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Effects of Microwave Drying on Lipid Oxidation of Stuffed Pasta

Teresa De Pilli · Roma Giuliani · Antonio Derossi · Carla Severini

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Abstract The aim of this work was to evaluate and compare the effects of conventional (hot air) and microwave drying on the lipid oxidation of stuffed pasta. Experiments were carried out on the pasta type ravioli dried by conventional and microwave treatments. Peroxide values and induction times were evaluated for the oil extracted from the stuffing. Peroxide values of the lipid extracts for the samples dried by conventional drying and microwave drying were similar (P-level = 0.51) and less than threshold acceptable value (25 mequiv O_2/kg oil). The use of microwave processing could be a remarkable energy saving measure as well as a way of lowering production costs without compromising product quality.

Keywords Drying · Lipid oxidation · Microwave · Stuffed pasta

Introduction

Industrial food processing carried out by microwaves may be classified in seven major unit operations: blanching, cooking, baking, drying, pasteurization, sterilization and tempering. Only few of these operations have been successful in food industry owing to the high costs of the plants and the non-homogeneous electromagnetic distribution in food. The different shapes and sizes of food differently influence the electromagnetic field distribution and thus the heating pattern.

The temperature distribution in food heated with a microwave oven is different from that corresponding to a conventional treatment which is also characterized by a non-homogeneous temperature pattern. In fact, in microwave-heated foods, the differences of temperature between the core and the surface is less significant since the heating occurs from the inner core to outer parts. So, other variables such as size, shape, water and salt contents become very important [1].

The most important characteristics resulting from the physical properties of microwaves and their interactions are the rapidity of heating and temperature heterogeneity different from that observed in conventionally heated products, thus differently affecting convenience, organoleptic quality and safety.

In order to satisfy consumer demand, new products have been put on sale, in particular pasta with different stuffings such as green vegetables, cheese, meat and their combinations. It is well known that a lot of organoleptic, physical, chemical and nutritional changes are deeply influenced by variables such as temperature, moisture and time depending on the method used for drying. Practically all quality attributes of food can be affected by food processing. Among the numerous reactions that take place during food processing, lipid oxidation has a determining role for the quality and safety of food. For example, the change of aroma as the result of new volatile odorous compounds, the flavour modifications caused by hydroxyl acids, the colour darkening as the result of condensation reaction between oxidation products and proteins, and finally, the new texture attributed to the oxidative induction of protein cross-linking are all results of lipid oxidation. The food, as a whole, is a particularly complex chemical

T. De Pilli (✉) · R. Giuliani · A. Derossi · C. Severini
Department of Food Science, University of Foggia,
Via Napoli 25, 71100 Foggia, Italy
e-mail: t.depilli@unifg.it

R. Giuliani
e-mail: ro.giuliani@unifg.it

matrix and it is clear that no one mechanism can be held exclusively responsible for the initiation of the lipid oxidation. Fundamental differences exist between the oxidation of lipids at high temperatures and the oxidation at room temperatures. The thermal activation can drastically change the further reaction pathways of the initial oxidation products [2]. The aim of this study was to evaluate and compare the effects of the conventional (hot air) and microwave drying on the lipid oxidation of stuffed pasta.

Material and Methods

This work was carried out on a model of stuffed pasta, type ravioli, made up of a sheet of dough, based on semolina-water, and a stuffing, based on olive oil, guar gum and water.

Raw Materials

Dough: durum wheat semolina (Divella S.p.A., Rutigliano, Bari, Italy), purchased on the local market, and running water were used.

Stuffing: extra virgin olive oil (De Santis, Bitonto, Bari, Italy), guar gum (Sigma-Aldrich Co., St. Louis, USA) and running water were used.

The untreated extra virgin olive oil showed the following oxidation index: 10.24 ± 0.85 peroxide value (mequiv O_2/kg oil) and $1,395 \pm 0.5$ induction time (minutes).

Samples Preparation

Sheet of dough: the dough was manually prepared with semolina and water (2:1 w/w); the water was slowly added favoring a better imbibition of semolina and obtaining an elastic and machinable dough. The sheets of dough were made using a rolling mill Imperia mod. SP 150 (IPS, Torino, Italy) with a manual thickness-selector at six positions. In order to obtain a sheet with a thickness of 1 mm, the dough was gradually laminated starting from the first position (corresponding to a thickness of about 6 mm) up to fifth position.

Stuffing: olive oil, gum guar and water for a total weight of 1 g were used. The ingredients were manually mixed to favor a homogeneous distribution of thickener. The gum guar and water ratio was maintained constant (1:1 w/w), while the ratios between olive oil and the mix were respectively, 2:1, 3:2, 1:1, 2:3, 1:2 (as they result from the factorial designs reported in Tables 1, 2).

Ravioli: a classic inox-aluminium ravioli mould (Raviolamp 36, Imperia Trading S.r.l., Torino, Italy) was used. The ravioli were prepared putting a sheet of dough on the mould, 1 g of stuffing into each piece and a second sheet as covering. The upside-down mould was submitted to a light pressure to easily separate the ravioli.

Table 1 Central composite design (A) relating to samples dried by hot air

Levels	Temperature (°C)	Pasta moisture (%)	Oil (%)
+1.681	86.81	24.043	66.81
+1	80	22	60
0	70	19	50
−1	60	16	40
−1.681	53.19	13.957	33.19

Table 2 Central composite design (B) relating to samples dried by microwave

Levels	Power (%)	Pasta moisture (%)	Oil (%)
+1.681	76.81	24.043	66.81
+1	70	22	60
0	60	19	50
−1	50	16	40
−1.681	43.19	13.957	33.19

Samples were dried using a climatic room Binder mod. KBF 240 (Tuttlingen, Germany) according to the following processing parameters:

- temperature range 53.18–86.82 °C ± 1;
- relative humidity of drying air 40%;
- air flow in the drier 100 m^3/h;
- drying time range 34–510 min.

A Sfornatutto DeLonghi mod. Combi&Functions Convenction microwave oven (Milano, Italy) with a maximum power of 850 watt was used to dry samples by microwave. The power percentage was varied through digital device.

Samples of pasta were placed on a metal mesh screen (1 cm pitch) in a single layer both into climatic room and microwave oven (Photo 1) prior to drying.

In order to estimate the maximum temperature attained by microwave, graduated heat sensitive paper strips (VWR International mod. MELB1762/MELB1763, Milano, Italy) were used. The maximum pasta temperature and drying time ranged from 95 to 152 °C and from 66 to 160 s, respectively.

After drying, pasta samples were immediately submitted to analyses. First of all the ravioli were manually opened in order to separate sheet from stuffing. Then, the sheet obtained was finely ground (particles < 300 μm) in a BUHLER ML 1204 mill (Germany). Moisture content and absorbance at 420 nm analyses were carried out on powdered sheet of pasta. The stuffing was subjected to oil

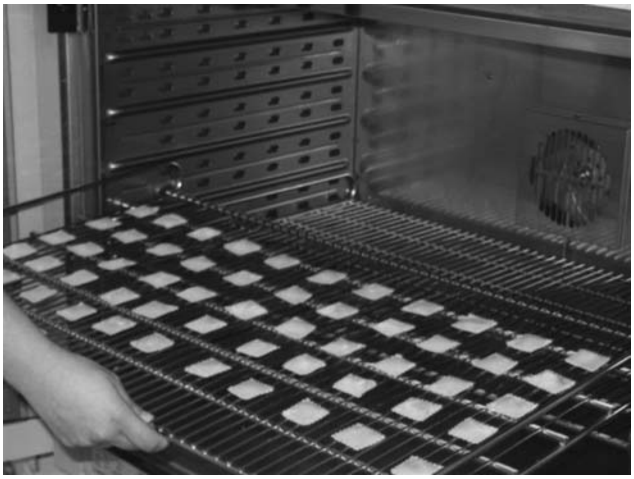

Photo 1 Pasta dried on a metal mesh screen

extraction. Stuffing (50 g) and hexane (300 mL) were placed into a 500 mL beaker and stirred for 30 min at room temperature. The mixing was filtered by a Whatman #1 filter paper to remove the solid portion. Then, solvent was evaporated under vacuum by a Rotavapor Büchi mod. R-114 (Switzerland) at 40 °C (bath temperature). The oil extract was submitted to the Rancimat test and peroxide value analyses.

Moisture %

The moisture content was determined according to the AACC methods [3].

Analyses of the Pasta Sheet

Absorbance at 420 nm

The spectrophotometric analyses were performed on the powdered sheet of pasta. The powder (10 g) was mixed with distilled water (50 mL) with a laboratory blender for 15 min (Warning Commercial, Torrington, CT, USA). The homogenate was centrifuged (ALC mod. 423R, Milan, Italy) for 10 min at 5,000 rpm at 24 °C and the liquid phase was filtered through a Whatman No.°1 paper. The absorbance values at 420 nm were measured using a UV/Vis spectrophotometer (Beckman DU 640, Fullerton, CA, USA).

Analyses of the Pasta Stuffing

Peroxide Value

The peroxide value was determined according to the AOCS methods [4] and results were expressed as mequiv of active oxygen/kg of oil.

Rancimat Test

Induction time was calculated by a Metröhm Rancimat mod. 743 (Herisau, Switzerland) and the results were expressed in minutes according to Läubli and Bruttel [5].

Oil extracts (3 g) were subjected to accelerated oxidation at high temperature (110 °C) under forced oxygenation (20 L/h). The oxidation curves obtained from the Rancimat test describe the resistance of oil to oxidation [6].

All the analyses were performed in duplicate.

Experimental Design

Two factorial designs, relating conventional and microwave drying, at three variables and five levels obtained by Central Composite Design (CCD) are reported in Tables 1 and 2 [7]. They were used to evaluate both the single influence of each processing variable and their possible interactions on lipid oxidation. Drying temperature (factorial design relating to conventional drying), power percentage of microwave oven (factorial design relating to microwave drying), moisture of pasta and olive oil percentages (both factorial designs) were the considered variables.

Seventeen trials with different combinations of the process variable values for each factorial design were obtained using the following equation: $n_{tot} = n_0 + n_c + n^* = 8 + 6 + 3 = 17$ where $n_0 = 2^n$ (n is the number of variables), n_c is the number of central point and n^* is the number of star point. The 17 combinations obtained for each Central Composite Design are reported in Tables 3 and 4.

All experiments were performed in triplicate. All experimental variables were chosen through preliminary tests.

Statistical Analysis

Data were submitted to statistical analysis, using Statsoft 6.0 (Tulsa, OK, USA) software. The analysis was carried out in two steps. The first step involved a stepwise regression to identify the relevant variables; the second step used a multiple regression (Standard Least Square Fitting) to fit a second order mathematical model, according to the following polynomial equation:

$$y = B_0 + \Sigma B_{i\chi i} + \Sigma B^2_{ii\chi ii} + \Sigma B_{ij\chi i\chi j}$$

where y is the dependent variable (peroxide value and induction time), B_0 is a constant value, χ_i and χ_j are the independent variables (drying temperature, power percentage of microwave oven, pasta moisture and percentage of olive oil) in coded values and B_i, B_{ii}, B_{ij} are the model regression coefficients. This model allowed the effects of

Table 3 Experimental factorial design relating to samples dried by hot air

Levels	Temperature (°C)	Pasta moisture (%)	Oil (%)
1	60	16	40
2	60	16	60
3	60	22	40
4	60	22	60
5	80	16	40
6	80	16	60
7	80	22	40
8	80	22	60
9	53.18	19	50
10	86.82	19	50
11	70	13.95	50
12	70	24.05	50
13	70	19	33.18
14	70	19	66.82
15[a]	70	19	50
16[a]	70	19	50
17[a]	70	19	50

[a] Central points

Table 4 Experimental factorial design relating to samples dried by microwave

Samples	Power (%)	Pasta moisture (%)	Oil (%)
1	50	16	40
2	50	16	60
3	50	22	40
4	50	22	60
5	70	16	40
6	70	16	60
7	70	22	40
8	70	22	60
9	43.18	19	50
10	76.82	19	50
11	60	13.95	50
12	60	24.05	50
13	60	19	33.18
14	60	19	66.82
15[a]	60	19	50
16[a]	60	19	50
17[a]	60	19	50

[a] Central points

the linear (χ_i), quadratic (χ_i^2) and combined ($\chi_i\chi_j$) terms of the independent variables to be assessed on the dependent variable.

The fitness of the mathematical model to the experimental data was evaluated by means of correlation coefficient (r), standard error of estimation (SE) and Fisher test (F) (and the derived P-level).

Isoresponse surfaces were developed to describe both individual and interactive effects of the independent variables of drying processing on peroxide value and induction time.

Standard deviations and variation coefficient were calculated by Excel for Office XP (Microsoft Corporation) software. Test T (for independent variables) was used to compare the peroxide values and induction times of samples dried by conventional and microwave drying. This test was carried out using the Statsoft 6.0 software.

Results and Discussion

Values of correlation coefficient (r), standard error of estimation (SE), Fisher test (F), P-level and general equations of each dependent variable considered for both factorial designs are shown in Tables 5 and 6. The values of r and P-level showed that each considered equation resulted significant; in fact they were always more than 0.81 and less than 0.05, respectively. In particular, the best fit equation was obtained for the data of peroxide values of samples dried by hot air ($r = 0.87$).

Effects of power percentage and moisture of pasta on the peroxide value of the oil extracted to stuffed pasta dried by microwave are reported in Fig. 1. This Figure shows that peroxide values were less than the threshold of acceptability (25 mequiv O_2/kg oil) [8, 9]. Moreover, peroxide values increased with increasing of power percentage at high moisture values of pasta (short time of treatment, i.e., 66 s), whereas for small moisture value (long time of treatment, i.e., 160 s) the peroxide values decreased with the increase of power percentages (Fig. 1). In addition, the relating partial equation showed an interactive factor between these variables on the peroxide value. In fact, the highest peroxide value was obtained at higher values of power percentages and moisture of pasta.

In contrast to what had been expected, the lowest values of peroxides were obtained for samples of oil extracted from pasta dried under the most drastic conditions (high power percentages, low values of pasta moisture and long treatment times) (Table 7). This behavior could be due to the formation of Maillard's reaction products (MRPs) which have an antioxidant activity [10] and a protective effect in pasta dried at high temperatures [11, 12]. This antioxidant activity was extensively studied both on model systems [13, 14] and real food [15], but the chemical structure of these compounds was still not sufficiently documented. In order to have confirmation of this statement, the absorbance at 420 nm was determined since this index measures the coloured compounds developed during

Table 5 General equations obtained by fitting multiple regression (backwards stepwise) to values of dependent variables relating to microwave drying

Dependent variables	General equation	*R*	SE	*F*
Peroxide value (mequiv O_2/kg oil)	$y = 62.41431 - 1.60369 \times P + 0.04613 \times P \times M + 0.01646 \times P \times O - 0.05207 \times M \times O$	0.84	1.6	7.05
Induction time (minutes)	$y = 2{,}444.800 - 175.610 \times M + 0.161 \times P^2 + 4.505 \times M^2 + 0.246 \times O^2 - 0.415 \times P \times O$	0.81	43.93	4.09

Significance $P < 0.05$

P oven power, *M* moisture of pasta, *O* olive oil percentage, *r* correlation coefficient, *SE* standard error, *F* Fisher test

Table 6 General equations obtained by fitting multiple regression (backwards stepwise) to values of dependent variables relating to conventional drying

Dependent variables	General equation	*R*	SE	*F*
Peroxide value (mequiv O_2/kg oil)	$y = 199.2827 - 1.7561 \times T - 8.4665 \times M - 1.7984 \times O + 0.0135 \times T^2 + 0.2151 \times M^2 + 0.0178 \times O^2$	0.87	1.78	5.34
Induction time (minutes)	$y = 663.9954 - 0.0845 \times O^2 + 0.1223 \times T \times O$	0.84	39.73	16.87

Significance $P < 0.05$

T drying temperature, *M* moisture of pasta, *O* olive oil percentage, *r* correlation coefficient, *SE* standard error, *F* Fisher test

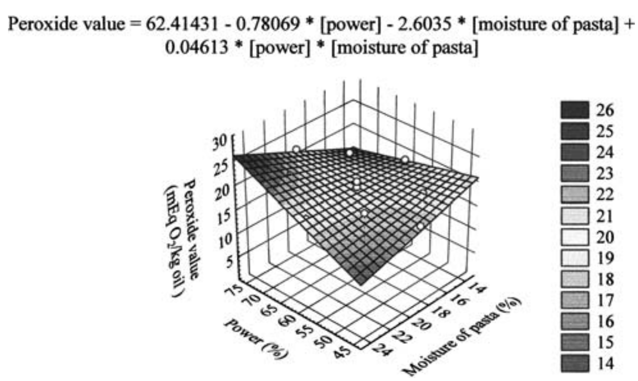

Fig. 1 Effect of power percentage and moisture of pasta on the peroxide value of oil extracted from samples dried by microwave

the Maillard reaction in food [16]. In fact, as shown in Fig. 2 the highest absorbance value was obtained for samples dried under the most drastic conditions.

Figure 3 shows the peroxide values of samples dried by microwave as a function of power and oil percentages. The peroxide value of samples containing low percentages of oil decreased with the increase of power percentage, probably due to the antioxidant activity of MRPs. It can be seen that the highest values of peroxides were obtained at high percentages of power and oil. This is because of the stuffing with the highest oil content has reached a very high temperature since the specific heat of oil is lower than that of water (2.0 and 4.2 KJ/kg °C, respectively) [17]. In fact, samples 13 and 14, on the same levels of power % and moisture %, showed that samples with 33.18% oil reached a temperature of 116 °C versus samples containing 66.82%

Table 7 Maximum temperatures reached into samples of pasta dried by microwave

Samples	Power (%)	Pasta moisture (%)	Oil (%)	Temperature (°C)
1	50	16	40	116
2	50	16	60	138
3	50	22	40	95
4	50	22	60	104
5	70	16	40	127
6	70	16	60	140
7	70	22	40	104
8	70	22	60	121
9	43.18	19	50	121
10	76.82	19	50	142
11	60	13.95	50	152
12	60	24.05	50	104
13	60	19	33.18	116
14	60	19	66.82	132
15	60	19	50	142
16	60	19	50	142
17	60	19	50	142

oil that reached 132 °C (Table 7). In this case, the high concentration of oxidation reagents caused the prevalence of the oxidation reaction over Maillard's reaction and the protective effect did not occur.

The isoresponse surfaces of peroxide value of oil extracted from samples dried by conventional drying processing are reported in Figs. 4 and 5. Again, peroxide value

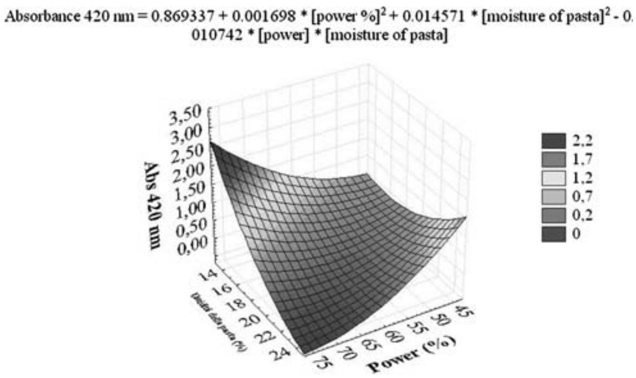

Fig. 2 Isoresponse surface relating to spectrophotometer analyses at 420 nm carried out on pasta sheets

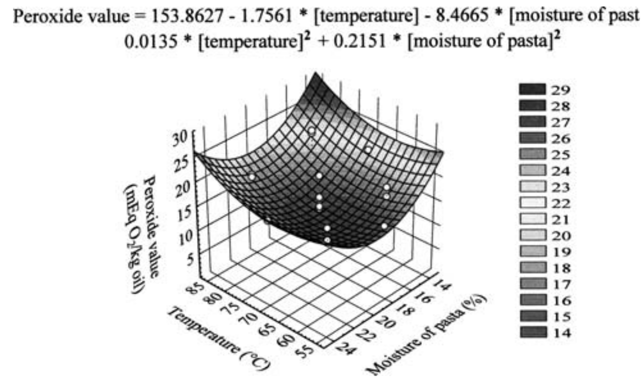

Fig. 4 Effect of temperature and moisture of pasta on the peroxide value of oil extracted from samples treated by conventional drying

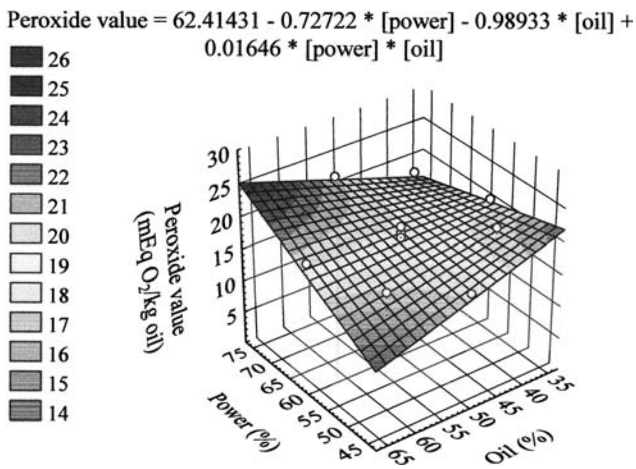

Fig. 3 Effect of power percentage and oil percentage on the peroxide value of oil extracted from samples dried by microwave

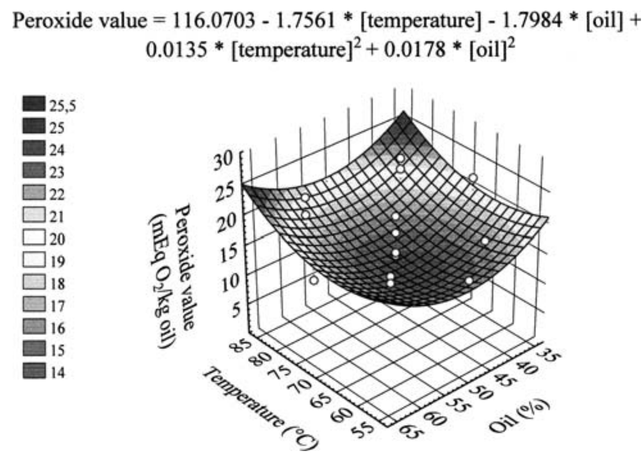

Fig. 5 Effect of temperature and oil percentage on the peroxide value of oil extracted from samples treated by conventional drying

of 23 mequiv O_2/kg oil was less than threshold of acceptability. The highest peroxide value was obtained at drastic condition of treatments, i.e., the highest values of drying temperature and the lowest values of pasta moisture (which correspond to the longest treatment times) (Fig. 4). The same trend is shown in Fig. 5 where the highest peroxide value was obtained at the highest value of drying temperature and the lowest percentage of oil. Also, the lowest percentages of oil increased the treatment time (Fig. 6) favoring its degradation.

Results of *T* test show that the differences between peroxide values of stuffing oil extracted from samples dried by microwave or hot air are not significant (Table 8). These data are in agreement with results reported by Caponio et al. [18].

The use of a very fast drying process such as the microwave one could lead to remarkable energy saving and a lowering of production costs without compromising the quality of the products.

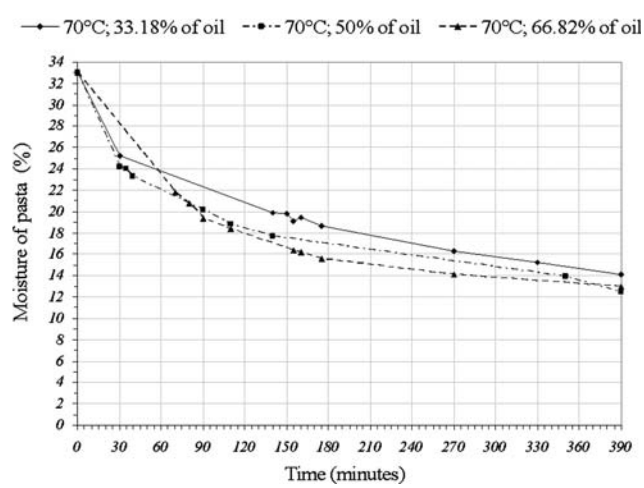

Fig. 6 Drying curves of samples of pasta with a stuffing at different percentages of oil (33.18, 50 and 66.82%) and conventionally dried at 70 °C

Table 8 *T* test of peroxide value (PV) of oil extracted from samples dried by microwave (*M*) and hot air (HA)

	Average M	Average HA	*T* value	Freedom degree	*P*-level	SD	
						M	HA
PV M versus PV HA	18.69	18.06	0.67	32	0.51	2.54	2.88

SD standard deviation

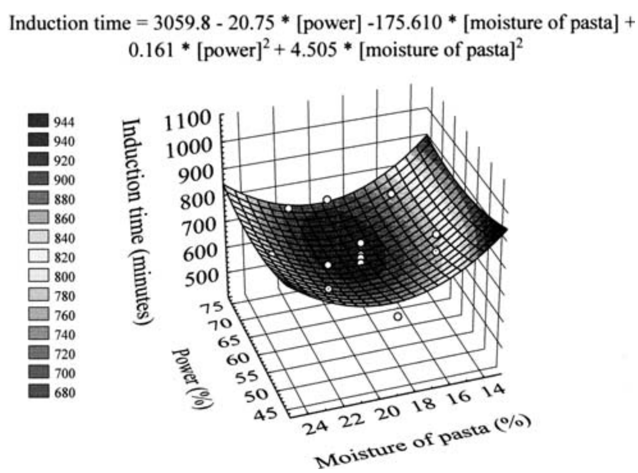

Fig. 7 Effect of power percentage and moisture of pasta on the induction time of oil extracted from samples dried by microwave

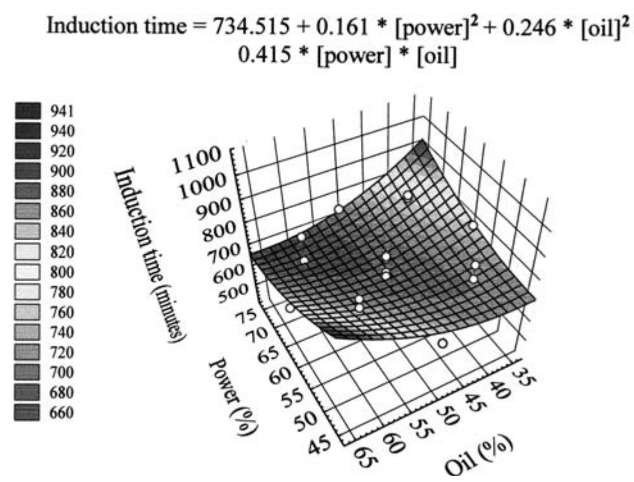

Fig. 8 Effect of power percentage and oil percentage on the induction time of oil extracted from samples dried by microwave

In addition, in order to evaluate the secondary lipid oxidation and to confirm the probable antioxidant action of MRPs, the Rancimat test was carried out.

Figure 7 shows the effect of power percentages and pasta moisture on the induction time of microwave dried samples. It can be observed that the highest values of the induction time were obtained at low values of pasta moisture: samples submitted to long treatment times (the maximum temperature reached inside the ravioli was 138–152 °C as shown in Table 7) caused less degradation. These results support our hypothesis that low peroxide values of samples dried at more drastic conditions could be due to the antioxidant action of MRPs rather than the degradation of peroxides in secondary oxidation compounds. The highest values of induction times were obtained respectively at more drastic conditions of treatment, i.e., low percentages of oil and high values of power percentages (at the same conditions low peroxide values were obtained, see Fig. 2) and high percentages of oil/low power as shown in Fig. 8. In contrast, samples with a high percentage of oil dried at high power percentages or samples with low percentage of oil dried at low power percentages resulted in more oxidization. It can be supposed that, under these conditions, the lipid oxidation prevailed against Maillard's reaction because of a high concentration of lipid oxidation reagents.

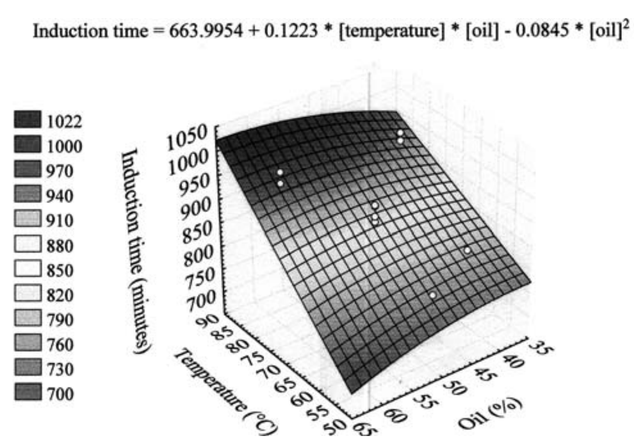

Fig. 9 Effect of temperature and oil percentage on the induction time of oil extracted from samples treated by conventional drying

The effect of oil percentages and drying temperatures on induction times of samples dried by hot air is shown in Fig. 9. From this figure and its relative partial equation, a positive interaction between both these processing variables can be found; in fact the highest value of induction time was obtained at the highest value of oil percentages and drying temperatures. Again this behavior could be attributed to the antioxidant action of MRPs.

Table 9 shows the induction time values of samples dried by microwave and hot air. The induction time of

Table 9 *T* test of induction time (IT) (minutes) of oil extracted from samples dried by microwave (*M*) and hot air (HA)

	Average M	Average HA	*T* Value	Freedom degree	*P*-level	SD	
						M	HA
IT M versus IT HA	751	874	−5.51	32	0.001	61.6	68.6

SD standard deviation

microwave dried samples was 751 min versus 874 min of hot air dried samples. Even if a statistical difference between them was found (as *T* test shown), in the absence of a reference scale, we are not able to determine how this difference influenced the lipid fraction quality of samples dried by both treatments.

However, since both microwave and hot air dried samples did not exceed the limits of acceptability, the use of microwave processing could be suitable for a remarkable energy saving and a lowering of production costs without to compromise the quality of products. In fact, the lowest lipid oxidation was obtained at the most drastic condition of drying which correspond to the shortest treatment time.

Moreover, although most of studies on the Maillard reaction pointed out its negative nutritional aspects such as the reduction of lysine (an essential amino acid) availability, the natural antioxidants developed during this reaction could be substituted by commonly used synthetic antioxidants in particular in pasta with high lipid stuffing (e.g., cheese). While the lack of essential amino acids could be balanced by other ingredients usually present as stuffing.

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