraction of lipid was repeated and the residue allowed to dry over night at room temperature. In order to disperse the solids, two to three ml of water were added to each tube and the tubes heated in a warm water bath for 30 minutes with stirring. After cooling, 25 ml of 60 percent H_2SO_4 were added. The mixture was allowed to stand for three hours with occasional stirring. The solutions were poured into 100 ml of water in 250 ml volumetric flasks, and the tubes washed out into the flasks with distilled water. The solutions were cooled, made to volume, and filtered through No. 31 Whatman papers. One ml aliquots of filtrate were made up to 100 ml with distilled water.

The total sugars were determined by a modification of the

phenol method of Dubois et al. (6).

The percentage transmittance readings were then transferred to the percentage of sugar on a 100 gram fresh weight basis with the aid of the standard curve made for the sugar determination.

Flame Analysis of Calcium

A 15 gram of sample was ashed in a platinum dish in the muffle furnace at 600° C for 14 hours. The ash was dissolved in 0.1 N HCl, filtered through Whatman No. 40 filter paper and brought to 25 ml volume in 0.1 HCl.

An attempt to remove phosphate from the original extract by a procedure similar to that of Denson (4) gave erratic results; therefore, two drops of concentrated H_3PO_4 were added to the samples before the flame analysis.

The calcium was determined at 554 m μ on a Beckman, Model DU, 2400 Spectophotometer fitted with a flame attachment. A hydrogen-oxygen flame was used, and the results were calculated from a previously determined calcium calibration curve ranging from 0 to 150 μ g per ml.

Flame Analysis of K

One ml aliquots of the acid extract of the ash samples were

taken from the solutions used for the calcium determination, put into 10 ml volumetric flasks and diluted with distilled water to volume.

The potassium content was determined at 768 m μ on the Beckman Spectophotometer with flame attachment.

The percentage of potassium was calculated from a previously determined calibration curve and reported on a fresh weight basis.

Colorimetric Determination of Phosphorus

This method is a modification of the "Rapid colorimetric determination of phosphorus in high explosive compositions following a wet-ashing procedure" (22).

Reagents:

1. Ammonium molybdate: 0.2 M. Dissolve 35.3 grams of ammonium molybdate tetrahydrate in distilled water. Dilute to one liter and filter if necessary.

Vanadate: 0.2 M in 0.4 M perchloric acid. Dissolve
 1.17 grams of ammonium metavanadate in 400 ml of distilled
 water containing 25 ml of 8 M perchloric acid. Dilute to 500 ml.

3. Eight M perchloric acid: dilute 345 ml of 70 percent perchloric acid to 500 ml.

Procedure:

Weigh a 15 gram sample and ash for 14 hours at 600° C. Take up the ash in 0.1 N HCl, filter through Whatman No. 40 filter paper and bring to volume with 1 N HCl. Put a two ml aliquot of this solution into a 50 ml volumetric flask. Add five ml of the vanadate reagent, mix, add ten ml of the molybdate reagent, mix and make up to volume with four M perchloric acid. Allow the sample to stand 20 minutes and read the absorbance vs a fresh reagent blank at 400 mµ.

Calculate the percentage of phosphorus from a previously determined calibration curve.

The Photovolt Tristimulus Colorimeter Determination of the Liquid Portion of the Processed Waterchestnut

Using the Photovolt tristimulus colorimeter, a three-number specification of color is obtained directly. This three-number specification is convertible to the tristimulus values, X, Y and Z, of the Standard ICI system.

The tristimulus values, X, Y and Z, are obtained by comparing directly an unknown light with an optical mixture of three primary lights. The Photovolt colorimeter cannot read directly in terms of X, Y and Z, but these values can be computed from the readings, R, G and B, of the colorimeter by the use of equations (16, p. 108-152).

The method of standardizing the colorimeter is with a standard white plaque (which is itself standardized against MgO).

After introducing the Amber filter into the light source, the instrument was adjusted to read 75.5, with the standard white plaque.

A series of readings on ten ml samples in 50 ml beakers with the Amber filter were obtained on the processing cover liquids.

The readings for the Blue and Green filters were obtained by standardizing the instrument to 78.0 and 76.5, respectively.

The tristimulus values X, Y, Z, and the chromaticity coordinates x and y were calculated (16, p. 108-152).

The Munsell values (Yv) were determined from the appropriate tables (16, p. 352-353).

RESULTS AND DISCUSSIONS

The procedures for the preliminary tests were followed, as outlined on page 16. The results are tabulated in Table 3. Among the 13 individual treatments which were duplicated in each case, the maximum force measurements ranged from 2.5 to 3.9 inches (one inch = 80 pounds force) and the total work varied from 263 to 494 inch-pounds. The measurements for the unprocessed corms were 4.2 inches maximum force and 474 inch-pounds total work as seen in Table 3.

Radiation had the most effect in softening the corms as shown by the texture measurements; however, the corms retained their crispness based on human judgments. Samples frozen at -18° F (-27.8° C) and 0° F (-17.8° C) for 22 hours and thawed at room temperature for 1.5 hours, the freeze-dried and reconstituted samples, the versene-treated samples and the calgon-treated samples showed little softening.

For the analysis of the Chinese waterchestnuts processed in various concentrations of hydrochloric acid and sodium hydroxide in tin containers, the methods described in the previous section were used. The average vacuum in the cans was 25.5 inches with a range of one inch. With regards to the texture, pH, degrees Brix, drained

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weight, and the percentages on a fresh weight basis of total solids, alcohol-insoluble solids, ash, total sugar, cellulose, starch, the three pectic fractions, the total pectins, the minerals--calcium, potassium, and phosphorus--and the color of the processed liquid, the following results were obtained.

1. Shear-press measurements.

The data tabulated in Table 4 show that the texture as measured by two methods, namely, total work and maximum force, decreased with increasing concentrations of both acid and alkali.

As seen in Figure 3, there is a linear relationship between the maximum force and total work in both the acid and alkali series. The changes in texture from the water pack to the 0.03 N acid and 0.03 N alkali were small.

Figure 4 shows the relationship between the maximum force and pH values. The acid series showed a linear relationship between the two factors, while the alkali series was more nearly linear with normality.

The work diagrams of all the processed samples were of similar shape. In the non-processed samples, a side-arm peak and several small peaks appear at the top of the work diagrams. The characteristic differences between the processed and non-processed work diagrams for the Chinese waterchestnut are shown in Figure 1.

		Tota inch		pH ³				
		I	Lot		Lot			
Normality ¹	A	B	С	Mean	A	B	С	
Acid Series								
0.031	326	283	352	320	3.10	3.10	3.25	
0.062	137	121	220	159	2.12	2.14	2.49	
0.125	74	87	51	71	1.82	1.87	1.73	
0,250	0	13	0	4	1.25	1.30	1.29	
0.500	0	0	0	0	0.93	1.00	0.90	
Alkali Series								
0.031	333	376	379	362	7.47	7.52	7.90	
0.062	304	326	300	310	8.75	8.84	8.80	
0.125	175	215	184	191	10.85	10.90	11.00	
0.250	115	93	61	90	11.80	11.78	11.90	
0.500	28	38	14	27	12.15	12.10	12.00	
Water Pack	361	326	334	340	5.80	5.80	6.05	
Fresh	523	538	509	523				

Table 4. The total work and pH of the Chinese waterchestnuts.

¹Strength of acid or base in 100 ml packing liquid.

²Samples on 100 g fresh weight basis.

³pH of processed liquid in cans.

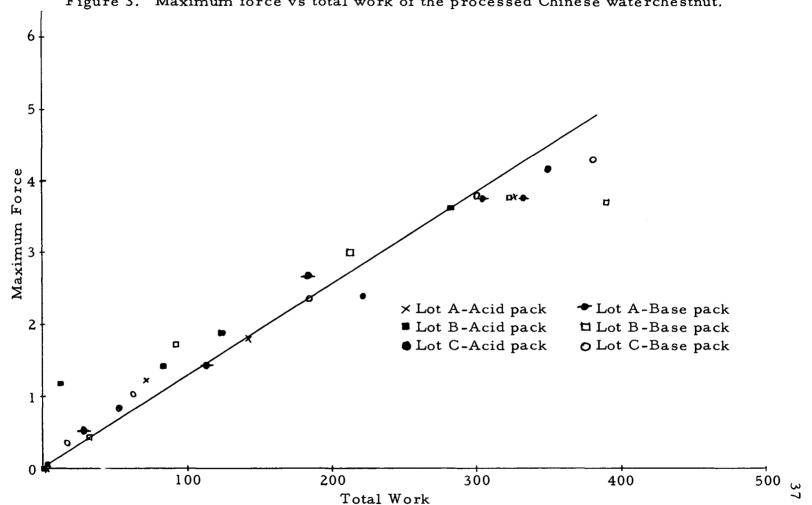


Figure 3. Maximum force vs total work of the processed Chinese waterchestnut.

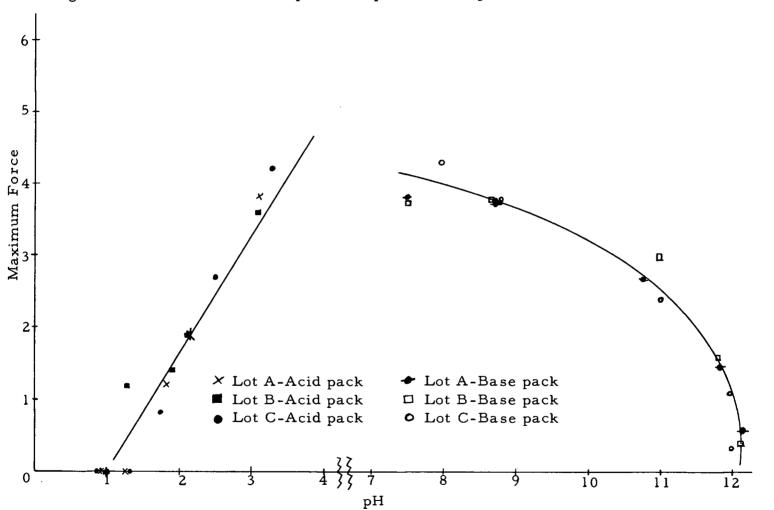


Figure 4. Maximum force vs pH of the processed liquid.

The means of the texture measurements, calculated on the basis of 55 grams of fresh weight, are tabulated in Table 4.

2. Drained weight.

The data in Table 5 show that the drained weights of the Chinese waterchestnuts processed in acid were less than those of the alkali packs. The range of the drained weights in the alkali-packed samples was small in comparison to the acid processed series.

The changes in drained weight from the water pack to the weakest acid and weakest alkali treated samples were small. A linear decrease in drained weight occurred in the acid pack up to the strongest acid (0.500 N) series. The increase in drained weight in the 0.500 N acid pack was caused by the water adsorptive capacity of the softened corms. The liquid drained incompletely from the screens and corms. The loss of total solids indicated that a large amount of water must be adsorbed on the separated cells. The overall texture of the corms was soft, since the corresponding shearpress readings were zero.

The drained weight of a processed product is an important index of commercial quality. Loss of soluble constituents is generally rapid when heat is applied (1, p. 32-36) and a softened texture occurs. Sterling (24) has reviewed the drained weight behavior in canned fruit in detail. The type and size of tissue (10, p. 78), the

	Percent Cellulose					ercen	t Total	Pectins	Percent Drained Weight				
	Lot				Lot				Lot				
Normality	A	В	С	Mean	A	В	С	Mean	A	В	С	Mean	
Acid Series													
0.031	0.88	0.82	0.69	0.80	90	63	62	72	98	101	96	98	
0.062	0.66	0.74	0.52	0.64	90	62	62	71	94	96	94	95	
0.125	0.52	0.54	0.20	0.42	90	62	62	72	96	95	93	94	
0.250	0.42	0.28	0.11	0.27	77	62	63	68	94	93	91	93	
0.500	0.30	0.20	0.10	0.20	69	64	56	62	95	98	93	95	
Alkali Series													
0.031	0.99	0.85	0.68	0.84	95	65	42	68	101	104	98	101	
0.062	0.98	0.70	0.60	0.76	89	70	42	67	101	102	99	100	
0.125	0.70	0.70	0.40	0.60	74	55	37	55	103	102	98	101	
0.250	0.60	0.62	0.40	0.54	73	50	51	58	102	103	98	101	
0.500	0.61	0.65	0.35	0.53	80	60	65	68	102	102	100	101	
Water Pack	0.98	0.89	0.70	0.86	90	62	62	71	102	105	96	101	
Fresh	1.00	0.90	0.75	0.88	87	86	84	85					

Table 5. The percentage of cellulose, total pectin and drained weight in Chinese waterchestnuts.¹

¹On 100 g fresh weight basis.

medium for packing (19), and the rate and duration of heating are important factors in determining the final drained weight. Any influence which will change the properties of the intercellular pectic compounds can greatly influence both texture and drained weight.

3. Effect of processing on pectins.

The susceptibility of the pectic substances to heat and chemical change during processing can be seen from the data in Table 6 and Figures 5 and 6. The change in total pectin was remarkably small, although marked changes occurred in the distribution of the total pectic substances between the three fractions.

As described by Kertesz (18, p. 136-140), the action of alkali and acid on the pectins will cause two primary reactions, namely a degradation of the pectic acid chain by a hydrolysis of the 1, 4 glycosidic bonds and a demethylation of the pectinic acids to give pectates. A third possible reaction with acids and alkalis is an ion exchange mechanism whereby cations are replaced in the insoluble pectates.

The acid reactions include the removal of bivalent cations, the hydrolysis of the cellulose-pectinic acid complex, simple hydrolysis of large water-insoluble pectic molecules, and finally, demethylation (14, p. 120-123). In more concentrated acids and at higher temperatures, noticeable degradation of the pectinic acids occurs. As the

	mg]	Percen	Pectin ²	mg Percent CS Pectin ³				mg Percent AS Pectin ⁴				
	Lot				Lot					Lot		
Normality	A	В	C	Mean	A	В	С	Mean	A	В	С	Mean
Acid Series												
0.031	54	11	15	27	24	29	20	24	12	23	27	21
0.062	54	18	22	31	24	24	19	22	12	20	21	18
0,125	61	20	30	37	22	24	22	23	7	18	11	12
0.250	54	38	4 0	44	20	17	19	19	3	7	4	5
0.500	53	47	39	46	12	9	13	11	4	8	4	5
Alkali Series												
0.031	56	38	22	39	17	16	8	14	22	12	12	15
0.062	59	49	24	44	13	9	6	9	17	12	12	14
0.125	53	37	20	37	10	7	5	7	11	11	12	11
0.250	54	30	25	36	17	9	10	12	8	11	16	12
0.500	40	34	33	36	20	11	18	16	14	15	14	15
Water Pack	56	32	30	39	12	15	12	13	22	15	18	18
Fresh	59	58	59	59	10	15	10	12	18	14	18	17

Table 6. The mg percentage of water-soluble, calgon-soluble and alkali-soluble pectin in Chinese waterchestnuts.¹

¹On 100 gram fresh weight basis.

 3 0. 4 percent calgon soluble pectins.

²Water soluble pectins.

⁴0.05 normal NaOH soluble pectins.

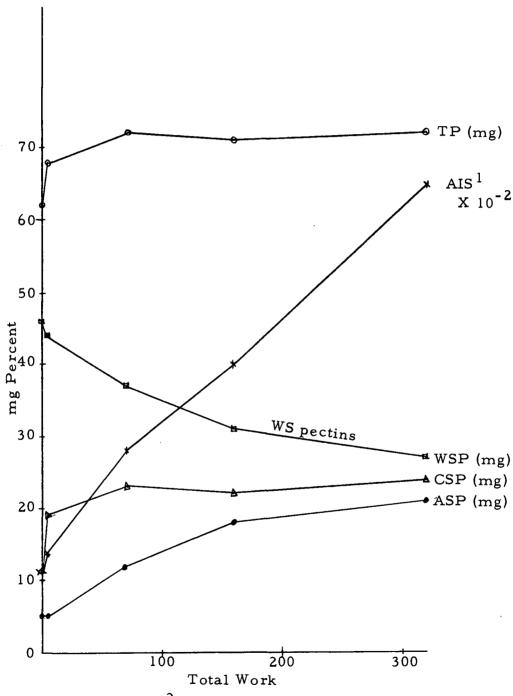
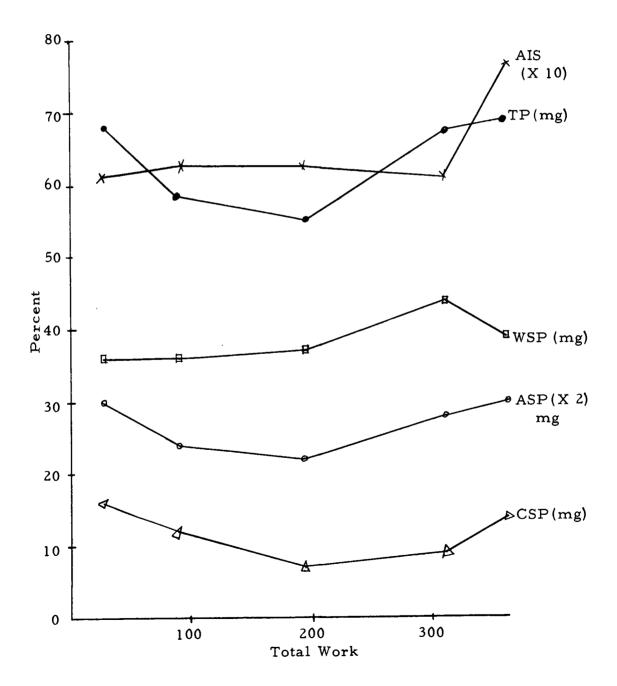


Figure 5. The pectic fractions and AIS content of the processed corms from the acid series.

 1 Values on graph 10^{-2} times analytical values.

Figure 6. Pectic fractions and AIS content of the processed corms from the alkali series.



concentration of the added acid and the reaction temperature are raised, decarboxylation will take place.

An excess of sodium hydroxide will solublize protopectin probably by a degradation of the molecular chain (9, p. 69-78). The pectins are rapidly demethylated in alkali above a pH of 8.5 and subsequently dissolve in the excess alkali.

A. <u>Water-soluble (WS) pectic fraction</u>. The data of the watersoluble pectin is tabulated in Table 6 and the mean value of the three series is plotted in Figures 5 and 6.

The decrease in water-soluble (WS) pectins from the fresh corm to the water pack indicated a diffusion of pectin molecules into the water medium, together with a small conversion of the WS pectins to a water-insoluble form. The diffusion of the WS pectins from the corms was far from the equilibrium value of about 20 mg percent indicating considerable hinderance to free diffusion by the cellular structure to molecules of colloidal size.

The decrease in the WS pectins from the water pack to the 0.030 N acid pack was a result of the conversion of WS pectin to calgon-soluble (CS) pectin and to alkali-soluble (AS) pectin. The CS pectin differs from the AS pectins in that the AS pectins are long chain polymers insoluble in 0.4 percent calgon.

The addition of dilute HCl apparently had a dual role in

changing the pectins. At the processing temperature of 240° F (115.6° C), the water-soluble pectic fractions are demethylated to an extent sufficient to make them sensitive to divalent cations. The second effect of the dilute acid is a solubilization of calcium thus making the calcium ion readily available for reaction with the water-insoluble, demethylated pectic fractions. The net result was a sharp increase in both the calcium content and the water-insoluble pectic fractions of the corms.

The gradual decrease of the water-insoluble pectins (CS pectins+AS pectins) with increasing strength of acid reflects a reversal of the divalent cation addition reactions. The conversion of CS pectin to WS pectin occurred as a result of the removal of calcium to form the more soluble acid pectates. The regular decrease in the AS pectins results from a chain degradation accompanied by some calcium removal. The conversion of AS pectin to WS pectin is due largely to a hydrolysis of the pectin-pectin glycosidic bonds.

The change in WS pectins from the water pack to the 0.030 N alkali series indicated a conversion of the AS pectins to the WS pectins as a result of alkaline degradation of the galacturonic acid chain.

The constant values for the WS pectins in the alkali series indicated little loss of WS pectin by diffusion from the cells, probably as result of adsorption and entrapment of this fraction by the swollen

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starch granules.

The WS pectins play a secondary role in texture. The WS pectins are dynamically intertied with the two water-insoluble fractions, serving as a transition state and material reservoir for changes between the CS fraction and the AS fraction.

The absolute level of the WS pectins at any time is a function of their relationship with the water-insoluble fractions and their rate of diffusion out of the cell. The diffusion rate, in turn, depends upon the molecular size of the WS pectins and the porosity of the cell or tissue (diffusion resistance).

B. <u>0.4 percent calgon-soluble (CS) pectin fraction</u>. As seen in Table 6, there was little change from the fresh to the water pack in this fraction.

The two fold increase in value of the CS pectins from the water pack to the 0.030 N acid series was a result of two reactions; the demethylation of the WS pectins and the solubilization of calcium salts giving calcium ions which react with the low methyl-ester pectins to form calcium pectinates which are calgon soluble.

The sharp decrease of the CS pectins in the higher acid concentrations was a result of the removal of the calcium ion by cation exchange and an acid degradation of the pectic chain at the 1, 4 glycosidic bonds. Calcium ions and short-chain, degraded pectins are the products of calcium pectate from acid hydrolysis. An increase in the WS fraction was a result of this reaction. A decrease in the CS pectins leads directly to cell separation and is definitely a factor in the texture change of the corms.

The CS pectins showed little change from the water pack to the 0.030 N alkali pack.

With increasing alkali concentrations (0.125 N), the CS fraction decreased to one-half its former value as a result of hydrolysis of the long-chain calcium pectates to short-chain pectates. The sodium pectate formed by this reaction is exterior to the primary cell wall and therefore dissolves in the processing liquid without a resulting increase in the WS fraction.

At the high alkali concentrations the trend towards decreasing amounts of CS pectins reversed and the CS fraction increased to its former level in 0.030 N alkali. This phenomenon is a direct result of the formation of short-chain, insoluble, calcium pectinates from the degraded pectic chains. Supporting evidence is seen in the concurrent rise in calcium levels from the 0.125 N to 0.500 N alkali packs. The source of this calcium is the calcium phosphate reservoir which is solublized at the higher alkali concentrations as is shown by the rapid decrease in phosphorus in the corms.

The texture softening resulting from the changes in the CS

fraction are marked. Initially, the texture rapidly softened because of cell separation as a result of the solublization of the intercellular pectins by the alkali. At the higher alkali concentrations the short chain fragments immediately react with calcium and contribute to the formation of a soft sticky mass which has completely lost its former, hard, crisp nature. In the low alkali concentrations the CS pectins bind the cells together in a normal tissue network, while in the 0.500 N alkali, the CS fraction of short chain lengths binds together the swollen starch granules freed from the cellular organization by the dissolution of the primary cell walls.

C. <u>Alkali-soluble (AS) pectic fraction</u>. The results for this fraction are shown in Table 6.

There is little change in protopectin from the fresh to the water pack.

The slight increase in AS pectins in the weak acid pack over the water pack probably represents a calcium binding of some of the longer chain WS pectins to the primary cell wall as binding sites become available as a result of demethylation. This corresponds to the initial calcium increase in the corms. The initial decrease in phosphate and potassium in the corms would tend to make the slightly soluble pectins more insoluble with a probable increase in the most insoluble fraction, namely protopectin (15). In the acid series the protopectin decreased in a regular manner with increasing acid strength. This was due to the removal of calcium, the hydrolysis of the pectic chain and the increase in phosphate bonds--all factors tending to increase the solubility of the AS fraction (protopectin).

There is a decrease in protopectin from the water pack to the dilute alkali pack arising from a hydrolytic cleavage of the chain.

The decrease in protopectin with increasing alkali concentration continued up to the 0.125 N pack due to chain hydrolysis. The increase at the highest alkali concentration of the AS fraction was a result of some recombination with calcium and the entrapment of the primary cell wall between an exterior layer of insoluble pectinates and the swollen starch granules on the cell interior. The progressive removal of phosphate from the macromolecule would increase the insolubility of the AS pectic fraction.

The primary cell wall is almost completely dissolved in the acid series at the two highest concentrations of HCl and this loss of primary structure is shown by the low shear-press values. Hydrochloric acid catalyzes the hydrolysis of the starch from within the cell, the hydrolysis of protopectin and cellulose in the primary cell wall and dissolves the intercellular pectins largely by calcium exchange. This creates an open, sponge-like texture which permits ready diffusion of the soluble constituents into the packing medium.

The final situation, as far as structure is concerned, is quite different in the alkali series. The insoluble pectins (CS and AS fractions) are readily degraded by the alkali, but the two fractions were not solubilized at the higher alkali concentrations. In addition, the starch granules located inside the cell are chemical modified and physically swollen, but were not broken down to soluble monomers. This created a sticky, gummy mass tied together with polyvalent cation bridges which has little firmness but great resistance to diffusion and dissolution.

As a result, the interpretation of texture based solely on analytical data becomes extremely complex and difficult. This fact is amply recorded in the pectin literature. The pectic substances hold a key position in controlling texture largely because of the dynamic and ever-changing role they play in the cell wall and as an intercellular cement. Because of this ready alteration in physical and chemical properties, artifacts are easily created which make final structural interpretations exceedingly difficult. The above interpretation should shed considerable light on past conflicts and again emphasizes the vital role of the pectic substances as the primary determinates of texture.

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D. <u>Total pectins</u>. A slight drop in the total pectin from the fresh corm to the water pack occurred because of the diffusion of the WS fraction into the packing medium.

The total pectin remained constant in the acid series indicating little removal, although a redistribution among fractions did occur as shown in Figures 5 and 6.

The alkali series showed slightly lower total pectin values indicating a more complete cleavage of the pectin molecule but decreased solubility due to calcium bonding, loss of phosphate bonding and hindered diffusion. This analysis is confirmed by the high-lowhigh trend in the total pectin figures with increasing strengths of alkali.

4. The AIS fraction.

Table 7 shows that considerable differences were found in the amounts of alcohol insoluble solids between treatments and lots. As shown in Table 7, the decrease in AIS from the fresh sample to the water pack was appreciable. This loss is reflected in a corresponding decrease in the starch fraction (see Figures 5 and 6).

In the acid treated samples, the linear decrease in AIS was a result of the hydrolysis of starch and cellulose.

	Percent Total Solid					Perce	nt Ash		Percent AIS				
		Lot				Lot				Lot			
Normality	A	В	С	Mean	A	В	С	Mean	Α	В	C	Mean	
Acid Series													
0.031	12.69	14.41	8.11	11.74	0.31	0.40	0.31	0.34	7.53	7,64	4.25	6.47	
0.062	11.01	11.24	7.98	10.07	0.30	0.42	0.32	0.35	4.89	3.81	3.29	3.99	
0.125	9.43	10.08	6.57	8.69	0.33	0.49	0.33	0.38	3.17	3.72	1.51	2.80	
0.250	7.98	10.68	6.70	8.45	0.35	0.48	0.35	0.39	1.17	2.01	0.94	1.37	
0.500	7.59	10.17	6.52	8.09	0.37	0.50	0.40	0.42	1.07	1.42	0.88	1.12	
Alkali Serie	S												
0.031	13.21	15.79	10.07	13.02	0.34	0.56	0.43	0.44	8.15	9.20	5.68	7.67	
0.062	13.00	13.76	9.23	11.99	0.48	0.56	0.53	0.52	6.46	7.10	4.62	6.06	
0.125	14.69	13.16	9.06	12.30	0.74	0.84	0.84	0.81	7.01	7.10	4.65	6.25	
0.250	13.47	13.82	9.40	12.23	1.29	1.29	1.15	1.24	7.32	7.27	4.29	6.28	
0.500	14.70	14.40	10.63	13.24	2. 91	2. 41	2.08	2.47	6.54	7.51	4.34	6.13	
Water Pack	11.93	13.44	9.81	11.72	0.26	0.45	0.36	0.36	7.16	8.33	5.42	6.97	
Fresh	19.14	22 . 36	15.09	18.83	0.83	0.86	0.82	0.84	8.62	9.61	5.46	7.89	

Table 7. The percentages of total solid, ash and AIS in Chinese waterchestnuts.¹

¹On 100 gram fresh weight basis.

The initial rapid decrease in AIS in the weak alkali, and a constant value at higher alkali concentrations followed the starch pattern, except at the highest alkali concentration where the starch decreased while the AIS remained constant. This apparently was a result of including a sodium salt in the AIS fraction. A high ash determination in the 0.500 N alkali samples confirmed this observation.

In the alkali treated samples, the AIS percentage remained within narrow limits, incomparison to the steady decrease in the acid series.

The texture of the 0.030 N alkali pack was firmer than that of the water pack. This is a result of the neutralization of the normal plant acids resulting in decreased starch hydrolysis and higher AIS values for the weakest alkali packs.

5. The total sugars.

Five gram samples of the slurry were used for the determination of the total sugar. The data obtained is tabulated in Table 8 and Figures 7 and 8. There was a positive linear relationship between the corrected degrees Brix and the sugar fraction. This would be expected, and indicated that the principal change in the soluble solids (corrected for NaOH and HCl) was a result of changes in the soluble sugars.

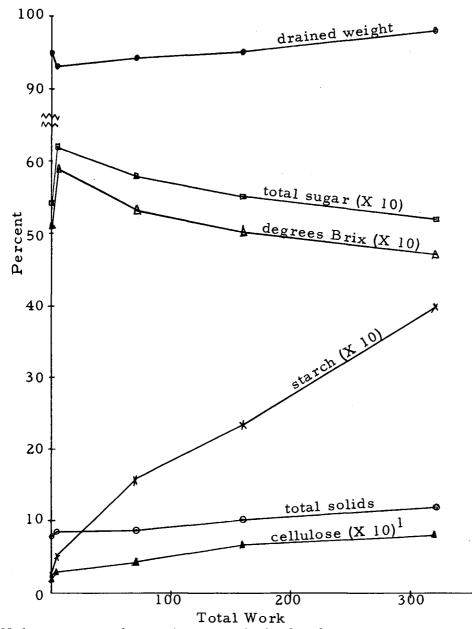
	P	ercent 7	Cotal Su	ıgar		Degrees Brix ²						
	Lot				Lot				Lot			
Normality	A	В	С	Mean	A	В	C	Mean	A	В	С	Mean
Acid Series												
0.031	5.43	6.88	3.25	5.18	4.26	5.01	2.68	3.98	5.3	5.3	3.5	4.7
0.062	6.94	7.35	4.18	5.47	2.45	2. 26	2. 27	2.33	6.1	5.6	3.9	5.0
0.125	7.18	7.05	5.28	5.77	2.00	1.91	0.80	1.57	5.9	5.8	4.2	5.3
0.250	6.72	6.99	4.55	6.19	0.12	1.20	0.25	0.52	6.2	6.8	4.7	5.9
0.500	6.22	6.84	3.17	5.41	0.12	0.30	0.20	0, 21	6.1	5.8	3.5	5.1
Alkali Series	5											
0.031	4.00	5.09	5.37	4.82	6.00	7.67	4.18	5.95	3.9	4.0	3.3	3.7
0.062	4.67	5.56	4.99	5.07	4.91	5.70	3.50	4.70	3.9	4.1	3.6	3.9
0.125	4.20	5.87	3.60	4.55	5.51	5.81	3.50	4.90	4.1	3.9	3.1	3.7
0.250	3.68	5.46	4.85	4.66	5.34	5.39	3.01	4.58	4.0	3.8	3.0	3.6
0.500	3.70	4.75	3.14	3.86	3.82	4.05	2.12	3.33	3.2	3.3	2.5	3.0
Water Pack	4.50	5.56	3.77	4.61	4.17	7.00	4.10	5.09	3.8	3.9	3.0	3.6
Fresh	10.44	11.17	9.24	10.28	6.27	7.83	4.26	6.12				

Table 8. The percentage of sugar, starch and degrees Brix in Chinese waterchestnuts.¹

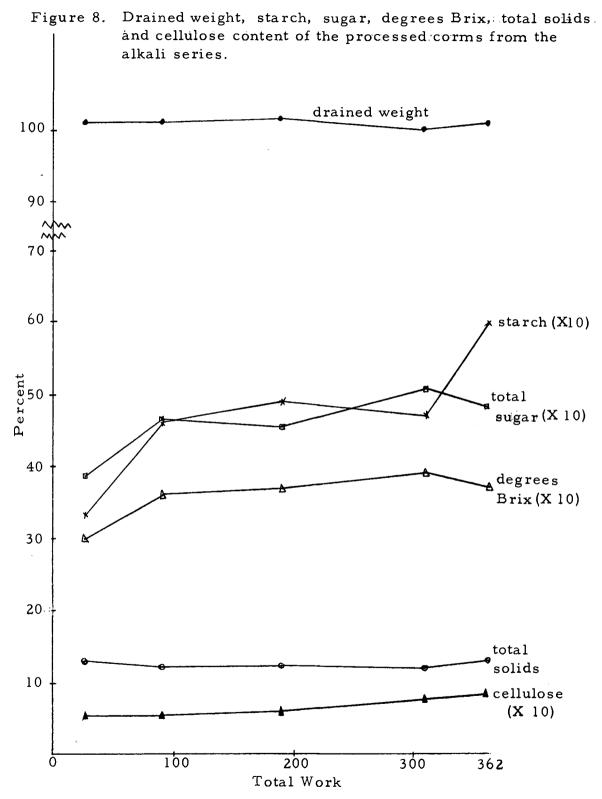
¹On 100 grams fresh weight basis.

²Measured from the processed liquid.

Figure 7. Drained weight, sugar, degrees Brix, starch, total solids and cellulose content of the processed corms from the acid series.



¹Values on graph ten times analytical values.



Acids serve as catalytic agents of hydrolysis. Stronger acids produce furfural from pentoses, and 5-hydroxy-methyl furfural or levulinic acid from hexoses (21, p. 69; 3, p. 40).

The action of alkalies follows two general courses namely isomerization at the reducing end of the molecules and fragmentation.

The higher analytical values for total sugars in the fresh corms than in the acid and alkali processed samples resulted from the dilution of the sugar by the liquid packing media used in the processed samples.

The corm/liquid dilution ratio was about 1.0/1.8 while the ratio of sugars in the water pack/fresh corms was 1.0/2.2, which is close to the water ratio between the corms and the packing medium.

The sugars increased in a regular fashion with increasing acid concentrations due to hydrolysis of both the starch and cellulose. The decreased concentration in the highest acid series represented the complete diffusion of sugars into the water unhindered by cellular structure and also some loss of sugars via degradation reactions.

Sugars remained relatively constant in the alkali series. Both starch and sugar fractions were constant, and there was no indication of a direct conversion of starch to sugar. No dextrins were formed. Seemingly, as the starch was degraded, these subunits form colored polymers. . The acid treated series had a higher sugar percentage than the alkali treated series at all levels of reagent concentration.

6. Total solids.

The total solids decreased in a regular manner with increasing concentrations of HCl as a result of losses in both cellulose and starch, although at a somewhat slower rate of decrease due to an increase in the ash content. The analytical data are shown in Table 7 and Figures 7 and 8.

In comparison to the analytical results of the acid series, the alkali treated series had more constant values.

The total solids in the alkali series followed the changes in AIS and starch with the exception of a higher total solids value at the highest alkali concentration. The high total solids was a result of the high ash content of this sample, the high contents of the calgonand alkali-soluble pectins and the pigmented polymers of the degradated and rebuilt starch molecules.

7. The starch fraction.

Table 8 and Figures 7 and 8 show the results of the analytical values for starch.

In the acid series, the starch decrease followed the pattern of the cellulose decrease. The acid hydrolysis of starch was more complete than the hydrolysis of the cellulose. In the acid series, the percentage of starch remaining in the corm was much less than in the alkali treated series. The expected starch hydrolysis pattern of starch to dextrin to sugar is evident as seen in the following chart.

	mg Percent of Dextrin							
Normality	Lot A	Lot B	Lot C					
0.031	94	53	43					
0.062	72	42	14					
0.125	58	37	0					
0.250	0	14	0					
0.500	0	0	0					

The starch in the alkali series remained fairly constant. Starch in the presence of alkali and heat swells, degrades and forms a gel. Alkali-treated starch has a sticky gummy texture. Though the starch values did not show a sharp change in percentage, the physical and chemical changes in the starch did affect the texture and structure of the product. This change in the starch granules may be a factor in crispness changes in the corm.

The starch value showed a drop in the 0.500 N NaOH processed corms. This may be due to the degraded starch rebuilding into macromolecules along with other chemical constituents resulting in insoluble pigments. Therefore, this portion of the starch may not be shown in the analytical values for starch. The existence of insoluble, pigmented molecules agrees with the observed increase in the total solids and the development of a brown color.

The starch content in many vegetables is related to the texture

of the processed product, but this relationship is complex as in the case of the white potato. There does not appear to be any trend in these data to indicate that a relationship existed between the analytical values for starch and the texture of the processed corms. However, there are definite changes in the physical nature of the starch, probably related to crystallinity changes, which may play a dominant role in crispness.

8. The cellulose fraction.

The results of the analysis of the cellulose fraction are shown in Table 5 and Figures 7 and 8. A definite trend of decreasing cellulose with increasing reagent concentration was shown in the samples subjected to both the acid and alkali treatments. In the acid treated series, the hydrolysis was more drastic than the fragmentation in the alkali series. Both the water pack and the treated samples showed a regular decrease in cellulose in comparison to the fresh samples.

The linear decrease of the cellulose in both series was the only common factor present in the analytical data of both the acid and alkali treated samples that correlated with the shear-press measurements.

Cellulose is a major constituent of all plant life. Together with the pectic substances, cellulose is the compound that gives strength

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to plant tissue. The two processing techniques which affect these structural compounds are heat and chemical treatment. Accompanying any textural change, there must be either a thinning of the cell walls and/or a separation of the cells as the cementing power of the intercellular material decreases.

Since the hydrolysis of cellulose correlated with an increase in softness, a reduction in thickness of the cell walls must be a primary factor in the loss of texture in both the acid and alkali packs. The rigidity of the cell walls is an important factor in the texture of the Chinese waterchestnut.

A change in the nature of the cell wall cellulose is apparent from a study of the shear press diagrams. The loss of the sharp secondary peak in all processed samples denotes a loss of fiber from the plant material. The fiber is composed mainly of cellulose and may be associated with the fibrovascular bundles or simply with the cell walls.

9. Total ash.

The high percentage of ash shown in the alkali treated samples was due to the introduction of large amounts of sodium in the samples. There was a marked decrease in ash in the weaker, sodium hydroxide, treated samples.

The steady upward trend of ash in the acid treated samples

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was due to the increase of the chloride ion from the hydrochloric acid.

The chloride salts diffused more rapidly into the water medium in the cans, thus the rate of ash increase was greater in the alkali series.

The data is tabulated in Table 7 and Figures 9 and 10.

10. Calcium, potassium and phosphorus.

The data in Table 9 and Figures 9 and 10 show the linear increase of phosphorus in the acid series with a decrease in both calcium and potassium. In the alkali treated series (Figure 10) the situation is reversed with an increase in calcium and potassium and a decrease in phosphorus.

The effect of the addition of HCl and NaOH on the minerals present in the corms is a relationship which depends upon the solubility of the salts formed, the rate of diffusion of these salts from the corms and the extent to which ion exchange occurs in the insoluble organic molecules.

In the acid processed samples, the decrease of both calcium and potassium was due to two dominant factors: (1) the acid hydrolysis of the macromolecules that are bridged by the calcium ions, thus setting the ions free, and (2) the ion exchange between the hydrogen and the calcium and potassium to form chloride salts which are

Normality	mg	g Perce	nt Calci	um	mg	mg Percent Phosphorus						
	Lot				Lot				Lot			<u> </u>
	A	В	С	Mean	A	В	С	Mean	A	В	С	Mean
Acid Series												
0.031	3.2	6.0	3.8	4.3	58	51	77	62	12	19	12	14
0.062	2. 2	5.3	3.1	3.5	55	47	69	64	15	25	15	18
0.125	2.0	5.0	2. 1	3.0	20	44	51	38	23	33	20	25
0.250	1.9	2.4	1.9	2.1	23	9	49	27	23	33	23	26
0.500	1.8	2.1	1.8	1.9	24	8	48	27	25	33	23	27
Alkali Series												
0.031	4.7	5.0	5.3	5.0	45	27	110	61	35	34	31	33
0.062	5.6	6.7	6.0	6.1	58	78	110	82	33	36	35	34
0.125	6.7	7.5	7.0	7.1	51	80	116	82	31	35	32	33
0.250	11.0	10.6	9.4	10.3	177	126	129	144	23	35	28	28
0.500	12.8	15.2	14.6	14.2	157	137	147	147	18	18	17	18
Water Pack	5.3	5.7	5.0	5.3	177	170	140	162	18	22	15	18
Fresh	8.1	10.2	8.0	8.7	223	3 26	409	319	40	36	44	40

Table 9. The mg percentage of calcium, potassium and phosphorus in Chinese waterchestnuts. l

On 100 g fresh weight basis.

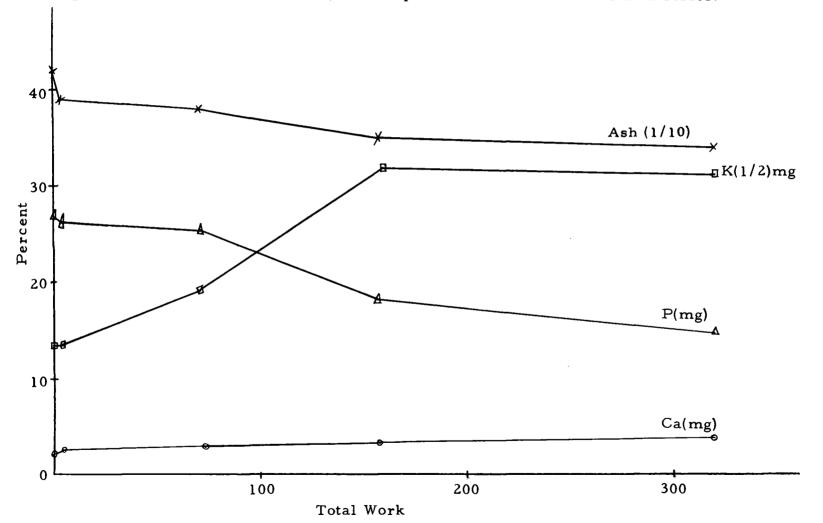


Figure 9 Ash and mineral contents of the processed corms from the acid series.

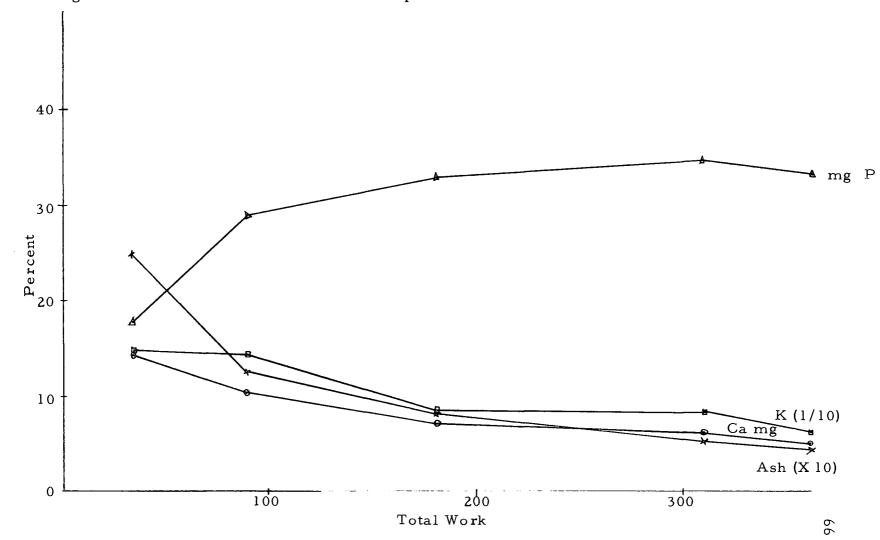


Figure 10. Ash and mineral content of the processed corms from the alkali series.

soluble in the processing liquid. The pectic acid formed by exchanging the calcium ion with the hydrogen ion is more soluble than the calcium pectate. This postulation is supported by the low amounts of calgon-soluble pectins and the low calcium values for the strong acid packs.

In the alkali treated series, the increase of calcium and potassium results from the demethylation and degradation of the pectin molecule with the subsequent addition of cations to form pectates only slightly soluble in alkali. While the potassium and calcium pectates form viscous solutions or gels, the effect on crispness is nil. This postulation ties in with the increase in calgon-soluble pectin at the higher alkali concentrations.

The decrease in phosphorus may be explained by the solublization of phosphorylated molecules and the ready diffusion of Na_3PO_4 from the corms.

11. Color evaluation of the processed liquid.

Ten ml of liquid from the canned corms was used for the color determination by the photovolt color reflection meter. The Y value is found by using the green filter corresponding to the table given in the literature (16, p. 354-355).

The hue and chroma were determined by using Munsell Color Charts. The calculated chromaticity co-ordinates, x and y, were used to locate a point on the proper Munsell color charts.

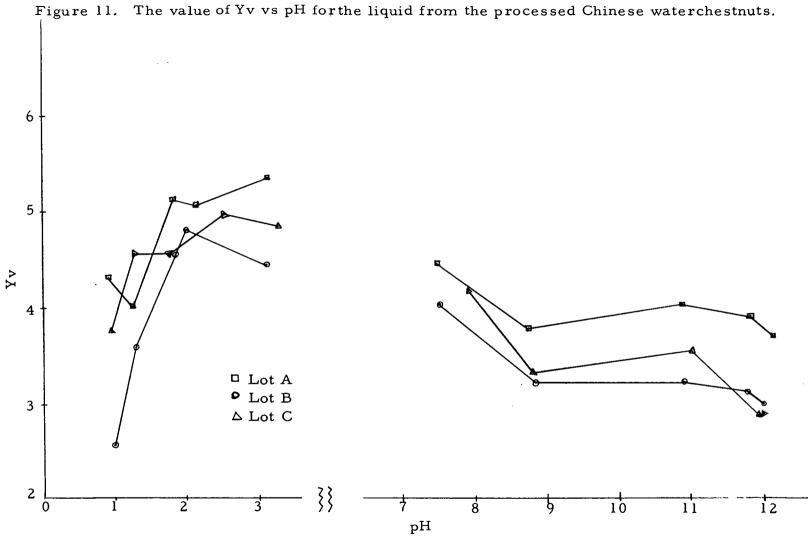
The results obtained are shown in Table 10.

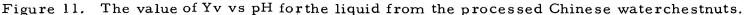
Figure 11 shows the relationships between pH and Yv, the value of the green filter.

Normality	Co-ordinate x				Co-ordinate y				Yv			
	Lot				Lot				Lot			
	A	B	С	Mean	A	В	С	Mean	A	В	С	Mean
Acid Series												
0.031	0.366	0.314	0.344	0.341	0.162	0.168	0.164	0.165	5.35	4.43	4.86	4.88
0.062	0.351	0.311	0.332	0.331	0.160	0.174	0.179	0.172	5.03	4.80	4.97	4.93
0.125	0.327	0.323	0.322	0.324	0.157	0.157	0.168	0.161	5.14	4.56	4.56	4.75
0.250	0. 297	0.378	0.350	0.341	0.161	0.152	0.176	0.163	4.00	3.59	4.56	4.05
0.500	0.361	0.338	0.303	0.334	0.147	0.141	0.139	0.143	4.30	2. 56	3.76	3.54
Alkali Series										•		
0.031	0.335	0.349	0.374	0.353	0.132	0.173	0.164	0.157	4.43	4.00	4.16	4.19
0.062	0.307	0.351	0.388	0.349	0.123	0.136	0.162	0.140	3.77	3.20	3.30	3.64
0.125	0.364	0.353	0.359	0.358	0.147	0.138	0.145	0.143	4.00	3.20	. 3. 52	3.49
0.250	0.355	0.342	0.339	0.345	0.151	0.156	0.156	0.155	3.84	3.10	2.75	3.23
0.500	0.350	0.387	0.316	0.351	0.164	0.147	0.166	0.159	3.68	3.00	2.87	2.18
Water Pack	0,371	0.319	0.329	0.339	0.158	0.156	0.174	0,163	5.54	4.94	5.20	5. 22

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Table 10. The chromaticity co-ordinates (x and y) and the value of Yv for the liquid from the processed waterchestnuts.





SUMMARY AND CONCLUSIONS

In this thesis, the effects of processing on the texture and chemical composition of Chinese waterchestnuts were studied. Texture was measured by the resistance to shear. Texture differences were created in the processed corms by canning in different concentrations of acid and alkali.

The change in total pectin was small, although marked changes occurred in the distribution of the total pectic substances among the three pectic fractions.

The WS pectins played a secondary role in texture. In dynamic equilibrium with the water-insoluble fractions (AS+CS), the WS pectins served as a transition state and material reservoir for changes between the two insoluble fractions. In the alkali series, the adsorption and entrapment of the WS fraction by the swollen starch granules made the values for the WS pectins at the highest and lowest alkali concentrations approximately equal.

In both series, cellulose was the only common factor present in the analytical data from both the acid and alkali series which correlated with the shear-press measurements. Together with the pectic substances, cellulose is the compound that gives strength to plant tissues. Heat and chemical treatment causes a thinning of the

cell walls and/or a separation of the cells as the cementing power of the intercellular material decreases and these treatments are accompanied by a textural change.

As mentioned by F. A. Isherwood,

Experience indicates, however, that all the factors tend to be interrelated and changes in texture are not caused by any one factor alone. This would be expected when one considers that the various constituents of the cell are in dynamic equilibrium with each other and that the soluble sugars, acids, phosphoric esters, phenolic substances, aminoacids, amides and other metabolites are being continuously produced from, and built into, the insoluble materials of the plant cell wall. Changes in texture must in the final instance be related to changes in the overall pattern of the cell composition and structure and, though the obvious approach may be to examine the cell-wall structure, a detailed analysis of the soluble metabolites may give more significant information (14).

On the basis of the experiments performed and the results

obtained, the following conclusions were drawn:

1. The Chinese waterchestnuts processed in both acid and

alkali lose their normal crisp texture.

2. A linear relationship existed between the pH of the acid

and texture as measured by total work and maximum force.

3. The percentage of total solids and AIS showed a significant

change between the control and processed samples.

4. Drained weights were more adversely affected by canning in acid than in alkali.

5. Resistance to shear in the Chinese waterchestnut inincreased with an increase in the percentage of cellulose. The corms gave a linear correlation between texture and cellulose.

6. The relationship between firmness of the corms and the pectic substances in this study emphasized the dynamic role of the pectic compounds in texture changes.

7. The treatments in the acid series showed more definite trends of change in chemical composition than the alkali series.

8. The color measurement by the Photovolt color meter showed that Chinese waterchestnuts, processed in both acid and base, were all in the region of reddish purple on the x, y chromaticity diagram. The higher Munsell Yv values were found in the weakest acid and alkali packs and the water pack.

9. The measurements of the degrees Brix in the processing liquid showed a consistent pattern with the total sugar of the corms.

10. Texture of a product, whether raw or processed, is intimately associated with its structural makeup. The changes in texture are not a result of one factor, but due to the interrelation of many factors.

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