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Evolution of surface temperature and its relationship with acrylamide formation during conventional and vacuum-combined baking of cookies

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A B S T R A C T

In this study, the mechanism by which vacuum baking reduces acrylamide in cookies was elucidated by continuous measurement of average surface temperature during conventional and vacuum-combined baking experiments. Heat transfer coefficient was also determined under atmospheric and vacuum baking conditions. Cookie dough samples (0.3 cm thickness, 5 cm diameter) were baked at 180 °C either completely under atmospheric pressure (101 kPa) or under a combination of atmospheric and reduced pressure (61 and 41 kPa). Baking times were adjusted accordingly to yield cookies with a moisture content between 3.0 and 3.5%. Surface and internal temperatures were recorded throughout baking treatments by using a thermal camera and a thermocouple, respectively. The degree of thermal load (energy input to the product) was then computed from the time - temperature profiles and related to acrylamide level in the finished product by using the total risk calculation approach. Acrylamide concentration of cookies prepared by vacuum-combined baking was found to be around 300 ng/g, a level which is about 30% lower in comparison to that of the conventionally baked sample (445 ng/g). A slight decrease in convective heat transfer coefficient was observed upon pressure reduction. Evaporative cooling effect created by sudden pressure reduction was found to be mainly responsible for limited temperature increase and hence acrylamide mitigation.

1. Introduction

Cookies, being among the foods with the highest amounts of acrylamide, are considered to be a significant contributor to acrylamide exposure of a large group of consumers (EFSA, 2015). Therefore, numerous studies have been conducted for reducing acrylamide level in cookies (Palazoğlu et al., 2012; Kocadağlı et al., 2012; Zhu et al., 2011; Açar et al., 2009; Anese et al., 2008; Gökmen et al., 2007; Graf et al., 2006). In these studies, it was found that baking time and oven temperature along with product formulation have been repeatedly reported to be the most important parameters affecting acrylamide formation.

Vacuum baking has recently been explored as an alternative baking technique due to its potential to reduce both baking time and temperature. In the limited number of studies conducted up to date (Palazoğlu et al., 2015; Mogol and Gökmen, 2014), effect of vacuum combination during baking on acrylamide level and quality characteristics of cookies has been investigated and vacuum-combined baking has been shown to be an effective acrylamide mitigation strategy. A 50% reduction in acrylamide level of cookies was achieved by Palazoğlu et al. (2015) by switching from atmospheric (101 kPa) to vacuum conditions (41 and 61 kPa) after 7.5 min of the 10-min baking process. It was concluded that vacuum application not only limited temperature increase, but also reduced the baking time (to provide a cookie with a moisture content of about 8%) which together was responsible for this observation. Also noted in the same study was that pressure reduction should take place after the crust setting is complete in order to avoid collapsing of the cookie dough. In another study conducted by Mogol and Gökmen (2014) cookie dough samples were partially baked

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conventionally at a higher-temperature (220 °C) before completing the baking process at a reduced temperature (180 °C) and pressure (500 mBar). They found that acrylamide level of the cookies was below the level of quantitation (<LOQ).

Although the above mentioned studies concluded that lower acrylamide levels were a result of the reduced thermal load (energy input to the product), they both lacked a reliable surface temperature measurement which was necessary to quantify it. In both studies, surface temperature was measured at a single point on the upper surface by using a thermocouple. Thermal load quantification is important as it forms the basis for total risk calculation approach which was first proposed by Açar and Gökmen (2010) in order to assess the level of risk arising from acrylamide formation caused by thermal processing. The approach is similar to the thermal process lethality calculation procedure, except that thermal process severity rather than thermal process lethality is calculated. Using this approach, the risk caused by a specific baking process can be evaluated provided that a maximum allowable acrylamide level for a no-risk product is defined.

A reliable surface temperature measurement during baking is clearly required in order to have a better understanding of the relationship between surface temperature profile and final acrylamide level and to accurately evaluate the risk associated with the baking process. The importance of surface temperature measurement comes from the fact that acrylamide formation primarily takes place on the surface (Gökmen and Palazoğlu, 2008). Therefore, the objective of this study was to measure the surface temperature of cookies during conventional and vacuum-combined baking experiments using a thermal camera and relate the surface temperature profile to acrylamide level in the finished product. The study also aims to determine heat transfer coefficient under conventional and vacuum baking conditions in order to assess the contribution of limited convective heat transfer caused by vacuum application to thermal load reduction.

2. Materials and methods

2.1. Chemicals and consumables

Acrylamide (99%) and L-asparagine (98%) was purchased from Sigma (Deisenhofen, Germany). L-Arginine, D-glucose, potassium hexacyanoferrate and zinc sulfate (all AnalaR grade) were purchased from Merck (Darmstadt, Germany). Formic acid and methanol was purchased from J.T.Baker (Deventer, Holland). 0.45 μm nylon membrane syringe filters, Oasis MCX and HLB (1 mL, 30 mg) solid phase extraction (SPE) cartridges, Acquity UPLC HSS T3 (100 mm, 2.1 mm id., 1.8 μm) and Atlantis T3 (250 mm, 4.6 mm id., 5 μm) columns were supplied by Waters (Millford, MA, USA).

Wheat flour, sodium bicarbonate, ammonium bicarbonate, high fructose corn syrup (HFCS) and shortening were supplied by local manufacturers, while other ingredients were purchased from a local market.

2.2. Preparation of cookies

Cookies were formulated based on the AACC method 10–54 (AACC, 2000) with 16.8 g sucrose, 0.4 g non-fat dry milk, 0.5 g NaCl, 0.4 g sodium bicarbonate, 16.0 g shortening, 0.6 g high fructose corn syrup, 0.2 g ammonium bicarbonate, 8.8 g deionized water, and 40.0 g wheat flour. The dough was prepared following the procedure given in method 10–54 with some modifications. Cookie dough was rolled out to a thickness of 0.3 cm and disks having a diameter of 5 cm were cut out using a plastic cutting mold. All baking experiments were conducted at 180 °C using an oven (Model OV-11, Jeio Tech, South Korea) coupled with a vacuum pump (Model PC 510 NT, Vacuubrand, Germany). Conventional baking of samples was conducted completely under atmospheric pressure (12 min under $P_{abs} = 101$ kPa), while vacuum-combined baking experiments took place partially under atmospheric pressure and partially under reduced pressure (8 min under $P_{abs} = 101$ kPa followed by 3 min under $P_{abs} = 61$ kPa, 8 min under $P_{abs} = 101$ kPa followed by 3 min under $P_{abs} = 41$ kPa). Baking pressure was controlled by regulating the vacuum pump. Pressure reduction from 101 to 61 kPa and from 101 to 41 kPa took 60 and 80 s, respectively. Baking times were adjusted accordingly to yield cookies with a moisture content between 3.0 and 3.5%, the level desired in the finished product (~3% by weight). Preliminary experiments showed that at least 8 min of conventional (atmospheric) baking was necessary before switching to vacuum conditions; otherwise the vacuum-combined baked products lacked a crumbly texture in the center as assessed visually and sensorily by the authors. Moisture content of samples was determined by drying 2.5 g of ground sample to constant weight at 105 °C (AOAC, 1975). Two parallel measurements were conducted. One cookie was baked at a time to avoid uneven baking, and three replications of each treatment were carried out.

2.3. Temperature measurement

Internal temperature of cookies along with air temperature inside the oven were acquired with an accuracy of 0.2 °C during baking experiments using 36-gauge type-T thermocouples (Omega Engineering, Inc., Stamford, CT) and a digital multimeter (Model 2700, Keithley, Cleveland, OH) coupled with a 20-channel multiplexer (Model 7700, Keithley, Cleveland, OH) as depicted in Fig. 1.

Surface temperature of cookies was measured using a thermal camera (Model PI200, Optris, Germany) positioned outside the oven (Fig. 1). The camera was able to see the whole upper surface of the cookie through an infrared window (Model IRW-2C, FLIR, Wilsonville, Oregon, USA) installed on the oven door. To do this, the oven was oriented so that the oven door was facing up. For the emissivity of cookies, the value reported by Baldino et al. (2014) for semi-sweet short dough biscuits (0.95) was used.

To determine the transmissivity of the infrared window, a copper disk (5 mm thickness, 50 mm diameter) painted with a matte black paint ($\varepsilon = 0.95$) (Vollmer and Möllmann, 2010) was placed inside the oven at 180 °C and brought to thermal equilibrium. The equilibrium temperature of the copper disk was measured simultaneously by the infrared camera through the window and a thermocouple. Then, the transmissivity setting of the infrared camera was adjusted until the temperature measured by the camera matched the true temperature measured by the thermocouple. Following this procedure, transmissivity of the infrared window at the baking temperature was determined to be 0.68.

2.4. Determination of heat transfer coefficient

Heat transfer coefficient was determined at 180 °C under 101, 61, and 41 kPa by the lumped capacitance method using both a painted cookie model and a polished cookie model made of copper ($\rho = 8940$ kg/m^3, $c_p = 390$ J/kgK). Emissivity values of the painted and polished cookie models were 0.95 and 0.03, respectively (Vollmer and Möllmann, 2010). The reason for using both a painted and a polished cookie model for determining heat transfer coefficient was to assess the individual contributions of radiation and convection to overall heat transfer. Both cookie models had a thickness of 5 mm and a diameter of 50 mm, and were coupled with 36-gauge type-T thermocouples (Omega Engineering, Inc., Stamford, CT) to measure their temperature. Experiments with the

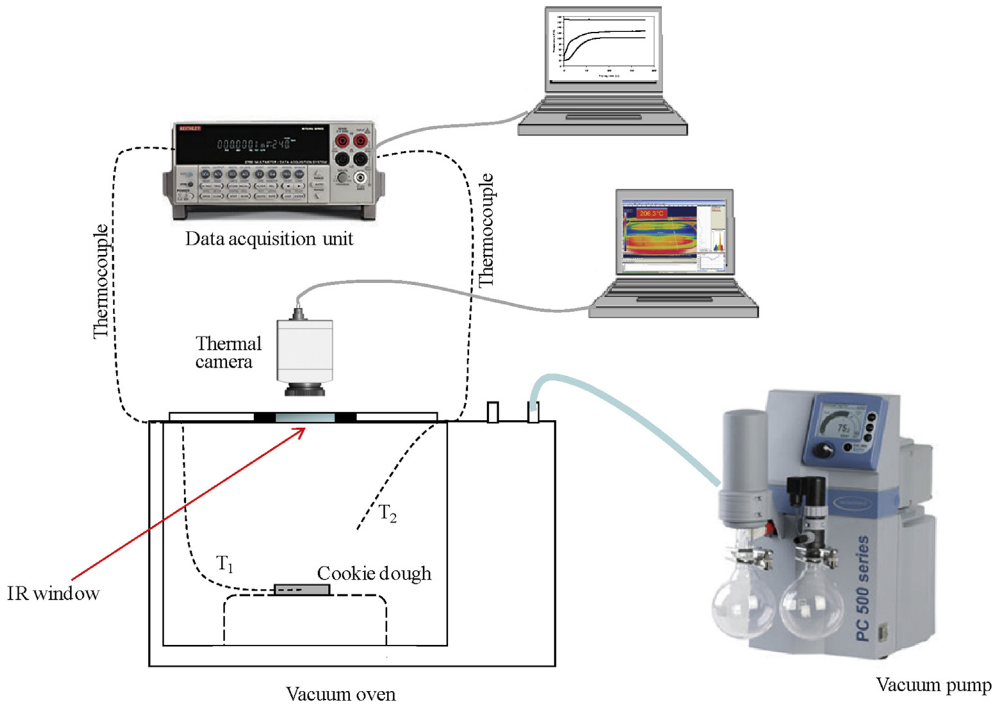

Fig. 1. Schematic of the experimental setup.

model cookies were conducted in duplicate, and convective and combined heat transfer coefficients were estimated from the slope of the $\ln\left(\frac{T(t)-T_\infty}{T_i-T_\infty}\right) - t$ plot (Eq. (1)).

$$\ln\left(\frac{T(t) - T_\infty}{T_i - T_\infty}\right) = -\frac{h\,A}{m\,c_p}t \tag{1}$$

capillary voltage of 0.75 kV; cone voltage of 21 V; extractor voltage of 4 V; source temperature at 120 °C; desolvation temperature at 450 °C; desolvation gas (nitrogen) flow of 900 L/h. Acrylamide was identified by multiple reaction monitoring (MRM) of two channels. Collision gas (argon) flow rate was 0.25 mL/min. The precursor ion $[M+H]+$ 72 was fragmented and product ions 55 (collision energy of 9 V) and 44 (collision energy of 12 V) were monitored. For all MRM transitions the dwell time was 0.2 s.

2.5. Acrylamide analysis

Acrylamide analysis was performed in the surface, which was prepared by removing a 1.5–2 mm (about 1.5 g) layer from the top surface by using a fine grater, and the remaining portion of the cookie separately. The samples were prepared for acrylamide analysis by multi-stage extraction strategy according to the procedure described by Gökmen et al. (2009). In brief, 1 g granulated cookie sample was extracted with 20 mL 10 mM formic acid in triple-stage (10, 5, 5 mL). Cold centrifugation (0 °C) was employed for the separation of fat from the sample at 5000 rpm for 10 min. The co-extracted colloids were precipitated by Carrez clarification and the combined extract was cleaned up by using Oasis MCX SPE cartridge (previously conditioned with 1 mL of methanol and 1 mL of water). First 8 drops of 1 mL extract were discarded to avoid any dilution and the remaining extract was collected in an autosampler vial. 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.

The samples were analyzed by using Waters Acquity UPLC system coupled to a TQ detector with ES + mode. Chromatographic separations were achieved by Acquity UPLC HSS T3 column using 10 mM formic acid with 0.5% methanol as mobile phase at a flow rate of 0.3 mL/min. The column was equilibrated at 40 °C and Waters ACQUITY FTN autosampler was held at 10 °C during the analysis. The electro-spray source had the following settings:

2.6. Total risk calculation

Total risk calculation is an approach developed by Açar and Gökmen (2010) in order to quantify the impact of a thermal process in regards to food safety associated with formation of thermal process contaminants. The approach is similar to thermal process lethality calculation procedure. First, risk formation rate (R_v) is computed using a reference temperature and the z-value by using Eq. (2). Then, the total risk (TR) is calculated as the area under the risk formation rate (R_v) versus time curve by using Eq. (3).

$$R_v = 10^{\frac{T-200}{z}} \tag{2}$$

$$TR = \int_{t_1}^{t_2} R_v dt \tag{3}$$

A total risk value that is lower than the time required for acrylamide concentration to reach the “risk threshold value” is desired. The risk threshold value was accepted to be 200 ng/g by Açar and Gökmen (2010), and time required to reach this value at 200 °C was determined to be 0.2 min. Total risk is calculated using 200 °C as the reference temperature and 30 °C for the z value. This z value was determined by Açar and Gökmen (2010) based on the formation and degradation kinetics of acrylamide within a temperature range of 100–250 °C during baking of cookies.

2.7. Statistical analysis

Acrylamide data reported are means of three replicates. Statistical analysis was performed using one-way ANOVA and Tukey's HSD post-hoc test where $p < 0.05$ was considered statistically significant.

3. Results and discussion

3.1. Temperature profiles

Fig. 2 shows the oven temperature and the internal and average surface temperatures of cookie dough during 12-min baking under atmospheric (101 kPa) pressure. By the time the oven lid was closed and the thermal camera was positioned after placing the cookie dough inside the oven, surface of the cookie dough experienced some temperature increase. This was the reason why the average surface temperature started at around 90 °C. At the end of baking, average surface and internal temperatures were 151.0 °C and 125.8 °C, respectively.

The highest average surface temperature attained during baking was reduced to around 140 °C (Fig. 3-a and b) when vacuum was introduced toward the end of the process. In the study conducted by Palazoğlu et al. (2015), the final surface temperature was measured to be slightly above 120 °C for the conventionally baked sample (205 °C–11 min). This proves that surface temperature of bakery products can be considerably underestimated when it is measured by thermocouples. A reliable measurement of temperature at the surface of bakery products during baking is only possible through use of a thermal camera as the thermocouple tip becomes buried in the dough as baking proceeds (as a result of dough rising) and from then on it can no longer measure the surface temperature. Furthermore, a thermocouple can measure temperature only at one point and the measured value should not be expected to be representative of the whole surface.

Surface of the cookie dough experienced a lower temperature drop when compared to its interior when the pressure was reduced. Two reasons for this observation can be proposed. First, the surface was drier than the interior and hence a lower degree of cooling resulted from the evaporation of less moisture. Second, the surface was able to be rapidly reheated since it was exposed to convection and radiation effects. It is obvious that more time was needed for this heat to penetrate into the interior by conduction.

One interesting finding of the present study was that after 167 s of baking, temperature was equal to or higher than 120 °C throughout the whole upper surface of the cookie and remained as such for the rest of the baking process in both conventional and vacuum-combined baking experiments. Although internal temperature exceeded 120 °C toward the end of atmospheric baking (Fig. 2), this temperature was not reached in the interior of the cookie under vacuum-combined treatments (Fig. 3-a and b). The temperature of 120 °C is especially important, because it is reported to be the temperature above which acrylamide formation is favored (Şahin and Barutcu, 2011).

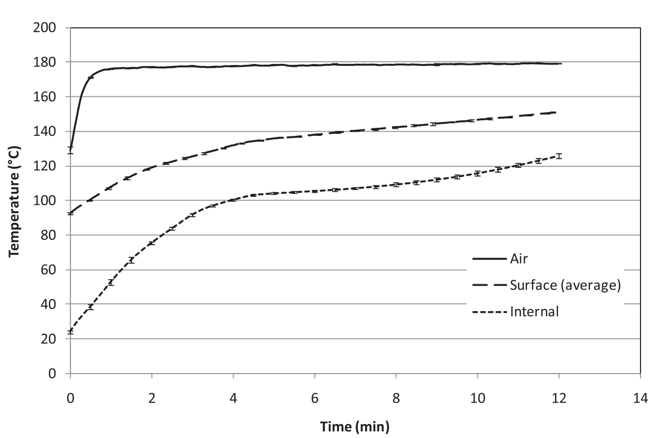

Fig. 2. Average surface and internal temperatures of the cookie sample along with air temperature during conventional baking (12 min under 101 kPa).

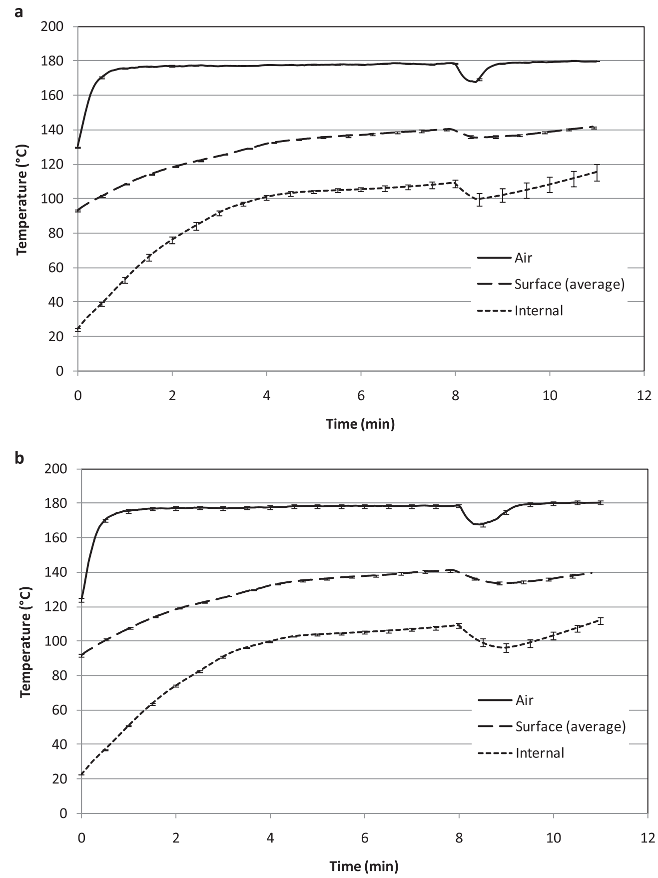

Fig. 3. Average surface and internal temperatures of the cookie sample along with air temperature during conventional-vacuum combined baking, a) 8 min under 101 kPa + 3 min under 61 kPa, b) 8 min under 101 kPa + 3 min under 41 kPa.

Fig. 4 shows the thermal images of the surface of cookie dough with 1-min interval during the baking experiments. It is clearly visible from these images (surface temperature distributions) that pressure reduction caused a substantial temperature drop on the majority of the surface. Already drier edge, where the maximum temperature exists, experienced a lower temperature drop as expected. An interesting observation for vacuum-combined baking is that when the baking was completed the average surface temperature was still below 143 °C (the value at the 8th minute, just prior to vacuum application).

3.2. Acrylamide content

Fig. 5 shows the acrylamide concentrations of the whole cookie, top surface, and the remaining portion separately for all treatments. No difference between these acrylamide concentrations, however, was found for any of the treatments. This observation may be attributed to the small thickness of the dough (0.3 cm) and baking it until the moisture content was down to 3% by weight.

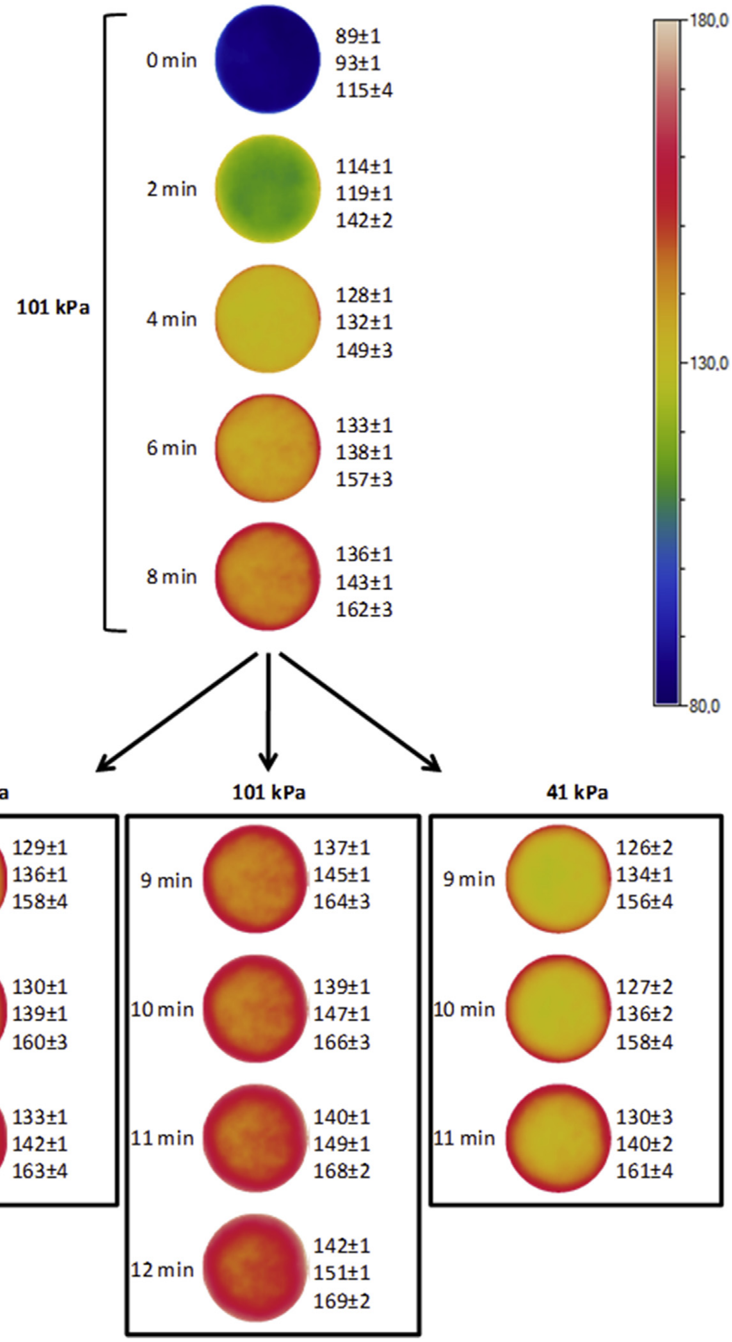

Fig. 4. Thermal images (surface temperature distribution) of cookies as a function of time for all baking treatments (the numbers on the right side of each thermal image indicate minimum (top), average (middle), and maximum (bottom) temperatures on the surface).

Acrylamide concentration of cookies prepared by vacuum-combined baking (~300 ng/g) was about 30% lower in comparison to that of the conventionally baked sample (445 ng/g). Palazoğlu et al. (2015) reported that when the last 2.5 min of the 10-min baking process at 205 °C was carried out under reduced pressure, the reduction in acrylamide level was 45% (for 61 kPa) to 53% (for 41 kPa). The thickness of the cookie dough in their study was, however, 7 mm resulting in a final moisture content of about 8% by weight. When the dough thickness is large and the moisture content of the finished product is high, a large difference in acrylamide concentration results between the interior and the crust, which in turn causes overall and surface acrylamide concentrations to be different.

3.3. Heat transfer coefficient

Combined (convective + radiative) heat transfer coefficient values determined under atmospheric pressure (Table 1) were in agreement with those reported by Sakin et al. (2009, 2009) estimated combined heat transfer coefficient values between 11 and 20 W/m^2K for atmospheric baking within a temperature range of 70–220 °C by using an oxidized aluminum ($\varepsilon = 0.33$) cylinder as the lumped system.

Radiation effect in the oven used in the present study can be extracted from Table 1. The values obtained with the polished copper disk do not include the radiation effect because of the very low emissivity of polished copper ($\varepsilon = 0.03$), and can therefore be

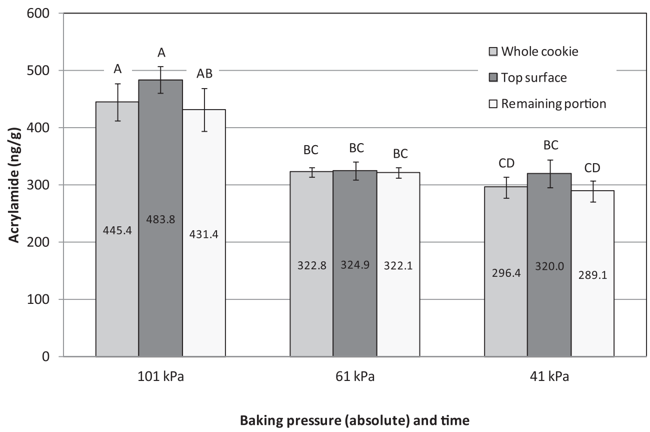

Fig. 5. Acrylamide levels of cookies (whole, top surface, and the remaining portion) prepared by conventional and vacuum-combined baking treatments (Means with the same letters are not significantly different (P > 0.05)).

considered as convective heat transfer coefficient. However, the values obtained with the painted copper disk include both convection and radiation effects (combined heat transfer coefficient). The difference between these two values is simply due to radiative heat transfer. It should be noted that the linear regression coefficient for all the data was greater than 0.999. In the present study, contribution of radiative heat transfer to overall heat transfer was found to be 46% under atmospheric pressure and 52% under reduced pressures (61 and 41 kPa). Sakin et al. (2009) also reported that at 220 °C under atmospheric pressure, 44% of overall heat transfer in baking oven was due to radiation. It is reasonable to expect the relative contribution of radiation to overall heat transfer to be larger for lower baking pressures since only convection is affected by pressure reduction. It was also reported by Saidi and Abardeh (2010) that reduced air pressure increases convective heat transfer resistance while it has no effect on radiation.

As can be seen from Table 1, a slight decrease in combined heat transfer coefficients was observed in this study when the oven pressure was reduced as expected, since convection was slowed down when there were fewer air molecules to carry heat inside the oven. The contribution of this reduction to limiting temperature increase during vacuum baking should however be minimal.

3.4. Total risk calculation

The total risk value calculated for the conventionally baked sample was 6.57 s (Fig. 6-a), which means that 12-min baking at an oven temperature of 180 °C under the atmospheric pressure resulted in a surface temperature profile which had a thermal impact equivalent to that of 6.57 s at 200 °C in terms of acrylamide formation. It should however be noted that the same baking time at the same oven temperature in a different oven and under different baking conditions may very well result in a different thermal impact. According to Açar and Gökmen (2010), a risk value of 6.57 s should translate into an acrylamide concentration lower than 200 ng/g, since it is lower than 0.2 min (time needed at 200 °C in order to reach the threshold value of 200 ng/g). However, acrylamide content of this sample was 445 ng/g.

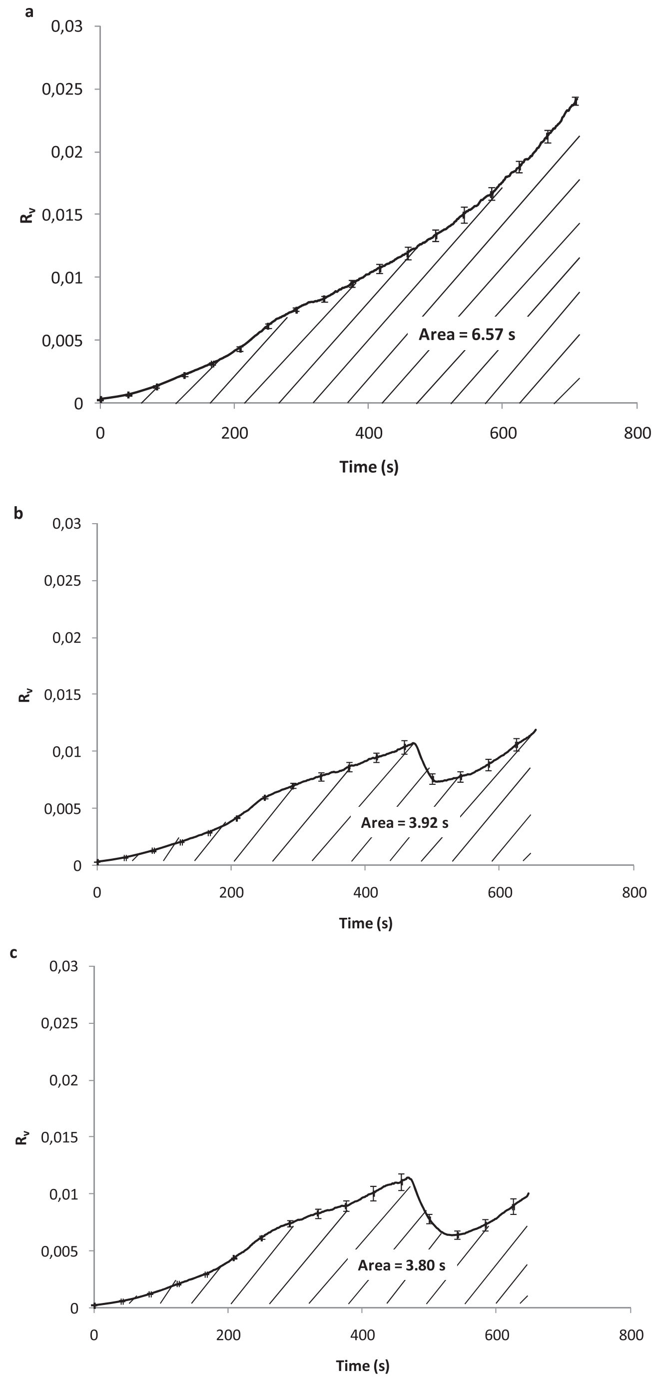

Fig. 6. Risk formation rate vs. time plots, a) conventional baking (12 min under 101 kPa), b) vacuum-combined baking (8 min under 101 kPa + 3 min under 61 kPa), c) vacuum-combined baking (8 min under 101 kPa + 3 min under 41 kPa).

Table 1
Heat transfer coefficient values (W/m^{2}K) under varying baking pressures.

Absolute pressure (kPa)	Combined (painted cookie model)	Convective (polished cookie model)
101	21.1 ± 0.2^a	11.5 ± 0.3^a
61	19.9 ± 0.1^b	9.6 ± 0.5^b
41	17.7 ± 0.1^c	8.6 ± 0.4^b

Means with the same superscripts in the same column are not significantly different (P > 0.05).

Lower risk values were calculated for vacuum-combined baked samples (3.92 s for 8 min under 101 kPa + 3 min under 61 kPa, 3.80 s for 8 min under 101 kPa + 3 min under 41 kPa) (Fig. 6-b and c). Acrylamide concentrations in the surface of these samples were also comparable (325 and 320 ng/g, respectively).

These results are not in accordance with those of Açar and Gökmen (2010), who generated their kinetic data by baking thin (<1 mm) cookie dough samples using the same cookie dough formulation as the one used in the present study. Discrepancies observed could possibly be due to the fact that the risk value was calculated using the average surface temperature, not taking into account the drier and hence hotter edges of the product surface where acrylamide formation can take place at a much higher rate. It is also possible that isothermal conditions may not have been maintained during the experiments conducted by Açar and Gökmen (2010). More reliable kinetic data (thermal formation time and z value) will need to be generated before the total risk calculation approach can be universally used in order to determine the risk associated with acrylamide formation in cookies. As stated by Nunes et al. (1993), accurate thermal process evaluation requires accurate kinetic parameters along with a reliable temperature measurement.

4. Conclusion

For the first time with this study, surface temperature of cookie dough during baking was measured reliably and continuously by using a thermal camera. Baking severity (thermal load) was then computed from the measured surface temperature profile and related to the resultant acrylamide level in the final product. The results showed that surface of the cookie dough in fact reaches temperatures as high as 150 °C (average) under conventional (atmospheric) baking conditions employed in this study, a value much higher than those previously measured by thermocouples. Heat transfer coefficient values under vacuum pressures were only slightly lower than that for atmospheric pressure, and apparently had little contribution to limiting thermal input to the cookie's surface.

Although a risk value calculated using the surface temperature profile may not always be representative of the whole cookie, it will give a conservative estimate since the whole cookie will always have a lower acrylamide concentration on the average in comparison to the surface within the normal baking times. It should also be noted that the risk value is independent of the oven and baking conditions when it is calculated using surface temperature profile provided that formulation and thickness of the dough are the same.

Practical application

Project findings will provide insight for development and optimization of new baking ovens and processes.

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