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Reduction of Acrylamide Level in French Fries by Employing a Temperature Program during Frying

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In this study, the effect of employing an oil temperature program during frying on the acrylamide content of French fries was investigated. The frying conditions that could lead to lower acrylamide levels in French fries were first simulated by means of an experimentally validated frying model. Then, experiments were conducted to test the simulated conditions in real frying process. Different time/temperature combinations (4 min at 170 °C, 2 min at 170 °C + 2 min at 150 °C, 1 min at 170 °C + 3 min at 150 °C, 1 min at 190 °C + 3 min at 150 °C) were employed for frying potato strips (8.5 × 8.5 × 70 mm), and the resultant acrylamide levels were determined with a gas chromatography−mass spectrometry (GC-MS) method. The results indicated that acrylamide levels in French fries can be reduced by half if the final stage of the frying process employs a lower oil temperature. Therefore, the method appears to be an effective way of controlling the acrylamide level in the final product.

KEYWORDS: French fries; acrylamide; oil temperature program; frying model

INTRODUCTION

Since its discovery in certain fried, baked, and roasted foods, acrylamide (a potential human carcinogen) and especially the limitation of its formation have been of great interest to researchers. Different approaches, including the addition of acid (*1–3*), enzyme (asparaginase) treatment (*4–6*), the addition of glycine (*2, 3, 7–9*), lactic acid fermentation (*10*), blanching (*11*), low-temperature vacuum frying (*12*), and recently the addition of calcium (*13*), have been tested as a potential way to reduce the levels of acrylamide in these foods. However, it remains a challenge to reduce acrylamide levels in the final product without sacrificing its quality.

The simultaneous heat and mass transfers leading to high-temperature and low-moisture conditions during these processes give rise to the formation of acrylamide as a result of the interaction between asparagine and reducing sugars, known as the Maillard reaction. Temperature and time have been repeatedly shown to be significant factors, along with the reducing sugar level of potato cultivar, affecting the amount of acrylamide formed in potatoes during frying (*14–20*). Gertz and Klostermann reported that acrylamide formation was accelerated at temperatures above 175 °C (*21*). Similarly, in its Proposed Draft Code of Practice for the Reduction of Acrylamide in Food, the FAO states that significant reductions in the acrylamide content of French fries can be achieved by frying at temperatures no higher than 175 °C (*22*).

Very little acrylamide was shown to form in French fries upon frying at 150 °C (*23*). Other researchers also reported significant reductions in acrylamide formation by lowering the oil temperature. Haase et al. achieved 50% reduction in acrylamide content of potato chips by lowering the oil temperature from 185 to 165 °C (*15*). Pedreschi et al. also reported 68 and 88% reductions in acrylamide content with a decrease in temperature from 190 to 170 and 150 °C, respectively (*11*). However, high drying rates observed during frying at higher temperatures were also reported to favor textural properties and to reduce the oil content of the final product due to faster development of the crust layer (*24*). This brings to mind that French fries of low acrylamide content and acceptable quality can be obtained if the frying process is composed of a high-temperature initial stage followed by a lower temperature final stage.

The results in the literature indicate that acrylamide is primarily formed toward the end of the frying process, when a steep increase in its concentration takes place, suggesting that acrylamide formation can be minimized by controlling the time and temperature of frying. Grob et al. suggested stopping the frying process before this steep increase (*18*). A better control of the process in terms of the acrylamide level and quality of the final product, however, can be achieved by applying an oil temperature program during frying. Amrein et al. suggested that lowering the oil temperature toward the end of frying may help reduce the acrylamide level in the final product while maintaining acceptable color formation (*19*). This makes a lot of sense because the conditions become more favorable (high temperature−low moisture) for acrylamide formation as the frying proceeds. A sudden drop in oil temperature during frying realized by transferring the potato strips from a high-temperature oil bath to a lower temperature one may prevent the surface

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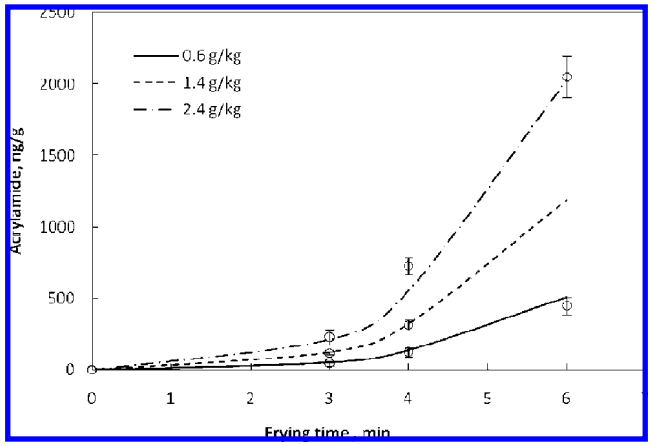

Figure 1. Effect of initial total reducing sugar content and frying time on acrylamide level in French fries. Both model (lines) and experimental (markers) results are shown (frying temperature = 170 °C).

from reaching excessively high temperatures, thereby limiting acrylamide formation. In this study, this hypothesis was tested by employing a temperature program during frying. To do this, different frying scenarios that can potentially yield lower levels of acrylamide in French fries were first simulated using an experimentally validated frying model, and then experiments were performed under the same sets of conditions to test the hypothesis in the real process.

MATERIALS AND METHODS

Chemicals and Consumables. Acrylamide (99+%) and $^{13}C_3$-labeled acrylamide (99% isotopic purity) were obtained from Sigma (Diesenhofen, Germany) and Cambridge Isotope Laboratories (Andover, MA), respectively. Methanol, hexane, potassium hexacyanoferrate, zinc sulfate, formic acid (98%), sodium thiosulfate pentahydrate, hydrobromic acid, potassium bromide, and bromine (99.8%) were of analytical grade and obtained from Merck (Darmstadt, Germany). Sodium thiosulfate pentahydrate, hydrobromic acid, potassium bromide, and bromine (99.8%) 9-fluorenylmethylchloroformate (FMOC) were also purchased from Merck. HPLC gradient grade acetonitrile was obtained from J. T. Baker (Deventer, The Netherlands). Ultrapure water was used throughout the experiments (MilliQ system, Millipore, Bedford, MA). The InnoWax (30 m × 0.25 mm, 0.15 μm film thickness) and Zorbax C_8 (150 × 4.6 mm) columns were purchased from Agilent Technologies (Palo Alto, CA). A Shodex Sugar SH-1011 (300 × 8.0 mm, 7 μm) column was supplied by Waters (Milford, MA). Microspin PVDF centrifuge filters (0.45 μm) were purchased from Alltech (Deerfield, IL).

Stock solutions of acrylamide (1 mg/mL) and $^{13}C_3$-labeled acrylamide (0.1 mg/mL) were prepared by dissolving in methanol. Carrez I solution was prepared by dissolving 15 g of potassium hexacyanoferrate in 100 mL of water and Carrez II solution by dissolving 30 g of zinc sulfate in 100 mL of water.

Frying of Potato Slices. Potatoes (Agria) used for frying experiments were purchased from local markets. Reducing sugars and asparagine contents were determined by high-performance liquid chromatography (HPLC). Potatoes were cut into strips (8.5 × 8.5 mm) using a French fry cutter, and the strip length was adjusted to 70 mm. Frying of potato strips was performed in sunflower oil by completely immersing the potato strip in the hot oil using a stainless steel wire assembly. Experiments were conducted employing different frying time−temperature combinations (4 min at 170 °C, 2 min at 170 °C + 2 min at 150 °C, 1 min at 170 °C + 3 min at 150 °C, 1 min at 190 °C + 3 min at 150 °C). The initial stage of frying (frying at 170 and 190 °C) was performed using a 5 L oil bath (Precisterm, J. P. Selecta, Spain), and the potato strip was quickly transferred into a 1 L oil bath comprising oil in a beaker on a temperature-controlled hot plate (MST basic, IKA-Werke, Staufen, Germany), where the final stage of frying (frying at 150 °C) was carried out. The temperatures of the oil baths were monitored during

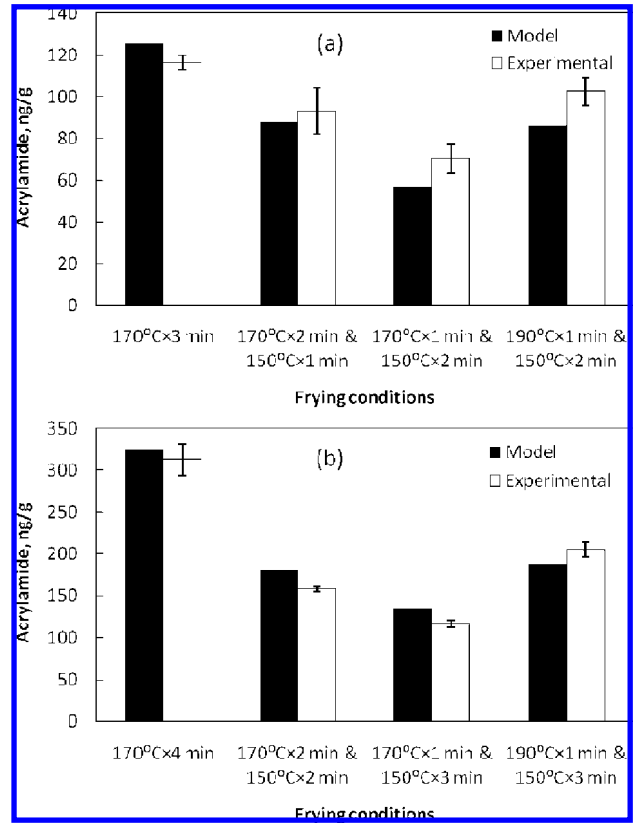

Figure 2. Acrylamide levels in French fries resulted from employing different temperature programs during frying. Total frying times: (**a**) 3 min; (**b**) 4 min (initial total reducing sugar content = 1.4 ± 0.2 g/kg, initial free asparagine content = 7.2 ± 0.6 g/kg).

frying. No changes in the oil temperatures were observed, because only one potato strip was fried at a time. All frying experiments were conducted in duplicate. Fried and control samples were analyzed for acrylamide content using a GC-MS method.

Model Development. A simultaneous heat and mass transfer model developed using an explicit finite difference scheme was used to simulate frying of potato strips. Heat conduction and mass diffusion equations in Cartesian coordinates (eqs 1 and 2) were numerically modeled with the appropriate initial (uniform initial temperature and moisture distribution) and boundary (convective boundary condition at the surface) conditions.

$$\frac{\partial^2 T}{\partial x^2}+\frac{\partial^2 T}{\partial y^2}+\frac{\partial^2 T}{\partial z^2}=\frac{1}{\alpha}\frac{\partial T}{\partial t} \quad (1)$$

$$\frac{\partial^2 C}{\partial x^2}+\frac{\partial^2 C}{\partial y^2}+\frac{\partial^2 C}{\partial z^2}=\frac{1}{D}\frac{\partial C}{\partial t} \quad (2)$$

The term $\lambda/k \times \partial\rho/\partial t$ (λ is the latent heat of vaporization of water at 103 °C, 2249 kJ/kg, and ρ is the moisture concentration, kg/m^3) was added to the left side of eq 1 to account for energy used to vaporize water at temperatures above 103 °C (boiling point of water in potato).

Modeling was performed for only for one-eighth of the potato strip, due to the symmetry of boundary conditions. A total of 40 nodes were used along the half-length of the strip (z-direction), whereas 20 nodes were used along the half-thickness (x- and y-directions). Volume change during frying was neglected, and constant thermophysical properties were assumed.

Heat and mass transfer parameters (heat transfer coefficient, mass transfer coefficient, and moisture diffusivity) used in the model were taken from elsewhere (*25*). These parameters were determined during frying of potato strips (8.5 × 8.5 × 70 mm) in sunflower oil at 150, 170, and 190 °C.

Kinetics of Acrylamide Formation/Degradation. The rate constants for acrylamide formation and degradation obtained by Gökmen and

Senyuva with a fructose−asparagine model system within the temperature range of 120−200 °C were used in the model (*23*). It has been reported that glucose and fructose had different kinetic behaviors (*26*). According to the multiresponse kinetic model suggested by these researchers, glucose is converted to an intermediate in a two-stage process that further undergoes to acrylamide, whereas fructose is converted to the same intermediate in a one-stage process. According to the simplified kinetic model by Gökmen and Senyuva, all reducing sugars are assumed to behave as a ketose sugar and the reaction proceeds as

$$A + B \xrightarrow{k_1} C \xrightarrow{k_2} D$$

where A, B, C, and D denote the reducing sugar, asparagine, acrylamide, and unknown degradation products, respectively (*23*). Because acrylamide formation was first order with respect to fructose and zeroth order with respect to asparagine, the above reaction can be reduced to the form

$$A \xrightarrow{k'_1} C \xrightarrow{k_2} D$$

where $k'_1 = k_1 C_B$. Now, the change in acrylamide and sugar concentrations can be given respectively by eqs 3 and 4:

$$\frac{dC_C}{dt} = k'_1 CA - k_2 C_C \quad (3)$$

$$\frac{dC_A}{dt} = -k'_1 C_A \quad (4)$$

Acrylamide and sugar concentrations after some incremental time step (Δt) were approximated by eqs 5 and 6, respectively:

$$[C_C]^{n+1} \approx \Delta t(k'_1[C_A]^n - k_2[C_C]^n) + [C_C]^n \quad (5)$$

$$[C_A]^{n+1} \approx \Delta t(-k'_1[C_A]^n) + [C_A]^n \quad (6)$$

Acrylamide accumulation and sugar depletion in any node were triggered if the temperature of that node was ≥120 °C. The model assumes a uniform initial sugar distribution within the potato strip. Sugar depletion and acrylamide accumulation in any given node were assumed to result only from the reactions given above (no transfer of sugar or acrylamide between nodes occurs).

Initial sugar content (C_{A_0}) used in the kinetic study by Gökmen and Senyuva was 5 μmol, and the temperature dependence of reaction rate constants for the formation and degradation of acrylamide was (*23*)

$$k'_1 = \exp[15.438 - 11284/T] \quad (7)$$

$$k_2 = \exp[7.4728 - 5950/T] \quad (8)$$

where T is the absolute temperature (Kelvin).

Measurement of Acrylamide by GC-MS Analysis. Acrylamide was analyzed as its brominated derivative by GC-MS using the method of Castle et al. with some modifications (*27*). The extracting medium was methanol, rather than water, because addition of water to the homogenized potato sample resulted in a thick slurry rendering extraction difficult (*28*). Two grams of homogenized sample was extracted with 2 × 20 mL of methanol. [$^{13}C_3$]Acrylamide (500 ng) was added to the sample as the internal standard, along with 15 mL of brominating reagent. The bromination was allowed to proceed overnight at room temperature (*29*).

The brominated extract (1 μL) was injected onto an Agilent 5973 GC-MS system (Agilent Technologies, Palo Alto, CA) in splitless mode at 200 °C. Helium carrier gas flow rate was maintained at 1 mL/min. An InnoWax capillary column was used for chromatographic separation. The oven temperature was 80 °C, rising at 10 °C/min to 250 °C and holding for 10 min. The transfer line was held at 250 °C and the ion source at 180 °C. Electron impact mass spectra were obtained at 70 eV. The mass spectrometer was operated in selected ion monitoring mode. Four ions were used to characterize brominated [$^{13}C_3$]acrylamide (*m*/*z* 108, 110, 153, and 155), and another four ions were used to characterize brominated acrylamide (*m*/*z* 106, 108, 150, and 152). The ion *m*/*z* 152 was used to quantify brominated acrylamide.

A stock solution of acrylamide at a concentration of 1.0 mg/mL was prepared in methanol. Working standards were prepared daily by diluting the stock solution to concentrations of 0.01, 0.02, 0.05, 0.10, 0.25, 0.50, and 1.0 μg/mL with methanol. They were brominated prior to GC-MS analysis using the procedure described above. Signal response was linear over a concentration range between 0.01 and 1.0 μg/mL. The limit of detection (LOD) and the limit of quantitation (LOQ) for acrylamide were 15 and 50 ng/g in French fries, respectively. The coefficient of variation was 10% or lower for three repetitive measurements of acrylamide in French fries.

Measurement of Reducing Sugars. Reducing sugars were analyzed by HPLC using the method described elsewhere with minor modifications (*30*). Five grams of potato was weighed into a 30 mL capped tube. A total of 23 mL of hot water at 70 °C, 1 mL of Carrez 1, and 1 mL of Carrez 2 solutions were added. Sugars were extracted by mixing the tube for 3 min using a homogenizer. After the tubes had stood for 3 min, the upper aqueous extract was transferred to a test tube. Remaining solid residue was re-extracted with 25 mL of hot water at 70 °C. Both extracts were combined and centrifuged for 10 min at 10000 rpm using microspin centrifuge filters (0.45 μm). The clear extract was injected onto an Agilent 1100 HPLC system (Waldbronn, Germany) consisting of a quaternary pump, a Rheodyne 7125 injector, a refractive index detector, and a temperature-controlled column oven. Chromatographic separations were performed on a Shodex Sugar SH-1011 column using 0.01 mM H_2SO_4 solution at a flow rate of 1.0 mL/min at 25 °C. Each sample was analyzed in duplicate, and the means of two measurements were reported. The LOD was determined to be 0.01 g/100 g of potato for glucose.

Measurement of Asparagines. Free asparagine was analyzed by HPLC using the method described elsewhere with minor modifications (*31*). Five grams of potato was homogenized with 50 mL of water for 3 min. Following centrifugation (10000 rpm × 10 min, 4 °C), clear supernatant was diluted with water to an approximate asparagine concentration of 10−50 mg/kg. Asparagine was converted to its FMOC derivative by mixing 0.1 mL of the extract with 0.6 mL of 30 mM FMOC reagent and 0.5 mL of pH 10.0 buffer for 2 min in a test tube. Excess of FMOC reagent was removed after derivatization by extraction with 2.0 mL of *n*-hexane. Twenty microliters of final test sample was injected into the HPLC system consisting of an Agilent 1100 quaternary pump, a Rheodyne 7125 injector, and a fluorescence detector set at 265 nm/340 nm. Chromatographic separation was performed on a Zorbax C_8 column using a gradient mixture of acetonitrile (A) and 50 mM, pH 4.2, aqueous acetate buffer (B) at a flow rate of 1.0 mL/min at 25 °C. Acetonitrile ratio was increased from 0 to 25% within 10 min and to 100% within 5 min. Each sample was analyzed in duplicate, and the means of two measurements were reported. The LOD was determined to be 0.1 mg/100 g of potato for asparagine.

RESULTS AND DISCUSSION

In this study, different frying scenarios were first evaluated using a frying model for their effect on reducing acrylamide level in French fries. Experimental work was then carried out to test these simulated conditions in real process. As the frying proceeds the surface of the strip reaches high temperatures as a result of simultaneous drying, promoting the Maillard reaction in this region. This is the reason that acrylamide formation is mainly a surface phenomenon. However, if the exposure of the strip surface to high temperatures (≥170 °C) is limited, excessive accumulation of acrylamide in potato can be prevented during frying. Therefore, the particular frying temperature and time combinations used were selected to see the effect of initial high oil temperature frying (170 or 190 °C) for shorter time (1 or 2 min) followed by relatively lower temperature frying (150 °C) for relatively longer time (2 or 3 min) on the accumulation of acrylamide in potato strips.

Figure 1 shows both model and experimental results for acrylamide formation in potato strips of different reducing sugar contents during frying at 170 °C. It is clear from these results that the effect of frying time on acrylamide formation is more pronounced for higher sugar contents. Although a reducing sugar content of 0.6 g/kg of fresh potato is rather low and one of 2.4 g/kg is moderately high, 1.4 g/kg can be considered a typical sugar content that may be encountered in real applications, which is also not too far from what is recommended by Biedermann-Brem et al. as an upper limit (1 g/kg) for potatoes destined for frying (*32*). It can also be seen from the figure that acrylamide concentration in potato strips tends to increase exponentially with frying time. No acrylamide formation is observed in the beginning of the frying process, because the conditions are not yet favorable. However, a steep increase in acrylamide concentration is observed as a result of high-temperature and low-moisture conditions that develop in the surface region as frying proceeds.

Figure 2 shows the effects of the different frying scenarios on acrylamide levels of French fries. It is clear from this figure that model and experimental acrylamide results compare well. Free asparagine and reducing sugars have been reported to be key participants in acrylamide formation (*33–35*). The potato tubers used in this study were first analyzed for these acrylamide precursors. Total reducing sugar content (sum of fructose and glucose) of the tubers was found to be 1.4 ± 0.2 g/kg, whereas asparagine was 7.2 ± 0.6 g/kg. It was simply considered from these results that asparagine is not a limiting factor, and the rate of acrylamide formation is mainly controlled by reducing sugars during frying. As also previously confirmed by others, free asparagine is the highest in potatoes among other free amino acids measured (*2, 12, 36–38*).

When compared to control treatments (170 °C × 3 min or 170 °C × 4 min), the temperature-programmed frying processes resulted in significant reductions in acrylamide concentrations. The effect was clearer for the total frying time of 4 min than for 3 min for all frying combinations tested. By employing a process starting with frying at 170 °C for 1 min and followed by frying at 150 °C for 3 min, acrylamide concentration of French fries can be reduced by 58%.

According to the industry, the residual moisture for an ideal product should be in a range between 38 and 45% (*20*). On the basis of our previous experience, the moisture content of potato strips decreases to this range within 3 and 6 min of frying at 170 and 150 °C, respectively (*17*). Hence, the total frying time of 4 min for temperature-programmed frying experiments used in this study was expected to meet this criterion related to final moisture content of French fries.

Simulation of the programmed temperature frying on the acrylamide content of French fries yielded results similar to those of the experiments. Of course, the effect of employing such a temperature program during frying on the quality characteristics should be evaluated. Results pertaining to the quality of French fries can be obtained once the kinetics of texture and color formation is taken into account in the model. Nevertheless, the frying model was shown to be an extremely useful tool for testing the effect of such frying scenarios on the acrylamide level in French fries before a decision is made as to whether the method being considered is viable and worth pursuing. In this way considerable time, money, and effort can be saved during process design and optimization.

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