

Heat Stability Testing of Milk Powders for UHT

PERFORMANCE

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Ingredients by



Dairy for life

Executive Summary

At Fonterra, we recognise the challenges faced by UHT milk manufacturers to effectively check that their milk powder ingredients are fit for purpose. As part of the Research and Development programme for NZMP Whole Milk Powder for UHT applications, a team at Fonterra Research and Development Centre (FRDC) evaluated the effectiveness of three common heat stability tests used by UHT manufacturers to assess the potential of milk powder to foul in UHT sterilisers and found significant shortcomings in all three tests. None of the tests we examined showed an ability to differentiate between good & bad powder performance in the UHT system based on powder performance in the test.



To overcome the shared shortcomings of these tests, the team developed two new test methods. The first – called the Fonterra Heat Exchanger Accelerated Fouling Rate Test – measures fouling rates of reconstituted WMP directly, using a pilot-scale UHT plant. The second – called the UHT Stability Test – is a rapid indirect test which produces results that correlate well with the above mentioned direct method.

This white paper introduces the Fonterra Heat Exchanger Accelerated Fouling Rate Test and the new UHT Stability Test and compares results with those from the three common industry tests. The advantage the UHT Stability Test offers over the common industry tests is its ability to screen out poor performing powders on the basis of the test results, giving greater confidence to powder producers and UHT milk manufacturers alike.



ASSESSING THE STABILITY OF DAIRY INGREDIENTS FOR UHT APPLICATIONS

Milk quality, either raw milk or milk powder, is an integral aspect of UHT manufacturing to ensure the production of consistent and high-quality shelf-stable milks. The quality of dairy ingredients has a large impact on the stability of both the UHT milk process and product. If good quality ingredients are not used this can lead to fouling of UHT sterilisers during manufacture and sedimentation and other defects in the final product. A common challenge faced by UHT milk manufacturers is how to effectively assess the quality of their milk ingredients and ensure they are well suited to, and improve, rather than impede, the processing and shelf-life characteristics of the final milk drink.

The only true and accurate measure of fouling is to process milk through a UHT plant. However, because this is expensive in terms of time and product, UHT milk manufacturers often use a heat stability test to assess the suitability of their dairy ingredients. These tests are based on a principle of assessing the milk's tendency to form aggregates as an indicator of 'instability', either

by applying heat or by some other means to provide an indirect measure of milk stability during industrial scale heating processes, such as UHT or retort. The three most commonly used heat stability test methods across the industry are:

- 1 Heat Coagulation Time (HCT)
- 2 Heat Stability Test
- 3 Alcohol Stability Test

There is a general belief that these tests can screen out milk powders with poor performance on a commercial plant. However most manufacturers acknowledge that they do not reflect true processing conditions – milk samples that show 'good' heat stability as measured by the test can demonstrate variable fouling performance during UHT; samples that show 'poor' heat stability during the test can perform well under UHT conditions. Here we demonstrate that Fonterra Heat Exchanger Accelerated Fouling Rate Test closely mimics the performance of WMP on commercial UHT plants and have used this tool to develop the UHT Stability Test which can be used as an effective grading test to ensure the performance of WMP in UHT applications.

ASSESSING THE EFFECTIVENESS OF COMMERCIAL INDUSTRY HEAT STABILITY TESTS FOR UHT APPLICATIONS

Test	Benefits	Shortcomings
Heat Coagulation Time (McSweeney et al ¹ , 2004)	<p>Multiple samples can be assessed simultaneously</p> <p>Oil bath operates at 140°C, more closely simulating UHT conditions</p> <p>Provides a measure of 'time taken to form insoluble material', which may provide a better indicator than 'volume of insoluble material' (over fixed time) provided by other tests.</p>	<p>Method requires a skilled operator to see the first signs of aggregation, and to operate the oil bath safely</p> <p>Only limited turbulence and shear applied</p> <p>No flow or replenishment of the fouling liquid</p>
Heat Stability Test ² (Fonterra variant)	<p>Simple test</p> <p>Doesn't take a lot of time</p> <p>Mostly operator independent</p> <p>Provides a measure of volume of failed material</p>	<p>Static test</p> <p>No flow, shear or refreshment of fouling liquid</p> <p>Oil-bath temperature is set at 121°C and thus doesn't represent UHT temperatures (~140°C).</p>
Alcohol Stability Test ³	<p>Does not require specialized equipment and training for a simple and rapid procedure</p> <p>Provides immediate results</p> <p>Only suitable for screening out very poor quality fresh milk (not powders)</p>	<p>Designed only for raw milk testing (TetraPak Handbook⁴)</p> <p>Widely criticized in literature as a poor index of the heat stability of milk</p> <p>Generates poorly reproducible results</p> <p>Highly dependent upon operator technique</p> <p>Does not simulate UHT conditions in terms of temperature, shear, flow and pressure conditions.</p>

Table 1. Benefits and shortcomings of three common commercial industry methods to assess milk powder stability

1. Samples are heated in an oil bath in sealed glass tubes at 140°C until the first signs of aggregation or gelation appear.

2. Fonterra's method Q2900-61 was developed in February 2012. Samples are pre-heated at 60°C for 10 mins in closed flasks, heated again at 120°C in stainless steel tubes in an oil bath for 15mins, diluted, centrifuged and the volume of sediment recorded.

3. Modified from Davies and White (1958): A range of predetermined concentrations of alcohol is first prepared. A portion of alcohol is added dropwise to an equal portion of the milk sample to be tested, at ambient temperature, by swirling the petri dish

simultaneously. Sample is observed for signs of coagulation. The milk is deemed to have failed the test if coagulation or grains of precipitation occurs in the mixture. The highest % ethanol concentration which does not coagulate the milk sample is recorded as the "alcohol number". Analysis is halted once the alcohol number is determined.

4. HANDBOOK: The role of raw milk quality in UHT production, Tetra Pak Processing Systems AB

INTRODUCING THE FONTERRA HEAT EXCHANGER ACCELERATED FOULING RATE TEST FOR UHT APPLICATIONS

A practical lab scale test, designed to emulate industrial scale fouling, was developed at FRDC (see Appendix One). This test – called the Fonterra Heat Exchanger Accelerated Fouling Rate Test – was based on the premise that to predict fouling, the reconstituted powders needed to be processed in an appropriate heat

exchanger using flow, shear and temperature conditions that are similar to those at scale in a UHT manufacturing plant. The test is called “accelerated” because more aggressive running conditions were deliberately used to get the heat exchanger to a fouled state in a reasonable time frame. The run length for the test is typically 2 hours compared to typical run lengths of 8-16 hours on commercial plants. For simplicity in this paper the term “fouling rate” is used as shorthand for “accelerated fouling rate”.

Using data from this test, fouling rate can be calculated using the equation:

$$\text{Fouling rate (}^{\circ}\text{C/hr)} = ((T_4(\text{at run finish}) - T_4(\text{at pre-start}))/\text{run length (min)}) * 60 (\text{min/hr})$$

(Where T₄ is the hot water feed temperature to the UHT plant)

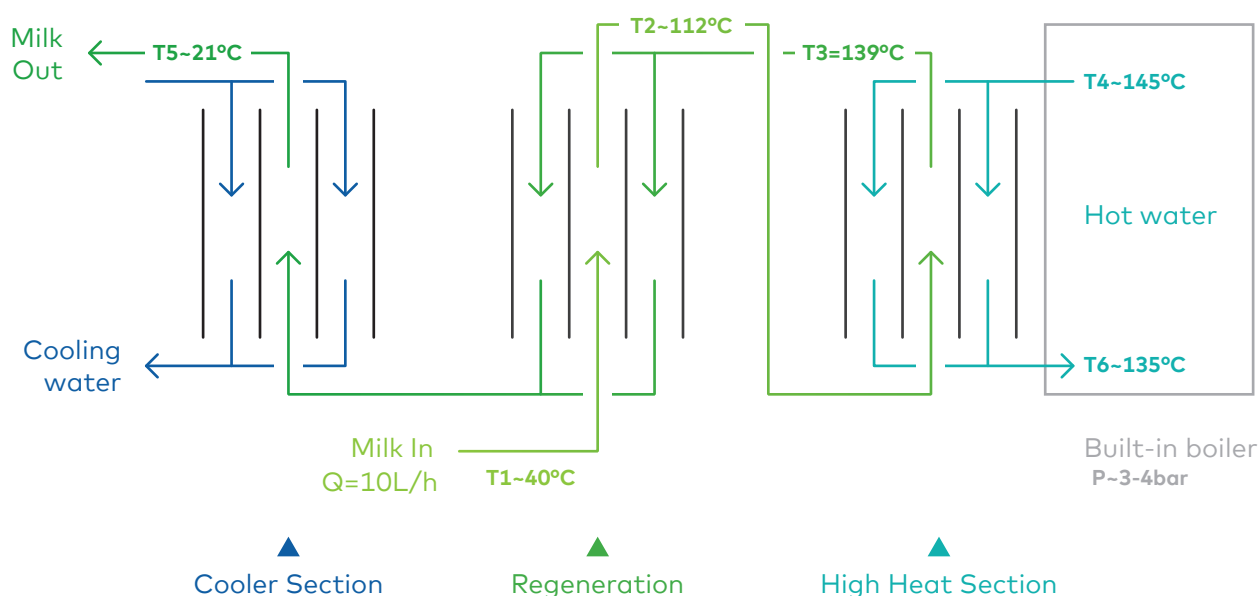


Figure 1: Flow diagram of the heat exchanger system used for the Fonterra Heat Exchanger Accelerated Fouling Test

As the plant fouls, a higher T₄ is required to maintain the same product temperature (at constant flow rates of both product and heating water). Monitoring the increase in T₄ over time therefore gives a measure of the rate at which the plant is fouling.

An excellent relationship was found between the fouling rate on the pilot UHT plant and that in several commercial UHT plants (see figure 2). Customer feedback indicates that powders with an accelerated fouling rate of less than 4°C/h perform well on commercial UHT plants.



Fonterra Heat Exchanger Accelerated Fouling Test vs Fouling on Commercial APV UHT Plant

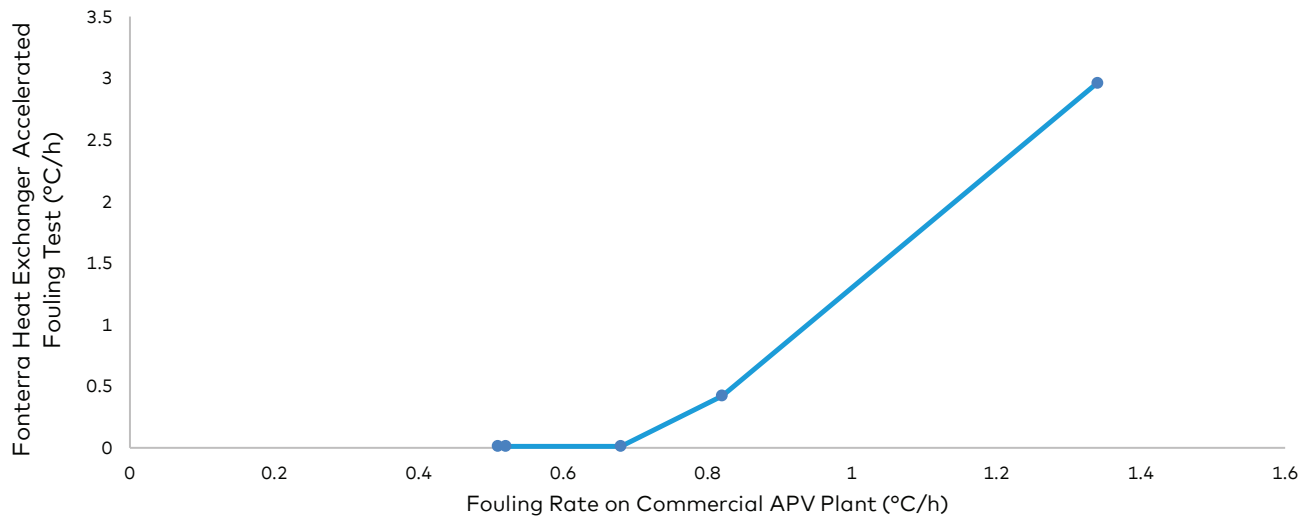


Figure 2: Relationship between the fouling rates of reconstituted whole milk powders measured on a commercial APV UHT plant (commercial beverage formulation) and the Fonterra Heat Exchanger Accelerated Fouling Rate Test (12.5% white milk formulation).

The results of this test for a set of whole milk powder samples were compared with results from three common industry heat stability tests for the same sample set. Details of the test methods investigated are outlined in Table 1. This comparison was conducted using a sample set of 54 commercial whole milk powders, designed to

represent New Zealand milks.

The general conclusion was that these three methods are poor at differentiating most powders with regards to their UHT performance, other than those powders at the extremes.



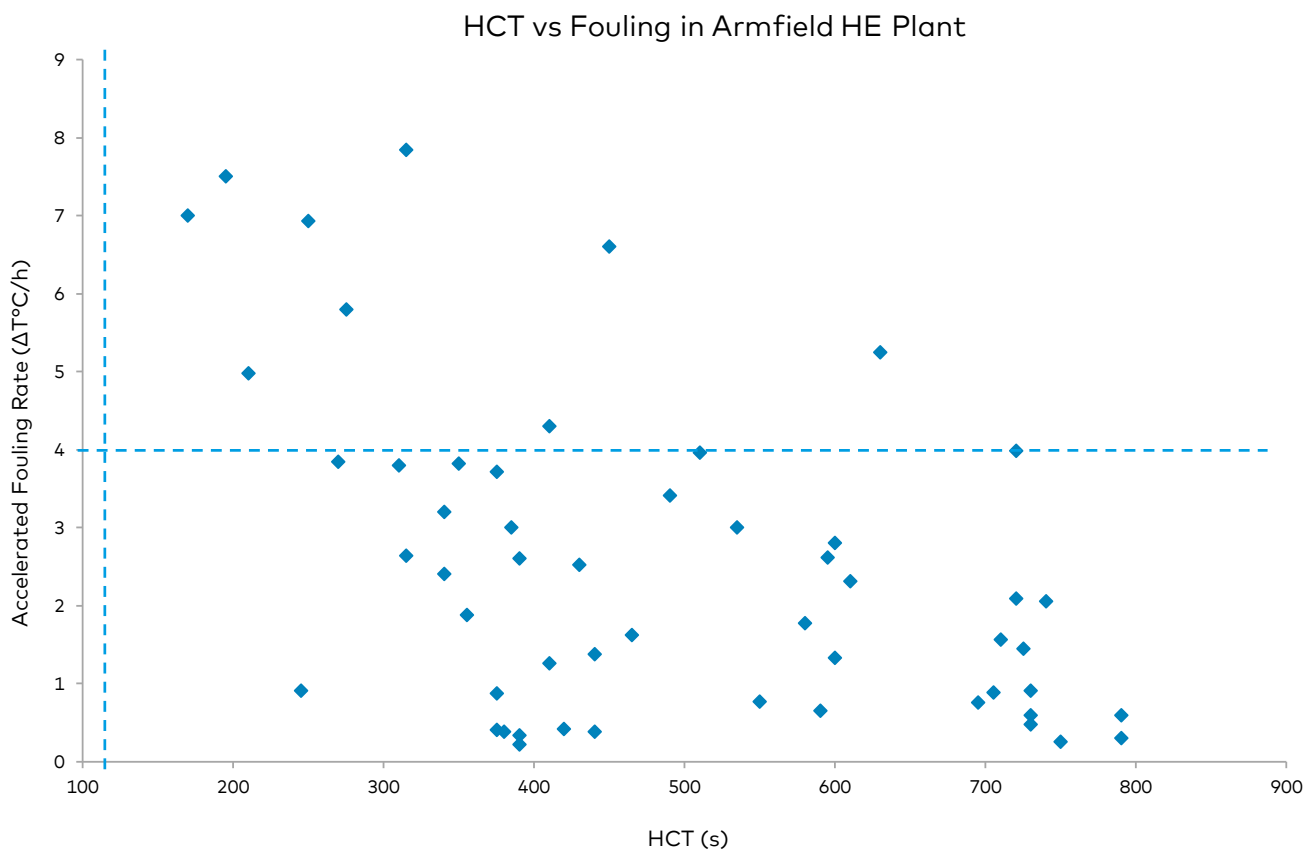


Figure 3: Relationship between the HCT method and the Fonterra Heat Exchanger Accelerated Fouling Rate Test.

A comparison of the whole milk powder results from the McSweeney et al HCT method (Figure 3) with the heat exchanger fouling rates indicated there was only a very weak correlation (linear regression R2 of 0.257). Although this might appear to fit an intuitive hypothesis of 'higher HCT relates to lower fouling'; samples of low fouling (4°C per hour and below) showed a widespread

of HCT results, that ranged from 250-800 seconds. Common practice on HCT would suggest an acceptable minimum of 120 seconds, yet based on results in Figure 3 all samples would pass, despite their fouling performance varying widely from 0-8°C/h. From a practical viewpoint, it would be very difficult to determine an acceptable HCT minimum threshold that an analyst could apply.



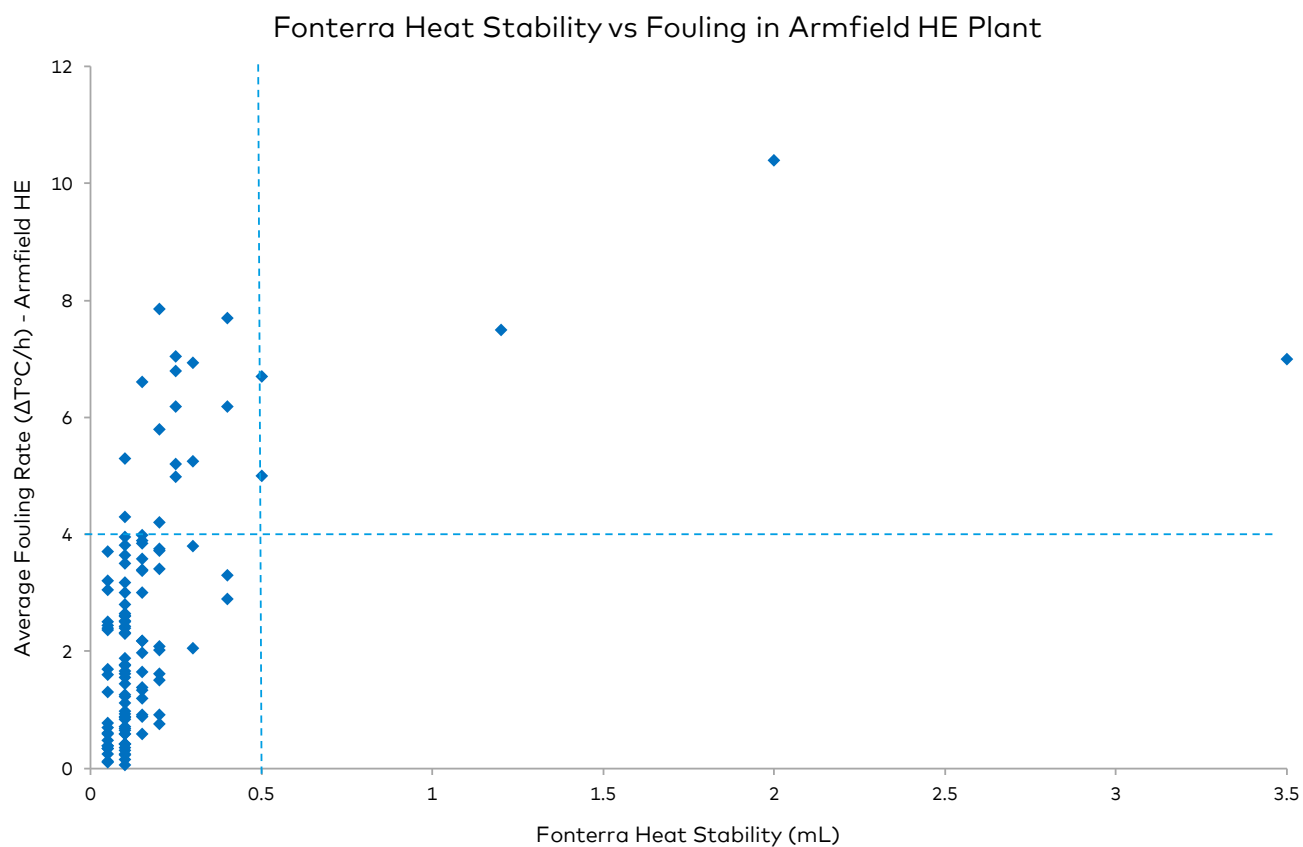


Figure 4: Relationship between Heat Stability test (Fonterra's variant) and the Fonterra Heat Exchanger Accelerated Fouling Rate Test.

In comparing the results from the Heat Stability test (Fonterra variant) with accelerated heat exchanger fouling rate data (Figure 4), there appeared to be a weak correlation. As with the Heat Coagulation test in the section above, this might fit a hypothesis of 'more sediment volume (mL) relates to higher fouling'. The

challenge, however, is in applying a practical rejection limit. If this limit was set at >0.5 mL, only 3% of powders tested would be rejected, according to the results in Figure 4. However, 15% of powders that passed the Heat Stability test grading (≤ 0.5 mL) fouled at a level of $>4^{\circ}\text{C/h}$, which would cause concern on a commercial UHT plant.



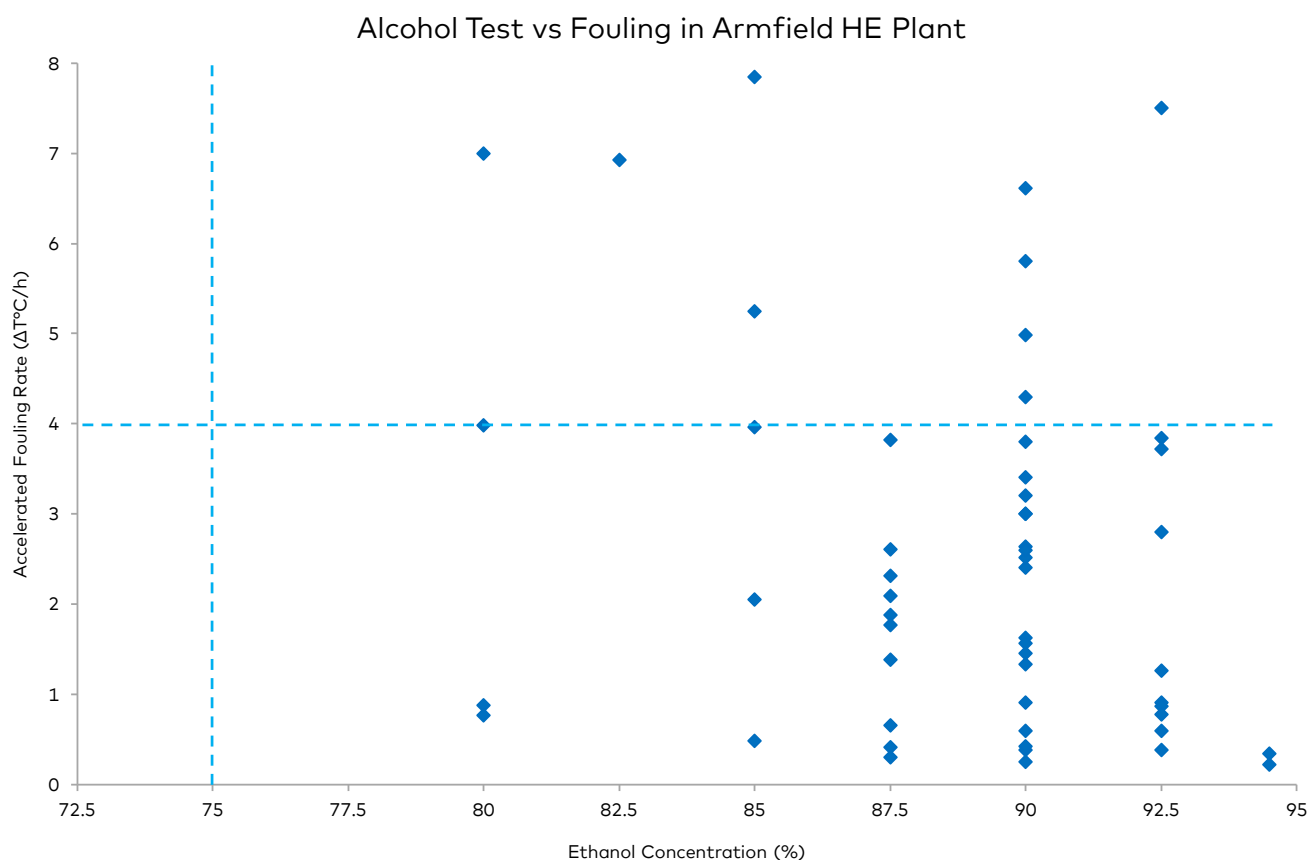


Figure 5: Relationship between the Alcohol Stability Test and the Fonterra Heat Exchanger Accelerated Fouling Rate Test.

There was no clear correlation between results from the Alcohol Stability Test and the Fonterra Heat Exchanger Accelerated Fouling Rate (Figure 5), nor any indication that an acceptance limit could be applied. Bylund (2003) reported that any sample with an alcohol number above 75% ethanol concentration was "protein stable". Yet based on this number, all samples tested in this study would pass. As with the HCT results, many would foul unacceptably, even at the higher levels of alcohol stability. We also observed that the alcohol test was inherently subjective, with a high degree of operator dependence and low reproducibility. For example, at a given concentration of alcohol, samples could either pass or fail the test based on differences in technique of alcohol addition to the milk. A dropwise addition, which was the

technique we applied, gave a different result from adding all the alcohol in one dose.

The objective of indirect benchtop tests is to give UHT milk manufacturers confidence that milk powders will perform well on a UHT plant, with minimal errors in sample acceptance or rejection. However, our findings reveal that common commercial industry tests introduce confusion to decision-making on powder acceptability. The results give weak correlations with fouling rates, and unacceptably high rejection rates based on arbitrary limits. While it has some limitations, the Fonterra Heat Exchanger Accelerated Fouling Rate test was found to be a direct, reliable, robust way of measuring the physical process of fouling that applies at commercial scale.



THE UHT STABILITY TEST – A BETTER FOULING DETECTOR

The capital outlay required for the Fonterra Heat Exchanger Accelerated Fouling Test and the turnaround time between testing each sample on the rig limits the number of samples processed per day. This leads to a situation where the cost per test for the Fonterra Heat Exchanger Accelerated Fouling Test is prohibitive to its use in grading commercial quantities of WMP.

The inconvenience and expense of the Fonterra Heat Exchanger Accelerated Fouling Rate Test and the unreliability of existing tests necessitated the development of a new test method for confirming suitability of WMP for UHT applications. The requirements of the new test are that it correlates with fouling rates, is able to differentiate between good & poor performing powders and better lends itself to use in grading commercial WMP. The result was the UHT Stability Test for WMP which measures the sediment

formed when a WMP sample is reconstituted in a hot acid environment. The conditions for the UHT stability test were chosen for the following reasons:

- It was hypothesised that the tendency of a whole milk powder to resist sedimentation in hot, low pH conditions would give an indication of the level of resistance of the powder to some of the major mechanisms of fouling during UHT processing.
- The test is an adaptation of an existing Fonterra grading test for coffee sediment in iWMP. Preliminary tests indicated a potential correlation of coffee sediment with fouling rate. Based on the above hypothesis the test was adapted to replace the coffee component of the test with HCl.

The UHT stability test was validated using a large set of whole milk powders (total of 135 whole milk powders investigated) and the results showed that powders with low fouling behaviour ($<4^{\circ}\text{C/h}$), were highly correlated with good performance ($<1.5\text{mL}$ sediment) in the new test.



UHT Stability Test vs Fonterra Heat Exchanger Accelerated Fouling Test

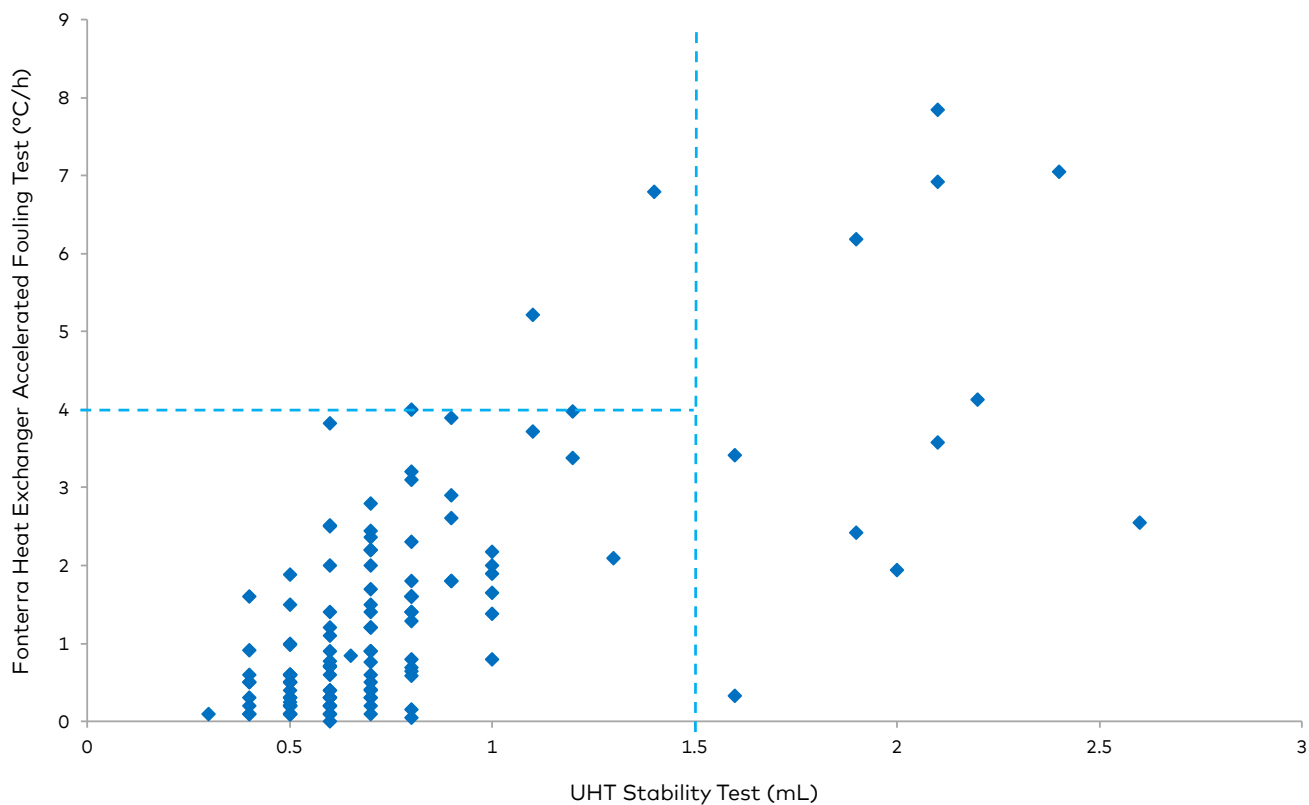


Figure 6: Relationship between the new UHT Stability Test and the Fonterra Heat Exchanger Fouling Rate Test.

Figure 6 illustrates the good correlation between the results of the UHT Stability Test and the Fonterra Heat Exchanger Fouling Rate Test and its effectiveness as a screening tool for high fouling powders when an acceptance limit of 1.5mL is applied.

Comparisons were made between the four indirect tests presented in this paper with respect to their efficacy

in screening out high fouling WMPs. The proportion of samples that pass the test but which produce an unacceptably high ($>4^{\circ}\text{C/h}$) fouling rate ("false positive") was compared to the proportion of samples that fail the test but produce an acceptable low ($<4^{\circ}\text{C/h}$) fouling rate ("false negative") for each test. The data for each test is given in Table 2 below:





Test	Heat Coagulation Time test	Heat Stability test	Alcohol Stability test	UHT Stability test
Limit	120s	0.5mL	75%EtOH	1.5mL
False Positive Pass (%) (passed high fouling rate powders)	17%	15%	17%	1.5%
False Negative Pass (%) (rejected low fouling rate powders)	0%	0%	0%	4.5%

Table 2. Proportion of "false positives" and "false negatives" with respect to fouling performance for four powder test methods (Heat Coagulation Time; Heat Stability test – Fonterra variant; Alcohol Stability; UHT Stability test).

The data in Table 2 demonstrates that the UHT Stability test is significantly (10 times) more accurate than the other tests in screening out high fouling powders, with only 1.5% of powders classified as false positives and 4.5% of the powders classified as false negatives.

We expect that optimised control of manufacturing conditions will reduce even further the incidence of false passes and the overall incidence of failed product (both false and correct fails).

Conclusion

The Fonterra Heat Exchanger Accelerated Fouling Rate test has been shown to be the best indicator of fouling behaviour of powders in UHT systems. However, this is impractical to implement commercially, so would be unsuitable for most UHT manufacturers. The three heat stability tests that were evaluated do not show any reliable correlation with the fouling performance of whole milk powders in a UHT system. In fact, the HCT and Alcohol stability tests passed 100% of the powders tested, regardless of their fouling performance. We cannot recommend any of these tests for predicting whole milk powder performance in a UHT plant. The UHT Stability Test for WMP does show a comparatively high

correlation with measured fouling behaviour. Heat stability tests are only indicators of UHT performance. The UHT Stability Test for WMP provides a point of final quality control, mitigating the variability of the raw material and processing controls. It does not readily apply to other WMP specifications, and for this reason, the method only provides accurate prediction of UHT performance when the WMP is designed for UHT applications and is specified in the product bulletin.

For more information on NZMP UHT Grade ingredients, contact your NZMP Account Manager or visit www.nzmp.com.



APPENDIX ONE: FONTERRA HEAT EXCHANGER ACCELERATED FOULING RATE METHOD

Apparatus:

The heat exchange equipment was the FT74XTS Armfield Heat Exchange model from the Armfield Corporation UK. Various modifications were made to the pump and sampling systems, to ensure consistent, robust performance, including:

- Replacing the single heat exchanger with three separate heat exchangers from SPX, as the plate seals in the original did not withstand dismantling for plate inspection
- Replacing the original pump with one that gave higher flow and turbulence CIP, increasing its effectiveness and flow during cleaning steps
- Introducing probes to monitor temperature inlet and outlet from each heat exchanger.
- Replacing the 5L hopper with a separate tank of 12 L capacity.

Limitations:

- Speed: the method is not fast enough to be a grading test at the point of powder manufacture, as typically only 2-3 runs per day can be achieved. A duplex system would reduce CIP time between runs and result in 4-6 runs per day;
- Scope: it is validated for a 12.5% w/v total solids WMP milk formulation; data on other formulations would need to be gathered, including repeatability etc. We note also that the measured fouling rate is sensitive to total solids concentration;
- Sensitivity: the ability of the fouling method to differentiate between a range of low fouling samples is modest at best. For example, a reconstituted milk powder that allowed a plant to run for 10-12 hours before cleaning would not be distinguished from one that ran for 15hrs.
- Cleaning: CIP is an important part of the procedure. It needs to be done thoroughly and it takes time.
- Expertise: fouling data requires some skill in interpretation, typically by a technologist or engineer, as there is a minimum level of computing involved.

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