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SEPARATION, FRACTIONATION, CONCENTRATION AND DRYING
OF FOOD PRODUCTS

Technology Progress Report for the Period October 1, 1984—March 3, 1987

By
W. W. Rose
L. D. Pederson
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November 1987

Work Performed Under Contract No. AC07-84ID12553

For
U. S. Department of Energy
Office of Industrial Programs
Washington, DC

By
National Food Processors Association
Dublin, California

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***Separation, fractionation,
concentration and drying of food
products: Technology progress
report, October 1, 1984-March 3,
1987***

Rose, W.W.; Pederson, L.D.; Merlo, C.A.; Brewbaker, P.L.

Nov 1987

National Food Processors Association, Dublin, CA (USA)

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FOR YOUR INFORMATION AND ACTION

Technical Progress Report - Separation, Concentration and Drying
of Food Products (October 1, 1984 to March 3, 1987)

The National Food Processors Association has completed Phases I and II of an effort to develop more energy efficient technologies for processing, containerizing, and transporting juice products.

This on-going effort has been partly supported by the Department of Energy's Office of Industrial Programs and is reviewed in the enclosed report. The concept to separate foods and concentrate the components at one site and then recombine at another is novel and could be very effective in reducing energy consumption and various other costs in the food industry.

Please review the report and provide comments and recommendations by phone or writing to:

A handwritten signature in black ink, which appears to read "William M. Sonnett", is written over the typed name.

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Your input will be greatly appreciated.

SEPARATION, FRACTIONATION, CONCENTRATION AND DRYING
OF FOOD PRODUCTS

Technology Progress Report
October 1, 1984 to March 3, 1987

by

W. W. Rose
L. D. Pederson
C. A. Merlo
P. L. Brewbaker

November 1987

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Prepared for
U.S. Department of Energy
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Office of Industrial Programs
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EXECUTIVE SUMMARY

This study was undertaken to assess the potential for developing more energy efficient technology for processing, containerizing, and transporting of juice products.

Container manufacturing and product transportation consume significant energy in conjunction with food production. Evaporation is presently used to concentrate food products, especially juice products; however, a large fraction of the final product is water, which is packaged and shipped with the food product. For example, tomato paste, which is produced by concentrating tomato juice from 5% total solids to 30% total solids, is over 60% water. Approximately 500,000 tons of tomato paste are produced, canned, and transported each year in the United States. In other words, over 300,000 tons of water are transported with tomato paste each year. Due to technical and economic limitations, evaporation cannot be used to further concentrate food products.

In this project, an advanced process is being investigated; this process, if successful, will greatly reduce the amount of water that must be packaged and shipped with food products. The research is described below.

In Phase I of this project (described in detail in this report), tomato puree was fractionated by conventional vacuum filtration, centrifugation, and crossflow microfiltration to produce a pulp fraction and a serum fraction. The resulting fractions from each separation were characterized. The pulp and serum fractions were recombined by homogenization, stomacher

blending, and static (in-line) mixing; the viscosity of the resulting recombined tomato purees were determined and compared to the viscosity of the original puree. In this study, viscosity was used as the most important quality characteristic of tomato puree. Centrifugation (for fractionation) followed by homogenization (for recombination) produced a recombined puree with viscosity equivalent to that of the original puree. In subsequent Phase I studies, the pulp fraction was rinsed with water to recover the soluble components of the pulp (producing a rinsed-pulp fraction) and the rinse water was added to the serum fraction (producing a rinse/serum fraction). The rinse/serum fraction was then concentrated by vacuum evaporation to produce a concentrated-rinse/serum fraction (50% and 21% total solids [TS]). The resulting fractions were characterized. The rinsed-pulp and concentrated-rinse/serum fractions were recombined (with water) by homogenization; satisfactory puree was produced.

In Phase II (described in detail in this report), the following fractionation sequence was investigated. Puree was fractionated by centrifugation and the resulting pulp fraction was rinsed to produce a rinsed-pulp fraction. The rinse water was added to the serum fraction to produce a rinse/serum fraction. The rinse/serum fraction was 1) concentrated by vacuum evaporation to 13% TS, or 2) further fractionated by ultrafiltration (with a 100,000 molecular-weight-cutoff membrane) to produce a high-molecular weight (MW) fraction and a low/intermediate-MW fraction. The resulting low/intermediate-MW fraction was then further fractionated by ultrafiltration (with a 2000-MW-cutoff membrane) to produce a low-MW fraction and an intermediate-MW fraction.

The low-MW fraction was treated by anion exchange to separate organic acids from sugars. The organic acid salts were recovered from the anion exchange resin and were regenerated by cation exchange.

The low/intermediate-MW fraction (pre-ion-exchange) was concentrated by vacuum evaporation to 74% TS.

Recombined puree was produced by homogenizing concentrated rinse/serum (13% TS) with pulp and water, and by homogenizing concentrated low/intermediate-MW fraction, high-MW fraction, pulp and water. The viscosity of these purees was close to the baseline puree; colors were lighter and yellower.

Phase II also included fractionation/recombination studies using fresh tomato juice. In the fresh juice studies, a puree containing 11% TS was produced by vacuum evaporation to serve as a baseline so that the viscosity measurement commonly used by industry (Bostwick) could be used to assess product quality. The fresh juice was centrifuged to produce a pulp fraction and a serum fraction. The pulp fraction was not rinsed in the fresh juice studies. The serum fraction was concentrated by vacuum evaporation to 13% and 70% TS. A satisfactory recombined puree was produced upon homogenizing the pulp, concentrated serum (70% TS), and water, or upon static mixing or homogenizing the pulp, concentrated serum (13% TS) and water.

Phase II also included product stability tests, in which recombined puree (made with 13% TS serum from fresh juice) was canned and stored (under refrigeration) and the cans were opened at one, four and eight months to examine the recombined puree for quality stability. Quality was stable for the samples studied.

ABSTRACT

This report describes the first and second phases of a three phase project, the object of which is to develop energy efficient separation, concentration, and drying processes for food products, especially juice products, in order to reduce energy requirements and their associated costs for processing, preservation, and transportation. Presently in juice processing, much water is eliminated through evaporation. However, there are significant limits to the current evaporation technology. If, however, the juice could be separated, prior to evaporation, into liquid and solid fractions, and the liquid concentrated further, the containerization and transportation costs could be significantly reduced.

In the first phase of this study, tomato puree was separated by three methods: conventional vacuum filtration, centrifugation, and crossflow microfiltration, producing pulp and serum fractions. The greatest separation by weight was obtained by centrifugation. The separated fractions were characterized. The clearest serum was obtained by microporous filtration. The separated fractions were recombined by three techniques (static mixing, stomacher blending, and homogenizing) and evaluated. The recombined puree consistently regained viscosity when homogenized, and in some cases, when static mixed.

In a subsequent part of the first phase tomato puree was separated by centrifugation and the pulp fraction was rinsed producing a rinsed-pulp fraction. The rinse water was added to the serum fraction producing a rinse/serum fraction. The rinse/serum fraction was concentrated by vacuum evaporation to

30, 50, and 70% total solids (TS). These fractions were characterized. Rinsing the pulp successfully lowered the sugar content of the rinsed-pulp.

The concentrated rinse/serum (50% and 21% TS) and rinsed pulp were recombined with the addition of water by homogenization, producing satisfactory puree.

The relation of viscosity to protein and pectin content of ripening tomatoes was also examined. The percent protein in the serum more closely followed the changes in viscosity than did percent pectin.

In the second phase, tomato puree was centrifuged to produce a pulp fraction and a serum fraction. The pulp fraction was rinsed producing a rinsed-pulp fraction and a rinse/serum fraction. The rinse/serum was then fractionated into high- and low/intermediate-MW (molecular weight) fractions by ultrafiltration with membranes of 100,000-MW-cutoff. The permeate stream (low/intermediate-MW fraction) from this ultrafiltration was then ultrafiltered with membranes of 2000-MW-cutoff producing intermediate- and low-MW (permeate) fractions. The low-MW fraction was treated by weak base anion exchange to remove organic acids from the fraction. The organic acid salts were recovered from the anion exchange resin and regenerated by cation exchange.

The low/intermediate-MW fraction was concentrated by vacuum evaporation to 74% TS. Satisfactory recombined puree was made by homogenizing rinsed pulp, concentrated low/intermediate-MW fraction, high-MW fraction, and water. Satisfactory recombined

puree was also made by homogenizing concentrated rinse/serum (13% TS) with rinsed pulp and water.

The second phase also included studies with fresh tomato juice. The juice was centrifuged into pulp and serum. Puree was made by vacuum evaporating juice to provide a baseline to compare with previous puree studies. Serum was concentrated by vacuum evaporation to 13% and 70% TS. A recombined puree was made by homogenizing pulp, concentrated serum (70% TS), and water, or by static mixing the pulp, concentrated serum (13% TS) and water. The viscosity was satisfactorily restored. The recombined puree (made with 13% TS serum) was canned and stored 8 months (under refrigeration) and quality was stable.

TABLE OF CONTENTS

	<u>Page</u>
Executive Summary	ii
Abstract	v
List of Figures	x
List of Tables	xii
Acknowledgements	xiv
Introduction	1
Testing and Results	4
Equipment	4
Analytical Procedures	11
Procedures and Results	14
Phase I	14
Separation and Recombination Studies	14
Separation and Characterization	15
Additional Microfiltration Studies	22
Recombination and Evaluation	27
Additional Recombination Studies	33
Additional Studies	37
Separation Technique Selection	37
Pulp Rinsing, Serum Concentration, and	
Recombination Studies	38
Pulp Rinsing	38
Serum Concentration	39
Recombination and Evaluation	42
Tomato Maturity Study	45
Phase II	53
Separation Study	53
Ultrafiltration and Concentration Studies	55
Initial 2000 MW Ultrafiltration	55
Permeate Concentration	57
100,000 MW Ultrafiltration	57
Permeate Concentration	62
2000 MW Ultrafiltration	64
Ion Exchange Studies	66
Initial Anion Resin Testing	66
Separation of Low MW Fraction	68
Initial Cation Resin Testing	69
Conversion of Organic Acid	70
Salts to Acids	

TABLE OF CONTENTS (Continued)

	<u>Page</u>
Mass Balance	75
Recombination Studies	77
Second Level Recombination	77
First Level Recombination	79
Pulp Drying Studies	79
Drying of Rinsed Pulp	80
Hygroscopy of Dried Pulp	82
Fresh Juice Studies	85
Puree Production	85
Serum Concentration	85
Recombination	87
Recombination with Low	
Level Solids Serum	87
Recombination with High	
Level Solids Serum	89
Storage Effects	91
Recombined Product Storage	91
High Level Solids Serum Storage	93
Ultrafiltration of Serum	93
Conclusions and Recommendations	101
References	102
Appendix A - Abbreviations	103
Appendix B - Metric to English Conversion Table	104

LIST OF FIGURES

<u>No.</u>		<u>Page</u>
1	Separation, fractionation and concentration schematic	3
2	Recirculation mode operation	5
3	Batch mode operation	5
4	Continuous mode operation	9
5	High performance liquid chromatogram of organic acids for three samples and a standard. The samples: original puree, microfiltered pulp, and microfiltered serum contained respectively: 0.91, 0.94, and 0.86% citric acid.	18
6	Microporous filter(0.2 micron)--Flux versus pressure and flow	23
7	Microporous filter(0.2 micron)--Flux versus pressure and temperature	25
8	Microporous filter(1.0 micron)--Flux, temperature, and % solids of permeate versus operating time	26
9	Bostwick viscosity (cm) versus stomach mixer mixing time(min) for unseparated puree (10.8% NTSS)	35
10	Bostwick viscosity (cm) versus homogenizing time (min) on unseparated puree (10.2% NTSS)	36
11	Bostwick viscosity (cm) versus homogenizing time (min) on unseparated puree (9.2% NTSS)	36
12	Percent protein in serum versus maturity stage (Bradford method)	47
13	Libby's efflux viscosity versus maturity stage	48
14	Serum viscosity versus maturity stage (Ostwald method)	49
15	Precipitate weight ratio versus maturity stage	50

LIST OF FIGURES (CON'T.)

<u>No.</u>		<u>Page</u>
16	Percent pectin in serum versus maturity stage (carbazole/H ₂ SO ₄ method)	51
17	Flux versus time - 100,000 MW ultra-filtration of rinse/serum	59
18	NTSS (%) versus time - 100,000 MW ultra-filtration of rinse/serum	60
19	Pressure drop versus time - 100,000 MW ultrafiltration of rinse/serum	63
20	pH vs. throughput for cation resin C-391b using NaOH regenerant from anion resin A-399b as feed	71
21	pH vs. throughput for cation resin C-391b using effluent >pH 2 (in Figure 8) plus "sweeten-off" backwash of resin in Figure 8; second feed is "NaOH-off" backwash from anion resin A-399b	72
22	pH vs. throughput for cation resin C-391b using NaOH regenerant from anion resin SM-73 as feed	74
23	pH vs. throughput for cation resin C-391b using effluent >pH 2 (in Figure 10) plus "sweeten-off" backwash of resin in Figure 10; second feed is "NaOH-off" backwash from anion resin SM-73	74
24	Hygroscopy - weight gain (%) versus time in tropical room	83
25	Hygroscopy - weight gain (%) versus time in prairie room	84
26	Flux versus time - 100,000 MW ultra-filtration in fresh juice serum	97
27	NTSS (%) versus time - 100,000 MW ultra-filtration of fresh juice serum	98
28	Pressure drop versus time - 100,000 MW ultrafiltration of fresh juice serum	100

LIST OF TABLES

<u>No.</u>		<u>Page</u>
1	Ultrafiltration Membrane Specifications	7
2	Separation Methods	16
3	Characterization of Puree and Separated Fractions	17
4	Characterization of Puree	19
5	Characterization of Diluted Pastes	20
6	Results of Static Mixer Recombination with Three Separation Techniques	29
7	Results of Stomacher Recombination with Two Separation Techniques	30
8	Results of Homogenizer Recombination with Three Separation Techniques	32
9	Consistency Measurements and Chemical Analyses of Homogenized Tomato Puree Samples	33
10	Change in Viscosity with Stomaching 2 Minutes and Sitting	34
11	Characterization of Puree and Rinsed and Concentrated Fractions	40
12	Original and Recombined Purees	43
13	Original and Recombined Purees after Different Homogenization Times	43
14	Original and Recombined Purees before and after Refrigerated Storage	44
15	pH of Tomato Juice and Serum Harvested at Six Stages of Maturity	52
16	Tomato Juice Hunter Color Readings of Tomatoes Harvested at Six Stages of Maturity	52

LIST OF TABLES (CON'T.)

<u>No.</u>		<u>Page</u>
17	Percent Total Solids in Tomato Juice Adjusted to 5.5 Brix from Tomatoes Harvested at Various Stages of Maturity	52
18	Characterization of Puree and Fractions	54
19	100,000 MW Ultrafiltration Testing with Rinse/serum	58
20	100,000 MW Ultrafiltration Temperature Scans with Rinse/serum	61
21	2000 MW Ultrafiltration Testing with Low/Int. MW Fraction	65
22	Acidity of Effluents from Cation Exchange with IWT C-391b of Organic Acid Salts Samples	73
23	Mass Balance	76
24	Results of Recombinations	78
25	Pulp Drying Results	81
26	Characterization of Fresh Juice and Fractions	86
27	Characterization of Fresh Juice and Recombined Samples	88
28	100,000 MW Ultrafiltration Testing with Fresh Juice Serum	96
29	100,000 MW Ultrafiltration Temperature Scans with Fresh Juice Serum	99

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INTRODUCTION

In 1970 the food processing industry used approximately 3.3 quads of energy for all aspects of food processing, including energy for processing the food, manufacturing paper, glass and metal containers, transporting food products, and manufacturing processing machinery and trucks and trailers for transportation. Of this total, 1.5 quads were used for manufacturing metal containers and for transporting food products (Loftness, 1978). Transportation and containerization are major costs associated with food processing.

In juice processing, for example, products are concentrated by evaporation prior to packaging and shipping to reduce containerization and transportation costs. The water content is reduced significantly. However, a significant amount of water remains in the concentrated product. For example, when tomato juice (~5% total solids) is conventionally concentrated to paste of 30%-40% total solids, it still contains 60%-70% water. Approximately 500,000 tons of conventionally concentrated tomato paste are produced in the United States each year, thus requiring transportation of 300,000 to 350,000 tons of water.

As tomato juice is concentrated by evaporation, the viscosity increases, thus limiting the amount of water which can be removed by evaporation. Refinements of the evaporation technology can only produce minor increases in achievable concentration.

In this project, a new approach is being studied (Fig. 1). In these studies, tomato juice was separated into pulp and serum fractions. This concept allows the individual fractions to be processed more efficiently. The pulp was rinsed to remove water soluble materials such as sugars and acids. These components may adversely affect the quality of the dried pulp. The rinse water

was added to the serum to produce a rinse/serum fraction. The resulting rinse/serum fraction was treated by UF to produce a high-molecular weight (MW) fraction and a low/intermediate-MW fraction. The permeate, or low/intermediate-MW fraction was ultrafiltered to produce a low-MW and an intermediate-MW fraction. These resulting fractions were concentrated by vacuum evaporation to further reduce water content. This separation, fractionation, and concentration process can provide substantial economic benefits by reducing the amount of water to be containerized and transported with the essential product. Three recombination techniques were studied: homogenization, stomacher blending, and static (in-line) mixing. Quality of the recombined product was analyzed in terms of viscosity, color, and syneresis. Additionally some studies were performed with fresh tomato juice instead of canned puree.

Fractionation of the rinse/serum fraction could allow concentration of individual components so that transportation of only "essential" ingredients is necessary. If practical, the sugars and/or organic acids will not be transported, and recombination may use locally available water, sugar, and organic acids.

The proposed approach may be applicable to the processing of tomato, orange, apple, and grape juices. Significant energy savings could result in several segments of the juice processing industry.

The overall objective of this project is the development of energy efficient separation, fractionation, and concentration processes for food products in order to reduce processing, preservation, and transportation energy requirements and their associated costs.

Figure 1 - Separation, fractionation, and concentration schematic.

TESTING AND RESULTS

EQUIPMENT

The following equipment was used for Phase I separation studies:

Centrifuge - Sorvall RC-5B and SS-3 centrifuges with GSA rotors used for separation by density. These centrifuge heads, when operated at 12,000 rpm, have a g-force (relative centrifugal force) of approximately $19,000 \pm 4000 \times g$ (gravity).

Conventional Filter - A conventional filter allowed separation by size. This consisted of a milk filter disc, 38 μ m pore size. This consisted of a milk filter disc, 38 μ m pore size in a Buchner funnel. Vacuum was used to drive the separation.

Microporous Filter - A ceramic microfilter (Norton Co.) also separated by size. Modules with 1.0 μ m and 0.2 μ m pore sizes were used. Each module (2.5 cm x 40 cm) contained eight ceramic tubes (2.8 mm ID) in a shell and tube configuration. The total surface area was 0.3 ft² per module.

Three different pumping units were used with the microporous filter: a Rannie A3 piston pump, a portable pumping unit (CARRE, INC.) with a centrifugal booster pump and a 3 hp high pressure diaphragm pump, a lab top pumping unit (Norton Co.) employing an external gear pump (Indiana General).

Two modes of operation were used. Recirculation mode (Fig. 2) is run with permeate and concentrate returning to the feed tank. Batch mode (Fig. 3) is run to effect concentration, with concentrate returning to the feed tank and permeate going to a permeate holding tank.

Figure 2 - Recirculation mode operation.

Figure 3 - Batch mode operation.

The following equipment was used for Phase II separation and fractionation studies:

Decanter Centrifuge - A Westfalia Model CA 220-110 Decanter was used for separating tomato puree. This centrifuge has a maximum g-force of ~3500.

Ultrafilter - A DDS/Niro Lab 20 Module with Rannie A3 piston pump was used. This is a plate and frame type module. Membranes used include:

2000-MW-cutoff value, Type GR90PP (polysulfone)
100,000-MW-cutoff value, Types GR40PP and PES-100
(polysulfone; polyether sulfone) (See Table 1 for specifications.)

Each membrane had 0.195 ft^2 (0.018 m^2) surface area.

Four modes of operation were used:

Recirculation mode and batch mode were run as described in the Microporous Filter section.

Batch diafiltration mode is used after batch mode has been used to produce a concentrate. Water is added to the concentrate in the feed tank and the UF unit is then run in batch mode.

TABLE 1. ULTRAFILTRATION MEMBRANE SPECIFICATIONS

Membrane Type	Component Type	Approx. MW-Cutoff Value	pH	Recommended Operation Limits			
				Temperature		Pressure	
				<u>°C</u>	<u>°F</u>	<u>MPa</u>	<u>psi</u>
GR40PP	Polysulfone	100,000	1-13	0-75	32-167	0-1	0-145
PES100	Polyethersulfone	100,000	NA	0-75	32-167	0-0.7	0-100
GR30PP	Polysulfone	2000	1-13	0-75	32-167	0-2	0-290

Batch diafiltration mode is used to wash low-molecular weight solutes out of the concentrate. The fraction of low-MW solutes removed is directly proportional to the amount they are diluted by the volume of water added to the feed tank.

Continuous mode (or feed and bleed) was run initially in batch mode to increase the concentration of the retentate to the level where the permeate SS (soluble solids) equalled the original feed SS. Then batch mode operation was continued, but with feed added periodically to the feed tank in amounts equal to the volume lost to permeate (Fig. 4). This was done to increase the amount of constituents passing through the membrane without diluting the permeate.

In some cases a temperature scan was made while in batch mode. During this test, the pressure and flow rates are held constant while the temperature is varied; flux and rejection data are monitored at various temperature settings.

Ion Exchange - Anion exchange resins, IWT SM-73 and IWT A-399b (Illinois Water Treatment Co.), were used for removing organic acids from sugars. The columns used were 2.3x27cm and 1.9x35cm respectively.

Cation exchange resin, IWT C-391b was used to convert organic acid salts to organic acids. The column used was 1.9 x 35 cm.

Figure 4 - Continuous mode operation.

The following equipment was used for serum concentration:

Hyperfilter - A spiral wound thin film composite membrane (FT-30) was examined for use in concentrating tomato serum. Each spiral wound module (6.4 cm diameter x 36 cm length) contains 0.52 m² membrane area. Two modules were used per pressure vessel. Pumping was accomplished by means of the portable pumping unit described in the Microporous Filter section. (Tomato serum for testing with the hyperfilter was produced by means of a tubular ZOSS [zirconium oxide stainless steel] ultrafilter [CARRE, Inc.] and two small pilot scale centrifuges [Alfa-Laval] [2000 and 18,000 rpm].)

Rotary evaporator - A Yamato Rotary Evaporator (Model No. RE-51B) and a Buchi/Brinkmann Rotovapor-R were used in connection with vacuum.

Pilot scale evaporator - A single effect Junior Plate Evaporator (APV) was used at ~160°F (70°C).

The following equipment was used for recombination of fractions:

Homogenizer - A Brinkmann Homogenizer PT-35 (also known as Polytron) with a PT-35/K probe generator which runs at 0-20,000 rpm was used. The homogenizer combines ultrasonic energy with mechanical shearing.

Static Mixer - A static mixer (Komax Systems, Inc.) was used. This inline mixer (1.2cm x 28cm) uses multivaned elements to divide and recombine a flowing process stream. Product flow was driven by a peristaltic pump (Sigmamotor T6S) at 0.5 gpm.

Stomacher - A Stomacher 80 laboratory blender (Dynatech Labs., Inc.) provided mixing by action of two reciprocating paddles on a Whirl-pak bag. This instrument is commonly used for mixing in the laboratory.

ANALYTICAL PROCEDURES

1. Natural tomato soluble solids (NTSS) - Samples were analyzed with a Bausch and Lomb 33-45-58 refractometer (Lamb, 1969). For some testing the term °Brix is used interchangeably with % NTSS, since Brix is read on the sugar scale (% sucrose) on a refractometer; % NTSS is also read as % sucrose on the sugar scale on a refractometer. Some samples were analyzed using an AO Hand Refractometer Model 10430.
2. Gross viscosity - The Bostwick procedure was used (NCA, 1968). In this test a low centimeter (cm) value indicates a high viscosity value.
3. Total Solids (TS) - Total solids were determined by drying at 70°C under vacuum for 4 hours (NCA, 1968). Samples were also analyzed using a microwave drying moisture solids analyzer (CEM Corp.) following the method of Chin et al. (1985).
4. Filtered Total Solids (FTS) - Samples were centrifuged at 10,000 rpm for 20 min. Supernatant was filtered with a Gelman Type A-E glass fiber filter. The filtrate was analyzed for total solids by the vacuum oven method.

5. Soluble Solids (SS) - The soluble solids was calculated from the total solids and filtered total solids determined by the vacuum oven method:

$$\%SS = \frac{\%FTS (100\% - \%TS)}{(100\% - \%FTS)}$$

This equation is derived from the following three equations:

$$\%FTS = \frac{\%SS}{\%SS + \%H_2O}$$

$$\%TS = \frac{\%SS + \%IS}{\%SS + \%IS + \%H_2O}$$

$$100\% = \%SS + \%IS + \%H_2O$$

6. Syneresis (Serum Separation) - Sample (40 gm) was weighed into a tube (26 mm i.d.) with a 60 mesh screen on the bottom. The tube was suspended over a 50 ml beaker. At time intervals (0.25, 0.5, 1, 2, 3, 4, 5 hrs) fluid that had accumulated in the beaker was weighed. Value reported is the weight of fluid at 1 hr divided by 40 gms x 100. This method was adapted from the method of N. Stoforos (University of California, Davis).
7. Color - A Minolta Chroma Meter CR-100 was used. A beaker (150ml) was filled to the rim with sample. The meter's measuring head was equipped with the glass light projection tube and was immersed ~1cm into the sample. Measurement ($L^*a^*b^*$) was made. (The instrument makes three measurements and records the mean

value.) The measuring head was removed from the sample, cleaned, and dried. The measurement process was performed a total of 10 times, and the overall mean was reported.

8. Titratable Acidity - The electrometric titration method, using 0.02N and 0.1N NaOH to pH 8.1, was used (NCA, 1968). The results were presented as the anhydrous citric acid equivalent.
9. pH - pH meters (IBC TropHy Meter and Yokogawa Model PH51 Pocket pH Meter) were used.
10. Conductivity - A conductivity meter (Yokogawa Model SC51 Pocket Conductivity Meter) was used.
11. Sugars - A Bio-Rad high performance liquid chromatographic (HPLC) system with column heater and refractive index detector was used. An HPX-87C column (Bio-Rad) was used with the HPLC system. Samples were diluted 1:10, mixed thoroughly, centrifuged, and the supernatant filtered through a 0.45 micron filter.
12. Organic Acids - (See Sugars procedure for instrument used.) An HPX-87H column (Bio-Rad) was used. Samples were treated as for sugar analysis, but with an additional clean-up step: an aliquot was passed through a Bond-Elut PSA column, rinsed with methanol and with water, and then eluted with 2N HCl.
13. Pectin - Samples were analyzed in the following manner. Sugars were removed and the samples defatted as per Southgate (1969). Pectin was extracted by pectinase following McCready and McComb (1952). The uronic acids

were determined colorimetrically using the method of Blumenkrantz and Asboe-Hansen (1973).

14. Protein - Samples were analyzed using the Kjeldahl method 2.055 - 2.057 (AOAC, 1984).
15. Lipid - Samples were analyzed using the Chloroform-Methanol Extraction method 43.276-43.277 (AOAC, 1984).
16. Molecular Weight Distribution of Pectins and Proteins (See Sugars procedure for instrument used.) TSK-40 and TSK-60 columns (Bio-Rad) were used. Samples of pectin extract or tomato fractions were diluted in mobile phase to yield about 0.1% as pectin. Mobile phase was acetic acid-sodium sulfate (~pH 4) to neutralize acid groups on pectins to negate ionic interactions on the column so separation would be by size only.

PROCEDURES AND RESULTS

Phase I

Studies were performed with commercially-prepared tomato puree (8-14% NTSS) because of its availability year-round, and because its viscosity can be easily measured by the Bostwick procedure which is commonly used in the tomato processing industry.

Separation and Recombination Studies

Tomato puree (~10% NTSS) was separated into two fractions (pulp and serum) by three methods:

centrifugation

conventional vacuum filtration

crossflow microporous filtration (or microfiltration)

Each of the resulting fractions, plus the original puree, were then characterized as described in the analytical procedures section.

Additional separation studies examined in a greater detail the microfiltration separation technique.

Recombined tomato puree was then prepared from the fractions produced by the three separation techniques utilizing three recombination methods:

- static (in-line) mixing

- stomacher blending

- homogenizing (or Polytron mixing)

These recombined purees were then evaluated, especially for viscosity.

Additional recombination studies examined the effect of the stomacher and homogenizer on the puree without prior separation.

In addition some studies examined the effect of microfiltration and homogenization upon tomato puree cells.

Separation and Characterization. Table 2 gives a brief comparison of the three methods used and the separation obtained. Table 3 gives results of the characterization of the original purees and the fractions obtained upon separation. Fig. 5 shows the HPLC output for organic acids of three samples and a standard. In addition the sub-contractor performed characterization studies on tomato puree (Table 4) and diluted tomato pastes (Table 5). The three separation methods are described in more detail following the tables.

TABLE 2. SEPARATION METHODS

	Centrifuge	Conventional Filter	Microporous Filter
Method	12,000 rpm (19,000 $\pm$ 4,000 x gravity Decant 2X	38 micron 23" vacuum 30 min.	0.3ft ² 1.0 micron 130psi, 64°C 5.3 gal/min
%NTSS Original	10.8	10.8	11.9
%Separation (by wt.)			
% Mass in Pulp	22	60	60
% Mass in Serum	78	40	40

TABLE 3. CHARACTERIZATION OF PUREE AND SEPARATED FRACTIONS

I.D.	Sample Number	NTSS, %	Protein, %	Pectin, %	Lipid, %	Sucrose, %	Glucose, %	Fructose, %	Citric Acid, %	Malic Acid, %	Pyroglu- tamic Acid %
<u>Puree</u>											
Centrifugation	1	10.8	1.79	0.75	0.20	<0.1	2.76	3.07	0.80	0.06	0.04
Conv.Filtration											
Microfiltration	9	11.9	2.00	0.86	0.32	<0.1	3.23	3.55	0.88	0.08	0.02
<u>Serum</u>											
Centrifugation	5	10.8	1.43	0.60	0.03	<0.1	2.60	3.11	0.92	0.06	0.02
Conv. Filtration	2	10.8	1.50	0.44	0.06	<0.1	3.11	3.54	0.88	0.06	0.02
Microfiltration	7	11.6	1.49	<0.1	0.02	<0.1	3.73	4.10	1.04	0.08	0.02
<u>Pulp</u>											
Centrifugation	6	NA	2.90	1.17	0.66	<0.1	2.67	3.07	0.88	0.09	0.05
Conv.Filtration	3	NA	1.93	0.84	0.29	<0.1	2.72	3.16	0.96	0.13	0.08
Microfiltration	8	NA	2.54	1.39	0.62	<0.1	3.14	3.51	1.00	0.12	0.03

Figure 5 - High performance liquid chromatogram of organic acids for three samples and a standard. The samples: original puree, microfiltered pulp, and microfiltered serum contained respectively: 0.91, 0.94, and 0.86% citric acid.

TABLE 4. CHARACTERIZATION OF PUREE

Juice Concentrate	Trial 1	Trial 2	Trial 3	x	s*
Bostwick Consistency(cm)	5.3	5.7	5.5	5.5	0.2
Brix (°)	13.0	13.1	13.3	13.1	0.15
Libby's Efflux Viscosity (catsup opening)(sec)	719	650	646	672	41
Precipitate wt ratio(%)	36.75	35.80	36.24	36.26	0.48
Total solids(%)	14.16	14.05	14.08	14.10	0.06
Ash	1.07	1.20	1.27	1.18	0.10
Hunter Color					
L	25.5	25.7	25.5	25.5	0.12
a	26.9	26.7	26.8	26.3	0.1
b	13.1	13.1	13.0	13.1	0.06
Titratable Acidity(ml)	11.4	11.3	11.5	11.4	0.1
pH	4.3	4.4	4.4	4.4	0.06
Total Carbohydrate(mg/ml)	52	68	NA	60	11
Serum					
Ostwald Serum Viscosity(sec)	93.14	94.7	94.7	94.2	0.9
Insoluble Solids					
Cell Wall Fractionation (gm/1gm solids)					
Ionically ass. pectin			0.1732		
Covalently bound pectin			0.1413		
Hemicellulose			0.1786		
Cellulose			0.4984		
% Recovery			99.15%		

* s = standard deviation

TABLE 5. CHARACTERIZATION OF DILUTED PASTES

(Same variety, harvest day, and identical treatment except hot vs. cold break; diluted to 12.1° Brix before analysis; reported values are averages for at least triplicate determination.)

<u>Juice</u>	<u>Cold Break</u>	<u>Hot Break</u>
Bostwick Consistency (cm. in 30 sec.)	11.7	3.4
Libby's Efflux Viscosity (sec)	9.1	1017.5
Precipitate Wright Ratio (gm)	20.12	33.70
Total solids (%)	11.74	11.94
Ash (%)	1.03	.96
Hunter Color	L=24.5 a=29.8 b=13.3 a/b=2.24	L=25.8 d=26.2 b=13.6 a/b=1.93
Titratable Acidity (ml)	13.8	9.6
pH	4.17	4.25
Total Carbohydrates (mg glucose/ml)	290	310
Protein (%, by Kjeldahl)	(wet basis) .31 (dry basis) 2.65	.48 3.98
Pectin (ug galacturonic acid/mg)	20.22	22.25
<u>Serum</u>		
Ostwald Serum Viscosity (sec)	14.0	373.5
pH	4.40	4.32
<u>Insoluble Solids</u>		
Cell Wall Fractionation (gm/lgm solids)		
Ionically Associated Pectin		.180
Covalently Bound Pectin		.158
Hemicellulose		.154
Cellulose		.378
% Recovery		88%

Centrifugation

Materials:

Tomato puree - 2 cans (425 gms @) (10.8% NTSS)
Laboratory centrifuge and cups

Method:

- 1) Place puree in centrifuge cups (150 gms/cup).
- 2) Centrifuge at 12,000 rpm, 30 min (19,000 $\pm$ 4000 x gravity).
- 3) Decant clear serum for characterization. Decant semi-clear serum into other centrifuge cups. Place pulp in container for characterization.
- 4) Repeat Steps 2 and 3 twice; on final decanting, all liquid goes to serum container.

Conventional filtration

Materials:

Tomato puree - 4 cans (425 gms @) (10.8% NTSS)
Buchner funnels - 186 mm and 155 mm i.d.
Vacuum filtration manifold
Milk filter discs to fit Buchner funnels
(38 micron pore size)

Method:

- 1) Filter 1/10 volume of puree (composited) in each funnel for 30 min. Repeat until all puree is filtered.
- 2) Adjust NTSS of filtrate to level of original puree with water (to make up for evaporation).

Microporous filtration

Materials:

Tomato puree (11,000 gms) (11.9% NTSS)
1 micron microfilter (Norton)
portable pumping unit (CARRE)

Method:

- 1) Heat puree to 60°C.
- 2) Run puree through microfilter pumping unit in batch (concentration) mode with permeate leaving the system, concentrate returning to the feed tank. Operating conditions: 130 psi (9 bar), 147°F (64°C), 5.3 gal/min (20 L/min). Length of test was 1.9-hr. (The flux obtained during this test was 52-48 gal/ft² day (88-81 L/m² hr)).
- 3) Water was not added to the serum to make up for the small amount of evaporation during the test.

Of the three separation methods studied centrifugation provided the most complete separation on a weight basis, microporous filtration provided the second most complete separation by weight. (See Additional Microfiltration Studies, below.) Micro-porous filtration provided the most complete separation of pectin (Table 2).

Additional Microfiltration Studies. Flux (or permeation rate) was studied versus pump flow rate (1.5 - 3 gal/min, 5.7 - 11.4 L/min) and average operating pressure (20-140 psi, 1.4 - 9.7 bar). The test was run at 73-86°F (23-30°C) in recirculation mode. Results are shown in Fig. 6. Increasing flow rate and pressure resulted in increasing flux rate.

Figure 6 - Microporous filter (0.2 micron)--Flux versus pressure and flow.

Flux was also studied versus temperature (81 and 140°F, 27 and 60°C) and average pressure (20-140psi, 1.4 - 9.7 bar). The test was run at 2 gal/min (7.6 L/min). Results are shown in Fig. 7. Increasing temperature and pressure resulted in increasing flux.

Figure 7 - Microporous filter (0.2 micron)--Flux versus pressure and temperature.

One test was run for 48 hrs in recirculation mode. Operating pressure was 116 psi (8 bar), temperature was 111-156°F (44-69°C), and flow rate was ~5 gal/min (19 L/min). Initially the flux was 54 gal/ft²day (92 L/m²hr). Mid-way through the test the feed was changed to fresh juice and the flux was 51 gal/ft²day (87 L/m²hr). Results are shown in Fig. 8.

Microfiltration of puree was also studied in batch mode. The inlet operating pressure was 88-120psi (6-8 bar), temperature was 115-149°F (46-65°C), and flow rate was 4.2 gal/min (16 L/min). The approximate separation by weight obtained was: 52% pulp, 48% serum. The flux varied from 54 to 83 gal/ft²day (92 - 141 L/m²hr) during the 45 min test.

Figure 8 - Microporous filter (1.0 micron)—Flux, temperature, and % solids of permeate versus operating time.

Recombination and Evaluation. The three recombination methods are described below, followed by the results of the recombination testing.

Static Mixer.

Both fractions (serum and pulp) were placed in a feed tank and the peristaltic pump was used at 0.5 gal/min to pump the feed through the static mixer. When recombining pulp and serum separated by centrifugation, the recombined fluid passed through the static mixer three times. The fluid recombined from the fractions produced by conventional filtration and microfiltration passed through the static mixer twice in both cases.

Results of the recombinations are given in Table 6. Separation by centrifugation or conventional filtration, followed by recombination by static mixing slightly decreased viscosity (+0.22cm and +0.18cm, +4.2% and +3.8%, respectively). Separation by microporous filtration, followed by recombination by static mixing, slightly increased viscosity (-0.17cm,-3.2%).

Stomacher.

Both fractions (serum and pulp) were placed in Whirl-pak bags and then in a protective bag and placed in the stomacher compartment. When recombining the centrifuge-separated serum and pulp, the stomacher was run for 2 min; with the conventional filter-separated fractions, it was run and samples tested after 0.5, 1, 3, and 5 min. Since the conventional filter technique gave a less sharp separation than the centrifuge, recombining with the stomacher was adequate after less time. Since other testing with the stomacher (see Additional Recombination Studies) showed

viscosity deterioration with its use, this technique was not used to recombine the microporous filter-separated fractions.

Results of the recombinations are given in Table 7. After separation by centrifuge, recombination by stomacher resulted in loss in viscosity (+1.1cm to 0.6cm, +26.5% to +10.9%); recombination by stomacher after conventional filtration resulted in no significant change in viscosity.

TABLE 6. RESULTS OF STATIC MIXER RECOMBINATION WITH THREE SEPARATION TECHNIQUES

	Centrifuge	Conventional Filter	Microporous Filter
% NTSS			
Original Puree	9.2	10.8	9.2
Recombined Puree	9.2	10.8	9.15
Viscosity (Bostwick, cm.)			
Original Puree	5.28	4.75	5.27
Recombined Puree	5.5	4.93	5.1
Viscosity Change—Gain or Loss(cm) (%)	Loss (+0.22 cm) (4.2%)	Loss (+0.18 cm) (+3.8%)	Gain (-0.17 cm) (-3.2%)
Separation Conditions	12,000 rpm 19,000 $\pm$ 4000 x gravity 30 min. Decant 1x	38 micron 30 min. 23" vacuum	0.2 micron 90-110 psi (6.2-7.6 bar) 104-140°F (40-60°C) 3.6-4.6 gal/min(13.6-17.4 L/min) 0.75 hr 57-77 gal/ft ² day (97-131 L/m ² hr)

TABLE 7. RESULTS OF STOMACHER RECOMBINATION WITH TWO SEPARATION TECHNIQUES

		Centrifuge		Conventional Filter
%NTSS				
Original Puree		12.9	8.9	10.8
Recombined Puree		12.9	8.9	10.8
Viscosity (Bostwick, cm.)				
Original Puree		4.15	5.5	4.95
Recombined Puree at:				
0.5 min				4.975
1 min				4.825
2 min		5.25	6.1	
3 min				5.2
5 min				5.2
Viscosity Change at:				
0.5 min				No Change (+0.025cm)[<1%]
1 min				Gain (-0.125cm)[-2.5%]
2 min		Loss (+1.1cm)[+26.5%]	Loss (+0.6cm)[+10.9%]	
3 min				Loss (+0.25cm)[+5.1%]
5 min				Loss (+0.25cm)[+5.1%]
Separation Conditions				
19,000 ± 4000 x gravity		13,000 rpm	13,000 rpm	38 micron
30 min.		30 min.	30 min.	30 min.
Decant 1x		Decant 1x	Decant 1x	23" vacuum

Homogenizer.

The homogenizer was used for recombining by placing serum and pulp (~300 gms) into a 400 ml beaker and homogenizing for 0.5 to 7 minutes (depending upon the test) while moving the beaker to distribute the homogenizing action throughout the beaker. In one study, the recombined puree was heated before the viscosity was measured.

Results of the recombinations are given in Table 8. After separation by all three methods, recombination by homogenizer for 5 min or longer resulted in viscosity gain (-0.43cm to -1.73cm, -8.9% to -33.0%); recombination by homogenizer for 3 min also caused a viscosity gain for all but the centrifuge separation method.

In addition the sub-contractor duplicated a centrifugation study with tomato puree adjusted to 11.6° Brix. Samples of the original puree and recombined centrifuged puree were characterized (Table 9).

All data points given in Table 9 are the average of duplicate or triplicate determination. Bostwick consistency, serum viscosity and precipitate weight ratio measurements indicate an increase in viscosity due to homogenization. All chemical measurements suggested that no real changes in chemical composition occur due to homogenization. The increase in viscosity that results from homogenization of tomato juice is hypothesized to be due to changes in physical properties of the chemical components.

TABLE 8. RESULTS OF HOMOGENIZER RECOMBINATION WITH THREE SEPARATION TECHNIQUES

			Centrifuge	Conventional Filter	Microporous Filter	
%NTSS						
Original Puree	9.0	8.8, 9.8	10.2	9.6	9.2	9.2
Recombined Puree	9.0	8.8, 9.8	10.2	9.6	9.15	NA
Viscosity (Bostwick, cm.)						
Original Puree	5.03	5.4, 5.0	4.85	5.25	5.27	5.27
Recombined Puree at:						
0.5 min	6.16	6.2*, 6.0*				
3 min			5.12	4.37	4.8	4.57
5 min			4.42			
7 min			3.93	3.52	4.75	4.37
Viscosity Change at:						
0.5 min	Loss(+1.13cm) [+22.5%]	*Loss,*Loss, (+0.8,+1.0cm) [+14.8%,+20%]				
3 min			Loss(+0.27cm) [+5.6%]	Gain(-0.88cm) [-16.8%]	Gain(-0.47cm) [-8.9%]	Gain(-0.7cm) [-13.3%]
5 min			Gain(-0.43cm) [-8.9%]			
7 min			Gain(-0.92cm) [-19.0%]	Gain(-1.73cm) [-33.0%]	Gain(-0.52cm) [-9.9%]	Gain(-0.9cm) [-17.1%]
Separation Conditions						
	13,000 rpm** 30 min	12,000 rpm** 30 min	12,000 rpm** 30 min	38 micron 30 min	0.2 micron 90-110 psi (6.2-7.6 bar)	0.2 micron 88-120 psi (6.1-8.3 bar)
	Decant 1x	Decant 1x	Decant 1x	23" vacuum	104-140°F (40-60°C)	115-149°F (46-65°C)
					3.6-4.6 gal/min 13.6-17.4 L/min)	4.2 gal/min (15.9 L/min)
					0.75 hr	0.75 hr
					57-77 gal/ft² day (97-131 L/m² hr)	54-83 gal/ft² day (92-141 L/m² hr)

*Recombined puree heated to 82°C for 35 min, before Bostwick measurement.

**19,000 ± 4000 x gravity

TABLE 9. CONSISTENCY MEASUREMENTS AND CHEMICAL ANALYSES OF
HOMOGENIZED TOMATO PUREE SAMPLES

Characteristic	Homogenization Time (Min)			
	0	3	5	7
Bostwick Consistency (cm)	5.5	5.5	5.3	5.1
Serum Viscosity (sec)	71	75	75	75
Precipitate Wt. Ratio (%)	32.05	36.15	37.05	37.40
Total Solids (%)	12.1	12.0	12.1	12.2
Ash (%)	0.9	0.9	0.9	0.8
pH	4.33	4.32	4.34	4.32
Titratable Acidity (ml)	13.3	13.9	11.5	14.8
*Total Carbohydrates (gm/gm)	0.329	0.311	0.316	0.325
*Total Pectin (gm/gm)	0.151	0.142	0.157	0.158
*Total Protein (Kjeldahl)(gm/gm)	0.158	0.163	0.159	0.156

*Values expressed on a wet weight basis.

Additional Recombination Studies. Studies were also undertaken examining the effect of the recombination technique alone upon the viscosity of the puree.

Stomacher.

The effect of stomaching on puree viscosity (2 min) was examined. Three sets of samples were tested. In each case the Bostwick viscosity decreased (0.8, 0.83, 0.68 cm increase) [+14.5, +15.4, +12.6%].

In addition the effect on puree viscosity of stomaching for 2 min and letting the puree sit before measuring viscosity was examined. Six cans (425 gms/can) of puree at 8.9% NTSS were used. The original puree had a Bostwick viscosity of 5.47 cm. Table 10 summarizes results. The puree viscosity did not improve with time.

TABLE 10. CHANGE IN VISCOSITY WITH STOMACHING 2 MINUTES AND SITTING

Time (min)	Viscosity Change from Original Puree
0	Loss (+0.65cm)[+11.9%]
36	Loss (+0.45cm)[+ 8.2%]
72	Loss (+0.55cm)[+10.1%]
132	Loss (+0.70cm)[+12.8%]

The effect of stomaching time on puree viscosity was also examined. Four cans (425 gms/can) of puree at 10.8% NTSS were used. The Bostwick of the original puree was measured. The rest of the puree was divided into 135 ml portions and pairs of portions were stomached for 1, 2, 4 and 8 min. A Bostwick measurement was made on each portion. Viscosity decreased with stomacher mixing time (Fig. 9).

Homoqenizer.

The effect of homogenization time upon puree viscosity was examined. Four cans (425 gms/can) of puree at 10.2% NTSS were used. The Bostwick of the original puree was measured. The rest of the puree was divided into 270 ml portions. One portion was homogenized for 1 min at setting no. 6 and Bostwick viscosity was measured. This process was repeated for homogenization times of 3, 5 and 7 min. The viscosity decreased initially and then increased above the original value at 5 and 7 min (Fig. 10).

The effect of homogenization time on the viscosity of puree was studied using a different puree (containing 9.2% NTSS) and Bostwick was measured after 3, 5 and 7 min of homogenization. The viscosity increased at 3 min with no further increase with longer mixing time (Fig. 11).

Figure 9 - Bostwick viscosity (cm) versus stomacher mixing time (min) for unseparated puree (10.8% NTSS).

Figure 10 - Bostwick viscosity (cm) versus homogenizing time (min) on unseparated puree (10.2% NTSS).

Figure 11 - Bostwick viscosity (cm) versus homogenizing time (min) on unseparated puree (9.2% NTSS).

Additional Studies. Several studies were made of the effect of the microfiltration separation technique and the homogenization recombination technique on intact cells in puree.

Samples that had been separated by microfiltration were recombined by homogenization. The microfiltration caused a 52% broken cell count after 49 min. However, in another study, 24 min of microfiltration caused a 59% broken cell count; an additional 23 min of operation increased the broken cell count to 69%. When recombined by homogenization there was only a slight increase in broken cells (to 57% in the first study; to 77% from 69% in the second study). Viscosity was restored upon recombination by homogenization.

Examination of cells after 3, 5 and 7 min of homogenization (without prior separation) showed no increase in the number of broken cells.

Separation Technique Selection. Centrifugation separated the most serum, which is highly desirable. Centrifuges are readily available and familiar to the food industry. Consequently, this was the separation technique chosen for further study.

The microporous filter in conjunction with recombination by static mixer is a viable alternative to the centrifuge. When recombining the two fractions produced by centrifugation, only a polytron homogenizer restored the viscosity to its original value. Homogenization would be an expensive recombination technique at the commercial scale. Microporous filters provided a good separation and the two fractions were recombined with a static mixer with full restoration of viscosity. Microfilters and static mixers are commercially available and may offer an economical alternative to centrifugation and homogenization. Further study of microfiltration and static mixing is warranted.

Pulp Rinsing, Serum Concentration and Recombination Studies

Tomato puree (~10% NTSS) was separated into two fractions by centrifugation. The pulp obtained was rinsed and the rinse water added to the serum. The rinsing step is designed to remove components from the pulp which might be damaged in drying. The rinse/serum fraction was then concentrated to 30, 50, and 70% NTSS by evaporation. These fractions were all characterized.

In addition, tomato serum was concentrated by hyperfiltration to approximately 20% NTSS. This fraction could be further concentrated by evaporation.

The two fractions obtained by centrifugation, rinsing, and rinse/serum concentration were recombined to reproduce the original puree solids level and evaluated.

Pulp Rinsing. Five cans (2900 gms/can) of puree were used for this step. The puree was centrifuged at 9500 rpm ($12,000 \pm 2700$ x gravity) for 40 min. Tomato serum was decanted and volume measured. Pulp was weighed for separation data. Water equal to approximately one-half the serum volume was stirred into the pulp (rinsing step). This rinsed pulp was recentrifuged as above, and rinse water decanted. The NTSS of the rinse water was measured and the rinsing repeated until the NTSS measured <1%. This process required 5 to 6 rinsing steps. The rinse water and serum were combined to produce a rinse/serum fraction.

The separation obtained on a weight basis from centrifuging and rinsing was 21.7% pulp, 78.3% serum. The original puree and the fractions obtained upon separation and rinsing were characterized and results are in Table 11. Samples were frozen for later use.

Serum Concentration. The rinse/serum fraction from the pulp rinsing step was concentrated to 30, 50 and 70% NTSS using a rotary evaporator (Buchi) with vacuum. The water bath temperature was 52-56°C during concentration. Batches of about 600 ml rinse/serum were concentrated to about 30 mls of 30% NTSS. Then several of these batches were combined to concentrate to 50 and 70% NTSS.

One hundred ml samples of each of the three levels were obtained. The remaining serum was concentrated to 50% NTSS and frozen. Characterization of these samples was performed and are listed in Table 11.

TABLE 11. CHARACTERIZATION OF PUREE AND RINSED AND CONCENTRATED FRACTIONS

I.D.	NTSS, %	Protein %	Pectin %	Sucrose, %	Glucose, %	Fructose, %	Citric Acid, %	Malic Acid, %	Pyroglu- tamic Acid
Puree	9.5	1.66	0.63	<0.1	2.63	2.92	NA	NA	0.17
Pulp Rinsed (11-25-85)	0.8	1.97	0.94	<0.1	0	0	0	0	0
Pulp Rinsed (No Date)	0.8	NA	1.35	NA	NA	NA	NA	NA	NA
Rinse/ Serum	5.2	0.66	<0.1	<0.1	0.81	0.91	NA	0	0.01
30% Rinse/ Serum	30 $\pm$ 3	4.22	1.44	<0.1	8.31	9.24	1.88	0.16	0.08
50% Rinse/ Serum	50 $\pm$ 4	7.50	2.49	<0.1	13.5	15.3	3.28	0.24	0.06
70% Rinse/ Serum	70 $\pm$ 5	10.40	3.51	NA	NA	NA	NA	NA	NA

Serum Concentration by Initial Hyperfiltration...

Studies were undertaken on the feasibility of using hyperfiltration to initially concentrate tomato serum prior to evaporation. Tomato serum produced by ultrafiltration and centrifugation were concentrated by hyperfilter. Fresh cold break tomato juice (~5% NTSS) was separated into serum and pulp fractions by ultrafilter (CARRE, Inc.) and fresh hot and cold break tomato juice was separated by centrifuge (Alfa-Laval). Most centrifuged serum was produced at 2000 rpm (2,200 x gravity), but one test (23 hrs.) involved serum which was recentrifuged at 12,200 rpm (maximum 20,000 x gravity).

Testing with the serum from ultrafiltration and 2000 rpm centrifugation lasted 89 hrs. Operating conditions were at 800 psi (55 bar) 104 and 140°F (40 and 60°C) in a recycle mode. The flux declined with time from 32 gal/ft²day (54 L/m²hr) to 7 gal/ft² day (12 L/m²hr). Mid-way through this test permeate was diverted from the system allowing concentration to take place to 20.9% NTSS. The flux declined from 12 gal/ft²day (20 L/m²hr) to 2 gal/ft²day (3 L/m²hr). Additional analysis of the data is necessary to predict economic feasibility.

Testing (23 hrs.) with the double centrifuged juice did not produce significantly different results. Operating conditions were the same at the higher temperature. The flux declined from 17 to 12 gal/ft²day (29 to 20 L/m²hr).

Rejection of NTSS was high during all testing. The permeate had 0.2 to <0.1% NTSS.

Recombination and Evaluation. The fractions of concentrated rinse/serum and rinsed pulp were recombined to make puree. Three studies were performed.

In the first study, the NTSS and TS of the fractions and original puree was measured to calculate the amounts to be combined with water. The calculated amounts were recombined and mixed lightly giving a product with a "juicelike" appearance. The recombined puree had NTSS of 8.8%; the original puree had NTSS of 9.5%. Consequently more rinse/serum and pulp were added to the mixture, based instead upon 50% NTSS and 50% TS in the rinse/serum, since these two analytical procedures are subject to larger experimental error when the solids are at such high levels. This mixture (No. 2) was homogenized for 3 min, followed by measurement of Bostwick and NTSS (Table 12).

A third mixture was made by adding 10 gm of rinsed pulp to Mixture No. 2 and homogenizing for 3 min. Mixture No. 4 was made by adding 10 gm of rinsed pulp to Mixture No. 3. Table 12 shows results of Bostwick and NTSS analysis. Mixture 4 Bostwick results are very close to that of the original puree. Mixture 4 also contains 21.5% pulp (by weight) which is very close to the percent pulp obtained in the separation upon centrifuging and rinsing (21.7%).

TABLE 12. ORIGINAL AND RECOMBINED PUREES

	Original				
	Puree	Mixture 1	Mixture 2	Mixture 3	Mixture 4
wt % rinse/ serum	- -	17.3	18.8	18.2	17.4
wt % pulp		13.7	15.2	17.9	21.5
Bostwick(cm)	5.5	(assumed >15)	11.4	8.3	5.8
NTSS (%)	9.5	8.8	9.5	9.6	9.3

The second study used fraction amounts close to those of Mixture No. 4 in the previous study, but with a slightly higher amount of rinse/serum, since the NTSS of Mixture No. 4 was somewhat lower than the original puree. The new mixture was homogenized for 3 min followed by measurement of Bostwick, NTSS and TS. A second batch of the mixture was made and homogenized for 7 min. Table 13 shows results. At 7 min homogenization, the Bostwick obtained was better than the original.

TABLE 13. ORIGINAL AND RECOMBINED PUREES AFTER DIFFERENT HOMOGENIZATION TIMES

	Original	Reconstituted	
	Puree	Homogenized	
		3 min	7 min
wt % rinse/serum	- -	18.8	18.8
wt % pump	- -	21.5	32.5
Bostwick(cm)	5.5	5.6	5.35
NTSS (%)	9.5	9.7	9.6
TS (%)	10.77	10.91	11.00

In the third study, the centrifugation, rinsing and concentration steps were repeated with careful monitoring of the addition and removal of water, so that a direct calculation could be made of the amount of concentrated rinse/serum (21% NTSS) needed to be used for reconstituting to the original puree solids level.

Using the weight fractions measured during the separation step as a guide, 82.99 gm rinsed pulp, 142.17 gm concentrated rinse/serum, and water to produce 350 gm of puree were combined and homogenized for 7 min. This was repeated with a second batch. Bostwick, NTSS, and TS were measured (Table 14). The samples were refrigerated for 3 days and Bostwick was remeasured. The first batch did not totally recover the viscosity, but the second batch did. Viscosity of both improved after refrigerated storage.

TABLE 14. ORIGINAL AND RECOMBINED PUREES
BEFORE AND AFTER REFRIGERATED STORAGE

	Original Puree	Recombined and Homogenized			
		Sample 1		Sample 2	
		Initial	Refrig.	Initial	Refrig.
Bostwick(cm)	5.45	6.33	5.65	5.50	5.30
NTSS(%)	9.1	9.1	-	9.2	-
TS(%)	10.81	10.84	-	10.96	-
wt % rinse/serum	-	40.6	-	40.6	-
wt % rinsed pulp	-	23.7	-	23.7	-

Tomato Maturity Study

The sub-contractor performed a study to quantitate changes in product viscosity and in protein and pectin contents as tomatoes ripen. To better control the consistency of tomato products, the factors which determine product viscosity must be known and understood. The changes in these factors during ripening and processing of the tomato must also be known. Pectic substances and proteins appear to both be important factors influencing tomato product consistency. However, these two components have not been well quantitated and characterized to examine how they relate to viscosity changes during maturation of the fruit.

Tomatoes of the variety 6203 were grown in the Purdue University horticulture greenhouses. The tomatoes were harvested at various stages of maturity, as measured by date of pollination. Tomatoes for Replication I and Replication II were harvested at stages 5 and 6 of maturity, respectively, as shown below:

<u>Stage of Maturity</u>	<u>Days after Blossoming</u>
1	34 (green)
2	41
3	43
4	45
5	47 (ripe)
6	49 (overripe)

All samples were subjected to the hot break process then analyzed for the following:

Libby's Efflux Viscosity
Serum Viscosity - Ostwald method
Precipitate Weight Ratio
Total Solids
pH
Protein Content
Pectin Content

All data points shown are the average of duplicate determinations. The percent protein in the serum (Fig. 12), Libby's efflux viscosity (Fig. 13), and serum viscosity (Fig. 14) decreased sharply between maturity stages 1 and 3. Levels were all minimized at stages 4 and 6, and increased slightly in stage 5. The pattern was similar for precipitate weight ratio (Fig. 15). While serum protein and all measures of viscosity were higher at stage 5 than at stages 4 and 6, serum pectin (Fig. 16) was minimized at stage 5. These results suggest that more than just pectin content determines viscosity. The percent protein in the serum more closely followed the changes in viscosity than did percent pectin. The pH of the juice or serum showed little change during maturation (Table 15). Hunter color changes occurred as expected during maturation (Table 16) after stage 1. Total solids in the juice generally decreased continually during maturation (Table 17).

Figure 12 - Percent protein in serum versus maturity stage (Bradford method).

Figure 13 - Libby's efflux viscosity versus maturity stage.

Figure 14 - Serum viscosity versus maturity stage.
(Ostwald method).

Figure 15 - Precipitate weight ratio versus maturity stage.

Figure 16 - Percent pectin in serum versus maturity stage
(carbazole/ H_2SO_4 method).

TABLE 15. pH OF TOMATO JUICE AND SERUM HARVESTED AT SIX STAGES OF MATURITY

Maturity Stage	Juice	pH (Replication II)
		Serum
1 (green)	4.51	4.50
2	4.35	4.33
3	4.31	4.27
4	4.28	4.28
5 (ripe)	4.36	4.34
6	4.28	4.32

TABLE 16. TOMATO JUICE HUNTER COLOR READINGS OF TOMATOES HARVESTED AT SIX STAGES OF MATURITY

Maturity Stage	Hunter Color Readings (Replication II)			
	L	a	b	a/b
1 (green)	48.8	-3.3	22.7	-.145
2	40.9	10.6	20.3	.522
3	30.7	25.6	14.1	1.82
4	28.1	28.3	13.1	2.16
5 (ripe)	27.0	28.6	12.7	2.25
6	26.1	29.8	12.7	2.35

TABLE 17. PERCENT TOTAL SOLIDS IN TOMATO JUICE ADJUSTED TO 5.5 BRUX FROM TOMATOES HARVESTED AT VARIOUS STAGES OF MATURITY

Maturity Stage	Total Solids (%) in Tomato Juice	
	Replication I	Replication II
1 (green)	6.46	6.45
2	6.29	6.21
3	6.09	6.06
4	5.92	5.95
5 (ripe)	5.96	5.91
6	---	5.85

PHASE II

Studies were performed with commercially-prepared tomato puree (9% NTSS) and with fresh tomato juice. Commercially-prepared tomato puree was used because of its year-round availability and because its viscosity can be easily measured by means of the Bostwick procedure.

Separation Study

Canned puree (320 lb) (140kg) was separated into pulp and serum fractions using a decanter centrifuge. Puree was fed to the centrifuge at ~1 gal/min (3.8L/min). Centrifuging was done at maximum rpm (~3500 x g) with slow scroll speed. Most of the pulp fraction was removed from the decanter (the centrifuge was not dismantled, so some pulp remained inside) and placed in a vat. Rinse water (~47°C) equal in weight to the serum fraction removed was added to the vat and mixed. This mixture was centrifuged as above. The rinse water leaving the centrifuge was added to the serum, producing rinse/serum. The rinsing procedure was repeated. A total of 480 lb (220kg) of water was added during the two rinse cycles, producing 710 lb (320kg) rinse/serum and 90 lb (40 kg) rinsed-pulp, thus ending with 28% of the original puree weight as rinsed-pulp. Results of analysis of samples are given in Table 18.

TABLE 18. CHARACTERIZATION OF PUREE AND FRACTIONS

Sample I.D.	TS	NTSS	Pectin	Protein	Fat	ORGANIC ACIDS		Pyro- glutam	Fruc- tose	SUGARS	
						Citric	Malic			Glu- cose	Su- crose
	%	%	%	%	%	%	%	%	%	%	%
Puree	10.7	9.1	0.65	1.63	0.26	0.75	0.13	0.25	NA	NA	NA
Rinsed Pulp	12.1	0.8	1.47	2.04	0.64	0.14	0.02	0.02	NA	NA	NA
Rinse/ serum	~4.0	4.0	0.19	0.65	<.01	0.34	0.07	0.12	NA	NA	NA
Concen. Rinse/serum	12.8	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
High- MW	5.6	~6	0.93	0.92	0.28	0.43	0.08	0.10	1.43	0.95	0.14
Low/ Int.-MW	3.5	3.5	0.02	0.53	<0.01	0.29	0.08	0.12	1.37	0.89	0.14
Concen. Low/Int.-MW	73.6	76	NA	NA	NA	NA	NA	NA	NA	NA	NA
Int.-MW	NA	~7.2	0.19	1.05	0.04	0.55	0.16	0.14	2.51	1.63	0.07
Low-MW	NA	3.2	<0.01	0.44	0.05	0.22	0.05	0.09	1.14	0.74	0.07
Sugars-1	NA	3.1	NA	NA	NA	0.073	<0.02	0.03	0.80	0.47	<0.02
Sugars-2	NA	NA	NA	NA	NA	0.104	0.116	0.053	NA	NA	NA
Organic Acids-1	NA	NA	NA	NA	NA	1.57	0.146	0.008	<0.05	<0.05	<0.05
Organic Acids-2	NA	NA	NA	NA	NA	2.70	0.139	0.011	<0.05	<0.05	<0.05

Ultrafiltration and Concentration Studies

The rinse/serum fraction was further fractionated by ultrafiltration. Some fractions obtained were concentrated.

Initial 2000-MW Membrane Ultrafiltration. In initial ultrafiltration studies, a 2000-MW membrane was chosen to fractionate the rinse/serum into a low-MW fraction and a high/intermediate-MW fraction. This approach was expected to remove a large fraction of the sugar from the rinse/serum, so that a sample suitable for molecular weight distribution analysis would be produced (Sugars interfere with the MW distribution determination).

A quantity of the rinse/serum (26.6 lb, 12.06 kg) was concentrated in batch mode for 4 hrs using GR90PP membranes (0.18m² total membrane area), at 25°C, 8 L/min, 290 psi (2 MPa). The flux decreased from 9 to 5 gal/ft²day (15 to 8 L/m²hr). Sugar rejection (as measured by NTSS) was higher than anticipated (45 to 57%); only 55 to 43% of sugars permeated. The initial rinse/serum concentration was 4% NTSS (Table 18); the resulting permeate had a concentration of 2.2 to 3.1% NTSS, and the resulting retentate (high/intermediate MW) had a concentration of 7.3% NTSS. The sugars may have formed complexes with high-molecular-weight components, thus producing a higher-than-expected sugar rejection (based upon the molecular-weight-cutoff of the membranes).

The membranes were cleaned with detergent and flux and sugar rejection were measured using a pure sugar solution (6% fructose) at 145 and 290 psi (1 and 2 MPa). The observed rejection (15 and 19% at the two pressures, respectively) was lower than that obtained with rinse/serum but higher than expected.

The retentate (obtained from batch mode runs with rinse/serum) was diafiltered in order to remove a larger fraction of sugars from the retentate. In order to do this water was added to the retentate in an amount equal to the weight of the permeate removed in the batch runs.

This solution (2.5% NTSS) was ultrafiltered in batch diafiltration mode for 4 hrs. Flux varied from 8 to 4 gal/ft²day (14 to 7 L/m²hr). Sugar rejection (by NTSS) was still high (60 to 64%). The permeate had a concentration of 1 to 1.7% NTSS and the final retentate had a concentration of 5.5% NTSS. The diafiltration procedure was repeated. Flux varied from 6 to 4 gal/ft²day (10 to 7 L/m²hr). Sugar rejection (by NTSS) was 54 to 73%. The final retentate had a concentration of 3.3% NTSS. During the two diafiltrations, permeate equal to 97% of the added water weight was removed.

The above procedure (batch run and two diafiltrations) removed about 70% of the sugars (by NTSS) from the feed (rinse/serum). The final retentate weight was 37% of the initial feed (rinse/serum) weight. The membranes were cleaned with an enzyme cleaner (Monarch DDS), which resulted in a significant water flux increase. However, a test with 6% fructose again showed only a slight decrease in rejection: 10 and 17% at 145 and 290 psi (1 and 2 MPa), respectively.

The above procedure was repeated with 28.5 lb (12.93 kg) of rinse/serum at the same operating conditions, for 7 hrs. The flux varied from 5 to 3 gal/ft²day (8 to 5 L/m²hr) over this period. Overall sugar rejection (by NTSS) was high (60 to 53%).

The 2000-MW-cutoff membrane was determined to be too tight for the initial ultrafiltration; use of 100,000-MW-cutoff membranes was therefore investigated.

Permeate Concentration.

Permeate from the initial 2000-MW-cutoff membrane ultrafiltration and two diafiltrations (40 lb, 18 kg) was concentrated using rotary evaporators at 104°F, 40°C and 27" Hg vacuum. The initial NTSS concentration was 1.5%. Twenty-six batches of this were concentrated to produce one solution with a concentration of 8.5% NTSS which was further concentrated in four batches to a concentration of 58% NTSS. The fluid color was initially light yellow; final fluid color was dark brown.

100,000-MW-Cutoff Membrane Ultrafiltration. Two types of 100,000-MW-cutoff membranes (GR40PP and PES100) were studied to fractionate the rinse/serum fraction into low-MW and high/intermediate-MW fractions. The higher MW-cutoff membranes were expected to exhibit higher flux and lower sugar rejection during fractionation of the rinse/serum fraction.

The data from four batch separation runs are summarized in Table 19. Unless otherwise indicated, all runs were made at 25°C, 8 L/min, 100 psi (0.7 MPa) with 0.108m² membrane area each of the two membrane types.

Each run (Figs. 17 and 18) started with the initial hour of run time in recycle mode to monitor the initial flux. Flux decline was time dependent as well as concentration dependent. After the first run the membranes were rinsed with water.

In the second run temperature scans were run (Table 20) following the initial recycle period (25 to 64°C) and the batch separation (25 to 62°C). At higher temperatures sugar rejection (by NTSS) was lower and the flux significantly higher. The membranes were cleaned with an enzyme detergent (Monarch DDS) following the second run.

TABLE 19. 100,000-MW-MEMBRANE ULTRAFILTRATION TESTING WITH RINSE/SERUM

Batch No.	Rinse/Serum (kg) [gal]	Separation Time (hr)	GR40PP		PES 100		Feed	NTSS% Reten-tate	Composite Permeate		Separation		Weight Retained %
			Recycle Start/End	Separat. Start/End	Recycle Start-End (L/m2hr) [gal/ft2day]	Separat/ Start-End			GR40	PES100	Sugar Permeated %	Acid Permeated %	
1	39.1 [9.8]	5.25	59/49 [35/29]	41/17 [24/10]	61/44 [36/26]	41/17 [24/10]	3.9	6.2	3.6	3.6	74	77	17
Rinse													
2	45.9 [11.5]	7	56/48 [33/28]	31/20 [18/12]	56/46 [33/27]	29/20 [17/12]	3.9	6.1	NA	NA	NA	NA	21
Enzyme Wash													
3	45.9 [11.5]	6.3	51/41 [30/24]	39/25 [23/15]	54/41 [32/24]	41/24 [24/14]	4.1	5.7	3.7	3.7	70	73	22
Rinse													
4	34.4 [8.6]	4.6	43/32 [25/19]	31/19 [18/11]	44/34 [26/20]	31/20 [18/12]	~4.1	5.4	3.8	3.8	~65	~66	28

Figure 17 - Flux versus time - 100,000-MW-cutoff membrane ultrafiltration of rinse/serum.

Figure 18 - NTSS (%) versus time - 100,000-MW-cutoff membrane ultrafiltration of rinse/serum.

TABLE 20. 100,000-MW-CUTOFF MEMBRANE ULTRAFILTRATION TEMPERATURE SCANS WITH RINSE/SERUM

Batch No.	No. of Membranes GR40/PES Inlet/Outlet	Rinse/ Serum (kg)	Scan Timing	Temp. (°C)	Flux		Reten- tate	NTSS(%) Permeate	
					GR40PP (L/m2hr) [gal/ft2day]	PES100		GR40PP	PES100
2	6/6	45.9	After Recycle	25	47[28]	46[27]	~4	3.0	3.4
				41	70[41]	65[38]	~4	3.6	3.7
				52	66[39]	61[36]	~4	3.6	3.6
				64	81[48]	79[47]	~4	3.6	3.6
			After Separation	25	22[13]	22[13]	NA	3.9	4.0
				40	27[16]	25[15]	6.0	NA	NA
				53	36[21]	34[20]	NA	4.0	4.1
				62	36[21]	39[23]	NA	4.1	4.4

The membranes were rinsed with water between the third and fourth runs. During the five runs, the rinse/serum flux declined during the run. If the membrane run lasted ~6 to 7 hrs (with rinse/serum) followed by rinsing with water, the flux of the rinse/serum returned to almost the original flux (at the beginning of that run). However, after ~16 hrs operation with rinse/serum, cleaning with a detergent was necessary in order to restore flux.

Flux was significantly higher with the 100,000-MW-cutoff membrane ultrafiltration than with the 2000-MW-cutoff membrane ultrafiltration. Sugar rejection was reduced so that without the use of diafiltration 65-74% of the sugar permeated. A slightly higher percentage of acid permeated (66-77%). The final retentate weight was 17-28% of the initial feed weight. This amount was limited mainly by the pressure drop across the membranes (Fig. 19) and the maximum operating pressure of the PES100 membranes (100 psi, 0.7 MPa). The performances of both membranes were similar in flux and rejection, although the PES100 membranes were on the outlet end of the ultrafiltration module and thus experienced lower pressures for most of the testing.

Permeate Concentration.

Permeate from the five batch separations made with the 100,000-MW-cutoff membranes (40 lb, 18 kg) was composited and concentrated using rotary evaporators at 158°F, 70°C and 22" Hg vacuum. The initial NTSS concentration was 3.5%. Thirty-one batches of this were concentrated to produce one solution with a concentration of 17.6% NTSS which was further concentrated in six batches to a concentration of 76% NTSS. Total evaporation time was 4-8 hrs. The fluid color was initially light yellow; final fluid color was dark brown.

Figure 19 - Pressure drop versus time - 100,000-MW-cutoff membrane ultrafiltration of rinse/serum.

2000-MW-Cutoff Membrane Ultrafiltration. Once the rinse/serum was fractionated into high- and low/intermediate-MW fractions, ultrafiltration membranes with a 2000-MW-cutoff were used to fractionate the low/intermediate-MW fraction into intermediate- and low-MW fractions.

New membranes of the same type used in the "Initial 2000-MW-Cut-off Membrane Ultrafiltration" (GR90PP) were obtained. The data from three batch separations are summarized in Table 21. Unless otherwise indicated, all runs were made at $23 \pm 2^\circ\text{C}$, 8 L/min flow rate, 290 psi (2 MPa) with 0.18m^2 total membrane area. The first and third batches consisted of an initial run in batch mode, followed by continuous mode operation. The second batch only ran in batch mode, because the retentate was lost at the end of the run. The membranes were rinsed with water after each of the three batches.

Flux ($26\text{--}14\text{ L/m}^2\text{hr}$, $16\text{--}8\text{ gal/ft}^2\text{day}$) was lower than the flux with 100,000-MW-cutoff membranes with rinse/serum ($61\text{--}17\text{ L/m}^2\text{hr}$, $36\text{--}10\text{ gal/ft}^2\text{day}$), but higher than the flux with 2000-MW-cutoff membranes with rinse/serum ($15\text{--}5\text{ L/m}^2\text{hr}$, $9\text{--}3\text{ gal/ft}^2\text{day}$). ~78% of the sugar (by NTSS) permeated in batches 1 and 3. The final retentate (intermediate-MW fraction) weight was ~11% of the initial feed (low/intermediate-MW fraction) weight showing good separation.

TABLE 21. 2000-MW-CUTOFF MEMBRANE ULTRAFILTRATION TESTING WITH LOW/INT.-MW FRACTION

Batch No.	Mode B/C ^a	Low/Int-MW (kg) [gal]	Recyle Time (hr)	Separation Time (hr)	Flux		Feed ^b	NTSS (%) Retentate	Composite Permeate
					Recycle Start/End (L/m ² hr) [gal/ft ² day]	Separat. Start/End (L/m ² hr) [gal/ft ² day]			
1	B	16.8 [4.2]	1	4.1	26/24 [16/14]	20/16 [12/10]	3.5	10.1	2.7
	C	16.4 [4.1]	0.5	6.2	15/18 [9/11]	14/17 [8/10]	7.2/ ^c 3.6	6.6	3.6
Rinse									
2	B	25.8 [6.5]	1	4.5	19/19 [11/11]	20/14 [12/9]	3.6	5.2	NA
3	B	22.4 [5.6]	0	7.0	No Recycle —	15/14 [9/8]	3.7	7.9	2.7
Rinse									
	C	10.5 [2.6]	0	6.1	No Recycle —	11/9 [6/5]	7.8/ ^c 3.6	7.6	3.7
Enzyme Wash									

^aB = Batch
C = Continuous

^bContinuous mode feed consisted of retentate from batch mode plus Low/Int-MW fraction.

^cBatch No. 1 continuous mode feed (retentate from batch mode) was diluted with additional Low/Int-MW fraction.

Ion Exchange Studies

The low MW- fraction was available for fractionation by ion exchange into sugars and organic acids. The purpose was to examine whether certain components (possibly sugars and/or organic acids) might be eliminated at the tomato processing plant, thus saving on containerization and transportation costs. These component(s) could then be obtained from local sources and added back at the point of remanufacture.

Initial Anion Resin Testing. Initially, the anion resins were studied with a model fluid. The testing with the model fluid and with the low-MW fraction involved the sequence of procedures below:

Feed Cycle - Feed entered at the top of the resin bed and passed downflow through the column exiting at the bottom. Flow rate was 20-25 ml/min. Column effluent was measured periodically for pH, conductivity, fructose and acidity. The feed cycle was terminated when citric acid content exceeded 0.2%.

Sweeten-off Backwash - Water (250ml) entered at the bottom of the resin bed and passed upflow through the column exiting at the top. This procedure was followed to purge the column of the feed solution.

Regeneration - Sodium hydroxide (4%) solution (150 ml) entered at the top of the resin bed and passed downflow through the column with a 45 min contact time exiting at the bottom. The effluent was called NaOH regenerant.

NaOH-off Backwash - Water (600ml) entered at the bottom of the resin bed and passed upflow through the column exiting at the top.

Final Rinse - Water entered at the top of the resin bed and passed downflow through the column until the pH of the effluent was less than 8.3. (An exception was made with the SM-73 resin. Rinsing was so slow that the final rinse was discontinued after several liters of water had passed through; the pH was ~10.)

A model system fluid (4% fructose [by NTSS], 0.4% citric acid [by titratable acidity], pH 2.5, conductivity = 1100 us/cm) was used as feed for testing. Two weak base anion resins (IWT SM-73 and A-399b) were used. Each column had 100 ml bed volume.

The two resins successfully removed citric acid from the model fluid (as measured by titratable acidity). The apparent capacity of the resins (in gm anhydrous citric acid removed from the feed per 100 ml bed volume, calculated from the volume of feed solution treated before the effluent reached 0.025% citric acid) is below:

Run No.	Flow (ml/min)	Apparent Capacity (gm citric acid removed/ bed volume [100ml])	
		SM-73	A-399b
1	20	6.5	NA
2	24	4.8	10.3
3	20	5.9	11.8

The A-399b resin had about twice the capacity of the SM-73 resin.

Separation of Low-MW Fraction. The feed for this testing was the low-MW fraction (3.2% NTSS, 0.223% anhydrous citric acid, pH ~4.1, conductivity = 3650 us/cm). SM-73 and A-399b were used. Each column had 100ml bed volume.

Both resins removed organic acids from the low-MW fraction (as measured by titratable acidity). The apparent capacity of the resins calculated from the volume of feed solution treated before the effluent reached 0.03 to 0.04% citric acid) is below:

<u>Resins</u>	<u>Flow</u> <u>(ml/min)</u>	<u>Apparent Capacity</u> (gm citric acid removed/ <u>bed volume [100ml])</u>
SM-73	18 _± 13	~7.5
A-399b	18 _± 9	~6.6

The SM-73 resin's capacity with the low-MW fraction was similar to that with the model system fluid. The conductivity of the low-MW fraction was three times as high as the conductivity of the model system fluid.

The A-399b resin's capacity with the low-MW fraction was lower than with the model system fluid. This resin permanently took on a yellow/grey color from the feed.

The citric acid was left in salt form, having been removed by the NaOH. These organic acid salts were then converted to organic acids by use of a cation resin.

Initial Cation Resin Testing. A model fluid was used for initial testing of the strong acid cation exchange resin (IWT C-391b). A formula for the model fluid was calculated from the theoretical concentration of sodium citrate in the NaOH regenerant produced with 0.22% anhydrous citric acid in the feed (low-MW fraction) to the anion resin; and the theoretical concentration of NaOH in the NaOH regenerant based upon that fluid's pH. This resulted in a model fluid of 6% sodium citrate and 0.4% NaOH. The pH of this solution was ~12.6, conductivity ~ 40,000 us/cm. The column had a bed volume of 100ml. The testing with the model fluid and with the NaOH regenerant (from the anion exchange of the low-MW fraction) involved the sequence of procedures below:

Feed Cycle - This procedure was the same as in anion resin testing. Column effluent was measured periodically for pH, conductivity, acidity. The feed cycle was terminated when pH reached 12.

Sweeten-off Backwash - See anion resin testing procedure.

Regeneration - Hydrochloric acid (7%) solution (150 ml) entered at the top of the resin bed and passed downflow through the column with a 30 min. contact time.

HCl-off Backwash - This procedure was the same as the NaOH-off Backwash in anion resin testing.

Final Rinse - This procedure was the same as in anion resin testing. The rinsing was discontinued when the pH of the effluent was greater than or equal to 4.3.

The resin was effective in converting the sodium citrate to citric acid. The apparent capacity of the resin is below (defined as the gm citric acid produced per 100 ml bed volume;

calculated from the volume of feed solution treated before the effluent dropped below ~0.1% anhydrous citric acid):

<u>Run No.</u>	<u>Flow (ml/min)</u>	<u>Apparent Capacity (gm citric acid produced/ bed volume [100ml])</u>
1	24	~4.5
2	20	~3.8

There appeared to be a decrease in capacity from the first run to the second.

Conversion of Organic Acid Salts to Acids. The first feed was the NaOH regenerant from the anion exchange of the low-MW fraction with the A-399b resin (pH ~12.6, conductivity = 52,500 us/cm, ~300 gm). pH and acidity of the effluent were monitored (Figure 20 and Table 22). The column was regenerated.

The next feed run through the column was the Sweeten-off Backwash from the above cation exchange. In addition effluent with pH ≥ 2 , from the above cation exchange, was added as feed. When this feed source was depleted, the column flow was stopped and the NaOH-off Backwash from the A-399b resin (~670ml) was added as feed. pH and acidity of the effluent were monitored (Figure 21 and Table 22). The column was regenerated.

Part of the Final Rinse (~50ml) from the A-399b anion resin was then used as feed. ~200ml water was added to wash out all the feed. The pH of the effluent ranged from 2.8 to 4.65; acidity ranged from 0.014 to 0.001% anhydrous citric acid. This (Final Rinse) effluent did not contribute significantly to the totality of organic acids.

The above procedure was repeated using the solutions produced from the anion exchange of the low-MW fraction with the SM-73 resin. pH and acidity of the effluent were monitored (Figs 22 and 23 and Table 22).

Figure 20 - pH vs. throughput for cation resin C-391b using NaOH regenerant from anion resin A-399b as feed.

Figure 21 - pH vs. throughput for cation resin C-391b using effluent $\geq$ pH 2 (from Figure 20) plus Sweeten-off Backwash of resin in Figure 20; second feed is NaOH-off Backwash from anion resin A-399b.

TABLE 22. ACIDITY OF EFFLUENTS FROM CATION EXCHANGE WITH
IWT C-391b OF ORGANIC ACID SALTS SAMPLES

Sample No.	Feed I.D.	Origin (Anion Exchange Resin)	Anhydrous Citric Acid (%)	pH	Quantity (gm)
1	NaOH Regenerant	A-399b	1.21	1.6	220
2	NaOH Regen. Effluent >pH2 + Sweeten-off from Sample 1	A-399b	0.46	1.9	335
3	NaOH-off Backwash	A-399b	0.11	2.3	600
5	NaOH Regenerant	SM-73	0.94	1.45	290
6	NaOH Regen. Effluent >pH2 + Sweeten-off from Sample 5	SM-73	0.37	1.7	320
7	NaOH-off Backwash	SM-73	0.19	2.35	670

Figure 22 - pH vs. throughput for cation resin C-391b using NaOH regenerant from anion resin SM-73 as feed.

Figure 23 - pH vs. throughput for cation resin C-391b using effluent >pH 2 (in Figure 22) plus Sweeten-off Backwash of resin in Figure 22; second feed is NaOH-off Backwash from anion resin SM-73.

Because some of the effluent was sent back through the column during the runs, the determination of the resin's apparent capacity was obscured. Approximations are made below:

<u>Feed</u> <u>Solutions</u> <u>From</u>	<u>Flow</u> <u>(ml/min)</u>	<u>Apparent Capacity</u> <u>(gm citric acid produced/</u> <u>bed volume [100ml])</u>
A-399b	22	2.6 - 3.0
SM-73	22	2.75 - 3.0

These capacities were lower than with the model system, but the capacity did not decrease significantly from the first run to the second run.

Mass Balance

An attempt has been made to calculate the mass balance of the above separation and ultrafiltration studies. Table 23 summarizes these calculations. As can be seen, after the first ultrafiltration step (100,000-MW cutoff), all of the total solids, sugars, and organic acids from the rinse/serum (+2.6%) were recovered. After the second ultrafiltration step (2000-MW cutoff), 95-98% of the total solids and sugars, and 87% of the organic acids of the rinse/serum were recovered.

TABLE 23. MASS BALANCE

Fraction	Weight gm	TS %	Sugar %	Acid %	TS gm	Sugar gm	Acid gm	TS %	Sugar Mass Balance	Acid
Puree	400	10.7	NA	1.13	42.80	NA	4.52	100	(% of Puree) NA	100
Water	600	0	0	0	0	0	0			
Rinsed-Pulp	62	12.5	NA	0.18	7.75	NA	0.11			
Rinse/Serum	938	3.9	2.43	0.53	36.58	22.79	4.97			
SubTotal	1000				44.33	NA	5.08	103.5	(% of Puree) NA	112.4
(Rinsed-Pulp + Rinse/Serum)										
High-MW	203.6	5.6	2.52	0.61	11.40	5.13	1.24			
Low/Int-MW	734.4	3.5	2.4	0.49	25.70	17.63	3.60			
SubTotal	1000				44.86	NA	4.95	104.8	(% of Puree) NA	109.5
(Rinsed-Pulp + High-MW + Low/Int-MW)										
SubTotal	938				37.11	22.76	4.84	101.4	(% of Rinse/Serum) 99.83	97.36
(High-MW + Low/Int-MW)										
Int-MW	97.9	6.7	4.21	0.85	6.58	4.12	0.83			
Low-MW	636.5	2.8	1.95	0.36	17.82	12.41	2.29			
SubTotal	1000				43.53	NA	4.477	101.7	(% of Puree) NA	99.05
(Rinsed-Pulp + High-MW + Int-MW + Low-MW)										
SubTotal	938				35.78	21.66	4.365	97.81	(% of Rinse/Serum) 95.04	87.81
(High-MW + Int-MW + Low-MW)										
Subtotal	734.4				24.38	16.53	3.123	94.85	(% of Low/Int-MW) 93.80	86.79
(Int-MW + Low-MW)										

Recombination Studies

After separating and fractionating the puree into fractions, reconstitution of these fractions was performed.

Second Level Recombination. Rinsed-pulp, high-MW, and concentrated low/intermediate-MW fractions were combined to make recombined puree. Using weights of fractions obtained during the separation/fractionation steps and the amount of solids in the original puree and fractions, calculations were made of how much of each fraction to add to make the puree. Since there had been spills during the initial centrifugation, exact tracking of fractions was not possible. Three preliminary trials were made homogenizing Trials 1 and 3 for 7 min, Trial 2 for 3 min. The samples were checked for solids and the final trial was tested for Bostwick, color, and syneresis. Results are summarized in Table 24. The amount of insoluble solids is apparently critical to viscosity. The three trials showed progressively better viscosity results and solids closer to the original solids level. The Bostwick results appeared promising. The color of the recombined product was lighter (higher L*), with slightly less red/purple (lower a*), and more yellow (higher b*). Syneresis results were similar to the original puree.

A second set of trials was made. A small amount of pulp (0.5% of the rinse/serum) which had been strained by cheese cloth from the rinse/serum prior to ultrafiltration was also added to the three fractions. All samples were homogenized 7 min.

TABLE 24. RESULTS OF RECOMBINATIONS

SAMPLE I.D.	TS	SS	IS	Bost- Calc. wick	Color			Color Std. Deviation			Syner- esis 1hr %Wt.	Rinsed Pulp %	Conc. Rinse/ Serum %	COMPOSITION		Water %	Strained Pulp %
					L*	a*	b*	L*	a*	b*				High MW %	Conc. Low/Int MW %		
Original Puree	10.65	8.56	2.08	5.35	31.62	21.90	19.44	0.08	0.15	0.14	5.93	--	--	--	--	--	--
Recombined Puree from Rinsed-Pulp, High-MW, and Low/Int.-MW Fractions (Second Level Recombination)																	
Trial 1	11.19	9.13	2.06	>24	NA	NA	NA	NA	NA	NA	NA	14.21	--	50.50	8.32	26.97	0
2	11.30	NA	NA	7.7	NA	NA	NA	NA	NA	NA	NA	17.00	--	48.85	8.05	26.10	0
3	10.76	8.05	2.71	5.68	35.07	20.22	24.40	0.08	0.13	0.11	6.64	22.00	--	44.16	7.28	26.56	0
Trial 4	10.93	8.85	2.08	6.95	34.33	19.29	23.73	0.07	0.13	0.07	4.68	15.20	--	48.73	8.26	26.80	1.01
5	10.83	8.52	2.32	4.5	35.09	19.52	24.55	0.06	0.04	0.11	2.84	18.00	--	46.63	7.91	26.50	0.96
6	10.32	8.66	2.26	5.33	34.91	19.35	24.25	0.08	0.03	0.09	2.35	16.97	--	47.38	8.04	26.63	0.98
7	11.18	8.95	2.23	5.23	34.87	19.53	24.34	0.09	0.13	0.13	2.45	16.97	--	48.22	8.18	26.63	0
Recombined Puree from Rinsed-Pulp and Concentrated Rinse/Serum (First Level Recombination)																	
Trial 1	10.91	8.81	2.10	6.72	35.10	19.34	24.57	0.04	0.07	0.04	4.19	15.90	68.00	--	--	--	--
2	10.87	8.57	2.30	5.45	35.69	19.45	25.15	0.14	0.12	0.13	3.2	18.00	66.00	--	--	--	--
3	10.85	8.60	2.25	4.3	35.92	19.42	25.46	0.17	0.15	0.10	NA	18.94	65.00	--	--	--	--

The results of these trials (Trials 4-7) are summarized in Table 24. The solids levels were similar to the original puree. The Bostwick viscosity results were good. The color results were similar to Trial 3 with slightly less red/purple. Syneresis results were better than with the original puree.

Thus, these trials showed that duplicating the solids of the original puree was difficult. Insoluble solids content is critical to the viscosity. Viscosity was better in the recombined puree than in the original puree, but insoluble solids were slightly higher than the original. If the solids could be exactly duplicated, firmer conclusions could be drawn.

First Level Recombination. Some rinse/serum was concentrated by rotary evaporation (2-2.5 hrs) at 45-50°C, 26-27" Hg vacuum to 12.8% TS. This concentrated rinse/serum was combined with rinsed-pulp to make recombined puree. Three trials were made, homogenizing all samples for 7 min.

Results are summarized in Table 24. The solids levels and Bostwicks obtained are similar to Trials 4-7 of the Second Level Recombination (also in Table 24). The color is slightly lighter with slightly more yellow than Trials 4-7 of the Second Level Recombination. Syneresis results were better than the original puree. These trials showed similar results to the above Second Level Recombination trials.

Pulp Drying Studies

Drying of the rinsed pulp would greatly facilitate its handling, storage, and transportation. To study this, tomato pulp was drum dried and evaluated for hygroscopy.

Drying of Rinsed Pulp. A twin drum laboratory dryer (Blau-Knox Co., Buflovak Equipment Div., Model ALC4) was used. This dryer had two drums, each 6 in. in diameter and 8 in. in length. The drums were driven by a Graham transmission model 175BR5. The dryer drums were heated internally by saturated steam; steam pressure determined drying temperature. Drum rotational speed determined product drying time, and the nip setting determined the product film thickness on the dryer.

Seven drying trials were conducted. The first was a preliminary evaluation trial. In this trial, it was decided that in order to obtain a feed consistency which could film on the dryer's surface, a slurry of rinsed-pulp and water was required. In the subsequent four trials, the amount of water added to rinsed-pulp was varied, and dryer settings were also varied, to obtain different dried pulps. The dried pulps were evaluated later by their ability to resist fungal spoilage in storage. The results are given in Table 25. The last two drying trials used unrinsed puree (9.6% NTSS) and a combination of tomato puree and paste which had an 18.6% NTSS to provide a dried tomato product which could be used as a sugar-containing control. This was compared with the dried rinsed-pulp, which contained little sugar. Results for these two trials are also given in Table 25.

Results in Table 25 show that the dryer was successful in producing a dried rinsed-pulp of 6% moisture content. The dried rinsed-pulp was orange in color, and resembled flakes of bran. Tomato puree could not be dried on the dryer, due to its low solids content, which caused the sticky product to roll up at the cut-off knives. The puree/paste blend was successfully dried to 4% moisture content; it was red in color.

TABLE 25. PULP DRYING RESULTS

Trial No.	Product	DRYER SETTINGS				Film Thickness (in)
		Moisture Content (%)	Steam Pressure (psi)	Steam Temp. (°F)	Drying time (sec)	
1	Prelim. Pulp: Water	89.5	55	302	26.2	0.009
2	Pulp: Water 2.33:1	90.5	55-70	302-316	26.2	0.009
3	Pulp: Water 2.33:1	89.3	65	312	26.2	0.007
4	Pulp: Water 3.51:1	87.6	65-68	312-315	33.7	0.006
5	Pulp: Water 5.00:1	87.7	66-68	313-315	28.1	0.006
6	Puree	89.5	55	302	28.1-33.7	.006-.008
7	Puree/Paste	79.2	50	298	13.7	0.008

Trial No.Dryer Results

1	Not dried sufficiently to avoid fungal spoilage
2	Dried well, no spoilage
3	Not dried sufficiently to avoid fungal spoilage
4	Dried well, no spoilage, moisture content: 6%
5	Dried well, no spoilage
6	Wouldn't dry, very sticky, rolled up at cut-off knives
7	Ran very well, no spoilage, moisture content: 4%

Hygroscopy of Dried Pulp. Hygroscopy is important because it relates to the storage stability, packaging needs, and handling requirements of dried foods. To study the hygroscopy of the dried rinsed-pulp (Trial 4) and the dried puree/paste (Trial 7), samples were placed in constant humidity rooms, and water uptake recorded as a gain in weight.

Two constant humidity rooms were used for the tests. The Tropical Room had 82°F dry bulb temperature and 80% relative humidity. The Prairie Room had 70°F dry bulb temperature and 45% relative humidity.

In the hygroscopy test, petri dishes were tared, and 5 gm samples of the dried product were weighed into the dishes. Six dishes (3 of dried pulp, and 3 of dried puree/paste) were placed in each room. The samples were weighed after 1, 2, 5, and 8 days in the rooms. The results, given as percent gain in weight versus storage time, are shown in Figs. 24 and 25.

Figure 24 - Hygroscopy - weight gain (%) versus time in tropical room.

Figure 25 - Hygroscopy - Weight gain (%) versus time in prairie room.

This study showed the hygroscopy of rinsed-pulp to be low, especially when compared with dried puree/paste. Samples of puree/paste stored in the Tropical Room absorbed enough water to permit mold growth after 10 days. The difference in hygroscopy reflects the difference in sugar content between the two samples, since at low moisture content, sugar has a great water binding capacity.

Fresh Juice Studies

In order to assess the effects upon fresh tomato juice of some of the techniques examined in this project, some studies were conducted with fresh product. For these studies fresh tomato juice (hot break: 210-200°F, 99-93°C) was obtained from a food processing plant along with serum and pulp produced from a production scale decanter centrifuge operating on fresh tomato juice.

Puree Production. The Bostwick procedure to evaluate viscosity is commonly used in the tomato processing industry. Since it is not possible to perform this test on tomato juice, because it is too thin, a baseline puree was made from the fresh juice. This was done in order to have a quality baseline that would include Bostwick for comparison with later studies results.

Fresh juice (4.9 lb, 2.2 kg) was concentrated using a rotary evaporator at 158°F, 70°C and 22" Hg vacuum. The initial NTSS was 5.0. Four batches of this were run and composited producing a final puree with 9.1% NTSS. The puree was analyzed and results are in Table 26.

Serum Concentration. It was necessary to concentrate the fresh juice serum to two solids levels. First, serum was needed at a solids level to be used for recombination to make puree; this

TABLE 26. CHARACTERIZATION OF FRESH JUICE AND FRACTIONS

SAMPLE I.D.	TS Vacuum %	SS Calc. %	IS Calc. %	NTSS %	Pectin %
Juice	5.86	4.78	1.08	5.0	0.32
Baseline Puree	10.59	8.59	2.00	9.1	--
Pulp	11.32 11.09	4.87 4.88	6.46 *1 6.21 *2	5.4 --	1.31 --
Serum	4.83	4.83	~0	4.9	0.18
13% Serum	12.76	12.58	0.18	13.0	--
23% Serum	--	--	--	20.8	--
70% Serum	70.61 71.92 73.61	Calc. 67.88 68.81 73.49	2.93 * 3.11 *4 0.13 *5	71.5 -- --	-- -- --

*1=Whole Bucket
 *2=2nd Frozen Tub
 * =Tub 1

*4=Composite of rest of
 Tub 1 and all Tub 2
 analyzed on 9/25/86
 *5=*4 analyzed on 10/16/86

would test whether separation alone affects quality. Then, serum was needed at a much higher solids level to be used for recombination to make puree; this would test whether separation and concentration affect quality. The highly concentrated serum would also be stored to assess shelf-stability.

Fresh juice serum (~20 gal, ~76 L) was evaporated in a pilot scale evaporator at ~160°F, ~70°C from 4.9% NTSS to 13% NTSS. Approximately 2.5 gal, 9.5 L was removed from the evaporator for recombination testing. The rest of the serum still in the evaporator was concentrated to 20.8% NTSS and removed. Rotary evaporators were used (~3-4 hrs) at ~160°F, ~70°C and 22" Hg vacuum to produce serum of 71.5% NTSS. Analysis results of fractions, juice, and puree are given in Table 26.

Recombination. The objective was to recombine the serum (at two solids levels) with pulp and water in order to assess the effect of separation, (concentration) and recombination upon quality. It was also necessary to produce enough of the puree made from low level solids serum to be used for a storage effect study.

Recombination with Low Level Solids Serum.

Pulp (113.16 gm), 13% TS serum (231.56 gm), and water (55.28gm) were combined to make 400 gm puree, homogenizing for 7 min. The NTSS (9.2%) and TS by microwave (10.5%) were measured to check solids levels against the baseline puree (9.1% NTSS, 10.3% TS). Since the solids were approximately the same as the baseline puree, larger batches were made using the same component ratios. Eleven batches (400 gms @) were made with the homogenizer for 7 min each and composited (Sample No. 1, Table 27). One batch (~12 lb, 5.4 kg) was made using the static mixer with two passes through it (Sample No. 2, Table 27). A portion of each of these two batches was tested for solids and quality factors. The rest was used in a storage effect study. (See Storage Effects.)

TABLE 27. CHARACTERIZATION OF FRESH JUICE AND RECOMBINED SAMPLES

Sample I.D.	Sample No.	TS %	SS %	IS Calc. %	NTSS %	Bostwick cm.	L*	Color a*	b*	Std. L*	Color Deviation a*	b*	Syner-esis 1 hr % Wt.
Baseline Puree		10.59	8.59	2.00	9.1	5.3	32.20	21.29	19.35	0.10	0.26	0.11	13.7
Homog. Puree	1	10.53	8.68	1.90	9.2	5.3	32.24	22.19	19.62	0.14	0.16	0.12	11.2F
Stat.Mix Puree	2	10.60	8.63	1.97	9.2	5.67	32.06	22.09	19.22	0.13	0.17	0.17	11.5F
HotF.1mo Homog.	3	11.15	9.15	2.00	9.62	4.47	31.59	21.76	19.30	0.12	0.10	0.14	6.2
HotF.4mo Homog.	4	11.12	9.07	2.05	9.77	4.7	30.94	21.82	19.25	0.15	0.25	0.36	8.3
HotF.8.5mo Homog.	5	NA	NA	NA	NA	4.5	31.51	21.26	19.23	0.11	0.10	0.09	10.1
HotF.1mo Stat.Mix	6	11.17	9.16	2.01	9.67	4.51	31.15	21.59	18.90	0.16	0.10	0.15	8.0
HotF.4mo Stat.Mix	7	11.21	9.12	2.10	9.8	4.36	30.74	21.57	18.80	0.27	0.21	0.24	8.3
HotF.8.5mo Stat.Mix	8	NA	NA	NA	NA	4.25	31.30	21.21	19.10	0.11	0.11	0.14	10.3
Homog. Puree-0	9 (Trial 1)	10.43	8.71	1.73	9.2	IN 6.0 ON 5.88 HT 8.0	31.91	20.97	19.57	0.21	0.16	0.18	6.0sF
	10 (Trial 2)	10.87	8.72	2.15	9.2	4.86	31.04	21.22	19.13	0.11	0.14	0.12	5.7
Stat.Mix Puree-0	11	10.34	8.51	1.83	9.2	6.45	30.69	20.54	18.60	0.18	0.18	0.16	11.5sF

IN=initial
HT=heated

ON=overnight
sF=Possibly slightly fermenting

F=Possibly fermenting

The solids of the recombined purees were nearly identical to the solids of the baseline puree. The Bostwick of the homogenized puree (mean of 4 determinations = 5.3 cm) was the same as the baseline puree cm) indicating no loss in viscosity. The Bostwick of the static mixed puree (mean of 3 determinations = 5.67 cm) was slightly higher (+0.37 cm, +7%) indicating a slight loss in viscosity.

The colors of the recombined purees were neither lighter nor darker (no change in L*), and had slightly more red/purple (higher a*) than the baseline puree.

Syneresis results of homogenized (11.25%) and static mixed (11.5%) purees were slightly lower than the baseline puree (13.7%) indicating no significant increase in syneresis or serum separation. There was some gas formation in the two recombined samples during the five hour test indicating possible fermentation. These samples had been refrigerated for four days before the syneresis test.

The results of these tests indicate that separation by centrifuge, and recombination by homogenizer does not adversely affect viscosity (Bostwick), color, or syneresis. Recombination by static mixer does not appear to adversely affect color or syneresis, but the Bostwick viscosity is not fully recovered.

Recombination with High Level Solids Serum.

Trial 1 - Pulp (226.3 gm), 70% TS serum (83.7 gm), and water (490.0 gm) were combined to make 800 gm puree, homogenizing for 7 min (Sample 9, Table 27). This recombination procedure was repeated with the same ratios of each component, using the static mixer (2 passes) making 950 gm puree (Sample 11, Table 27). The samples were tested for solids and quality factors.

The total solids of the recombined purees (10.43 and 10.34%) were slightly lower than that of the baseline puree (10.59%). The Bostwick viscosity of the homogenized puree (mean of five determinations) was higher (6.0 cm) than the baseline puree (5.3 cm) indicating a slight viscosity loss (+0.7 cm, +13%). The Bostwick of the static mixed puree (mean of three determinations) was also higher (6.45 cm) than the baseline puree, indicating a viscosity loss (+1.15 cm, +22%). The samples were refrigerated overnight and the homogenized sample (No. 9) was tested for Bostwick. There was a slight improvement (5.88 cm). Both samples were heated to 92°C, cooled, and tested for Bostwick giving poorer results (8.0 and 7.9 cm). The lower viscosity values may have been related to the lower solids values; thus a second trial was made.

The color of the static mixed recombined sample (No. 11) was darker (lower L*); and had slightly less red/purple (lower a*) and less yellow (lower b*) than the baseline puree. The color of the homogenized sample (No. 9) was nearly the same as the baseline puree.

Syneresis results of the homogenized puree (6.0%) were significantly lower than the baseline puree (13.7%). The static mixed puree (11.5%) was approximately the same as the baseline puree. There was some gas formation in the two recombined samples during the latter part of the five hour test. This might indicate fermentation, however chemical analysis revealed no alcohol or lactic acid, possible fermentation products.

Trial 2 - Pulp (128.0 gm), 70% TS serum (28.2 gm), and water (233.7 gm) were combined to make 400 gm puree, homogenizing for 7 min (Sample 10, Table 27). The sample was tested for solids and quality factors.

This time the total and insoluble solids of the homogenized puree (10.87 and 2.15%) were slightly higher than that of the baseline puree (10.59 and 2.0%). The Bostwick viscosity of the homogenized puree (mean of 3 determinations) was lower (4.86 cm) than the baseline puree (5.3 cm) indicating a slight viscosity gain (-0.44 cm, 8%). If the recombination could reproduce the actual baseline solids, the baseline Bostwick value could probably also be reached. Quantity limitation prevented a third trial.

The color of Sample 10 was slightly darker than the baseline puree.

Syneresis of the Trial 2 homogenized puree (5.7%) was lower than the baseline puree (13.7%).

The results of these tests indicate that separation by centrifuge, concentration of serum to a high level, and recombination by homogenization will most likely give a puree with adequate viscosity, less serum separation, and slightly darker color. The total and insoluble solids content of the recombined product is critical to its viscosity. Since the static mixed recombination was not repeated, it is difficult to draw firm conclusions about its quality.

Storage Effects. The effect of time upon both recombined product and high level solids serum was studied.

Recombined Product Storage.

As mentioned previously, some of the recombined puree made with low level solids serum was used in a storage effect study, to see what effect separation and recombination would have upon the long term quality of the recombined product.

Sixteen #303 cans were filled with recombined product which had been heated to ~190°F, 88°C. The cans were refrigerated to prevent flat sour spoilage. Eight cans contained static mixed product; eight cans contained homogenized product (one of these cans was only half full). The cans were stored for subsequent opening at various time intervals for product quality assessment.

At 1 month two cans of each recombination technique were opened and composited. Analysis results are in Table 27 (Sample Nos. 3 and 6). The solids of these two purees were higher than those of the baseline puree because of the evaporation which took place while heating the recombined purees prior to filling the cans. The Bostwick viscosity determination gave similar results for both products (4.47 cm, s.d. = 0.01 [the mean of 3 determinations] for the homogenized; 4.51 cm, s.d. = 0.16 [the mean of 8 determinations] for the static mixed). These values show an increase in viscosity (~-0.8 cm, 15%) over the baseline puree (5.3 cm) which is at least partly due to the increase in solids (~0.6% TS increase). The Bostwick of the static mixed puree was approximately the same as that of the homogenized puree, which is significant since these two values differed (0.37 cm, 7%) prior to heating and holding.

The hot filled purees had higher solids than the baseline puree. This, in addition to the heat treatment, may account for their darker colors (lower L*). The static mixed hot filled puree (Sample No. 6) had slightly less yellow color (lower b*).

Syneresis results are better (lower) than the baseline puree and the recombined purees prior to heating and holding. Some of this may be due to the increase in solids content, and some may be due to the heat treatment.

At 4 months two cans of each recombination technique were opened and composited. Analysis results are in Table 27 (Sample Nos. 4 and 7). The Bostwick viscosities were similar to those at 1 mo. The colors of both recombined products were slightly darker than at 1 mo, but there were no other significant changes in color. The syneresis results showed a slight increase in serum separation, but the values were less than the original baseline puree.

At 8.5 months two cans of each recombination technique were opened and composited. Analysis results are in Table 27 (Sample Nos. 5 and 8). The Bostwick viscosity values of the recombined products were similar to or lower than those at 1 and 4 months. Colors of the recombined products at 8.5 months were similar to the color at 1 month. Syneresis results were higher than at 1 and 4 months, but lower than the baseline puree.

These storage effect results indicate acceptable storage capability. Provision should be made in subsequent testing to assess storage of the product at temperature(s) commonly used for product storage (70°F).

High Level Solids Serum Storage.

The 72% solids serum was stored at room temperature in a plastic tub for 8 months. There was no sign of mold growth. The outer layer appears darker in color than the inner portion.

Ultrafiltration of Serum. The same 100,000-MW-cutoff ultrafiltration membranes that had been used with rinse/serum were used for testing with fresh juice serum.

Two batch separation runs were made and data is summarized in Table 28 and Figs. 26 and 27. All runs were made at 100 psi (0.7 MPa), 8L/min, and 25°C (except during the two temperature scans

in the second batch) with 0.108m² membrane area each of two membrane types (GR40PP and PES100). Each run began with an hour run time in recycle mode, to monitor initial flux. This showed that flux decline was time dependent as well as concentration dependent. After the first batch, the membranes were cleaned with an enzyme detergent (Monarch DDS).

During the second batch, temperature scans followed the initial recycle period (25-65°C) and the batch separation (25-65°C) (Table 29). At the higher temperatures, as in the tests run with rinse/serum, sugar rejection (by NTSS) was lower and the flux significantly higher.

The flux with fresh juice serum was in the same range as that with the rinse/serum but was somewhat lower, possibly for the following reasons: the initial fresh juice serum feed had a higher osmotic pressure, due to its higher concentration, (5% NTSS) than the initial rinse/serum feed (4% NTSS); the membranes had operated previously for 29 hr before operating on fresh juice serum.

The flux from both membrane types was very similar even though the PES100 membranes were operating at a lower pressure (since they were on the outlet end of the module). Sugar rejection with the PES100 membranes was lower than with the GR40PP membranes. The lower rejection, a desirable characteristic, may be related to the lower operating pressure of the PES100 membranes, or the characteristics of the membrane. Reversing the order of the membranes on the module could test whether the lower operating pressure was the cause of the lower rejection.

During the runs with fresh juice serum, 53-57% of the sugar permeated and 55-60% of the organic acid permeated. The final retentate weight was 37-41% of the initial feed weight. The runs

with rinse/serum permeated more sugar (65-74%) and acid (66-77%), and produced a lower retentate weight (17-28% of the initial feed weight). The runs with fresh juice serum could not reach the lower retentate weight reached with rinse/serum. This may have been due to the higher pressure drop across the membranes which occurred at the beginning of the runs with fresh juice serum (62 and 80 psi) versus the initial pressure drop with rinse serum (32, 43, 52, and 62 psi), (Figs. 19 and 28) which built up with operation time.

TABLE 28. 100,000-MW-CUTOFF MEMBRANE ULTRAFILTRATION TESTING WITH FRESH JUICE SERUM

Batch No.	Serum (kg)	Separation Time (hr)	Temp. Scans Time (Hr)	Flux				Feed	NTSS (%) Reten- tate	NTSS (%) Composite Permeate		Sugar Permeated %	Acid Permeated %	Separation Weight Retained %
				GR40PP		PES100				GR40	PES100			
				Recycle Start/End	Separat. Start/End	Recycle Start/End	Separat. Start/End							
				[L/m ² hr] [gal/ft ² day]										
1	39.5	4.5	0	33/30 [19/18]	28/19 [16/11]	35/32 [21/19]	29/19 [17/11]	5.0	5.9	4.6	4.7	53	55	41
2	26.1	4.7	1.8	30/25 [18/15]	18/15 [10/9]	34/27 [20/16]	19/15 [11/9]	5.0	6.1	4.5	4.7	57	60	37

Figure 26 - Flux versus time - 100,000-MW-cutoff membrane ultrafiltration of fresh juice serum.

Figure 27 - NTSS (%) versus time - 100,000-MW-cutoff membrane ultrafiltration of fresh juice serum.

TABLE 29. 100,00-MW-CUTOFF MEMBRANE ULTRAFILTRATION TEMPERATURE SCANS WITH FRESH JUICE SERUM

Batch No.	Serum (kg)	Scan Timing	Temp. (°C)	Flux (L/m2hr) [gal/ft2day]		Reten- tate	NTSS (%) Permeate	
				GR4OPP	PES100		GR4OPP	PES100
2	26.1	After Recycle	25	23[15]	27[16]	5.0	4.2	4.4
			38	31[18]	33[19]	4.9	4.2	4.4
			51	35[21]	36[21]	4.8	4.3	4.5
			65	41[24]	41[24]	5.1	4.5	4.6
		After Separation	25	15[9]	15[9]	5.6	4.4	4.8
			38	18[10]	18[10]	5.8	4.5	4.8
			48	18[10]	18[10]	5.9	4.7	4.9

Figure 28 - Pressure drop versus time - 100,000-MW-cutoff membrane ultrafiltration of fresh juice serum.

CONCLUSIONS AND RECOMMENDATIONS

Tomato puree was successfully separated by centrifuge into pulp and rinse/serum fractions. Rinse/serum was successfully fractionated into high-, intermediate-, and low-MW fractions. Further study on the performance economics of the ultrafilter is recommended. Fractions were successfully concentrated (rinse/serum [to 13% TS] and low/intermediate-MW fraction [to 74% TS] and recombined by homogenizer to produce a puree. The recombined puree had satisfactory viscosity and was lighter in color than the control puree. Ion exchange successfully produced sugar and organic acid fractions from the low-MW fraction. Further work is needed to demonstrate the possibility of recombining using the sugar and organic acid fractions.

Fresh tomato juice was successfully separated by centrifuge into pulp and serum fractions. The serum was successfully concentrated to two levels (13 and 70% TS) and recombined with pulp and water to produce a puree by homogenization and by static mixing. The homogenized recombined puree had satisfactory viscosity at both serum concentration levels; the static mixed recombined puree had satisfactory viscosity at the lower serum concentration level. The recombined puree was darker in color than the control puree at the higher serum concentration level, and the same color as the control puree at the lower serum concentration level. It is recommended that a storage study of fractions and recombined product be conducted at ambient temperature.

Performance of host site studies is recommended.

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APPENDIX A

ABBREVIATIONS

Btu	British thermal unit
°C	degree Celsius
cm	centimeter
cP	centipoise
°F	degree Fahrenheit
ft	foot
gal	gallon
gal/ft ² day	gallon per square foot per day
gal/min	gallon per minute
gm	gram
h	hour
hr	hour
hp	horsepower
in	inch
J	joule
K	kelvin
kg	kilogram
kJ	kilojoule
L	liter
lb	pound
L/m ² hr	liter per square meter per hour
L/min	liter per min
m	meter
mPa	milliPascal
MPa	megaPascal
psi	pound per square inch
rpm	revolutions per minute
s	second
w	watt

APPENDIX B

METRIC TO ENGLISH CONVERSION TABLE

<u>To Convert From</u>	<u>To</u>	<u>Multiply By</u>
L/m ² hr	gal/ft ² day	5.891×10^{-1}
bar	psi	1.45×10^1
mPa	psi	1.45×10^{-7}
MPa	psi	1.45×10^2
L	gal	2.642×10^{-1}
L/min	gal/min	2.642×10^{-1}
cm	in	3.937×10^{-1}
m	in	3.937×10^1
m	ft	3.2808
cm ²	in ²	1.55×10^{-1}
m ²	ft ²	1.0764×10^1
m ³	ft ³	3.5313×10^1
m ³	gal	2.642×10^4
gm	lb	2.2046×10^{-3}
kg	lb	2.2046
kg	t	1.023×10^{-3}
w	hp	1.3405×10^{-3}
J	kwh	2.7778×10^{-7}
kJ	Btu	9.478×10^{-1}