APPARENT VISCOSITY OF FORMULATIONS OF INULIN CHICORY EXTRACT  
(Cichorium intybus L ) WITH MODIFIED AND HYDROLYZED STARCHES

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João Alexandre Bortoloti ⁴

ABSTRACT

Rheological properties of chicory root extracts were determined at 25°C, 40°C and 55°C. Samples consisted of pure concentrated extract and concentrated extract formulated with hydrolyzed and modified starch. All suspensions exhibited a pseudoplastic behavior, with an index value (n) varying from 0.42 to 0.97 in the shear rate range of 14 to 264 s⁻¹. Statistical models indicate that the apparent viscosity of suspensions, formulated with the two different starches, are affected principally by the proportion of hydrolyzed starch, the proportion of modified starch and the fraction of the two starches in the total mixture.

Keywords: fat replacement, food ingredient, rheology, chicory extract

VISCONSIDADE APARENTE DE EXTRATOS DE INULINA DE CHICÓRIA (Intybus de cichorium L.) FORMULADOS COM AMIDOS MODIFICADO E HIDROLIZADO

RESUMO

As propriedades reológicas dos extratos de raízes de Chicória foram determinadas a 25°C, 40°C e 55°C. As amostras consistiram de extrato puro concentrado e extrato concentrado formulado com o amido hidrolisado e modificado. Toda a suspensão apresentou um comportamento pseudoplastico, com um valor de índice reológico (n) variando de 0,42 a 0,97 na faixa de deformação de 14 a 264 s⁻¹. Modelos estatísticos indicam que as viscosidades aparentes das suspensões formulados, com dois diferentes amidos, são afetadas principalmente pela proporção de amido hidrolisado, amido modificado e fração dos amidos na mistura total.

Palavras-chave: substituto de gordura, ingrediente alimentício, reologia, extrato de Chicória

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INTRODUCTION

Chicorium intibus is a plant of great economic potential due to high concentrations of fructoligossacharide, known as inulin, in its roots, which is used as a replacement ingredient for sugar and fat. Inulin is a polysaccharide consisting of a chain of fructose units with a terminal glucose unit. Vegetables such as asparagus, onion, garlic, dahlia and chicory are the predominant source for commercial inulin, (Silva 1996). Commercially, inulin is extracted from chicory root. The process involves extraction by infusion in hot water, evaporation of excess water, and spray drying of the juice to obtain inulin powder, commercially named as raftiline, (Teeuwen and others 1992). It is also possible to obtain other selected products from partial enzymatic hydrolysis, commercially explored as raftilose. These products are resistant to digestion in the upper gastrointestinal tract, but readily fermented in the colon, where they serve as selective growth substrates for bifidobacteria. This process results in a very important physiological property for inulin causing nutritional benefits similar to those associated with a high fiber diet with stimulation of bifid bacteria growth decreasing, for example, blood cholesterol levels according to Roberfroid and others (1993) and Silva (1996). Inulin is particularly suitable for fat replacement in low fat or fat free products, such as chocolate, confectionery, cheese and ice cream dressing, because it has a fat creamy form, gelling capacity and good body, texture and mouthfeel. Raftiline and raftilose are used as dietary fiber, having the additional advantages of being able to replace fat and sugar in foods, (Hewitt 1994; Cândido and Campos 1995).

The rheological behavior of fluid foods is essential for designing and evaluating food processing equipment and it is an important parameter for quality control for consumer acceptability. Regarding engineering calculations, knowledge of applicable flow models is important for designing flow systems, (Rao and Anantheswaran 1982).

It was observed that some biological fluids such as suspensions, proteins or polysaccharides do not obey the Newtonian law of viscosity. Vegetable juices and purees containing great masses of insoluble particulate material, such as pectin, in suspension are highly non-Newtonian due to high macromolecular concentrations that directly affect their rheological behaviors. Rao and others (1974) presented examples of non-Newtonian behavior with pseudoplastic characteristics related to banana, guava and papaya purees. The effect of temperature and concentration on apparent viscosity must be known to understand unitary operations such as heat and mass transfer of fluid foods, (Vitali and Rao (1982); Rao and others (1984)). Hassan and Hobani (1998) studied flow properties of Hibiscus sabdariffa L concluding that the power law provided a good fit for pseudoplastic flow behavior. The rheological behavior of chicory extract was analyzed based on that class of material.

The objective of this work is to study the rheological behavior of mixtures of chicory extract containing two types of starches and to simultaneously analyze for the effects of process and mixture variables on the apparent viscosity using statistical models.

MATERIALS AND METHODS

Material

The chicory was harvested, the roots separated and washed before slicing, and placed in an autoclave for extraction at 120°C for 20 minutes. The ratio of extraction was one part of sliced roots to two parts of distilled water according to Figueira (2000). After that time period solid and liquid phases were separated from each other by filtration, and concentrated in a pilot vacuum evaporator [PRECISION PS CIENTIFIC GCA CORPORATION] at conditions of 20inHg and 55°C, until 22°Brix. The concentrated extracts were put into clean PET bottles and stored in a freezer to prevent deterioration prior to the viscometric measurements.

Starches

Commercial ingredients such as modified starch (Dextrina 17) and hydrolyzed starch (Loremalt 2002) supplied by the Lorenz Company in Brazil were used.

Experimental design

Standard process variable and mixture models (Barros Neto and others, 1996) were multiplied to obtain combined process-mixture variable models (Cornell,1988). The proportions of hydrolyzed starch, x1, and...
modified starch \( x_2 \), the mixture variables, were studied at different total percentage starch levels and different temperatures, the process variables. Experimental designs were executed at different proportions of modified-hydrolyzed starch (1:0), ( \( \frac{1}{2} : \frac{1}{2} \)) and (0.1) permitting determination of linear and quadratic mixture models. Three temperature levels, 25°C, 40°C and 55°C and six total starch percentage levels were investigated for the process variables. Apparent viscosity measurements were made at all possible combinations of levels of the process and mixture variables.

**Preparation of formulated suspensions**

The extract was thawed and weighed in beakers on a semianalytical balance [MARTE model AS2000] to form 20.00g aliquots. To this extract, modified, hydrolyzed or a 50%:50% mixture of these starches were slowly added until complete dispersion in quantities necessary to make up 0%, 5%, 10%, 15%, 20%, 25% and 30% of the total mixture. This suspension was transferred to the viscometer for which readings were taken after the temperature was stabilized at 25°C, 40°C and 55°C.

**Viscometric measurements**

All measurements reported here were taken with a single spindle Brookfield type viscometer model LV, sensor system SC-18 and SC-34, covering all ranges of combinations and temperature conditions. The SC-18 sensor was used for suspensions containing 5%, 10% and 15% starch at 25°C and 5%, 10%, 15% and 20% of starch at 40°C and 55°C. The SC-34 sensor was used to measure suspensions containing 20%, 25% and 30% of starch at 25°C and 25% and 30% at 40°C and 55°C. The use of both SC-18 and SC-34 sensors for the measuring system was necessary so that readings within the security intervals could be made at all the different mixture and temperature conditions. The viscometer was connected to a thermostatic bath circulator to keep the sample at constant temperature. The data acquisition system registered shear stress and apparent viscosity at temperatures of 25°C, 40°C and 55°C. A rotating spindle velocity within 50 to 200 rpm was used to obtain shear rates ranging from 14 to 264 s\(^{-1}\).

**Data processing**

The choice of rheological model is based on the fluid characteristics of the material under study. Empirical models, showing the relationships between shear stress and shear rate, provide descriptions of rheological behavior.

\[
\tau = K \cdot \dot{\gamma}^n
\]

(1)

Since, \( \eta = \frac{\tau}{\dot{\gamma}} \), equation (1) can be rewritten as (Morris 1995):

\[
\eta_{\text{app}} = K \cdot \dot{\gamma}^{(n-1)}
\]

(2)

Lapasin and Pricl (1995) state that the transport properties and specially the rheological behavior of complex and real materials with polysaccharide systems can be significantly affected by different factors such as solvent medium, concentration and temperature. For homogeneous systems viscosity is a monotonically decreasing function of temperature.

The relative errors were calculated according to equation 3, showing the differences between predicted values obtained from the model and experimental values of shear stress and apparent viscosity. According to Lomauro and others (1985) the errors are considered very acceptable for values of \( ERM \) below 5%.

\[
ERM = 100 \frac{\sum \text{VE} - \text{VP}}{\text{VE}}
\]

(3)

**Statistical mixture-process variable models**

Multiple linear regression of apparent viscosity on the various levels of the mixture variables (proportions of hydrolyzed and modified starches, \( x_1 \) and \( x_2 \)) and on the process variables (% total starch and temperature, \( z_1 \) and \( z_2 \) scaled values) were carried out to obtain a model capable of predicting apparent viscosity as a function of these variables. Since replicate experiments were not plausible to perform, approximate estimates of errors in the regression coefficient models were obtained from cumulative probability plots of weighted values of the model’s coefficients. A second estimate was obtained from regression residuals for a model not showing evidence of lack of fit.

**RESULTS AND DISCUSSION**

The apparent viscosity curves (\( \eta_{\text{app}} \)) showed similar behaviors at all three temperatures and
all total starch percentages. Figure 1 shows the typical behavior where the apparent viscosity decreases with increasing of shear rate ($\dot{\gamma}$). Such a decrease is more significant for the pure extract, showing a non-Newtonian flow behavior with pseudoplastic characteristics, as is indicated by the flow index, $n$, situated in the 0.42 to 0.65 range, for 25°C, 40°C and 55°C, as shown in Table 1.

![Figure 1 - Typical behavior of apparent viscosity curve for suspension from chicory extract with and/or hydrolyzed starches](image)

**Table 1 - Consistency and flow behavior index**

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>% Starch</th>
<th>Hydrolyzed</th>
<th>Modified</th>
<th>Hydrolyzed + Modified</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$K$ (Pa s$^n$)</td>
<td>$n$</td>
<td>$R^2$</td>
</tr>
<tr>
<td>25</td>
<td>0</td>
<td>0.48 0.65 1.00 1.93</td>
<td>0.48 0.65 1.00 1.93</td>
<td>0.48 0.65 1.00 1.93</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.15 0.95 0.99 0.71</td>
<td>0.15 0.94 0.99 0.44</td>
<td>0.19 0.84 0.99 1.50</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.19 0.93 0.99 0.83</td>
<td>0.17 0.95 0.99 0.42</td>
<td>0.16 0.97 0.99 1.41</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>0.27 0.95 0.99 0.71</td>
<td>0.20 0.96 0.99 0.46</td>
<td>0.33 0.90 0.99 2.49</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.55 0.82 0.99 2.13</td>
<td>0.40 0.88 0.99 1.29</td>
<td>0.63 0.90 0.99 1.44</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>0.94 0.92 0.99 1.39</td>
<td>0.62 0.84 0.99 0.70</td>
<td>0.75 0.86 0.99 0.84</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>1.11 0.92 0.99 0.43</td>
<td>0.78 0.92 0.99 1.02</td>
<td>1.02 0.91 0.99 0.99</td>
</tr>
<tr>
<td>40</td>
<td>0</td>
<td>1.04 0.36 0.97 4.11</td>
<td>1.06 0.42 0.97 4.11</td>
<td>1.06 0.42 0.97 4.11</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.09 0.96 0.99 0.54</td>
<td>0.15 0.91 0.99 1.59</td>
<td>0.26 0.75 0.99 2.20</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.11 0.94 0.99 0.96</td>
<td>0.15 0.96 0.99 0.33</td>
<td>0.23 0.84 0.99 1.15</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>0.16 0.95 0.99 1.03</td>
<td>0.17 0.96 0.99 0.29</td>
<td>0.25 0.94 0.99 0.61</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.22 0.96 0.99 0.33</td>
<td>0.22 0.97 0.99 0.38</td>
<td>0.30 0.93 0.99 0.67</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>0.44 0.87 0.99 0.91</td>
<td>0.34 0.93 0.99 1.84</td>
<td>0.49 0.92 0.99 1.27</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>1.09 0.81 0.99 0.96</td>
<td>0.73 0.83 0.99 1.14</td>
<td>0.84 0.86 0.99 1.45</td>
</tr>
<tr>
<td>55</td>
<td>0</td>
<td>0.55 0.46 0.95 6.49</td>
<td>0.55 0.46 0.95 6.49</td>
<td>0.55 0.46 0.95 6.49</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.06 0.93 0.99 0.83</td>
<td>0.07 0.95 0.99 0.53</td>
<td>0.12 0.84 0.99 1.87</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.09 0.91 0.99 0.76</td>
<td>0.10 0.95 0.99 0.52</td>
<td>0.13 0.88 0.99 1.84</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>0.10 0.96 0.99 0.54</td>
<td>0.12 0.95 0.99 0.98</td>
<td>0.15 0.93 0.99 0.60</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.13 0.94 0.99 0.63</td>
<td>0.21 0.96 0.99 0.24</td>
<td>0.31 0.89 0.99 0.61</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>0.42 0.78 0.99 1.21</td>
<td>0.43 0.85 0.99 1.61</td>
<td>0.65 0.83 0.99 2.84</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>0.54 0.84 0.99 1.14</td>
<td>0.59 0.85 0.99 1.56</td>
<td>0.81 0.89 0.99 1.21</td>
</tr>
</tbody>
</table>
The decrease of apparent viscosity with increasing shear rate, for 5%, 10% 15%, 25% and 30% hydrolyzed starch at 25°C, with $n$ values ranging from 0.92 to 0.95 as shown in Table 1, is less significant than the curve for 20% starch, with $n$ equal to 0.82 characterizing a pseudoplastic behavior. For temperatures of 40°C and 55°C, $n$ values are situated in the 0.91 to 0.96 range for 5%, 10% 15% and 20% of hydrolyzed starch, and from 0.78 to 0.87 for 25% and 30% of hydrolyzed starch.

The decrease of apparent viscosity with increasing shear rate for temperatures of 25°C, and 5%, 10%, 15% and 30% of modified starch, is less significant than the decreases observed for 20% and 25% of modified starch, with $n$ values ranging from 0.92 to 0.96 and from 0.84 to 0.88 respectively, as can be seen in Table 1. The first group shows $n$ values near unity, showing a tendency toward Newtonian flow behavior. For a temperature of 40°C with 5%, 10% 15%, 20% and 25% of modified starch and for a temperature of 55°C with 5%, 10%, 15% and 20% of modified starch, the $n$ values ranged from 0.91 to 0.96. Their apparent viscosity decreases with increasing shear rates are less significant than the one observed for the curve for 30% starch and 40°C levels, and for those with 25% and 30% of modified starch at 55°C, where the $n$ values ranged from 0.83 to 0.85.

The suspension of chicory extract with a 1:1 mixture of hydrolyzed and modified starch showed non-Newtonian pseudoplastic behavior characteristics. The $n$ values fell in the 0.83 to 0.97 range as shown in Table 1.

The combination of temperature and the modified and hydrolyzed starches added to chicory extract, in the isolated forms or in the mixture form, resulted in a stable system associated with the complex structure of inulin, presenting a pseudoplastic behavior, with a Newtonian behavioral tendency with little variation in apparent viscosity.

The magnitude of the determination coefficient near unity and the relative average error below 5% observed in Table 1 for three different temperatures indicates, according to Lomauro and others (1985), that these models can be employed with confidence in the range of the fitted data.

To perform an analysis of the effects of the process and mixture variables on the apparent viscosity a deformation rate of 264 s$^{-1}$ was used for each set of experimental conditions studied (process temperature, total starch and mixture component percentages). Table 2 indicates the percentages of A and B in the mixture, the percentages of total starch (% MP) and the temperatures used in the process. This procedure could be adopted in this work since the tendencies of the apparent viscosity behavior for the different formulations are analogous to those observed in the entire range of the experimental deformation rates used.

A computer program (Bortoloti, 2001) written in one of our laboratories and the SAS program (SAS Institute, 2000) were used to perform the calculations. The following models were tested: (a) linear model in the process variables and linear in the mixture variables (b) linear model for process variables and quadratic for mixture variables, (c) bilinear for process variables and linear for mixture ones, and (d) bilinear for process and quadratic for mixture variables The models were determined by multiple linear regression and their errors were estimated in a approximate manner by making cumulative probability graphs. Model lack of fit was judged qualitatively by examining residual graphs since replicate data was not available to perform an ANOVA of the data.

The linear-linear, linear-quadratic and bilinear-linear models all showed evidence of lack of fit. Graphs of the residual values against the predicted apparent viscosities have points with a definite parabolic form. As such these models were all rejected (Barros Neto and others 1996; Box and others, 1978).

Table 2 - Apparent viscosity values ($\eta_{app}$) of the A and B components used on inulin chicory extract
as function of temperature and formulation percentage.

<table>
<thead>
<tr>
<th>Starch (%)</th>
<th>η&lt;sub&gt;app&lt;/sub&gt; (mPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25°C</td>
</tr>
<tr>
<td>Total A</td>
<td>5</td>
</tr>
<tr>
<td>A B 25°C</td>
<td>11.10</td>
</tr>
<tr>
<td>5 0 10</td>
<td>10.82</td>
</tr>
<tr>
<td>5 2.5 10</td>
<td>7.75</td>
</tr>
<tr>
<td>10 5 15</td>
<td>12.98</td>
</tr>
<tr>
<td>10 2.5 15</td>
<td>16.34</td>
</tr>
<tr>
<td>15 7.5 15</td>
<td>19.11</td>
</tr>
<tr>
<td>20 25 20</td>
<td>20.32</td>
</tr>
<tr>
<td>20 0 25</td>
<td>20.69</td>
</tr>
<tr>
<td>20 10 20</td>
<td>34.86</td>
</tr>
<tr>
<td>25 25 0</td>
<td>60.61</td>
</tr>
<tr>
<td>25 0 25</td>
<td>25.7</td>
</tr>
<tr>
<td>25 12.5 25</td>
<td>34.87</td>
</tr>
<tr>
<td>30 30 0</td>
<td>71.19</td>
</tr>
<tr>
<td>30 0 30</td>
<td>49.25</td>
</tr>
<tr>
<td>30 15 30</td>
<td>63.24</td>
</tr>
</tbody>
</table>

The combination of the bilinear model for the process variables and the quadratic model for the mixture variables furnishes the model given by equation 4. Figure 2 shows the graph of the residuals versus the predicted apparent viscosity values. This graph does not present strong evidence of lack of fit since the points are almost randomly distributed although the points for high apparent viscosity values are more disperse than those for lower values indicating heteroscedasticity in the data. 80% of the residual values are within ±5 mPa of the experimental values with most deviations larger than this occurring for predicted apparent viscosity values of over 30 mPa. As such this model:

\[
\hat{y} = -19.54x_a + 0.15x_b + 16.54x_a x_b 
\]

\[
+ (0.36x_a + 0.02x_b - 0.3x_a x_b)z_1 + 
\]

\[
(3.97x_a + 1.69x_b - 1.40x_a x_b)z_2 - 
\]

\[
(0.07x_a + 0.02x_b - 0.72x_a x_b)z_1z_2
\]

\[
(4)
\]

can be accepted as being well adjusted to the experimental data for the purpose of understanding the important factors governing the apparent viscosity values. Ideally replicate experiments should be made to allow a rigorous determination of lack of fit in an attempt to obtain a more accurate quantitative model. However this would increase the already large number of experimental determinations that would need to be made.

Figure 3 contains a graph of cumulative probabilities for a normal distribution versus regression coefficient values that have been divided by their corresponding covariance matrix weights. In this way the regression coefficients that are not significant will provide estimates of the measurement variance and are expected to fall on a straight line that is centered close to the origin. The significant regression coefficients are expected to fall far from this line. The graph in Figure 3 shows that three regression coefficients clearly cannot be considered to form part of any possible linear array of points in the middle of the graph. Excluding all other terms with statistically insignificant coefficients in equation 4 leads to the following simple model

\[
\hat{y} = -19.54x_a + 16.54x_a x_b + 3.97x_a z_2
\]

\[
(5)
\]

This model suggests there are three contributions to the variation in the apparent viscosity, a linear one depending on the proportion of hydrolyzed starch, an interaction term involving this proportion and the proportion of modified starch and finally an interaction between the hydrolyzed starch proportion and the percentage of total starch in the mixture.
Figure 2. Graph of the residual values (predicted – experimental apparent viscosities) versus the predicted values of the apparent viscosity for the bilinear-quadratic model.

An increase in the proportion of hydrolyzed starch at the cost of modified starch causes a decrease in the apparent viscosity value. However, the interaction effect of the modified and hydrolyzed starches is synergistic, increasing the apparent viscosity value. To a lesser degree, as indicated by its smaller coefficient, the interaction effect of the hydrolyzed starch proportion with the percentage of total starch in the mixture reinforces the synergic effect between the two starch proportions. This indicates that the effect of substituting hydrolyzed starch by modified starch increases the apparent viscosity more at high total starch percentages than at low ones as might be expected.

It is interesting that this model indicates that the temperature does not affect the apparent viscosity. This was confirmed by the apparent viscosity values reported here. The formulation composition influence on viscosity is greater than its variation with temperature range.

**CONCLUSION**

Pure and formulated chicory extracts with
hydrolyzed and modified starches exhibited non-newtonian flow behavior, namely pseudoplastic characteristics. The rheological index values of the power law model for formulated chicory extracts are higher than those for pure extract, minimizing the pseudoplastic characteristics of the fluid under study. The rheological index values varied from 0.42, corresponding to pure chicory extract, to 0.97, corresponding to the formulated chicory extract. Multiple linear regression to determine combined models involving both process and mixture variables has shown to be a useful tool for understanding these complex processes. The model determined here shows that the apparent viscosities of suspensions containing chicory extract formulated with two different starches are principally influenced by the fractions of hydrolyzed and modified starches, their interaction and the percentage of total starch in the system. Temperature does not appear to affect the apparent viscosity of this system.

ACKNOWLEDGMENTS

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BIBLIOGRAPHY REFERENCE


Nomenclature

\textit{ERM} \quad \text{Mean relative deviation, \%} \\
K \quad \text{Consistency index, mPa.s} \\
\textit{n} \quad \text{Flow behavior index, dimensionless} \\
\textit{ne} \quad \text{number of experimental data} \\
R^2 \quad \text{Determination coefficient} \\
T \quad \text{Temperature, °C} \\
\textit{VE} \quad \text{Experimental value} \\
\textit{VP} \quad \text{predicted value} \\
X_A \quad \text{percentage of component A (modified starch) in the starch mixture} \\
X_B \quad \text{percentage of component B (hydrolyzed starch) in the starch mixture} \\
\textit{z_1} \quad \text{codified temperature values} \\
\textit{z_2} \quad \text{percentage mass fraction of total starch (both components A e B) in the mixture} \\
\eta_{\text{app}} \quad \text{Apparent viscosity, mPa.s} \\
\hat{\gamma} \quad \text{predicted apparent viscosity, mPa.s} \\
\dot{\gamma} \quad \text{Shear rate, s}^{-1} \\
\tau \quad \text{Shear stress, mPa}