

SciVerse ScienceDirect

Procedia Food Science 1 (2011) 1272 – 1277



11th International Congress on Engineering and Food (ICEF11)

Temperature Integrators as tools to validate thermal processes in food manufacturing

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Abstract

Characterisation of thermal processes remains is challenge as it involves rotating parts and reasonably high temperatures thus making use of measurement techniques such as thermocouples unusable. Time Temperature Integrators (TTIs) has been suggested as an alternative that would provide information that would allow process design and validation. In this work we aim to validate the use of TTIs as measurement tools to validate both the effect but also the uncertainty of thermal processes in conditions relevant to industrial processing. P values estimated using TTIs compared favourably with those obtained from thermocouple measurements for a range of temperature profiles relevant to food processing. Efficiency of large mixing vessels has been quantified in a pilot scale vessel. Factors such as fluid viscosity; fill level and heating options were examined. The results show that the free TTIs show higher P values than the thermocouple situated in the centre of the vessel (but similar to the thermocouple positioned on the wall of the vessel) while the TTIs fitted in balls correlate well with the centre thermocouple. The results indicate that the mixing performance is dependent on the fluid viscosity, the fill level and the heating options. As the free TTIs follow the fluid path they gave a more accurate representation of the real thermal impact on the food product. This work demonstrates that TTIs can be successfully used as a tool for validation of efficiency and uncertainty of industrial processes.

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Keywords: Thermal processing, validation, time temperature integrators

1. Introduction

The main objective of food manufacturers is to offer products that are safe while have the highest possible nutritional value. Despite the lack of clear understanding food manufacturers still have the challenge of producing large quantities of safe foods. This in practice is typically achieved by over

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Selection and/or peer-review under responsibility of 11th International Congress on Engineering and Food (ICEF 11) Executive Committee.

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processing resulting in a reduction of both nutritional and sensorial aspects of foods. With a current pressure in fresher, higher quality products over processing is a less attractive option and a better understanding of the significance of the processing variables should be obtained.

Characterisation of thermal processes remains is critical in this direction and still remains a challenge as it involves rotating parts and reasonably high temperatures. Typically Thermocouples and data loggers are used as measurement tools. Temperature is measured as a function of time inside the food container. This time temperature history is translated into an equivalent time at a reference temperature. This validation technique is straightforward to apply and the data analysis is relatively fast and easy. However, thermocouples and data logger are not convenient for all types of thermal processes. In some cases, they can interfere on the movement of the fluid and lead to incorrect time temperature history and sometimes it is impossible to put them at the coldest location of the container.

Time Temperature Integrators (TTIs) has been suggested as an alternative that would provide information that would allow process design and validation [1]. TTIs are devices that contain a thermally labile substance, which under a heat treatment undergoes irreversible changes that can be quantified as a F or P value [2]. TTIs are relatively small (a few cm in size), shock resistant monitoring devices, which can be made neutrally buoyant from materials with the same thermal conductivity as the food. Their shape can be modified to simulate food particles and they can be put inside a particle without interfering with the fluid motion.

Enzymatic TTIs are commonly used in determining the effectiveness of heat treatments, since they do not cause any food safety issues, unlike microbiological TTIs [2]. However, the selection of the enzyme can be difficult since the kinetics of enzyme inactivation has to be known and the *z* value should match the one of the organism of interest (typically $z = 8 - 10^{\circ}$ C) in order to be used as a food safety tool [3-4].

In this work we aim to validate the use of enzymatic TTIs as measurement tools to validate both the effect but also the uncertainty of thermal processes in conditions relevant to industrial processing.

2. Materials and Method

TTIs were prepared using α -amylase from *Bacillus amyloliquefaciens* and from *Bacillus licheniformis*.

A technique using a Peltier stage device has been developed to generate a range of temperature-time profiles used to determine the accuracy of Time Temperature Indicators [5].

The performance of large mixing vessels was quantified in a 250 lt, pilot scale vessel (Guisti, Campden Burton on Trent, UK) using 32 TTIs; one data logger fixed in the centre of the vessel and one thermocouple measuring the wall temperature. 20 TTIs were put at the start of the experiment together with 12 TTIs fitted in balls to avoid them becoming overcooked by passing too close to the hot vessel wall. The vessel was brought up to the set up temperature (83°C or 85°C) using the jacket heating with a holding time of 15 min [5]. The cooling was performed by both using the jacket and the vacuum cooling. In a separate set of experiments information about the flow field was obtained using Particle Image Velocimetry (PIV) and Positron Emission Particle Tracking (PEPT) in a scaled down version of the equipment [6].

3. Results and Discussion

It is of importance to establish the capability and accuracy of TTIs to measure the effect of thermal processing. TTIs were subjected to a range of temperature profiles relevant to industrial processes [7] using a Peltier stage. In Figure 1 one can see the P values estimated from the TTIs and the thermocouples. As one can see the results indicate that there is very good agreement between values predicted from thermocouples and those predicted from TTIs for a range of non isothermal processes. One can also see that when the holding time increases, the variability in P values estimated from both TTIs and

thermocouples increase. The increase in variability is lower in the case of thermocouples. Overall the error in the evaluation of thermal processes is less than $\pm 20\%$ when TTIs are used.

In practice, the accuracy of the TTIs will be constrained by (i) a lower limit of P, where there is sufficient thermal lag between the TTIs and the process, so that the TTI value is not accurate, and (ii) a higher limit of P, where the value of the enzyme activity is so low that it is not sensitive to the change in P. In between lies the operational window in which measurements can be taken with sufficient accuracy which is approximately 2 to 8 minutes at 85°C for *Bacillus amyloliquefaciens* and 5 to 30 minutes at 85°C for *Bacillus licheniformis*.



Fig. 1. P values of the thermocouples versus the P values of the TTIs for industrial time

Once evaluated the TTIs were then used to evaluate the performance of a pilot scale processing equipment. In Figure 2 P values as obtained from TTIs and thermocouples are shown. One can see from 2 that P values using tomato soup and a 5% starch suspension are similar. The P value of TTIs added after the heating time (marked HT TTIs) is similar to those added at the start of the experiment. This indicates that most of the heat treatment is achieved during the holding time. Once steam injection is used the difference between the *P* values of the thermocouples located on the wall and in the centre decreased, while the P values measured by the TTIs and the wall thermocouple increased, indicating a more uniform temperature field. As the TTIs that are in the cases (marked GB TTIs and TC TTIs) are more protected from direct steam the P values are significantly lower to those measured from the free TTIs. When a high viscosity fluid is used (Figure 2 (c) and (d)) wall thermocouple tends to overpredict the thermal treatment experienced from the fluid and the centre thermocouple. This was expected, as a high viscosity fluid would result in a low convective heat transfer. Steam injection appears to improve the heat transfer performance resulting in an increasing P value recorded by the TTIs.

The results indicate that the TTIs that were allowed to flow have a higher P values when compares to the values obtained from the thermocouple situated in the centre of the vessel (but similar to the thermocouple positioned on the wall of the vessel). The TTIs fitted in balls correlate well with the value measured from the thermocouple positioned in the centre of the vessel.

The overall results of the study indicate that the mixing performance is dependent on the fluid viscosity, the fill level and the heating options. As the free TTIs follow the fluid path they gave a more accurate representation of the real thermal impact on the food product.



Fig. 2 Experiments performed with tomato soup and with a holding temperature of 83°C and experiments performed with 5% gelatinised starch and with a holding temperature of 85°C. Error bars show one standard deviation

This work demonstrates how different types of TTI can be used to study the effects of a process. The results suggest that:

• TTI particles that are free to be close to the steam-heated wall show the highest P value; these are likely to be representative of the processing the fluid has received.

•TTI particles that have constrained to be away from the wall show lower values, indicating the processing received by fluid that is not heated by the wall.

•The process should be designed so that the two sets of results give P values that are as close as possible. The influence of overfilling shows that mixing profiles changes and that greater variation was observed; it is probable that the TTI results seen here show the overprocessed fraction only, and that underprocessing can also occur.

In order to fully characterise the transport phenomena in the vessel velocity profiles and particle paths were obtained. In Figure 3 typical velocity profiles obtained using PEPT are shown. As one can see the velocity obtained for TTIs are very similar to those of the free tracers that represent the velocity of the fluid. Differences are observed across position D where TTIs appear to move downwards faster than the fluid. This was attributed to a sedimentation effect. PIV and PEPT measurements indicate the flow is laminar/transitional the through bulk of vessel [4]. The only regions where significant flow instabilities were generated was at the free surface and at the trailing edge of the impeller. Mixing throughout the bulk was therefore be expected to occur by laminar mechanisms with some mixing by eddy diffusion present at the free surface.



Fig. 3 Comparison of the velocity fields of the free tracer and the TTI tracer for the experiment performed with 4% starch at 33 rpm. Each subfigure show the various positions of the velocity measurement (a) Position A, (b) Position B, (c) Position (C) and (d) Position D [6]

4. Conclusion

As food manufacturers are required to deliver safe high quality products it is essential to develop methodologies that will accurately and reliably evaluate thermal processing. Traditional techniques such as thermocouples have significant limitations as they are not able to represent a typical fluid element. Time Temperature Integrators (TTIs) have been developed and evaluated over the last decades as an alternative method. Despite being a mature technique, aspects of their behaviour during processing such

as flow behaviour has yet to be fully understood. For viscous fluids there is little difference between the flow of the TTIs and of the fluid, so the results are relevant. Specially designed TTIs allow the effect of local wall heating to be assessed – here the need is to ensure that the path of the TTI and the fluid is the same.

This work demonstrates that TTIs can be successfully used as a tool for validation of efficiency and uncertainty of food processes. Their use in a real plant processes requires care. For viscous fluids there is little difference between the flow of the TTIs and of the fluid, so the results are relevant. Specially designed TTIs allow the effect of local wall heating to be assessed – here the need is to ensure that the path of the TTI and the fluid is the same.

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Presented at ICEF11 (May 22-26, 2011 - Athens, Greece) as paper AFT1183.