



Color changes in fish during grilling – Influences of heat transfer and heating medium on browning color

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ARTICLE INFO

Article history:

Received 29 June 2012

Received in revised form 12 November 2012

Accepted 22 November 2012

Available online 7 December 2012

Keywords:

Color

Fish

Browning

Kinetic analysis

Far-infrared radiation

Superheated steam

ABSTRACT

Samples of red sea bream (*Pagrus major*) were grilled under radiant (far-infrared radiation, FIR) and convective (superheated steam, SHS) heating. The temperature and color (CIE L^* , a^* , and b^* values) of the sample surface were monitored over time, using SHS, dry air, and N_2 as heating media. The rate of L^* changes was evaluated by treating the browning reaction as first-order. Color changes based on a^* and b^* values were effectively correlated with the L^* value, using empirical equations. A slower reduction in L^* for heating with SHS rather than FIR was obtained, probably because of different activation energies (31.5 and 50.7 kJ mol⁻¹) and frequency factors (8.2 and 4759 s⁻¹). The order of reductions in L^* was dry air > N_2 = SHS. The absence of O_2 in the heating medium could be the reason for the delay in the browning reaction during heating using N_2 and SHS.

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1. Introduction

The challenge of developing advanced thermal processes for the food industry continues, in line with the demand for enhanced food safety and quality, because thermal processing is always associated with some undesirable degradation of heat-sensitive quality attributes. Quality degradation as a result of excessive heating, especially at high temperatures, and its prevention require individual attention. The effect of thermal processing on sensory quality in marine products is difficult to predict because of intra- and inter-specific differences in fish species and factors such as appearance, odor, color, flavor, and texture.

Consumers often assess the initial quality of a product by its color and appearance, and color serves as a clue as to how well food is cooked. The analysis of color changes during heating therefore has a great influence not only on the quality but also on the safety of food products. Using a color difference meter, a sensor that touches the food, color is determined by measuring the reflection of emitted light. Several researchers have contributed to this field (Kaida et al., 1999; Pedreshi et al., 2006; Kong et al., 2007; Yan et al., 2008; Braeckman et al., 2009). Moreover, if the changes in food color, including the browning process, during cooking can be predicted and effectively modeled, appropriate cooking methods such as grilling can be improved. For grilling processes in the food industry, conduction and convective heat transfer have

mainly been used, as well as radiant heat transfer, e.g., from an infrared (IR) lamp (Sakai and Mao, 2006). Shibukawa et al. (1989), Sato et al. (1992), and Sugiyama et al. (1993) reported the influence of surface color on browning formation, as well as the influence of the ratio of radiant heat transfer to the radiation wavelength on scorching color. After comparing food cooked in a convection oven and that cooked in a radiation oven, Sato et al. (1992) claimed that the heat transfer system did not directly affect the browning color. However, the heat transfer system influences the surface temperatures of foods, and surface temperature differences affect the browning color.

Recently, superheated steam (SHS, steam-spray type) has been widely used as a heat source in steam convection ovens in hotels, kitchens, and elsewhere. A steam convection oven features high reproducibility and can process large amounts of food. The characteristics of fish grilled by SHS and by propane-gas heating were compared by Hamada et al. (2006). They obtained better results with the former than with the latter; in particular, the samples were juicier in the former case and the grilling time was shorter. Ohishi and Shibukawa (2008) found that a cake baked in an SHS oven was darker than a cake baked in a steam-free convection oven, indicating a shorter baking time. They observed a considerable transfer of heat during the initial stage of heating in an SHS oven.

Developing a kinetic model of food color changes during cooking is an important challenge in food processing. If the rate and temperature dependence of a reaction are known, they can, in principle, be predicted and therefore controlled (Martins et al.,

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Nomenclature

E_a	activation energy (kJ mol ⁻¹)	<i>Subscripts</i>	
k_0	frequency factor (s ⁻¹)	<i>c</i>	calculated
R	molar gas constant (J K ⁻¹ mol ⁻¹)	<i>f</i>	final
t	grilling time (s)	<i>i</i>	initial
T	surface temperature (K)	<i>m</i>	measured
Y	dimensionless parameter related to color lightness (-)	<i>cs</i>	calculated at same surface temperature
a^*	fish surface color position on red/green axis (-)	<i>msi</i>	measured at similar surface temperature
b^*	fish surface color position on blue/yellow axis (-)		
L^*	fish surface color lightness (-)		

2001). In the browning of bakery products, the model suggested by Broyart et al. (1998) for the baking of crackers is widely used. They proposed a non-isothermal kinetics approach to modeling color development on the food surface during baking; in the model, the thermal history and moisture content are taken into account. With fish, however, obtaining the water activity (or moisture content) variation during grilling is complicated. Such measurements require the setting up of a destructive method for accurate sampling while the fish is still hot and deformable, and water losses by evaporation from cut surfaces must be minimized (Wagner et al., 2007; Purlis and Salvadori, 2009).

Onishi et al. (2011) evaluated the browning on the surface of baked bread using computer vision, numerical simulations, and non-isothermal kinetics, and modeled the development of browning color formation. They did not consider weight loss as a factor in their model, because they found a high correlation between weight loss and the CIE L^* value only at the highest baking temperature, that is, within the temperature range where browning ends and initial carbonization begins. Nakamura et al. (2011) analyzed the browning of fish samples during grilling on the basis of the relationships among color, sample surface temperature, and grilling time using near-IR (NIR) heating. The proposed method enabled prediction of the L^* value from the surface temperature history, as well as the CIE a^* and b^* values, from empirically developed equations obtained using an NIR heating grill, and thus enabled the characteristic trajectory of color changes for grilled fish in colorimetric space (L^* , a^* , and b^*) to be drawn. However, there are very few reports on the effects of other heat transfer modes during grilling processes, or on the differences among various heating media in browning color simulation.

In the present study, we examined the influence of the heating medium used for grilling on browning color formation in fish, using two different heat transfer systems, namely radiant (far IR, FIR) and convective (SHS) heating, and three convective heating media: SHS, dry air, and nitrogen (N₂).

2. Materials and methods

2.1. Raw material

Red sea bream (*Pagrus major*), a white-flesh fish (cultivated in Ehime, Japan), was used as the sample material. Raw fillets were purchased on the day of the experiment. The skin and bones were removed, and the fillets were cut into pieces of size 5 × 6 × 2 cm³ (width × length × thickness) for all the experiments. The samples were wrapped in wrapping film and refrigerated (5 °C) until they were used in the experiments. The initial moisture content of the samples was approximately 77% w/w (wet basis).

2.2. Experimental conditions

Two experimental apparatuses were used to evaluate the color changes of the samples during grilling under different heat transfer

systems: an FIR oven (radiant) and a convection-type oven (convective). Schematic diagrams of these apparatuses and their heating sources (manually assembled oven, laboratory-scale) are shown in Fig. 1A and B for FIR heating (100 V/750 W) and convection-type heating (steam-spray type), respectively.

2.2.1. FIR-type oven

The FIR heater (electric ceramic plate heater PLC-328, Noritake Co., Aichi, Japan) was square in shape (12 × 12 cm²). The IR energy was irradiated downward from the heater. The samples, which were positioned approximately 8 cm below the heat source, were placed on an electronic balance. The radiation energy, measured by a radiation sensor (RF30 Captec, Villeneuve d'Ascq, France) at the sample position, was 2.7 × 10⁴ W m⁻². Sampling of the grilled samples was conducted at prescribed times of 0, 2, 4, 6, 8, and 10 min.

2.2.2. Convection-type oven

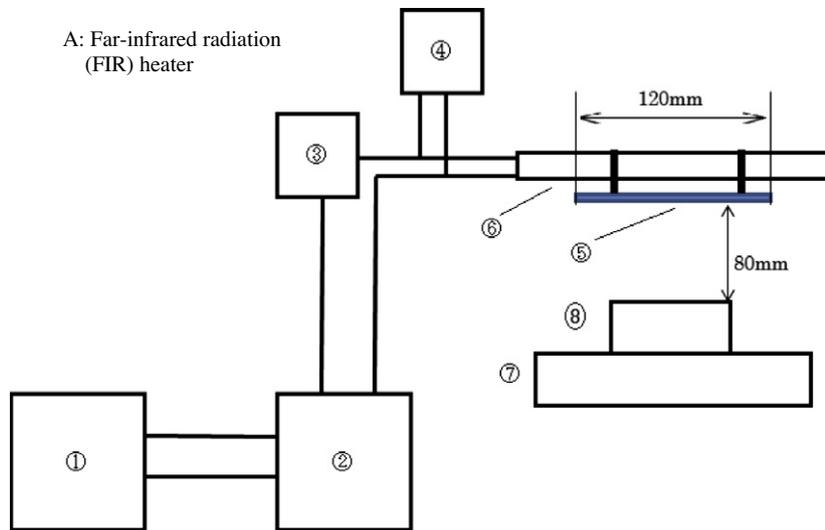
Fish samples that were similar in shape and size to those used in the FIR experiments were prepared in the same manner as described above. Three heating media were tested: SHS, dry air, and N₂. The samples were positioned approximately 8 cm below the hot-gas outlet. In the case of SHS, steam was generated by a boiler and superheated from 100 °C to 240 °C by gas heating. The steam was sprayed onto the sample at a flow rate of 2.5 kg h⁻¹. Dry air or N₂ was injected via a pipe connected to the super-steamer (see Fig. 1B), instead of via a direct pipe connection to the steam boiler, as in the case of SHS.

To compare the heating media used in this oven, similar surface temperature histories were used for the three heating media. Although the internal oven temperature was set at 200 °C (in all cases), SHS was sprayed at 200 °C–240 °C with a flow velocity of 27.4 m s⁻¹, whereas dry air was sprayed at 200 °C–220 °C with a flow velocity of 15 m s⁻¹. For the experiments with N₂, the conditions were similar to those used for dry air. Sampling of the grilled samples for color and surface temperature measurements was conducted at prescribed times of 0, 1, 2, 4, 6, 8, 10, 12, and 14 min.

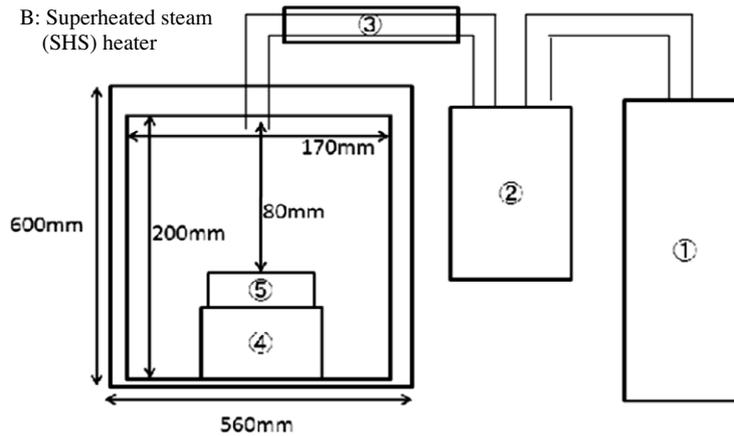
2.3. Surface temperature measurements

The surface temperatures of the samples were measured using a K-type thermocouple ($\phi = 0.5$ mm). A personal computer, a data-logger (Thermovac 5001A, Eto Denki Co., Tokyo, Japan), and software (Thermovac-E/Ef 2.6, Eto Denki Co.) were used to collect the temperature data.

To justify the use of K-type thermocouples instead of IR sensors, recognized as a more robust device for surface temperature measurements, the accuracy of the surface temperature profiles collected using K-type thermocouples was verified by comparison with profiles collected with IR sensors, using SHS heating under the conditions described above, and in the temperature range 110–150 °C. The surface temperature profiles were similar, with a variation of ±0.5 °C in the worst case. In the comparison of the



1. Power supply-unit (100V). 2. Power controller. 3. Ammeter. 4. Voltmeter. 5. Infrared heater. 6. Heater holder. 7. Electronic balance. 8. Sample.



1. Steam boiler. 2. Super-steamer. 3. Micro heater. 4. Sample stand. 5. Sample.

Fig. 1. Experimental grilling device. A: far-infrared radiation (FIR) heater, B: superheated steam (SHS) heater.

whole profile, the mean of the absolute values of the relative error ($\overline{|RE|}$) was 0.96%, with a standard deviation ($SD \overline{|RE|}$) of 0.49%. The method for evaluating the error determination was described in detail in Llave et al. (2012).

2.4. Measurement of color

Changes in the color values of the fish surface were measured using the L^* , a^* , and b^* (L^* , lightness; a^* , red/green; b^* , yellow/blue) system at the same position as the surface temperature measurements, using a spectrophotometric color difference meter (NF333, Nippon Denshoku Industries Co., Ltd., Tokyo, Japan). The L^* , a^* , and b^* values were measured using a D65 light source with a viewing field angle of 2° . The color results represent the mean value of at least three samples, excluding the maximum and minimum values.

2.5. Kinetic study of browning reaction during grilling

The browning process is a Maillard-type reaction and is considered to involve a series of complex reactions. It is difficult to

identify the elementary reaction and to obtain the rate constant of an individual reaction. The present study used a kinetics theory similar to those reported by Broyart et al. (1998), Nakamura et al. (2011), and Purlis and Salvadori (2009).

Assuming that an original substance A (reactant A) is transformed into a browning substance P by heating, and that the production rate of P is proportional to the concentration of A remaining, the reaction rate can be expressed by the following equations:



$$\frac{dC_P}{dt} = -\frac{dC_A}{dt} = kC_A \quad (2)$$

Initial conditions:

$$C_A = C_{Ai}, \quad C_P = C_{Pi}, \quad \text{at } t = 0 \quad (3)$$

where C_A and C_P are the concentrations of A and P , respectively, k is the rate constant, and subscripts i and f represent the initial and final values, respectively. Assuming that initially there is no

browning substance and that all of A finally changes into P, the initial and final concentrations are represented as follows:

$$C_{Ai} = C_0, \quad C_{Pi} = 0$$

$$C_{Af} = 0, \quad C_{Pf} = C_0, \quad C_0 = C_A + C_P \quad (4)$$

where C_0 is the total concentration of A plus P. As C_P increases from C_{Pi} to C_{Pf} , the sample darkens, that is, L^* decreases from L_i^* to L_f^* . Assuming the decrease in L^* is proportional to the concentration of P, the dimensionless parameter Y is defined by the following equation:

$$Y = \frac{C_{Pf} - C_P}{C_{Pf} - C_{Pi}} = \frac{L_f^* - L^*}{L_f^* - L_i^*} \quad (5)$$

Substituting Eq. (4) into Eq. (5), the experimental value of Y can be calculated from Eq. (6):

$$Y = \frac{C_0 - C_P}{C_0} = \frac{C_A}{C_0} = \frac{L_f^* - L^*}{L_f^* - L_i^*} \quad (6)$$

Substituting Eq. (6) into Eqs. (2) and (3) yields the following equation:

$$\frac{dY}{dt} = -kY \quad (7)$$

Although this dimensionless parameter does not have any physical meaning (van Boekel, 2008), Eq. (7) was chosen because browning was modeled through the color change intensity and did not directly involve chemical compounds. If the surface temperature is constant, Eq. (7) can be easily integrated as Eq. (8), and the plot of $\ln Y$ versus time provides the rate constant k from its gradient:

$$\ln Y = -kt \quad (8)$$

However, Purlis and Salvadori (2009) claimed that browning is a non-isothermal process. Therefore, since the surface temperature of the fish depends on the grilling time, an analytical expression for lightness cannot be obtained from Eq. (7) or Eq. (8). Dolan (2003) reported that a highly temperature-dependent reaction (such as browning) would be expected to have a greater error if the time-temperature history is neglected when the kinetic parameters are estimated. We assumed that the temperature dependence of the rate constant follows the Arrhenius equation:

$$k = k_0 \exp\left(-\frac{E_a}{RT}\right) \quad (9)$$

where k_0 is the frequency factor, E_a is the activation energy, and R is the gas constant. We then evaluated the frequency factor and the activation energy as follows. When Eq. (9) is substituted into Eq. (7), the following equation is obtained:

$$\frac{dY}{dt} = -k_0 \exp\left(-\frac{E_a}{RT}\right) Y \quad (10)$$

If the frequency factor and the activation energy are provided, and if the experimental time-temperature history is used, Y can be calculated by the numerical integration of Eq. (10). The frequency factor and the activation energy were determined, as they showed agreement with the measured values in the grilling experiment controlled by surface temperature. The method for determining these values was described in detail in a previous paper (Nakamura et al., 2011).

3. Results and discussion

3.1. Color changes of fish sample surfaces

Fig. 2 shows the surface color changes (L^* , a^* , and b^* values) and surface temperatures of the samples during FIR and SHS heating (Fig. 2A and B, respectively).

According to Nakamura et al. (2011), the color change during grilling consists of four steps: (1) protein denaturation, (2) water evaporation, (3) a browning reaction, and (4) a carbonization reaction.

To analyze color changes during grilling by FIR heating, each sample was grilled from its raw state until the surface temperature reached approximately 180 °C (Fig. 2A). The fish was white and bright up to 95 °C, at which point the color began to change as a result of protein denaturation; 95 °C was also the point at which L_m^* peaked. Above that temperature, L_m^* decreased gradually as water evaporated. At the same time, the a_m^* and b_m^* values increased slightly as a result of the browning reaction. Finally, when the surface temperature was close to 150 °C, the samples began to darken, a_m^* peaked, and L_m^* was approximately 30. This was considered to be the starting point of the carbonization step. Similar results were reported by Nakamura et al. (2011) using the same fish under NIR heating. Above this point, as the surface temperature continued to rise, L_m^* , a_m^* , and b_m^* decreased.

Fig. 2B, shows the color changes of fish samples during grilling by SHS heating; the changes were similar to those presented in Fig. 2A, although the temperature was higher (240 °C instead of

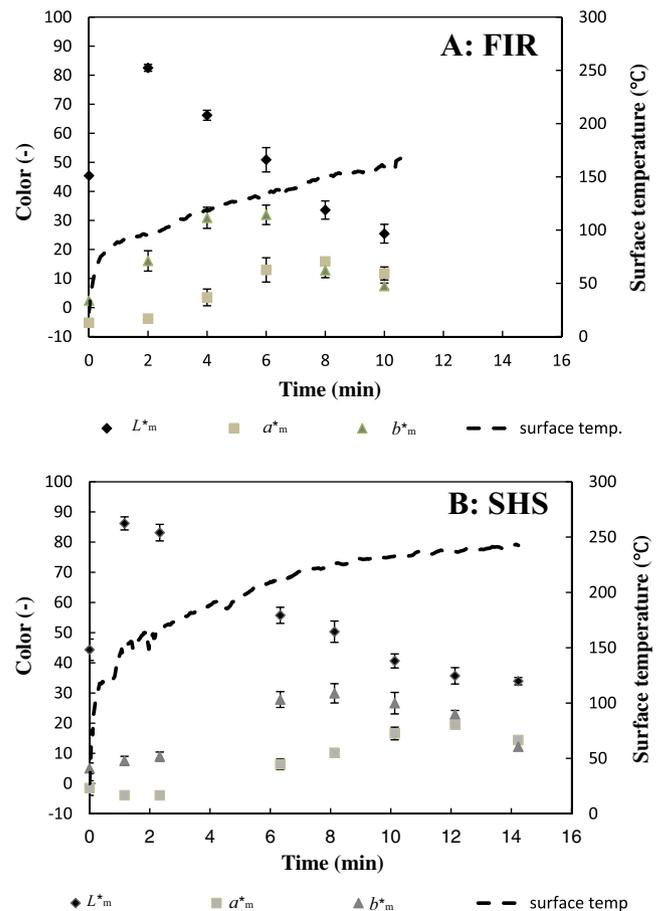


Fig. 2. Measured values of surface color changes during grilling. A: far-infrared (FIR), B: superheated steam (SHS). Bars indicate the standard deviation from five determinations.

200 °C in the case of FIR heating). Despite the higher temperature, the heating time was longer for SHS than for FIR heating. The carbonization phase started after 12 min instead of 8 min. With SHS, the need to use higher temperatures has been reported, because

SHS provides lower rates of heat transfer while the steam is superheated (Spirax-Sarco, 2007). It was reported that, as a result, it is necessary to prolong the grilling time with SHS heating, as was observed in the present study. The results presented in Fig. 2A and B

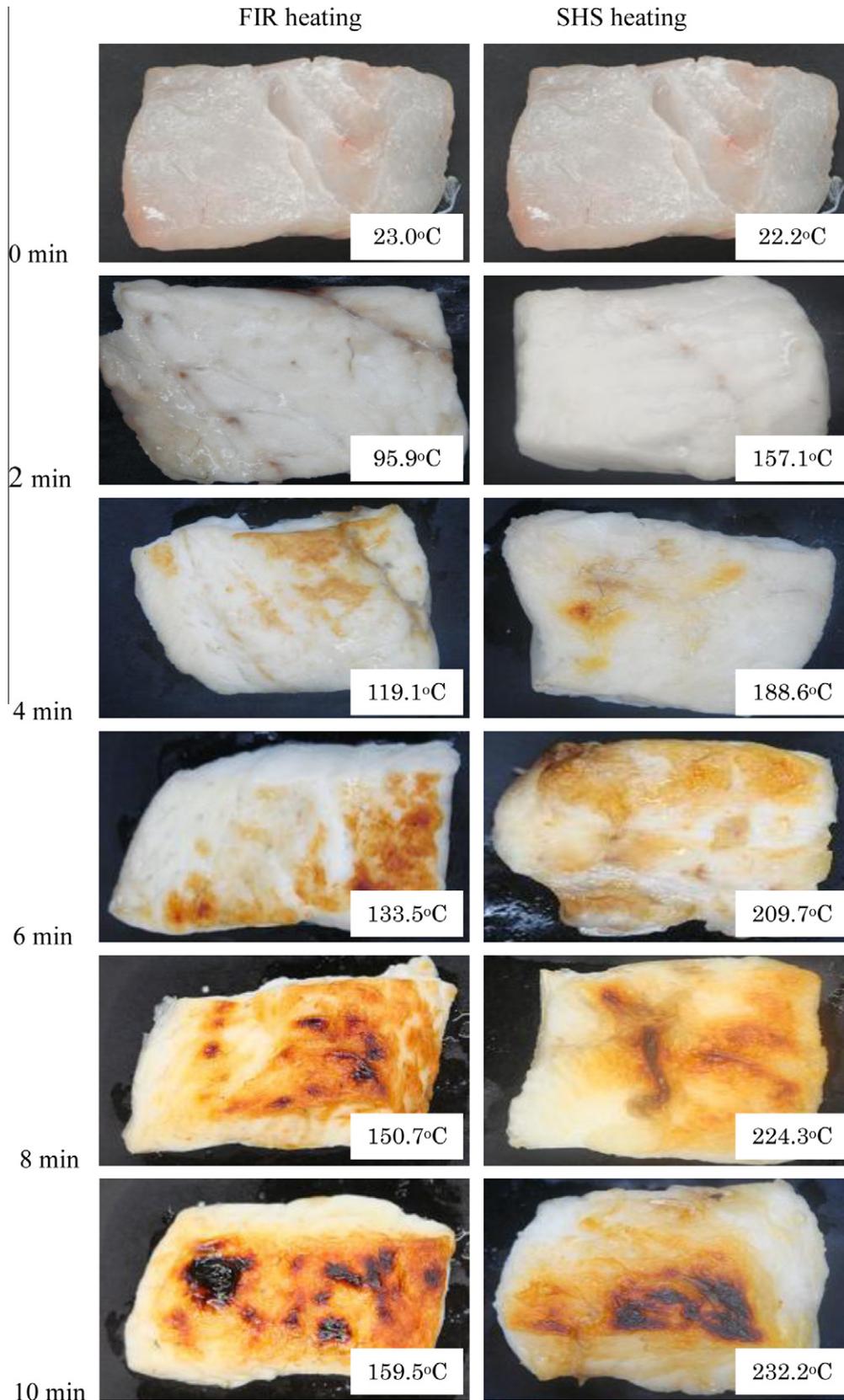


Fig. 3. Surface color and temperature changes of samples during grilling, at defined time steps. Comparison of far-infrared (FIR) and superheated steam (SHS) heating.

are confirmed in Fig. 3 by comparing the photographs of the color changes at the sample surface during FIR and SHS grilling.

To compare color changes, the trajectory through color space (using L^* , a^* , and b^* values as color-space coordinates) is frequently used. In this study, the browning colors induced in samples using different heat transfer systems during the grilling process were effectively compared. The L_m^* , a_m^* , and b_m^* plotted data (color values after protein denaturation occurs) used here were data obtained from FIR and SHS heating, along with the NIR heating results reported by Nakamura et al. (2011). The color changes follow almost the same spiral trajectory through the color space, regardless of the heat transfer mode. This result confirmed that the surface browning color developed in same way and is independent of the heat transfer mode, as was reported by Sato et al. (1992) and Shibukawa et al. (1989). Therefore, if L_m^* declines, a_m^* and b_m^* change accordingly, although it was observed that the heating rate accelerates or delays color changes. For a better visualization of the color behavior during grilling, two-dimensional projection drawings of the L^*-a^* , L^*-b^* , and b^*-a^* planes are shown in Fig. 4A–C for the

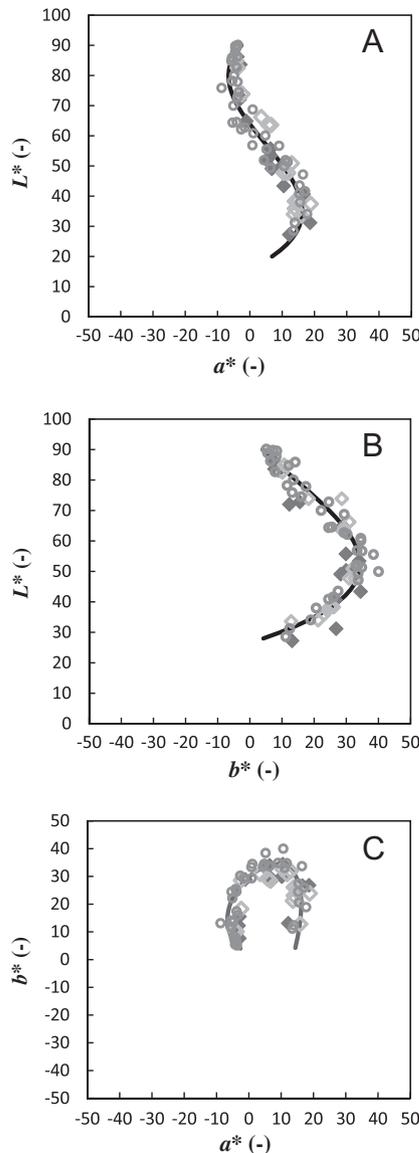


Fig. 4. The relationships between $L_m^*-a_m^*$ (A), $L_m^*-b_m^*$ (B), and $b_m^*-a_m^*$ (C). Solid line represents the correlation of NIR calculated values; \blacklozenge far-infrared, \blacklozenge superheated steam, \circ near-infrared.

three different heating methods (FIR, SHS, and NIR). Fig. 4A and B show the correlations between the measured data for L^*-a^* and L^*-b^* , respectively, plotted together with the reported data for calculated color values using NIR heating, shown by a solid black line. To estimate a_c^* and b_c^* , the empirical equations developed by Nakamura et al. (2011) were used:

$$a_c^* = 4.84 \times 10^{-4} \times (L_c^*)^3 - 8.7 \times 10^{-2} \times (L_c^*)^2 + 4.48 \times (L_c^*) - 54.2 \text{ at } 28 < L_c^* < 90 \quad (11)$$

$$b_c^* = 4.76 \times 10^{-4} \times (L_c^*)^3 - 1.14 \times 10^{-1} \times (L_c^*)^2 + 8.02 \times (L_c^*) - 141.4 \text{ at } 28 < L_c^* < 90 \quad (12)$$

where L_c^* was obtained from the E_a and k_0 kinetic parameters of the browning reaction during grilling. A good approximation to the measured data was found using the calculated values, even though they were obtained using different heat transfer systems. Thus, when the value of L_c^* decreases, the a_c^* and b_c^* values change simultaneously, according to Eqs. (11) and (12), respectively. The relationship between the measured values of a_m^* and b_m^* with the calculated NIR values (solid line Fig. 4C), confirms the results presented in Fig. 4A and B.

3.2. Kinetic analysis of browning reaction during grilling

In Fig. 5A and B, the measured data presented in Fig. 2A and B are shown together with the calculated values (L_c^* , a_c^* , and b_c^*), using FIR and SHS heating, respectively. The kinetics parameters (E_a and k_0) of the reduction rate of L_c^* during grilling using FIR and SHS

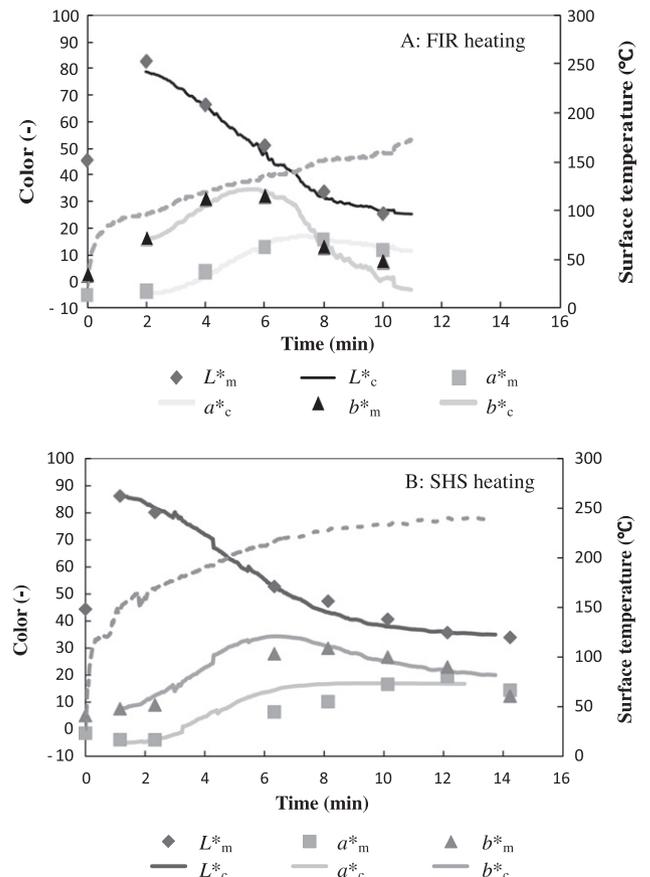


Fig. 5. Comparison of calculated and measured L^* , a^* , and b^* values. A: far-infrared (FIR). B: superheated steam (SHS). Dashed lines represent the surface temperature profile.

Table 1
Comparison of kinetics parameters, E_a and k_0 , of the reduction rate of L^* value during grilling using far-infrared (FIR) and superheated steam (SHS) heating.

	SHS ^a	FIR ^a
E_a (kJ mol ⁻¹)	31.5 ± 0.19	50.7 ± 0.20
k_0 (s ⁻¹)	8.2 ± 0.03	4759.0 ± 51.87

^a Mean ± standard deviation of the kinetics parameters values ($n = 3$).

heating are presented in Table 1. The curve of the L^* values was calculated by the RKG method, and drawn only at temperatures over 110 °C, because the color change is caused by the browning reaction from this level, as reported by Mondal and Datta (2008), Purlis and Salvadori (2009), Onishi et al. (2011), and Nakamura et al. (2011). The mean values of L_m^* , a_m^* , and b_m^* and the calculated ones were found to be in good agreement, in both cases. It was therefore demonstrated that color changes can be calculated from surface temperature using the method described above, regardless of the heat transfer mode.

For a better understanding of the comparison of the different heating modes, the same surface temperature (SHS heating profile) and the estimated k_0 and E_a kinetics parameters presented in Table 1 for each heating mode were used to calculate the L_{cs}^* values presented in Fig. 6. The results show a slower reduction of L_{cs}^* values using SHS than using FIR, and this is attributable to the significant differences (one-way analysis of variance, $p > 0.05$) between the kinetics parameters E_a and k_0 for the two types of heating.

Through an analysis of the effects of radiant and convective heat transfer modes on the browning of cookies during baking, Shibukawa et al. (1989) claimed that as the surface temperature increases, the surface color becomes darker, regardless of the heat transfer mode. They also found that the difference in color depended only on the surface temperature. Thus, the difference between the rates of color development for the two heat transfer modes was a result of the difference between the rates of increase of the surface temperature, even when the same air temperature was used in the oven. Similar results were obtained in the present study.

However, the atmosphere during grilling is important. The absence of oxygen (dioxygen, O₂) in the grilling environment can be one reason for the low color reduction rate under SHS heating. This condition cannot occur using FIR heating. O₂ participates in

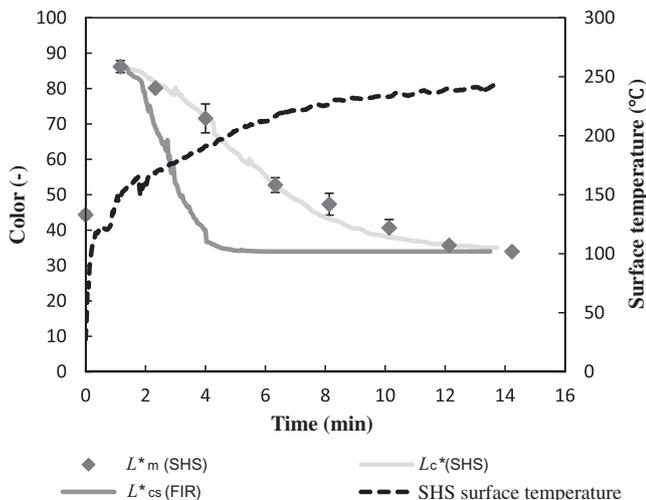


Fig. 6. Comparison of calculated L_{cs}^* values [far-infrared (FIR) and superheated steam (SHS)] for the same surface temperature. Bars indicate the standard deviation from three determinations.

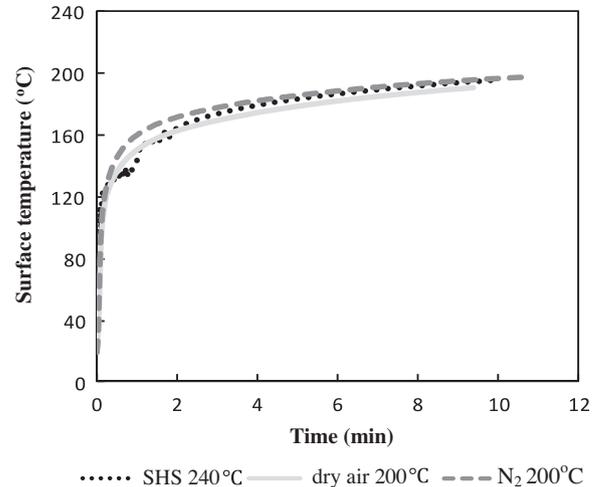


Fig. 7. Surface temperature histories of fish grilled under different heating media [superheated steam (SHS), dry air, and N₂].

the Maillard reaction, causing oxidation, and accelerates browning. Indrasena and Barrow (2011) claimed that a high temperature accelerates the oxidation of lipids if appropriate initiators and oxygen are present. Thus, the composition of the heating medium plays an important role in browning of fish during grilling.

3.3. Comparison of browning color during grilling under different heating media

The surface temperature histories of fish grilled under three heating media (SHS, dry air, and N₂) were obtained using a convection-type oven. The heat flow rate and the temperature setting were adjusted to obtain a similar surface temperature history, as shown in Fig. 7. The surface temperature profiles using dry air and N₂ were compared with the profile using SHS as the heating medium. It was observed that they were very similar, attaining an $|\overline{RE}|$ value of 2.7% with a standard deviation of 5.9% for dry air, and an $|\overline{RE}|$ value of 4.1% with a standard deviation of 1.4% for N₂. In the case of the SHS medium, the surface temperature was slightly higher in the early stages of heating compared with those in the other two media. Similar results for an SHS oven were reported by Ohishi and Shibukawa (2008). Above 130 °C, however, all of the surface temperature histories were similar. This last

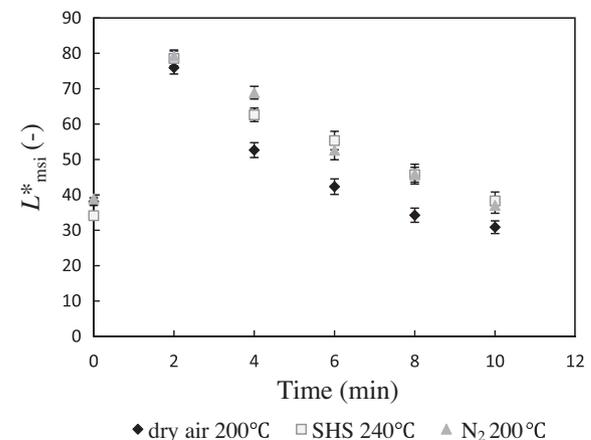


Fig. 8. Comparison of measured L_{msi}^* values of fish grilled using similar surface temperatures under different heating media [superheated steam (SHS), dry air, and N₂]. Bars indicate the standard deviation from three determinations.

region is recognized as the one affected by the browning reaction (Mondal and Datta, 2008; Purlis and Salvadori, 2009; Onishi et al., 2011; Nakamura et al., 2011).

Color changes during fish grilling under the three heating media at similar surface heating temperatures are presented in Fig. 8. In the dry air medium, the color changes showed a faster reduction of L_{msi}^* than those in the SHS and N_2 media. As explained above, this can be caused by the presence of remaining air and O_2 in the grilling environment. This condition cannot occur when N_2 is the heating medium, because the air in the environment is fully replaced by N_2 gas. Thus, the absence of O_2 in the heating medium could be the reason for the delay in the browning reaction during heating using N_2 . Similar results were observed for SHS, confirming the effective displacement of air by SHS.

4. Conclusions

The traditional method of grilling fish in convection air ovens, a widely used practice in the food industry, excessively darkens the fish surface. An alternative process for effective cooking is needed to obtain grilled fish of an attractive color. The present study therefore analyzed the use of SHS and FIR heating, and several heating media were tested. We obtained the following conclusions.

1. As browning occurs, the trajectory through color space followed almost the same pattern under radiant FIR heating as that under convective SHS heating, regardless of the sample surface temperature.
2. Color changes were appropriately estimated. The reduction in the L_c^* value was successfully calculated using the surface temperature and estimated kinetics parameters; a_c^* and b_c^* values were calculated using empirical equations, and were in good agreement with the measured color, and independent of the heat transfer mode.
3. The composition of the grilling environment could be the reason for the differences between color development in the heat transfer modes evaluated in this study.
4. The effect of the heating medium on color changes shows reductions in L_{msi}^* values in the order dry air > N_2 \approx SHS. The absence of O_2 in the heating medium was recognized as the reason for the delay in the browning reaction during heating using N_2 and SHS.

Acknowledgements

This work was supported in part by a Grant-in-Aid for Scientific Research (C) from the Ministry of Education, Culture, Sports, Science and Technology of Japan (21500753).

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