i’Tec_S11
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Keywords: Distribution, Residence time, Time, Treatment, Principle.

Measuring treatment times
Concept of residence time distribution

1. Industrial context

Several studies on animal feed heat treatments have revealed that Salmonella bacteria in such products can be destroyed by applying a time/temperature scale (moisture content).

Sector industrials generally apply these treatments in the form of continuous processes. Their effectiveness depends mainly on the control of time and temperature factors; the time factor is the most difficult to measure and control, due to spread in residence time distribution patterns.

When a product is treated in a continuous system (or reactor-mixer), particles do not all pass through the system at the same speed resulting in differing treatment times; this is the phenomenon responsible for residence time distribution spread or RTD.

This phenomenon is of major importance in a thermal destruction process targeting microorganisms as the particles with the shortest residence times may leave the facility before decontamination is complete.

It is therefore essential to have accurate data on residence time distribution in order to be able to qualify the length of treatment times in a continuous process.

Measuring particle residence times in the process and grouping this data by category makes it possible to:

- plot the distribution of residence time categories on a graph,
- use parameters to describe particle populations based on their residence times.

2. The various flow types in a reactor-mixer

A range of flow types has been identified by applying residence time distributions to research into how mixes flow in a continuous mixer; these types include 2 optimal flows, plug flow and optimal mix flow.

- With plug flow, the product passes through the reactor area at a constant and uniform speed. Each particle that enters the reactor moves forward section-by-section without mixing with other particles of different ages.
- With an optimal mix flow, the mix composition is uniform throughout the whole mixer-reactor volume.

In practice, any mixer-reactor will often show intermediate curves between these two models.
These 3 flow models are illustrated in Figure 1 and Figure 2.

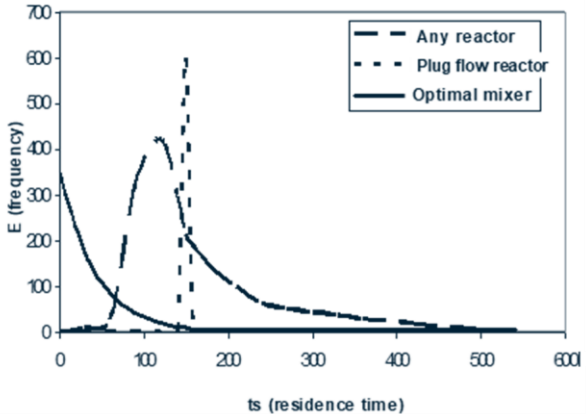

Figure 1: Various flow models - Frequency density of residence times

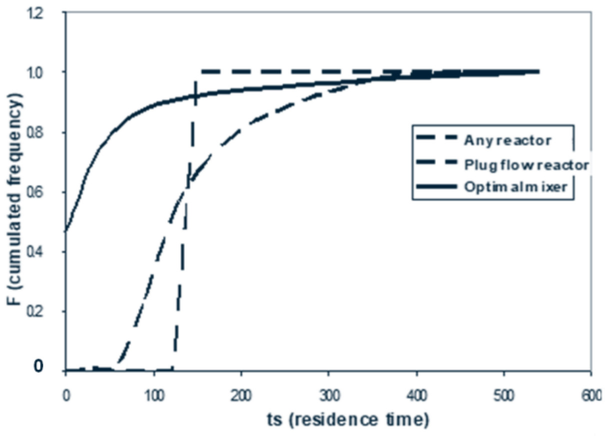

Figure 2: Various flow models - Cumulated frequency of residence times

3. Using tracers for experimental determination of RTD

3.1. Principle

The method consists in injecting a tracer at the mixer-reactor input and detecting it as it exits.

3.2. Injecting the tracer

There are two injection methods - step injection and pulsed injection.
In step injections, the tracer is injected throughout the test period; this requires a large quantity of tracer together with precision control over the rate at which the tracer is injected.
In pulsed injections, a given quantity of tracer is injected at the reactor-mixer input over a very short time period, less than 0.01 of the throughput time (J. Villermaux, 1993). This is the method used by the animal feed industry as tracer flow rates do not have to be controlled and the injection only uses a small amount of tracer.
Tracer injection conditions have been described by J.J. Bimbenet (1995) and J. Villermaux (1993, 1996). These authors consider that injections should only be performed when the system is stationary and the tracer does not disturb reactor operation. It is preferable to inject tracers into small, turbulent areas thus ensuring that the tracer mixes into the product. Tracer injection should be distributed over the whole cross-sectional area of the input point for the system under study. The quantity of injected tracer is determined based on the amount of tracer that can be analysed at conditioner output and the tracer injection method used.

3.3. Taking samples at the reactor-mixer output and tracer dosage

Samples are taken as close as possible to the mixer-reactor in order to avoid having to take account of disturbances generated by downstream circuits and equipment when determining the reactor-mixer's RTD.
With pulsed injections, the duration of the sampling phase should be longer than the treatment time and the time taken between two samples should be calculated so as to obtain approx. 30 to 40 samples. This is the number of samples required to obtain a representative residence time distribution.
The tracer dosing method is selected according to which tracer is injected into the reactor-mixer (Tecaliman 2004 a).

3.4. Processing pulsed injection data

Once processed, this data can be used to construct RTD curves (Loncin 1976) and to calculate a certain number of variables used to characterise residence time distribution.

3.4.1. Frequency density curve

The x-axis represents the age categories "Ti" or residence time category in the process.
The y-axis represents the ratio of particles belonging to a given age category "Ti". (Figure 3).

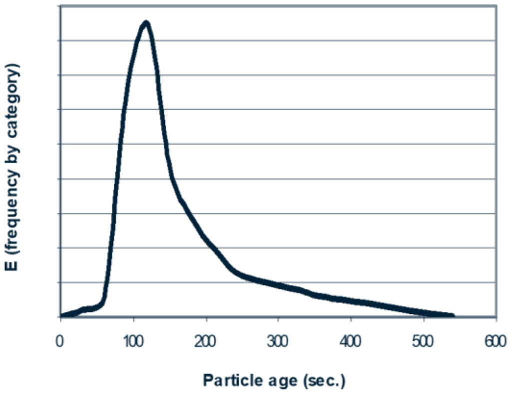

Figure 3: RTD type curve - Function E (residence time frequency density)

3.4.2. Cumulated frequencies curve

The x-axis represents the age categories "Ti" or residence time category in the process.
The y-axis represents the cumulated probability densities or cumulated frequencies "F" of particles classified according to their external age (Figure 4).

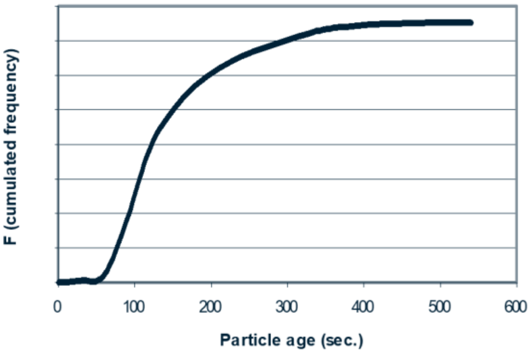

Figure 4: RTD type curve - Function F (residence time cumulated frequencies)

3.4.3. Standardised curves

This illustration of RTD gives time and concentration scales that are independent of reactor flow rate and of the amount of tracer injected or recovered (Figure 5 and Figure 6).
In this graph, the x and y axes are as follows:

- x-axis - reduced time, which is the ratio between the actual time and the throughput time (Tp, throughput time, is equal to the mass of product in the process divided by the process rate - Tecaliman 2004 c).
- Y-axis - reduced concentration, which is the tracer concentration divided by the concentration that the tracer would have if it was evenly distributed throughout the product in the

reactor-mixer.

The method used to calculate reduced times and concentrations is described in **i'Tec_S13** (Tecaliman 2004 b).

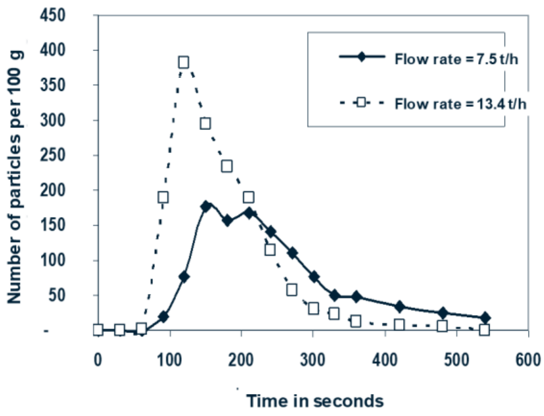

Figure 5: RTD curves - Function E (residence time frequency density) - Raw data

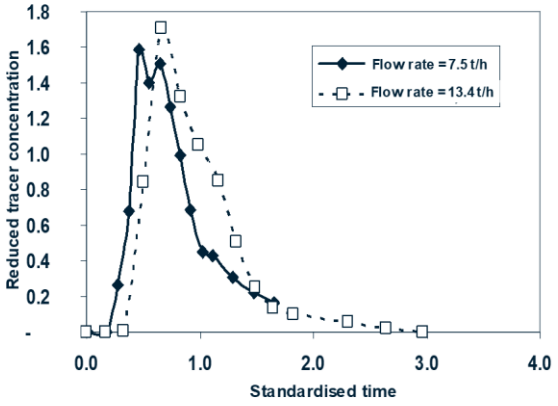

Figure 6: RTD curves - Function E (residence time frequency density) - Standardised data

3.4.4. Variables used to characterise RTD

3.4.4.1 General case

3.4.4.1.1 Tracer recovery rate

The recovery rate equals the ratio between the amounts of tracer injected into and recovered from the mixer. It is expressed as a % and should lie between 80 and 110%.
A recovery rate of under 100% may indicate that:

- the tracer in the mixer-reactor has been partially destroyed,
- the amount of tracer injected has been overestimated,
- systematic dosage errors have been made in samples taken at conditioner output,
- the sampling does not represent product flow at conditioner output (e.g. low or irregular sampling frequency or incorrect measurement of the time taken between samples),
- the sampling period is too short, i.e. sampling ceased while some tracer particles still remained in the conditioner.

A recovery rate of over 100% may indicate that:

- the amount of tracer injected has been underestimated,
- the treated product already contained some tracer prior to injecting the tracer in the reactor-mixer,
- systematic dosage errors were made in samples taken at reactor-mixer output,

3.4.4.1.2 Mean particle residence time in the process: $\bar{X}$

This is an estimate of the mean treatment time.

3.4.4.1.3 Median time: $\mathbf{Me}$

This indicates the time required for 50% of the marked particles to pass through the mixer-reactor.

3.4.4.1.4 Dominant value or mode: $\mathbf{Mo}$

This corresponds to the most frequent residence time.

3.4.4.1.5 Minimum residence time

The minimum product residence time in the reactor-mixer is the time at which the first tracer particles are identified at the reactor-mixer output.

3.4.4.1.6 The period between the time where 84 and 16% of particles were resident in the process

This criterion is used to evaluate the residence time distribution spread. It is calculated based on cumulated data using times T16% and T84% as the reference boundaries, which corresponds to a particle population of 68% (84-16=68). A low value for this criterion would indicate a strongly centred residence time distribution, or a steep cumulated function curve (F).

3.4.4.2 Normal distribution of residence times

3.4.4.2.1 Mean, mode and median

In this case, the mean, the median and the dominant (modal) value are all very close (B. Scherrer 1984); various statistical parameters can be calculated in order to characterise RTD.
The residence time distribution curve (Function E) can be characterised by comparing the mean against the mode and the median:

- Where $\bar{X} = \mathrm{Mo} = \mathrm{Me}$, the distribution curve is symmetrical
- Where $\mathrm{Mo} \langle \mathrm{Me} \langle \bar{X}$, the distribution curve is asymmetrical, right - it shows a tail.
- Where $\bar{X} \langle \mathrm{Me} \langle \mathrm{Mo}$, the residence time distribution is asymmetrical, left.

3.4.4.2.2 Variance and standard deviation

These two parameters are used to evaluate the spread of the residence time distribution frequency curve. High variance or standard deviation values indicate a large residence time distribution spread.

3.4.4.2.3 Skewness coefficient for the residence time distribution curve: $\alpha 3$

A coefficient equal to 0 indicates a symmetrical Function E curve, while a coefficient greater than 0 indicates that the residence time spread tails out (the curve spreads to the right), and a coefficient less than 0 indicates a distribution that tails out to the left.

3.4.4.2.4 Flattening coefficient for the residence time distribution curve: $\alpha 4$

This coefficient is independent of the distribution variance and measuring unit and system. It is used to compare distributions one against the other (Function E).

- Where $\alpha 4 = 3$, the flattening of the residence time distribution curve indicates a normal distribution.
- Where $\alpha 4 \rangle 3$, the residence time distribution curve is narrower than a normal curve.
- Where $\alpha 4 \langle 3$, the residence time distribution curve is wider than a normal curve.

4. Applying the residence time distribution concept in the animal feed industry

4.1. Equipment design

In a continuous process, the spread of residence time distributions increases as the usable volume of the reactor-mixer increases (Villermaux 1996). As the treatment time depends on the ratio between the flow rate and the actual volume used, this means that large-volume reactors-mixers have to be used for high flow rates.

In the animal feed industry, several types of equipment have been designed to address this problem:

- double jacketed thermal conditioner with co-rotating shafts,
- conditioners connected in series,
- conditioner connected to a multi-stage soaking vessel.

4.2. Comparison between equipment or equipment operation

Converting raw data on residence time distributions into standardised data makes it possible to compare various equipment or determine mean residence times at varying flow rates in a process with a known RTD (J. Lamoine 1999).

5. Bibliography

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