Effect of operating conditions on physical characteristics of extruded rice starch

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Summary

The effect of screw speed, flow rate, barrel temperature and added moisture on physical characteristics of high moisture extruded rice starch was studied. The product physical characterization was made by measuring density, final moisture content and the maximum load force in a puncture test. Rice flour containing approximately 1.5% protein and 6% moisture was mixed with 55–65% (w/w) water and extruded in a co-rotating twin screw extruder with a slit die. A two level factorial design was used to analyse each parameter. It was found that added moisture was the most important variable affecting the force-deformation curve maxima, with the interaction between added moisture and barrel temperature exhibiting a less significant effect. Added moisture and flow rate each affected density, with the interaction between added moisture and screw speed showing a less significant effect. Temperature and flow rate affected final moisture interactively with added moisture, which was evidently the most important variable.

Keywords

Extrusion process parameters, gelatinization, high moisture doughs, product density, texture viscosity, wet extrusion.

Introduction

High temperature–short time extrusion cooking is widely used in the food industry to produce a variety of products, being an extremely versatile operation.

The effect of extrusion conditions on the quality of the final product is extremely important and has been generally studied: Mercier & Fellet (1975) and Mercier (1977) analysed, respectively, the effect of barrel temperature on the expansion of corn starch and potato starch. Owusu-Ansah & Voort (1984) investigated textural and microstructural changes in corn starch as a function of extrusion variables. Chiang & Johnson (1977) studied the effects of operating conditions on starch gelatinization. Launay & Lisch (1983) related the viscoelastic properties of melted starch to its expansion volume. Chinnaswamy & Hanna (1987) concluded that the die nozzle length-to-diameter ratio affected the expansion of corn starch. Linko *et al.*

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(1981) and Faubion *et al.* (1982) described the physical and chemical properties of extruded products based on extrusion conditions. The use of extrusion technology to modify the characteristics of starchy materials is mainly based on empirically acquired knowledge (Faridi & Faubion, 1982), due to the complexity of the process. For this reason, experimental results are very important. There is little information available concerning rice starch extrudates and therefore obtaining such information for this material was one objective of this work.

Another aspect of increasing importance is the study of the extruded doughs, not just of the very dry products that are the most important extruded products (breakfast cereals, etc.). The extrusion of high moisture doughs (also called wet extrusion) is a promising method of making better meat analogs, and providing new ways of improving texture and modifying taste. The extrusion of defatted soy flour, to obtain a meat analog, and the extrusion of various marine products have been reported in the literature (Noguchi, 1989). Another emerging application of wet extrusion is in the use of extruders as bioreactors. The first application in this area was the liquefaction of starch to produce oligosaccharides, for subsequent saccharification to sugar syrups (Ofoli *et al.*, 1987, Linko *et al.*, 1983, Halukin *et al.*, 1983, van Zuilichem *et al.*, 1990). These works have shown that a high moisture feed (up to 70%) resulted in increased starch hydrolysis and that the resulting oligosaccharide solution significantly increased the overall efficiency of the saccharification, compared to conventional enzymic reactors. This observation would be particularly applicable when the objective was the fermentation of the resulting sugars to obtain alcohol (Linko *et al.*, 1983). The possibility of producing modified starch, with a controllable degree of modification, would also lead to a variety of additives, like bonding and thickening agents.

The objective of this study was to analyse the effect of some of the most important operating variables on physical characteristics of a high moisture rice starch extrudate. Screw speed, added moisture, flow rate and barrel temperature were the operating variables or process parameters considered, and force-deformation curve maxima, moisture content and density the product characteristics measured. The force-deformation curve, obtained with a puncture test, is an empirical measurement that can be related to textural characteristics of the product and is widely used in the field of textural analysis. This measurement was performed after preliminary experiments showed that it would lead to more reproducible results than other alternatives.

The process parameters control other variables that can be called system parameters: specific mechanical and thermal energies and residence time distribution. The system parameters are preferable to relate observations to modifications occurring in the dough.

A good statistical design decreases experimental requirements and provides a solid basis for discussion. A non-replicated factorial design with factors at two levels was used. This method allows for the identification of the operating variables (factors) that significantly influence each product characteristic (response) and of the relevant interactive effects.

Materials and methods

Raw material

A rice starch mixture containing 86% (minimum) starch (California Natural Products, Lathrop, CA) with a 1.5% protein content and a 6.0% moisture content

Table 1. Limits of the operating variables

Limit	W ₀ added moisture (% w/w)	T barrel temperature (°C)	N screw speed (r.p.m.)	Q flow rate (10 ⁻⁶ m ³ s ⁻¹)
low (-)	55	60	175	1.25
high (+)	65	100	275	2.0

was used. The starch powder was blended with tap water (hardness: 30 mg l⁻¹ CaCO₃) in a Hobart mixer, in the amount defined by the added moisture to be incorporated.

Extrusion

Experiments were conducted in a laboratory size torque rheometer (Sys 90, Haake Buchler Company) and co-rotating twin-screw extruder (MPC/V-30, APV Baker Ltd, Staffordshire, UK) combination, attached to a slit die (Haake Buchler Company). The screws had four sections: a feed section with deep flights and 222-mm axial length, a kneading section of 33 mm axial length, a compression section with a decreased pitch and 98-mm axial length and a metering section with very shallow flights and 28.5-mm axial length. The temperature along the extruder barrel and slit die was controlled by electrical heating and air cooling systems. This screw configuration was suitable for high moisture doughs. The extruder could be opened up easily, when stopped, and this feature was used to verify screw filling. Screws appeared to be full at all operating conditions, in spite of the absence of reversed sections, and this may be due to the high moisture content of the dough.

The extruder operating conditions were characterized by four independent variables (Table 1): screw speed, barrel temperature, feed rate and added moisture (the water added to the rice starch mixture, expressed as added water weight/rice feed weight).

The physical characteristics of the extruded rice starch considered were density, final moisture content and an empirical measurement of textural characteristics. Preliminary experiments showed that a puncture test gave more reproducible results than other tests more related to rheological properties, such as a compression test, and was therefore chosen. The maximum load force (the force-deformation curve maximum) was also the most reproducible parameter in these tests. Although of an empirical nature, puncture tests are widely used in the field of texture analysis (Bourne, 1982). A puncture test basically measures the resistance offered by a sample to the penetration of a narrow flat-end cylindrical punch. Since no correlation was made between these measurements and texture perception, it was preferred to maintain 'force-deformation curve maximum' throughout the text instead of 'texture', 'textural characteristics' or other words linked to texture perception, since these are open to ambiguity.

Experimental design

In order to analyse the influence of each factor (operating variable) independently and possible interactions between factors, a non-replicated factorial design with factors at two levels was used (Box *et al.*, 1978; Gacula & Singh, 1984). This methodology minimizes experimental requirements and allows the results to be interpreted with a

solid statistical basis. Low and high levels were defined for each factor, covering the range of interest (see Table 1). A total of 2^4 (16) experimental runs was required, conducted in random order. Samples of products were collected for each run, after allowing the extruder to reach a steady state, determined by constant temperature and pressure. The configuration of the screws and of the slit die was the same in all runs.

For each response, the effect of a factor or an interaction between factors can be determined by a normal plot of the effects which gives the probability effect. The equations used for calculating the effects can be found in Box *et al.* (1978) and Gacula & Singh (1984).

In general terms, the normal plot is simple to interpret: all factors and interactions that have no significant effect on a response will fall on a straight line. Points that fall outside this line correspond to variables or interactions that significantly affect the response.

Analysis of the extrudates

Extrusion at these high moisture levels leads to extrudates that are semi-rigid viscoelastic pastes, harder or softer depending on the exact operating conditions. It is important to note that these extrudates are not dry porous solids. The product leaves the extruder as a thick sheet, which expands slightly at the die exit, and can be cut as desired.

Moisture content

Extrudate moisture was determined in triplicate per extruder run on samples collected in aluminium dishes, using method no. 925.09 (AOAC, 1990).

Bulk density

Extrudate density was determined using a pycnometer, following method no. 936.13 (AOAC, 1990) for measuring densities in solids. Each value was an average of three different samples.

Force-deformation curve maxima

Samples for analysis were collected in plastic trays and covered with aluminium foil to prevent moisture loss. Samples were analysed after 6 h, at room temperature, in an Instron Universal Testing Machine, Model 1122 (Instron Corporation, Canton, MS, USA). The maximum load force in a puncture test was the parameter selected to quantify the textural characteristics of the samples. A 2-kg load cell was used. The crosshead was set to drive a 10-mm diameter flat-end cylindrical punch into samples with a surface area of 50×100 mm, at 50 mm min^{-1} , to 70% of its original height. The maximum load force was recorded by a computerized data acquisition system attached to the Instron (Instron Corporation, Canton, MS-Series IX, v. 4.10 utilities). Each value was an average of five tests, performed at different locations in a sample.

Results and discussion

Effect on force-deformation curve maxima

For the purpose of this discussion, it is accepted that a higher maximum load force corresponds to a harder sample. Although intuitive for this type of product,

Figure 1. Normal plot of the effects on the maximum load force (Kg).

Figure 2. Interactive effect of added moisture (W_o) — temperature (T) on the maximum load force (Kg). Values in bold at each corner indicate the mean maximum load force (Kg) for the corresponding levels of W_o — T (see Table 1). Values in italic indicate the variation in the maximum load force (Kg) between corners, represented proportionally by the bold lines.

this relation was not experimentally proven with these samples, which would require a measurement of true hardness.

Figure 1 shows the normal plot for the maximum load force. It can be seen that added moisture and temperature are the only variables with a significant effect and that temperature has an interactive effect with added moisture. Figure 2 visualizes the interaction of the two levels of moisture and two temperatures as the sides of a square. If all other variables have no significant effect, then experiments with screw speed or flow rate as independent variables are replicates of the same relevant conditions. The average can be taken for all runs with the same added moisture and temperature combination. The corners indicate the four possible combinations and the average value of the maximum load force is indicated in bold characters. Bold

lines are drawn on the sides of the square according to the magnitude and direction of the increase in the maximum load force. The length of these bold lines is proportional to the variation of the parameter (with the largest variation having 100% of the length). The lines are drawn from left to right or bottom to top if the variation is positive (the response increases) and from right to left or bottom to top if the variation is negative (the response decreases with increase in the variable). The absolute value of the change in maximum load force is also indicated in italic.

The length of the bold lines shows that the effect of the added moisture was more important at the higher temperature. The effect of temperature depended dramatically on the added moisture. For low added moisture, the effect was more significant and analysing the direction of the bold lines it can be seen that at the lower added moisture increasing temperature increased the resistance of the sample to penetration, while at high added moisture increasing temperature had an opposite effect. For added moisture, the effect was always negative: increasing it led to lower force-deformation curve maxima.

This square form of representation describes the limits but nothing in between; points of maximum or minimum could occur. Higher level factorials would be required to analyse this. Alternatively, one could try to interpret results phenomenologically and infer from the analysis whether this is likely to be the case.

It is known that water has a plasticizing effect, lubricating starch chains. Therefore, chain mobility is decreased with a decrease in moisture (Halek *et al.*, 1989). This chain-stiffening results in higher resistance to compression, which may also correspond to a higher resistance to penetration for this type of material, and hence a higher maximum load force for the lower added moisture level. The temperature by added moisture interaction can be related to the range of moisture contents used. All values correspond to high moisture contents, with excess water and therefore lower shearing. Temperature controls material conversion at these conditions. Since the feed contained proteins, at low moisture, protein coagulation might have taken place, particularly at the higher temperature. This would have a stiffening effect, which would explain the observed higher maximum load force at minimum moisture and higher temperature. The absence of such a significant extent of protein coagulation, as moisture increases, would diminish this effect and eventually temperature changes would not affect texture very significantly and the response to the penetration test would therefore be reasonably constant. The reverse effect (-0.085) found at high moisture was small and can be considered negligible. Therefore, temperature only affected the result at low moisture content.

The following conclusions can therefore be made, noting that for this type of product it is acceptable that resistance to penetration might be directly related to perceived hardness to bite:

1. For an increased resistance to penetration, the added moisture should be reduced. This will have a greater effect at higher barrel temperatures.
2. If a lower resistance to penetration is required, the added moisture should be increased, with a greater effect at higher temperatures. Alternatively, if the added moisture is low, a temperature decrease will also result in a decrease in resistance to penetration.
3. At intermediate moistures it may be possible to change barrel temperature without affecting the force-deformation curve significantly.
4. Screw speed and flow rate can be changed without affecting the force-deformation curve.

Figure 3. Normal plot of the effects on density (Kg/m^3).

Figure 4. Individual effect of (a) added moisture (W_0), (b) flow rate (Q), and (c) interactive effect added moisture (W_0) – screw speed (N) on extrudate density (Kg/m^3). Values in bold at each corner indicate the mean density (Kg/m^3) for the corresponding levels of W_0 – N (see Table 1). Values in italic indicate the variation in the density (Kg/m^3) between corners, represented proportionally by the bold lines.

Effect on density

It can be observed from the normal probability plot for density, shown in Fig. 3, that the density of the extrudate was affected by the added moisture of the feed and the flow rate. The interaction of the screw speed with the feed moisture was also significant, but to a lesser extent. These effects are shown in Fig. 4. The individual

effects of added moisture and flow rate are shown separately, since the use of squares is conventionally linked to interactive effects (Box *et al.*, 1978). Since temperature and screw speed effects could be neglected, runs where only one of these variables has changed can be interpreted as in the texture analysis; they are replicates of similar experimental conditions. The less relevant added moisture-screw speed interactive effect is however also interesting to analyse. In this case, the values at each corner of Fig. 3 are not an average of (approximate) replicates, since flow rate is an important effect. These values are simply an indication of the importance of varying only one of the interactive variables at one extreme of the other variable.

As illustrated in Fig. 4(b), density increased with increasing flow rate and decreasing added moisture. The interactive effect shows that the effect of moisture was more important at higher screw speed and that increasing the screw speed increased density at low added moisture while decreasing it at high added moisture.

These observations can be interpreted by considering that increasing moisture increases the mobility of protein molecules present in the mixture feed, thereby increasing the probability of reactive sites getting close enough to form cross-links between adjacent molecules, and thus inducing an expandable structure (lower density). Moreover, moisture increase leads to a viscosity decrease at the die which may have permitted higher expansion and reduced density. Such observations were reported by Holay & Harper (1982). Therefore, one would expect the moisture content effect to be gradual, without a maximum or minimum.

In relation to the flow rate effect, it can be said that as the feed rate increases, the shear rate increases, causing starch molecules to stretch, weakening bonds (Bhattacharya *et al.*, 1986) and resulting in a lower expansion and denser product.

The interactive effect of screw speed with added moisture may be explained by the fact that an increase in screw speed increases the shear stress and shear rate and therefore the viscosity of the dough (for a pseudoplastic behaviour). Similar observations have been reported by Faridi & Faubion (1982). The reduced viscosity leads to reduced density. Therefore, increasing screw speed enhances the effect of added moisture on product density. This effect should have a decreased relevance as moisture content increases since most starch gels exhibit a more significant shear thinning behaviour and a higher consistency the higher the starch concentration. The fact that increasing screw speed increases viscosity at low moisture contents may also be explained by this. The reverse effect at high moisture content can be attributed to a change in the rheological characteristics of the fluid resulting from the different conditions. Low moisture was linked to more significant protein coagulation and therefore it is possible that the different dough characteristics relate to different rheological behaviours. The reverse effect could be associated with the appearance of dilatant behaviour, where increasing the shear rate increases the viscosity and therefore density is decreased.

To summarize, it can be concluded that:

1. To increase density, flow rate can be increased. This effect is not changed significantly by the other variables. Alternatively, moisture can be decreased, which will have a more significant effect at higher screw speeds.
2. Screw speed can also be changed to adjust density slightly, but only for high or low added moisture. This effect depends however on which limit of added moisture is used: increasing screw speed increases density at low added moisture and decreases it at high added moisture.

Figure 5. Normal plot of the effects on the extrudate moisture content (%).

3. Temperature can be changed without affecting density within the experimental range considered.

Effect on the final moisture content

Figure 5 shows the normal plot for the final moisture content. Added moisture was obviously the most important effect. Temperature also showed a significant individual effect. These are the only independent variables that fell outside the 95% confidence region. However, the effect of added moisture is so large that it can distort the other observations. Although less relevant, it would be important also to analyse the added moisture and flow rate interaction and the added moisture, temperature and flow rate interaction. In this case, the normal plot shows more clearly the rationale for considering these effects than the calculation of the probability values for each effect. Overall, the analysis can be carried out simply by analysing the triple effect, since it includes all variables that are significant. A triple effect can be analysed easily with a cube (Fig. 6). The sides of each face show the effects of two variables for one extreme of the third variable, and the convention used is the same as with the squares used for double effects in Figs 2 and 4. Since the effect of added moisture was much more significant than the others, the added moisture scale is broken for better comparison with the effects of the other variables. Values where only screw speed varied are considered as duplicates, with the average of both being the value shown in each corner of the cube.

As illustrated in Fig. 6, added moisture affected final moisture directly: as feed moisture increased, higher extrudate moisture was obtained due to the higher amount of water present in the dough. This effect was not influenced by flow rate at lower temperatures while at higher temperatures it became more significant for high flow rate and less significant for low flow rate.

The flow rate only affected results at high temperatures. Due to the interactive effects, increasing the flow rate decreased product moisture content for low added moisture and increased it for high added moisture.

Increasing temperature decreased product moisture, but the effect was only

Figure 6. Interactive effect of added moisture (W_o) — temperature (T) — flow rate (Q) on extrudate moisture (%). Values in bold at each corner indicate the mean extrudate moisture (%) for the corresponding levels of W_o - T - Q (see Table 1). Values in italic indicate the variation in the extrudate moisture (%) between corners, represented proportionally by the bold lines.

significant for high added moisture at low flow rate and for low added moisture at high flow rate. Temperature did not significantly affect final moisture when the added moisture and the flow rate were both high or both low.

All the observations mentioned can be related directly to the flash of water that occurs as the product leaves the die. The flash can be influenced both by temperature and by flow rate. When temperature increases, a higher vapour pressure is created in the dough and hence an increased flash of moisture (Badrie & Mellows, 1991). The effect of flow rate can be related to the input of specific energy. Colonna *et al.* (1984) have identified a reduced input of the specific energy with increasing flow rate. Hsihe *et al.* (1990) have reported lower vapour pressures in the dough as a result of the reduced input of specific energy and hence a lower flash of moisture. Increased final moisture content as a result of increasing flow rate was observed at high moisture content only, while at lower moisture content the effect was reversed. It should be noted that this reverse effect was obtained for the high temperature only and therefore one can speculate that it was due to the interactive effect of the three variables. However this effect was statistically small and could be neglected.

The following conclusions can therefore be made:

1. For controlling the extrudate moisture content, the most straightforward way is obviously to change the added moisture in the same direction. For this purpose, it is beneficial to work at high flow rate and temperature, where the effect of the added moisture is maximum.
2. A small adjustment of the final moisture can be made by varying the flow rate, but only at high temperatures. Increasing flow rate increases moisture content for high added moisture and decreases it for low added moisture.

3. Temperature also allows for an adjustment of the final moisture. Temperature increases cause a decrease in the moisture content, but this action is only significant for high flow rate and low added moisture or high added moisture and low flow rate.
4. Screw speed can be changed without affecting the product moisture.

Conclusions

Overall conclusions can be obtained by analysing the effect of changing operating conditions in the three parameters (force-deformation curve maxima, density and final moisture content).

The textural characteristics of the extrudate can be affected in such a way that the maximum load force in a puncture test can be increased by decreasing moisture content. This is likely to imply that the hardness is increased. However, this decreases both the density and the final moisture content. To compensate partly for this effect, the flow rate can be increased, adjusting the density without affecting the other physical characteristics considered. However, for the lower moisture contents, an even greater decrease in the final moisture is caused by the increasing flow rate if temperature is high. Therefore, to increase physical resistance to puncture it is better to work at low temperatures, in spite of the lower effect of the decrease in added moisture for this region. It is suggested that this corresponds to a harder material.

To decrease the density, flow rate can be decreased without affecting the other physical characteristics, but only at low temperatures. At high temperatures changing the flow rate will also slightly affect the final moisture content. Density can also be decreased significantly by decreasing the added moisture, but this has a stiffening effect (higher viscosity) and significantly decreases the final moisture as well.

For controlling the product moisture content, temperature and flow rate can be varied, but have only a limited effect, being more adequate for small adjustments. The best option is to change the added moisture, although this also affects the other physical characteristics considered.

For the range of experimental values, the screw speed can be changed without significantly affecting the results, except for the small effect of making density a little more sensitive to added moisture changes. For energy efficiency reasons, one can therefore work at lower screw speeds.

Energy saving would also be improved by working at lower temperatures, which can be done without affecting the product response to a puncture test. Under these conditions, the force-deformation curve maxima and the final moisture content are determined by the added moisture only. The effect of added moisture on the maximum load force in a puncture test is, however, less significant than if the temperature was high.

Increased productivity would suggest higher flow rates, which will not affect the force-deformation curve maxima nor the final moisture content, if low temperatures are used. However, there will be an effect on density.

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