DETERMINATION OF ACRYLAMIDE AND HYDROXYMETHYLFURFURAL (HMF) VALUES AS AFFECTED BY FRYING DURATION AND TEMPERATURE LEVELS DURING THE PREPARATION OF TRAY KADAYIF DESSERT

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Abstract. In this study, a wire kadayıf on a tray was fried at 175, 200 and 225 °C for 40, 50 and 60 min. The temperature levels affected hydroxymetylfurfural (HMF) content, color values (L, a, b), outward appearance, interior color, taste, texture, aftertaste and general acceptability values at significant levels ($p \leq 0.01$); as well as pH and aroma ($p \leq 0.05$). As the temperature increases the HMF content, color value a, outward appearance, interior color, taste, odor, aroma, texture, aftertaste and the general acceptability values have increased. Whereas the moisture, pH and L color values decreased. The frying time had an effect on moisture. The changes in the color brightness (L), red color (+a), and the yellow color (+b) values of the kadayıf were highly significant ($p \leq 0.01$); and the changeswere also significant ($p \leq 0.05$) on the HMF content, texture, aftertaste and general acceptability values. As the frying time increases HMF content, a color value, texture, aftertaste and the general acceptability values increased whereas the moisture, L and b color values decreased.
Keywords: *acrylamide, HMF, frying temperature, frying duration, tray kadayıf*

Introduction

Kadayıf is a delicious Turkish sweet pastry, consumed in different provinces of Turkey such as Mersin, Hatay, Erzurum, Diyarbakir, and Gaziantep. Tray kadayıf and creamy kadayıf can both be prepared with either whole hazelnut, walnut or pistachio (Anon, 2012). According to the “standard of tray kadayıf” (T.S-10344, 1992), it is a semi-processed product made by adding drinking water to sifted flour and baking it on a plate. However, tray kadayıf is offered in raw and fired varieties in markets. Flour for making Kadayıf should have the following properties: humidity 14.5% (max), protein 8-10% (F = 5.70), the essence of age 22% (max), water holding capacity 54% (max), stability 2 min (max), softening temperature 150 BU (max) and minimum value of the biscuits perchance show maximum level in kadayıf. In recent years, tray kadayıf has mostly been produced in small enterprises, but has also been manufactured in large quantities to a lower extent (Pekak, 2006).

Acrylamide was first prepared in 1893 by Moureu at 10 °C on a saturated solution of acrylic chloride in benzene, followed by adding dry ammonia and boiling for mean duration, then ammonium chloride was refluxed by filtrating, cooling, and precipitating acrylamide (Carpenter and Davis, 1957). Neurotoxic effects of acrylamide were detected in a laboratory with animal testing. In addition, genotoxic and carcinogenic effects of acrylamide have been identified in recent years, its presence in many processed foods has been a hazard to human health (Joint, 2005). Acrylamide as a chemical substance was synthesized in 1949, it is white, odorless, crystalline, solid and

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soluble in water (255 g/L of water) (Thomas and Thomas, 2012), its melting and boiling point is 87.5 °C and 125 °C, respectively (Gölükçü and Tokgöz, 2005). It has no burn inducing or irritating properties, low acidity, and weak basic characters. It has a monomeric structure of two different functional groups; double bond and weak amid group as the electron, and these groups are joined through chemical reactions (Can, 2007). In food items, acrylamide has a simple structure which occurs as a result of reactions taking place among carbohydrates, proteins, amino acids, lipids, or other food components (Gölükçü and Tokgöz, 2005). It is usually formed during frying, baking, and roasting (Svensson et al., 2003). The content of acrylamide in the product can be identified by some factors, such as preparation methods, compositions, ambient temperature, and pH. Asparagine appears to be the primary amino acid involved in the production of acrylamide via the Millard reaction (LoPachin and Canady, 2004).

According to Zyzak et al. (2003), there is a linear relationship between free asparagine contents and forming of acrylamide in the product. Gölükçü and Tokgöz (2005) indicated that asparagine content of the product is the most important factor in the formation of acrylamide in cereals. The duration of heating initiates the Millard reaction, in which sugars (simple monosaccharides capable of carrying out reduction reactions) present in starchy foods are reduced with amino acids to produce acrylamide. Applying high temperature (more than 120 °C) caused a raise in the acrylamide contents above 1 mg/kg in food items with high contents of carbohydrates. Therefore, there is a relationship between acrylamide in the food and temperature. Controlling the temperature range can reduce the amount of acrylamide formation. Food which is prepared through boiling has a low content of this compound, thus it should be preferred to frying, baking, roasting, and grilling; using methods such as frying or roasting at elevated temperatures for a long duration should also be avoided (Gölükçü and Tokgöz, 2005).

Beside acrylamide, hydroxylmethylfurfural (HMF) is another harmful compound which occurs as a result of the Millard reaction in biscuits. HMF is formed from an aromatic alcohol, aromatic aldehyde, and furan ring, and as an intermediate product in the hydrolysis of hexose in an acidic medium or in the Millard reaction (Seyyed Cheraghi, 2014). The concentration of HMF varies significantly and in some food items it can even reach 1 g/Kg (Rada-Mendoza et al., 2004; Akkan et al., 2001). The formation and amount of HMF are affected by temperature and pH value, also the quality deterioration of a wide range of edibles is determined by Hydroxymethylfurfural (HMF) (Gökmen and Şenyuva, 2006). Beside the harmful compounds which are the result of Maillard reactions, antioxidant compounds are also formed (Yıldırım, 2010). Some researchers have stated that HMF has possibly genotoxic and mutagenic effects on human cells, possibly inducing liver and colon cancer (Svendsen et al., 2007; Monien et al., 2012; Zhang and An, 2017); so the objective of this study was to determine the effect of temperature and duration on acrylamide and hydroxymethylfurfural formation during the preparation of tray kadayıf.

Material

As the ingredients of tray kadayıf, butter and hazelnut were acquired in the markets (Erzurum, Turkey). The ratio of grout which has been used was 2:1 sugar mixed with water. After boiling, approximately (2.0 ml) lemon juice was added to prevent

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crystallization. The tray kadayıf had the following values: pH = 6.43, humidity = 20.53%, L (96.19), +a (0.53) and +b (16.89).

Method

Processing of tray kadayıf

200 g of butter was melted on a very low temperature and was then mixed with the kadayıf for a 4-5 min period; and about 500 g was laid with a homogeneous thickness. After seating on the tray, a special medium-density fiberboard (MDF) wood and an overall 10 kg heavy weight was placed on it. The tray kadayıf was fried at 175 °C, 200 °C and 225 °C for 40, 50, 60 min respectively. After that, the previously prepared syrup was poured onto it; then sliced and crushed nuts were placed on top, and was then presented to panelists. The analysis samples were prepared from the upper part of the fried tray kadayıf samples after grounding it into very small pieces.

The analyses performed on tray kadayıf samples

Measurement of the color intensity

In the above-mentioned samples, the color densities, which are brightness of kadayıf color (L; 0-100: dark-bright) red color (+a), yellow color (+b) were measured using three parallel Minolta Colorimeter device. Results were evaluated according to the International Commission on Illumination (CIE Lab, Commission Internationale de l’Eclairage; Anonymous, 2012).

Acrylamide analysis

The analysis of acrylamide was performed according to Robarge et al. (2003). During the preparation of the sample the following steps were taken.

The acrylamide standard solutions, used as the sample calibration solutions were 1000 ppb, 500 ppb, and 50 ppb 100 ppb, and were subjected to the same processes to obtain a calibration curve.

Extraction: very small particles of tray kadayıf samples (1 g) were put into 10 ml demineralized water in a 50 ml Erlenmeyer, then was magnetically stirred for 20 min, at 5500 rpm and centrifuged for about 10 min. After centrifuging the supernatant, it was filtrated through a 0.45 µm nylon filter, then 200-300 µL 0.1 normality (N) potassium bromate ($KBrO_3$) was slowly added to 3 ml of the filtrate, followed by stirring and 1 hour of rest in an ice bath. The ice bathing tubes received one drop of 1.0 N sodium thiosulfate ($Na_2O_3S_2$) and were mixed slowly with, followed by adding 2 ml of ethyl acetate ($C_4H_8O_2$). The tubes which had Ethyl acetate added were centrifuged at 5500 rpm for an additional 10 min, then the vials were transferred for supernatant analysis.

Sample extracts and the standard solution were measured using a Gas Chromatography-Mass Spectrometry (GC-MS, Agilent) analysis device. Positron effect was analyzed with imaging method of the GC-MS, DB-225 column (30 m × 0.25 mm × 0.25 µm) and helium gas. The oven temperature was increased from 40 to 200 °C and in this analysis set which increasing 30 °C in a minute split-less mode is used.

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Sensory analysis

The sensory analysis of tray kadayıf was carried out at the laboratory of the Faculty of Agriculture, Food Engineering Department, Ataturk University, and panelists were selected from among the graduate and postgraduate students of the Food Engineering Department faculty. Slides which were cut from each tray kadayıf were served on coded plastic plates with drinking water to eight panelists, who were asked to evaluate them. The tray kadayıf was left waiting for 24 h at room temperature. Tray kadayıf samples' taste, aroma, color, texture and overall acceptability were subjected to evaluation. There was a 9-point hedonic scale for the evaluation of these parameters (1 = very bad, 9 = very good) for the panelists to rate each tray kadayıf (Kramer and Twigg, 1973).

Determination of hydroxymethylfurfural (HMF)

Hydroxymethylfurfural analysis was performed with a High-Performance Liquid Chromatography (HPLC) on samples prepared according to the method given by Rada-Mendoza et al. (2002). According to the aforementioned method, a 2 g sample was transferred to a 50 ml Erlenmeyer, to which 4 ml each of carrez I [15 g potassium hexacyanoferrate (II) trihydrate ($K_4[Fe(CN)_6] \times 3H_2O$) mixed with 100 ml ultra-pure water] and carrez II [30 g zinc sulfate heptahydrate ($ZnSO_4$ x $7H_2O$) mixed with 100 ml ultra-pure water] were added and flask was filled to 50 mL with deionized water. After stirring for 30 min, the contents of the Erlenmeyer were passed through a 0.45 µm filter. The samples were analyzed using the HPLC, Agilent 1100 system. UV-VIS detector and Nucleosil 5C18 (250 × 4,6 mm) column (Hichrom, Reading Berkshire, England) were used in this system. The samples were then placed at room temperature, and 10:90 (v/v) methanol-water was used with a flow rate of 1 ml/min to keep it mobile. Hydroxymethylfurfural concentration curve was measured using external calibration (55690-5-HMF, Fluka Chemika) at 280 nm.

Determination of pH

The homogenized 3 g tray kadayıf sample was placed into pure water and its pH was assessed. The sample's pH was previously measured to be pH 4.00 and pH 7.00 using an INOLAB pH 720 brand of Microprocessor (Torley et al., 2008).

Determination of dry matter

Homogenous samples of about 8-10 g were taken from the total samples of both acrylamide and HMF and then dried in a drying oven at 110 °C until a constant weight was achieved (Kotancılar, 2013).

Experimental plan

Trials were conducted with factorial arrangement by three different frying temperatures (175, 200 and 275 °C) and three different frying durations (40, 50 and 60 min) in complete randomized design (CRD) with two replicates (3 × 3 × 2).

Statistical analysis

The raw values received from trials were subjected to variance analysis in SPSS (SPSS1999), and the mean of the sources of the main variation has been found

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important, as compared with Duncan Multiple Comparison Test (Yıldız and Bircan, 2003).

Results and discussion

Acrylamide values in tray kadayıf

In the present study, variance analysis (*Table 1*) indicated that there were significant ($p \leq 0.01$) differences among different temperatures and different durations based on acrylamide values. As shown in *Table 2,* the temperature increase does not cause any significant changes on the acrylamide content, as the lowest amount of acrylamide was (130.5 µg/kg) at 200 °C, and the highest amount was (137.7 µg/kg) at 175 °C. Mottram et al. (2002) determinate that in the asparagine/glucose model systems the increase of temperature from 120 °C to 170 °C increases the amount of acrylamide and above 170 °C it is reduced. The acrylamide content increases with the frying duration. The lowest amount of acrylamide (132 µg/kg) was determined after 40 min and the highest amount (137 µg/kg) was obtained with 60 min of duration. Tuta (2009) stated that if the temperature rises above 100 °C and the duration of frying increases in parallel, then the acrylamide content decreases. The highest value was achieved with a high-temperature heat treatment of low duration.

Table 1. *Variance analysis of acrylamide, L, +a, +b color values at different temperatures and duration*

SOV	SD	F			
		Acrylamide	**L**	**+a**	**+b**
Frying temperature (°C)	2	2.54	410.00**	12810.00**	3184.00**
Frying duration (S)	2	1.06	120.00**	3253.00**	976.00**
C × S	4	1.29	20.00**	59.00**	1467.00**
Error	9				

*, **: Significant at $p \leq 0.05$ and $p \leq 0.01$, respectively

Table 2. *Mean comparison of acrylamide, L, +a, +b color values at different temperatures and duration*

Frying temperature (°C)	n	Means			
		Acrylamide (µg/kg)	**L**	**+a**	**+b**
175	6	137.70 a*	66.67 a	2.72 c	25.10 b
200	6	130.50 a	60.16 b	7.81 b	26.77 a
225	6	135.10 a	45.05 c	13.52 a	15.81 a
Std. error		± 2.3	± 0.547	± 0.048	± 0.105
Frying duration (S)	**n**				
40	6	132.00 a	63.36 a	5.45 c	25.44 a
50	6	134.00 a	57.16 b	7.72 b	23.23 b
60	6	137.00 a	51.36 c	10.87 a	19.01 c
Std. error		± 2.3	± 0.547	± 0.048	± 0.105

*The average shown with the same letters are statistically indistinguishable from each other ($p < 0.05$)

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Color parameter's values of the tray kadayıf

Variance analysis (*Table 1*) of the tray kadayıf L (Brightness), +a (Redness), and +b (Yellowness) color values influenced by the frying temperature, duration and C × S interactions was highly significant ($p \leq 0.01$). The important variables of the frying temperature and duration including L, +a, and +b color values can be found in *Table 2*. The lowest amount of L color value (45.05) was assessed at 225 °C and the maximum amount (66.67) was obtained at 175 °C temperature. By increasing the duration of frying, L color values decreased. The lowest L color value (51.36) was measured after the 60 min mark and the maximum amount (63.36) was obtained after 40 min of duration. The lowest amount of +a color value (2.72) was detected at 175 °C and the maximum amount (13.52) was obtained at 225 °C temperature. By increasing the frying duration, +a color value would increase. The lowest amount of +a color value (5.45) was assessed after 40 min and the highest amount (10.87) was obtained at the 60 min mark. The lowest amount of b color value (15. 81) was measured at 225 °C and the highest amount (26.77) was obtained at 200 °C temperature. By increasing the frying duration, b color values decreased. The lowest amount of b color value (19.01) was detected after 60 min and the maximum amount (25.44) was obtained during the 40 min duration. The interaction between C and S, and its effect on L, +a, and +b color values are given in *Table 1*.

The result showed that the reduction in the L color value as a result of the increased temperature caused the tray kadayıf to gain a dark color. In parallel, +a surface color value has increased and has become more of a reddish color, while in +b value there was an increase at first, followed by a serious decline. While o the dark and intense red color was obtained at 225 °C the brightest and least red surface color was obtained at 175 °C. Colored compounds formed as a result of Maillard reaction are high molecular weight macromolecules called Melanoidins and low molecular weight molecules which have two or three heterocyclic rings (Ames et al., 1998). Color formation, temperature, duration, increase with pH and moisture content in the medium (aw = 0.3 -0.7). As shown in *Table 2*, with the increase in temperature and duration caused a decrease in the L value of tray kadayıf, which means the surface color got darker. In parallel, the increase in a value was observed, which means that the intensity of red increased. The brightest and pinkish colors were obtained when the tray kadayıf was fried for 40 min and the darkest red was obtained by frying for 60 min. By increasing frying duration b color value decreased. A decrease was observed in yellow color as well. In a study by Özkaynak (2006) each of the three oil varieties developed darker colors depending on the frying duration as a result of variations in the formation of acrylamide, and L was found to decrease.

Sensory analysis values of tray kadayıf

Variance analysis values of appearance, interior color, taste, odor, flavor, texture, aftertaste and overall acceptability are shown in *Table 3*. The effect of frying temperature on appearance, interior color, taste, texture, aftertaste and the overall acceptability of tray kadayıf was statistically highly significant ($p \leq 0.01$) whereas that on the aroma and smell was significant at a different level ($p \leq 0.05$). The frying duration's effect on the overall acceptability was highly significant ($p \leq 0.01$); on the texture and aftertaste it was significant ($p \leq 0.05$) while on the appearance, interior color, test, smell and aroma it was not significant. The effect of the interaction between

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frying temperature and frying duration on the outward appearance of tray kadayıf, taste, texture, aftertaste and the general acceptability was highly significant ($p \leq 0.01$), while that on smell value was significant ($p \leq 0.05$) and was not significant on the interior color and the aroma. Significance values of frying temperature can be seen in *Table 3*, while the Duncan multiple comparison test results are shown in *Table 4*.

Table 3. *Variance analysis of tray kadayıf features at different temperatures and durations*

S.O.V	SD	F							
		Appearance	Interior color	Taste	Smell	Aroma	Texture	Aftertaste	General acceptability
Frying temperature (°C)	2	82.08**	41.17**	8.08**	3.56*	4.77*	39.56**	15.13**	12.99**
Frying duration (S)	2	1.25	3.33	1.47	0.22	1.90	4.27*	4.72*	3.51**
C × S	4	13.40**	2.71	8.00**	3.93*	1.60	7.76**	7.99**	9.75**
Error	9								

*, **: Significant at $p \leq 0.05$ and $p \leq 0.01$, respectively

Table 4. *Mean comparison of tray kadayıf features at different temperatures and durations*

Frying temperature (°C)	n	Means							
		Appearance	Interior color	Taste	Smell	Aroma	Texture	Aftertaste	General acceptability
175	6	2.83 c	3.79 c	4.87 b	5.21 b	4.67 b	4.33 c	4.38 b	4.50 b
200	6	5.96 b	6.08 b	5.87 a	5.96 ab	5.88 a	5.67 b	5.79 a	5.96 a
225	6	7.00 a	7.54 a	6.21 a	6.25 a	6.04 a	6.75 a	6.17 a	6.04 a
Std. error		± 0.24	± 0.29	± 0.24	± 0.34	± 0.34	± 0.19	± 0.24	± 0.24
Frying duration (min)	**n**								
40	6	5.00 a	5.63 a	5.30 a	5.58 a	5.00 a	5.16 b	4.87 b	5.00 b
50	6	5.58 a	5.68 a	5.70 a	5.89 a	5.67 a	5.62 ab	5.54 ab	5.62 ab
60	6	5.18 a	5.79 a	5.95 a	5.93 a	5.92 a	5.96 a	5.91 a	5.87- a
Std. error		± 0.24	± 0.29	± 0.24	± 0.34	± 0.34	± 0.19	± 0.24	± 0.24

*The average shown with the same letter are statistically indistinguishable from each other ($p < 0.05$)

As shown in *Table 4*, increase in the temperature of tray kadayıf influenced the appearance, interior color, taste, odor, flavor, texture, and aftertaste positively and was also more appreciated by panelists. While the highest values of appearance, interior color, taste, odor, flavor, texture, aftertaste and general accessibility were obtained at 225 °C, the least admired was the tray kadayıf fried in 175 °C.

HMF, pH, and surface moisture values of tray kadayıf

Variance analysis of HMF, pH and the surface moisture under different frying temperatures and durations of tray can be seen in *Table 5*. As shown in *Table 5* the effect of frying temperature on HMF and humidity values was highly significant ($p \leq 0.01$), while on pH value was significant ($p \leq 0.05$). The effect of frying duration on humidity values was highly significant ($p \leq 0.01$) while on HMF values was significant

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($p \leq 0.05$). As shown in *Table 6*, the increase in the temperature caused an increase in HMF values (the lowest value of HMF was achieved at 175 °C, while the highest value was observed at 225 °C), and a decrease in pH and the amount of humidity on the surface. Depending on the increase in the temperature the moisture content decreased accordingly. Normal moisture content in particular decreased at temperatures above 50 °C and pH 4-7, while HMF formation rate increased (Borrelli et al., 2002; Oral, 2011).

Table 5. *Variance analysis of HMF, PH and humidity values under different temperatures and durations*

S.O.V	SD	F		
		HMF	pH	Humidity
Frying temperature (C)	2	14.34**	5.59*	239.00**
Frying duration (S)	2	2.89*	2.22	108.00**
C × S	4	1.40	0.64	24.00**
Error	9			

*. **: Significant at $p \leq 0.05$ and $p \leq 0.01$, respectively

Table 6. *Mean comparison of HMF, PH and humidity values under different temperatures and durations*

Frying temperature (°C)	n	Mean		
		HMF (µg/kg)	pH	Humidity (%)
175	6	8.70 b	5.70 a	2.29 a
200	6	54.30 b	5.60 b	0.78 b
225	6	342.90 a	5.50 b	0.56 c
Std. error		± 47.8	± 0.1	± 0.1
Frying duration (min)	**n**			
40	6	53.00 b	5.68 a	1.87 a
50	6	135.00 ab	5.61 a	1.16 b
60	6	216.00 a	5.52 a	0.61 c
Std. error		± 47.8	± 0.1	± 0.1

*The averages shown with the same letters are statistically indistinguishable from each other ($p < 0.05$)

The increasing in frying duration lead to a pH decrease. However, this change was not statistically significant. The HMF value proved to be dependent on frying duration. The lowest HMF value was measured during the 40 min frying period, while the highest value was detected after the 60 min cooking (*Table 6*). Cooking duration in the production of dry fruit pulp is extremely important. The determination of HMF and acrylamide contents significantly increased with the increase in cooking duration (Boz, 2012). As a result of the increase of frying duration, moisture content decreased. Elmore et al. (2005) investigated how much the moisture content depends on frying duration of baked cakes of rye and wheat flour and potatoes. The moisture contents of potatoes and cakes made from rye and wheat flour in the first minute were 59.73, 41.60, and 39.60, respectively. With the 10 min duration, the ratio under 180° C decreased to 24.25, 3.86,

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and 10.33. After 50 min, it decreased to 0.75, 0.70, and 0 (Elmore et al., 2005). The reduction was observed in the moisture content when the temperature of tray kadayıf was increasing.

As displayed in *Table 6*, with the increase of temperature, at the end of all three periods there was an increase in moisture content of tray kadayıfs. At 175 °C, 200 °C and 225 °C and with 40 min of frying duration the highest moisture content, but after 60 min it reached the lowest value. These properties were observed after the 50 and 60 min periods. When the temperature was raised from 175 °C to 200 °C, there was a rapid decrease in the moisture content. At 220 °C and 40 min of frying, there were not many changes to moisture content.

Conclusions

None of the factors has a statistically significant effect on the value of acrylamide. Increase in temperature does not cause any change on acrylamide content. While raising frying duration caused an increase. Increase in temperature levels of tray kadayıf reduced its *L* color value and a darker color was obtained.

The increase in temperature levels, caused an increase in HMF values, a decrease in pH and moisture content on the surface. Depending on the temperature rise a reduction in moisture content was observed. Increasing the frying duration caused an increase in pH values. Depending on higher temperature levels and longer frying duration, HMF values raised. With the increase of frying duration, the moisture content was reduced.

In this research, *a* color value of the surface increased and the kadayıf appeared much redder. In *b* color value there was an increase at first and then it had a serious decrease. Raising frying duration caused a decrease in *L* color value of tray kadayıf, which means that the surface color got darker. In parallel, an increase in the *a* color value was observed, which meant a more intensive red color. The *b* color value decreased by increasing frying duration. An increase in temperature levels had positively influenced the appearance, interior color, taste and smell and caused it to be appreciated by panelists. Increasing the duration of frying had no effect on the aroma of tray kadayıf and had a positive effect on texture, aftertaste and general acceptability.

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