# Characterization of deep-fat frying in a wheat flour-water mixture model using a state diagram

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# Abstract

BACKGROUND: Crispness is an important characteristic to be controlled in deep-fat fried products. The physical state of food polymers influences the development of cellular structure and textural qualities of fried food. Glassy state is believed to play an important role in the mechanical properties of low-moisture food. Therefore, an understanding of the physicochemical phenomena in the development of fried food structure using a state diagram of the frying process is discussed.

**RESULTS:** Wheat flour models containing 400 and  $600 \text{ g kg}^{-1}$  initial moisture content were fried in frying oil at 150 °C for 1–7 min. Thermal properties of wheat flour, structure alteration and textural properties of fried samples were evaluated. The porous structure continuously enlarged when the sample was in the rubbery state. As the frying time was prolonged, the state of the product became glassy due to a decrease in water content, resulting in the ceasing of porous enlargement.

CONCLUSION: The results revealed that physicochemical changes during frying influence the alteration of microstructure and quality of fried food, and the state diagram could be applied to explain the formation of microstructure during the frying process and used as a decision-making tool to choose the proper conditions to provide desirable qualities in fried food.

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Keywords: deep-fat frying; state diagram; porous structure; crispness; wheat flour-water mixture

# INTRODUCTION

Crispness is a unique characteristic and an important parameter to be controlled in deep-fat fried products. The physical state of food polymers influences the development of cellular structure and consequently the textural qualities of fried food during both processing and storage. The development of microstructure during frying is one of the predominant factors affected by oil absorption.<sup>1</sup> Pore development was found to influence the final oil uptake during frying.<sup>2</sup> Moreover, the porosity that occurs in the crust during frying corresponds to the surface roughness, consequently affecting oil absorption in the post-frying process.<sup>3,4</sup>

The glass transition of polymer foods is believed to be a factor in the formation of cellular structure in extruded foods and baked products.<sup>5,6</sup> Important changes in the physical properties of food polymers, i.e., molecular mobility, viscosity, and elasticity, occur as they pass through their glass transition temperatures ( $T_g$ ). The importance of the rubbery and glassy states in understanding the behavior of high-solid foods has become extensively appreciated, leading to the food-polymer approach to the study of structural properties.<sup>7,8</sup> Molecular mobility influences the structural formation and consequently food quality in extruded foods and baked products. For instance, cellular formation and crispness in the extruded product are desirable properties; porosity formation ceases when the extrudate temperature becomes lower than its  $T_{\rm g}$ ;<sup>5</sup> keeping the food well below the  $T_{\rm g}$  condition can prolong crispness and shelf-life.

Based on thermal analysis,  $T_g$  plays an important role in the mechanical properties of low-moisture food. The formation of microstructure in fried foods is similar to that in extruded foods and baked products. The alteration of physical state and water vapor expansion inside food material, by heating in the hot oil, bring about the cellular structure as well. Therefore, an understanding of the physicochemical phenomena in the development of fried food structure can help food manufacturers create conditions that enhance fried food quality. A state diagram, which shows physicochemical changes as a function of temperature and water content,



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illustrates the transformation that takes place during both processing and storage. The state diagrams for cereal food products, such as extrusion foods, biscuits and cookies, and bread, also were prepared.<sup>6,8–11</sup> However, there are very few approaches characterizing fried products using state diagrams.

Among the few published studies on frying state diagrams, Vitrac et al.12 proposed a state diagram that was constructed from fried food temperature and residual water content to predict the principal reactions of materials during the frying process; this diagram, however, was not constructed using practical physicochemical properties. Kawas and Moreira<sup>13</sup> created  $T_{\rm g}$  curves for raw tortilla and tortilla chips; however, only the relationship between the glass transition temperature and the water content was displayed, with no data on the alterations in phase transformation throughout the process. In this study, the physical state relating to the alteration of structural characteristics in fried samples was made clear by using a simple wheat flour-water mixture model. The physical state for such a model system was investigated using thermal analysis as fundamental information, and the diagram was constructed. In addition, the state diagram of the deep-fat fried food for the wheat flour model was constructed to characterize the alteration of microstructure during the frying process and the crispness of fried samples.

# MATERIALS AND METHODS

# Thermal properties of wheat flour

Differential scanning calorimetry (DSC) (DSC-50 instrument, Shimadzu, Japan) was used to determine the glass transition  $(T_g)$  and the melting temperature of commercial wheat flour. For calibration of temperature and heat capacity in DSC measurement, indium (Wako Pure Chemical, Osaka, Japan) and distilled water were used.  $\alpha$ -Alumina was used as the reference material. The wheat flour was defatted by 750 g kg<sup>-1</sup> *n*-propanol, and the moisture content was adjusted by exposure to the saturated salts K<sub>2</sub>SO<sub>4</sub> (0.973*a*<sub>w</sub>), KNO<sub>3</sub> (0.924*a*<sub>w</sub>), KCl (0.842*a*<sub>w</sub>),  $Mg(NO_3)_2.6H_2O$  (0.528 $a_w$ ),  $CH_3COOK$  (0.224 $a_w$ ), and LiCl  $(0.11a_w)$ . Samples with higher moisture content were prepared by mixing defatted wheat flour and distilled water. The 20 mg samples were weighed into an aluminium pan. The scanning temperature was 0-200 °C with a heating rate of 5 °C min<sup>-1</sup>. Average values of two replicate samples were taken as  $T_{\rm g}$  and melting temperature. The moisture content of samples was determined by difference in weight before and after the pitted DSC pans were dried after the second scanning. Glass transition temperature was determined from the onset of an endothermic shift of baseline using TA-60 software interfaced with the calorimeter.

# Sample preparation and frying

Model samples were prepared using wheat flour at initial moisture levels of 400 and  $600 \text{ g kg}^{-1}$  (wet

basis). These levels were achieved by mixing the flour with distilled water at room temperature  $(25 \,^{\circ}\text{C})$  for 15 min. The 400 g kg<sup>-1</sup> sample was re-formed between two rollers of a pasta maker (Imperia, Turin, Italy) until a final thickness of 0.8 mm was achieved. The sample was subsequently cut using a circular stainless steel mold into a 5 cm diameter circular disk weighing  $3.00 \pm 0.05$  g. The batter-like 600 g kg<sup>-1</sup> moisture sample was weighed into a 5 cm diameter Teflon-coated circular mold weighing  $4.50 \pm 0.05$  g, as illustrated in Fig. 1. The weights of these two types of sample were strategically chosen so that they would contain equal quantities of wheat flour.

All samples were then deep-fat fried in an oil bath (TM-4, As One, Osaka, Japan) containing 4.5 L soybean oil (Nisshin, Tokyo, Japan) at  $150 \,^{\circ}$ C for 1, 3, 5, or 7 min. Preliminary tests indicated that there were no significant differences in the amount of free fatty acid in the frying oil when it was used for less than 3 h. Therefore, the oil used in this study was discarded after 3 h of frying. The residual moisture content of the fried samples was determined by drying in a hot-air oven at  $105 \,^{\circ}$ C for 12 h or until the weight remained constant, in accordance with AOAC methods.<sup>14</sup> The residual moisture content (wet basis) was calculated for each sample. Duplicate samples were used at each frying time, and the average is reported.

# **DSC** of fried samples

The fried samples were defatted by homogenizing with a 2:1 chloroform-methanol solution for 5 min and then filtered through filter paper (Whatman no. 1). The defatted fried sample was dried in a 25°C oven for 12h. The moisture content of the fried samples was adjusted to be nearly similar to their former residual moisture content, i.e., the residual moisture content before the lipid extraction, by exposing them to saturated salts. The 20 mg samples were weighed into an aluminium pan. The scanning temperature was 0-200°C with a heating rate of 5°C min<sup>-1</sup>. Average values of two



Figure 1. Teflon-coated circular mold used for frying the 600 g  $\rm kg^{-1}$  sample.

replicate samples were taken as  $T_{\rm g}$  and melting temperature of the fried samples at each frying time.

# **Temperature profile determination**

The temperature profile at the centers of the two samples was measured using a thin thermocouple (Type E,  $0.4 \times 0.7$  mm, Omega Engineering, Stamford, CT, USA). For the 400 g kg<sup>-1</sup> sample, the thermocouple was inserted at the center of the sample. In the case of the 600 g kg<sup>-1</sup> sample, the thermocouple was installed at the center of the mold, where it could directly contact the sample. Time–temperature data were recorded during the frying process using a data acquisition system (Thermodac 5001A, Etodenki, Tokyo, Japan).

# **Texture measurements**

All samples were deep-fat fried for 1, 3, 5, or 7 min. The fried samples were kept in Ziploc bags (Asahi Kasei, Tokyo, Japan) when the temperature of samples had decreased to room temperature and the texture determination was conducted within 1 h after frying. Tensipresser (TTP-50BX, Taketomo, Tokyo, Japan) was used to evaluate the texture of the fried samples. Puncture tests were performed (at 25 °C) by using a 3 mm diameter flat-ended cylindrical probe. The samples were placed on a platform with a 10 mm diameter hole; cross-head speed was  $1 \text{ mm s}^{-1}$ . The plunger was pressed through the sample to a depth of 1.5 cm and repeated 15 times for each treatment.

# Scanning electron microscopy (SEM)

Samples from all treatments were fried in oil at 150 °C for 1, 3, 5, or 7 min. After frying, the samples were immediately dipped in petroleum ether for 24 h to extract the oil from the samples.<sup>15</sup> The defatted samples were dried, mounted on stubs, and coated with platinum gold. The cross-sectional surface of the coated samples was observed using a scanning electron microscope (S-4000 model, Hitachi, Japan) with an accelerating voltage of 15 kV.

# RESULTS

#### State diagram of wheat flour

The thermal properties of defatted wheat flour, as assessed by DSC, are shown in Fig. 2. On the first scan large relaxation peaks were observed, whereas on the second run the transitions were less clear and broader. Two different  $T_g$  values were also identified in the second scan. Previously published studies<sup>16–20</sup> and our unpublished results on the  $T_g$  values of starch and gluten have indicated that these two transitions probably correspond to a starch-rich phase for the higher-temperature transition and to a gluten-rich



Figure 2. Typical DSC power-time curves of defatted wheat flour.



Figure 3. State diagram of wheat flour.

phase for the lower-temperature transition. However, several studies have reported only a single apparent  $T_g$  for wheat flour by the DSC studied.<sup>6,10</sup> Kalichevsky and Blanshard<sup>16</sup> found two glass transitions in a mixture of amylopectin and gluten due to the two polymers, where there is sufficient difference between the  $T_g$  values of these two components for them to be detected separately. Oil removal from the wheat flour in this study probably induced the immiscibility of gluten and the starch fraction, making it possible to observe two different glass transitions.

Figure 3 shows the wheat flour state diagram that was constructed from the thermal properties as a function of moisture content of wheat flour. From the obtained result, the apparent  $T_{g}$  values of gluten and starch decreased with increasing moisture content. The depression of  $T_{\rm g}$  and melting temperature as moisture content increases indicates that water is a good plasticizer for wheat flour. In the highermoisture content sample, the melting endothermic peak appeared at a lower temperature. At a moisture content of more than  $650 \,\mathrm{g \, kg^{-1}}$ , melting temperature was independent of the moisture level (known as gelatinization). The detectable individual  $T_{\rm g}$  values of starch and gluten could be observed in wheat flour containing moisture contents of  $70-250 \text{ g kg}^{-1}$ . Only one glass transition temperature at maximal freezeconcentration  $(T_g')$  was observed when moisture content was more than 250 g kg<sup>-1</sup>. This result may be caused by the greater miscibility at higher water contents.16

# State diagram of deep-fat fried sample

Thermal properties of samples containing 400 and 600 g kg<sup>-1</sup> moisture that were fried for 0, 1, 3, 5, and 7 min were assessed. In the first heating scan, the fried sample was heated to a temperature higher than the frying temperature in order to delete the thermal history of the frying process.  $T_g$  and melting temperature of the fried sample are shown in Fig. 4. In the 400 g kg<sup>-1</sup> sample (Fig. 4a), the melting temperature of starch granules in samples fried for 1 or 3 min was observed. That is, the amount



**Figure 4.** Thermal properties of fried sample containing (a) 400 g  $kg^{-1}$  and (b) 600 g  $kg^{-1}$  initial moisture.

of water in this sample was not sufficient to cause gelatinization. However, the residual moisture content of samples fried for 5 or 7 min was too low to detect the melting temperature of starch. For the 600g  $kg^{-1}$  sample (Fig. 4b), no melting temperature was observed because the starch granules were already destroyed by gelatinization during frying. A single apparent  $T_g$  was observed in both samples; this may be the result of some compounds that developed during the frying process, leading to more a complicated composition in these fried samples. The  $T_g$  at both moisture content levels increased as a function of frying time due to decreasing residual moisture content in the frying process. However, the  $T_{\rm g}$  of fried samples at both moisture levels was lower than the starch  $T_{\rm g}$  and gluten  $T_{\rm g}$  curves in the wheat flour state diagram (Fig. 3) at the same moisture content.  $T_{g}$ depression may be affected by starch gelatinization during heating in the frying process. Chung et al.<sup>21</sup> reported that the  $T_g$  of gelatinized starch is lower than that of native starch. The crystalline regions suppress the mobility of the amorphous molecules because they behave as cross-linkages between amorphous regions of the polymeric network.<sup>22</sup>

# The alteration of fried food structure

The structural alteration of the fried wheat flour model containing 400 and  $600 \text{ g kg}^{-1}$  initial moisture

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Figure 5. SEM photos of fried samples containing (a)  $400 \text{ g kg}^{-1}$  and (b)  $600 \text{ g kg}^{-1}$  initial moisture (×60, original magnification).

at various frying times was observed by SEM, as shown in Fig. 5. For the 400 g kg<sup>-1</sup> sample (Fig. 5a), small pores were formed at the beginning of frying and then became larger as the frying time was prolonged. However, no difference in microstructure was observed in the sample after frying for 3-7 min. In the 600 g kg<sup>-1</sup> sample (Fig. 5b), porous structure was continuously altered for 5 min. At 7 min frying time the pore size distribution was similar to that at 5 min. These observations suggested that the extension of porous structure ceased in the first 3 and 5 min of frying in the 400 and 600 g kg<sup>-1</sup> samples, respectively.

# Texture profile of fried food

Crispness is a desirable quality in deep-fat fried food. The crispness of snack foods is often evaluated by determining their fracture force and the number of peaks.<sup>23</sup> Fracture force is the force at the first significant break in the first peak in one bite area in the texture profile.<sup>24</sup> A lower force to fracture the sample and high number of peaks correspond to higher fracturability; i.e., the sample can be broken more easily.

The texture profiles and fracture force of the 400 and  $600 \text{ g kg}^{-1}$  fried samples are depicted in Figs 6 and 7, respectively. The texture profiles of all samples



**Figure 6.** Texture profiles of fried sample containing (a)  $400 \text{ g kg}^{-1}$  and (b)  $600 \text{ g kg}^{-1}$  moisture at various frying times.

in each treatment are shown in the same chart. It was seen that the longer frying time resulted in a lower force to break the samples. The force at the point where the probe went through the sample was lower. From the texture profile of the 400 g kg<sup>-1</sup> sample in Fig. 6(a), all samples fried at 1 min have no fracture behavior. A jagged line in the texture profile was observed for the samples fried for 3, 5, or 7 min; i.e., the fracture behavior appeared at 3 min of frying. At 3 min of frying, the force required to pass through the 3 min fried sample was observed. It was observed that the fracture force of the 7 min fried sample was lowest throughout the frying process (Fig. 7). In the texture profile of the fried sample with 600 g kg<sup>-1</sup> initial moisture content, a remarkable fracture peak was observed in samples fried for 5 or 7 min (Figs 6b and 7). At 7 min of frying time, a higher number of peaks and a lower fracture force were observed.

# DISCUSSION

#### State diagram for the deep-fat frying process

Glassy foods are formed in processes that allow a sufficiently short time for removal of water or cooling of concentrated solids. Deep-fat frying is considered to be the one process in which water is rapidly removed by heating in hot oil. The phase transition of starch and gluten controls the development of structure during



**Figure 7.** Fracture force of fried sample containing (a) 400 g kg<sup>-1</sup> and (b) 600 g kg<sup>-1</sup> moisture at various frying times.



**Figure 8.** Pathway of the frying process state diagram of fried sample containing  $400 \text{ g kg}^{-1}$  initial moisture.

processing and product quality during storage. The first step in achieving the proper conditions is to establish a state diagram to explain physicochemical changes in the pathway of frying for samples containing 400 and 600 g kg<sup>-1</sup> moisture, as shown in Figs 8 and 9, respectively. The pathway in this state diagram was established from the moisture content and the center temperature of the fried sample at various frying times on the wheat flour state diagram. It was evident that the rapid decrease in moisture content due to water evaporation induced samples at both moisture levels to transition to the glassy state by the end of frying.

The consistency of the 400 g kg<sup>-1</sup> sample was like that of dough, which was induced by the continuous gluten network filling out with starch granules, and was in the rubbery state in the initial stage (before frying). The obtained results confirmed that this level of moisture was insufficient for gelatinization. Therefore, gluten was considered to be the dominant factor in the structure formation. Le Meste *et al.*<sup>25</sup> reported that the  $T_g$  value of white pan bread, assessed by thermomechanical analysis (TMA), was similar to that of gluten. This suggests that gluten governs structural formation in a dough. Once this sample was fried, the temperature of the sample was higher than 100 °C. As shown in Fig. 5(a), many small pores



Figure 9. Pathway of the frying process state diagram of fried sample containing  $600 \text{ g kg}^{-1}$  initial moisture.

were observed in SEM photographs of the  $400 \,\mathrm{g \ kg^{-1}}$ sample at 1 min of frying time. The porous structure became larger in the 3 min fried sample, and then the size of pores remained constant as the frying time was prolonged. The morphology of the porous structure in the samples that were fried from 3 to 7 min was nearly the same. From the frying pathway, the sample at 3 min almost went to the glassy state. However, the alteration of porous structure in the  $400 \text{ g kg}^{-1}$  sample stopped before it reached the  $T_{\rm g}$  of gluten. Generally, gluten, which governs the formation of the final structure in dough, is sensitive to changes in moisture and temperature.<sup>26</sup> Kokini et al.<sup>9</sup> reported that, on heating to above the glass transition, gliadin and glutenins become entangled polymers at about 70-135°C by disulfide crosslinkage, and then become softened when heated to over 135°C. This temperature was independent of moisture content in the moisture range 200-400 g  $kg^{-1}$ .<sup>9</sup> Therefore, the decrease in molecular mobility due to entanglement and the concomitant dramatic decrease in residue moisture content, which results from rapid evaporation during frying, brought about no changes in the porous structure even though the sample existed at a temperature above  $T_g$  (after 3 min of frying time) in this sample.

The 600 g kg<sup>-1</sup> sample, which is regarded as batter, was also in the rubbery state initially. During heating for 1 min in hot oil, the amount of water in this sample was sufficient for it to go through gelatinization (Fig. 9). The porous structure in this sample is generated by vapor evaporation from the starch gel, which can trap the evaporated water temporarily, resulting in continuous development in both size and quantity of the cellular structure. This took place until the moisture content of the fried sample decreased and the samples reached the glass state (at 5 min of frying). The expansion of porous structure ceased when the system existed below  $T_{\rm g}$  because of the limitation of molecular mobility.<sup>5</sup> These results coincide with the expansion of porous structure in extruded products and baked bread.<sup>6,10</sup>



Figure 10. Pathway of fried sample containing  $400 \text{ g kg}^{-1}$  initial moisture of the texture assessment.



**Figure 11.** Pathway of fried sample containing  $600 \text{ g kg}^{-1}$  initial moisture of the texture assessment.

#### State diagram for texture assessment

Glass transition is considered to correspond to crispness in low-moisture snack food.<sup>27</sup> The loss of crispness due to the water acting as a plasticizer during storage leads to a decrease of palatability in this product. State diagrams, as shown in Figs 10 and 11, were constructed from the moisture content of the fried sample at room temperature on the wheat flour state diagram. The texture profile of  $400 \,\mathrm{g \ kg^{-1}}$ initial moisture content (Figs 6a and 7) revealed that no fracture peak developed in 1 min of frying even though the moisture content of this sample at room temperature existed in the glassy state (Fig. 10). The jagged line in the texture profile began to appear in the 3 min fried sample. In the  $600 \text{ g kg}^{-1}$  sample, at 1 min of frying the sample was still in the rubbery state (Fig. 11); no fracture peak appears in the texture profile (Figs 6b and 7). In the 3 min fried sample, the residual moisture content in this sample reached the glassy state but slight fracture was observed. The jagged line in the texture profile started to appear in the 5 min fried sample. The reduction of elasticity probably results from the stiffening of thin-walled air cells with the longer frying time.

The glassy state of the structural elements of gel networks of proteins and polysaccharides corresponds to a high concentration of chain segment interactions.<sup>28</sup> The fried sample in the first stage still possesses the high moisture content that it had in the rubbery state. In this state, the sample had a leathery behavior; only the rupture peak appeared in the texture profile. As the frying time was extended, the moisture within the sample decreased and the sample consequently went through the glassy state even though there was a difference in initial moisture content. The samples in the final state behaved like glass; they were brittle and cracked easily. The faster they moved into the glassy state, the more rigid their structure. However, the moisture content of the  $400 \,\mathrm{g \ kg^{-1}}$  sample fried for 1 min and the  $600 \text{ g kg}^{-1}$  sample fried for 3 min existed in the glassy state, but no brittleness was observed. Castro et al.29 reported that the glass transition temperature itself is not a good predictor of mechanical properties in the amorphous starch system. The water content in the samples probably also influences the textural characteristics of fried food. The obtained results may be due to water and absorbed oil (in the fried sample), which is considered as the plasticizer for food biopolymers, so that the effect of plasticization can probably influence the crispness in fried food. The presence of a small amount of water in the crisp food resulted in a higher requirement for force to break the intermolecular bonding of starch, forming small crystalline-like regions.<sup>30</sup> Further research will be needed to elucidate the relationship between glass transition and crispness in fried food systems.

# CONCLUSIONS

Physicochemical changes during frying influence the alteration of microstructure and quality of fried food. From the results obtained it was evident that the morphology of the porous structure differed at the two moisture levels studied. In the 400 g kg<sup>-1</sup> initial moisture content sample, a small-pore structure was formed. On the other hand, larger pores were observed in the  $600 \text{ g kg}^{-1}$  sample. The alteration in cellular structure in these two samples is considered to be governed by the arrangement of gluten during heating, or starch gelatinization. A state diagram can be applied to explain both the formation of microstructure during the frying process and the textural characteristics of the fried samples. This state diagram is a tool for understanding microstructure formation during the frying process.  $T_{\rm g}$  values help to describe the porosity development that gives good textural characteristics of fried food. However,  $T_g$  depression alone could not clearly explain the brittleness in the fried sample. Plasticization is also involved. A residual moisture content of less than  $50 \text{ g kg}^{-1}$  in the fried sample provided good textural characteristics in the present study.

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