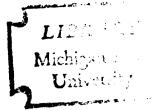
RHEOLOGICAL PROPERTIES OF SLUSH FROZEN ORANGE JUICE CONCENTRATE

Thesis for the Degree of M. S. MICHIGAN STATE UNIVERSITY PANAGIOTIS ATHANASOPOULOS 1975

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ABSTRACT

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RHEOLOGICAL PROPERTIES OF SLUSH FROZEN ORANGE JUICE CONCENTRATE

by

Panagiotis Athanasopoulos

Design problems for cooling and heating, for pumping, and transportation of liquid foods are associated with knowledge of their flow characteristics. The objectives of this study were 1) to determine the rheological parameters of slush frozen orange juice concentrate, and 2) to determine the influence of percent frozen water on flow characteristics of the frozen orange juice concentrate.

Commercial frozen orange juice concentrate (45° Brix) was used in this experiment. Measurements were accomplished with a capillary tube viscometer. The experiments were conducted under constant temperature conditions. Data obtained from the experimental studies were computer analysed. According to results obtained, the flow behavior index increased with decreasing temperature; the consistency coefficient decreased with decreasing temperature, while the apparent viscosity increased with decreasing temperature. Two empirical functions were developed to correlate the rheological parameters to percent of frozen water. In addition a chart was prepared to correlate the friction factor and the generalized Reynold's number. This chart should be useful for pumping and transportation design problems.

RHEOLOGICAL PROPERTIES OF SLUSH FROZEN ORANGE JUICE CONCENTRATE

bу

Panagiotis Athanasopoulos

A THES IS

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Panagiotis Athanasopoulos

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NOMENCLATURE

A	Area
а	Constant in equation (12)
В	Intergration constant in equation (7)
Ъ"	Distance between two parallel heated surfaces cr
¥	Shear rate sec
Ср	Specific heat of the product cal/gro(
P	Pressure gradient dynes cm ⁻²
D	Tube diameter
Ea	Activation energy cal/mole
F	Friction force dyne
f	Friction factor
GR e	Generalized Reynold's number
ћс	Convective heat transfer coefficient cal/cm ² min.oc
KE	Kinetic energy cm gr/g
K	Reaction rate constant
k	Thermal condutivity
L	Tube length cr
L'	Latent heat cal/gr
۲	Coefficient of viscosity points
На	Apparent viscosity points
m	Consistency soefficient dynes cm ² sec ^{-r}
mb	Consistency coefficient at the bulk properties

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INTRODUCTION

The methods used in freezing fruit juices may be divided into two categories depending upon the rate of freezing. They are the slow and quick freezing categories. Juices containing small quantities of colloidal and suspended matter, such as apple juice, may be frozen slowly without causing any particular physical change in them. When the frozen juices are thawed and agitated, they are completely reconstituted. These juices can be solidly frozen in large containers by using an air blast freezer.

In slowly frozen juices containing a considerable amount of colloidal and suspended matter, such as orange juice, much of the colloidal and suspended matter is coagulated. When thawing a slowly frozen juice, the coagulated material usually settles. On the other hand, in quick frozen juices, less colloidal and suspended matter is coagulated. In this case, the coagulated particles are much finer and do not settle so rapidly when the juice is thawed (Tressler et. al 1968). As a rule, the more rapidly the juice is frozen, the less the clearing or settling after thawing. Rapid freezing also permits much less chemical, enzymatic and microbiological changes than slow freezing does. Continuous slushfreezing methods are usually used for these juices. Juice is slushfrozen, and is automatically filled into cans which are closed and frozen solid by passing through a freezing tunnel. The votator is widely used for slush-freezing; both for single strength and concentrated juices.

Frozen slushes are the basis of a new technique developed during the last years which can be used as a concentration process. According

to this method, called "slush evaporation", the evaporator is fed by slush-frozen juice, and water is removed by both vaporization and sublimation.

Since slush-frozen juices have to be mechanically handled, it is important to know the rheological properties of these slushes. The flow behavior index (n) and consistency coefficient (m) must be considered when machinery or equipment are designed.

No investigations have been attempted, on the rheological properties of frozen slushes. Therefore, the objectives of this study were to establish the flow behavior of slush-frozen orange juice concentrate by determining if it is a Newtonian or a non-Newtonian liquid; to determine the rheological parameters of slush-frozen orange juice concentrate at temperatures as low as those used by industry; and to determine the influence of percent frozen water on flow characteristics of the slush-frozen orange juice concentrate. This knowledge could then be utilized to improve freezing procedures, and pumping, and also for designing fast transportation systems.

LITERATURE REVIEW

The term rheology has been defined as a "science devoted to the study of deformation and flow" (Reiner, 1960). Rheology encompasses the area of fluid flow which is so important in many segments of the food industry. It should not be assumed that the theories developed for other materials will apply directly and ideally to food products (Anon., 1973). Many added parameters tend to make the situation much more complex. The rheological properties of food products may be influenced by factors such as temperature, humidity, and chemical or microbiological reactions. So, parameters are frequently experimentally measured and concepts are developed which are not utilized in any other field.

A. PROPERTIES OF FLUID FOODS

The consistency of a fluid is the property which governs its flow characteristics, which in turn, are essential in estimation various engineering quantities, such as heat transfer coefficients, evaporation rates, and pumping and mixing requirements. Since these flow characteristics are dependent upon the properties of the fluid, it is necessary to discuss methods utilized in measurement of these properties. Because fluid foods exist in variable conditions the methods to be discussed will not be adequate for all situations. These methods and the parameters used to describe fluid properties are the best available and can lead to acceptable results in many design situations.

I. Viscosity

Viscosity has been defined as the internal resistance of a liquid to flow, and it is indicated by the coefficient of viscosity given by the equation

$$\tau = -\mu \, \underline{\mathrm{du}} \tag{1}$$

In this equation (7) is the shear stress, P is the coefficient of viscosity and du/dy is the velosity gradient which exists between two surfaces. The model in Fig. I) can be used for theoretical determination of shear-rate of shear (which is equivalent to du/dy) relationship. As shear stress has been defined the ratio F/A = 7, and as rate of shear the ratio F/A = 7.

Equation (I) describes Newtonian fluids. The plot of τ vs -du/dy is a straight line passing through the origin. The slope of the line is the viscosity coefficient or viscosity. Since ν is a constant, a single determination of it can completely characterize the flow behavior of the liquid. (Heldman, 1975)

There are many liquids employed in the food industry which do not hold on to this simple relationship. Such liquids are often suspensions of solids or emulsions of liquids in a liquid medium. Both can be called dispersions. The discrete particles (the discontinuous phase) may interact with each other and also with the medium (the continuous phase) by which they are surrounded. If the interaction depends on the flow rate then the coefficient of viscosity is no longer a constant, and for such systems a one point measurement is no longer adequate, because it does not characterize the flow rate. Such liquids are called non-Newtonian. As the amount of pectin as well as other suspend and colloidal material increases the products and become increasingly non-Newtonian (Holdsworth, 1973;

Muller, H.G. 1973). They also show continuous flow even for the smallest applied force. The Bingham-plastic behavior is one in which a finite yield (7y) is required before a viscous response is obtained.

The relationship between the shear stress and rate of shear for a variety of fluids, considered to be non-Newtonian, is illustrated in Fig. (2). The Newtonian behavior is described by the equation (I). The plot shear stress vs rate of shear, for Bingham-plastic behavior, is a straight line described by equation (2)

$$\tau = m \left(-\frac{du}{dy} \right) + \tau y \tag{2}$$

Where (m) is referred to as plastic viscosity, and (\(\tau_y\)) is the yield stress required before a viscous response is obtained.

Equation (3) is a two parameter model which can be used to describe pseudoplastic and dilatant fluids.

$$\mathcal{T} = m \left(-\frac{du}{dy} \right)^{n} \tag{3}$$

Mixed type or quasi-plastic fluids can be described by equation (4).

$$\mathcal{T} = m \left(-\frac{du}{dy} \right)^n + \mathcal{T} y \tag{4}$$

Which is a general power-low equation. For these fluids, three parameters are required to describe the flow characteristics of the fluid, which would have either pseudoplastic or dilatant response after an initial yield point is reached.

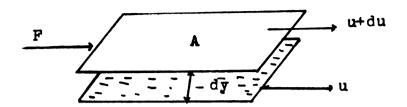


Fig. I Model for theoretical determination of shear-rate of shear relationship (from Charm 1971).

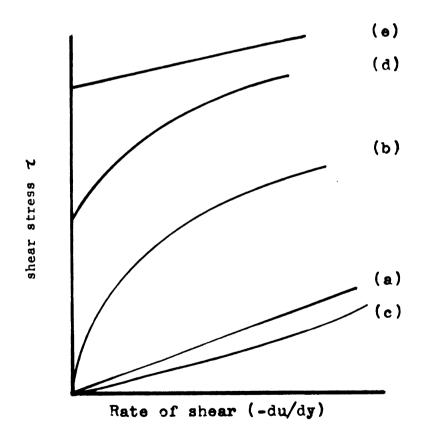


Fig. 2 Characteristic flow behavior of fluids:

(a) Newtonian, (b) Pseudoplastic, (c)

Delatant (d) Mixed type, (e) Binghamplastic (from Heldman 1975).

2 Apparent Viscosity

The term "apparent viscosity" is used to describe the viscous property of non-Newtonian fluids. Apparent viscosity is expressed in absolute units and is a measure of the resistance to flow at a given rate of shear. It represents the viscosity of a Newtonian liquid exhibiting the same resistance to flow at the chosen rate of shear.

In most common cases of a pseudoplastic fluid the apparent viscosity decreases with increasing shear rate. The ratio,

$$\frac{\mathcal{T}}{=du/dy} = \mu a \tag{5}$$

may be considered to be the apparent viscosity of the fluid (Charm, 1971).

3. Non-Newtonian time dependent fluids

If the shear stress becomes a function of time the fluid is considered time-dependent.

If a fluid is subjected to a constant rate of shear for a given period of time and its apparent viscosity increases to some maximum value, which means the shear stress increases (ratio 5), the phenomenon is called rheopecty. This rarely occurs in food stuffs (Van Wazer et.al, 1963).

A behavior which may occur among food products is a decrease in shear stress with time. This phenomenon is called "thixotropy." Translated the Greek term thixotropy means change by touch, indicating the material decreases in shear stress, but builds up again when at rest. Some fluids revert to their original viscosity almost immediately while others will recover after several hours (Russopoulos, 1956).

4. Factors affecting rheological properties

Product composition affects directly rheological properties of a

fluid. Temperature is the other factor which influences considerably the rheological parameters.

a Total solids

Total solids have a tremendous effect on consistency coefficient (m) and a little effect on flow behavior index. At high concentrations, higher than 50 brix, the flow behavior index (n) decreases significantly with the increasing of total solids (Harper et. al 1965; Holdsworth, 1973). Figure 4 shows how the rheological parameters are affected by total solids in pear pure at 150°F (Harper and Leberman, 1964) and in table I values of (m) and (n) are given as they were calculated from Fig. 4 at different total solids concentration.

b <u>Temperature</u>

One of the widely used methods of describing the influence of temperature on reaction rate was presented by Arrhenious in 1899 when he proposed that the following expression should describe the influence of temperature on the reaction rate constant:

$$\frac{d(LnK)}{dT} = -Ea - R_{g}T^{2}$$
 (6)

by intergrating equation (6) the following one is obtained:

$$L_{n}K = -\frac{E_{a}}{R_{g}T} + L_{n}B$$
 (7)

Where (K) is the reaction rate constant, (E_a) is the activation energy, (R) is the gas constant, (T) is the absolute temperature and (B) is a constant of integration.

To develop a relationship between temperature and the rheological parameter (m) equation (7), will be written as:

$$L_{n}m = L_{n}B - \frac{Ea}{R_{g}T}$$
(8)
By determining consistency coefficient (m) at a number of



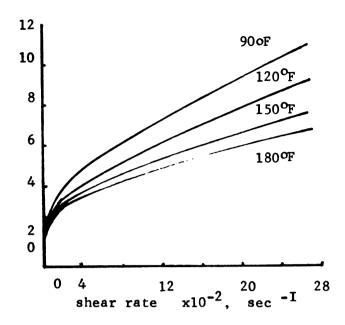


Fig. 3. Flow behavior curves for Pear Purees at different temperatures (Harper and Lebermann, 1964).

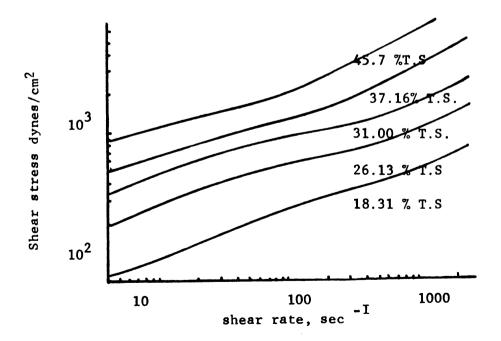


Fig. 4. Flow behavior curves for Pear Purees at 150°F (Harper and Lebermann, 1964).

TABLE I--Effect of total solids on

(m) and (n) for Pear Puree
from Harper and Leberman, 1964

Total solids %	m	n
18	50	.32
26	118	.33
31	205.7	.37
37	272.0	.35
45	407.4	.37
L		

TABLE 2--Effect of temperature on

(m) and (n) for Pear Puree from Harper and Leberman, 1964

Temper.OF	m	n			
90	37.15	.423			
120	31.65	. 420			
150	25.12	. 430			
180	23.43	.425			

temperatures (at least 3) and plotting the results obtained we evaluate constants ($-\frac{E_a}{R_g}$) and B. After that we can predict (m) values at different temperatures using equation (8).

The flow behavior index (n) is not significantly affected by temperature. Fig.4 shows plots of shear stress vs shear rate at different temperatures for pear pured. Since the slope of all the curves is about the same, the values of flow behavior index (n) are not significantly different. Table 2 shows how rheological parameters (m) and (n) are influenced by temperature. Results of this table have been calculated from Fig. 3.

5. Some applications of rheological parameters

a Design of transport systems

The most important factors taken under consideration in pumping calculation are friction and Reynolds number.

In most applications, friction is defined as a force which opposes the flow of the fluid in a certain system. The following expression, for a unit mass of fluid, has been used to define friction

$$F = A (KE) f (9)$$

where F is the force, A the area, KE the kinetic energy, and f is a friction factor. The friction factor (f) for non-Newtonian liquids and for laminar flow can be evaluated from the following expression (Heldman, 1975).

$$f = \frac{16m \left(\frac{3n+1}{2}\right)^{n} 2^{n-3}}{p\bar{u}^{2} - n}$$
 (10)

where (m) is the consistency coefficient, (n) is the flow behavior index, (p) is the density, $(\bar{\mathbf{u}})$ is the mean velocity, and (D) is the tube diameter. Laminar flow occurs when the generalized Reynolds number (GRe)

is less than 2100. In the case in which GRe > 2100, a certain chart developed by Dodge and Metzner (1959) can be used for evaluation of (f). The (GRe) is defined as follows (Heldman 1975):

GRe =
$$-\frac{p\bar{u}}{2^{n-3}} \frac{2-n}{m} \frac{ph}{n}$$
 (11)

The following expression is generally used for kinetic energy computation, for a power-low fluid

$$KE = -\frac{\mathfrak{a}^2}{a} \tag{12}$$

where (a) is constant equal to two (a=2) during turbulent flow but varies according to following equation when laminar flow occurs (Heldman, 1975)

$$a = \frac{(4n+2)(5n+3)}{3(3n+2)^2}$$
 (13)

From this last expression it is apparent that parameter (a) is not affected by consistency coefficient (m) but is determined by flow behavior index (n).

b Cooling and heating processes

The most important factor in design problems dealing with heat transfer is the computation of convective heat transfer coefficients for pseudoplastic fluids.

There are several expressions proposed by investigators which can be used for convective heat transfer coefficient, when tubular heat exchanger is used for heating or cooling. One of them is the following, presented by Charm and Merrill (1959).

$$\frac{\text{fic D}}{\text{K}} = 2 \begin{bmatrix} \frac{\text{WCp}}{-1} \\ \frac{\text{KL}}{\text{KL}} \end{bmatrix} = \begin{bmatrix} \frac{1}{3} \\ \frac{\text{mb}}{\text{ms}} & \frac{3n+1}{2(3n-1)} \end{bmatrix}^{0.14}$$
(14)

Where hc is convective heat transfer coefficient, D tube diameter, K

is product thermal conductivity, W is flow rate, Cp is product specific heat, L' is tube length, n is flow behavior index, mb and ms is consistency coefficient at the bulk properties and at the mean wall temperature.

Other expressions and examples are given by Heldman (1975).

B. VISCOMETERS

Viscosity measurements are accomplished indirectly by using instruments measuring time, force, or flow rate. A large number of instruments have been utilized to measure the rheological properties of fluid food products. All these instruments can be classified in two categories: (a) rotational rheometers including the coaxial-cylinder type and the cone and plate type, and (b) capillary tube rheometers.

The solid components, present in the orange concentrate, have an effect on its flow behavior. The dimensions of these components are not always negligible with respect to the gap between inner and outer cylinder of a rotational viscometer; therefore measurements in the rotational viscometer might be erroneous, (Rozeme et al. 1974). So in this experiment a capillary tube rheometer was used. The theory about this instrument is somewhat complicated and an analysis of it in detail would be beyond the purpose of this study. However, it is intentional that some basic information pertaining to general concepts of this methodology is included herewith in favor of a sufficient communication.

For capillary tube rheometers the pressure gradient and the volumetric flow rate of the fluid are measured. From these measurements shear rate and shear stress are obtained. The following assumptions are

inherent to all capillary viscometers; (a) flow is steady, (b) properties are time independent, (c) flow is laminar, (d) fluid velocity has no radial or tangential components, (e) the fluid exhibits no slippage at the wall, (f) the fluid is incompressible, (g) the fluid viscosity is not influenced by pressure, and (h) the measurement is conducted under isothermal conditions (Heldman, 1975).

To obtain a laminar flow at a steady state and to have a negligible entrance and exit flow-effects we have to use a small diameter tube (normally less than 1/4 inc.), and the length to diameter ratio must be high (usually higher than 100) (Saravacos, 1968).

In most cases a tube viscometer can be used to determine the viscometric constants of various fluid foods. Gravity-flow capillary viscometers have been used widely in the laboratory for Newtonian fluids. A pressure tube viscometer is always used for the determination of the rheological parameters for non-Newtonian liquid foods.

For a non-Newtonian fluid, obeying the power-law equation (3), the flow through a capillary tube is given by the equation (Brown, 1961)

Observing equations (3) and (15) we obtain the following expressions for shear stress

$$\mathcal{T} = \frac{\text{RAP}}{2\text{L}}$$
(16)

and for shear rate

$$y' = \left(\begin{array}{cc} \frac{I+3n}{---} \right) \left(\begin{array}{c} Q \\ -\frac{Q}{4} \end{array}\right)$$
 (17)

By rearranging equation (15) as

$$\log \left(\frac{AP}{2L} \right) = \log m - n \log \pi \left(\frac{n}{3n+1} \right) - (3n+1) \log R + \log Q$$
 (18)

and by plotting log (AP/2L) versus log Q the value of (n) can be determined from the slope of the resulting curve, while the parameter (m) can be evaluated from the intercept.

The tube viscometer used in these experiments for determining rheological properties of slush-frozen orange juice is described in the next chapter.

C. FLOW PROPERTIES OF ORANGE JUICE

Orange juice concentrate is a suspension of particles in a liquid called orange juice serum. The latter contains a significant amount of pectin, besides sugar acids and other soluble materials.

Both whole orange juice and orange juice serum show non-Newtonian behavior and have been characterized as non-Newtonian, pseudoplastic, thixotropic liquids. This behavior was defined by Ezell(1959) and confirmed by Charm (1963).

The rheological properties of concentrated orange juice may be explained on the basis of the contribution on the flow behavior of each one of the component systems (Mizrahi and Berk, 1970). Depectinized orange juice serum is a Newtonian liquid, and can be considered as the basic liquid medium. The soluble pectic substances impart to the serum non-Newtonian behavior and thixotropic properties. The suspended material seems to be responsible for most of the non-Newtonian behavior and time dependence of flow properties. The disintegration of coarse pulp particles results the irreversible decrease in apparent viscosity.

Flow characteristics of slush-frozen orange juice are not

presented in the literature. To our knowledge no studies have been attempted in this temperature range.

METHODS AND PROCEDURES

All experiments were conducted in a refrigerated room in which temperature could be controlled. The apparatus, and all instruments used, were placed in this room.

a <u>Sample</u>. Commercial frozen orange juice concentrate, in 12 fluid oz. (354 cm³) paper cans was used. This juice was shipped from Indian River, Florida. It was prepared by TREESWEET PRODUCTS CO. It was held in a refrigerator at 15° F. about five days after the day it was bought.

TREESWEET PRODUCTS CO. provided the following information on the lot of orange juice concentrate. The source was Valencia oranges extracted by Brown machine-reamers, and concentrated in triple effect vacuum pans (temperature not exceeding 80° F.). Its pulp was 9 to 11% in reconstituted juice, acidity in concentrate 2.32 to 2.56%, pH in reconstituted juice, approximately 3.80 - 3.90. This orange juice concentrate was packed on July 18, 1974.

The content of six cans was emptied into a glass beaker and placed in the experimental room at 20° F. (-6.67°C) to equalize with the room temperature. A portion of this sample of about 1.7kg (3.4 lb.) was used for making the experimental measurements. The sample was thoroughly mixed by stirring before measurements were made. To avoid any loss of moisture, the sample was covered with aluminum foil

b <u>Pressure.</u> Air under pressure was used in order to obtain flow of the orange juice concentrate through the capillary tube. Compressed air for the experiment was obtained from the air line system in the

laboratory and was transferred to the air pressure vessel at about I8 psig. A steel vessel, normally used for gas transportation, suitable for pressure up to 120 psia was used. The dimensions of this vessel are: height 107 cm, inside diameter 26 cm, and the volume 56781 cubic cm. This vessel was equipped with an air valve near the bottom for connecting to the air pressure line, and with components near the top, as illustrated in Fig. 5.

- 1 Pressure regulator, to regulate air pressure to desirable level.
- Pressure gauge, A MARSHALLTOWN, IOWA USA MFG CO, Oto 30 psig gauge was used.
- 3 Air valve, to connect the air pressure vessel and the viscometer (sample vessel).

The air pressure vessel was held overnight in the refrigerated room to allow the compressed air to reach the room temperature.

Since measurements were obtained at pressures of 14, 12, 10, 8, and 6 psig when the air vessel was refilled, a considerable amount of cooled air (6 psig) existed. Time of about 3-4 hours was estimated as sufficient for the new air to obtain the desirable temperature, so three measurements per day were scheduled.

c The apparatus. Tube viscometer, illustrated in Fig. 5, was used in this experiment. It consisted basically of a stainless steel reservoir for the orange juice sample; a tin vessel surrounding this reservoir, working as a constant temperature bath; a short piece of stainless steel tube between the reservoir and the capillary tube; and a straight copper capillary tube.

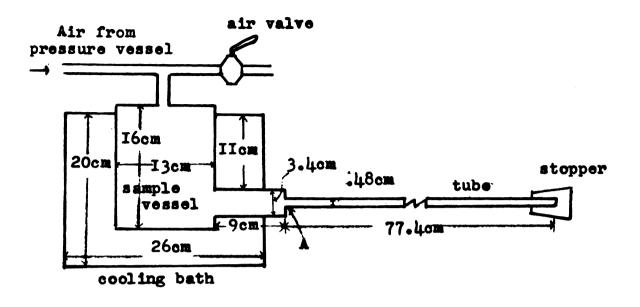
As reservoir, a stainless steel vessel 16 cm high and 13 cm inside diameter was used. Four bolts supported the reservoir in a

center position inside the tin vessel which served as a bath. In the space between these two vessels coolant (ethylene glycol) was recirculated from the constant temperature cooler bath.

For all measurements, smooth wall copper tubing with an inside mean diameter of 0.48 cm and a 77.4 cm in length was used. The mean diameter was computed from the volume of the distilled water required to fill it completely. A stainless steel tube, with 3.4 cm inside diameter, and 9 cm long, welded horizontally near the bottom of the reservoir, was used to connect the reservoir and the capillary tube. The latter was connected to the apparatus through a rubber stopper as illustrated in Fig. 6 in detail.

The end of the capillary tube was stopped using a rubber stopper (Fig. 5) while a piece of string of about 30 cm long (I foot), ending in a loop was used to prevent entrance of the juice into the capillary tube before starting measurements were begun. For this purpose the loop-end of the piece of string acted as a stopper for the capillary tube (Fig. 6). The apparatus was then connected to the cooling bath so all the components were held at constant temperature; the mean room temperature. An AMINCO SILVER SPRING, Maryland (serial 17845 - 20, Volt II5, wt 1250, 60-cycles) bath cooler was used, which had a temperature range from -140° F. to +360° F. This bath cooler was equipped with a small pump to recirculate liquid coolant (ethylene glycol).

d Measurements. Approximately 1.7 kg (3.4 lb) of orange juice concentrate was removed from the sample, after a gentle agitation, and was placed in the reservoir of the apparatus. The slush-frozen orange juice was gently agitated by a mixer. A GT2I laboratory mixer (Gerald



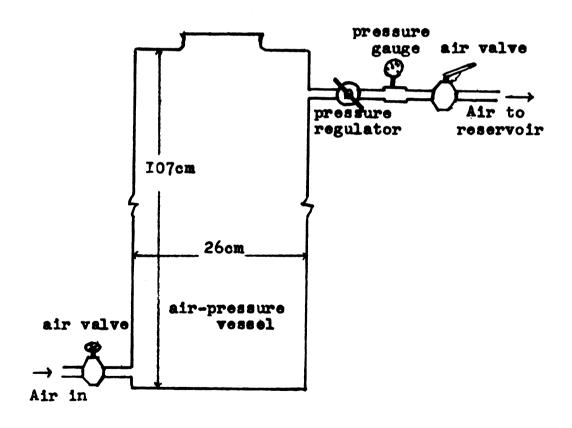
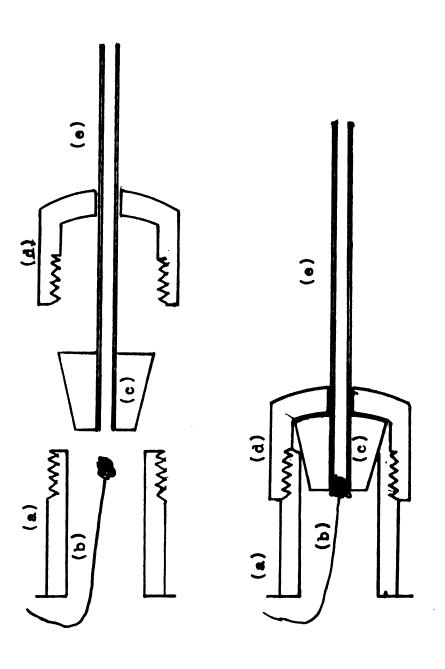


Fig. 5. Tube viscometer equipment arrangement.



Details for connecting sample reservoir and copper tubing at point A: F18. 6.

(a) Stainless steel tube, (b)String, (c) Rubber stopper,

(d) Cup, (e) Copper capillary tube.

K. Heller Co. -Las Vegas) was used with variable speed. Speed was controlled by using a GT 21 motor electronic controller able to control speed in a range of 0 to 277 RPM (slow shaft) or from 0 to 5,000 RPM (fast shaft). The temperature was checked from time to time, using a 0 to 230° F., 2°, Partial imm. (SARGENTWELTH, SCIENTIFIC CO.) thermometer. When the temperature of the orange juice was constant, the mixer was removed from the reservoir. After removing the piece of string, the cover was placed on the vessel and secured with wing nuts. The air pressure vessel was connected to the apparatus to provide the desired pressure. The rubber stopper was attached to the discharge end of the capillary tube.

Using the regulator, the air pressure was adjusted to the desired level and the air valve was opened. After a delay of 2 to 3 minutes for pressure equilibration measurements were obtained. At this time the components and orange juice concentrate were at temperature equilibrium, and practically isothermal flow could be expected.

The rubber stopper was then removed, and after 3 to 4 seconds, when a steady flow was obtained, samples were collected in preweighed small glass beakers. The beakers were placed in the refrigerated room for at least 2 hr. before making the measurements in order to avoid increase in the collected juice temperature. This permitted reusing the product for subsequent trials. The procedure prevents changes in the physical properties of the juice due to temperature changes. A temperature change of 2 to 4° F. (I.8-3.6° C) was observed.

Samples were weighed and re-used by mixing with remaining portions of the orange juice. Weight was converted to volume by dividing with density (p). This was determined by weighing a certain volume of orange juice concentrate at these low temperatures. It was found, that one cubic centimeter had an average weight of 1.174 grams. The density increases with decreasing of the temperature, but at the region below to 4°C it decreases as the temperature is decreasing. The average measured density was used to compute the volumetric flow rate.

In computing pressure difference, ΔP , the gravitational affect was added, and the factor 6.7 x 10^4 was used to convert psig to dynes per square centimeter.

Each experiment, including one sample at each pressure and at a given same temperature, conducted in triplicate. Samples were collected at seven different temperatures, namely, 20,22, 24, 26, 28, 30, and 32° F. (-6.67, -5.56, -4.45, -3.34, -2.23, -1.12, 0° C), and for five different pressures at each temperature, 14, 12, 10, 8, and 6 psig. The time for running each experiment was in a range of 10 to 30 seconds. A GRALAB Universal Timer, model 1971, (Dimco-Gray Company, Dayton, Ohio), capable of timing any interval from one second to sixty minutes, used to measure time intervals during experiment. The weight from the capillary tube ranged from about 50 to 270 grams. The weight was determined by analytical balance (Mettler P5N) to the second significant figure.

The experiments were conducted by beginning with the highest pressure followed by experiments at lower pressures.

- e <u>Soluble solids</u>, For soluble solids determination an ABBE refractometer was used. The juice was found to be 45 Brix.
- f <u>Total solids</u>. For total solids determination the vacuum oven (Vacuo) official method was used. The orange juice contained 47.3% total solids.

RESULTS AND DISCUSSION

For each experimental measurement the pressure difference (AP) and the volumetric flow rate (Q) were calculated. The pressure difference, measured in p s i, was converted to dynes per sq. cm by multiplying by the appropriate conversion factor (6.7×10^4) . The average volumetric flow rate (Q) for all the temperature values (Table 3) was plotted vs. $\Delta P/2L$ resulting in the curves shown in Fig. 7. These rheograms indicate that slush-frozen orange juice concentrate is a non-Newtonian liquid, as expected. If the liquid was Newtonian, the curves in Fig. 7 would be straight lines. Plotting of log (AP/2L) vs. log (Q), (Table 4) results in a characteristic straight line (Fig. 8). The average of the three trials from Table 3 was used in log (Q) computation. Both rheograms, Fig. 7 and Fig. 8, were obtained by using Wang computer (700 series). Least squares fit-power curve $(\Delta P/2L = KQ^{n})$ and linear regression analysis ($log \Delta P/2L = log K + n log Q$) programs were used for Fig. 7 and 8 respectively. The slopes of the curves from the first program are slightly different than that from the latter one (Table 5). Both columns in Table 5 should be identical. The slight difference due to the fact that logarithms for AP/2L and for Q were taken to the third significant figure. Since the results obtained by using least square fit-power curve program are more accurate, the computer outputs for this program were used in the calculations. Correlation coefficients for each curve and for both plots are presented in Table 6.

The slope of each curve, Fig. 7 and 8, represents the flow behavior index (n). To evaluate the consistency coefficient (m),

		1
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TABLE 3--Flow rate at different temperatures and \mathcal{O}^{P}

P 2L				Flow	Rate, c	cm ³ .sec		
dyn.		20° F	220 F	24° F	260 F	28° F	30° F	320 F
cm ³		(-6.67 °C)	(-5.56 OC)	(-4.45°C)	(-3.34° C)	(-2.23° C)	(-1.12 oc)	(0 °C)
9 09	max.	11.168	11.831	12.307	12.660	13.468	13.649*	15.463
	aver.	11.108	11.266	12.135	12.573	13.415	13.610	15.354
	min.	11.020	10.831	11.889	12.484	13.315	15.570	15.290
5 195	max.	8.484	9.158	10.031	10.169	10.363	11.314	12.075
	aver.	8.379	8.978	9.816	10.142	10.251	11.243	12.048
	min.	8.290	8.767	9.668	10.121	10.141	11.175	12.022
4 345	max.	6.659	6.888	7.667	7.729	8.091	8.730*	9.224
	aver.	6.631	6.764	7.632	7.722	8.058	8.460	9.105
	min.	6.612	6.528	7.593	7.717	8.025	8.190	9.035
3 480	max.	4.966	5.210	5.567	5.756	5.991	6.372	6.704
	aver.	4.923	5.112	5.458	5.635	5.927	6.248	6.483
	min.	4.851	5.055	5.315	5.503	5.878	6.124	6.257
2 610	max.	3.227	3.428	3.486	3.491	3.683	3.746*	4.235
	aver.	3.233	3.294	3.404	3.431	3.601	3.734	4.156
	min.	3.221	3.193	3.321	3.395	3.467	3.722	4.080

One measurement has been eliminated.

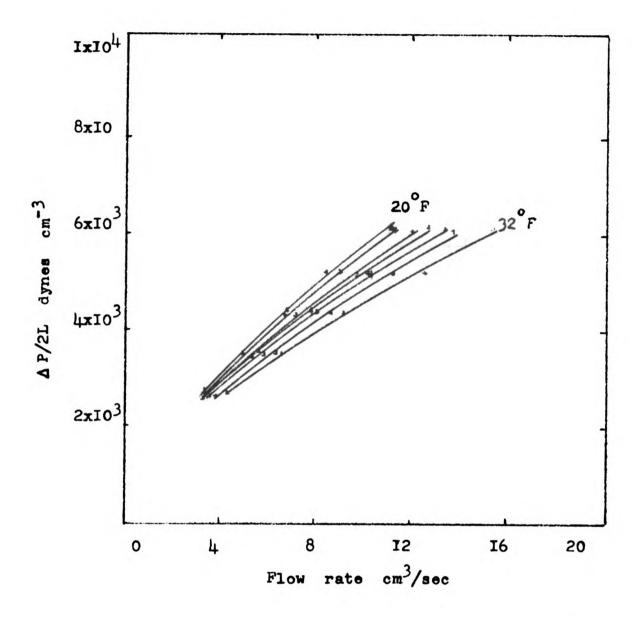


Fig. 7. Orange juice concentrate rheograms at different temperatures.

TABLE 4. Log Q at different temperatures

P 108		1 o 8 Q (Q	is the aver	age of the th	Q (Q is the average of the three trials - Table 3)	Table 3)	
7P	20° F	22° F	24° F	26º F	28 ⁰ F	30° F	32º F
3.785	1.046	1.052	1.084	1.099	1.128	1.134	1.186
3.716	0.923	0, 953	0.992	1.006	1.011	1.051	1.081
3,638	0.822	0.830	0.864	0.888	906.0	0.927	0.959
3.542	0.692	602.0	0.737	0.751	0.773	0.796	0.812
3.417	0.510	0.518	0.532	0.535	0.556	0.572	0.619

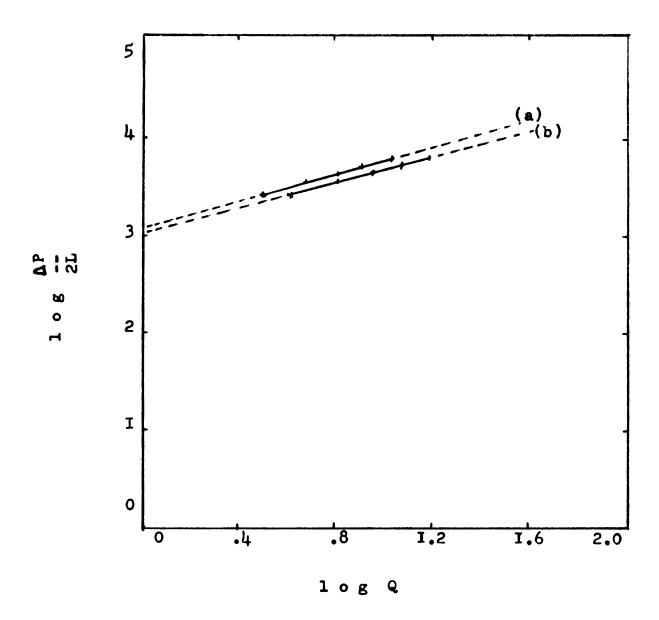


Fig. 8. Orange concentrate rheograms at (a) 20°F and (b) 32°F.

TABLE 5-- Slope of curves obtained by Wang computer

Temper.	s 1 o	pen*
o _F	△P/2L vs Q	log d P/2L vs log Q
20(-6.67°C) 22(-5.56°C)	.6991 .6837	. 6987 . 6930
24(-4.45°C)	.6675	. 6670
26(-3.34°C) 28(-2.23°C)	.6536	. 6534 . 6547
30(-1.12°C)	.6532	. 6522
32(0°C)	.6406	. 6486

^{*} The difference between the two columns due to the fact that log. were taken to the third significant figure (Table 4).

TABLE 6-- Correlation coefficients obtained by Wang computer

Temper.	correl.	coeff.
o _F	AP.2L vs Q	log AP/2L vs Q
20(-6.67°C)	. 999	.990
22(-5.56°C)	. 999	. 9995
24(-4.45°C)	.999	.999
26(-3.34°C)	. 9989	. 9989
28(-2.23°C)	.9988	.9987
30(-1.12°C)	. 9980	. 998
32 (0°C)	.9995	. 999

the following relationship (Equ. 18), between pressure difference and volumetric flow rate was used.

$$\log(\frac{AP}{2L}) = \left[\log m - n \log \pi - n \log(\frac{n}{3n+1}) - (3n+1) \log R\right] + n\log Q$$
 (19)

This equation is of the type:

$$\log\left(\frac{\Delta P}{2I}\right) = \log K + n\log Q \text{ or } \frac{\Delta P}{2I} = KQ^{n}$$
 (20)

In equation (20), (K) is the same in the brackets as in equation (19).

$$l_{cy} K = logm - nlog \pi - nlog (\frac{n}{3n+1}) - (3n+1) log R$$
 (21)

Values of (K) are obtained directly from computer for each curve, therefore the consistency coefficient (m) can be evaluated from equation (21). Table 7 summarizes the rheological parameters of orange juice concentrate in temperature, a range from 20° F to 32° F. (0° C). These results are in accordance with that reported by Saravacos (1968) for mixture of 2% of citrus pectin and 50% sucrose.

The rheological properties of orange juice concentrate are influenced by the soluble pectic substances and suspended materials. Both of them impart non-Newtonian behavior to the juice. Only a slight influence of temperature on the flow behavior index was expected, because the pectin gel structure is almost completely insensitive to temperature (Mizrahi et al 1970). Generally the flow behavior index will decrease with temperature. In this experiment, it was found that the flow behavior index (n) increased as the temperature decreased within the range below the freezing point.

Since the predominant change in the orange juice concentrate at temperatures below the freezing point is ice crystal formation, a

behavior index (n) was attempted. The temperature of 26° F was estimated as the initial freezing point of orange juice concentrate. This temperature defined by using data reported in Ag-handbook No. 66 - USDA (1948) for sugar solutions at different concentrations, and the percent frozen portion of the water was computed (Appendix 1), for each temperature, on this basis. Table 8 presents the percent of frozen water at different temperatures and Fig. 10 shows the plot of flow behavior index (n) vs. percent of frozen water at temperatures from 20° F (6.67°C) to 26° F (-3.34°C). The Wang computer was used for this plot, and the correlation coefficient obtained was 0.938. These results illustrate that the flow behavior index (n) is directly related to the amount of ice crystal formation.

Consistency coefficient; (m) normally increase with decreasing temperature. At the low temperatures investigated, a decrease in consistency coefficient (m) with temperature was observed. Fig. 11 illustrates that both parameters (m) and (n), are influenced by temperature. A correlation between (m) and percent frozen water was attempted. The plot (Fig. 9) obtained by using the Wang computer, shows the relationship between these variables. A negative correlation coefficient equal to -0.947 was computed. The decrease in consistency coefficient (m) and increase in percent frozen water with decreasing temperature is shown in Fig. 12.

Ice crystals, and generally percent of frozen water influences
the constitutes responsible for the non-Newtonian behavior or orange juice
concentrate, namely soluble pectin and suspended materials. The exact

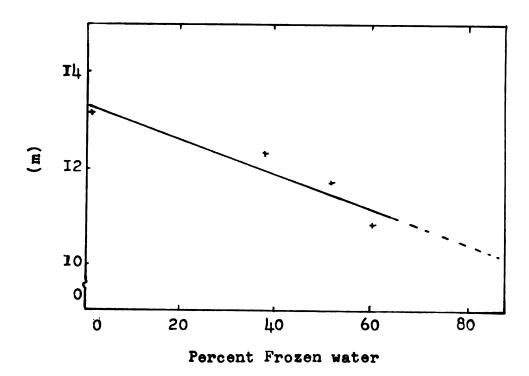


Fig. 9. Relationship between (m) and percent frozen water.

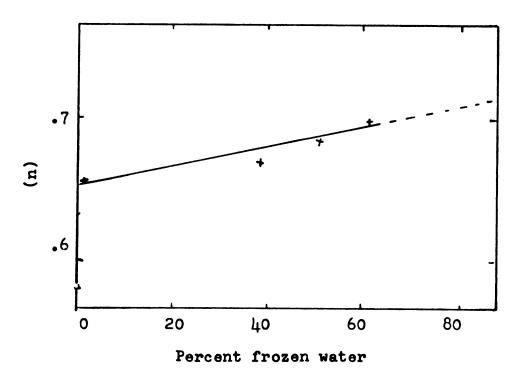


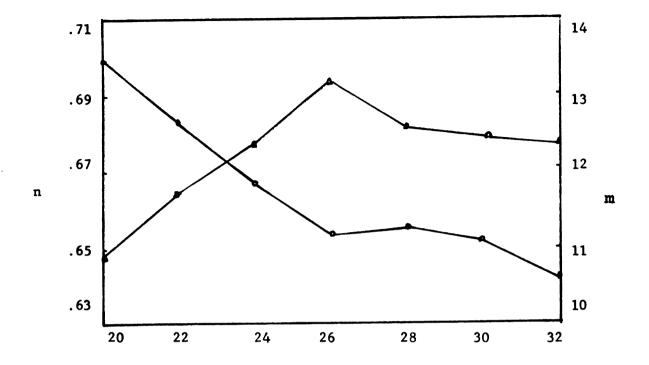
Fig. IO. Correlation between (n) and percent frozen water.

TABLE 7. Effect of temperature on n and m
(n and m for the average Q-Table 3)

Temperature OF (OC)	n	dyn sec ⁿ
20 (-6.67)	. 69913	10.86
22 (-5.50)	. 68367	11.74
24 (-4.45)	. 66745	12.38
26 (-3.34)	.6536	13.20
28 (-2.23)	. 65598	12.61
30 (-1.12)	. 6532	12.45
32 (0)	.6406	12.36

TABLE 8. Effect of temperature on percent frozen water

Temperature OF (OC)	% Unfrozen Portion	% Frozen water
20 (-6.67)	19.956	62.13
22 (-5.56)	24.887	52.77
24 (-4.45)	32.106	39.08
26 (-3.34)	51.768	1.77



Temperature, ${}^{O}F$ Fig. 11. Influence of temperature on (n) and (m) .

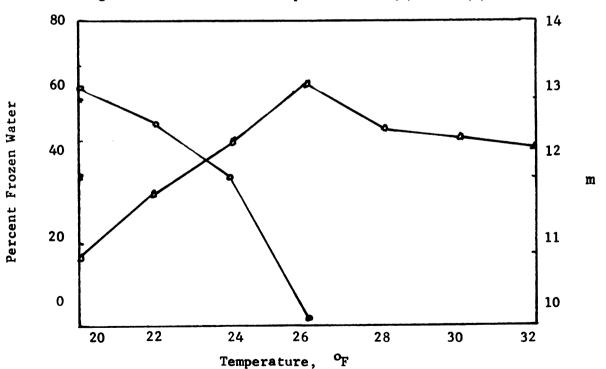


Fig. 12. Change of (m) and percent frozen water with temperature $\$

influence of these components on flow characteristics is not obvious. The molecules of pure water arrange themselves very regularly into hexagonal crystallization units when slow freezing occurs. In the freezing of solutions, instead of solid masses of crystalline ice, skeletons of ice bathed in a concentrated solution, are being formed (Luyet, 1968). These skeletons in connection with the rag-like coarse, may form an open lattice which can be crossed by water and sugar but not by soluble pectin. The drained more concentrated liquid, low in soluble pectin and coarse, shows less pseudoplasticity (Mizrahi et al 1970), and may work as a lubricant between the particles influencing their mobilization. These may exlain why (n) increases and (m) decreases with decreasing the temperature. In addition, during the flow through the tube a thin layer of syrup is probably formed in contact with tube walls, creating what is called "slippage."

As the slow behavior index (n) increases and tends to become equal to unity, the liquid shows less and less pseudoplasticity resulting in higher pumping requirements. On the other hand as consistency coefficient (m) decreases, smaller pump size is required. In this experiment, a simultaneous change of both flow behavior index (increases) and consistency coefficient (decreases) with decreasing in temperature was observed resulting in an increase in the apparent viscosity.

Two empirical equations were derived from the correlation of percent frozen water and the rheological parameters. Equation (22) correlates the flow behavior index (n) and percent frozen water (x), and equation (23) shows the relationship between consistency coefficient (m) and (x).

$$n = (0.64877) + x (0.698) 10^{-3}$$
 (22)

$$m = (13.425) - x (35.41) 10^{-3}$$
 (23)

Equation (22) indicates that the higher the percent frozen water, the higher the (n) value. On the other hand, consistency coefficient (m) decreases with an increase in percent of frozen water (equation 23).

Using equations (22) and (23), rheological parameters can be predicted at any temperature below the freezing point. Percent frozen water can be computed by following the procedure shown in appendix I. These equations are appropriate for orange juice concentrate containing the same amount of pulp and soluble pectin as the orange juice as used in this investigation.

The apparent viscosity of the orange juice concentrate, at various shear stresses and temperatures, was calculated using equation (5). Shear rate (-du/dy) at various temperatures and flow rates were computed from equation (17). Table 9 summerizes the results of these calculations. The apparent viscosity is affected by temperature at various shear rates as illustrated in Table 10. Shear rate is plotted vs. shear stress (Fig. 13) at three temperatures at 6° F (-3.34°C) intervals. It is interesting to note that a higher shear stress results in lower apparent viscosity values (Fig. 14). In addition, all three curves in Fig. 14 show a flattened segment near the freezing point (24° to 26° F). A three dimensional illustration (Fig. 15) was prepared to illustrate the influence of temperature and shear stress on apparent viscosity simultaneously. The surface obtained indicated the manner in which the apparent viscosity is affected by temperature and shear stress.

TABLE 9--Influence of temperature on shear rate at different shear stress

shear stress			Rate of	s he s r,	sec -I		
dyn.cm ²	20° F (-6.67° C)	22° F (-5.56° C)	2 4º F (4.45º C)	2 60 F (-3.340 C)	2 80 F (-2.230C)	30° F (-1.12° C)	320 F (00 C)
1 462.8	1133.728	1158.250	1257.528	1312.113	1369.316	1420.675	1613.328
1246.8	855.195	923.022	1017.214	1058.415	1046.355	1164.639	1265.949
1042.8	676.787	695.402	758.247	805.865	822.508	883.063	956.712
835.2	502.462	525.561	565.603	588.066	686.409	652.172	681.204
626.4	329.973	338, 654	352.750	358.058	367.567	389.759	436.694

TABLE 10--Influence of temperature on apparent viscosity

T							
shear		A P	Apparent	Viscosity,	83 7	o O	
dynes cm ² 2	20° F (-6.67°C)	22° F (-5.56°C)	24º F (-4.45ºC)	26° F (-3.34°C)	28° F (-2.23°C)	300 F (-1.12°C)	320 F (00 C)
1462.8	129.0	126.3	116.3	111.5	106.8	103.0	90.7
1246.8	145.8	135.1	122.57	117.8	119.2	107.1	98.5
1042.8	154.1	150.0	137.5	129.4	126.8	118.1	109.0
835.2	166.22	158.9	147.7	142.0	138.1	128.1	122.6
626.4	189.80	185.0	177.6	174.9	170.4	160.7	143.4

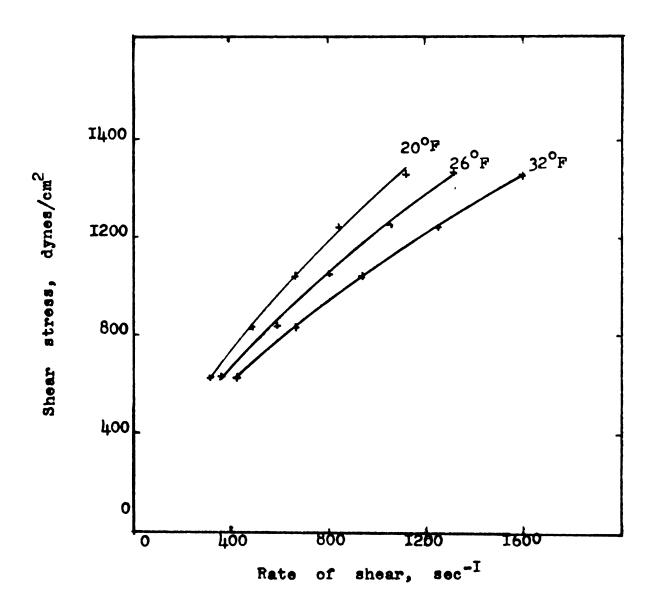


Fig. I3. Effect of temperature on shear stress-rate of shear relationship.

A correlation between apparent viscosity and percent frozen water was attempted, at different shear stresses. The correlation coefficient obtained was 0.896 for the lowest shear stress (626.4 dyn cm⁻²) and 0.931 for the highest shear stress (1462.8 dyn. cm⁻²). Apparent viscosity changes with shear stress, so we can not develope an equation correlating the apparent viscosity and the percent frozen water. Fig. 16 shows the plot of the apparent viscosity vs. percent of frozen water at three different shear stresses.

The velocity of the orange juice concentrate was computed for different conditions. In Table II the effect of temperature at different shear stresses on the velocity is illustrated. The relationship between shear rate and velocity is shown in Fig. 17, on log - log co-ordinates. The plot is a characteristic straight line.

The friction factor (f) for laminar flow can be evaluated from equation (10), or using the expressing:

$$f = 16$$
 / GRe (24)

Using the equation (11) the GRe was computed at the highest shear stress, and in turn, friction factors were evaluated using the above relationship (Table 12). Friction factor (f) was plotted vs. GRe on log - log co-ordinates, as shown in Fig. 18.

At a given temperature, (m) and (n) can be computed using equations (22) and (23) and used in equation (11). Using the value for GRe obtained and graph (18), the friction factor (f) can be evaluated. After finding the friction factor, the friction loss may be computed from the following common expression:

$$\frac{\Delta P}{P} = f \frac{u^{-2}L}{g_c R}$$
 (25)

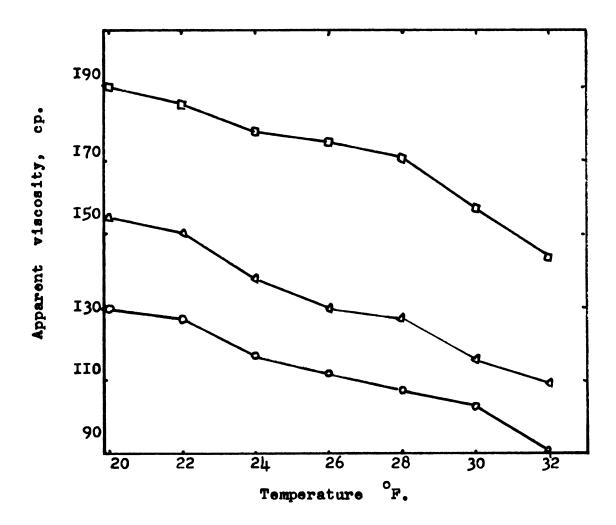


Fig. I4. Influence of temperature on apparent viscosity at different shear stress.

D 626.4 dynes/cm²

Δ IO42.8 dynes/cm²

0 1462.8 dynes/cm²

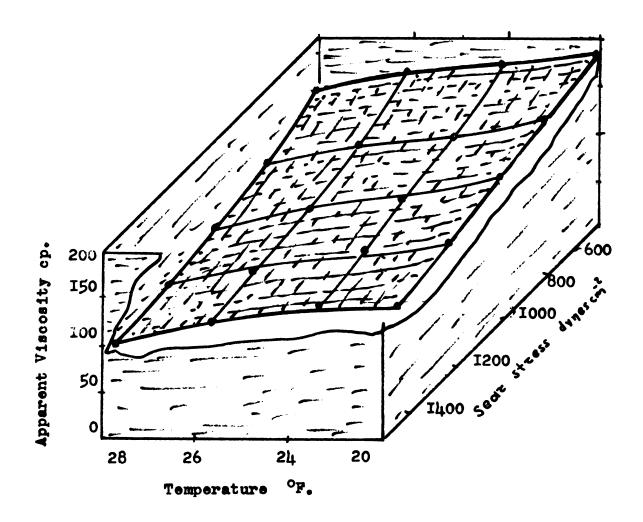


Fig. 15. Influence of shear stress and temperature on apparent viscosity.

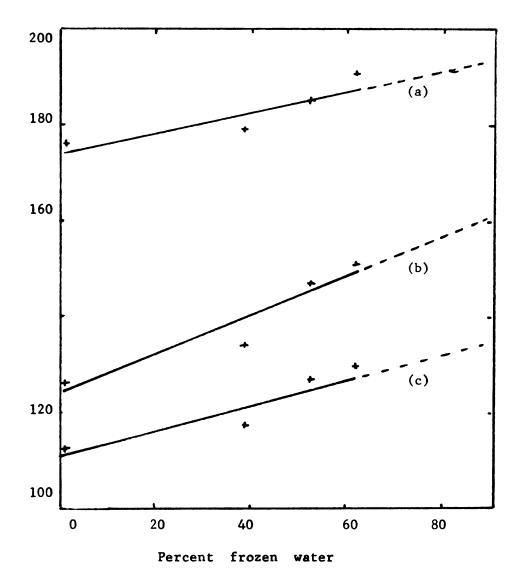


Fig. 16. Correlation between apparent viscosity and percent of frozen water.

- (a) at 626.4 dyn. cm⁻²
- (b) at $1042.8 \text{ dyn. cm}^{-2}$
- (c) at $1462.8 \text{ dyn.} \text{ cm}^{-2}$

TABLE II--Influence of temperature on velocity at different \$\Delta P/2L\$

2 <u>T</u>			Velocity		U = Q/A c m/s e c		
dyn cm	20° F (-6.67°C)	22 ⁰ F (-5.56 ⁰ C)	24° F (-4.45°C)	26 ^o F (-3.34 ^o c)	28° F (-2.23°C)	30° F (-1.12°C)	32° F (0°C)
6 095	61.71	62.59	67.42	69.85	74.53	75.19	85.30
5 195	46.55	49.88	54.54	56.63	56.95	62.46	66.94
4 345	36.84	37.58	40.65	42.90	44.77	46.74	50.58
3 480	27.35	28.4	30.32	31.31	32.93	34.52	36.02
2 610	17.96	18.30	18.91	19.06	20.00	20.63	23.09

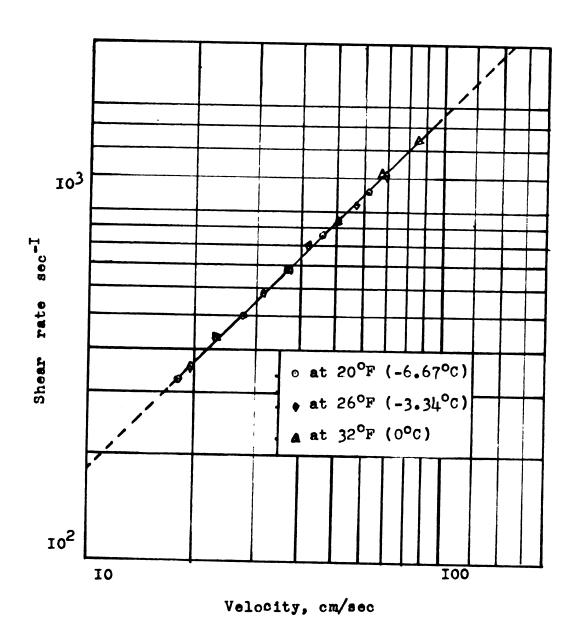


Fig. 17. Effect of shear rate on velocity of orange juice concentrate at different temperatures.

TABLE I2--Influence of temperature on GRe and on friction factor f (at shear stress 1462.8 dyn cm⁻²)

Temp. OF	ū	GRe	f= 16/GRe
20 (-6.67°C)	61.71	129.9	.0829
22 (-5.50°C)	62.59	193.7	.0826
24(-4.45°C)	67.42	218.9	.073
26(-3.34°C)	69.85	227.9	.0702
28(-2.23°C)	74.53	258.3	.0619
30 (-1.12°C)	75.19	252.7	.0616
32(0°C)	85.30	338.8	.0472

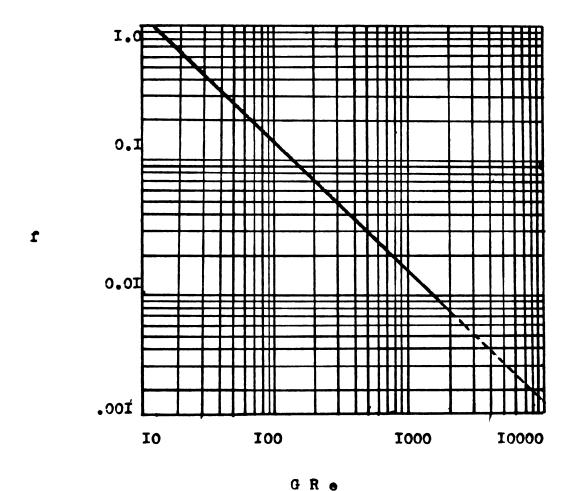


Fig. 18. Friction factor (f) against generalized

ReynoldS number (GRe) for laminar flow

of slush frozen orange juice concentrate

computed from f=16/GRe(Data from Table 12)

The results may be used in pumping and transportation calculations. In addition, the convection heat transfer coefficient, he, is needed in slush-freezing of orange juice concentrate either for packing as frozen product or for freeze concentration. The mean heat transfer coefficient can be evaluated from the following expression, presented by Pigford (1955).

$$\bar{N}u = 1.75 \frac{3n+1}{4n} \frac{1/3}{(Gz)}$$
 (26)

where n is the flow behavior index, Nu is the mean Nusself number, $Nu = \frac{F_0 cD}{k} \text{ and } Gz \text{ the Graetz number, } G = \frac{w^C p}{kL}, \text{ where D is the tube diameter, L is the length and k is the thermal conductivity. Equation}$ (14) can be used as well for hc determination in tubular heat exchangers.

For plate heat exchangers the following equation has been proposed by Skelland (1967).

$$\frac{\bar{h}cb''}{c} = Nu = 6/j (n+1/2n+1)$$
 (27)

where b'' is the distance between the two parallel heated surfaces and

$$j = 5/4 - \frac{2n}{2n+1} + \frac{3n}{4n+1} - \frac{n}{5n+1}$$
 (28)

This equation can be used for heat transfer coefficient evaluation.

The source of errors in this experiment can be evaluated by considering the following:

- a. The same sample was used for all measurements; freezing and thawing may have affected the results obtained.
- b. The same density was used to convert the sample weight to volume for all temperatures.
- c. Weighing accuracy of the samples which were collected.
- d. Inherent errors in instruments used in this research.

Despite the possible errors mentioned above, reasonable results were obtained. These may be used in design problems for pumping and transportation of frozen orange juice concentrate. Convective heat transfer coefficient can be evaluated as well, as it is explained in the above paragraphs, and may be used for problems dealing with design systems for cooling and thawing processes (Heldman 1975). Tubular heat exchangers are usually used for chilling orange juice concentrate, but recently plate-type heat exchangers were used successfully (Wells 1974). Freeze concentration and slush evaporation (Lowe et.al 1974) are processes in which rheological parameters can be used, either in slush-freezing step (by using the convective heat transfer coefficient) or in pumping the slush-frozen juice (using GRe and f), and in evaporation (Harper, 1760).

SUMMARY

A tube viscometer was constructed using a stainless steel reservoir and a capillary copper tubing. The rheological parameters of slush frozen orange juice concentrate of 45° Brix were evaluated by measuring the pressure gradient and the flow rate at certain time intervals.

Data obtained were computer (Wang computer, 700 series) analyzed in order to evaluate flow behavior index (n) and consistency coefficient (m). At temperatures below the initial freezing point, 26° F (-3.34°C), (m) decreased and (n) increased with a decrease in temperature.

The percent frozen water at different temperatures was computed and evaluation of its influence on the values of the rheological parameters was attempted. A linear relationship between percent frozen water and both (m) and (n) was obtained. The correlation coefficients were -0.947 and 0.938 respectively.

Two empirical equations were derived relating the percent frozen water and the rheological parameters (n) and (m); they were:

$$n = (0.64877) + x (0.698) 10^{-3}$$

$$m = (13.425) - x(35.41) \times 10^{-3}$$

These equations indicate the higher the percent frozen water, the higher the (n) and the lower the (m).

Apparent viscosity increased with a decline in temperature. The apparent viscosity was highest at low shear stresses. A correlation between the apparent viscosity and the percent of frozen water was observed. The correlation coefficients were 0.896 at the lowest shear

stress and .931 at the highest one.

The plot of shear rate and velocity in log-log coordinates resulted in a straight line.

Finally a graph was prepared relating the friction factor (f) to generalized Reynolds number (GRe). This graph is useful for pumping and transportation calculations.

Further studies are necessary with products containing different amount of pulp and soluble pectic substances in a range covering the commercial orange juice concentrations. Equations may be developed, by addition to equations (22) and (23), to obtain relationships between rheological parameters and pulp or soluble pectin contents.

BIBLIOGRAPHY

- 1. Anonymus 1973. Physics in the food Industry. Food Manufacture 48, No 7, 23.
- 2. Bogue, D.C. 1959. Entrance effects and prediction of turbulence in mon-Newtonian flow. Ind. Eng. Chem. 51: 874.
- 3. Brown, R. 1961. Designing laminar flow systems. Chemical Engineering June 12, 243.
- 4. Charm, S.E. 1960. Viscometry of non-Newtonian food materials. Food Research 25, 351.
- 5. Charm, S. E. 1963. The direct determination of shear stress-shear rate behavior of foods in the presence of yield stress. Food Science 28, 107.
- 6. Charm, S. E. 1971. The fundumentals of Food Engineering 2nd addition. AVI Publishing Co., Westport, Conn.
- 7. Charm, S. E. and Merrill, E. W. 1959. Heat transfer coefficients in straight tubes for pseudoplastic fluids in stream flow. Food Research 24, 319.
- 8. Dodge, D. W., and Metzner, A. B. 1959. Turbulent flow of non-Newtonian systems. Am. Inst. Chem. Engin. J. 5, 189.
- 9, Ezell, G.H. 1959. Viscosity of concentrated orange and grapefruit juices. Food Tech. 13, 9.
- 10. Harper, J. C. 1960. Viscometric behavior in relation to evaporation of fruit pures. Food Tech. No 14, 557.
- 11. Harper, J. C., and Leberman, K. W. 1964. Rheological behavior of pear pure's. Proc. First Intern. Congr. Food Science and Technology pp. 719-728.
- 12. Harper, J. C., and El Sahrigi, A. E. 1965. Viscometric behavior of tomato concentrates. J. Food Science 30, 470.
- 13. Heldman, D.R. 1975. Food Process Engineering. AVI Publishing Co., Westport, Conn.
- 14. Holdsworth, S. D. 1973. Consistency and Texture of Fruit products. Food Manufacture 48, No 7, 25.

- 15. Lowe, C. M., and King, C. T. 1974. Slush evaporation: A new method for concentration of liquid foods. J. Food Science 39, No 2, 248.
- 16. Luyet, B. 1968. In the freezing preservation of foods, eddited by Tassler et al., vol. 2, chp. I., AVI Publishing Co., Westport, Conn.
- 17. Mizrahi, S. & Berk, Z. 1970. Flow behavior of concentrated orange juice. Journal of Texture Studies I, 342.
- 18. Mizrahi, S. & Berk, Z. 1972. Flow behavior of concentrated orange juice: Mathematical treatment. Journal of Texture Studies 3, 70.
- 19. Muller, H. G. 1973. An Introduction to Food Rheology. Crane, Russak & Companu, Inc., New York.
- 20. Pigford, R. L. 1955. Non-isothermal flow and heat transfer inside vertical tubes. Chem. Eng. Progr. Symp. Ser. 17 51, 79.
- 21. Ram, A. & Tamir, A. 1964. A capillary viscometer for non-Newtonian liquids. Industrial and Engineering Chemistry 56 No 2, 47-53.
- 22. Reiner, M. 1960. Deformation, Strain and Flow. Lewis, London.
- 23. Rozema, H. & Beverloo, W. 1974. Laminar Isothermal flow of non-Newtonian Fluids in a Circular Pipe. Food Science + Technology vol. 7, No 4, 223.
- 24. Russopoulos, N. V. 1956. Geoponiki Chimia, Part I, p. 146 (Greek).
- 25. Saravacos, G. 1968. Tube viscometry of fruit purees and juices. Food Tech. 22, No 12, 89.
- 26. Skilland, A. H. 1967. Non-Newtonian flow and heat transfer. John Wiley and Sons, New York.
- 27. Treesweet Products Co. Personal communication.
- 28. Tressler, Van Arson Copley 1968. The freezing preservation of foods. Vol. 2. AVI Publishing Co., Westprot, Conn.
- 29. USDA Ag. Handbook No. 66, 1948.
- Van Wazer, J. R., Lyons, J. W., Kim. K. Y. and Colwell, R.E. 1963. Viscosity and Flow measurement. New York-London-Sydney Interscience Publishers a division of John Wiley and Sons, Inc. New York.
- 31. Wells, J. E. 1974. Chills concentrate from 60°F to 15°F more efficiently, uniformily, Food processing 35, No 12, 28.

APPENDIX

COMPUTATION OF UNFROZEN WATER RECENT

The following equation was used (Heldman 1975) to determine unfrozen water percent:

$$\frac{L}{Rg} \begin{bmatrix} I - I \\ T_A \end{bmatrix} = Ln X_A$$
 (I)

where L is the product of latent heat of fusion per unit mass and molecular weight of water, Rg the gas constant 1.987, T_A is the freezing point of pure water 492° K, T_A is the freezing point of the orange juice 486° K, and X_A is mole fraction of water in solution.

Using the appropriate values in the equation (I) we obtain:

Ln
$$X_A = \frac{144 \times 18}{1.987} = \frac{1}{492} = \frac{1}{486}$$
 I 1 I 1 1 2 2 3 3 3 3 3 4 3 4 5 5 5 6 6 7 3 6 7

The next step is the equivalent molecular weight E M W determination. From the definition of moll fraction:

$$X_A = \frac{Wu / 18}{Wu/18 + T.S.\%/EMW}$$
 (2)

where the Wu is the unfrozen portion percent. The total solids in orange juice we worked with have been determined: T.S. =47.3%, and $H_20\%$ is 100 - 47.3 = 52.7%. So, EMW can be computed from (2). We found EMW = 101.28.

Using temperatures from $20^{\circ}F$ to $26^{\circ}F$ in equation (I), one value of X_A for each temperature was computed, and from equation (2), the unfrozen portion of the water was evaluated.

Since the original product contains 52.7% water, the percent water frozen will be:

$$(52.7 - Mu) / 52.7 \times 100$$

