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Influence of dimethylpolysiloxane addition to edible oils: dependence on the main variables of the frying process

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SUMMARY

Influence of dimethylpolysiloxane addition to edible oils: dependence on the main variables of the frying process

Experiments were carried out in the absence of food to define the influence of significant variables of the frying process, i.e. oil unsaturation, temperature, surface-to-oil volume ratio, type of heating and dimethylpolysiloxane (DMPS) addition. Special emphasis was placed on clarifying the action of the addition of low concentrations of DMPS to frying oils. From statistical evaluation based on the content of polar compounds, the strong influence of the variables surface-to-oil volume ratio, DMPS addition and oil unsaturation was deduced. However, changes due to 10°C difference in temperature were not significant. Analysis of two-way interactions demonstrated that interactions between type of heating/DMPS addition, type of heating/oil unsaturation, surface-to-oil volume ratio/DMPS addition and surface-to-oil volume ratio/oil unsaturation were significant, thus indicating the complexity of the frying process. Finally, it was found that increase in specific groups of compounds formed through oxidative polymerization as well as loss of natural antioxidants depended mainly on the total content of polar compounds and, at a second level, on the oil unsaturation.

KEY-WORDS: Dimethylpolysiloxane – Frying – High oleic sunflower oil – Polar compounds – Sunflower oil – Thermoxidation.

1. INTRODUCTION

At the high temperatures of frying, fats and oils become highly susceptible to degradation and tend to develop undesirable foam due to oxidative polymerization. The use of silicones as antifoaming agents in aqueous systems is well-known and its action can be explained by the surface chemical nature of the polymer (Miura, 1993). The addition of silicones, specifically dimethylpolysiloxane (DMPS), to oils at very low concentrations has been demonstrated to be useful to increase oil stability at the high temperatures of the frying process. Under these circumstances, the antifoaming effects found might also be an indirect result of the lower incidence of oxidation (Zwobada, 1979).

According to previous studies, DMPS forms a monolayer on the oil surface protecting the oil against oxidation (Ohta et al., 1987) and it has been found that concentrations as low as 0.06 mg/Kg were enough to provide an effective protection to the oils (Freeman et al., 1973). Although the precise mode of action has not been conclusively verified, inhibition of the convection currents created when there is a temperature gradient seems to play an important role (Kusaka et al., 1985).

It is interesting to observe that the extent of the effect found in previous studies was very variable, going from a drastic decrease of the alteration (Freeman et al., 1973; Augustin et al., 1987) to small differences between oils without and with DMPS added at the usual concentrations (Tian and White, 1994). The differences between the results obtained may be due to the complexity of the frying process as oil degradation is influenced by a high number of variables and parameters. In this respect, changes from antioxidant to prooxidant activity of DMPS depending on the type of heating (either oven or plates) have been reported (Rock et al., 1967) although other authors have found a positive effect in all the experiments with oils heated in plates deteriorating more rapidly (Kusaka et al., 1984). Also, the influence of DMPS has been reported to be dependent on the temperature (Kusaka et al., 1977) and the oil unsaturation (Yuki and Morimoto, 1982).

On other hand, the analytical methods used to evaluate fat degradation, i.e. carbonyl value (Yuki and Morimoto, 1982; Kusaka et al., 1984), iodine value (Rhee, 1978; Augustin et al., 1987), viscosity (Rhee, 1978; Yuki and Morimoto, 1984), volatiles (Snyder et al., 1986), room odor and flavor scores (Rhee, 1978; Frankel et al., 1985), triglyceride oxidation (Freeman et al., 1973; Yan and White, 1991) polar compounds (Augustin et al., 1987), etc, may have also contributed significantly to the variability found as deduced from studies wherein several analytical methods were used. In general, these results would suggest interactions between the variables of the frying process and dependence on the analytical method applied.

The main objective of this study was to clarify the effect of DMPS and its dependence on the main known variables influencing the frying process, i.e. temperature, surface-to-oil volume ratio, type of heating and unsaturation degree. Initially, the influence of DMPS was defined under thermoxidative conditions as a better control of the variables could be thus established.

Analytical evaluations include total degradation products by means of polar compound determination as well as quantitation of oxidized and polymeric compounds.

2. EXPERIMENTAL PART

Samples

Conventional sunflower oil (SO), genetically modified high oleic sunflower seed oil (HOSO) and a 50% mixture of both of them were used (SO/HOSO).

Sample treatment

Samples without and with 2 mg/Kg of DMPS, were heated in 150 mL beakers using either 25 or 50 mL of oil, which allowed an excellent control of the surface-to-oil volume ratio variable, 1 and 0.5 cm^{-1}, respectively. Two temperatures, i.e. 170°C and 180°C, were selected. Heating of the samples was carried out either in oven or plates. The main difference between the two treatments consisted of the occurrence of a temperature gradient when the sample was heated in plates while the bulk of the sample is at the same temperature in the oven. This permits analysis of the influence of the convection currents created due to the temperature gradient. Finally, depending on the values of the main variables, oils were heated for 5, 10 and 20 hours. Nevertheless, for all the combinations, 5 hour heating samples were prepared.

Analytical determinations

Quantitation and distribution of polar compounds.

The amount of polar compounds was determined by silica column chromatography following the method proposed by the IUPAC (IUPAC,1987) with two slight modifications: a mixture of hexane:diethyl ether (90:10) was used to elute the non-polar fraction while a final elution of the column with methanol improved the recovery of the sample. The polar fractions were further submitted to a fractionation by high-performance size-exclusion chromatography (Dobarganes et al., 1988). Briefly, the samples were analysed in a Konik 500 A chromatograph with a 10 µl sample loop. A Hewlett Packard 1037 A refractive index detector and two 100 Å and 500 Å Ultrastyragel columns (Waters Associates, Mildford, MA) connected in series operated at 45°C. The columns were 25 cm x 0.77 cm I.D., packed with porous highly cross-linked styrene divinylbenzene copolymer. HPLC-grade tetrahydrofuran was used as the mobile phase at a flow rate of 0.5 mL/min. Samples were injected in tetrahydrofuran (15-20 mg/mL).

Quantitation of tocopherols

Tocopherols were quantitated by high-performance liquid chromatography following the standard IUPAC Method (IUPAC, 1992).

Oil stability index

Oil stability index were determined using a Rancimat apparatus at 100°C following the standard AOCS Method (AOCS, 1994).

Statistical evaluation

Analysis of variance (ANOVA) was applied to a full 2^5 factorial design to study the main effects of the variables and two-way interactions.

3. RESULTS AND DISCUSSION

Table I show the initial values for total polar compounds, their distribution in groups of compounds related to polymerization, oxidation and hydrolysis, content of tocopherols, and susceptibility against oxidation.

Table I
Chemical evaluation of the initial oils

SAMPLES	Polar compounds (wt% on oil)	Polar compound distribution TGD (mg/g)	oxTGM (mg/g)	DG (mg/g)	FA (mg/g)	Tocopherols (mg/Kg) α	γ	Oil Stability Index (hours)
SO	3.2	5.5	8.8	11.6	6.1	603	28	9.0
SO DMPS	3.3	5.7	9.1	12.0	6.2	598	29	8.0
SO/HOSO	3.3	4.6	9.4	12.8	6.2	627	23	10.0
SO/HOSO DMPS	3.1	4.0	9.0	11.7	6.3	634	19	11.3
HOSO	2.9	2.1	6.1	14.4	6.4	650	13	20.8
HOSO DMPS	2.9	2.5	6.8	13.8	5.9	654	17	19.9

Abbreviations: SO: sunflower oil; HOSO: high oleic sunflower oil; DMPS: dimethylpolysiloxane
TGD: triglyceride dimers; oxTGM: oxidized triglyceride monomers; DG: diglycerides; FA: fatty acids

As can be observed, the content of total polar components was very low in all the oils which is indicative of good initial quality. Only small differences were found in the distribution of polar compounds and content of tocopherols while stability against oxidation was dependent, as expected, on the unsaturation degree.

Table II shows polar compound percentages after 5 hours of heating for all the combinations of the variables selected. Values for the different variables were those normally used in thermoxidation studies simulating frying condition (Gere, 1983). The action of temperature, surface-to-oil volume ratio and unsaturation level of the oils, followed the expected pattern, i.e. the higher the level of the variable, the higher the oil degradation (Gere, 1982; Gere, 1983; Dobarganes et al., 1993), although the extent of the action of each variable was different.

Table II
Influence of the main variables during thermoxidation of oils: Evaluation of polar compounds (% on oil) after heating for 5 hours

Type of heating	T (°C)	S/V (cm^{-1})	DMPS (ppm)	HOSO	HOSO/SO	SO
PLATE	170	0.5	0	18.0	19.3	22.4
			2	5.5	6.7	6.8
		1	0	30.8	36.5	38.7
			2	8.3	10.4	11.6
	180	0.5	0	22.7	25.4	15.1
			2	6.7	8.9	8.9
		1	0	38.6	41.8	40.8
			2	10.9	13.2	14.6
OVEN	170	0.5	0	5.4	8.4	12.9
			2	5.5	8.5	13.5
		1	0	11.9	15.1	25.1
			2	9.7	23.0	26.5
	180	0.5	0	8.3	9.9	14.8
			2	9.5	13.7	16.0
		1	0	13.3	15.5	21.0
			2	13.0	22.0	27.1

For abbreviations, see Table I

In general, results indicated that the effect of an increase of 10°C in temperature was not important and even some unexplained results in polar compound levels, confirmed in duplicate assays, were found, i.e. SO heated in plate at 180°C gave significantly lower polar compounds (15.1%) than HOSO under the same conditions (22.7%) or SO at 170°C (22.4%).

Among the results obtained, the strong effect of the surface-to-oil volume ratio stood out. This is in part due to the considerable differences between the two values selected although it is not strange to find that surface-to-oil volume ratio doubles in small fryers or frying pans from the first frying operation until the point at which the oil is discarded (Pérez-Camino et al., 1987). It is interesting to remark that the importance of this variable in oil degradation contrasts with its lack of definition in frying studies, which renders it impossible to obtain replicate experiments and to compare results from different studies.

On other hand, the effect of the unsaturation degree seemed to depend on the type of heating as much lower differences between the oils were found by heating in plates than in oven.

Special comments are required for the results found for DMPS action. As can be observed, for the same values of the rest of the variables, strong differences were obtained when samples were prepared in plates, while no clear differences were found for samples heated in oven, indicating a strong interaction between the two variables. Values of polar compounds obtained in plates were between 3 and 4-fold lower than those found in oven, clearly suggesting that DMPS addition had a drastic positive effect on frying fat alteration when a temperature gradient was established. The differences found would demonstrate the existence of a strong protection against oxidation during heating in plates as other reactions contributing to increase the polar compound levels under frying conditions, like thermal polymerization or cyclization -due to the high temperature-, are expected to be of the same order, while hydrolysis was avoided because of the absence of food moisture.

For statistical evaluation of the results obtained, a full 2^5 factorial design was considered. Table III shows the analysis of variance for the main effects and two-way interactions. Data corresponding to the intermediate level of oil unsaturation (SO/HOSO) were not included to maintain two levels for each variable.

Regarding the main effects of the variables and taking into account the probabilities associated to the null hypothesis included in the last column, no significant differences could be attributed to the temperature at the most usual significance level (0.05) while the influence of surface-to-oil volume ratio, type of heating, addition of DMPS and oil unsaturation on the level of polar compounds was found to be highly significant.

The direction of the estimated effects clearly indicated that a combination of the low oil unsaturation, the low surface to oil volume ratio, addition of 2 mg/Kg of DMPS and heating in plates would produce the lowest level of polar compounds at high temperature.

Table III
Analysis of variance for main effects and two-way interactions

Effect[a]	SS [b]	DF[c]	Mean Squares	F	P
A	25.56	1	25.56	2.436	0.1381
B	701.25	1	701.25	66.839	0.0000
C	140.28	1	140.28	13.370	0.0021
D	666.12	1	666.12	63.491	0.0000
E	297.68	1	297.68	28.373	0.0001
AB	0.66	1	0.66	0.063	0.8049
AC	0.45	1	0.45	0.043	0.8383
BC	22.11	1	22.11	2.107	0.1659
AD	2.88	1	2.88	0.274	0.6075
BD	83.20	1	83.20	7.930	0.0124
CD	816.08	1	816.08	77.784	0.0000
AE	23.12	1	23.12	2.203	0.1571
BE	50.00	1	50.00	4.765	0.0442
CE	123.24	1	123.24	11.747	0.0034
DE	5.95	1	5.95	0.567	0.4622
Residual	167.86	16	10.49		

[a] A: Temperature; B: Surface-to-oil volume ratio; C: Type of heating; D: DMPS addition; E: Oil unsaturation

[b] SS: Sum of squares

[c] DF: Degrees of freedom

Particularly interesting were the results for the two-way interactions. Among the ten possible combinations of the variables, only four were of statistical significance: type of heating/DMPS addition, type of heating/oil unsaturation, surface-to-oil volume ratio/DMPS addition and surface-to-oil volume ratio/oil unsaturation. The interactions between the type of heating and either DMPS addition or oil unsaturation are of great importance for a correct interpretation of simulation studies in the absence of foods when samples are heated in oven, but have no interest in frying practice where a temperature gradient is always created. Thus, the most significant expected interactions in frying practice should be those found between surface-to-oil volume ratio and either the DMPS addition or the oil unsaturation which suggested that surface-to-oil volume ratio should be as low as possible, particularly if there is no DMPS addition and/or the oil unsaturation is high.

Apart from evaluation of total polar compounds it is interesting to know their distribution in oxidation, polymerization and hydrolytic compounds. Table IV and V include selected results of distribution of polar compounds, showing the effect of the main variables which have been found of major importance in the frying process: surface-to-oil volume ratio (S/V), oil unsaturation and DMPS addition. Data corresponding to 170°C have been selected and two periods of heating were controlled. They were established depending on surface to oil volume ratio, i.e., 5 and 20 hours for S/V = 0.5 cm^{-1} and 5 and 10 hours for S/V = 1 cm^{-1}.

Table IV
Influence of DMPS addition, oil unsaturation and period of heating on total polar compounds (wt% on oil) and polar compound distribution (mg/g oil)

Samples*	Period of heating (h)	Total	Distribution				
			TGP	TGD	oxTGM	DG	FA
SO	5	22.4	33.4	98.8	69.0	13.8	9.0
SO DMPS	5	6.8	3.7	28.6	19.0	10.0	6.7
SO/HOSO	5	19.3	26.1	84.9	66.9	11.6	3.5
SO/HOSO DMPS	5	6.7	2.8	26.1	19.2	12.7	6.2
HOSO	5	18.0	24.5	65.5	70.9	13.3	5.8
HOSO DMPS	5	5.5	2.4	18.2	16.7	12.4	5.3
SO	20	56.8	234.0	170.9	142.6	10.8	9.7
SO DMPS	20	18.4	28.5	90.5	49.3	10.1	5.6
SO/HOSO	20	51.8	198.4	155.0	148.1	12.4	4.1
SO/HOSO DMPS	20	16.6	21.7	76.9	50.5	12.1	4.8
HOSO	20	52.2	186.4	138.8	176.9	13.1	6.8
HOSO DMPS	20	14.9	22.5	56.9	48.6	14.3	6.7

* Samples heated in plates at 170°C and S/V = 0.5
For abbreviations, see Table I

Table V
Influence of DMPS addition, oil unsaturation and period of heating on total polar compounds (wt% on oil) and polar compound distribution (mg/g oil)

Samples*	Period of heating (h)	Total	Distribution				
			TGP	TGD	oxTGM	DG	FA
SO	5	38.7	100.6	159.1	110.6	10.5	6.2
SO DMPS	5	11.6	9.4	57.3	34.1	10.0	5.2
SO/HOSO	5	36.5	114.6	109.5	125.5	11.7	3.7
SO/HOSO DMPS	5	10.4	8.2	47.0	32.7	11.5	4.6
HOSO	5	30.8	66.8	105.6	114.9	13.6	7.1
HOSO DMPS	5	8.3	6.5	30.2	28.6	13.1	4.7
SO	10	59.9	246.2	179.7	158.3	10.6	4.2
SO DMPS	10	20.2	28.3	98.6	60.2	10.3	4.6
SO/HOSO	10	55.1	208.8	137.8	189.3	11.8	3.3
SO/HOSO DMPS	10	19.0	28.5	86.1	58.7	12.2	4.6
HOSO	10	51.3	170.8	141.1	181.7	13.2	6.2
HOSO DMPS	10	14.3	16.9	56.5	51.2	14.0	4.4

* Samples heated in plates at 170°C and S/V = 1
For abbreviations, see Table I

As can be observed, similar polar compound levels were found after 10 and 20 hours, at surface-to-oil volume ratio of 1 and 0.5 cm^{-1}, respectively, confirming the importance of the latter variable in polar compound formation. Although frying fat alteration is limited to 25% polar compounds in some European countries (Firestone, 1993), oils were heated until realistic levels of alteration, as oils and fats above 50% polar compounds can be found in real frying practice (Gertz, 1986; Sebedio et al.,1987; Dobarganes et al., 1995; Skrökki, 1995).

Regarding polar compound distribution, results indicated that the higher the increase in polar compounds, the higher the content of oxidation and polymerization compounds, while diglycerides and fatty acid remained at the initial levels. Also, it is interesting to observe that distribution of polar compounds primarily depended on the alteration level, as deduced from the comparison between samples with similar levels of polar compounds and different values for the rest of the variables. The distribution seemed also to be modulated by oil unsaturation as higher amounts of polymerization compounds were found for similar alteration levels in the most unsaturated oil while the less unsaturated one had a higher proportion of oxidized triglyceride monomers. From these results it seems that the effect of DMPS was to decrease oil degradation which in turn would delay the development of oxidative polymerization.

With respect to the loss of α-tocopherol, Table VI summarizes the results obtained after heating for five hours. Initial values were similar for the three oils, as shown in Table I although, as found for oxidation and polymerization compounds, the loss of α-tocopherol depended on the polar compound level and oil unsaturation.

As can be observed by comparing parallel results in Table II and VI, the loss of α–tocopherol was very high in the less unsaturated oil in spite of the low level of polar compounds and total exhaustion was found at polar compound levels about 12% for HOSO. In contrast, α–tocopherol was clearly detected at polar compound contents higher than 20% in SO. As previously reported (Yuki and Ishikawa, 1976), these results indicated that destruction of tocopherol was more rapid in the less unsaturated oils. More over, the rapid loss of α–tocopherol in the less unsaturated oil would suggest the absence of effective protection at high temperature as no acceleration in polar compound formation after total destruction of tocopherol was detected. These findings might indicate a certain capacity of α–tocopherol to become altered, apparently intermediate between that of HOSO and SO.

In conclusion, results obtained in these simulation experiments would indicate the complexity of the frying process and the possibilities of decreasing oil degradation not only based on the knowledge of the primary effect of the variables but also on their interactions. Later experiments carried out on discontinuous and continuous laboratory frying have been of complementary aid to confirm the results under practical conditions and to define more clearly the circumstances under which DMPS would have a positive action (Jorge et al., 1996).

Table VI
Influence of the main variables during thermoxidation of oils: Quantitation of α-tocopherol (mg/Kg) after heating for 5 hours

Type of heating	T (°C)	S/V (cm^{-1})	DMPS (ppm)	HOSO	HOSO/SO	SO
PLATE	170	0.5	0	n.d.[a]	tr.[b]	10
			2	485	509	531
		1	0	n.d.	n.d.	n.d.
			2	214	353	378
	180	0.5	0	n.d.	n.d.	305
			2	361	460	485
		1	0	n.d.	n.d.	n.d.
			2	179	189	292
OVEN	170	0.5	0	355	284	166
			2	374	219	131
		1	0	tr.	61	20
			2	65	11	18
	180	0.5	0	195	335	145
			2	150	110	120
		1	0	n.d.	22	33
			2	n.d.	n.d.	n.d.

[a] nd., not detectable
[b] tr., traces
For abbreviations, see Table I

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