Viscosity of Ice Cream Mix at Pasteurization Temperatures

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ABSTRACT

To calculate the holding times of ice cream mixes in the tubes of HTST pasteurizers, accurate viscosity data are needed for the temperatures and the shear rates of interest. Apparent viscosity was determined for ice cream mixes at 80°C using shear rate sweeps in a Carri-Med rheometer. Mixes varied in fat percentage, milk SNF, sweetener content, type of polysaccharide (guar, locust bean gum, carboxymethyl cellulose, xanthan), polysaccharide concentration, and blended proprietary stabilizer type and concentration. The effect of dissolution time was also studied. Data were fitted to the power law model, and the best estimate of flow behavior index was .7 for all mixes except those with xanthan gum, in which case the estimate was .5. The consistency coefficient is reported for all combinations described and provides useful data for equipment design and regulatory needs. The effect of the consistency coefficient on Reynolds number in holding tubes is also discussed.

(**Key words**: stabilizers, polysaccharides, viscosity, pasteurization

Abbreviation key: **n** = flow behavior index of the power law equation, **K** = consistency coefficient.

INTRODUCTION

We (8, 9) previously reported that ice cream mixes being processed through HTST pasteurizers under certain conditions of holding tube diameter and flow rate may exhibit laminar flow in the holding tube. In laminar flow, the ratio of mean velocity to maximum velocity is .5. In turbulent flow, the ratio of mean velocity to average maximum velocity is larger and depends on the extent of turbulence (near 1 for isotropic turbulence) (7). Turbulence may increase because of a temperature increase by its effect on reducing viscosity. At the same mean velocity, the minimum holding time for laminar flow is shorter than the minimum holding time for turbulent flow. This characteristic could potentially lead to insufficient holding time during pasteurization if adjustments are not made to flow rate to account for differences in viscosity, especially when holding time has been determined using a salt injection into water, which has a low viscosity. Pasteurization regulations vary by jurisdiction; however, all regulations state that every particle of milk or milk product must be held at a minimum temperature (e.g., 80°C for ice cream mix in Ontario) for a minimum time (e.g., 25 s for ice cream mix in Ontario) (11).

The flow pattern exhibited within pipeline flow can be estimated by calculation of the Reynolds number (5). One of the difficulties associated with appropriate calculations of Reynolds number is obtaining reliable data on ice cream mix viscosity at pasteurization temperatures as a function of composition and preparation procedure. Arbuckle (1) stated that mix viscosity is a function of composition (mainly fat, stabilizer, and salts), processing conditions (mainly pasteurization, homogenization, and aging), and temperature. He also suggested that mix viscosity comprises apparent viscosity, which dissipates with agitation, and basic viscosity, which ranges from 50 to 300 mPa·s. This "apparent" viscosity at rest or at low shear rate is partly due to phenomena (e.g., aggregation of fat globules) that cause shear rate thinning behavior and partly due to the thixotropic character of the mix. Arbuckle (4) did not define the shear rate at which basic

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Received November 10, 1993.
Accepted March 15, 1994.

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viscosity values were reported, however, and most stabilized ice cream mixes demonstrate non-Newtonian, pseudoplastic behavior (4, 9).

Cottrell et al. (4) studied the effects of sodium carboxymethyl cellulose, carrageenan, guar gum, and locust bean gum on ice cream mix viscosity between 30 and 0°C. They (4) demonstrated a nonlinear increase in apparent viscosity with concentration of all four stabilizers studied and used a power law model (Equation [2]) to describe these effects. The flow behavior index (**n**) of the power law model ranged from .8 at .1% to .48 at .3% for locust bean gum and from .94 at .1% to .68 at .3% for guar gum in ice cream mixes containing 38.75% total solids. Smith et al. (13) reported on the effect of sweeteners on mix viscosity at 2°C. Using the power law model, values were reported for the consistency coefficient (**K**) of between 4.8 and 6.7 $Pa{\cdot}s^n$ and for n between .48 to .55 in an ice cream mix containing 10% milk fat, 37.3% total solids, and .3% of a proprietary stabilizer blend. Another factor greatly influencing mix viscosity is the dissolution time of the polysaccharide in solution. Polysaccharide polymers interact with water and require time to become hydrated fully (2). Polymer hydration is reduced in the presence of sugars, leading to interactions between polymers and increases in apparent viscosity of the solution (6).

In the presence of non-Newtonian behavior, when viscosity varies with shear rate,

$$N_{GRe} = \frac{\rho D^n V^{2-n}}{K 8^{n-1} \left(\frac{3n + 1}{4n}\right)^n} \qquad [1]$$

where N_{GRe} = generalized Reynolds number, ρ = fluid density, D = diameter of tube, V = average velocity, and n and K are the power law parameters (7). To use Equation [1] to determine whether laminar ($N_{GRe} < 2100$) or turbulent flow may occur, the power law parameters for viscosity are needed.

Thus, ice cream mix viscosity is an important parameter in determining flow behavior, and many factors contribute to the viscosity of an ice cream mix. Hydrocolloids play an important role, and their interactions with water and other solutes are complex. The viscosity value that is critical for determining flow behavior in the holding tube of a continuous pasteurizer is at an intermediate step in the processing of ice cream mix and thus may not be the same as that determined when the mix is at equilibrium viscosity. Comprehensive data on ice cream mix viscosity at pasteurization temperatures are difficult to find. Thus, the purpose of this paper is to report the results of a study of mix viscosity at 80°C under typical processing conditions.

MATERIALS AND METHODS

Manufacture of Ice Cream Mixes

The standard formulation used for all preparation procedure studies consisted of 11% milk fat, 10.5% milk SNF, 12.5% sucrose, 2.5% corn syrup solids, and .3% stabilizer. Unstabilized cream (Wm. Neilson Ltd., Halton Hills, ON, Canada), instant skim milk powder (Gay Lea Foods, Guelph, ON, Canada), water, sucrose (Redpath Sugar, Toronto, ON, Canada), 42 dextrose equivalent coarse corn syrup solids (Casco, Inc., Etobicoke, ON, Canada), and guar gum (Food Specialties, Mississauga, ON, Canada) were used in the preparation of these mixes. The stabilizer was blended with the sugars and added to the prewarmed milk ingredients and water at 30°C. The mixture was then processed in one of two ways. Mixes of .5 kg were mixed with a laboratory mixer (Tissumizer, Terkmar, Cincinnati, OH) for 60 s and held at 30°C for 30 min, prior to rapid heating to 80°C in the cell of the rheometer. Mixes of 7 kg were held at 30°C for 20 min, heated to 50°C, homogenized in a two-stage Cherry-Burrell Superhomo (Chicago, IL) at 17.3 MPa (2500 psig) for first stage and 3.5 MPa (500 psig) for second stage, and then transferred to the rheometer for heating to 80°C within 30 min after blending. The holding time in each case simulated commercial blending conditions prior to HTST pasteurization. Variables that were examined included time of mixing (30 to 120 s), holding time prior to heating (0 to 30 min), mix temperature (30 or 80°C), and effect of homogenization.

The fat, SNF, and sweetener components of this standard formulation were varied in the following ways, holding all other compositional variables constant: fat content, 8 or 14%; SNF, 9 or 12%; corn syrup solids and sucrose, 0 and 15% or 5 and 10%. Mono- and diglycerides (.15%; Food Specialties), carrageenan (.02%; Food Specialties), and the following polysaccharides (.13 and .28%) were added to the standard mix: guar gum, locust bean gum, sodium carboxymethyl cellulose, and xanthan gum (Food Specialties). The following blended stabilizer-emulsifier preparations (.3 and .45%) were also added to the standard mix: Emulbilizer 300 (mono- and diglycerides, guar gum, Polysorbate 80, carrageenan, locust bean gum; Food Specialties); Essenoid HT84 (mono- and diglycerides, locust bean gum, carboxymethyl cellulose, guar gum, Polysorbate 80, and carrageenan; Food Specialties); and GS1143 (mono- and diglycerides, carboxymethyl cellulose, guar gum, Polysorbate 80, and carrageenan; Davis-Germantown, Scarborough, ON, Canada). All mixes were prepared in duplicate.

Viscosity Measurement

Viscosity was measured using a constant stress rheometer (Carri-Med CSL 100, Dorking, Surrey, UK) equipped with an aluminum Mooney-Ewart (Carri-Med) concentric cylinder geometry (4-cm diameter, 1-mm gap between cylinders, truncated cone, 2°C angle, .77-μm gap). The samples (8 ml) were heated to 80°C in the preheated Carri-Med concentric cylinder, which was connected to a circulating water bath to maintain the sample temperature. Heating time for the mix in the concentric cylinder was very rapid (2 to 3 s), as determined with a thermocouple. The gap was set for each sample while the entire measurement system was at 80°C, thus accounting for thermal expansion of the geometry. Shear rate sweeps from 20 to 200 s^{-1} were performed immediately after the sample was loaded into the rheometer.

Data Analyses

The power law model was used to characterize the viscosity of the experimental ice cream mixes for typical shear rates in pasteurization systems:

$$\sigma = K\dot{\gamma}^n, \qquad [2]$$

where σ = shear stress (millipascals), $\dot{\gamma}$ = shear rate (per second), K = consistency coefficient (millipascal seconds^n), and n = flow behavior index (3).

A nonlinear regression procedure (12) was used to calculate least squares estimates of the parameters K and n based on the shear sweep data over a range of shear rates from 20 to 200 s^{-1}. Because of the form of the power law model, interaction between the parameters occurred, and the estimate of one parameter was highly sensitive to the estimate of the other. This result also made it difficult to estimate the appropriate joint confidence region for the parameters. Asymptotic 95% confidence intervals for the estimates of n were estimated by the SAS regression procedure, and most included the value n = .7. For this group of samples, n was fixed at .7, and K was estimated by linear, least squares regression using the shear stress and shear rate data directly. Samples that did not exhibit this shear thinning behavior included the mixes containing xanthan gum (.13 and .28%). For these samples, the value of n was fixed at .5.

RESULTS AND DISCUSSION

To model the rheological characteristics at typical HTST temperatures (80°C), the effects of shear rate, temperature, sample preparation procedure, holding time, and measurement time on the flow behavior were investigated. All of the ice cream mixes, including the control mixes without polysaccharide stabilizers, exhibited non-Newtonian flow, as was expected (2, 4). Apparent viscosity increased as shear rate decreased over the shear rate range of 20 to 200 s^{-1}, indicating the importance of specifying shear rate conditions in any measurement or tabulation of ice cream mix viscosity data. Mix viscosity also decreased with increasing temperature, as was expected (1, 2). In the standard mix at 20 s^{-1}, viscosity ranged from 130 mPa·s at 30°C to 70 mPa·s at 80°C, and, at 200 s^{-1}, viscosity ranged from 70 mPa·s at 30°C to 30 mPa·s at 80°C.

The conditions of incorporation of stabilizer into the mix affected viscosity. When ingredients were mixed for 60 s with the laboratory mixer or homogenized in the pilot-scale homogenizer, the repeatability of the data was greatly improved, presumably through better homogeneity or better dispersion and faster dissolution of the powders. Mixing for 120 s with the laboratory mixer produced similar results to the 60-s mixing. With guar gum,

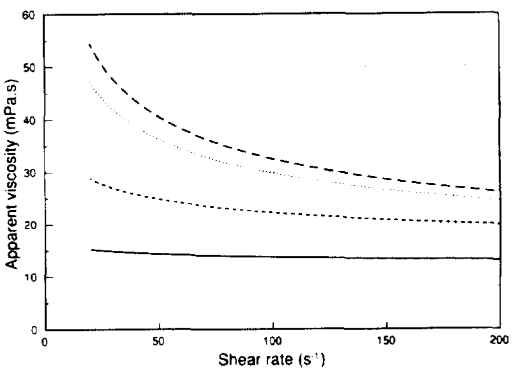

Figure 1. The effect of holding time of the blended standard mix at 30°C prior to heating on apparent viscosity, measured at 80°C. Holding times: 30 min (——), 20 min (· · ·), 10 min (– – –), or 0 min (----).

either the laboratory mixing procedure or homogenization led to the same viscosity shear rate profile; however, homogenization was necessary to incorporate fully many of the less soluble stabilizers.

A further complicating variable was the time needed for full dissolution of the stabilizer in the ice cream mix because viscosity increases as a result of hydration. Although it was important to allow the polysaccharides time to hydrate, it was also important to simulate commercial processing conditions. In a processing environment, up to 30 min may pass between the blending and the pumping of the final blended product through the HTST system. Mix viscosity of the standard mix at 30°C, measured at a constant shear rate of 100 s^{-1}, increased from 23 to 61 mPa·s over 20 min in the rheometer, probably because of the time of dissolution of the stabilizer. Water loss from evaporation at this temperature was not expected. Figure 1 shows the effect of holding time of the mix at 30°C on the apparent viscosity, measured at 80°C. Viscosity steadily rose from 0 to 30 min, but the differences between holding times were less as time increased. Thirty minutes seemed appropriate to allow full or nearly complete dissolution of the stabilizer at this temperature.

A final experimental variable to control was the measurement time in the rheometer. Viscosity at a constant shear rate increased more rapidly in the rheometer from 0 to 4 min at 80°C than at 30°C over 20 min. This effect may have been related to polysaccharide hydration and interactions between polymers (6) but may have also been due to evaporation of moisture from the sample. However, an increase in total solids in the mix as a function of time in the rheometer could not be demonstrated. In commercial practice, mix is only held at 80°C for a minimum of 25 s. Thus, shear rate sweeps were performed from 20 to 200 s^{-1} for all compositional variables in the up direction only over 2 min, and immediately upon reaching 80°C, as equipment limitations would not allow shorter times. Replicate sample variation exceeded the variation seen when up versus down sweeps were compared for the same sample. Shear rates at the wall within a commercial holding tube at typical processing conditions have been calculated as 20 to 200 s^{-1} (9).

The main objective of this research was to determine the effect of compositional variables on the rheological characteristics of the ice cream mixes at pasteurization temperatures and processing conditions. The power law model was used to describe the apparent viscosity as function of the shear rate raised to an exponent of $(n - 1)$. Thus, a solution with n less than unity exhibits decreasing apparent viscosity as the shear rate increases, which is referred to as pseudoplastic behavior. Not surprisingly, ice cream mixes containing complex polysaccharide stabilizers are pseudoplastic; particles may disaggregate, and the large polymer molecules tend to disentangle and possible align in the flow field as the shear rate increases, offering less resistance to flow (2).

Initially, nonlinear regression was used to estimate values for n and K simultaneously for each composition of ice cream mix based on the shear stress and shear rate data for two replicates. In all cases, the statistical analysis indicated high cross-correlation between the two parameters, and a joint confidence region for the two parameters was difficult to estimate. For most of the ice cream mixes, the estimates of n were between .66 and .74, and all 95% confidence intervals for the estimates included n = .7. Therefore, it was decided that n could be estimated to one significant digit, .7, which was used to describe the shear thinning behavior of most of the solutions containing polysaccharide stabilizers. Once the parameter n was fixed, the values of K for each ice

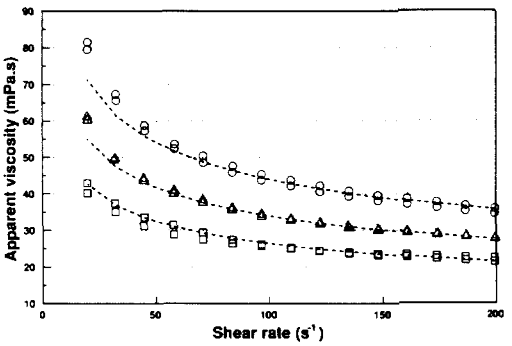

Figure 2. The effect of fat content of the mix on mix viscosity at 80°C. Fat: 8% (□), 11% (△), and 14% (○). Data points represent the results of both trials. The fitted curves according to the power law equation are shown by the dashed lines.

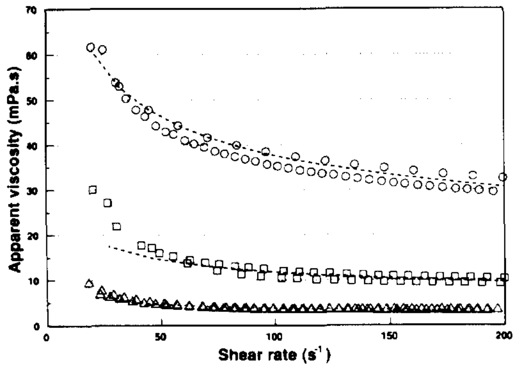

Figure 3. The effect of two percentages of guar gum on mix viscosity at 80°C compared with viscosity of a control mix with no stabilizer. Guar gum: 0% (control; △), .13% (□), and .28% (○). Data points represent the results of both trials. The fitted curves according to the power law equation are shown by the dashed lines.

cream mix were calculated by least squares linear regression using the shear stress and shear rate data directly.

The effect of increasing fat percentage on the apparent viscosity is illustrated in Figure 2. Figure 3 presents both experimental values and fitted curves for different percentages of guar gum in the mix. The control sample represented in Figure 3 contained .15% monoglycerides, diglycerides, and a minimal amount of carrageenan (.02%). The fitted curves in both figures are based on a flow index of .7 and the appropriate K value for the particular composition (including the control sample). The apparent viscosity increased substantially as typical commercial levels of polysaccharide (.1 to .3%) were incorporated. Both figures indicate that the assumption of a constant flow index gives a reasonable prediction of the effect of shear rate on apparent viscosity. Although experimental observations were made at low shear rates (<50 s^{-1}), the calculated apparent viscosities were subject to the largest experimental errors due to the low stresses involved.

The estimates of K for mix compositional variables are summarized in Table 1. Apparent viscosity increased as fat and SNF increased in the mix. However, in these experiments, the total solids also changed with the adjustments in fat and SNF. The effect of corn syrup solids was not statistically significant ($P > .05$) in the set of experiments conducted with total sweetener content of 15%. Although this result may not have been expected, the effects of corn syrup solids on increasing mix viscosity reported by others (2) may become more evident as mix temperature is lowered after pasteurization.

The estimates of K and n for the mixes containing the various polysaccharides are summarized in Table 2. As polysaccharide concentration was increased from .13 to .28%, apparent viscosity significantly increased with all polysaccharides. Carboxymethyl cellulose produced the lowest apparent viscosity at .28%, guar gum and locust bean gum were similar, and xanthan produced the highest apparent viscosity in the mixes at .13%. Also, xanthan produced a lower n, .5, than the other polysaccharides, no doubt because of its peculiar structure; demonstrated higher pseudoplasticity in solution (10); and showed a lower concentration dependence than the other polysaccharides, which as such is remarkable. In model solutions, such a difference is not observed. Monoglycerides and diglycerides (.15%), and carrageenan (.02%) were added to the polysaccharide mixes to bring them to levels similar to those in the blended stabilizers; the total percentage of stabilizer plus emulsifier was constant in each case. Table 3 summarizes the estimates of K for the blended stabilizers. All three blended stabilizers were based on proprietary combinations of guar,

TABLE 1. Effect of mix compositional variables on the consistency coefficient (K) as estimated by the power law model.[1]

Mix composition[2]	Estimated K $\bar{X}$	Estimated K 95% CI	r^2	Apparent viscosity at 100 s^{-1}
	(mPa·s^n)	(mPa·s^n)		(mPa·s)
Control[3]	135	1	.999	33.9
Fat				
8%	105	1	.995	26.4
14%	175	2	.994	43.9
SNF				
9%	128	2	.987	32.1
12%	180	7	.916	45.2
CSS[4]				
0% + 15% sucrose	131	2	.998	32.9
5% + 10% sucrose	150	20	.925	37.7

[1] Flow behavior index = .7. The 95% confidence interval (CI) for K and the r^2 value for the power law fit to the data are also shown. The apparent viscosity at 100 s^{-1} has been calculated from the estimates of the model. All mixes contained .3% guar.

[2] All other mix composition variables were held constant.

[3] Control contained 11% fat, 10.5% SNF, 12.5% sucrose, 2.5% CSS.

[4] Corn syrup solids.

locust bean gum, and CMC. However, the blended stabilizers produced a lower apparent viscosity than the pure polysaccharides, ranging from 7.8 to 26.9 mPa·s at 100 s^{-1}. The percentages of polysaccharide and emulsifier were likely different in each case.

The range of K when n = .7 for all ice cream mixes was from 31 to 180 mPa·$s^{.7}$, and apparent viscosity of mixes that included a polysaccharide stabilizer ranged from 7.8 to 45.2 mPa·s at 100 s^{-1}. The typical range of processing conditions for HTST systems includes processing rates of 1820 to 40,950 L/h (400 to 9000 Imp. gal/h) and holding tube diameters between 5.1 and 10.2 cm (2 and 4 in). For these conditions and mix characteristics, generalized Reynolds numbers (Table 4) were calculated as outlined by Goff and David-

TABLE 2. Effect of mix polysaccharides on the consistency coefficient (K) as estimated by the power law model.[1]

Mix composition[2]	Estimated K $\bar{X}$	Estimated K 95% CI	r^2	Apparent viscosity at 100 s^{-1}
	(mPa·s^n)	(mPa·s^n)		(mPa·s)
n = .7				
No stabilizer	15	.2	.905	3.8
.13% CMC	44	3	.997	11.0
.28% CMC	99	1	.997	24.9
.13% Guar	43	3	.938	10.8
.28% Guar	149	6	.984	37.4
.13% LBG	50	1	.991	12.6
.28% LBG	139	2	.994	34.9
n = .5				
.13% Xanthan	217	5	.914	21.7
.28% Xanthan	251	13	.791	25.1

[1] The 95% confidence interval (CI) for K and the r^2 value for the power law fit to the data are also shown. The apparent viscosity at 100 s^{-1} has been calculated from the estimates of the model.

[2] All mixes contained 11% fat, 10.5% SNF, 12.5% sucrose, 2.5% corn syrup solids, .15% mono- and diglycerides, and .02% carrageenan. CMC = Sodium carboxymethyl cellulose; LBG = locust bean gum.

TABLE 3. Effect of commercial blended stabilizers on the consistency coefficient (K) as estimated by the power law model.[1]

Mix composition[2]	Estimated K $\overline{X}$	Estimated K 95% CI	r^2	Apparent viscosity at 100 s^{-1}
	(mPa·s^n)			(mPa·s)
.30% E300[3]	35	4	.824	8.8
.45% E300	48	3	.911	12.1
.30% GS1143	31	.4	.980	7.8
.45% GS1143	43	.3	.896	10.8
.30% HT84	52	3	.925	13.1
.45% HT84	107	4	.972	26.9

[1] Flow behavior index = .7. The 95% confidence limits for K and the r^2 value for the power law fit to the data are also shown. The apparent viscosity at 100 s^{-1} has been calculated from the estimates of the model.

[2] All mixes contained 11% fat, 10.5% SNF, 12.5% sucrose, and 2.5% corn syrup solids.

[3] The compositions of Emulbilizer 300 (E300), Essenoid HT84, and GS1143 are listed in the Materials and Methods section.

son (9). These calculations indicated that the potential exists for laminar or transitional flow for mixes with the highest K over all processing conditions and also for mixes with low K in smaller systems (small holding tubes and low flow rates). Further research is being conducted on a pilot-scale HTST pasteurizer to determine the effects of mix properties and processing conditions on the holding time.

ACKNOWLEDGMENTS

Appreciation is expressed to the Ontario Ministry of Agriculture and Food, Ault Foods, Beatrice Foods, and Universal Flavors for financial support and to Helmuth Hager and Ashley Stone for technical assistance.

TABLE 4. Generalized Reynolds numbers (N_{GRe}) for low (K = 40 Pa·s^n) and high (K = 180 Pa·s^n) viscosity mixes at typical holding tube diameters and flow rates.[1]

Diameter	Flow rate	N_{GRe} K = 40 Pa·s^n	N_{GRe} K = 180 Pa·s^n
(cm)	(L/h)		
5.1	1820	1200	260
	4550	3800	840
6.4	4550	2400	540
	9100	8000	1800
7.6	11,375	5500	1200
	18,200	10,000	2300
10.2	18.200	6000	1300
	40,950	17,000	3700

[1] Flow behavior index (n) = .7.

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