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Two-Phase Slug Flow Heat Exchanger for Microbial Thermal Inactivation Research

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A continuous two-phase (air-liquid), slug flow, tubular heat exchanger was developed for microbial thermal inactivation research to give exposure times and temperatures in the range of high-temperature, short-time milk pasteurization as well as heat-treated sample volumes of at least 2 ml. The use of air to compartmentalize the liquid in the capillary tubing prevented the development of laminar flow, which enabled precise identification of the residence time of the fastest flowing particles in the heating, holding, and cooling sections of the instrument. Residence time distributions were quantitated by measuring the degree of spreading of radioactive tracers for water, whole milk, chocolate milk, cream, and ice-cream mix with holding temperatures from 50 to 72 C, holding times from 2 to 60 sec, and heating and cooling times of 1.7 sec each. For a holding time of 60 sec and a fastest particle velocity of 10.2 cm/sec, the velocity ratios of the fastest moving particle to the median particle were 1.05, 1.05, 1.10, and 1.13 for whole milk, chocolate milk, cream, and ice-cream mix, respectively. With shorter holding times, these velocity ratios were even closer to unity. These velocity ratios indicated that the instrument would be an effective tool for thermal inactivation research on microorganisms suspended in homogeneous fluids with a viscosity of 15 centipoises or less at the exposure temperature.

The devices which have been used to study thermal inactivation of microorganisms range from simple thermal death-time tubes to sophisticated heat exchangers capable of automatic operation. With the general trend in the food industry toward processing at higher temperatures for shorter holding times, laboratory instrumentation such as the thermoresistometer (5) and its modifications for holding temperatures below 100 C (2) were developed to be effective at the new processing conditions.

For studies on thermal inactivation of viruses in dairy products, an instrument was needed which would be effective in the temperature range of the modified thermoresistometer (50 to 90 C); however, large sample volumes of heat-treated material (2 ml) were needed for virological assay. The sample volumes from the modified thermoresistometer are limited to 0.01 ml; consequently, it could not be utilized in this study. Relatively large sample volumes of liquids with short heating, holding, and cooling times are easily obtained from a tubular heat exchanger (4); however, the laminar flow profile commonly found in this type of heat exchanger precludes precise identification of holding time. The use of two-phase flow has been shown to effectively break up the laminar flow profile in capillary tubing; consequently, it enabled precise identification of the fastest particle residence times (7). The purpose of this study was to develop and evaluate a two-phase (air-liquid), slug flow heat exchanger for thermal inactivation research with short heating, holding, and cooling times and sample volumes of at least 2 ml.

MATERIALS AND METHODS

Design. A diagram, which shows the principal sections of the instrument, is presented in Fig. 1. The microbial suspension was transported by regulated pressurized air from reservoir storage through a flow meter to the mixing tee where it was mixed with air to establish stable slug flow (no air bubbles agglomerated). The enlarged section of nylon tubing (0.24 cm inside diameter; Imperial Eastman 22 SN) in Fig. 1 shows the nature of the two-phase slug flow during operation. The compartmentalized slugs were heated to the desired holding temperature in 1.7 sec with hot water in the stainless-steel heating section (0.24 cm inside diameter). The heating water temperature and flow rate were controlled with a constant temperature circulator. The slugs then traveled through a submerged nylon holding tube of known length to give a predetermined holding time. From the holding section, the temperature of the slugs was reduced to ambient in 1.7 sec with tap water in the stainless-steel cooling section (0.24 cm inside diameter).

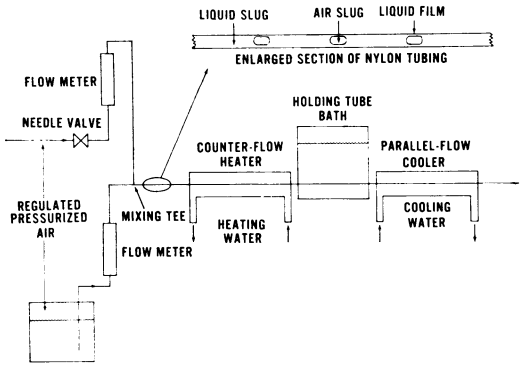

FIG. 1. *Flow diagram of the slug flow heat exchanger.*

Operation. Water, whole milk, chocolate milk, cream (38% milk fat), and ice-cream mix (16% milk fat) were tested with holding temperatures from 50 to 72 C, holding times from 2 to 60 sec, and fastest particles velocities of 10.2 and 20.3 cm/sec. With these two velocities, the volumetric flow rates of liquid were 20 and 40 ml/min, respectively. The liquid-to-air ratio was approximately 5 for all tests with a liquid-slug length of 0.6 cm and an air-slug length of 0.12 cm.

Liquid velocity through the instrument was controlled by varying the air pressure on the liquid in the stainless-steel reservoir. Air flow through the instrument was controlled by varying the pressure drop of the air through the needle valve. Fastest particle residence times were determined by visual observation of dye injections, which necessitated that the nylon tubing be transparent.

Temperature measurement. Slug temperatures were monitored by miniature thermocouples (Baldwin-Lima-Hamilton #TCC-FS-50), which were mounted into the nylon fittings used to connect the various sections of the capillary tubing. Heating and cooling water temperatures were measured with 0.3-cm armored, stainless-steel thermocouples. A temperature profile through the instrument during operation is presented in Table 1.

Residence time distribution measurement. Measurements for residence time distribution were obtained by injecting a pulse input of a radioactive tracer (iodine-131 carrier free) prior to the heating section and quantitating the degree of spreading of the tracer at the exit of the exchanger. A gamma-sensitive scintillation detector (no. 821330) coupled with a pulse-height analyzer (no. 8742) and a ratemeter (no. 8732), all manufactured by Nuclear Chicago Corp., Des Plaines, Ill., were used to measure the spreading of the tracer. After the injected mixture (approximately 5% iodine-131, 10% food dye, and 85% test product by volume) had traveled through the heating, holding, and cooling sections, the flow was stopped in a 150-cm section of nylon tubing by capping each end of the auxiliary section. The food dye was used to enable easy identification of the leading edge of the tracer mixture. The scintillation detector was placed on each 5-cm section of tubing from the leading edge of the tracer to the end of the auxiliary section, and the counts per minute were manually recorded from the ratemeter dial for each 5 cm. Each of the readings of counts per minute was then converted into a percentage of the total pulse input (approximately 32,000 counts/min) from which the complete distributions could be calculated.

TABLE 1. *Temperature profile during operation*

Position	Temp (C)
Liquid in reservoir	3
Slugs at the heater inlet	20
Slugs at the heater inlet with preheater[a]	45
Exposure temp	50 to 72
Heating water	3.3 > exposure temp
Cooling water	20
Slugs at the cooler outlet	23

[a] Preheater used with cream and ice-cream mix only.

RESULTS AND DISCUSSION

Heating. One advantage of slug flow over laminar flow is the shorter time required for heating and cooling. When heat transfer occurs during laminar flow, continuity, momentum, and energy differential equations can be written for a given system. The Graetz solution as proposed by Hausen (3) for a constant wall temperature and parabolic velocity distribution system predicts a mean Nusselt number of 4.7 for water flowing at 20 ml/min in a circular tube (0.24 cm inside diameter and 16.5 cm in length). The predicted heat transfer coefficient for laminar flow calculated from this Nusselt number is 1025 kcal/m² hr °C. The experimental heat transfer coefficients were in the range of 950 kcal/m² hr °C. Although heat transfer coefficients for slug flow can not be estimated from theoretical considerations, the experimental coefficients were in the range of 2200 kcal/m² hr °C for water flowing at 20 ml/min.

At the higher exposure temperatures (70 C) after prolonged usage, whole milk tended to foul the surface of the stainless-steel heaters, which reduced heat transfer efficiency. Treatment with phosphoric acid solution and pipe cleaners was successful in removing this scale from the affected heaters. The fluids with a high percentage of milk fat, such as cream, did not exhibit this problem.

Liquid-to-air ratio. Several ratios (the length of a liquid slug divided by the length of an air slug) were tested during preliminary evaluations of the instrument. These ratios were measured by volumetric calibrations from the flow meters

or with a ruler when the flow was stopped. At high ratios (> 10), the stable slug flow pattern tended to break down (air slugs agglomerated), which made identification of the fastest particle residence time difficult. Continuous temperature measurements at the holding inlet showed uneven heating (± 2 C) at these high ratios. At low ratios (< 2.5), the pressure losses in the capillary tubing were prohibitively high during operation, and the liquid flow control was unstable because of the large volume change of the air phase in the tubing during heating and cooling.

From these tests, the ideal liquid-to-air ratio was low enough for even slug heating at the holding inlet and for development of stable slug flow and high enough for stable liquid flow control. A ratio of approximately 5 was found to be an acceptable compromise for all fluids tested.

Higher viscosity fluids. Attempts to test ice-cream mix on the instrument were unsuccessful because of the high pressure losses in the capillary tubing. By preheating the fluids to approximately 45 C, the pressure losses were reduced sufficiently due to the reduction in viscosity, so that cream and ice-cream mix could be tested satisfactorily.

Residence time distributions. The use of air to compartmentalize the liquid broke up the laminar flow profile that would exist in single-phase flow. Some liquid particles, however, still had a higher velocity than other particles because of a film of liquid adhering to the tube surface in the air phase of the flow. As the liquid moved through the tube, it picked up material at its leading edge and deposited material from its trailing edge. Consequently, all liquid particles in one slug at the holding inlet would have a residence time distribution rather than one single residence time. It was necessary to quantitate this distribution for a series of selected test conditions so that the microbial thermal inactivation curves could be corrected for this effect (Fig. 2).

Abbreviated residence time distribution data are commonly reported as velocity ratios (the velocity of the fastest flowing particle divided by the velocity of the average flowing particle). In this study, the velocity of the median flowing particle was used instead of the velocity of the average flowing particle because of the significant tailing of the distributions (Fig. 2). For each fluid tested, maximum velocity ratios calculated from the radioactive tracer data are shown for holding times up to 60 sec in Table 2.

The higher viscosity fluids, such as ice-cream mix, deviated more from perfect slug flow (velocity ratio of 1.00) than did the lower viscosity fluids. Tests also showed that the same fluid deviated more from perfect slug flow at a velocity of 20.3 than at 10.2 cm/sec. When the velocity ratios of the slug flow system, however, were compared to those of single-phase laminar flow (Table 2), the use of air to compartmentalize the liquid was found to be an effective technique of approaching perfect slug flow.

Holding time corrections. Due to the inherent

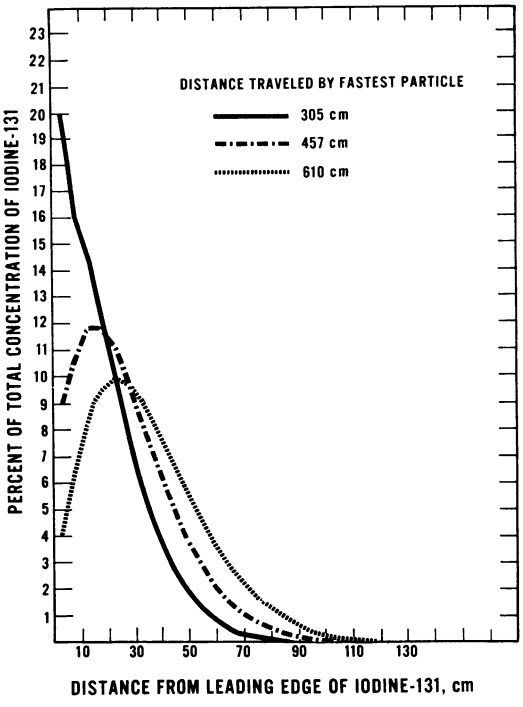

FIG. 2. *Residence time distributions for three holding-tube lengths at a velocity of 10.2 cm/sec for whole milk.*

TABLE 2. *Maximum velocity ratios of the fastest to the median particle for each fluid through 610 cm of slug flow*

Fluid	Fastest particle velocity (cm/sec)	Velocity ratios (V_{max}/V_{med})[a]
Milk	10.2	1.05
Chocolate milk	10.2	1.05
Cream[b]	10.2	1.10
Ice-cream mix[b]	10.2	1.13
Milk	20.3	1.08
Chocolate milk	20.3	1.08
Water	20.3	1.05

[a] Velocity ratio for fully developed laminar flow is 2.0.

[b] Data taken on modified instrument with preheater.

characteristics of continuous-flow heat exchangers, both heating (come-up) time and residence time distribution effects necessitate exposure time corrections on the microbial thermal inactivation curves. For example, Fig. 3 shows an uncorrected viral inactivation curve (6) in ice-cream mix and the corrected curve which properly accounts for the heating and residence time distribution effects. The uncorrected curve is the equation of a second-order polynominal which was fitted to the log survivors versus fastest particle exposure time data by the method of least squares.

The time correction for heating effects, calculated by the method of Ball and Olson (1), was 0.3 sec for the lower viscosity fluids and 0.6 sec for the higher viscosity fluids. The increase in the magnitude of the correction for the higher viscosity fluids was due to the use of the preheater which increased the fluid temperature at the heater inlet. A z value of 5.6 C was assumed for the purpose of calculating these heating time corrections.

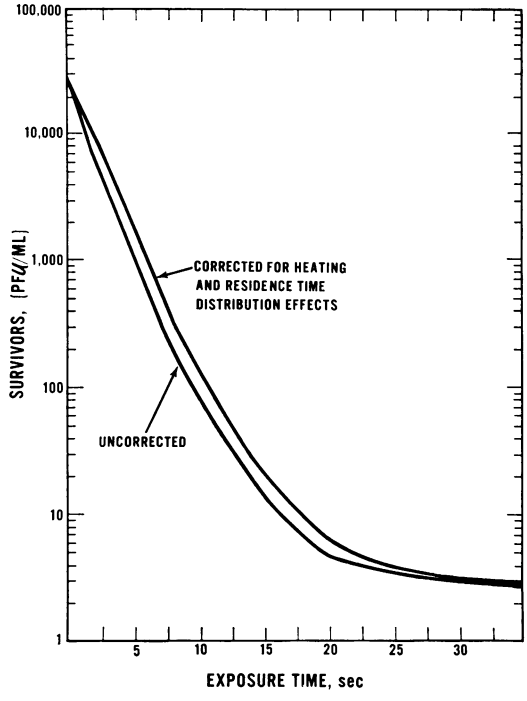

FIG. 3. *Thermal inactivation curve of a virus in ice-cream mix corrected for heating and residence time distribution effects.*

The correction for residence time distribution effects was calculated by utilizing Simpson's rule. For ice-cream mix as the test liquid, this correction ranged from 0.2 sec at a 2-sec exposure to 3.5 sec at a 30-sec exposure. Correcting for the lower viscosity fluids would be performed in a similar manner; however, the magnitude of the correction would be less than that for ice-cream mix because of less deviation from perfect slug flow. Comparison of the uncorrected and corrected curves in Fig. 3 shows that the distribution effects necessitate an approximate 15% increase in the measured fastest particle exposure time for ice-cream mix. Similar comparisons for cream, milk, and water necessitate approximate 12, 10, and 7% increases in the measured fastest particle exposure time, respectively.

In summation, the slug flow heat exchanger appeared to be an effective tool for thermal inactivation research on microorganisms suspended in homogeneous fluids with a viscosity of 15 centipoises or less at the exposure temperature.

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