Polydimethylsiloxane Shows Strong Protective Effects in Continuous Deep-Frying Operations

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Abstract: Polydimethylsiloxane (PDMS) was previously reported to show no protective effect in continuous deep-frying. In this study, we used canola oil with/without added PDMS to deep-fry shredded potato at 180°C either continuously or with 10-, 20-, or 30-min intervals between frying sessions for 6 h. In continuous deep-frying in canola oil not containing PDMS, far more oil vapor was generated from the oil and the water in the potato compared to frying with 20- and 30-min intervals between sessions and the oil in the fryer accordingly had a lower polar compound content (PC). The longer the oil was used to deep-fry potato, the more steam was generated from potato. Thus, polar compounds evaporated into the air in the steam, resulting in a low PC value of oil in the fryer. In contrast, both thermal deterioration and oil vaporization were remarkably inhibited in canola oil containing PDMS regardless of the frying pattern, and the PC value of the oil in the fryer increased in proportion to the amount of potato deep-fried. Canola oil with/without added PDMS was heated at 180°C for 6 h to confirm the effect of water released from potato on the oxidation of oil. A large increase in PC was observed in canola oil not containing PDMS when heated without water but this increase was inhibited to some extent when water was supplied continuously. On the other hand, the PC of canola oil containing PDMS was far lower than that of oil not containing PDMS, but the addition of water promoted an increase in PC. In conclusion, we observed superior protective effects of PDMS regardless of the deep-frying pattern employed, but the PC value nonetheless increased as the amount of food deep-fried increased. In addition, we confirmed that water in potato strongly correlates to PC increase of oil in the fryer.

Key words: polydimethylsiloxane, network, continuous deep-frying, oxidation, steam

1 INTRODUCTION

Silicone oils are added at ppm concentrations to oil used for industrial purposes to act, for example, as a smoke point enhancer, antifoaming agent, or color inhibitor¹⁻³⁾. Warner *et al.*⁴⁾ reported that methyl silicone reduced the altered odor of thermally treated oil.

Polydimethylsiloxane (PDMS) exhibits a far higher protective effect than do natural and other synthetic antioxidants^{5,6)}. The protective effect of PDMS in deep-frying operations is reported to be mainly due to PDMS particles dispersed in the oil⁷⁾. Zeta potential is a measure of the magnitude of the electrostatic or charge repulsion/attraction between particles, and is one of the fundamental parameters known to affect stability. The zeta potential of PDMS droplets suspended in canola oil was very high and thus the negatively charged PDMS droplets should attract nearby low molecular weight compounds. It was suggested

that this attraction disturbed the motion of oxygen molecules and prevented their attack against unsaturated fatty acid moiety. This would be the reason why PDMS suppressed the oxidation reaction of oil.

However, there are other interpretations regarding the protective mechanism of PDMS¹⁾. Gerde *et al.*⁸⁻¹⁰⁾ heated soybean oil statically (without stirring) with added PDMS in a crystallizing dish and found out that the addition of 100 ppb PDMS to soybean oil, enough to form a continuous layer over the surface of the oil, reduced the oxygen concentration when compared to a soybean oil control containing no added PDMS at temperatures ranging from 93 to 180°C. This suggests an oxygen barrier effect of PDMS, which was also confirmed by our storage experiment using canola oil with added PDMS at 60°C¹¹⁾. On the other hand, we made a thermal treatment of canola oil covered with a layer of PDMS (0.06 µg/cm²) under high agitation (200 rpm)

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mimicking practical deep-frying. The result showed that oxidation proceeded vigorously regardless of the presence or absence of a PDMS layer⁷⁾. In addition, Freeman *et al.*⁵⁾ reported that droplets of excess PDMS will appear in the oil if concentration is greater than ca. 1 ppm. Thus it was proposed that PDMS droplets mainly exhibited the protective effects in the practical deep-frying, while a PDMS layer had the effect in oil heated without disturbance.

Marquez-Ruiz *et al.*^{12–14)} and others¹⁵⁾ studied the effect of PDMS addition to oils used for continuous and discontinuous frying of potatoes under laboratory conditions. PDMS had a strong positive action in discontinuous frying, and dramatic differences between samples with and without added PDMS were observed. However, PDMS was not effective in simulated continuous frying. Comparison of the results obtained for the discontinuous and continuous frying of potatoes indicated that in continuous frying, the surface of the oil was well protected from the penetration of oxygen by steam generated from the frying potatoes. The authors suggested that the addition of PDMS was unnecessary for continuous frying^{16–18)}.

We previously investigated a method for inhibiting the oxidation of oil heated extensively at frying temperatures by blanketing the oil surface with flowing nitrogen and carbon dioxide¹⁹⁾, in accordance with a report by Aladedunye²⁰⁾, but observed that carbon dioxide, which is heavier than oxygen, was partially effective with a reasonable amount of flow in protecting the surface of the oil from oxygen penetration. In addition, we investigated why used oil recovered from food manufacturing industries after being used for deep-frying showed low carbonyl values²¹⁾ and suggested that steam generated from water in deep-fried food distilled out decomposition products in oil during the frying process.

In the present paper we again examined the protective effect of PDMS in continuous and discontinuous deep-frying operations and investigated both the volatile oxidation products distilled out from the oil and the characteristics of PDMS dispersed in the oil.

2 EXPERIMENTAL

2.1 Materials

The canola oil used was a product of J-Oil Mills Ltd. (Tokyo, Japan) and had the following composition: 0.1% myristic acid, 4.1% palmitic acid, 0.2% palmitoleic acid, 1.8% stearic acid, 64.0% oleic acid, 18.7% linoleic acid, 8.8% α -linolenic acid, and 2.3% other lipids as determined by gas chromatography²²⁾. The canola oil did not contain PDMS. All solvents and reagents were purchased from Wako Pure Chemical Industries Ltd. (Osaka, Japan).

2.2 Chemical properties of the oil

The polar compound content (PC) of oil was determined using a digital edible oil tester (testo270®, Testo Japan, Yokohama, Japan) by placing the sensor into oil contained in a 15-mL test tube and heated to 50°C for the determination. Good correlation was observed between values obtained by the tester and those obtained by the Japan Oil Chemists' Society standard method for determining the PC of oil. See 2.5 and 3.4.

2.3 Continuous and discontinuous deep-frying operations in canola oil with and without the addition of PDMS

The deep-frying conditions were as described by Jorge *et al.*¹³⁾. Potato was peeled and shredded (3200 g). Fresh canola oil (3000 g) with or without PDMS added to 2 ppm⁶⁾ was poured into the tub (23 cm $\times$ 30 cm $\times$ 15 cm deep) of an electric fryer (KCEF-S; Chubo Center, Chiba, Japan). A wire net covered with nonwoven rayon fabric (AS ONE, Tokyo, Japan) was installed over the fryer tub to catch oil vapor released during deep-frying. The surface to volume ratio (SVR) of the oil in the tub was 0.21 cm⁻¹. Canola oil was heated from room temperature to 180°C in 20 min, then 100 g potato was deep-fried for 10 min. This deep-frying operation was repeated with fresh potato 32 consecutive times. After the last batch, the oil was heated another 20 min (6 h heating in total) to ensure the oil temperature was at 180°C. Throughout, the oil in the fryer was not replenished with fresh canola oil. The PC of the residual oil was determined using the testo270®. The residual oil, fried potato, and nonwoven fabric were weighed. The fabric was dried using silica gel under reduced pressure at 2 kPa. Oil on the nonwoven fabric was extracted with hexane.

In the same manner, 100 g potato was deep fried for 10 min with 10-min (16 consecutive batches only for canola oil without added PDMS), 20-min (11 consecutive batches) or 30-min (9 consecutive batches) intervals between each frying operation, each for a total operation time of 6 h. The oil was not replenished in any of these deep-frying experiments.

2.4 Thermal treatment of oil with a continuous supply of water

Takasago *et al.*^{23, 24)} reported that water dissolved in oil was closely related to oxidation of the oil, and that oxidation stability of oil increased with decreasing amounts of dissolved water. To confirm if the exclusion of water from potato influences the increase in PC in heated oil, a small amount of water was added continuously for 6 h to canola oil heated at deep-frying temperatures. A 600-mL, 9 cm diameter beaker containing 300 g fresh canola oil with or without 2 ppm PDMS was placed in an oil heater and heated from room temperature to 180°C in 40 min. Water was supplied to the oil as follows. A ceramic pot 4.5 cm in diameter containing many small holes was used as the

water container. All the holes were plugged with tightly rolled Kimwipes (Wipers S-200, Nippon Paper Crexia Co., Ltd. Tokyo, Japan). Water seeped through the paper plugs at a rate of 60 mL/h. The ceramic pot was gently submerged into the canola oil in the beaker. The PC of the oil was determined using the testo270[®] every hour during heating. The initial surface to volume ratio was 0.199 cm⁻¹.

2.5 Correlation between the PC values determined using a testo270[®] and by column chromatography

The PC values of many oil samples were measured with a testo270[®]. This instrument uses an electrostatic capacitive type sensor and simply and conveniently determines the PC values of thermally treated oils. These values were compared to those obtained by chromatography and compared to find a correlation between the two methods^{25, 26}.

2.6 Viscosity measurement

The ratio of the viscosity increase is a good indicator of the polymerization of oil under thermal treatment²⁷. The viscosities of the oil samples obtained in 2.3 were determined at 25°C using a Viscotesta VT-06 (RION, Tokyo, Japan).

2.7 Gardner color measurement

According to the Japan Oil Chemists' Society standard method for determining the color (2.2.1.3), oil color was determined.

2.8 Statistical analyses

All experiments were conducted in triplicate. All values for PC are provided as mean ± SD and were analyzed using one-way analysis of variance with Dunnett's multiple comparison post hoc test. Results were considered significant at $p < 0.05$.

3 RESULTS and DISCUSSION

3.1 Continuous and discontinuous deep-frying operations with canola oil with and without the addition of PDMS

Table 1 shows the oil volume, SVR, and the highest/lowest temperature during the deep-frying operation. The SVR of canola oil with or without the addition of PDMS and kept at a temperature of over 160°C remained almost unchanged after each 6 h deep-frying operation. The highest (overrun temperature) and lowest temperature when potato was given to oil, shows the oil temperature range observed during 6 h deep-frying operation.

The ratio of potato deep-fried/potato raw was similar in the seven deep-frying operations.

Figure 1 shows the weights of oil recovered from the nonwoven fabric used to cover the fryer. Continuous deep-frying in canola oil without added PDMS gave rise to a large

Fig. 1 Oil recovered from nonwoven fabric covering the electric fryer used for deep-frying potatoes at 0- (continuous), 10-, 20-, or 30-min intervals for 6 h with and without added PDMS. Values are expressed as mean ± SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

Table 1 Conditions and results for continuous and discontinuous deep frying operations.

	Continuous	10-min interval	20-min interval	30-min interval	PDMS continuous	PDMS 20-min interval	PDMS 30-min interval
Oil 0 h (g)	3000	3000	3000	3000	3000	3000	3000
Oil 6 h (g)	2717 ± 14 ^f	2824 ± 10 ^d	2869 ± 7 ^c	2896 ± 3 ^b	2756 ± 14 ^e	2877 ± 7 ^c	2921 ± 2 ^a
SVR (cm ⁻¹) 0 h	0.212	0.212	0.212	0.212	0.212	0.212	0.212
SVR (cm ⁻¹) 6 h	0.234 ± 0.001 ^a	0.225 ± 0.001 ^c	0.222 ± 0.001 ^d	0.220 ± 0.001 ^d	0.231 ± 0.001 ^b	0.221 ± 0.001 ^d	0.218 ± 0.001 ^e
The highest temperature (°C)	184	182	183	184	181	183	184
The lowest temperature (°C)	164	163	164	162	163	164	164
Potato (g)	3200	1600	1100	900	3200	1100	900

PDMS: polydimethylsiloxane, SVR: surface to volume ratio. Values of Oil 6 h (g) and SVR (cm⁻¹) 6 h are expressed as mean ± SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

amount of oil vapor whereas the amount of oil vapor generated in discontinuous deep-frying varied according to the amount of potato fried. Interestingly, the addition of PDMS to canola oil resulted in little oil vaporizing in both continuous and discontinuous deep-frying. In addition, the weight of canola oil with added PDMS left-over in fryer was greater than that of the corresponding oil without added PDMS (Table 1). PDMS particles dispersed in oil show a high zeta potential and form a network⁷⁾ which would greatly hinder oil volatilization. Kusaka *et al.*²⁸⁾ also reported that silicone oil disturbed water evaporation in frying oil. Babayan²⁹⁾ patented the process for the addition of methyl and ethyl silicones to oil to increase the smoke point by 8–19°C, consistent with our present results. Deep-frying plants and outlets using oil with added PDMS do not have oil stains (private communication). Furthermore, we previously reported that PDMS particles reduced the volatilization of low molecular weight compounds in thermally deteriorated canola oil at ambient temperature⁷⁾, in agreement with the report by Warner *et al.*⁴⁾.

As shown in Fig. 2, the PC of canola oil without added PDMS used in discontinuous frying increased significantly regardless of the interval length. In addition, the longer the interval, the higher the PC value, because oxidation products are vigorously distilled out by steam generated when food is deep-fried²¹⁾. Canola oil containing PDMS showed remarkably inhibited increases in PC, and the longer the interval, the lower the PC value. Consequently, the increase in PC with respect to the interval was reversed as com-

Fig. 2 Polar compound content of canola oil with and without added PDMS used for deep-frying potato at 0- (continuous), 10-, 20-, or 30-min intervals for 6 h. Values are expressed as mean $\pm$ SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

pared to canola oil not containing PDMS. In general, increases in heating time and in water contained in food used for hydrolysis increase the PC value while steam generated from food decreases PC value due to steam distillation. In addition, PDMS dispersed in oil drastically inhibits oil volatilization, as described above.

Our previous thermal experiment at 180°C for 5 h using canola oil with and without PDMS, containing 672 $\mu\text{g/g}$ natural tocopherols showed slight decrease of total tocopherols in PDMS-canola oil, whereas very large decreases were observed with pure canola oil, with 92% of the tocopherols destroyed after 5 h heating⁶⁾. Tocopherols are antioxidants that should function independently in eliminating radicals regardless of the presence or absence of PDMS in canola oil, assuming that PDMS does not interfere with tocopherol activity. However, residual total tocopherols were obviously high in PDMS-canola oil. In the oxidation of oil, fatty acid radicals initially generated are attacked by oxygen to form peroxide radicals, then tocopherols react with the radicals, resulting in stable products³⁰⁾. This process involves oxygen consumption; tocopherols decrease with the radical elimination and thermal decomposition. On the other hand, PDMS should neither react with the radicals described above nor consume oxygen. Thus, PDMS inhibited the oxidation of canola oil in a manner^{6,7)} different from the radical elimination of tocopherols and prevented the consumption of tocopherols.

The Gardner color values of canola oil heated for 6 h are shown in Fig. 3. The color of all heated oil samples not containing PDMS deteriorated markedly and there was no statistical difference among the samples, suggesting that the color deterioration of oil not containing PDMS was

Fig. 3 Gardner color of canola oil with or without added PDMS used for deep-frying potato at 0- (continuous), 10-, 20-, or 30-min intervals for 6 h. Values are expressed as mean $\pm$ SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

related more to the heating time rather than to the amount of potato fried. Oil with added PDMS showed significantly less color deterioration and again there was no statistical difference among the samples.

The concentration of PDMS (2 ppm at the start of deep-frying) could have changed as the deep-frying procedure proceeded because of the absorption by potato and the thermal decomposition of PDMS. However, this decrease was not determined because the quantification of PDMS is very difficult at concentrations below 5 ppm¹¹⁾.

3.2 Effects of a continuous supply of water on oil with and without added PDMS

The conditions used for this thermal treatment experiment resembled that of section 2.3 in terms of the ratio of water added to the oil heated for 6 h; that is, the water ratio to oil was close to that of continuous deep-frying. Table 2 shows the SVR after 0 and 6 h of heating and the temperature range measured during heating.

The continuous supply of water during the first 3 h of heating canola oil without added PDMS had no effect on the measured values but the increase in PC was retarded after 5 h (Fig. 4) due to the steam distillation of decomposition products generated during heating, as shown in Fig. 2. When canola oil containing PDMS was heated, much lower increases in PC values were observed compared to canola oil not containing PDMS. The continuous supply of water to canola oil containing PDMS resulted in a gradual increase in PC after 2 h of heating, in contrast to the findings of Marquez-Ruiz *et al.*^{12–15)}. PC values indicate the balance of the amount of oil-degradation compounds and that of steam-distilled during deep-frying, so that the amount of steam-distilled degradation compounds has a direct influence on the size of PC values. In our experiments the hydrolysis/ decomposition of oil exceeded the amount of product distilled out by steam because the PDMS particles reduced the evaporation of volatile oil-degradation compounds during deep-frying.

Fig. 4 Polar compound content of canola oil with or without added PDMS heated at 180°C for 6 h with and without a continuous water supply. Values are expressed as mean $\pm$ SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

3.3 Polymerization of oil in deep-frying operations

The polymerization of canola oil was estimated by the increase in viscosity during deep-frying²⁷⁾. The viscosity of canola oil without added PDMS increased by 20% in continuous and 10-min interval frying, by 40% in 20-min and 30-min interval fryings. From this result it was proved that thermal oxidation proceeded vigorously during idle time of interval frying in canola oil without added PDMS. However, canola oil containing PDMS showed no detectable increase in viscosity (Fig. 5), indicating that thermal oxidation and polymerization via oxidized triacylglycerols was largely inhibited using the current deep-frying conditions³¹⁾. When the interval frying was carried out in canola oil containing PDMS, PDMS layer over the surface of the oil inhibited oxygen penetration from air during idle time, and thermal oxidation hardly proceeded as well as polymerization.

Table 2 Thermal treatment of canola oil containing PDMS and water.

	Simple heating	Heating with water	PDMS heating	PDMS heating with water
SVR (cm ⁻¹) 0 h	0.199	0.199	0.199	0.199
SVR (cm ⁻¹) 6 h	0.219 $\pm$ 0.002 ^b	0.229 $\pm$ 0.002 ^a	0.219 $\pm$ 0.002 ^b	0.226 $\pm$ 0.002 ^a
The highest temperature (°C)	182	183	182	182
The lowest temperature (°C)	175	166	174	162

PDMS: polydimethylsiloxane, SVR: surface to volume ratio. Values of SVR (cm⁻¹) 6 h are expressed as mean $\pm$ SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

Fig. 5 Viscosity increase of canola oil with or without added PDMS used for deep-frying potato at 0- (continuous), 10-, 20-, or 30-min intervals for 6 h. Values are expressed as mean $\pm$ SD. Values with non-common superscript letters differ significantly ($p < 0.05$) by Dunnett's multiple comparison post hoc test.

3.4 Correlation between PC determined using a testo270[®] and by column chromatography

The Japan Oil Chemists' Society standard method for determining the PC of oil requires equipment such as a glass column, a separatory funnel, silica gel 60, sea sand, and developing solvent (hexane/diethyl ether, 87:13 v/v). For column chromatography, oil is eluted using the developing solvent and solvent is removed from the eluent containing triacylglycerol using a rotary evaporator. The entire process takes about 3 h. In addition, the triacylglycerol fraction must be kept at 2 kPa overnight to completely remove the solvent. The PC value is calculated using the formula $(A-B)/A \times 100$, where A is the weight of oil sampled (2.5 ± 0.1 g) and B is the weight of recovered oil (intact triacylglycerol). In the present study, PC values ranged 0-25%, which are within allowable limits according to EU and US regulations³². Incomplete removal of solvent from the triacylglycerol fraction provides erroneously small PC values. **Figure 6** shows that the PC values obtained using the testo270[®] were larger than those obtained by chromatography.

These two methods typically provide somewhat different PC values because separation is achieved using different properties of the polar compounds. Testo 270[®] separation is based on the electrostatics of polar compounds, while the standard method is based on the low polarity of intact triacylglycerol. However, **Fig. 6** shows that the value obtained using the testo270[®] correlates linearly with that obtained by the standard method, indicating that values obtained using the former method are satisfactory for our investigation as long as we do not compare the values obtained by the two methods. We determined the PC values of oil in sections 2.3 and 2.4 exclusively using the testo270[®].

Fig. 6 Correlation between polar compound content obtained by column chromatography and using a testo270[®]. Values are expressed as mean $\pm$ SD.

Conclusion

Canola oil with/without added PDMS was used to deep-fry shredded potato at 180°C either continuously or with 10-, 20-, or 30-min intervals between frying sessions for 6 h. In continuous deep-frying using canola oil without added PDMS, much more oil vapor was generated from the oil and water in the potato compared to when there were 20- and 30-min intervals between frying and the PC value of the oil was accordingly lower. In contrast, the addition of PDMS drastically decreased thermal deterioration and oil vaporization in both continuous and interval frying, and the slight increase in PC value was proportional to the amount of potato deep-fried. PDMS particles dispersed in canola oil have a high zeta potential and form a network in oil⁷, thus greatly inhibiting oil volatilization. We suggest that fewer polar compounds, such as decomposition products and hydrolyzed triacylglycerols, evaporated from oil containing PDMS compared to canola oil not containing PDMS.

In conclusion, we observed superior protective effects of PDMS regardless of the pattern of deep-frying and the PC value increased gradually according to the amount of food deep-fried.

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