

STEAM INJECTION FOR GLOBULE SIZE
REDUCTION IN AN EMULSION

Thesis for the Degree of Ph. D.
MICHIGAN STATE UNIVERSITY
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1962



This is to certify that the

thesis entitled

Steam Injection for Globule Size
Reduction in an Emulsion

presented by

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has been accepted towards fulfillment
of the requirements for

Ph.D. degree in Agricultural Engineering

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2000-10-10
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**STEAM INJECTION FOR GLOBULE
SIZE REDUCTION IN AN EMULSION**

By

Victor A. Jones

ABSTRACT

**Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of**

DOCTOR OF PHILOSOPHY

Department of Agricultural Engineering

Approval _____

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ABSTRACT

STEAM INJECTION FOR GLOBULE SIZE REDUCTION IN AN EMULSION

by Victor A. Jones

A 2.65% corn oil-in-water emulsion was recirculated through an injector where steam was introduced from four ports drilled tangential to the flow cross section and at a 30° angle with the flow path. The homogenizing effect was measured by the optical transmittance of 1020 mμ light through 1 cm of standardized diluted emulsion. Transmittance, as a measure of globule size distribution, was determined at several levels of steam energy input for various conditions investigated. Increased transmittance above a minimum level was indicative of globule size reduction.

The variable factors studied were steam-injection temperature, emulsion pressure at discharge from the injector, emulsion flow rate, and initial globule size.

Homogenizing effect increased substantially with temperature of injected dry saturated steam in the range 237° to 347°F. The 347°F steam-injection treatment of 175 Btu/lb of emulsion produced transmittances equivalent to 1800 psi homogenization in a laboratory-model conventional milk homogenizer.

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Victor A. Jones

An optimum emulsion pressure on the discharge side of the injector was observed, although emulsion pressure effects were much less pronounced than steam temperature. Increasing the discharge pressure from zero to 50 psi produced a slight increase in homogenizing effect; whereas, 80 psi decreased the size reduction at a given energy level when 347°F steam was injected.

The changes in velocity and Reynolds number obtained with different flow rates through a particular injector did not affect the size reduction of globules, as determined with the optical test with steam at 347°F.

Emulsions prehomogenized at zero, 1500, 3000, and 4500 psi in a conventional milk homogenizer underwent further size reduction when subjected to the 347°F steam treatment. Emulsion transmittance changed less per unit steam energy input as prehomogenization pressure increased, suggesting less efficient homogenization of small globule emulsions.

The globule size reduction accompanying steam injection was attributed to the cavitation effect produced by the rapid collapse of injected steam bubbles.

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Victor A.^{lan} Jones

A THESIS

**Submitted to
Michigan State University
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for the degree of**

DOCTOR OF PHILOSOPHY

Department of Agricultural Engineering

1962

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Doctor of Philosophy

Final Examination: October 15, 1962, Agricultural Engineering
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ACKNOWLEDGEMENTS

The writer is sincerely grateful for the opportunity of pursuing this study at Michigan State University. The writer desires especially to recognize and express appreciation to a few individuals for their interest, cooperation, and guidance.

Dr. C. W. Hall, Agricultural Engineering Department, as Chairman of the Guidance Committee, provided encouragement throughout this study. As a dedicated teacher and researcher, he will forever remain an inspiration to this student. The other Guidance Committee members, Dr. I. J. Pflug and Dr. G. M. Trout of the Food Science Department and Professor D. J. Renwick, Mechanical Engineering Department, contributed constructive criticism, suggestions, and encouragement.

The cooperation of Dr. A. W. Farrall, Head, Agricultural Engineering Department, and the entire Agricultural Engineering staff in making available facilities, equipment, and technical information is acknowledged with gratitude. The cooperative assistance of Drs. J. R. Brunner, T. I. Hedrick, C. M. Stine, and I. J. Pflug of the Food Science Department and Dr. R. S. Emery of the Dairy Department in providing additional equipment, instrumentation, and information was a major contribution.

The writer is indebted to his wife, Maryetta, and family for their understanding, patience, and moral support during graduate study.

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TABLE OF CONTENTS

	Page
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF SYMBOLS	x
INTRODUCTION	1
OBJECTIVES	3
LITERATURE REVIEW	4
Theories and Mechanisms of Homogenization	4
Energy Requirements for Homogenization	6
Steam Injection and Injectors	7
Effectiveness of Homogenization Tests	13
Microscopic examination	14
Fat separation tests	14
Turbimetric methods	17
Electric sensing-zone particle analyzing	21
ANALYSIS OF FACTORS THAT MAY INFLUENCE HOMOGENIZATION	
BY STEAM INJECTION	22
EQUIPMENT AND APPARATUS	26
Steam Injector	26
Steam-injection Homogenization System and Equipment	29
DEVELOPMENT OF EXPERIMENTAL METHODS	34
Emulsion Preparation	34
Determination of Effectiveness of Homogenization	36

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TABLE OF CONTENTS (Continued)

	Page
Emulsion Prehomogenization and Preliminary Optical Tests	38
PROCEDURE	50
CALCULATIONS	59
RESULTS AND DISCUSSION	67
Effect of Steam Temperature on Globule Size Reduction	67
Effect of Emulsion Pressure on Discharge Side of Injector on Globule Size Reduction	72
Effect of Flow Rate on Globule Size Reduction	77
Effect of Initial Globule Size on Globule Size Reduction	77
CONCLUSIONS	82
FUTURE RESEARCHES SUGGESTED BY THIS STUDY	84
REFERENCES	85
APPENDIX	93

LIST OF TABLES

Table		Page
1	Summary of steam-injection test variables	51
2	A sample data assembly and calculations table	66
3	Water meter calibration, I	94
4	Water meter calibration, II	95

LIST OF FIGURES

Figure		Page
1	Heat content per unit volume of steam at various temperatures	25
2	Photograph of the steam-injector assembly	26
3	Steam-injector assembly drawing	27
4	Steam-injection system and equipment	29
5	Schematic diagram of the steam-injector system	30
6	Relationship of transmittance and homogenization pressure of corn oil-water emulsion at various wave lengths following prehomogenization at 200 psi	39
7	Theoretical transmittance of milk with globules of uniform size	41
8	Relationship of transmittance and homogenization pressure of corn oil-water emulsion at various wave lengths following prehomogenization at 800 psi. (Results obtained with three different batches of emulsion for 1020 mμ wave length readings and two batches of emulsion for 700, 800, and 900 mμ wave length readings. Each point represents one test)	42
9	Transmittance as a measure of stability of emulsion homogenized in a conventional pressure homogenizer	44
10	Transmittance as a measure of stability of emulsion receiving steam-injection treatment	45
11	Effect of 24 hr dilution on emulsion transmittance	46
12	Effect of steam energy input errors on transmittance	47

LIST OF FIGURES (Continued)

Figure		Page
13	Comparison of transmittances of corn oil-water emulsion and of milk homogenized at various pressures. (Three batches of corn oil emulsion served as a source of emulsion for the tests. Each point represents one test)	49
14	Planimeter area factors for various temperatures on the 50° to 350°F potentiometer chart	58
15	A typical mass flow rate-temperature graph	61
16	Time-temperature plot produced on potentiometer chart showing various points used for calculations. (a) Complete test. (b) Expanded area between any two samples	62
17	Effect of steam energy input and steam temperature on emulsion globule size reduction by steam injection	68
18	Emulsion transmittance of four replicate steam-injection tests	69
19	Effect of steam temperature on emulsion size reduction by steam injection	71
20	Effect of steam energy input and emulsion pressure on discharge side of injector on globule size reduction, Test Series B	73
21	Effect of steam energy input and emulsion pressure on discharge side of injector on globule size reduction, Test Series C	74
22	Effect of emulsion pressure on discharge side of injector on globule size reduction. (Steam energy input--175 Btu/lb of emulsion)	75
23	Effect of emulsion flow rate on globule size reduction by steam injection	78
24	Effect of initial globule size on subsequent size reduction by steam injection	79
25	Rate of transmittance increase with steam energy input as a function of emulsion prehomogenization pressure	81

LIST OF SYMBOLS

- α = $2\pi r/\lambda$
- a = absorptivity (or extinction coefficient); the ratio of the absorbance to the product of concentration and length of optical path; $a=A/bc$
- a = acceleration, ft/sec²
- A = absorbance (or optical density); the logarithm of the reciprocal of the transmittance; $A=\log_{10} (1/T)$
- A_p = planimeter area, planimeter area units
- A_s = surface area of globules in an emulsion, cm²
- b = internal cell length, cm
- B = planimeter area factor, °F-min/unit planimeter area
- c = concentration
- C = specific heat, Btu/lb-°F
- d = prefix indicating derivative
- D = diameter, ft
- E = surface energy associated with a change in surface area, ergs
- γ = interfacial tension, dyne/cm
- G = water meter correction factor, actual volume in gal/gal meter reading
- h_f = enthalpy of liquid water, Btu/lb
- h_g = enthalpy of water vapor, Btu/lb

LIST OF SYMBOLS (Continued)

- i = number of any arbitrary sample under consideration or the number of the time interval between the i and $(i-1)$ samples; frequently used as a subscript
- I_{λ} = turbidity index at wave length λ ; $I_{\lambda} = yA/bx$
- k = thermal conductivity, Btu/hr-ft-°F
- K = turbidity per unit concentration of fat
- λ = wave length of light, $m\mu$
- μ = viscosity, lb/ft-sec or lb/ft-hr
- m = ratio of refractive indices m_1 and m_2 ; $m = m_1/m_2$
- m_1 = refractive index of the disperse phase of an emulsion
- m_2 = refractive index of the continuous phase of an emulsion
- M = water meter reading, gal
- ΔM = difference between two meter readings, gal
- n = number of the sample for which a calculation is being made
- π = 3.1416
- P = transmitted radiant power of an emulsion
- P_0 = transmitted radiant power of a reference cell (water)
- q = total steam energy treatment per unit weight of 2.65% emulsion, Btu/lb of emulsion
- q_a = steam energy treatment per unit weight of 2.65% emulsion immediately preceding the injector, Btu/lb of emulsion
- q_b = steam energy injected per unit weight of 2.65% emulsion in passing through the injector once, Btu/lb of emulsion
- ρ = density, lb/gal

LIST OF SYMBOLS (Continued)

- ρ_1 = density of continuous phase of an emulsion, lb/ft³
- ρ_2 = density of disperse phase of an emulsion, lb/ft³
- r = radius of globule, ft
- Σ = prefix indicating summation
- $\Delta\theta$ = time interval between meter readings, min
- t = temperature, °F
- t_A = arithmetic average of the preinjector and discharge temperatures at the midpoint of the time interval between samples, °F
- t_B = temperature average of the initial and highest temperature of the sample, °F
- t_D = temperature of the emulsion immediately after the injector, °F
- t_p = temperature of the emulsion immediately before the injector, °F
- Δt_n = temperature increase in passing through the injector when sample n was taken, °F
- T = transmittance, $T=P/P_0$
- V_g = specific volume of steam, ft³/lb
- V = velocity, ft/sec
- w = mass flow rate, lb/min
- x = fat percentage in undiluted emulsion, %
- y = volume dilution factor, volume of water/unit volume of emulsion
- Y = pounds of injected steam/pound of 2.65% emulsion
- z = wave length exponent

INTRODUCTION

The injection of steam into food products has become increasingly important in food processing operations in recent years. Steam injection has become especially useful for dairy and other liquid food products where steam comes into direct contact with products in flavor removal and in pasteurizing and sterilizing operations. Several investigators have observed a size reduction of fat globules associated with steam treatments. Practically no effort has been employed in studying or in utilizing this accompanying size reduction to emulsify or homogenize products, however.

With the trend toward higher temperature treatments of fluid milk products, the time requirements for bringing a product up to temperature and for cooling attain increased significance. Steam injection permits extremely rapid temperature increases without burn-on of product on heat exchanger surfaces. The vacuum cooling, which always follows steam injection in commercial processing of liquid foods to remove excess water resulting from condensed steam, is also a very rapid cooling process. When product quality factors such as flavor and shelf life are considered, steam injection becomes competitive with other heat transfer mechanisms.

Present homogenizers are less than 1% efficient in converting mechanical energy into surface energy associated with the increased surface area of the globules. Although homogenization costs are only a fraction of a cent per quart, the huge volume of product homogenized requires continuous investigation of more efficient processes.

Much of the work involving steam contact with foods has been done abroad, where homogenization is not practiced as widely as in the United States. More work has been done in reducing the homogenizing effect because of its detrimental effect in products such as cream where excessive butterfat is lost in making butter.

Possibilities for combining the homogenization process with pasteurization or sterilization offer challenging incentives for investigation of the homogenizing effect of injected steam. Fundamental information regarding heat transfer mechanisms may also be obtainable by observing the size reduction effects on the globules in an emulsion subjected to steam injection.

OBJECTIVES

- 1. To prepare an emulsion satisfactory for globule size reduction studies.**
- 2. To develop a method for determining the extent of size reduction of globules in an emulsion.**
- 3. To design a steam injector and assemble auxiliary equipment and instrumentation for studying globule size reduction in an emulsion.**
- 4. To investigate factors influencing the globule size reduction in an emulsion subjected to steam injection.**

LITERATURE REVIEW

Theories and Mechanisms of Homogenization

The first milk homogenizer was designed by Gaulin about 60 years ago and today nearly all of the 50 billion pounds of fluid milk consumed in the United States are homogenized. Product is forced through a small orifice with pressures near 2500 psi. Although numerous studies have been reported on various design factors influencing homogenization, the homogenization process is substantially the same as first introduced by Gaulin. The physical process by which homogenization is accomplished is still an unsolved phenomenon, however. Trout (1950) discussed five of the theories which have been advanced: explosion, impingement, shearing, acceleration and deceleration, and cavitation. A wiredrawing or attenuation theory has since been proposed by Wittig (1949, 1950, 1953). A brief description of each of the theories follows.

Explosion--rupture of the globule upon sudden reduction or release of pressure as the emulsion leaves the homogenizer valve.

Impingement, impact, shattering, or splashing--disrupting action caused by a high-velocity stream striking a solid surface.

Acceleration and deceleration--globule disruption attributed to violent changes in the velocity of the emulsion.

Shearing or grinding--tearing apart of the globule which protrudes into adjoining layers of a liquid that are traveling at different velocities.

Cavitation--shattering of globule caused by the instantaneous collapse of cavities formed when the increased velocity at the vena contracta is accompanied by a reduction in product pressure to its vapor pressure.

Wiredrawing or attenuation--friction between the liquid particles causes wiredrawing of the lipoplasts, and turbulence produces dissociation by displacement.

In recent years, other means of homogenization have received attention. Centrifugal force is employed by some European dairies to homogenize (Storgårds, 1956), and high voltage electrical energy has also been studied as a homogenization agent in tests in Russia (Surkov et al., 1959). The homogenization effect of sonic and ultrasonic waves has been studied by (Anderson, 1937; Chambers, 1937, 1938; Brown, 1941; Burger, 1954; Newcomer et al., 1957; Surkov et al., 1960; Pietermaat and Lescrauwaet, 1961; and Heide, 1961). The emulsifying action of ultrasonic waves is considered to result from cavitation, as first explained by Bondy and Söllner (1935).

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A cavitation effect produced by injecting superheated steam into various emulsions was reported by Polatski (1946) and Kudryavtsev (1959), and numerous investigators have noted a reduction in size of globules or a loss of butterfat in cream when direct contact of steam and product were used in deodorizing, pasteurizing, or sterilizing operations.

Energy Requirements for Homogenization

Wenrich (1946) analyzed the energy requirements for homogenization of milk based upon surface energy considerations. The surface energy associated with a globule surface area change is given by the equation,

$$E = \int_{A_S} \gamma dA_S,$$

where E = surface energy associated with the surface area change, ergs

γ = interfacial tension at the oil-aqueous phase interface, dyne/cm

A_S = surface area, cm^2 .

The surface area depends upon the number and size of globules. Reducing the globule size by a given fraction increases the surface area by the reciprocal of the same fraction if a constant volume of oil is assumed. Wenrich calculated an energy requirement of 38×10^{-6} erg or 3.6×10^{-15} Btu per globule to reduce a 4 μ globule to sixty-four 1 μ globules, based upon an interfacial tension of 25 dynes/cm. This is equivalent to the

kinetic energy associated with a 52.2 ft/sec velocity. The pressure required to produce this velocity head is 19.2 psi. He concluded that little of the energy employed in homogenization was utilized in subdividing the fat globules.

If all the fat in 1 lb of 4% milk were in 4 μ globules with a specific gravity of 0.9, the number of globules would be 6×10^{13} . The heat energy equivalent for homogenization would be 0.0216 Btu/lb of emulsion.

Although the waste of energy associated with the homogenization process has long been recognized, Mitten and Preu (1959) furnished additional experimental evidence that nearly all of the energy required for homogenization was used to overcome friction and was dissipated as heat. Temperature rise in a conventional pressure homogenizer was equal to the calculated temperature increase based upon the thermal equivalent of the hydraulic energy input.

Steam Injection and Injectors

Steam-injection applications have become increasingly important in food-processing operations in recent years. Steam may come into direct contact with liquid food products in flavor removal and in pasteurizing or sterilizing operations. As a side effect, several investigators have observed a size reduction of fat globules associated with steam-injection processing.

Sargent (1935) first noted that fat globules in Vacreator¹-treated cream had a smaller average diameter than globules in flash pasteurized cream from the same bulk supply. McDowall (1953), summarizing some earlier work, suggested the fat content of buttermilk was approximately 0.3% fat higher for vacuum-pasteurized than for flash-pasteurized cream. He stated (McDowall, 1953, p. 891) that "Treatment of cream in the Vacreator at very high intensities gives higher fat losses, probably due to excessive disruption of the fat globules." Intensity in this case referred to the amount of steam that condensed in the product. No attempt was made to correlate intensity with variations in fat losses. Dolby (1953, 1957a, 1957b) found that Vacreator treatment caused a considerable increase in the proportion of fat present as globules less than 2 μ in diameter (from 63 to 76% of globules or from 1.6 to 2.9% of weight). Breakup of the globules took place where cream and steam flow at high velocity. By modifying the steam inlet funnel to give less turbulence, globule sizes were not reduced as much. Howey (1959) reported no increased fat losses on churning steam-injected cream, but the cream was only heated to 105°F. The fat would not have been liquid when the steam was injected.

Mohler (1952) and Zollikofer (1952) mentioned the homogenizing effect of the Swiss uperization process. Mohler

¹Trade name of vacuum pasteurizer unit developed by Murray Vacuum Pasteurizer, New Zealand, and distributed in the United States by Cherry-Burrell Corp., Cedar Rapids, Iowa.

suggested that size reduction of globules resulted from expansion following steam injection. Grosclaude (1960) reported that 86% of the fat globules in milk were less than 2 μ in diameter after processing in a Laguilharré sterilizer. Kaliba et al. (1960a, 1960b) described a steam-injection unit that produced vibrations of 2 to 12 kc/sec during milk processing and that broke up fat globules. The homogenization effect in cream was so pronounced that a significant increase resulted in butterfat content in buttermilk. An increase in the Gibson and Herreid (1958) effectiveness of homogenization index, indicating an increase in size of globules, was reported by Graves et al. (1962) when milk was subjected to Vac-Heat¹ treatment. This test is not sensitive to size reduction, if this takes place simultaneously with size increase, as suggested by Dolby (1953, 1957a, 1957b). Hedrick and Trout (1959) also reported an increase in the fat-plug formation on milk treated in a Vac-Heat unit, but did not investigate size reduction effects.

Although the above investigators reported on size reduction associated with steam processes, Polatski (1946) was the first to investigate the utilization of injected steam for emulsification and size reduction. A number of oil-in-water emulsions were treated by injecting superheated steam at 150°C (saturation temperature, 132°C) through a 0.5 mm opening

¹Trade name of a vacuum unit manufactured by Creamery Package, Chicago, Ill.

into a flask of emulsion held at 18°C. Maximum size of globules following treatment was 3 μ . Saturated steam also gave a homogenizing effect, but superheated steam was reported as more effective. The basis of comparison for stating that superheat was more effective was not given. Although these tests were conducted with very small samples, Polatski suggested steam injection as a method for obtaining highly dispersed pharmaceutical emulsions and suspensions and for preparing agricultural insecticide emulsions.

Kudryavtsev (1959) also investigated the dispersing action of superheated steam in an oil-water emulsion. Steam under 0.2 atm (2.94 psig) pressure injected for 0.5 min through jets of 0.6 to 1.3 mm diameter into a layer of 0.3 ml of transformer oil on 15 ml of water produced a finely dispersed emulsion. Dispersion of the oil droplets appeared to be little affected by jet diameter. When oil and water were fed into a multistage system of overflow vessels, each equipped with a steam injector, most of the droplets were reduced to less than 5 μ diameter on reaching the third vessel. Sixteen to 18 g of steam/kg of emulsion were used in each vessel and the emulsion temperature increased from the 18°C feed temperature to 28°, 38°, and 48°C in the successive vessels. Steam-port diameter had little effect on globule size.

Studies of the collapse of injected steam bubbles or vapor bubbles produced by other means include theoretical analyses, high-speed photography, and acoustical

investigations. Rayleigh (1917) proposed equations to estimate the instantaneous local pressure developed when a spherical cavity collapses in a liquid. This analysis was based upon initial bubble size and the rate of collapse. Plesset and Zwick (1954) and Zwick and Plesset (1954) expanded Rayleigh's theory by relaxing some of the simplifying assumptions and by considering heat transfer effects.

Mohler (1952) observed both sonic and ultrasonic waves resulting from steam-bubble condensation. A microphone and oscillograph were employed to record ultrasonic noise intensity. Mohler reported that increased temperature differences between liquid and injected steam increased the intensity. Guth (1954), by photographing the collapse of single-injected steam bubbles at speeds up to 65,000 frames/sec and by measuring the sound frequency, found that the fundamental tone frequency corresponded to the number of bubbles leaving the nozzle. Shock waves produced by the collapse of the steam bubbles were observed with Schlieren photographs. Kustova and Kudryavtsev (1959) studied the impact waves originating from collapsing steam bubbles. The pressure of the impact wave increased almost linearly with increase in the steam-injection nozzle diameter and with steam pressure.

Levenspiel (1959) produced vapor bubbles by boiling at below atmospheric pressures. The collapse of the bubble upon subjection to atmospheric pressure was observed by photographs. The rate of bubble collapse increased with

instability temperature difference, defined as the difference in boiling points before and after the pressure change. Morgan (1960) found sound frequency decreased with increasing temperature differentials between liquid and injected steam. Correlation of frequency or intensity of waves with homogenization effects has not been attempted.

Mohler (1952) suggested that shape and angle of the injected steam to the liquid flow were important but did not present enough data to permit conclusions to be drawn. Several sizes of nozzles were used, but again no conclusions were reached.

Brown et al. (1951) designed a steam injector for food products for which Morgan (1960) and Morgan and Carlson (1960) investigated heat transfer rates, sound frequency associated with the injector, and various conditions for optimum operation. Morgan and Carlson's suggestions for successful operation of this heater, in which steam was injected tangential to the liquid flow stream, were summarized as follows:

1. Liquid flow should be turbulent in the injector, preferably with Reynolds number above 10,000.
2. Liquid flow rate should be independent of pressures in the injector.
3. Pressure within the injector must be between 3 and 15 psi higher than the saturation pressure for the attained temperature.

4. Heat transfer surfaces with steam on one side and liquid on the other side should be avoided.
5. The axis of the steam jet into the liquid should not form a right angle with any of the solid surfaces containing the liquid.
6. The system should be as free as possible from non-condensable gases.

Initial vapor-bubble size influences the rate of collapse, as hypothesized by Rayleigh (1917) and supported by investigations of Knapp and Hollander (1948), Harrison (1952), and G  th (1954). An injected steam bubble splits into several smaller bubbles (G  th, 1954; Kornfeld and Suvorov, 1944). It is the smaller bubbles which collapse in accordance with the Rayleigh theory.

Effectiveness of Homogenization Tests

The term "homogenization efficiency" is used in two entirely different senses. First, this term is used as a measure of the ability of globules to remain uniformly distributed in a sample; and, second, it is used as a measure of the energy consumption in homogenization. In this paper, the term, "homogenization effectiveness," will be used for the former. "Homogenization efficiency" will be restricted to energy relationships.

Numerous tests have been proposed for determining the effectiveness of homogenization or size distribution of fat globules in fluid milk products. These methods can be grouped

into the following four categories, based upon principle of operation.

1. Microscopic examination
2. Fat separation tests
3. Turbimetric methods
4. Electric sensing-zone particle analyzing

Microscopic examination

Trout (1950) and Dolby (1953) have reviewed the various techniques employed for microscopic testing, including type and extent of dilution, depth of film, and direct and photographic counting. Pearce and Seagraves-Smith (1957) suggested a new staining technique for providing better detail of globules. In general, the microscopic methods require a skilled and experienced technician; they are subject to sampling errors, especially in measuring the small number of large globules containing a large proportion of the fat in relation to their weight; and thousands of globules must be measured to determine size distribution. The Farrall Homogenization Index (Farrall, 1941) requires relatively few measurements, since only globules 2 μ in diameter and over are measured, but it does not provide complete size distribution data.

Fat separation tests

Fat separation tests are based upon the tendency of fat globules to rise because of their lesser density as compared

with the aqueous portion of milk. The velocity of rise for a given size globule is given by Stokes' law:

$$V = 2ar^2(\rho_1 - \rho_2)/9\mu,$$

where V = velocity of rising particle, ft/sec

a = acceleration, ft/sec²

r = radius of globule, ft

ρ_1 = density of serum, lb/ft³

ρ_2 = density of fat, lb/ft³

μ = viscosity of serum, lb/ft-sec.

Trout (1950) reviewed the tests in use up to 1949. These included the U.S. Public Health Service (USPHS) (1953) test with 48 hr of quiescent storage and the accelerated centrifugal tests, each of which has numerous modifications.

Wittig (1950) and Ridgeway (1957) have described burette methods similar to the USPHS method except that the fat content of the original and of the lower portion is determined.

The centrifugal methods can be divided into those requiring a fat test of a specific portion of the sample following centrifugation and those read directly as the volume of cream in a Babcock milk test bottle after centrifugation.

Maxcy and Sommer (1954) determined the percentage enrichment of the top 10 ml of a 50 ml homogenized milk sample after centrifuging under prescribed conditions. Seiberling (1955) compared this method with the Snyder and Sommer (1942, 1943) method, the USPHS method, and the Farrall Index (Farrall,

1941) method. Maxcy and Sommer also used a gravity fat separation test with evaporated milk for determining the percentage enrichment of the top portion removed in a specified manner. Pearce (1959) adapted Maxcy and Sommer's centrifugal test for use with low-viscosity evaporated milk.

Dolby (1957a) used a centrifugal test on 35 to 40% cream diluted with 60°C water to 10% fat for estimating the proportion of the total fat as small globules (1 μ and under). Dolby also employed a modified Andreasen particle-size apparatus to estimate the fat present in large globules (7 to 8 μ and over). By sampling with a pipette 1 cm from the bottom of the flask, he determined the proportion of fat rising 1 cm in 60 min when the cream was diluted to 10% fat and held at 60°C.

The centrifugal method of Snyder and Sommer (1942, 1943), using a standard Babcock milk test bobbie, was modified by Gibson and Herreid (1958) by adding 1 ml of Sudan III solution to give a more distinct cream volume. They also used a lower temperature (110°F) and centrifuged only once. Each 0.1% cream volume was approximately equal to 1.0% difference in the USPHS index.

Most fat separation tests give a homogenizing index or value but do not give much information concerning the size distribution of globules. Puri et al. (1952) applied Stokes' law to find the size distribution by taking samples in a flask at various depth and time intervals.

The primary advantage of the centrifugal methods for determining effectiveness of homogenization is that this test can be conducted immediately following homogenization.

Turbimetric methods

The theoretical foundation for turbimetric studies was established by Rayleigh's classical theory of light scattering and on the subsequent generalization by Mie (1908). A detailed discussion of light-scattering theory is presented by van de Hulst (1957). Factors affecting light scattering by a dilute suspension of uniform particles include

1. Radius of particle, r
2. Wave length of light in medium surrounding particles,
 λ
3. Refractive index of the disperse phase of an emulsion, m_1
4. Refractive index of the continuous phase of an emulsion, m_2
5. Number or concentration of particles
6. Length of light path in suspension, b
7. Angle between direction of propagation of scattered light and reversed direction of incident beam.

Factors 1, 2, 3, and 4 can be grouped into two parameters,

$$m = m_1/m_2; \quad \alpha = 2\pi r/\lambda.$$

Haugaard and Pettinati (1959) claim that potential sources of error caused by the small natural variations in m , the ratio

of refractive indices, can be disregarded if the sample is properly diluted. Factors 5, 6, and 7 can be held constant or corrected, thus leaving particle size, r , and wave length, λ , as important factors in determination of size by optical means.

Turbidimetric methods for determining effectiveness of homogenization have been proposed by Ashworth (1951), Lagoni and Merten (1955, 1956), Deackoff and Rees (1957), Goulden (1958c, 1958d, 1960), and Haugaard and Pettinati (1959). In addition to fat globules, casein micelles also act as scattering agents in optical investigations. Their effect can be overcome by dissolving the micelles with ammonium hydroxide (Ashworth, 1951; Deackoff and Rees, 1957), with sodium hydroxide or other alkali (Goulden, 1958d; Písecký, 1955) or with chelating agents (Haugaard and Pettinati, 1959). Deackoff and Rees (1957) and Rees (1962) used the longer wave lengths to reduce or nullify the scattering effects of casein micelles and reduced the color effects of milk. Leviton and Haller (1947) also demonstrated that scattered radiation of casein micelles decreased at longer wave lengths. Ashworth (1951) calculated a "K-value," turbidity per unit concentration of fat, from transmittance values obtained with a 515 m μ filter in a colorimeter. He correlated K-values with homogenization pressure; percentage of milk homogenized in a mixed sample, a known portion of which had been homogenized; and effectiveness of homogenization values obtained by the USPHS method.

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Lagoni and Merten (1955, 1956) based their test upon the Mie theory relationship that z in the equation,

$$a\lambda^z = \text{constant},$$

where a is absorptivity and λ is wave length in millimicrons, is a function of particle size. " z " was determined from the slope of the $\log a / \log \lambda$ curve at wave lengths from 380 to 800 $m\mu$ for various homogenization pressures and dilution rates.

Deackoff and Rees (1957) determined transmittance at wave lengths between 660 and 1020 $m\mu$ for various homogenization pressures, and compared these values with effectiveness of homogenization values obtained by the USPHS method. They found 1020 $m\mu$ light to be most sensitive to homogenization pressure changes.

Goulden (1958d) expressed effectiveness of homogenization by a turbidity index, I_λ , determined at wave length λ and defined as

$$I_\lambda = \frac{y}{bx} \log \frac{P_0}{P} = \frac{y}{bx} A,$$

where y = volume dilution factor, volume of water/unit
volume of milk

b = light path length in emulsion, cm

P = transmitted radiant power of emulsion

P_0 = transmitted radiant power of water

x = fat percentage in undiluted milk, %

A = absorbance.

Calibration-corrected I_{900} values ranged from 14 to 20 for commercially homogenized milk, as measured with a colorimeter with a filter of maximum sensitivity at 900 m μ . In another test, undiluted milk samples in thin absorption cells were checked for transmission in the wave length region from 900 to 2400 m μ . Results by this method were compared with results of tests by the Deackoff and Rees method and Lagoni and Merten method. The Deackoff and Rees method was the most sensitive and the Lagoni and Merten method the least sensitive to homogenization conditions at pressures used for commercial homogenization. In a later test, Goulden (1960) claimed his " I_{λ} " values to be more reproducible than effectiveness of homogenization values obtained by the Ridgeway (1957) centrifugal method. Goulden (1958b) also investigated various factors influencing turbidity tests and showed that scattering maxima in the wave length range 450 to 1050 m μ became more intense and moved to shorter wave lengths as globule size was reduced.

Haugaard and Pettinati (1959) measured parallel transmitted and total transmitted light from a 500 m μ layer of milk diluted one part in four with a solution containing chelating agent and emulsifier. Both equivalent homogenization pressure and percentage of fat content of the sample were read from a prepared nomogram with reasonably good accuracy. Transmitted light was read at 600 m μ with a milk glass standard reference. The effect of instrument design factors on turbidity measurements was studied by Goulden (1960). He emphasized the importance of small angles of acceptance at the detector.

The ease of conducting the test and the short time required are the primary advantages of the turbidity test. The Pettinati and Haugaard (1959) test required but 5 min for effectiveness of homogenization as well as fat content determination.

Electric sensing-zone particle analyzing

A relatively new instrument offers perhaps the most promise in evaluating size distribution of globules, at least from a research standpoint (Orr and DallaValle, 1959; Ulrich, 1960; Kinsman, 1962). Particles are diluted in an electrolyte and forced through a small orifice with an electrode on either side. An electrical pulse is created as the conduction changes during the passage of a particle through the orifice. Large numbers of particles can be counted in a very short time and the size distribution by number of particles, weight, or surface area can be calculated.

ANALYSIS OF FACTORS THAT MAY INFLUENCE HOMOGENIZATION BY STEAM INJECTION

The cavitation effect associated with the collapse of a vapor bubble in a liquid is a complex phenomenon. Although studies have been reported on the formation and collapse of vapor bubbles, including size, rate of growth and collapse, sound frequency associated with steam injection, and shock waves accompanying the collapse, the limited information available is primarily empirical in nature and laws governing the phenomenon are nonexistent.

From the information concerning steam injection, bubble collapse, instantaneous local pressures, and accompanying shock waves, as previously described, the speed of bubble collapse, the proximity of the globules to a collapsing bubble, and the air content of the steam or product were recognized as potential sources of influence on any homogenizing effect of steam injection. Outlined below are some of the factors that may affect each of these.

I. Speed of bubble collapse

A. Initial heat content of the newly formed bubble

1. Size of the bubble

a. Steam properties (see A2 below)

b. Product properties (see A3 below)

- c. Size, shape, number, and placement of steam-injection ports
 - 2. Steam properties
 - a. Temperature
 - b. Pressure
 - c. Quality
 - d. Superheat
 - e. Air content
 - 3. Product properties
 - a. Temperature
 - b. Pressure
 - c. Velocity
 - d. Reynolds number
 - e. Air content
 - f. Thermal properties
 - 4. Rate of heat transfer from developing bubble (same as for B below)
- B. Rate of heat transfer from bubble
- 1. Bubble size and shape (see A1 above)
 - 2. Reynolds number of product ($VD\rho/\mu$)
 - 3. Prandtl number of product (C_p/k)
 - 4. Temperature differential between bubble and liquid
 - 5. Air content of bubble or product
- C. Pressure differential between bubble and liquid
- 1. Steam and product thermal properties and factors affecting heat transfer as outlined above (see B)
 - 2. Pressure applied upon liquid

- 3. Injector design affecting flow characteristics
 - D. Air content of steam and product
 - E. Initial size of steam bubble (see A1 above)
- II. Proximity of collapsing cavity to globules
 - A. Injector design
 - B. Rate of heat transfer from developing bubble (see IB above)
- III. Air content of steam and product (absorption of shock waves)

The easily controlled factors, such as steam temperature and pressure and product temperature, pressure, velocity, etc., are seen to influence the bubble collapse in a number of ways. For example, a low-pressure steam would appear to be desirable for a bubble of minimum heat content per unit volume, as indicated in Fig 1. On the other hand, a high-temperature steam appears advantageous for rapid collapse of bubbles from a heat transfer standpoint. Similar contradictory conclusions can be drawn for many of the factors listed above. The interaction of these variable factors may further complicate analysis of experimental data. It is conceivable that a change in one variable of a given set of optimum conditions for homogenization could require changing a number of other variables for successful operation.

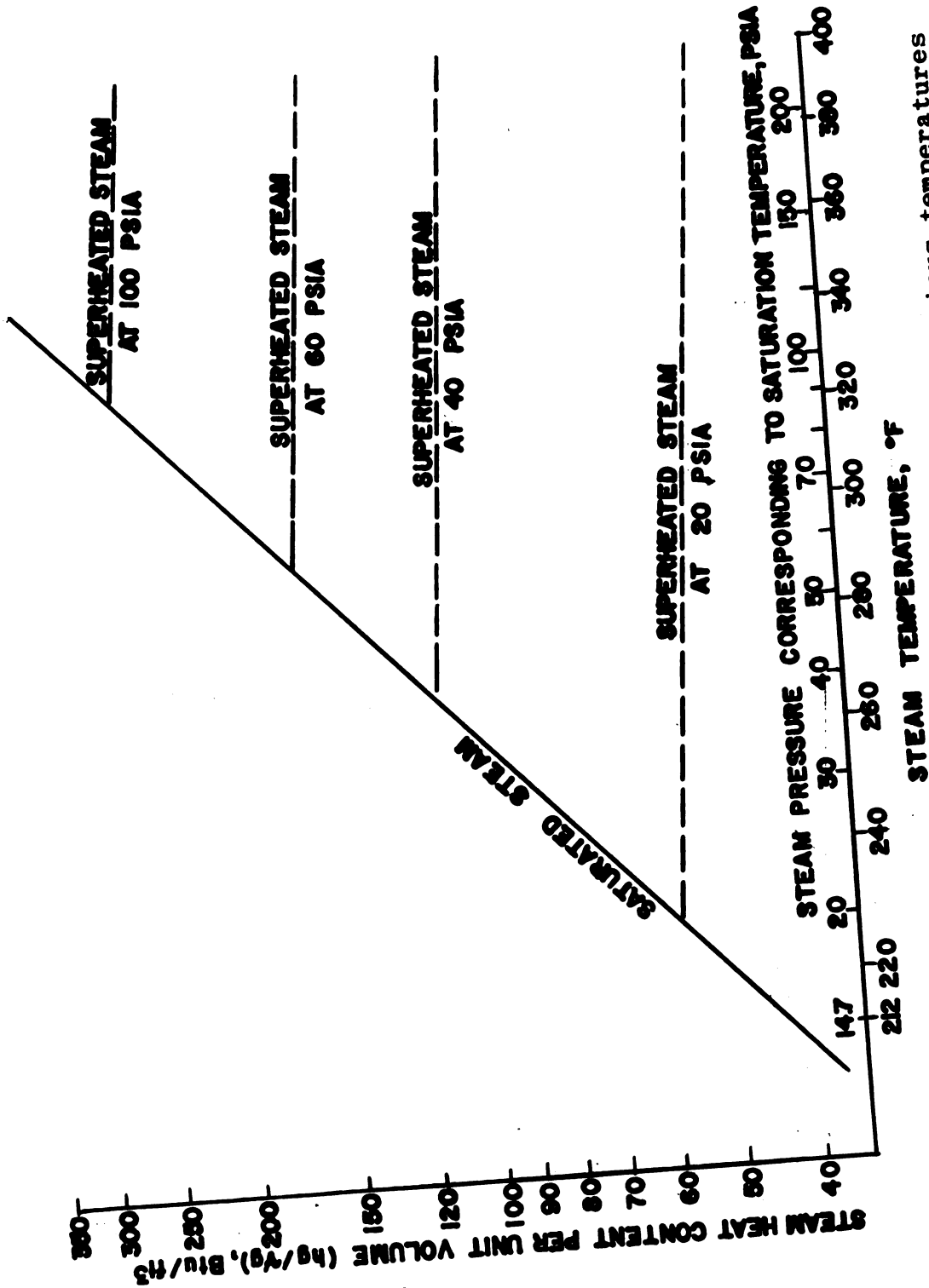


Figure 1. Heat content per unit volume of steam at various temperatures

EQUIPMENT AND APPARATUS

Steam Injector

An aluminum steam injector was constructed to fit into a 1.5 in. stainless steel sanitary "T," as shown in Fig 2 and 3. The emulsion flow cross section was reduced to 0.25 in. diameter by a 60° included angle entrance. Steam was fed to the injector through the perpendicular portion of the sanitary "T." It was injected into the emulsion at the 0.25 in. diameter constriction through four 0.0625 in. diameter steam

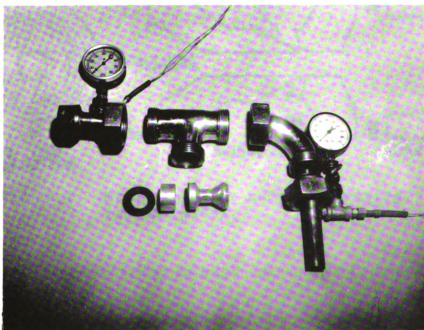


Figure 2. Photograph of the steam-injector assembly

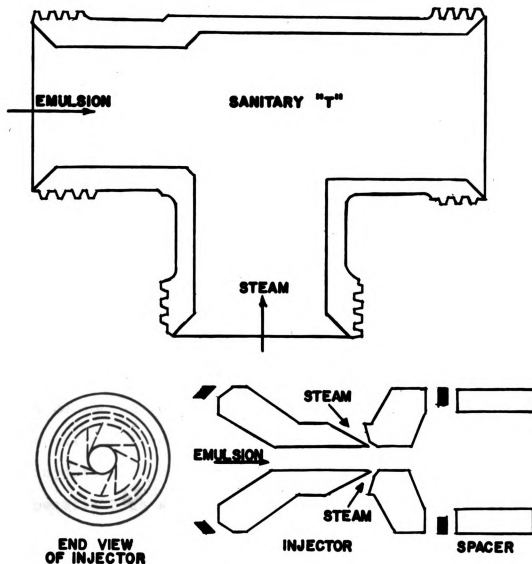


Figure 3. Steam-injector assembly drawing
(actual size)

ports. The steam-injection ports were drilled at a 30° angle to the flow path of the emulsion and tangential to the emulsion flow cross section. About 0.25 in. downstream from the steam ports, the cross section was expanded to 1 in. diameter by a 120° included angle.

With emulsion flow rates of less than 1 gpm, Reynolds numbers above 10,000 as suggested by Morgan (1960) for steam injectors are realized. The reduced cross-sectional area at the injection ports produces a low-pressure area and may permit steam to enter with less heat per unit volume. The reduced area at the injector may also cause steam bubbles to be formed closer to any given oil globule than would a larger cross-sectional area. The 60° entrance angle was incorporated in the design to reduce pressure drop through the injector and yet permit construction with available equipment. The tangential injection of steam also follows Morgan's recommendations. This permits high heat-transfer rates without objectionable noise. Although Mohler (1952) suggested that angle of injection affects shock wave intensity, no evidence was given as to what the effect was. The 30° angle was arbitrarily selected. Because pressure on the steam bubble would appear to speed collapse, the downstream expansion of the cross section will tend to increase pressure and possibly aid in the collapse of the steam bubble.

Steam-Injection Homogenization System and Equipment

Fig 4 and 5 show a photograph and a schematic diagram of the equipment and system used in the homogenization studies. Steam was produced by a Dutton Econotherm¹ 20 hp boiler whose pressure could be regulated by thermostatic control to any level up to 115 psi. The emulsion was pumped from a 5-gal milk can used as a reservoir by a Model TX10 Union Triplex² pump with a Varidrive³ motor. The flow rate

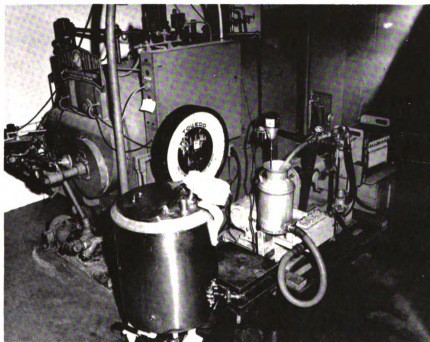


Figure 4. Steam-injection system and equipment

¹Manufactured by C. H. Dutton Co., Kalamazoo, Mich.

²Manufactured by Union Pump Co., Battle Creek, Mich.

³Manufactured by U.S. Electric Motors, Inc., Milford, Conn.

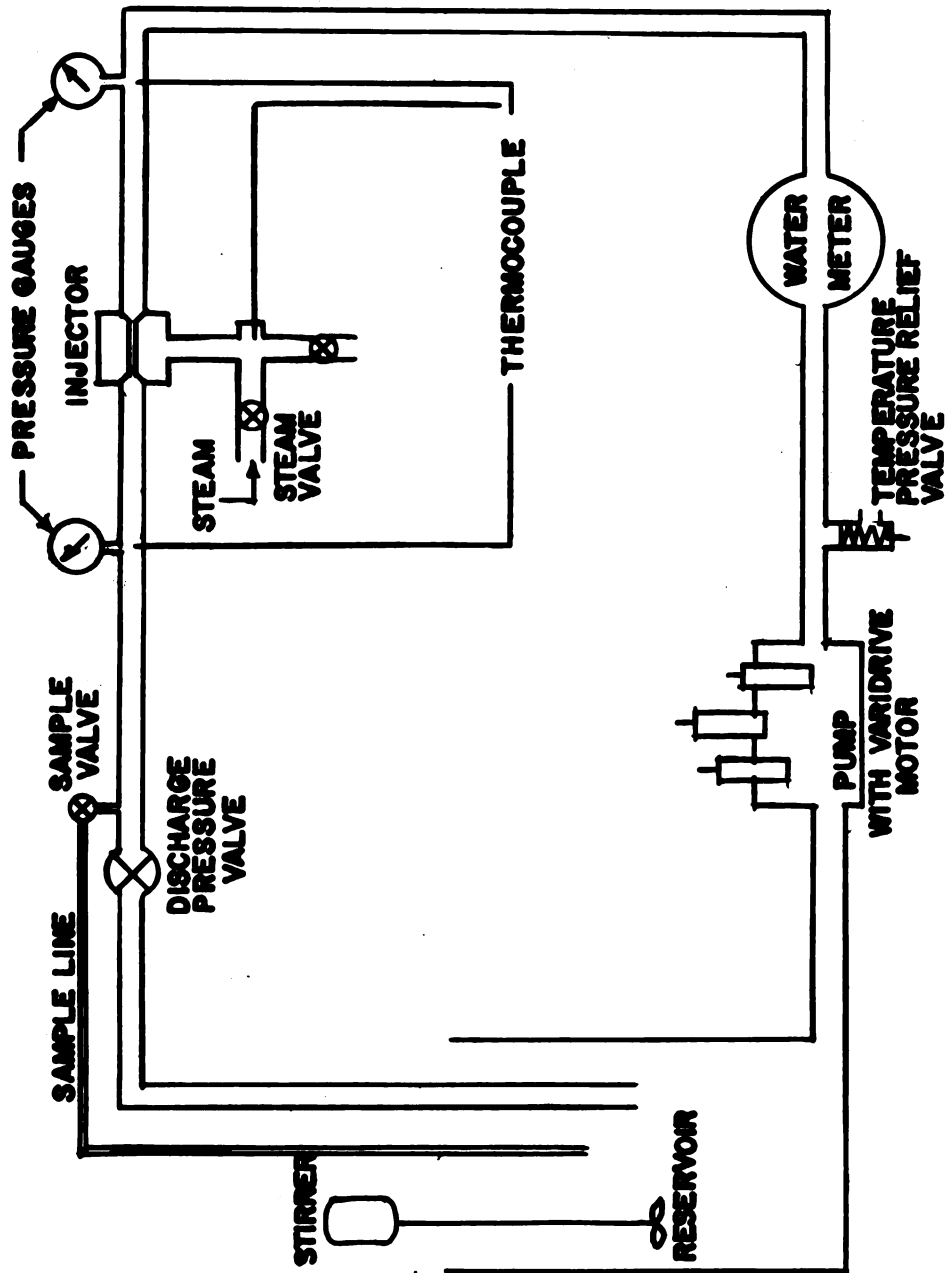


Figure 5. Schematic diagram of the steam-injector system

could be varied from approximately 1.5 to 10 gpm with pressures up to 100 psi. From the pump, the emulsion bypassed a temperature and pressure relief valve and flowed through a Badger¹ hot-water meter which could be read to the nearest 0.1 gal. Emulsion in the reservoir was kept thoroughly mixed with a small mechanical stirrer. A 200 psi Bourdon tube pressure gauge and a copper-constantan 24-gauge wire thermocouple located in the center of the 1 in. sanitary pipeline were installed between the meter and injector. Steam was delivered to the injector through the perpendicular portion of the 1.5 in. sanitary "T" from below to reduce the possibility of movement of any entrained water droplets into the emulsion through the injector. A stand pipe directly below the injector provided space for condensed steam. A second copper-constantan thermocouple for measuring the steam temperature was located between the injector and the stand pipe, approximately 4.5 in. below the steam ports in the injector. A third thermocouple, similar to the other two, was located at the center of the 1 in. sanitary tube leading from the injector and approximately 2.625 in. downstream from the injector ports. Brown et al. (1951) had shown previously that a thermocouple 2.5 in. downstream from the injector gave the same temperature as thermocouples located 11.5 in. downstream with considerably higher temperature increases than recorded in

¹Manufactured by Badger Mfg. Co., Milwaukee, Wis.

this paper. A Bourdon tube 80 psi pressure gauge was located at the same position as the downstream thermocouple. A valve for withdrawing samples from the system was placed immediately before a 1 in. pipe globe valve used to control the emulsion pressure on the discharge side of the injector. Thermocouples were attached to a 12-point Brown-Electronik¹ recording potentiometer with a temperature range of 50° to 350°F and set to record 1 point every 5 sec. The preinjector thermocouple was connected to recording points 1, 3, 5, 8, and 10. The steam temperature thermocouple was connected to point 7 and point 12 and points 2, 4, 6, 9, and 11 were attached to the thermocouple on the discharge side of the injector.

The accuracy of the water meter was tested by pumping water through the meter at various flow rates, temperatures, and pressures. Flow was diverted to a weigh tank simultaneously with an initial meter reading. A final meter reading was taken when approximately 20 gal had flowed through the meter, and the flow was diverted from the tank at the same instant. The volume of water, based upon its weight and density, was calculated. The results obtained immediately following installation of the meter are presented in Table 3 (Appendix). The meter correction factor, G, the ratio of gallons based upon weight to gallons meter reading, was 1.035 ± 0.02 for pump settings of 3 and above (flow rates of

¹Brown Instrument Division, Minneapolis-Honeywell Regulator Company, Philadelphia, Pa.

3.4 gpm and above). The meter factor increased at lower rates, indicating some slippage. Temperatures in the range of 56° to 160°F and pressures up to 80 psi had little influence on the factor.

Flow rate, based upon the meter readings, decreased substantially with temperature in preliminary steam-injection trials. Therefore, a second meter calibration test was conducted at various temperatures to determine if the meter was malfunctioning or if the pump output was varying. The results of this test are presented in Table 4 (Appendix). Only 1.4% difference in meter factors in the temperature range of 65° to 190°F resulted. The meter factor was slightly higher than the previous test indicated, but the meter has been used considerably. For calculation of flow rates, a meter calibration factor of 1.05 was used for all subsequent steam-injection tests.

DEVELOPMENT OF EXPERIMENTAL METHODS

Emulsion Preparation

An emulsion with the following properties was desired for investigation of the homogenizing effect of steam injection:

1. Physically stable emulsion; no change of globule size distribution with time
2. Oil globules in liquid state at room temperatures
3. Nonperishable
4. No proteinaceous or other ingredients with particles which could influence homogenization tests
5. An emulsion which could be prepared with relatively large globules
6. Inexpensive.

Emulsion physical stability was desired to facilitate homogenization effectiveness tests and analyses. No emulsion systems' globule size distribution remains constant indefinitely. The time of testing for effectiveness of homogenization becomes an important factor in analyzing data for an unstable emulsion with rapidly changing globule size distributions. Minimal instability is imperative if time corrections are to be avoided. Information concerning emulsion stability is presented later in this section.

Corn oil was selected as the oil phase because of its low melting point (0° to 14°F) (Eckey, 1954) and because it was a readily available, edible oil which could be used without

objection in sanitary equipment. It is well-known that oils must be in a liquid state for efficient homogenization in conventional homogenizers. A low melting point oil was desired for this study for two reasons. First, preheating of the emulsion was avoided by starting with an emulsion whose oil phase was liquid. Second, heat transfer analysis and the results of Mohler (1952) and Levenspiel (1959) indicated that the temperature difference between the vapor bubble and the surrounding liquid had great influence on the rate of collapse. Mohler showed the effect of temperature difference on the intensity of the shock wave associated with the collapse. The lower the temperature of the emulsion at the time of steam injection, the greater the speed of collapse and the more intense the shock wave.

Preliminary investigations with various concentrations of a number of emulsifiers, including monoglycerides, lecithin, a polysorbate, and detergents, were conducted to determine a satisfactory emulsion composition. Three percent corn oil emulsions with various emulsifiers were homogenized with a hand homogenizer at room temperature and samples were observed for free fat, formation of a layer of high oil content emulsion, and other properties at various time intervals and during heating to approximately 190°F. The detergent, Tide,¹ gave the most satisfactory emulsion of the emulsifiers tried,

¹Product of Proctor and Gamble Co., Cincinnati, Ohio.

but foam was a problem. Antifoam AF Emulsion¹ at the rate of 100 ppm was used in subsequent tests to control foam. An emulsion with the following composition by weight percentages was used for all further tests:

Corn oil	3.00%
Tide	0.50%
Antifoam AF Emulsion	0.010%
Water	96.5%

The detergent and the antifoam were combined with a volume of water approximately equal to the volume of corn oil in the emulsion. The antifoam-detergent solution was combined with the corn oil in a 1 gal jar and shaken to give a concentrated emulsion. The concentrated emulsion was diluted with an appropriate amount of water to produce a 3.00% oil-in-water emulsion. Ingredient costs were less than 6¢/gal of 3% emulsion. The emulsion could be held several months at room temperature without visible microorganism growth.

Determination of Effectiveness of Homogenization

The Deackoff and Rees (1957) optical method for determining effectiveness of homogenization of milk was simplified for testing the corn oil-water emulsion. The method used in this investigation was as follows:

¹ A silicone defoamer produced by Dow Corning Corp., Midland, Mich.

1. One ml of emulsion was pipetted into a 250 ml Erlenmeyer flask at room temperature.
2. The emulsion was diluted with distilled water at room temperature to give 0.012% oil content.
3. The diluted emulsion was permitted to stand at least 30 min but not more than 2 hr.
4. The sample was poured into a standard 1 cm cuvette and the transmittance¹ was read, using distilled water as a reference, in a Beckman² Model B spectrophotometer with 1020 mμ wave length light.

The NH_4OH treatment for dissolving the casein micelles, as suggested by Deackoff and Rees, was omitted because no proteinaceous material was present in the emulsion. In diluting the emulsion in step 2 above, compensation was made for the previous dilution effect of the injected steam. Calculation of the quantity of injected steam and of the quantity of distilled water required in step 2 to give a 0.012% oil content is covered in a later section. Evidence presented later in this section confirmed the use of 1020 mμ wave length light for determination of effectiveness of homogenization.

Attempts were made to compare the optical test with fat separation tests, using the Babcock test for milk fat to

¹Transmittance may be defined as the ratio of the transmitted radiant power of the emulsion, P , to the transmitted radiant power of distilled water, P_0 , in a matched reference cell. $T=P/P_0$.

²Manufactured by Beckman Instruments, Inc., Fullerton, Calif.

determine oil content of the top and bottom portions of the emulsions. A white flocculent material in the fat column interfered with fat content readings. A similar flocculent material was observed in tests using a detergent solution with no corn oil. It was concluded that the interfering substance originated from the detergent and no further tests of this type were attempted.

Emulsion Prehomogenization and Preliminary Optical Tests

The 3% corn oil-water emulsion was homogenized with a Manton-Gaulin Type 75K¹ homogenizer with 200 psi first-stage valve pressure to prevent rapid formation of a layer of high oil content emulsion and to give a more uniform globule size emulsion. Portions of this prehomogenized emulsion were rehomogenized at various pressures, using the first stage valve only. The transmittances of these samples were determined at a number of wave lengths between 650 and 1020 mμ. Transmittance was plotted versus homogenization pressure for various wave lengths in Fig 6.

The transmittance of samples homogenized at pressures of 1700 psi and above were anticipated on the basis of Deackoff and Rees' (1957) results with milk. The results at homogenizer pressures of 1125 psi and below in which the transmittance decreased

¹Manufactured by Manton-Gaulin Mfg. Co., Inc., Everett, Mass.

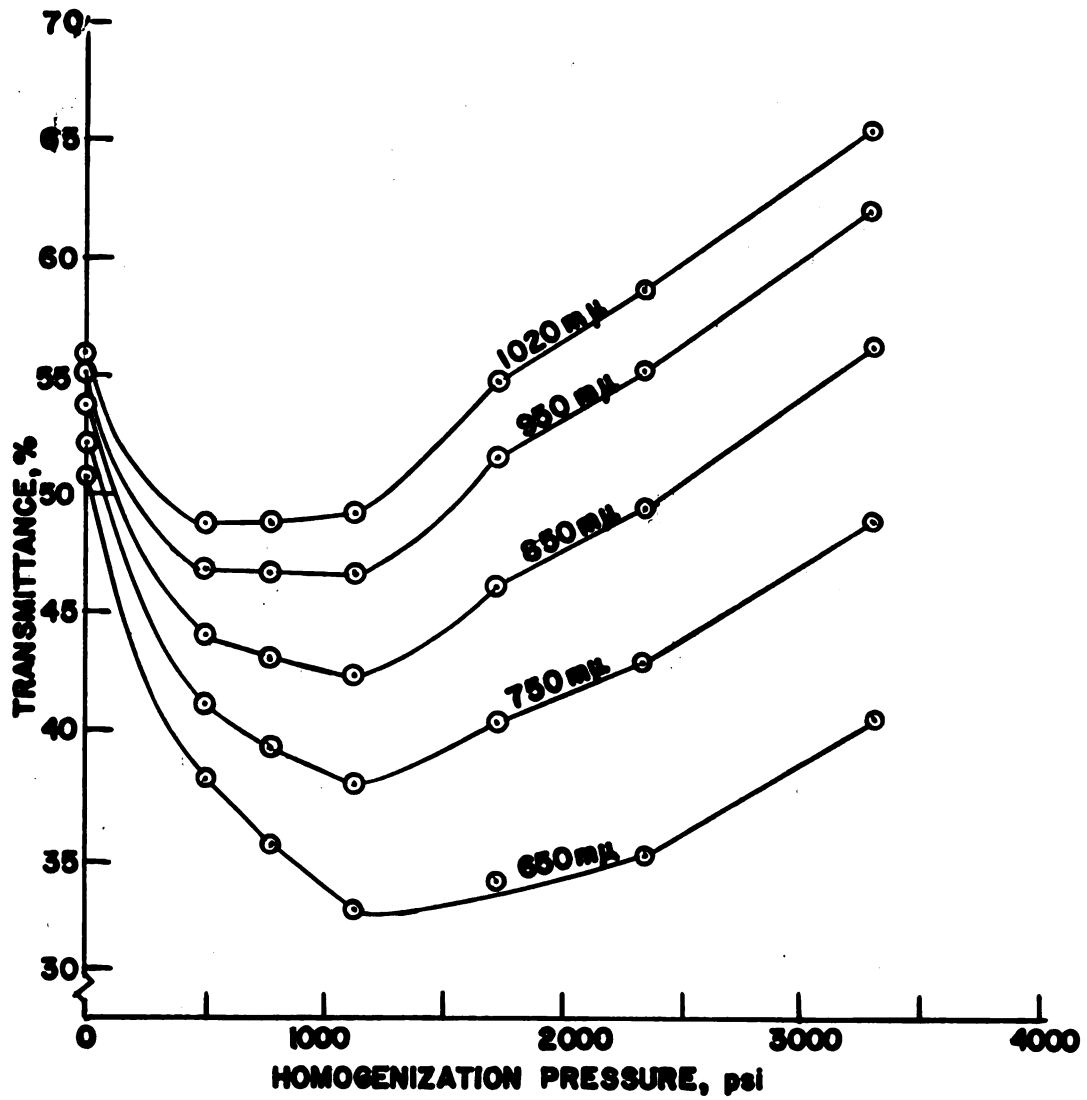


Figure 6. Relationship of transmittance and homogenization pressure of corn oil-water emulsion at various wave lengths following prehomogenization at 200 psi

or remained constant with increasing pressures were attributed to the large size of globules in the 200 psi prehomogenized emulsion as compared with milk. Transmittance is dependent upon the wave length-globule diameter relationship, as mentioned in "Literature Review."

Fig 7, plotted from calculations based upon the theoretical approach of Goulden (1958), indicates that large globules as well as small globules produce high transmittances.

In an attempt to produce transmittance-pressure curves which would permit evaluating effectiveness of homogenization at lower pressures, emulsion was prehomogenized at 800 psi and then rehomogenized at various pressures. Fig 8 presents the results obtained with three different batches of emulsion. The 1020 mμ wave length gave the most satisfactory curve for comparison of effectiveness of homogenization. Deackoff and Rees (1957) have shown that this is also the best wave length for evaluating homogenized milk.

Since prehomogenization at 800 psi permitted optical differentiation between samples down to approximately 500 psi homogenizer pressures, all emulsions for steam-injection tests were prehomogenized at this pressure. This treatment produced an emulsion with globules in the range of 1 to 10 μ. A large portion of the globules was in the 4 to 6 μ range. From theoretical considerations, the energy requirements for homogenization are proportional to surface area increases. Large globules for the steam-injection test appeared to be desirable because the diameter of large globules would be reduced more by a given energy input than would the size of small globules.

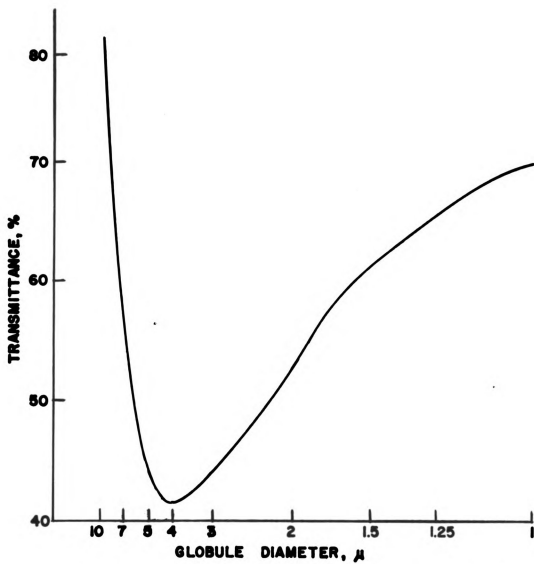


Figure 7. Theoretical transmittance of milk with globules of uniform size

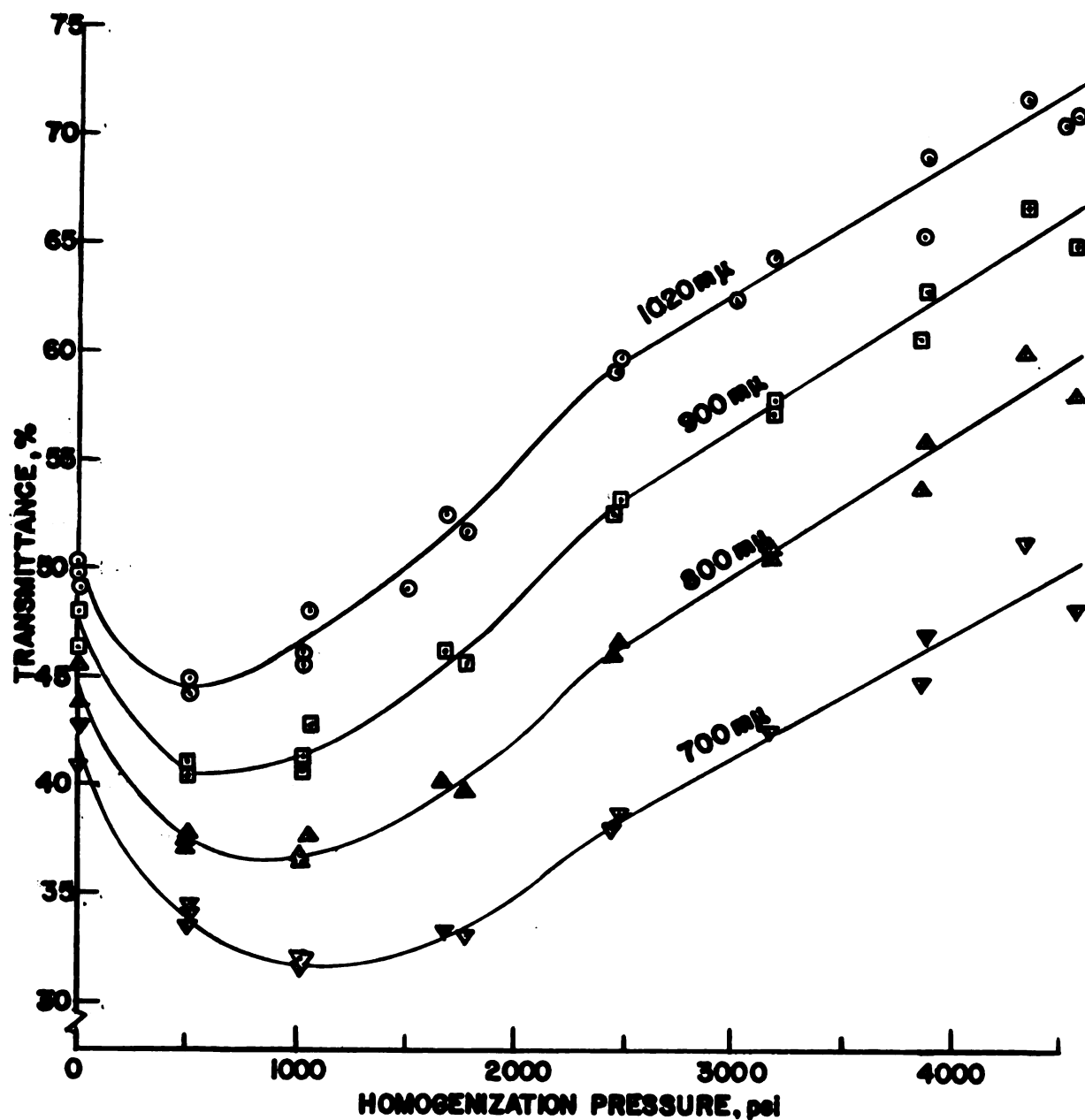


Figure 8. Relationship of transmittance and homogenization pressure of a corn oil-water emulsion at various wave lengths following prehomogenization at 800 psi. (Results obtained with three different batches of emulsion for 1020 mμ wave length readings and two batches of emulsion for 700, 800, and 900 mμ wave length readings. Each point represents one test)

Because of the inherent physical instability of emulsions, the optical effectiveness of homogenization test was repeated at various time intervals following the first test. Representative results for both conventional pressure homogenization and steam-injection homogenization are presented in Fig 9 and 10. The transmittance-pressure or energy curves were substantially the same for the emulsions for periods up to at least six days. Transmittances of emulsions receiving little or no homogenization showed more variation than emulsions with smaller globules.

Small droplets of free oil were observed on the surface of the emulsion diluted to 0.012% for the optical test when the sample had received only the 800 psi prehomogenization. Since the time between emulsion dilution and transmittance reading could influence the results, the transmittance of a number of samples was read 24 hr after dilution for comparison with initial readings. Fig 11 contains representative results. Again, the emulsions with little or no homogenization showed the greatest variation in transmittances and very little difference in transmittance at higher homogenization pressure.

Since the dilution of emulsion for the effectiveness of homogenization test was based upon steam energy input, which in turn was calculated from a number of measurements that were subject to experimental error, emulsions were diluted at various levels to show the effect of errors in calculated steam energy input. These data are presented in Fig 12. Large errors in the

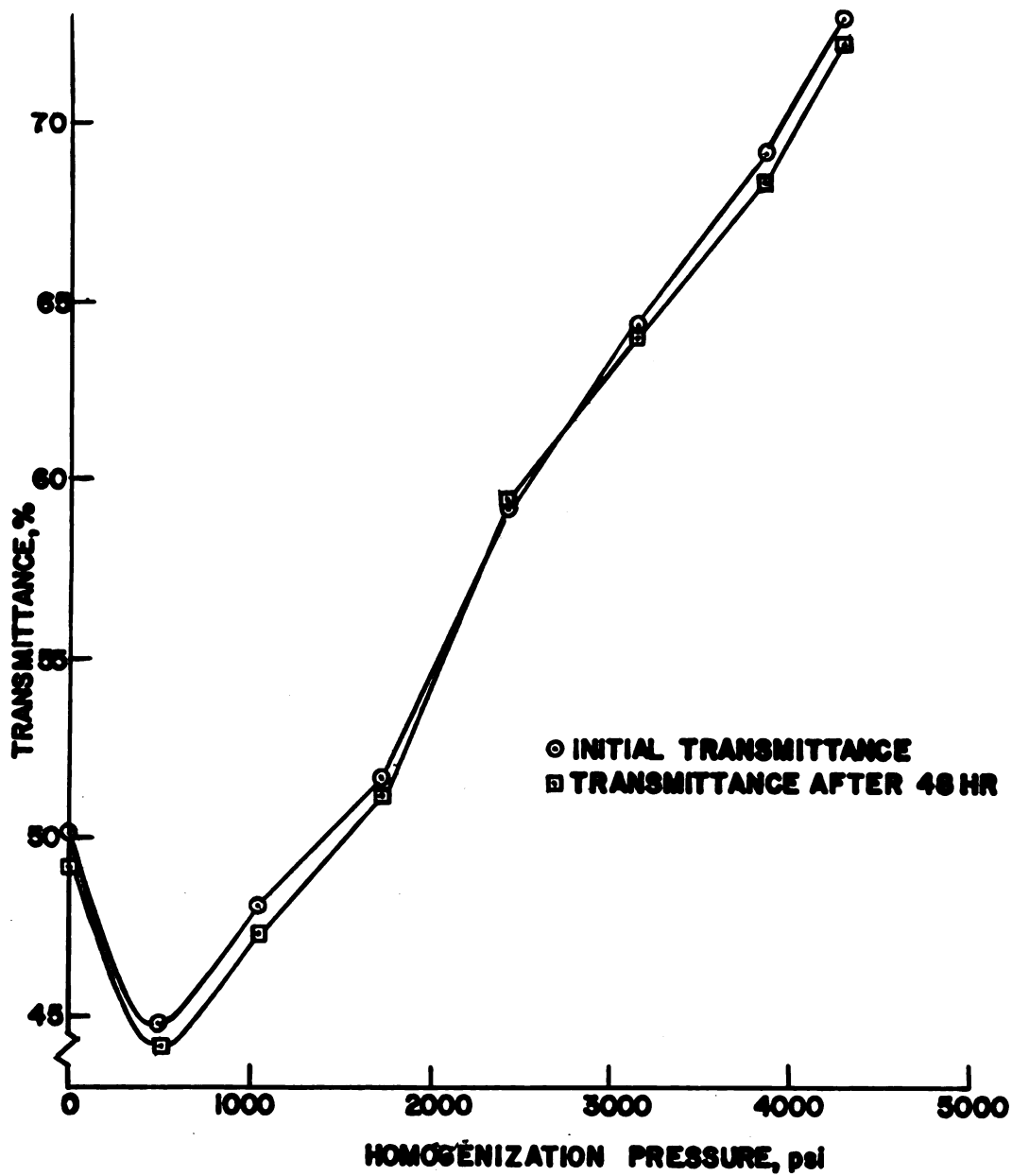


Figure 9. Transmittance as a measure of stability of emulsion homogenized in a conventional pressure homogenizer

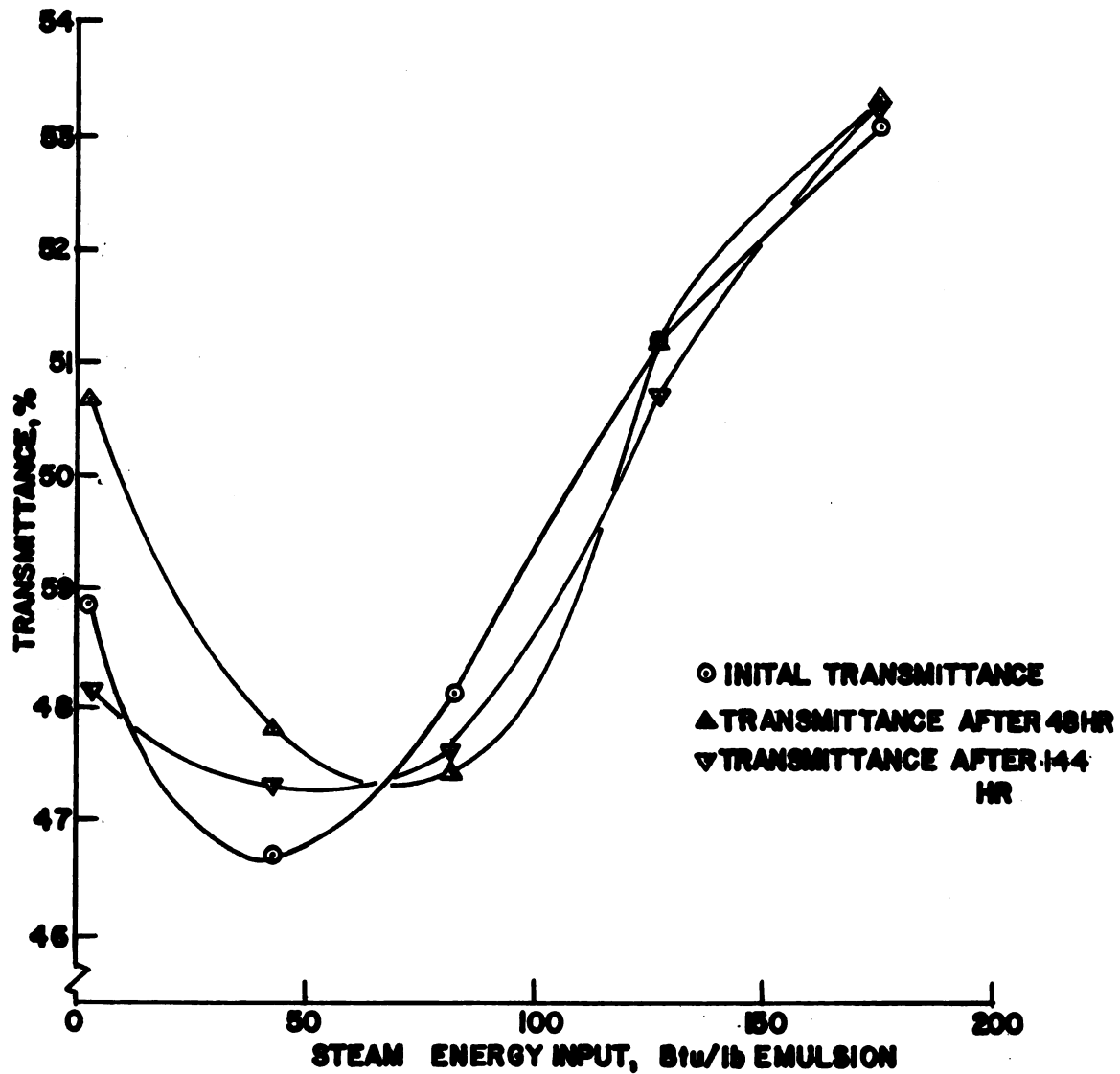


Figure 10. Transmittance as a measure of stability of emulsion receiving steam-injection treatment

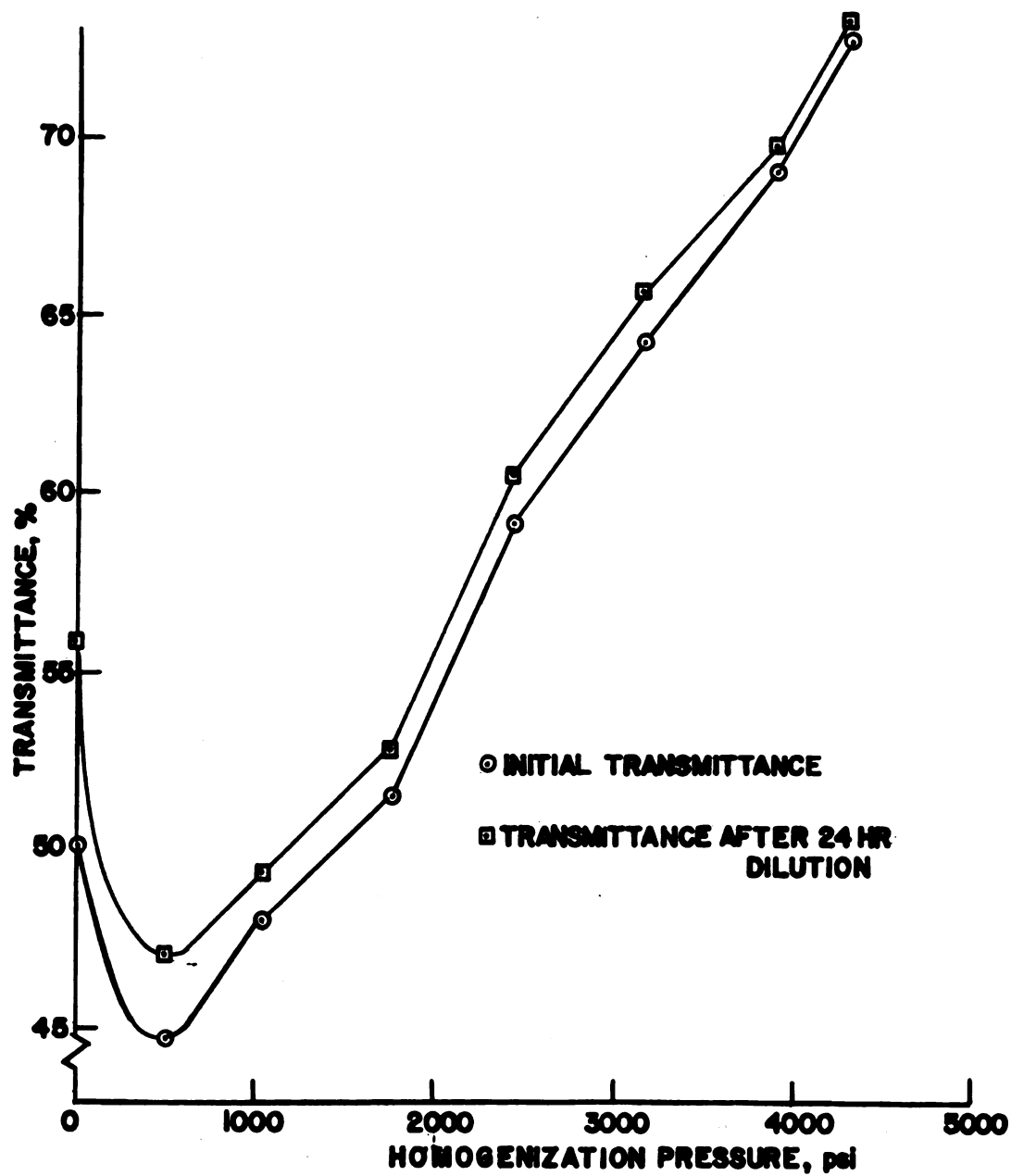


Figure 11. Effect of 24 hr dilution on emulsion transmittance

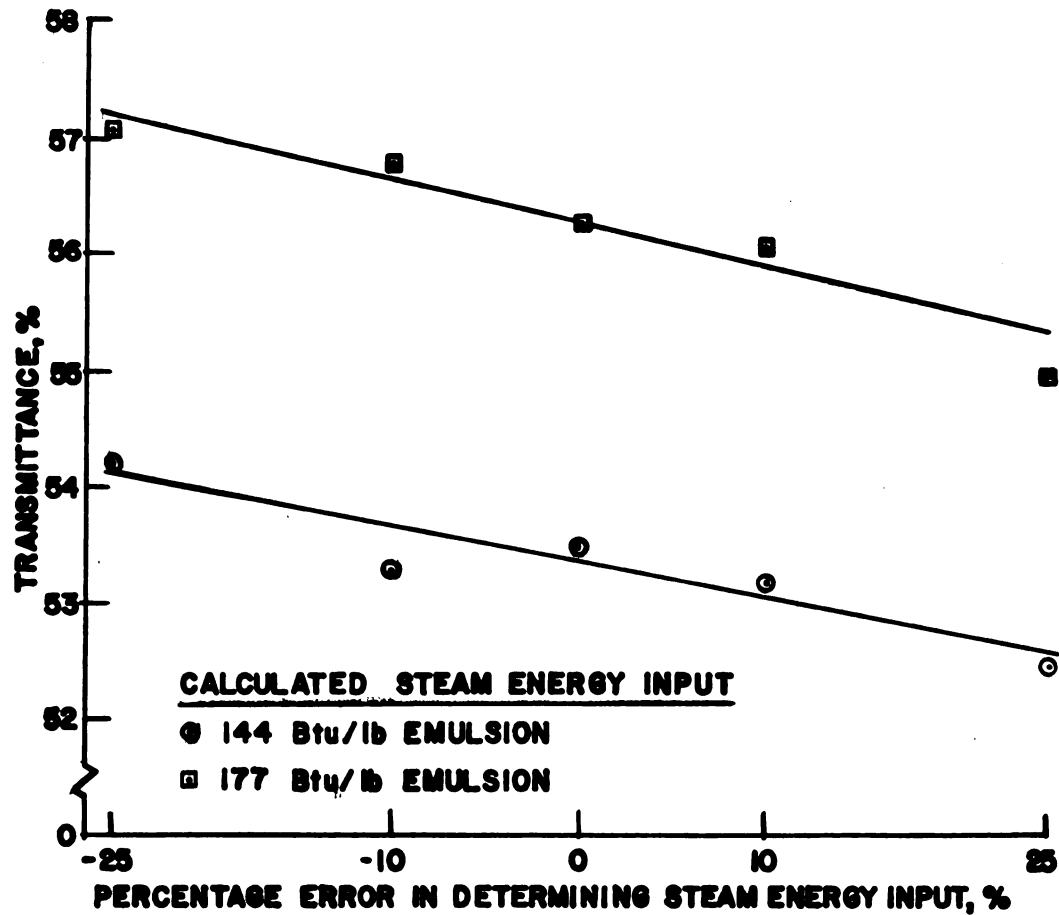


Figure 12. Effect of steam energy input errors on transmittance

steam energy input resulted in very small differences in transmittance.

For comparative purposes, 3.5% milk was pasteurized at 144°F for 30 min and homogenized at various pressures. The effectiveness of homogenization was determined by the optical method of Deackoff and Rees (1957). The transmittance-homogenization pressure plot for milk is presented in Fig 13 with the results for the corn oil emulsion tested by the optical method described previously.

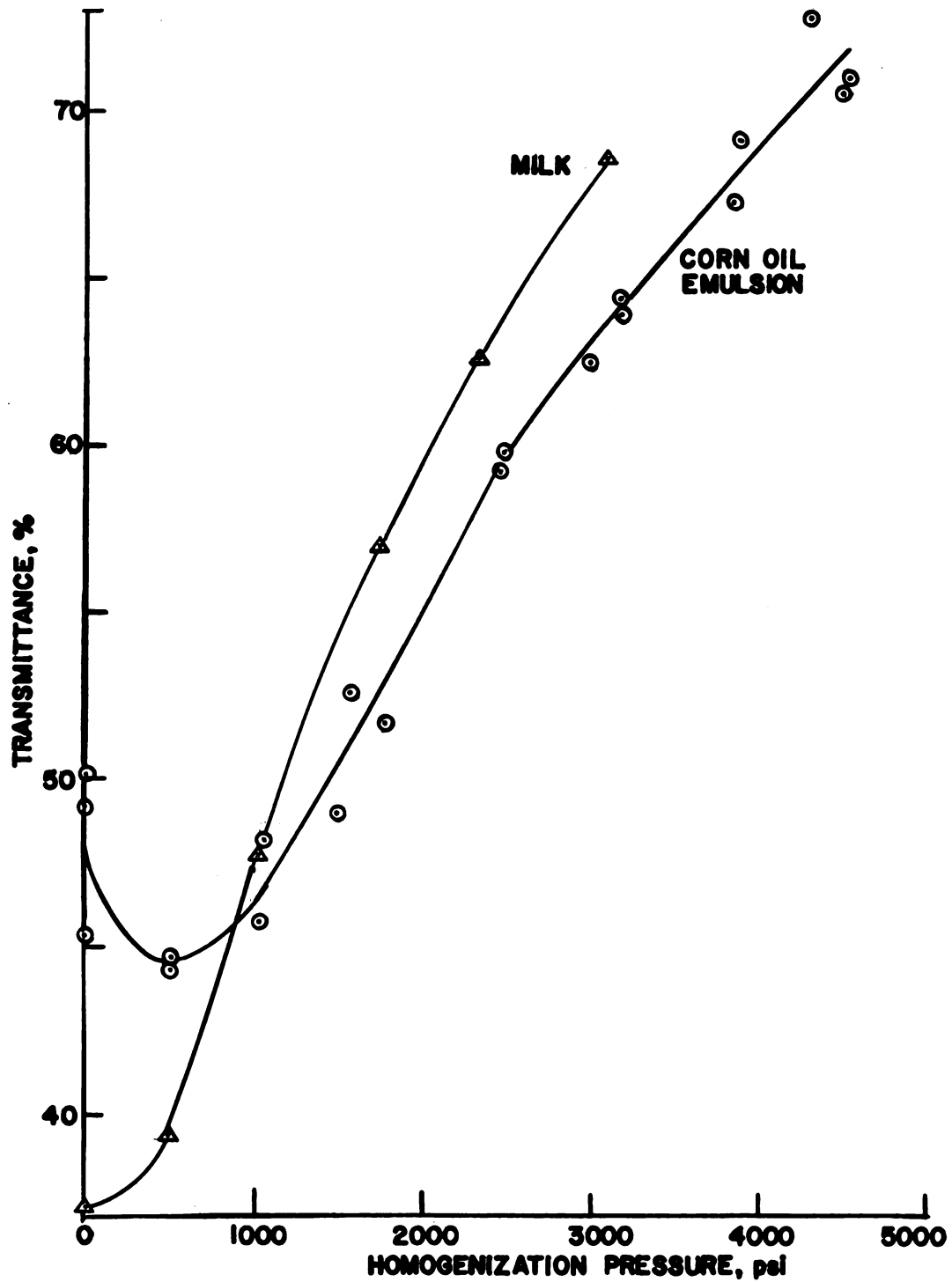


Figure 13. Comparison of transmittances of corn oil-water emulsion and of milk homogenized at various pressures. (Three batches of corn oil emulsion served as a source of emulsion for the tests. Each point represents one test)

PROCEDURE

Five series of four tests each were conducted to determine the effect of various factors on the size reduction of emulsion globules subjected to steam treatments. Emulsion for a given test was recirculated through the injector system and samples were withdrawn to represent various levels of steam treatment. The variables studied in the five series of tests were:

Series A--Steam-injection temperature

Series B--Emulsion pressure at discharge from injector

Series C--Emulsion pressure at discharge from injector

Series D--Emulsion flow rate

Series E--Initial globule size.

Table 1 contains a summary of test conditions and variables.

Following the step-by-step procedure used for all tests as outlined below, further explanation of some of the steps is presented.

1. Emulsion sufficient for the four tests of a series was prepared (see "Development of Experimental Methods").

2. The boiler pressure regulator was adjusted to give the desired steam temperature. (Steam temperatures for each test are shown in Table 1.)

Table 1. Summary of steam-injection test variables

Test series number	Pre- homogenization pressure psi	Injector discharge pressure psi	Mass flow rate lb/min	Steam temperature °F
A-1	800	0	57	237
A-2	800	0	57	273
A-3	800	0	57	306
A-4	800	0	57	347
B-1	800	0	57	347
B-2	800	25	57	346
B-3	800	50	57	346
B-4	800	80	57	346
C-1	800	0	57	346
C-2	800	25	57	346
C-3	800	50	57	346
C-4	800	80	57	346
D-1	800	0	27	346
D-2	800	0	42	346
D-3	800	0	57	346
D-4	800	0	75	346
E-1	800+0	0	57	346
E-2	800+1500	0	57	346
E-3	800+3000	0	57	346
E-4	800+4500	0	57	346

3. Cold water was pumped through the system, and the flow rate was adjusted by means of the Varidrive motor with the steam valve closed (see Fig 5).

4. Emulsion pressure on the discharge side of the injector was adjusted to 24 in. water by means of the globe type discharge valve.

5. The sample valve was adjusted to give a sample flow rate of about 14 ml/sec.

6. The pump was stopped.

7. Water in the reservoir, between the injector and the reservoir on the discharge side and between the reservoir and the pump suction valves, was drained.

8. A weighed portion (30.9 lb) of emulsion was placed in the 5 gal reservoir.

9. The pump and the potentiometer were started simultaneously.

10. A meter reading was taken and recorded on the potentiometer chart.

11. A control sample was taken after circulation of the emulsion for at least 1 min.

12. The steam valve was opened and the time was recorded on the potentiometer chart.

13. Emulsion pressure on the discharge side of the injector was adjusted to the desired level (Series B and C tests only).

14. The sample valve was readjusted to give the proper sample flow rate.

15. Meter readings were taken and recorded on the potentiometer chart at various intervals as the emulsion heated.

16. Four or five samples were taken at various intervals during the heating of the emulsion to represent various levels of steam treatment.

17. Steps 2 through 16 were repeated for the other tests of each series.

18. The steam energy input per pound of emulsion and the dilution of each emulsion sample was calculated (see "Calculations").

19. Each sample was diluted with water to a 0.012% oil content and the transmittance was measured with a spectrophotometer (see "Development of Experimental Methods").

Preceding each test, cold water was circulated through the steam-injection system to flush old emulsion, to cool the system, and to adjust the flow rate. Because of the detergent content, high flow rate, and temperature of the emulsion at the conclusion of a test, a cold-water rinse for at least 5 min was considered adequate to clean the system. The water in the reservoir, between the injector and the reservoir and between the reservoir and the pump suction valves, was drained. No attempt was made to remove the remaining water from the system because this would have required complete dismantling of

the pump and disconnecting and draining the water meter. The residual water in the system weighed 4.1 lb.

The 3% corn oil emulsion for a given series of tests was thoroughly mixed in a large container so that the starting emulsion for each test of a series was identical. For each test, a 30.9 lb portion of the emulsion was weighed and placed in the 5 gal milk can reservoir. The emulsion was continuously recirculated and the residual water in the system diluted the emulsion to a calculated 2.65% oil content.

The recording potentiometer was employed as a timing device for taking samples and for determining flow rate. The potentiometer and the pump were started simultaneously. A water meter reading was taken immediately after the pump was started and at various times throughout each test. The meter readings were recorded on the potentiometer chart paper at the corresponding time of the reading.

Emulsion was continuously flowed through the sample hose to the reservoir. The rate of flow in the sample line was regulated by the sample valve so that approximately 4 sec were required to draw each sample of 50 to 60 ml into a small bottle. Calculations indicated that the sample withdrawn at the midpoint of the 4 sec interval was representative of the emulsion in the injector at the time recorded on the potentiometer chart.

A sample that had received no steam treatment was taken after circulation of the emulsion for at least 1 min. Since

emulsion in the reservoir was kept well-mixed and since 35 lb of emulsion in the system were circulated at more than 55 lb/min in all but two of the twenty tests, the emulsion was considered to be uniformly diluted by the residual water in the system when the control sample was taken. For the lowest flow-rate test of 28 lb/min, the emulsion was circulated for 1.8 min before the control sample was taken.

The emulsion temperature at the start of the steam treatment was approximately 75°F for all tests. The steam valve was opened after the control sample was withdrawn and the time was recorded on the potentiometer chart. As the emulsion recirculated through the system, the temperature was permitted to increase to between 192° and 208°F. Four or five samples were taken during steam injection to represent different levels of steam treatment. The time of withdrawal was recorded on the potentiometer chart for each sample.

The pressure on the discharge side of the injector was adjusted to 24 in. of water before the start of each test by means of a globe valve to provide sufficient pressure for withdrawing the sample. In the series of tests in which emulsion discharge pressure was the variable, the pressure was adjusted immediately after the steam valve was opened. Although it would have been desirable to have the pressure adjusted before the start of the steam injection treatment, emulsion was forced through the steam ports by the pressure. The maximum time required for adjusting the pressure after the steam

injection was commenced was less than 0.5 min. Approximately 3.5 min were required to increase the emulsion temperature from about 75° to 200°F for tests in which discharge pressure was the variable.

In Test Series E all emulsion was prehomogenized in the Manton-Gaulin homogenizer at 800 psi and then rehomogenized at the pressure specified in Table 1.

The accumulated heat energy which a unit weight of 2.65% emulsion absorbed from the injected steam is hereafter called "steam energy input," and its units are Btu/lb of 2.65% emulsion. The steam energy input was used as a basis for evaluating the test variables. Steam energy input was plotted versus transmittance as a measure of size reduction (effectiveness of homogenization). The steam energy input was also employed to determine the dilution required for producing the 0.012% emulsion for the optical test.

The steam energy input was calculated from the mass flow rate and temperature-time potentiometer chart data. Details of the calculations are covered in the following section entitled "Calculations."

Mass flow rate decreased by more than 10% as the temperature of the emulsion increased from room temperature to about 175°F. Therefore, mass flow rate was plotted versus temperature for each test or series of tests. From this plot, the mass flow rate at any temperature of emulsion was available.

Areas formed by the temperature-time plot on the potentiometer chart were planimetered. Since the temperature scale on the potentiometer chart was not linear, temperature time values represented by a unit area varied with temperature. A planimeter area factor graph relating planimeter area to temperature-time values for various temperatures is presented in Fig 14.

The quantity of steam involved in heating the product was calculated from steam enthalpy data taken from steam tables. The steam was assumed to be dry saturated steam. The quantity of injected steam permitted the calculation of the volume of water necessary for dilution to 0.012% corn oil for the optical test. Transmittances of all samples were determined between 24 and 36 hr following steam injection and transmittance was plotted versus steam energy input per pound of 2.65% emulsion for each series of tests.

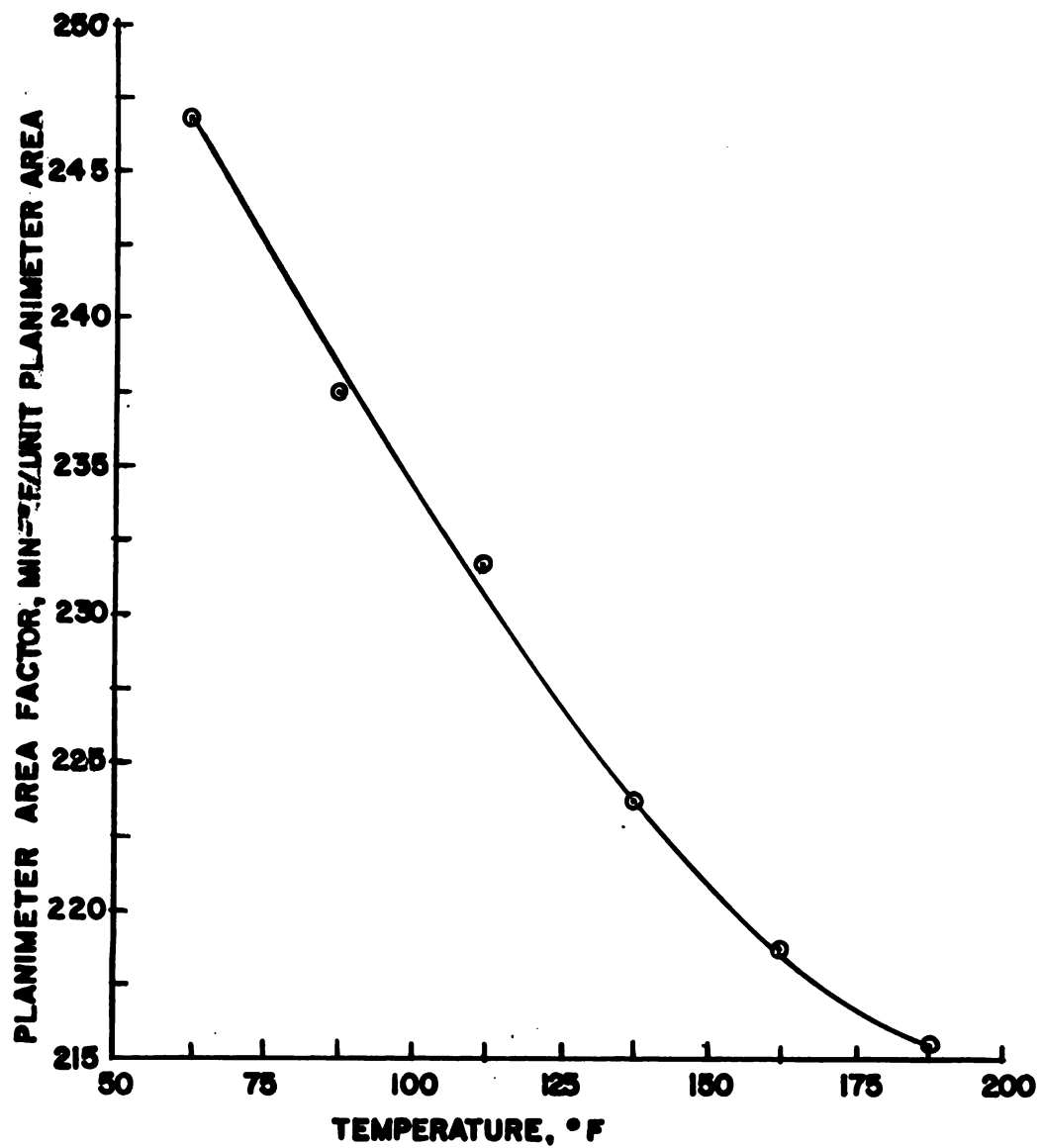


Figure 14. Planimeter area factors for various temperatures on the 50° to 350°F potentiometer chart

CALCULATIONS

The emulsion samples were drawn immediately following the injector where the steam energy input per pound of 2.65% emulsion was higher than in the remainder of the emulsion. The total treatment to which the emulsion sample had been subjected was calculated as follows:

$$q = q_a + q_b,$$

where q = total steam energy treatment per unit weight of 2.65% emulsion, Btu/lb of emulsion

q_a = steam energy treatment per unit weight of 2.65% emulsion immediately preceding the injector, Btu/lb of emulsion

q_b = steam energy injected per unit weight of 2.65% emulsion in passing through the injector one time, Btu/lb of emulsion

To simplify the calculation of q_a , all globules and condensed steam were considered to be uniformly distributed in the system at the instant of steam injection. The error resulting from this simplification was small, as will be shown.

The values of q_a and q_b were determined from the data on the potentiometer chart. From the meter reading-time data the mass flow rate, w , was calculated as follows:

$$w = Gp(\Delta M/\Delta \theta)$$

where w = mass flow rate, lb/min

G = meter correction factor (1.05)

ρ = density of emulsion, lb/gal

ΔM = difference between two meter readings, gal

$\Delta \theta$ = time interval between meter readings, min.

Since the flow rate was found to vary with temperature, mass flow rate was plotted against the preinjector temperature at the midpoint of the time interval between meter readings for each test or series of tests. This same temperature was used to determine emulsion density, ρ , which was considered to be the same as for water. An example of a mass flow rate-temperature plot is shown in Fig 15. The mass flow rate for a given calculation was read from the mass flow rate-temperature curve at the preinjector temperature at the midpoint of the time interval between samples, t_{p_1} (see Fig 16).

The area between preinjector and discharge temperatures, t_p and t_D , recorded on the potentiometer chart and within the time interval between any two samples, (i-1) and i, was planimeted (Fig 16). The planimeter area factor for converting planimeter area to °F-min was read from Fig 14. The temperature, t_{A_1} , corresponding to the arithmetic average of the preinjector and discharge temperature at the midpoint of the time interval between samples was used to determine the planimeter area factor representative of the area.

The quantity of heat injected per pound of emulsion entering the injector was determined for each interval between samples. This quantity was calculated as follows:

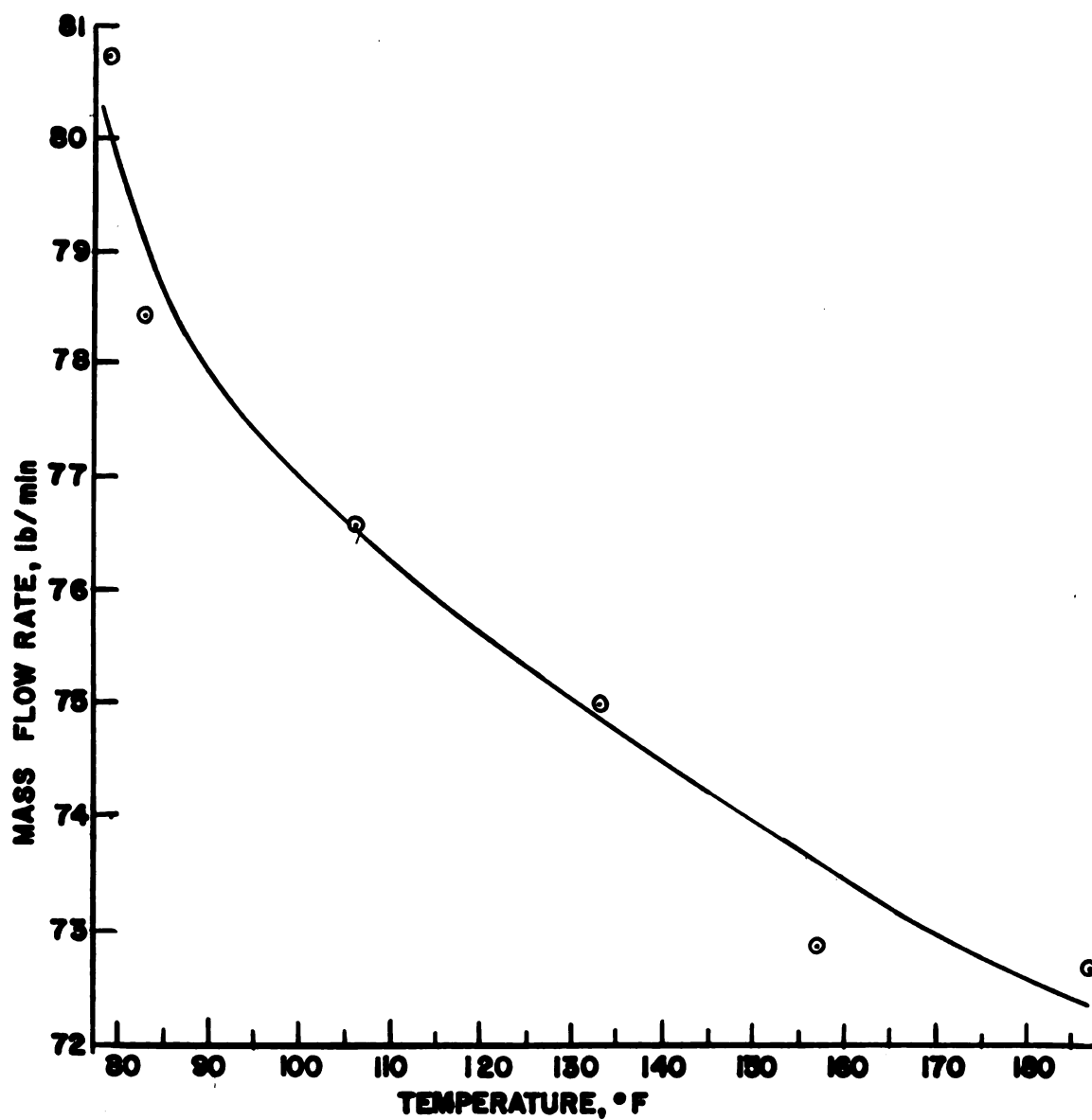


Figure 15. A typical mass flow rate-temperature graph

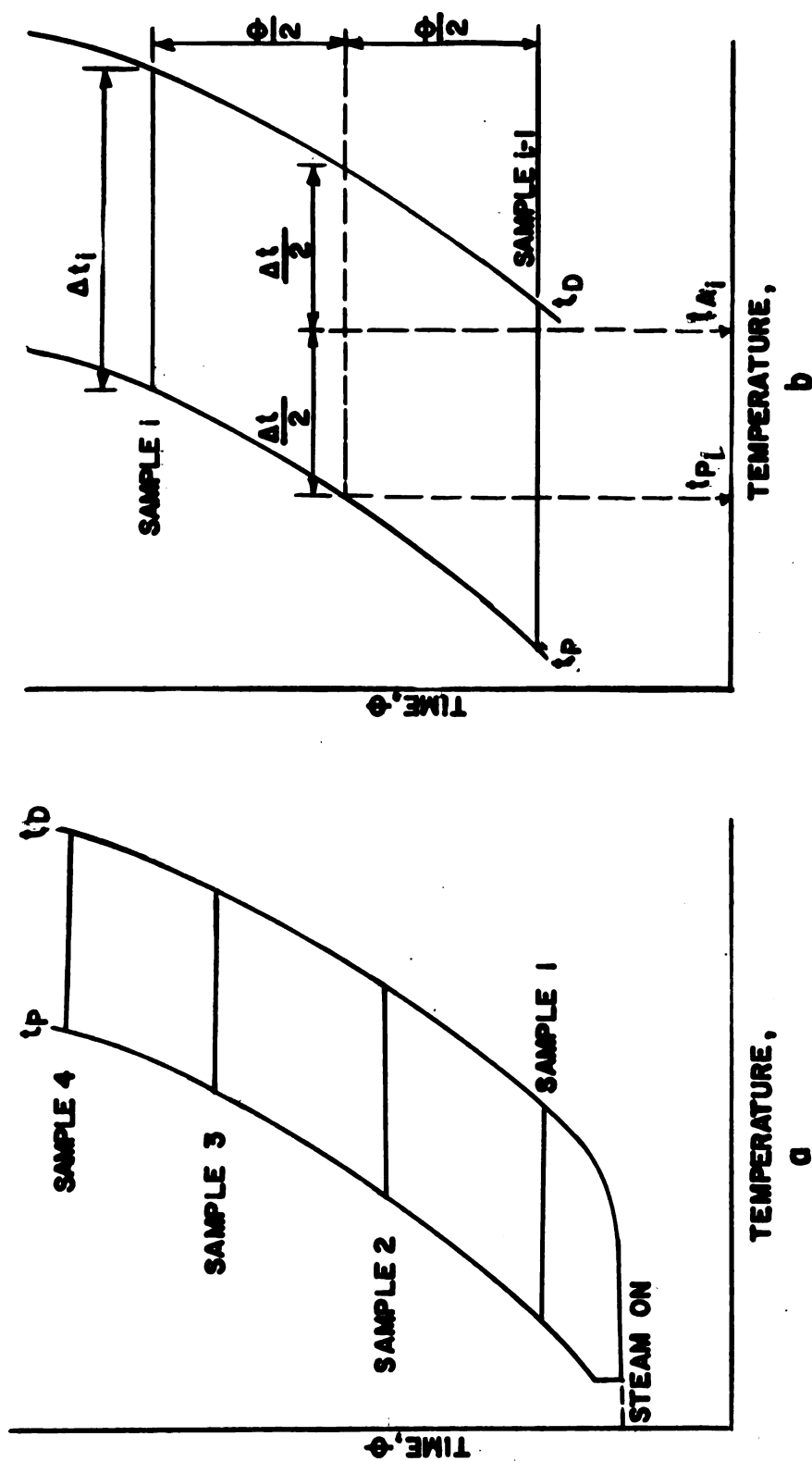


Figure 16. Time-temperature plot produced on potentiometer chart showing various points used for calculations. (a) Complete test. (b) Expanded area between any two samples

$$q_{a_i} = B_i A_{p_i} w_i / 35,$$

where q_{a_i} = steam energy treatment per unit weight of 2.65% emulsion in the time interval between samples, (i-1) and i, Btu/lb of emulsion

B_i = planimeter area factor for this time interval, °F-min/planimeter area unit

A_{p_i} = planimeter area for this time interval, planimeter area units

w_i = mass flow rate for this time interval, lb/min

The number 35 represents the weight of 2.65% emulsion in the system. The specific heat of the emulsion was estimated as 1 Btu/lb-°F. Since the value will be the same for all tests, any error in this assumption would not affect the evaluation of the variables. The sum of the injected energy between samples is given by

$$q_a = \sum_{i=0}^{i=n} q_{a_i},$$

where n is the number of the sample for which the calculation was made.

The quantity of heat added to each element of emulsion in passing through the injector is given by

$$q_{bn} = \Delta t_n,$$

where Δt_n is the temperature increase (°F) in passing through the injector when sample n was taken when the specific heat is taken as 1 Btu/lb-°F.

In calculating q_a , the more intense treatment of the 0.9 lb of emulsion between the injector and the reservoir was

neglected. If this had been included in the calculation, the energy added to the system in the interval between the last two samples under consideration would have been

$$q_{a_n} = \frac{P_n A_n w_n - 0.9 \Delta t_n}{35}$$

and the value of q would have been

$$q = \sum_{i=0}^{i=n-1} q_{a_i} + q_{a_n} + q_{b_n}.$$

Substituting for q_{a_i} and q_{a_n} from above,

$$q = \sum_{i=0}^{i=n-1} \frac{P_i A_i w_i}{35} + \frac{P_n A_n w_n - 0.9 \Delta t_n}{35} + \Delta t_n,$$

which reduces to

$$q = \sum_{i=0}^{i=n} \frac{P_i A_i w_i}{35} + 0.973 \Delta t_n.$$

In neglecting the more intense treatment, the maximum error is less than 2.6%, and in the majority of tests the error was less than 1%.

The quantity of water added to the emulsion by steam injection was calculated from the enthalpy of dry saturated steam at the injection temperature. The enthalpy of the emulsion into which the steam was injected was continually rising because of the increase in temperature. Therefore, emulsion enthalpy was arbitrarily taken at the temperature average of the initial emulsion and the highest temperature of the sample, T_B . The enthalpy of the emulsion was considered to be the same as for water since the water content was 96.5%. The

small error resulting from this assumption would be the same for all tests.

The quantity of injected water vapor per pound of 2.65% emulsion was calculated as

$$Y = q/(h_g - h_f),$$

where Y = injected steam per pound of 2.65% emulsion,
lb steam/lb of emulsion

h_g = enthalpy of dry saturated steam, Btu/lb

h_f = enthalpy of emulsion, Btu/lb.

The volume of water required to dilute the 1 ml sample of emulsion to give a 0.012% oil content emulsion for the optical test was calculated by equating the required oil content with the calculated content,

$$0.00012 = 0.0265/y(1+Y),$$

where y is the volume of water in ml to add to 1 ml of emulsion. Solving for y ,

$$y = 221/(1+Y).$$

A sample data assembly and calculations are presented in Table 2.

Table 2. A sample data assembly and calculations table^a

Test series: <u>E</u>		Pump setting: <u>6.75D</u>				Steam temperature: <u>346</u>								
Test number: <u>1</u>		Discharge pressure: <u>0</u>				Steam enthalpy: <u>1192</u>								
Pot ^b	Sample code	Ap ^c	t _{A1}	B _i	t _{p1}	w _i	q _{a1}	q _{b1}	q _i	t _{B1}	h _g -h _f	Y _i	y _i	T _i
108	E-1-0	--	--	--	--	--	0	0	0	--	--	0	221	45.3
209	E-1-1	.0765	103	233	87	58.6	29.8	28.75	59	104	1120	.053	210	45.3
306	E-1-2	.0893	126	226	112	56.7	32.7	29.25	92	116	1108	.083	204	46.9
404	E-1-3	.104	151	221	137	55.6	36.6	28.25	127	129	1095	.116	198	48.8
504	E-1-4	.125	181	216	168	54.9	42.3	27.00	168	141	1083	.155	191	51.8

^aSee "List of Symbols" (p. x) and "Calculations" section for meaning of symbols and methods of calculating various quantities.

^bPot - potentiometer time. First digit indicates time in minutes from beginning of test. Last two digits indicate which of the 12 points was recording.

^cAverage of two or more planimeter readings.

RESULTS AND DISCUSSION

Effect of Steam Temperature on Globule Size Reduction

The temperature of injected steam had more effect on the size reduction of globules in the corn oil emulsion than the other factors investigated. The results of Test Series A in which steam temperatures of 237°, 273°, 306°, and 347°F were employed, are presented graphically in Fig 17. The characteristic decrease in transmittance with small energy input, followed by increased transmittance, was observed for all but the lowest steam temperature injection. The 193 Btu/lb of emulsion of 237°F steam was insufficient to produce an increase in transmittance. The initial decrease in transmittance agrees with the theoretical analysis of Goulden (1958a) (Fig 7).

The transmittance differences of control samples (samples receiving no steam treatment) were found to be of minor importance. Fig 18 presents the results of four replicate tests with emulsions prepared and prehomogenized at different times. Although the transmittances of control samples varied from 44.0 to 48.9%, the differences between samples was only about 2% for steam inputs of 50 to 150 Btu/lb of emulsion. At higher steam energy inputs, the differences were even less. Control sample transmittance differences within a test series were smaller than the differences between the replicate test emulsions of Fig 18. Correspondingly, less variation in

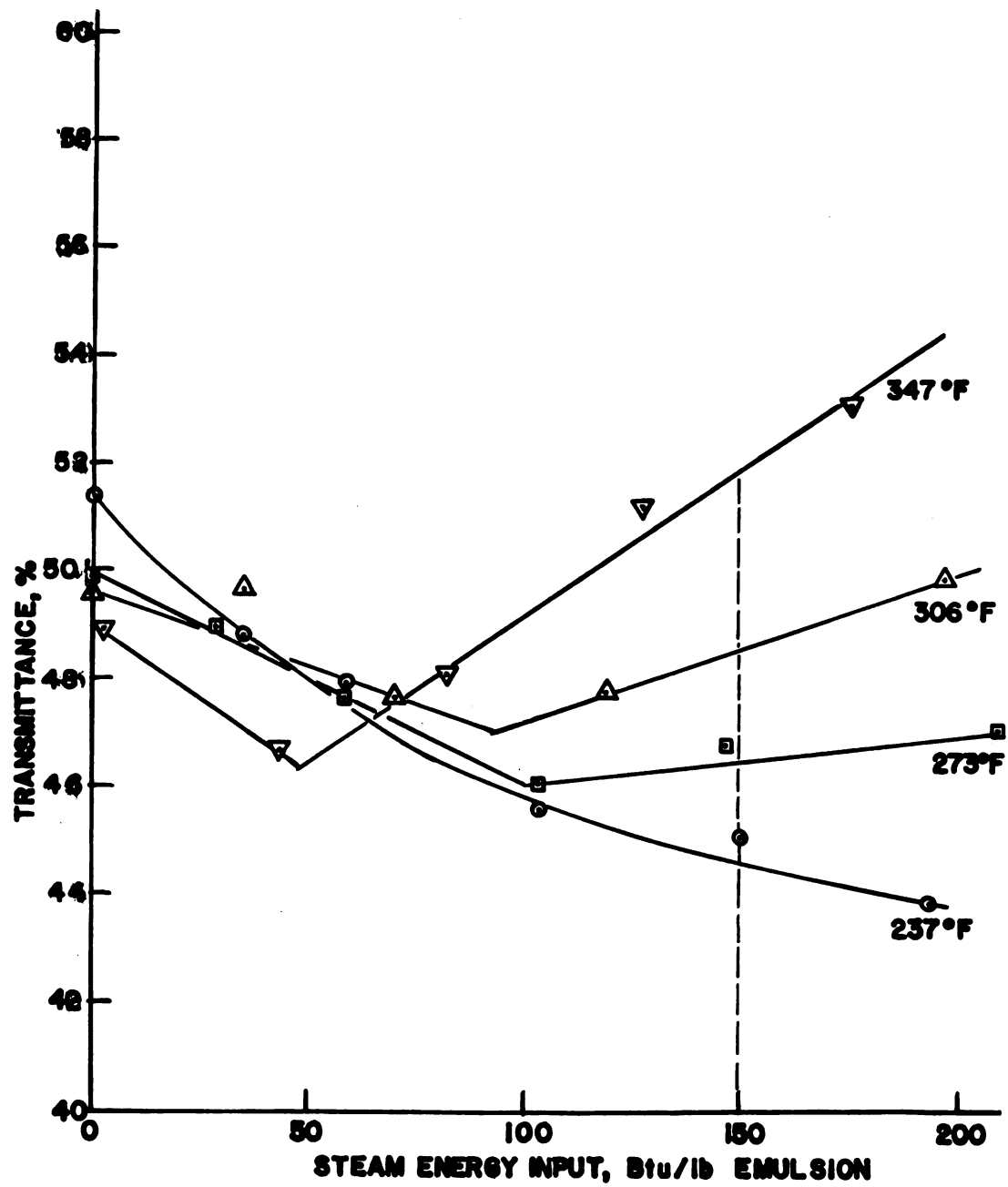


Figure 17. Effect of steam energy input and steam temperature on emulsion globule size reduction by steam injection

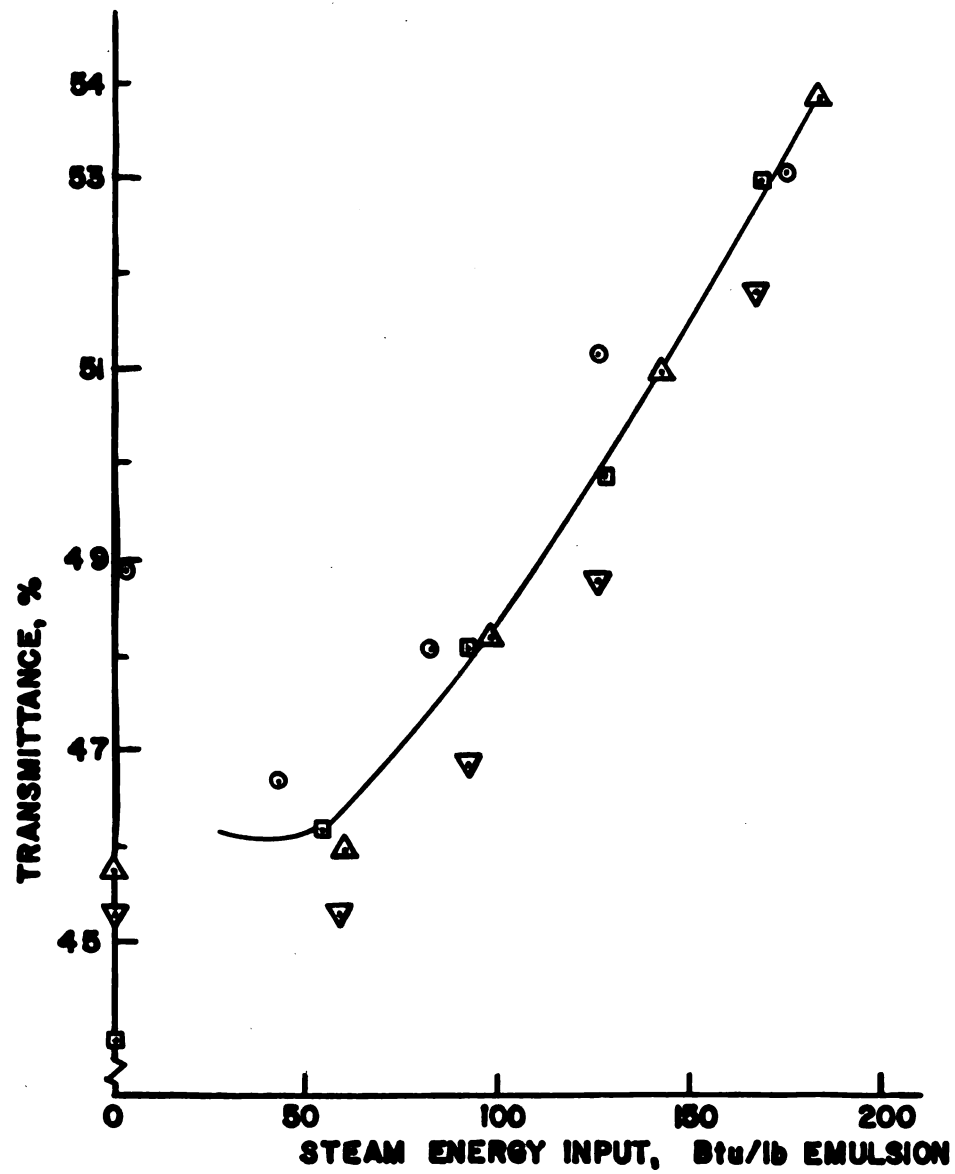


Figure 18. Emulsion transmittance of four replicate steam-injection tests

transmittances for emulsions within a test series would be expected at the higher steam energy inputs.

The fact that the 237°F steam-treatment emulsion reached a transmittance 2% lower than any of the other emulsions is probably due to a reduction in size of large globules, which would produce a decrease in transmittance with very little reduction of the medium-sized and small globules which would produce greater transmittance. It would appear that the low-temperature steam was more selective in breaking up only larger globules than the higher temperature steams.

A transmittance-steam temperature plot (Fig 19) permits better visualization of temperature effects. Both the transmittance and rate of transmittance increased with steam temperature. The temperature difference between injected steam and emulsion had much greater influence on the collapse of the steam bubbles than the quantity of heat per unit volume of bubble (Fig 1). If it is assumed that speed of collapse and accompanying shock waves produce globule size reduction, the temperature effect supports the findings of Levenspiel (1959), who showed that greater temperature differentials gave more rapid collapse of vapor bubbles, and of Mohler (1952) and G  th (1954), who found more intense shock waves associated with greater temperature.

A comparison of Fig 18 with Fig 13 indicated that a steam treatment of 175 Btu/lb of emulsion gave equivalent homogenization to about 1800 psi in the laboratory-model conventional homogenizer.

The size reduction of globules resulting from circulation of emulsion at various temperatures without steam injection was

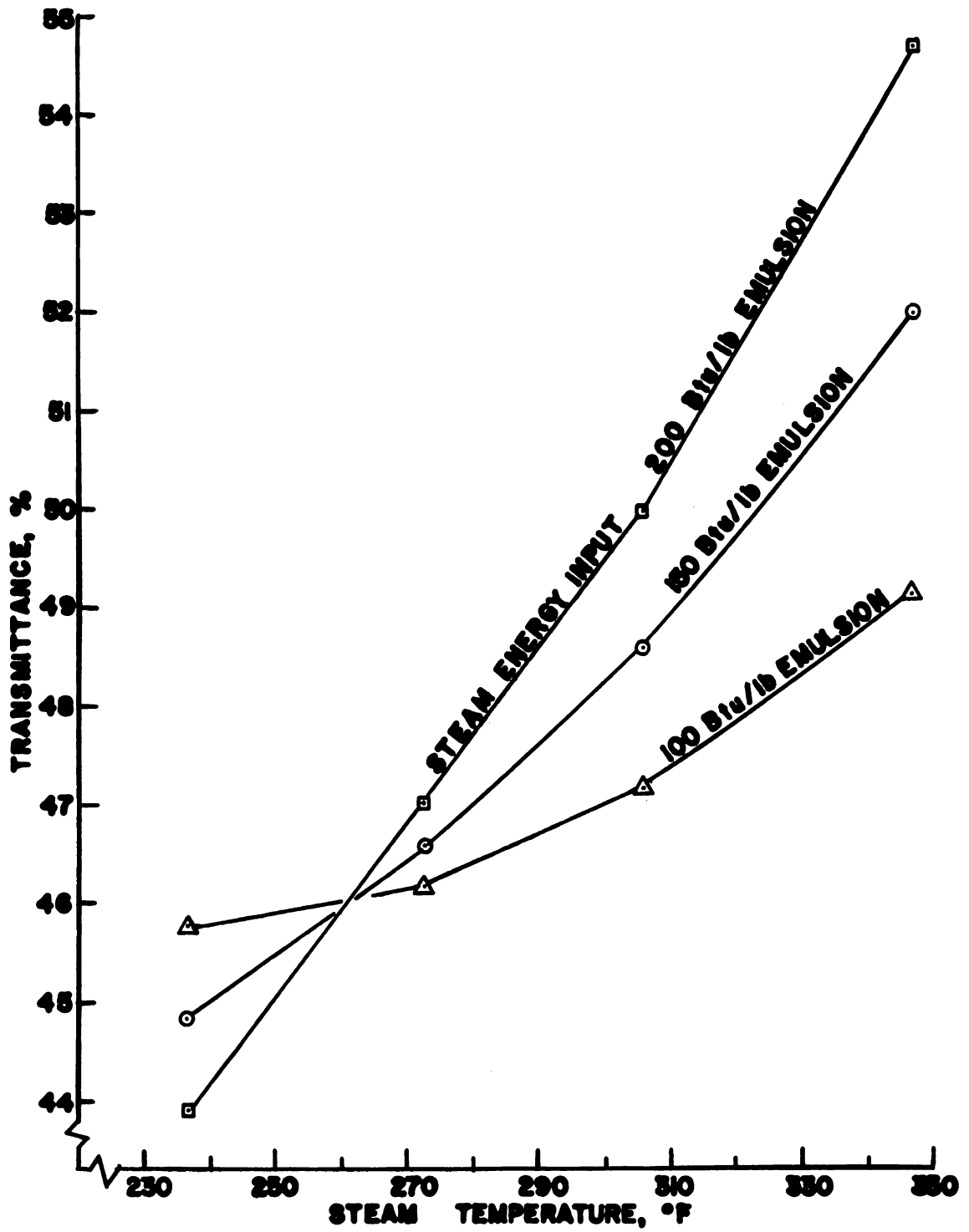


Figure 19. Effect of steam temperature on emulsion size reduction by steam injection

not investigated in the present series of tests. It is significant, however, that the emulsion subjected to 237°F steam required 22.7 min of circulation during steam injection, while only 3.2 min were required with 347°F steam. The fact that the high-temperature steam reduced globule size considerably more than the low-temperature steam supports the finding of some preliminary tests. The preliminary tests showed size reduction associated with circulation of emulsion in the system to be negligible when an effectiveness of homogenization test different from the optical test employed in this study was used.

Effect of Emulsion Pressure on Discharge Side of Injector on Globule Size Reduction

The results of Test Series B, depicted graphically in Fig 20, were close enough to make differentiation between emulsion discharge pressures difficult or impossible. Since preliminary results with a number of tests had indicated emulsion pressure on the discharge side of the injector influenced globule size reduction, the discharge pressure series of tests was repeated. Results of Test Series C, presented in Fig 21, give clearer differentiation. When transmittances for both series of tests at steam inputs of 175 Btu/lb of emulsion were plotted on a transmittance-discharge pressure curve (Fig 22), the two tests were observed to be in close agreement. The homogenization produced by 175 Btu/lb of emulsion with the 346°F

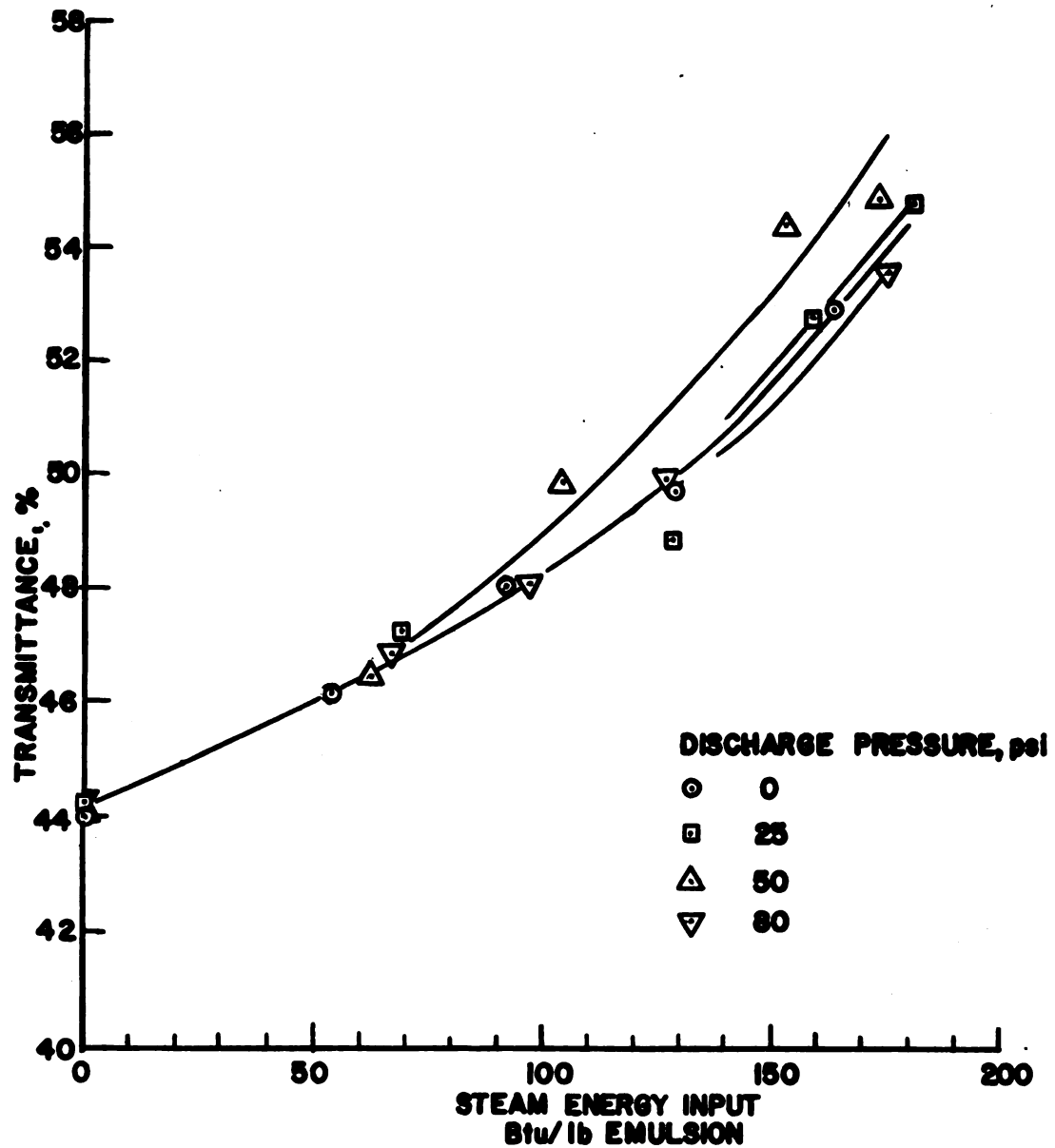


Figure 20. Effect of steam energy input and emulsion pressure on discharge side of injector on globule size reduction, Test Series B

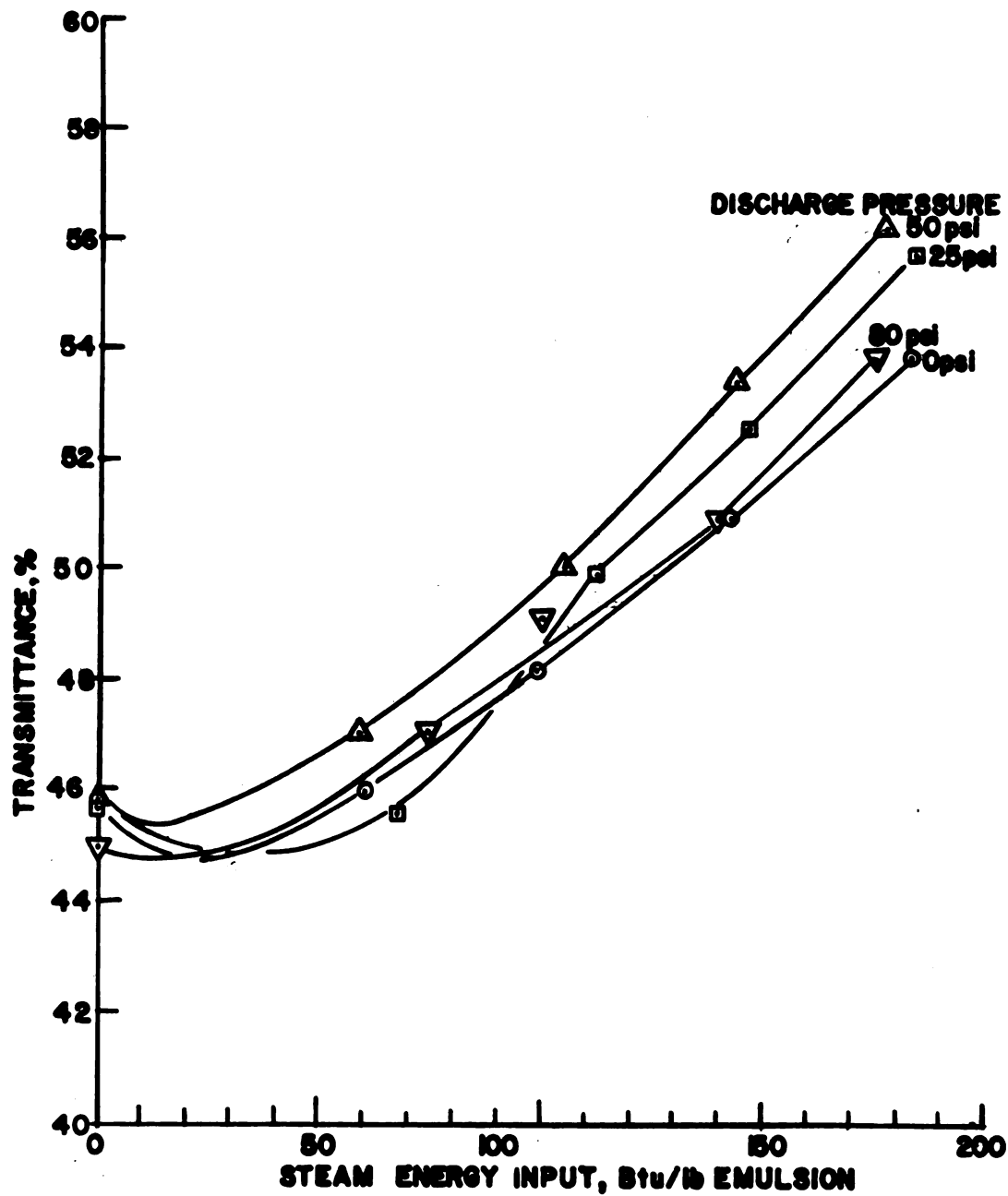


Figure 21. Effect of steam energy input and emulsion pressure on discharge side of injector on globule size reduction, Test Series C

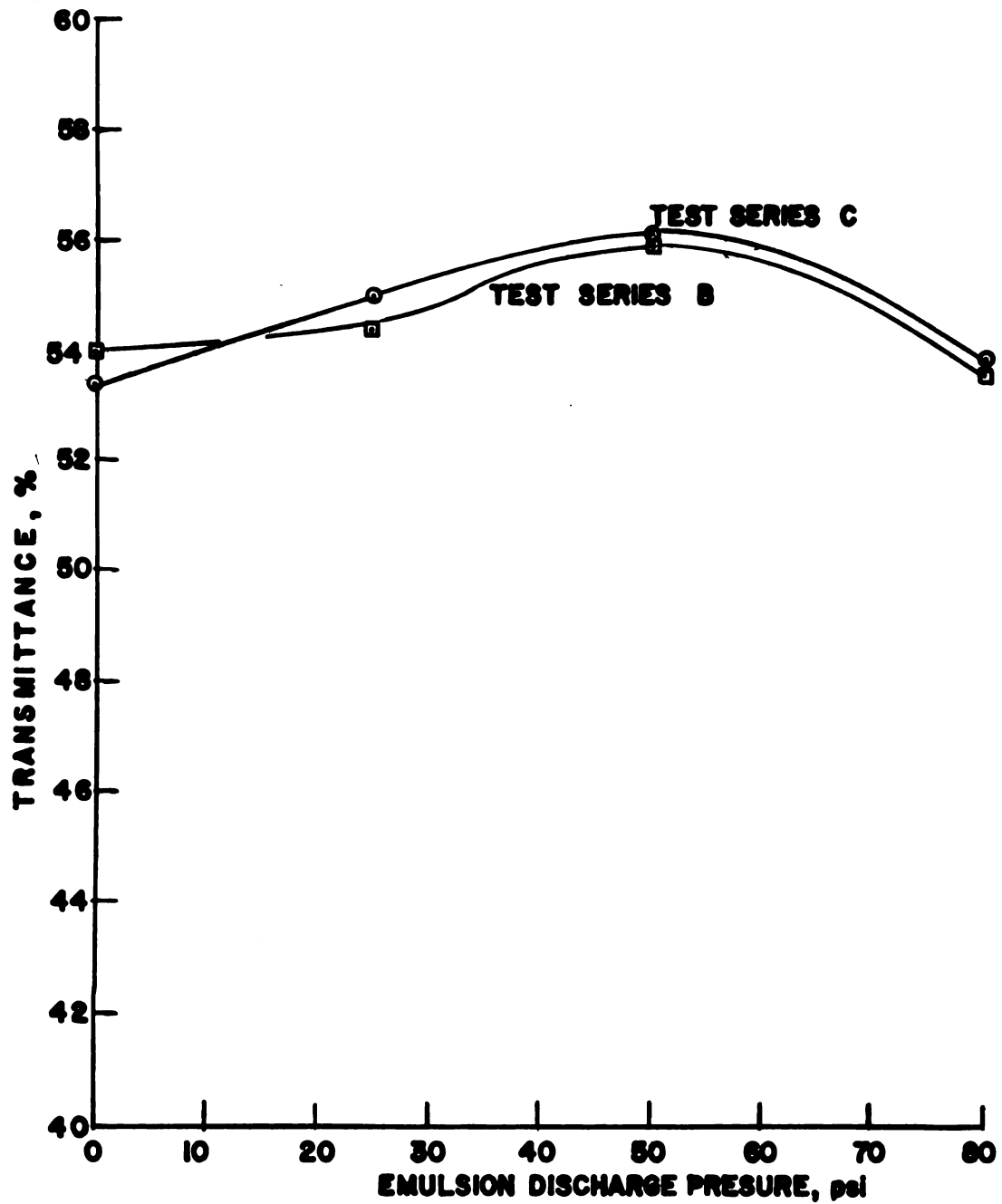


Figure 22. Effect of emulsion pressure on discharge side of injector on globule size reduction. (Steam energy input--175 Btu/lb of emulsion)

injected steam with 50 psi discharge pressure was equivalent to 2050 psi in the laboratory milk homogenizer.

Since the effects of discharge pressure on globule size reduction were compared at the same level of steam input, the differences in homogenizing effect must be attributed to the mechanisms surrounding the formation and/or collapse of the injected steam bubbles. Loo (1952, 1953), using a valve designed to produce cavitation in a pressure homogenizer, found that some back pressure on the valve increased the homogenizing effect, but an optimum pressure was reached after which pressure reduced the homogenizing effect. The increase in homogenization with back pressure was attributed to a more intense striking effect of the collapsing cavity and the decrease in homogenization above the optimum pressure to suppression of vapor cavity or bubble formation.

The increased homogenization associated with increased discharge pressure in the steam injection tests is explained similarly by a more rapid bubble collapse and more intense shock wave. The decrease in homogenization above the optimum pressure is not immediately apparent. Since the same quantity of steam is injected, it would appear that the size and the penetration of injected steam bubbles might be influenced by emulsion pressure and thus affect size reduction. Information concerning those factors was not available. More definite knowledge concerning the cause of the optimum pressure must

await further investigation of the bubble injection and collapse phenomenon.

Effect of Flow Rate on Globule Size Reduction

The velocity and Reynolds number of the emulsion at the injector were noted as potential sources of influence on steam bubble formation and collapse and was presented in the section, "Analysis of Factors That May Influence Homogenization by Steam Injection." Emulsion velocity may affect initial bubble size and shape, and Reynolds number may affect the rate of heat transfer from the steam bubble. Both velocity and Reynolds number varied directly with flow rate in the injector and both factors were expected to increase the homogenizing effect if they were important variables.

The results of Test Series D, presented in Fig 23, indicated that velocities (20 to 60 ft/sec) and Reynolds numbers (38,000 to 115,000) obtained with flow rates of 3 to 9 gpm did not affect size reduction under the test condition employed.

Effect of Initial Globule Size on Size Reduction

Emulsions prehomogenized at zero, 1500, 3000, and 4500 psi to give different initial globule size distributions were further homogenized by the 347°F steam-injection treatment. The transmittance of these samples from Test Series E is presented in Fig 24. Since the points formed a straight line for all except the zero psi homogenized emulsion the slope of the curves, including the slope of the straight line portion of

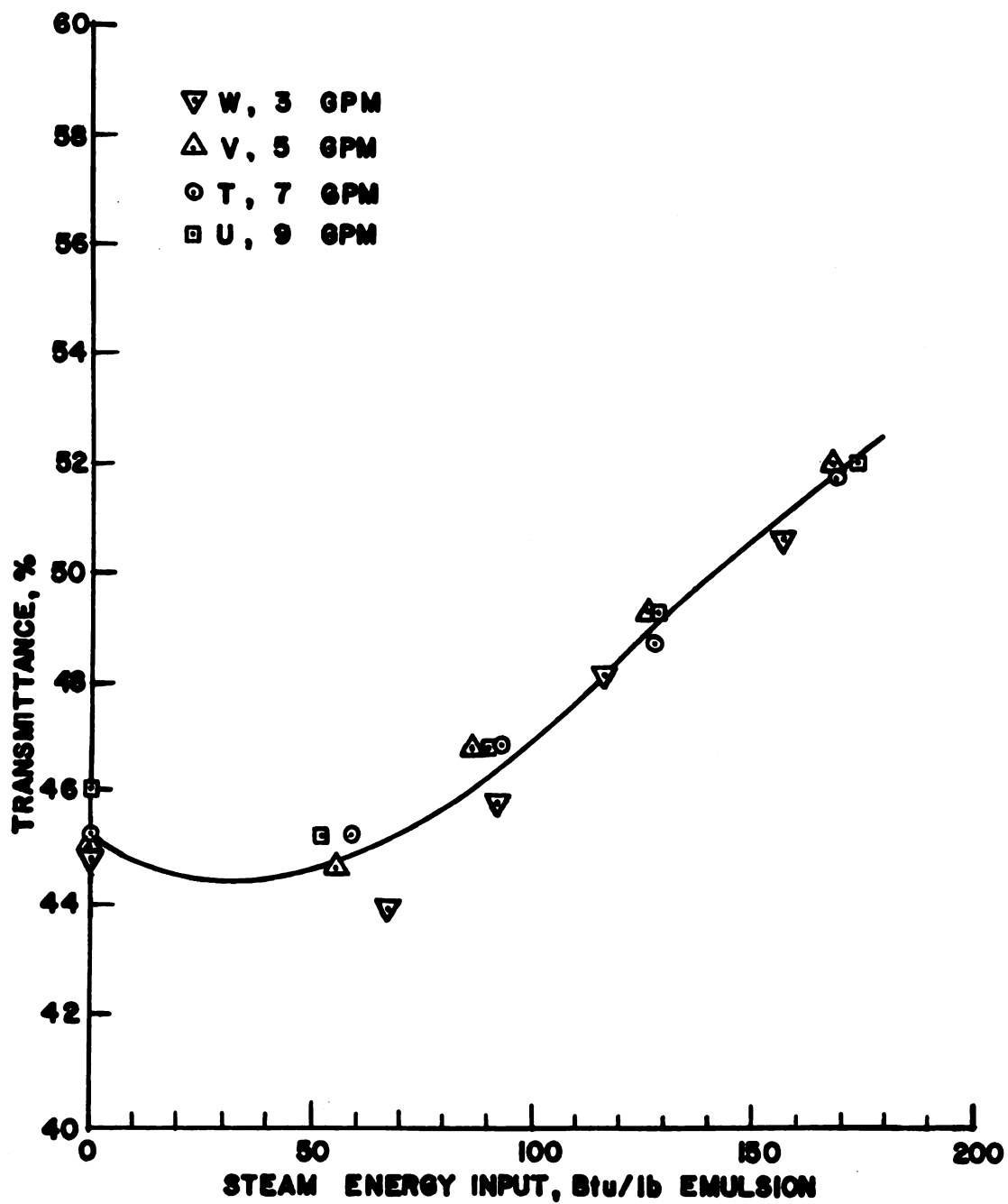


Figure 23. Effect of emulsion flow rate on globule size reduction by steam injection

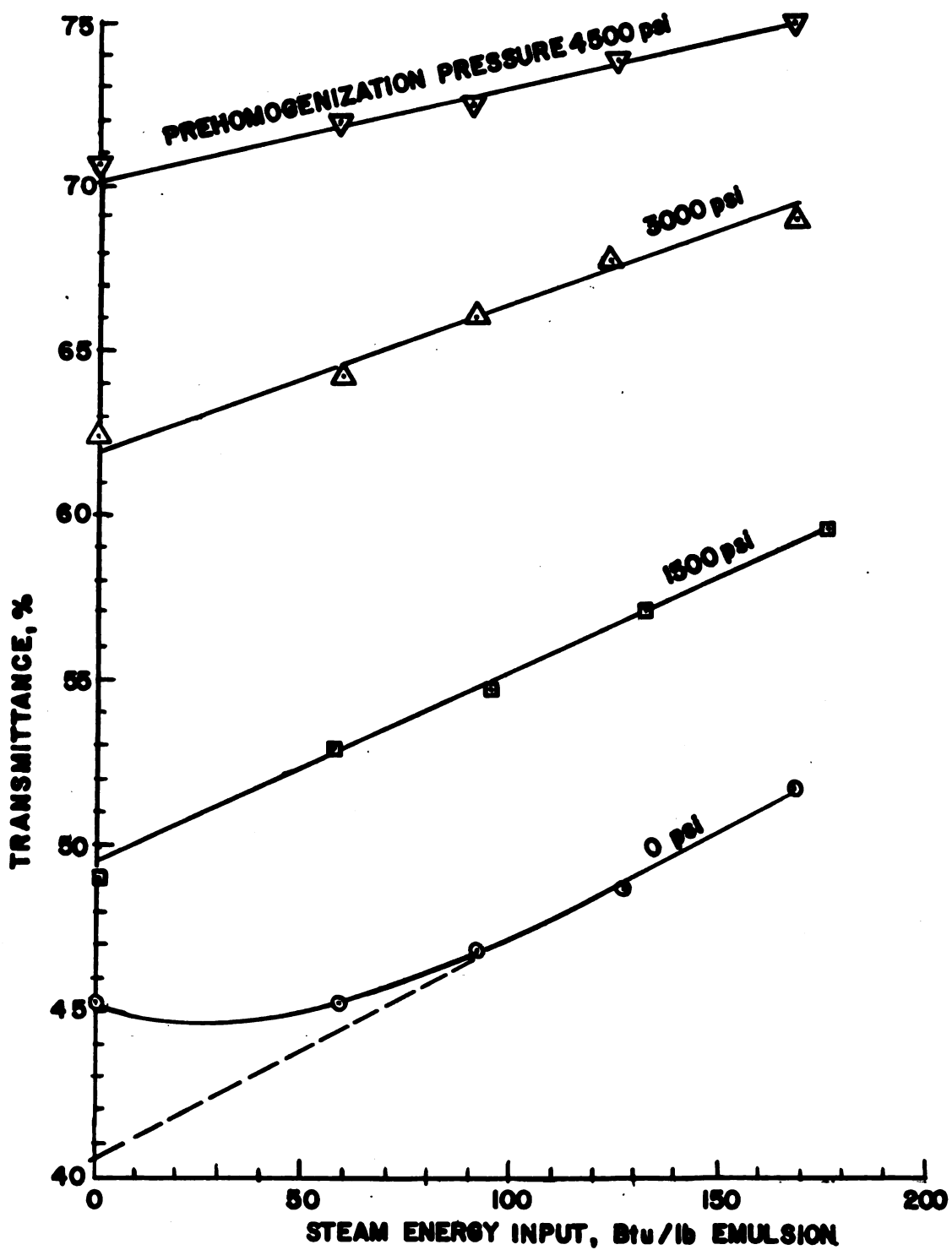


Figure 24. Effect of initial globule size on subsequent size reduction by steam injection

the zero psi homogenized sample, was plotted versus homogenization pressure in Fig 25. This transmittance-energy slope versus homogenization pressure curve also appeared to be a near straight line. The decrease in transmittance-energy slope with increased homogenization pressure suggests less efficient globule size reduction by steam injection of small globule emulsions.

Although the slope of the transmittance-steam energy input curves decreased with increasing homogenization pressures, the straight-line relationship of transmittance and steam energy above 1500 psi suggests that higher prehomogenization pressures could be employed successfully in future studies.

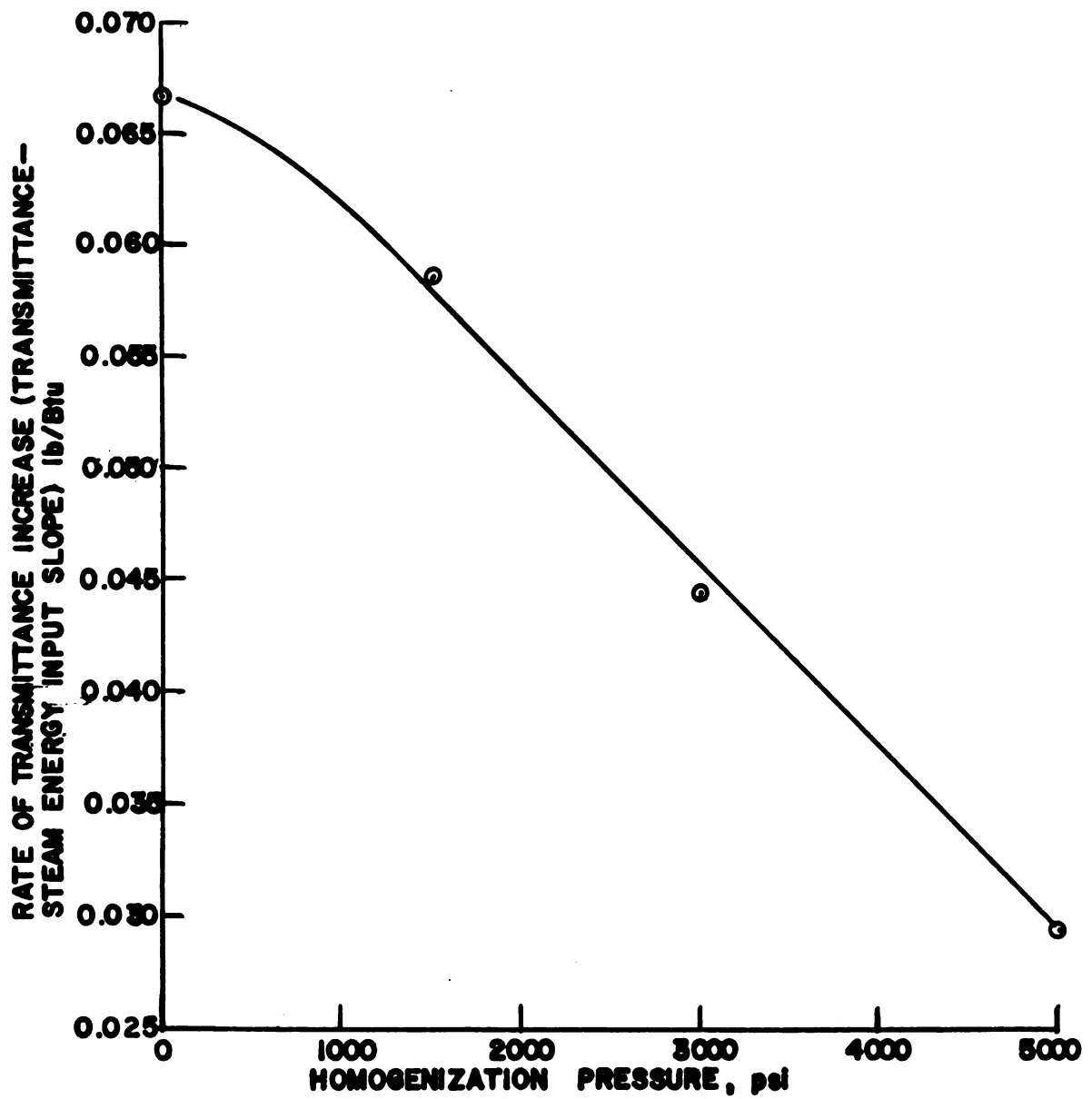


Figure 25. Rate of transmittance increase with steam energy input as a function of emulsion prehomogenization pressure

CONCLUSIONS

1. Homogenizing effect increased substantially with temperature of injected dry saturated steam in the range of 237° to 347°F. The 347°F steam-injection treatment of 175 Btu/lb of emulsion produced transmittances equivalent to 1800 psi homogenization in a laboratory-model conventional milk homogenizer.
2. Emulsion pressure on the discharge side of the injector had much less effect on globule size reduction than steam temperature. Although the differences in transmittance were not large, there appeared to be an optimum discharge pressure for size reduction. A discharge pressure of 50 psi with 346°F steam at the rate of 175 Btu/lb of emulsion gave homogenization equivalent to 2050 psi in a laboratory-model conventional milk homogenizer. With pressures of zero, 25, and 80 psi, the homogenizing effect was slightly less.
3. The changes in velocity and Reynolds number obtained with different flow rates through a particular injector did not affect the size reduction of globules as determined with the optical test with steam at 346°F.
4. Emulsions, prehomogenized at zero, 1500, 3000, and 4500 psi in a conventional milk homogenizer, underwent further

size reduction when subjected to the 346°F steam treatment. Emulsion transmittance changed less per unit steam energy input as prehomogenization pressure increased, suggesting less efficient homogenization of small globule emulsions.

5. Since the factors which improved globule size reduction effects are known to increase steam bubble collapse rate and shock waves, the globule size reduction accompanying steam injection was attributed to the cavitation effect produced by the rapid collapse of injected steam bubbles.
6. A combination homogenization-sterilization process employing steam injection into liquid food products appears to be feasible when high enough temperature steams can be used. Since only a few of the factors affecting globule size reduction were investigated, further study of optimum conditions for homogenization are necessary for efficient operation.

FUTURE RESEARCHES SUGGESTED BY THIS STUDY

1. A comparison of optical effectiveness of homogenization results with globule surface area as determined with a electric sensing-zone particle analyzer.
2. An evaluation of globule size reduction effects with steam temperatures above 347°F.
3. A study of the relationship of sound frequency and intensity, shock waves, and homogenizing effect of steam injected into an emulsion.
4. A determination of the effect of air incorporation in the emulsion and in the injected steam on the size reduction of globules.
5. An investigation of the relative merits of saturated and superheated steam for homogenization.
6. A study of injector design factors influencing homogenization by steam injection.

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APPENDIX

Table 3. Water meter calibration, I

Pump varidrive setting	Temp. °F	Pres- sure psi	Approx. flow rate gpm	Meter reading gal	Water weight lb	Meter factor gal wt gal meter
0	56	0	1.1	10.0	112.38	1.337
1.5	64	0	2.2	20.0	172.06	1.033
1.5	65	0	2.2	20.0	171.19	1.028
1.5	72	0	2.2	21.0	286.56	1.068
1.5	157	0	2.2	20.0	177.94	1.089
1.5	66	80	2.2	20.0	172.00	1.033
1.5	66	80	2.2	19.5	169.44	1.044
3.0	57	0	3.4	15.0	126.81	1.015
3.0	61	0	3.4	20.0	169.50	1.018
3.0	64	0	3.4	20.0	170.19	1.022
3.0	72	0	3.4	22.0	188.38	1.030
3.0	156	0	3.4	21.0	182.56	1.064
3.0	67	80	3.4	21.0	183.44	1.050
3.0	68	80	3.4	19.5	170.56	1.029
5.9	58	0	6.2	19.0	161.19	1.018
5.9	62	0	6.2	20.0	169.69	1.019
5.9	64	0	6.2	20.0	169.50	1.018
5.9	73	0	6.2	23.0	196.13	1.025
5.9	159	0	6.2	23.0	195.50	1.044
5.9	68	80	6.2	19.7	168.75	1.029
5.9	69	80	6.2	20.0	171.75	1.032
9.0	58	0	9.7	20.0	171.44	1.029
9.0	62	0	9.7	20.0	170.38	1.023
9.0	63	0	9.7	20.0	170.19	1.022
9.0	74	0	9.7	23.0	196.38	1.027
9.0	160	0	9.7	20.0	170.69	1.048
9.0	70	80	9.7	20.0	169.75	1.020
9.0	70	80	9.7	20.0	170.00	1.021
9.0	75	80	9.7	23.0	195.50	1.022

Table 4. Water meter calibration, II

Pump varidrive setting	Temp. °F	Pres- sure psi	Approx. flow rate gpm	Meter reading gal	Water weight lb	Meter factor gal wt gal meter
6.75	64	0	7.0	20.0	172.56	1.036
6.75	64	0	7.0	20.0	172.69	1.037
6.75	110	0	7.0	20.0	173.75	1.052
6.75	162	0	7.0	20.0	170.44	1.047
6.75	190	0	7.0	20.0	169.02	1.050