

Influence of Thermal Processing Conditions on Acrylamide Generation and Browning in a Potato Model System

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Fried potato products such as French fries and chips may contain substantial amounts of acrylamide. Numerous efforts are undertaken to minimize the acrylamide content of these products while sensory properties such as color and flavor have to be respected as well. An optimization of the frying process can be achieved if the basic kinetic data of the browning and acrylamide formation are known. Therefore, heating experiments with potato powder were performed under controlled conditions (moisture, temperature, and time). Browning and acrylamide content both increased with heating time at all temperatures and moisture contents tested. The moisture content had a strong influence on the activation energy of browning and acrylamide formation. The activation energy strongly increased at moisture contents below 20%. At higher moisture contents, it was very similar for both parameters. At low moisture contents, the activation energy of acrylamide formation was larger as compared to the one for browning. This explains why the end of the frying process is very critical. Therefore, a lower temperature toward the end of frying reduces the acrylamide content of the product while color development is still good.

KEYWORDS: Acrylamide; browning; activation energy; moisture content; potato

INTRODUCTION

Fried potato products such as chips (United Kingdom, crisps) and French fries are very popular and consumed in large amounts worldwide (1). During the frying of raw potatoes, significant changes within the potato matrix occur including reduction of water content, uptake of oil, starch gelatinization, formation of a crispy crust, and generation of color and flavor (2, 3). The detection of acrylamide in heated food revealed that fried and baked potato products may contain up to a few milligrams of acrylamide per kilogram (4). Because acrylamide is probably carcinogenic to humans (5) and chips and French fries are very popular, numerous studies were performed to lower the acrylamide content in these products. Although free asparagine delivers the backbone of the acrylamide molecule (6), the content of glucose and fructose in the raw material was found to determine the acrylamide formation in heated potatoes (7–9). On the process side, the frying temperature and time as well as the shape of the product were identified as key factors for acrylamide formation (10–12). During heating, the acrylamide formation occurs in parallel to the drying of the matrix (13–15) and a low moisture content may favor the acrylamide formation (13, 15, 16). The culinary quality of the product is determined by the color, flavor, and crispness and is primarily influenced by the frying process and the composition of the raw material. The “acrylamide toolbox” set up by the CIAA

comprises several mitigation options for potato products (17). The Maillard reaction is responsible for the development of color and flavor (18) and the formation of acrylamide (19); thus, these factors are related. To prepare products of desired quality with low acrylamide content, process conditions should allow for sufficient browning without enhancing acrylamide formation.

To optimize process conditions, kinetic data on the acrylamide formation and browning, obtained under well-controlled conditions, are needed. Some kinetic data on acrylamide formation are available for model systems (20, 21) and for fried potatoes (22, 23). Likewise, kinetic data on nonenzymatic browning are available for various foods, including potatoes and food model systems (24–27). It is important to note that the browning rate at constant temperature is influenced by the water content of the system, and at the same time, the water content also influences the activation energy of the browning reaction. In consequence, for modeling browning and acrylamide formation in processes such as deep fat frying in which product temperature and water content continuously change over time, reaction rates for each temperature/moisture combination of the process must be known.

In the present investigation, freeze-dried potato powder was prepared, adjusted to different water contents, and then heated in a hermetically sealed reactor at different temperatures for varying lengths of time. The extent of browning and the formation of acrylamide in these samples were determined, and the rates and activation energies thereof were derived. In view of the aim to apply these data in process optimization for

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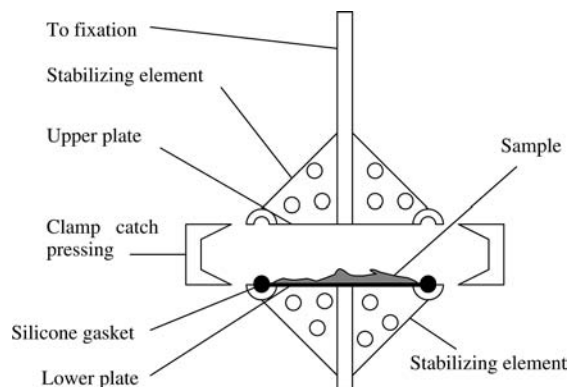


Figure 1. Scheme of the reactor used for the heating experiments.

mitigation of acrylamide, overall reaction rates rather than detailed mechanisms of Maillard reactions and acrylamide formation were taken into account. The obtained data present the kinetic basis for the optimization of potato frying in terms of acrylamide and color.

MATERIALS AND METHODS

Preparation of Potato Powder. Potato powder was prepared from a lot of fresh potatoes from the 2003 harvest (cultivar Agria), which has been characterized in a previous study (9). The potatoes were washed, peeled, and cut into cubes of 10 mm edge length. The cubes were blanched at 85 °C for 3 min and frozen at -24 °C overnight. After they were freeze dried (Secfroid, Lausanne, Switzerland) for 72 h, the material was ground to a particle size of ≤ 0.5 mm (Mu4 Ultrazentrifuge B13, Retsch, Arlesheim, Switzerland). The residual moisture was determined with an infrared dryer (LP16, Mettler, Greifensee, Switzerland), and the powder was then stored in airtight plastic bags. The water contents of the samples were adjusted by adding deionized water onto the potato flour in a mixer (Küchenblitz, Betty Bossy, Zürich, Switzerland) to obtain a homogeneous distribution of the water: 5.0, 8.7, 11.7, 17.6, 21.7, 40.6, and 60.7 g of water per 100 g wet weight. The samples were packed into airtight plastic bags, stored at room temperature for 3 days to allow for equilibration of the added water within the potato powder, and then stored in a refrigerator at 4 °C until use. Samples with a high water content were not stored but heated within 24 h in order to prevent spoilage.

Heating Experiments and Kinetic Modeling. The heating experiments were performed in a hermetically sealable stainless steel reactor, which had been designed and built by Ufheil (28) for fast heating and cooling cycles without loss of moisture by water evaporation (Figure 1). Measurement of temperature within the reactor showed that the temperature reached the oil temperature after 30–40 s, which was virtually independent of the sample moisture (28). The reactor was tightly closed by a silicone gasket (surrounding the sample; diameter, 100 mm; thickness, 1.6 mm), which was fixed by a clamp catch pressing the two plates (1 mm thickness) together. Five grams of potato powder was spread evenly on the lower plate and then sealed in the reactor. The reactor was immersed into a preheated oil bath (containing 15 L of sunflower oil) equipped with a stirrer, temperature controller, and heater (thermostat). The samples were heated at 119, 143, and 167 °C for 1–40 min, which reflects temperature–time combinations applied during the frying of potatoes (2). The powder was heated for three different lengths of time at every temperature. After the powder was heated, the reactor was immediately cooled in an ice–water bath in order to stop further reactions.

For the kinetic modeling, pseudo zero-order and pseudo first-order reactions were assumed. As a first approximation, the temperature within the reactor was presumed to be equal to the oil temperature after the immersion. The reaction rate for browning and acrylamide formation was determined by reading the slope of a trend line fitted to the data sets (see slopes in Figure 2 as examples). The obtained reaction rates were plotted as natural logarithm against inverse temperature (1/

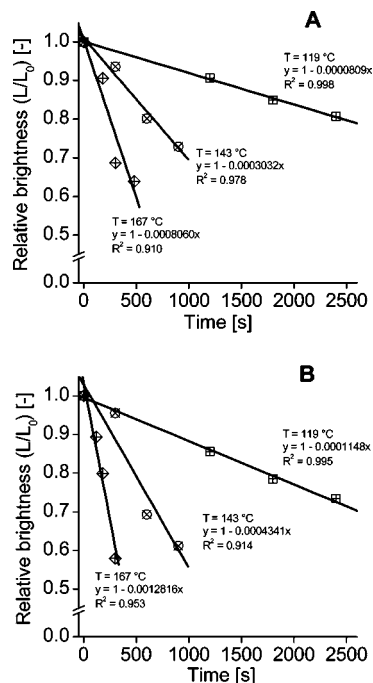


Figure 2. Relative brightness (L/L_0) determined in heating experiments at three different temperatures and for two different moisture contents: (A) 11.7 g moisture/100 g and (B) 21.7 g moisture/100 g.

K), and out of this plot, the activation energy was obtained by using the Arrhenius equation.

Analytical Procedures. The browning was determined after heating using an optical L, a, b analyzer (Chroma-Meter CR 310, Minolta, Dietikon, Switzerland). Each sample was measured in triplicate. The relative brightness (L/L_0) was calculated from the ratio of the L value of a heated sample to the L value of the unheated sample and used as a measure for the extent of browning. Analogous calculations were made with the a value (redness). Kinetic calculations were also done with absolute L and a values.

Glucose and fructose were determined with an enzymatic kit (Scil, Martinsried, Germany) as described previously (9). Free amino acids were determined by cation-exchange chromatography followed by postcolumn derivatization with ninhydrin using norleucine (Fluka, Buchs, Switzerland) as an internal standard (2500 mg/kg). Extraction, cleanup, separation, and quantification were performed as published earlier (9, 14). Acrylamide was determined with the gas chromatography–mass spectrometry (GC-MS) method described by Biedermann et al. (29) using $^{13}\text{C}_3$ -acrylamide (CIL, Andover, United States) and methacrylamide (Fluka) as internal standards (both added at 500 $\mu\text{g}/\text{kg}$). GC-MS involved an 8000 series gas chromatograph (Fisons Instruments, Milan, Italy) with on-column injector coupled to an SSQ 710 quadrupole mass spectrometer (Finnigan MAT, San Jose, United States). The precolumn (TSP deactivated, i.d. = 0.53 mm) and the separation column (BGB-Wax, 12 m, i.d. = 0.25 mm) were both from BGB Analytik (Böckten, Switzerland). GC-MS conditions were as described earlier (14, 29). All data shown refer to wet weight. The limit of quantification was determined to be 25 $\mu\text{g}/\text{kg}$.

RESULTS AND DISCUSSION

Characterization of the Potato Powder and the Heating Process. The potato powder was analyzed for reducing sugars and free amino acids (Table 1). The water loss during freeze drying amounted to 78 g per 100 g of blanched potatoes, which corresponded well with the dry matter content determined in the previous study (9). Spiking experiments with asparagine and glucose (1000 mg/kg) gave recoveries of 99 and 104%, respectively. The relative standard deviations for multiple analyses ($n \geq 3$) were <5% for amino acids and <15% for

Table 1. Content of Reducing Sugars ($n = 2$) and Free Amino Acids ($n = 4$) in the Unheated Potato Powder^a

| compound | concentration (mg/kg) |
|------------------------|-----------------------|
| glucose | 210 |
| fructose | 90 |
| free asparagine | 9330 |
| total free amino acids | 21170 |

^aData refer to the powder as is (residual moisture, 3.8 g/100 g). Relative standard deviations between analyses amounted to <15% (reducing sugars) and <5% (free amino acids).

Table 2. Repeatability of the Heating Process under Various Conditions ($n = 4$)^a

| moisture content (g/100 g) | heating condition | acrylamide | | <i>L</i> value | |
|----------------------------|-------------------|---------------------------|---------|----------------|---------|
| | | mean ($\mu\text{g/kg}$) | RSD (%) | mean (–) | RSD (%) |
| 8.7 | 143 °C, 15 min | 3450 | 3.5 | 72.9 | 0.9 |
| 11.7 | 167 °C, 8 min | 11630 | 1.9 | 57.7 | 0.4 |
| 21.7 | 119 °C, 30 min | 830 | 16.4 | 69.8 | 3.4 |

^aRSD, relative standard deviation. Values refer to wet weight.

sugars. Referring the data in **Table 1** to fresh weight showed that 77% of the reducing sugars and 19% of the free asparagine were lost during blanching. Losses of reducing sugars may also have occurred during storage of the fresh potatoes since the potato powder was prepared some weeks after the analyses from the cited study and the content of reducing sugars decreased during storage (9). In addition, the potatoes used were from the 2003 harvest (hot and dry season) and thus contained more reducing sugars, which were partly degraded during storage (9). Thus, the used model system corresponds to a potato with a low content of reducing sugars, which is desired in the production of chips and French fries. Asparagine was the major free amino acid accounting for 44% of the pool of free amino acids, which was also reported in other studies (7, 9). Asparagine, glutamine, glutamic acid, and aspartic acid represented about three-quarters of the total free amino acids.

The repeatability of the heating process was checked by performing experiments at three different heating conditions with four separate samples at each condition. The results in **Table 2** demonstrate that the process was well-reproduced in terms of acrylamide content and browning (*L* value). Therefore, only one sample was heated for one particular process condition for the kinetic modeling. The considerably larger variation in the acrylamide content for the heating at 119 °C for 30 min might be due to inhomogeneities caused by the increased moisture content or due to the eventual elimination of acrylamide at long process times (13).

Temperatures of the oil bath were stable at the three temperatures tested. The maximum deviation was recorded during an experiment at 167 °C and amounted to 0.5% (relative standard deviation). Normally, the variation was <0.2% for all temperatures. The temperatures and the water contents used for these measurements were equivalent to the conditions in the outer layer, i.e., the crust of French-fried potatoes (2, 30), which explains the relatively high acrylamide contents.

Kinetic Data of Browning. To characterize the kinetics of browning, heating experiments at 119, 143, and 167 °C were performed for seven different moisture contents ranging from 5.0 to 60.7 g per 100 g wet weight. The browning was

Table 3. Browning Rates (s^{-1}) According to the Relative Brightness (L/L_0) Obtained at the Different Process Conditions by Assuming a Reaction of Pseudo Zero-Order

| moisture content (g/100 g) | 119 °C | 143 °C | 167 °C |
|----------------------------|------------|------------|------------|
| 5 | –0.0000102 | –0.0000753 | –0.0004029 |
| 8.7 | –0.0000396 | –0.0002328 | –0.0006188 |
| 11.7 | –0.0000809 | –0.0003032 | –0.0008060 |
| 17.6 | –0.0001006 | –0.0003163 | –0.0006581 |
| 21.7 | –0.0001148 | –0.0004341 | –0.0012816 |
| 40.6 | –0.0001696 | –0.0007572 | –0.0016540 |
| 60.7 | –0.0001396 | –0.0005106 | –0.0012808 |

Table 4. Rates of Acrylamide Formation (s^{-1}) Determined at the Different Process Conditions by Assuming a Reaction of Pseudo Zero-Order^a

| moisture content (g/100 g) | 119 °C | 143 °C | 167 °C |
|----------------------------|--------|--------|---------|
| 5 | 0.2450 | 2.3076 | 19.7781 |
| 8.7 | 0.3350 | 3.5331 | 21.7902 |
| 11.7 | 0.5216 | 4.5790 | 19.0372 |
| 17.6 | 0.4166 | 3.1792 | 12.934 |
| 21.7 | 0.4955 | 3.1898 | 6.8328 |
| 40.6 | 0.2630 | 1.2610 | 2.1393 |
| 60.7 | ND | ND | 0.5456 |

^aND, no acrylamide detectable.

determined by measuring the *L*, *a*, and *b* values of the heated potato powder. **Figure 2** shows two data sets for the browning (relative *L* value) determined at 11.7 g moisture (graph A) and 21.7 g moisture (graph B) per 100 g wet weight, respectively. These two moisture contents reflect the moisture content in potatoes during the critical phase of frying (2, 11). Browning increased with process temperature and time as indicated by the slopes of the trend lines shown in **Figure 2**. Comparison of the data from the two different moisture contents (**Figure 2**) shows that browning proceeded faster at the higher moisture content (graph B) for all three temperatures tested. All browning rates according to the relative brightness are summarized in **Table 3**.

Generally, the browning rate, determined as relative brightness (*L* value), decreased with lower moisture contents (range 40.6 to 5.0 g moisture/100 g) at all three temperatures. However, the browning rates at 60.7 g moisture/100 g were lower than the rates at 40.6 g moisture/100 g. For neither the *a* value nor the *b* value, a similar relation was found as follows: The browning rates according to the *a* value or to the *b* value were rather stable at 119 and 143 °C but varied strongly at 167 °C (no data shown). The *a* value (absolute values) increased in a linear manner with heating time for all temperatures and moisture contents tested (data not shown). The higher the temperature was, the faster the changes of the *a* value were, which was also observed in fried potato slices (23) and French fries (2).

Kinetic Data of Acrylamide Formation. Acrylamide was determined in samples of different moisture contents (range, 5.0–40.6 g moisture/100 g) heated at three different temperatures. All obtained rates for acrylamide formation obtained in this study are summarized in **Table 4**. **Figure 3** shows data from two experiments with potato powder containing 11.7 g (A) and 21.7 g (B) of moisture per 100 g, respectively. Generally, acrylamide formation rates decreased with higher moisture contents for all temperatures tested. Thus, the dryer

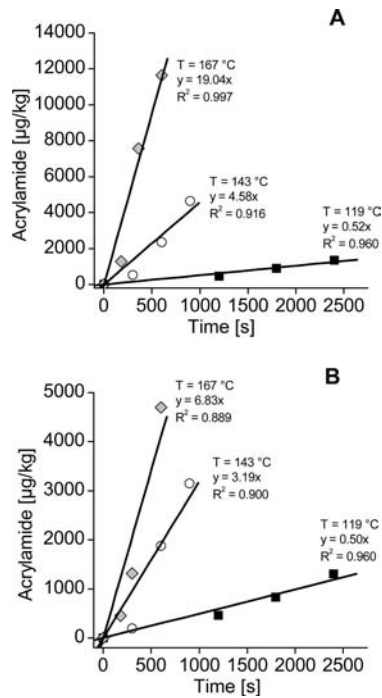


Figure 3. Acrylamide formation determined at three different temperatures for two different moisture contents: (A) 11.7 g moisture/100 g and (B) 21.7 g moisture/100 g. Note the different scales of y-axes.

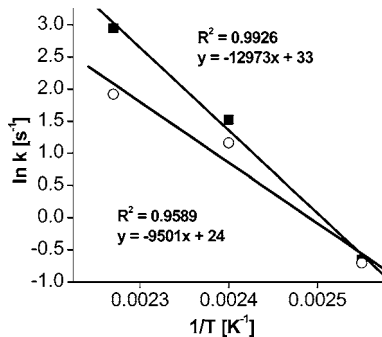


Figure 4. Arrhenius plots derived from the data shown in Figure 3. Filled squares, 11.7 g moisture/100 g; circles, 21.7 g moisture/100 g; T , absolute temperature; and k , reaction rate.

the product gets (from 40 to 10% moisture), the faster the acrylamide formation proceeds, which was also described by other groups (13, 15). The extent of acrylamide formation at 11.7 g moisture/100 g was greater than at 21.7 g moisture/100 g for all three temperatures (Figure 3). However, the highest acrylamide formation rates were found at low moisture contents, i.e., around 10%. At very low (5 g/100 g) and at higher moisture contents (> 15 g/100 g), lower acrylamide formation rates were determined. In samples of constant moisture content, the acrylamide formation increased strongly with higher temperature (see Figure 3), which indicates a strong temperature dependency of this reaction. Figure 4 shows two Arrhenius plots, which were derived from the data shown in Figure 3. The data fit the trend line well as indicated by the coefficient of correlation.

The used acrylamide data have the inherent drawback that only the net acrylamide formation was determined, which does not take elimination into account. However, no decreasing acrylamide contents were determined for long heating times, which indicates that formation still outnumbered elimination.

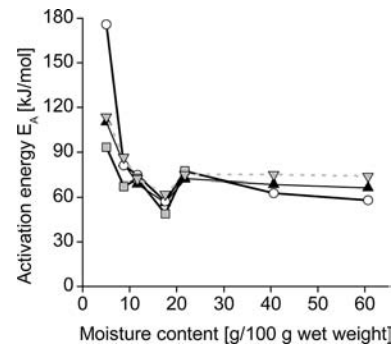


Figure 5. Influence of moisture content on the activation energy of browning (calculated with relative L values L/L_0). Data are shown for pseudo zero-order kinetics for L value (black triangles), a value (white circles), b value (grey squares), as well as for pseudo first-order kinetics for L value (grey triangles with dotted line).

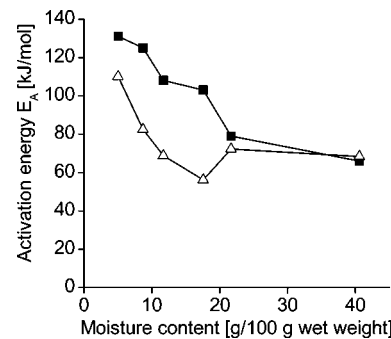


Figure 6. Influence of moisture content on the activation energy of acrylamide formation (black squares) and browning (white triangles). For browning, data according to relative brightness (L/L_0) are shown. Kinetic data were obtained assuming pseudo zero-order kinetics.

The acrylamide content of the heated potato powder correlated linearly with the L value as well as with the a value ($R^2 > 0.9$) at the three temperatures for all investigated moisture contents. The darker the powder was (lower L value), the more acrylamide it contained, and the more red the powder was (higher a value), the more acrylamide it contained. Similar findings were published by Pedreschi et al. for fried potato slices and potato strips (23, 31). However, these correlations were only observed if samples with the same moisture content were compared but not between samples of different moisture content. This indicates that the moisture content itself affected the color of the powder. Furthermore, the moisture content influences the activation energy of acrylamide formation and browning in a different manner (see below; Figure 6); therefore, correlations between acrylamide and color are lower if data from different moisture contents are compared.

Influence of Moisture Content on Activation Energy of Browning and Acrylamide Formation. The kinetic data from experiments with different moisture contents explained the influence of water on the browning and the acrylamide formation. Figure 5 shows data for the activation energy for browning (relative L , a , and b values) whereby calculations were considered as follows: Browning rates were determined for relative L , a , and b values assuming a reaction of pseudo zero-order, as well as assuming a reaction of pseudo first-order for the relative L value (L/L_0). Generally, all four different variants of calculation gave similar results indicating that the nature of the model may not be decisive for the outcome. The activation

energy for the browning was found to increase at low moisture contents whereas no large differences were found at moisture contents above 20 g/100 g.

Activation energies for browning were also determined with absolute L and a values, respectively. As observed in the calculation with relative color values, the activation energy was found to increase at low moisture contents (<20 g/100 g) for both L and a values while no large differences were found at higher moisture contents. The activation energy for browning (L and a value) differed $\leq 10\%$ if calculated with relative or absolute values, which shows that the way of determination was not critical for the outcome. For example, the activation energy amounted to 79 kJ/mol for the absolute a value and to 81 kJ/mol for the relative a value (both at 8.7 g moisture/100 g). An exception was the activation energy according to absolute a values at a moisture content of 5 g/100 g: In this case, calculation with absolute values resulted in an activation energy of 105 kJ/mol, which is over 60% less as compared to the results from relative values (176 kJ/mol) but very close to the values obtained from L and b values. However, for this moisture content, only two a values were available, which might have caused this discrepancy.

As a conclusion, browning becomes particularly temperature-dependent at low moisture content; that is, the temperature is a very critical parameter for browning toward the end of the frying process. These results are in agreement with data from model systems for Maillard reactions where browning strongly increased at low water contents (32–34). For example, at 2.9% water content, the activation energy for the formation of Amadori products amounted to 125.6 kJ/mol, whereas it decreased to 77.5 kJ/mol at 12.6% water content (33). However, our results are in contrast to the values published by Pedreschi et al. who found that the activation energy for color changes of chips decreased at lower water contents (23). It may be pointed out that the lowest moisture content considered in the study of Pedreschi was 44 g/100 g fresh weight, which is above the moisture range investigated in the present study. For a water content of 50 g/100 g fresh weight, they found an activation energy of 75 kJ/mol (23), which fits to our data as shown in **Figure 5**. Knol et al. found an activation energy of 40 kJ/mol for the formation of melanoidins in an aqueous model system (20), which supports our data as shown in **Figure 5** (low activation energy at high water content).

Figure 6 shows the influence of moisture content on the activation energies of acrylamide formation and browning (relative L value) if reactions of pseudo zero-order are assumed. In both cases, the activation energy increased at lower moisture contents. At moisture content of <20 g/100 g, the activation energy of the acrylamide formation was larger than the one of browning. Therefore, at low moisture contents, the acrylamide formation is more sensitive to temperature changes than browning. At low moisture content (<20 g/100 g), the activation energy for acrylamide formation amounted to 80–120 kJ/mol; thus, it was up to 80% higher than the activation energy for browning. As a consequence, the temperature has a particularly strong influence in products with low water content, i.e., chips, and generally toward the end of frying when products become dry. This corresponds well to findings from different studies on acrylamide formation during frying of potatoes: Most acrylamide is formed in the last phase of the frying process, i.e., when the water content of the product reaches low values. A lower frying temperature, particularly toward the end of the frying process, reduces the acrylamide formation in French fries and chips (10, 11, 35).

The assumption of a reaction of pseudo zero-order for the acrylamide formation may be questionable. However, some aspects support this assumption. (i) The amount of precursors (reducing sugars and free asparagine) present in potatoes outnumbers the amount of formed acrylamide by several orders of magnitude (7, 9, 15). (ii) The acrylamide contents increased in a fairly linear manner with time in most of the experiments performed in this study (e.g., see **Figure 3**). (iii) The acrylamide content of the unheated powder (time = 0 s) was not measurable (below detection limit), which is contradictive to an exponential curve (i.e., first-order kinetics). The kinetics of the formation of hydroxymethylfurfural in milk were also found to follow zero-order kinetics (36). Nevertheless, calculations were also performed assuming a reaction of pseudo first-order. Again, the activation energy was highest (70 kJ/mol) at the lowest moisture content and the lowest value (35 kJ/mol) was found for the highest moisture content. However, the values were generally substantially lower as compared to the values derived from zero-order kinetics (results not shown). A possible explanation for these differences could be the used data set. For pseudo zero-order kinetics, all data, i.e., no acrylamide at time zero, were included, while these data points had to be omitted in the calculations for pseudo first-order kinetics.

In literature, different values for the activation energy of acrylamide formation were published. Claeys et al. found activation energies ranging from 140 to 170 kJ/mol in aqueous sugar–asparagine models (21, 37). Sadd and Hamlet determined a value of 120 kJ/mol in a model for cereal products (16). Granda and Moreira reported an activation energy for acrylamide formation of 61 kJ/mol during traditional frying of potato slices (22). Knol et al. published values from 58 to 95 kJ/mol for the activation energy depending on the step in the pathway of acrylamide formation (20). Hence, there is some considerable variation in the available data for the activation energy of acrylamide formation. The differences in the investigated systems (aqueous vs dry, pure substances vs real food matrix, and open vs closed) and the chosen approach for the calculation may be responsible for a large part of the discrepancies. As we have shown, the moisture content plays a key role; therefore, differences between open or closed models and between models with different water content have to be expected and are easily explicable. The present data offer the advantage of using a real food matrix heated under controlled and well-reproduced conditions, which allowed for demonstrating the strong influence of the moisture content on the kinetics of acrylamide formation and browning. The investigated range of moisture contents is comparable to the moisture content in a potato during frying (3), which is a clear advantage over aqueous models. However, the potato powder presents a very complex system with several unknown factors. The presence of 3-aminopropanamide (38), the composition of the pool of free amino acids (7), and the elimination of acrylamide (13) may also be important but were not considered in this study. The findings provide some kinetic basis to explain why the end of the process is the most critical phase during frying of potatoes. Interestingly, several studies on fried potatoes support our findings (10, 11, 22, 35, 39, 40). In practice, many other factors such as raw material, pretreatments, and additives are important as well. However, because the activation energy for browning (L value) is smaller than the one for acrylamide formation at moisture contents <20 g/100 g, a reduction of the frying temperature toward the end of the frying process decreases acrylamide formation more strongly as compared to browning. Thus, French fries or chips of good color and moderate acrylamide concentration can be prepared

through this adjustment of the frying process. However, our model did neither take flavor, nor crispness, nor oil uptake into account, which are equally important for product quality. For a holistic optimization of fried potato products, data on culinary factors such as crispness and flavor need to be taken into account as well in future investigations.

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