Quantifying Tailing in Liquid Confectionery Fillings using Texture Analysis and Extensional Viscosity

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ABSTRACT
A general method to quantitatively evaluate the degree of tailing in a liquid confectionery filling is proposed. The method centres on texture analysis, whereby an adhesion type test is performed. The results describe the formation and subsequent breakage of a filament and the degree of tailing can be evaluated by calculation of the angle between baseline and peak height. The angle is taken as that between the baseline and points drawn on the curve between 50%, and 75% of the maximum height expressed as time in seconds. The lower the angle the lesser the degree of tailing. The method is easy to use and has well defined limits, an angle of 90° represents the perfect maximum, i.e. no tailing; and 180° represents the idealised minimum, i.e. complete tailing. The results are backed up and corroborated by extensional viscosity measurements of filament stretching and subsequent breakage.

INTRODUCTION
Tailing; the formation of a filament when filling, e.g. chocolate cups by means of depositing liquid fillings, is a major problem for the confectionery industry. Tailing is caused by a number of issues; depositing at too high viscosity, at too cold a liquor temperature, at too high soluble solids, at too high a hydrocolloid concentration, or at the wrong pH1. Ideally, one wants no tailing, such that when one chocolate cup is filled and the depositor stopped, the liquid filling similarly stops, and should not form filaments. The degree of tailing can be reduced by introducing suck-back of the depositing nozzles, or by using a lifting depositor mechanism1. However, formation of these filaments causes production down time, unattractive finished products and leads to rework.

Production of liquid fillings, for this market, needs to balance the textural requirements of the filling with the ability to reduce the degree of tailing. Until now this property has been limited to subjective evaluation based on operator opinion and could not be satisfactorily quantified.

The method proposed here aims to address this issue with a simple texture analysis test that can easily evaluate the degree of tailing for a given sample. The method is based on texture analysis, is easy to use and the results adequately describe a sample’s relative degree of tailing. The method is flexible and can be adapted to mimic production conditions reflecting different deposition rates. The effect of the filament stretching is similarly examined by means of extensional rheometry and compared to the trends observed from texture analysis.

MATERIALS AND METHODS
Four liquid fillings were tested, with the following hydrocolloids; GRINDSTED®
Filling 430, GRINDSTED® Filling 420, GRINDSTED® Pectin CF 251B and GRINDSTED® Pectin LA 110. These represent combinations of pectin / xanthan and pectin / LBG, low ester conventional pectin and low ester amidated pectin respectively.

The texture experiments were carried out on a Stable Micro Systems Texture Analyser TAXT-Plus equipped with EXPONENT™ software, and located at Coloplast A/S, Humlebæk. The test used was an adhesion type test, with a 40mm plate. The adhesion test was chosen because it had previously been demonstrated by Coloplast A/S to give information regarding the tailing of skin type adhesives. Therefore, it seemed suitable for use in this application.

The plate was brought into contact with the sample and depressed to a depth of 10 mm and left to equilibrate for 10 seconds before the probe being drawn away at an arbitrary speed of 5mm / s. Simultaneously to the measurement being performed, a video film was recorded. From this film it was possible to see the filament formation and subsequent filament breakdown. With software control it was possible to synchronise the film with the texture analysis data, and thereby physically see which portion of the curve described the phenomenon of tailing.

The extensional viscosity measurements were made using a CaBER 1 Extensional Rheometer instrument from Haake, courtesy of the University of Lund’s Chemistry Department and JK Lab, Sweden. The sample was loaded between the plates, such that the sample diameter was 6.0mm, and the sample height was 3.0 mm. The plates were then drawn apart such that the Hencky strain was 1.25 and the final distance between the plates was 10.5mm.

RESULTS AND DISCUSSION

Figure 1 shows a schematic diagram giving the general shape of the texture analysis curve, the three main areas of the curve and the tangents required to calculate the angle of degree of tailing. The first section of the curve, phase I, is where the probe is brought into contact with the sample, depressed to a depth of 10mm and left to equilibrate for 10 seconds. The second phase, II, is the actual tailing portion of the curve, showing the angle of tailing and the third phase, III is the final baseline of the curve. The degree of tailing can be obtained from the angle between the downslope and the final baseline, as seen in phase II.

![Figure 1 Schematic diagram of the curve obtained from the texture analyser after running an adhesion type test.](image)

It can be envisaged that if the angle is 90°, then there has been fast immediate breakdown of the filament, in effect no filament was formed, and hence no tailing. As the angle becomes more and more obtuse, the breakdown of the filament is slower and more gradual, indicating the trend towards considerable filament stretching and therefore tailing. The upper value is limited to 180°, and in practise will never exist.

An arbitrary post-test speed of 5mm per second was chosen and the curves for all samples are given in Figure 2. The curves show the initial contact of the probe with the
Figure 2 Adhesion type tests for liquid fillings; GRINDSTED® Filling 430, GRINDSTED® Filling 420, GRINDSTED® Pectin CF 251B and GRINDSTED® Pectin LA 110 conducted at a post-test speed of 5mm per second.

The sample, seen as a negative on the graph, decreasing to a minimum after 5 seconds.

The probe is then left in contact with the sample for 10 seconds and the curve can be seen to level out over this equilibration period, corresponding to phase I in Figure 1. 15 seconds after the test is started the probe is withdrawn from the sample at a speed of 5 mm per second. This is the portion of the curves that are interesting and can be used to evaluate the degree of tailing, as phase II from Figure 1.

The curves continue to give the final baseline as seen in phase III.

Pictorial evidence in the form of stills from the video films is shown in Figures 3. They are taken at the point, which best shows the indication of filament formation. Long filaments can clearly be seen for GRINDSTED® Filling 430 and GRINDSTED® Filling 420, hardly any filament formation for GRINDSTED® Pectin CF 251B, and obvious filament formation for GRINDSTED® Pectin LA 110, although considerably less either of the GRINDSTED® Filling products.

The main problem is to establish a method whereby the angle can be calculated by means of objective methods.

Using time as the axis of reference and taking GRINDSTED® Filling 430, (Danisco 430), in Figure 2 as an example the protocol for evaluating the degree of tailing is achieved as follows.

The onset of final baseline of Danisco 430 begins after some 33 seconds. The maximum height of the same curve occurs after some 18.5 seconds. To set the fixed points of the second tangent the difference between the baseline and maximum is used and divided by 4. Hence we have (33-18.5) / 4 = 3.625. The second tangent is then fixed by marking points on the curve representing 50% and 75% of the time between maximum peak and baseline onset. This results in a tangent that is as close to the apex of the curve as possible and can best describe the angle in question.

Figure 3 Still images from the video films shot simultaneously with the texture analysis measurements for the four confectionery liquid fillings; (a) – GRINDSTED® Filling 430, (b) – GRINDSTED® Filling 420, (c) – GRINDSTED® Pectin CF 251B, (d) – GRINDSTED® Pectin LA 110
The pictures shown in Figure 2 are in good agreement with the trends reported in the filament stretching work of Kolte\textsuperscript{2} and Kolte and Szabo\textsuperscript{3}, which showed the extension of a filament before subsequent breakage.

**PRODUCT IMPLICATIONS**

The results in Table 1 are gained from the protocol set out above and clearly show differences in the angle reported. An increasing angle indicates an increase in the degree of tailing. GRINDSTED\textsuperscript{®} Filling 430, a combination of xanthan and pectin shows the highest degree of tailing, which is largely consistent with the well known, characteristically long texture of xanthan. GRINDSTED\textsuperscript{®} Filling 420 substitutes xanthan with LBG, compared to GRINDSTED\textsuperscript{®} Filling 430 and this results in a lower degree of tailing, but it is arguable if this difference is significant. The results indicate that these two products can essentially be thought of as the same.

Considerable differences are seen when only pectin is used. Pectin is noted for its short texture and this can be seen well with GRINDSTED\textsuperscript{®} Pectin CF251 B, which has a degree of tailing value of 109°, only some 19° from the ideal minimum of 90°. Showing less degree of tailing than either GRINDSTED\textsuperscript{®} Filling 430 or GRINDSTED\textsuperscript{®} Pectin LA 110 still displays a considerable degree of tailing of, 143°. The difference between the two pure pectin types is that GRINDSTED\textsuperscript{®} Pectin LA 110 is low ester amidated in nature compared to the low ester conventional GRINDSTED\textsuperscript{®} Pectin CF 251 B. Low ester pectin is noted for forming flexible systems, and this might explain why the low ester amidated pectin, which has a much lower DE% than GRINDSTED\textsuperscript{®} Pectin CF 251 B has as a greater tendency for tailing than its higher low ester counterpart.

**EXTENSIONAL VISCOSITY**

Studies of the filling samples using the Haake CaBER extensional rheometer, which, in principle, runs as the liquid filament rheometer\textsuperscript{4} were performed to confirm the filament breakdown and also measure the extensional viscosity. The filament diameter, which records the lifetime of the filament until breakage, is measured according to the principles outlined by Bach et al, using laser analysis\textsuperscript{5}, and is also described on the ThermoHaake website for the CaBER\textsuperscript{6}. Results for the sample GRIDNSTED\textsuperscript{®} Pectin LA 110 were not incorporated here due to difficulties with measurement. The results for filament breakdown are presented in Figure 3.

<table>
<thead>
<tr>
<th>Filling Type</th>
<th>Baseline Onset</th>
<th>Max. Height</th>
<th>Delta 50</th>
<th>Delta 75</th>
<th>Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>GRINDSTED\textsuperscript{®} Filling 430</td>
<td>33s</td>
<td>18.5s</td>
<td>25.75s</td>
<td>29.37s</td>
<td>165°</td>
</tr>
<tr>
<td>GRINDSTED\textsuperscript{®} Filling 420</td>
<td>24s</td>
<td>17s</td>
<td>20.5s</td>
<td>22.25s</td>
<td>161°</td>
</tr>
<tr>
<td>GRINDSTED\textsuperscript{®} Pectin CF 251B</td>
<td>20.5s</td>
<td>17.55s</td>
<td>18.87s</td>
<td>19.68s</td>
<td>109°</td>
</tr>
<tr>
<td>GRINDSTED\textsuperscript{®} Pectin LA 110</td>
<td>22s</td>
<td>16.8s</td>
<td>19.40s</td>
<td>20.70s</td>
<td>143°</td>
</tr>
</tbody>
</table>

Table 1 Gives baseline onset, maximum height and delta 50 and 75 together with the angle used to describe tailing.
The results from Figure 3 shows that the filament for GRINDSTED® Pectin CF 251B broke quicker than those filaments for GRINDSTED® Filling 420 and 430, with a breakage time of 11, 35 and 73 seconds respectively. This is the same trend as shown for results expressed from texture analysis with regards to the angle measured. The results confirm here that sample with a high angle similarly take longer time to break. There was a suspicion that samples that had either a high angle or a long filament lifetime, i.e. a high degree of tailing would also have appreciable higher extensional viscosities. This hypothesis was also tested. Due to the nature of the measurement the extensional strain rates measured were sample dependent. However, the results given in Figure 4 provide clear enough indication that increasing the degree of tailing increases the extensional viscosity.

Figure 4 clearly demonstrates that the filling samples with the higher degree of tailing possess the greater extensional viscosity values, that for GRINDSTED® Filling 430 being some 1.5 orders of magnitude higher for the corresponding strain rate of $0.1s^{-1}$ compared to GRINDSTED® Pectin CF 251B. Although not shown, this is suggests, based on earlier extensional work, that the Trouton ratios will likely also be considerably different, and values for GRINDSTED® Filling 430 probably approaching 100.

Figure 3 Shows the distance versus time graph for the filling samples.

CONCLUSION

The degree of tailing can quickly and easily be quantified by using the texture analysis methodology. The method is simple and can be confirmed by means of more complex extensional rheometry, where not only filament breakdown but extensional viscosity itself were able to be measured. This further allows characterisation of Trouton ratios, should such information be necessary. However, for the simpler quality
control type measurements the results from the texture analyser are more than adequate. While applied here only to confectionery liquid fillings, it is quite feasible that the methods can be extended to other food and non-food systems alike where tailing can be an issue, e.g. bakery fillings, ripples, sauces, salad dressings, printing inks, paints and coatings.

ACKNOWLEDGEMENTS
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REFERENCES
6. www.ThermoHaake.com