Total Polar Compounds and Acid Values of Repeatedly Used Frying Oils Measured by Standard and Rapid Methods

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ABSTRACT

Soybean oil and palm olein were used to fry French fries, chicken leg fillets and pork chops. Total polar compounds were measured by column chromatography and two rapid-measuring devices (Ebro FOM 310 and Testo 270). Acid value was determined by titration method and a 3M Shortening Monitor. The results showed that the content of total polar compounds and acid value in both soybean oil and palm olein increased linearly with frying time. The influence of oil type on the content of total polar compounds and acid value in used oil was significant, but the effect of food type on these parameters was not observed. The rate of total polar compounds generation in palm olein was lower than that in soybean oil. All rapid methods used in this study had results that highly correlated with those from column chromatography, but moderately correlated with the titration method. We found that Ebro FOM 310 was more suitable for monitoring the quality of soybean oil during frying, while Testo 270 was suitable for palm olein. These results provided the basis for choosing the proper rapid-measuring device to control the quality of frying oil in restaurants.

Key words: deep frying, total polar compounds, acid value, rapid measurement

INTRODUCTION

Deep frying is commonly utilized for food preparations such as frozen pre-fried foods, snack foods and fast foods. Fried foods are far more popular today in many places and this can be observed from the rapidly increasing number of fast food restaurants and vendors in the last few decades. Deep frying of foods at high temperature creates the welcome special flavor, golden brown color and crispy texture. It is noted that frying causes oil to undergo hydrolysis, oxidation and thermal reaction, and consequently numerous byproducts such as free fatty acids, alcohols, cyclic compounds, dimers and polymers[1] can be produced. Some products of decomposition in used oil have been identified to have adverse effects on human health[2-4], as they may have a higher chance of absorption into the fried foods[5]. Therefore, it is important to understand the factors affecting the deterioration of frying oil and to monitor the quantity of products of decomposition for ensuring the quality of fried foods.

The mechanism of thermal degradation of frying oil is complicated. Variables involved in the process include frying conditions, replenishment of fresh oil, original oil quality, food materials, and fryer type[6]. Cooking oils with more saturated fatty acids such as lard and palm oil are usually more stable for frying. On the other hand, soybean oil with more unsaturated fatty acids is less stable, and decomposes easily at high frying temperature. In Taiwan, soybean oil is one of the most commonly used cooking oils at home and is also used by many small snack vendors for frying. Palm oil is mostly used commercially to prepare fried potato foods[1]. Among frying oils, those with high oleic acid content such as sunflower oil and palm olein[1,7] have better health profile and heat stability.

Moisture in foods induces and accelerates oxidation with the hydrolytic compounds. Foods with high water content like potato and foods with breading or battering materials cause faster hydrolysis of frying oil. During frying, the fat of chicken or pork that is released into frying oil alters the fatty acid composition and increases the degradation rate of the oil[8]. The release of amino acids from food into oil can prevent oil degradation during deep frying, but the starch has not been observed to have a similar effect[9]. However, only very few researches related to the influences of oil and food types on frying oil quality can be found.

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The content of total polar compounds and acid value are the most predominant indicators for oil quality and are widely used in many international regulations[10,11]. For public health concerns, the content of total polar compounds and acid value in frying oil are regulated at not more than 25% and 2.0 mg KOH/g, respectively, in Taiwan[7,12]. Determination of total polar compounds in frying oil provides a more robust measurement on the extent of deterioration in most situations[10] due to its higher accuracy and reproducibility. The contents of free fatty acid (FFA) and total polar compounds were commonly used for initial oil quality assurance and after-use frying oil quality assessment, respectively[12]. Nevertheless, the standard analytical procedure for oil quality evaluation needs to be done in a laboratory with proper equipment by skilled technicians[13]. It is not suitable for a small industry or vendor to use on site.

Rapid methods have been developed to measure oil quality based either on its chemical properties such as FFA, iodine value, carbonyl value and the content of total polar compounds, or on its physical characteristics such as oil color, viscosity and dielectric constant. Establishing the correlation between the standard and rapid methods for the content of total polar compounds and acid value in used oil offers the necessary supports for the food company to monitor oil quality with a proper rapid method. The objectives of this study were to investigate the effects of oil variety and food type on the oil quality during frying, and to compare the correlations between the standard methods and the rapid methods.

MATERIALS AND METHODS

I. *Experimental Design*

The deep frying process was continued for 48 h for two kinds of oils (soybean oil and palm olein) and three of the most common frying foods in Taiwan (French fries, chicken leg fillet and pork chop). Used oil samples were taken every 6 h for quality analysis. The rapid measurements on the content of total polar compounds and acid value of used oil were performed on site, and the same parameters were assessed again using standard methods later for comparison. The effects of oil types and foods on the content of total polar compounds and acid value in used oils were determined by statistical analysis.

II. *Materials*

(I) *Deep Frying*

Soybean oil and refined palm olein were purchased from Uni-President Enterprises Co. (Tainan, Taiwan) and Chang Guann Co., Ltd. (Kaohsiung, Taiwan), respectively. Frozen French fries used in this study were imported by Disheng Co., Ltd. (Kaohsiung, Taiwan). The French fries were quarter inch in diameter without battering. Marinated

chicken leg fillets were provided by Charoen Pokphand Enterprise Co., Ltd. (Taipei, Taiwan) and were breaded manually with potato starch before frying. Seasoned and battered pork chops were manufactured by Shang Lee Food Co., Ltd. (Nantou, Taiwan). The French fries and pork chops were stored at -20°C, and the chicken leg fillets were refrigerated at a temperature below 7°C.

(II) *Chemicals*

Silica gel with a particle size of 0.063 - 0.200 mm (70 - 230 mesh, No. 7734) was purchased from Merck Co., Ltd. (Darmstadt, Germany). Chromatographic grade isopropanol was purchased from SK Chemicals Co., Ltd. (Ulsan, Korea). Potassium hydroxide was supplied by Panreac Co. Ltd. (Barcelona, EU). Methylisobutylketone (MIBK), light petroleum (boiling point of 35 - 60°C) and diethyl ether were obtained from Tedia Co., Ltd. (OH, USA). All other chemicals were of analytical grade.

III. *Deep Frying Protocol*

The frying conditions of Abdulkarim *et al.*[14] and Manral *et al.*[15] were modified as described below. An electronic deep fat fryer (IL, USA) was a gift from the McDonald's branch company in Taiwan. Each of the two separated troughs in the fryer has a capacity of 15 L. Fresh soybean oil or palm olein was loaded into the fryer and heated to 180°C before frying. Each batch of French fries, chicken leg fillets and pork chops were fried for 3, 6 and 3 min, respectively. A 1:20 (kg/L) ratio of food to oil was applied. There was a total of 24 batches for frying in 6-h cycles and a total of 8 cycles were performed, resulting in 48 h of frying.

The frying oil was not replenished but filtered with filter paper (Part No. 803-0170, Frymaster, Shreveport, LA, USA) after a frying cycle. Control samples were obtained by heating oil without food and following the same frying protocol as above. Rapid measurements were taken while there was no moisture (bubbles) in the frying oil after each cycle. After fully agitating the frying oil, 50-mL samples were collected. Hot oil samples were filtered by passing through the filter paper (*Whatman No.4*) with the aid of an aspirator to remove food residues. Thereafter, the samples were kept in glass bottles and flushed with nitrogen gas before sealing. They were stored at -20°C prior to analysis[13].

IV. *Rapid Measurements of Oil Quality*

The Food Oil Monitor 310 (FOM 310, Ebro Inc., Germany) and the Testo 270 Deep-frying Oil Tester (Testo Inc., Germany) were used to rapidly measure the content of total polar compounds in used oil. The 3M Shortening Monitor (3M, USA) was used to quickly assess the acid value in used oil.

Before taking any measurement, the test mode (liquid, semiliquid or solid) of FOM 310 was selected depending on the state of the oil sample, and the sensor was calibrated

using the "fresh state" oil at a temperature above 100°C. Within the operation temperature of 160 - 200°C, the sensor of FOM 310 was submerged into the oil sample and the oil was stirred gently for 20 s to allow even distribution. FOM 310 typically showed a stable reading of the content of total polar compounds (%) within 1 min[17]. The testing procedure of Testo 270 was very similar to that used by the FOM 310, except the test mode selection and the sensor calibration were not necessary for the Testo 270[18]. These rapid methods detect the dielectric constant of oil. This constant was converted to the content of total polar compounds (%) based on the formula set up by the manufacturer.

The 3M Shortening Monitor was in the form of a test paper strip with four bands. All the bands of the test strip were immersed directly into the hot oil (165 - 190°C) for 3 s. After 30 s, the acid value was determined apparently by comparing the response color band to the synopsis[16].

V. *Standard Methods of Measuring Oil Quality*

The content of total polar compounds in oil sample was also determined based on the methods of AOCS Cd_20-91[20] and ISO 8420[21]. A glass column (35 cm in length and 2.1 cm in diameter) was used for chromatography. The eluent was a mixture of petroleum and diethyl ether in the ratio of 87 : 13 (v/v). The oil sample (2.5 g) was loaded into the packed column and the non-polar fraction was eluted by the eluent. The content of total polar compounds (%) was calculated as the mass fraction of the total polar compounds in the oil sample in percentage.

The CNS 3647[19] method was used for acid value evaluation. An oil sample of 10 g was dissolved in 50 mL of methyl isobutyl ketone (MIBK). The sample solution was titrated against 0.01 - 0.05 N of potassium hydroxide-isopropanol solution to pH 10. The acid value (mg KOH/g) was calculated from the KOH titration volume.

VI. *Statistical Analysis*

Measurements of duplicate samples were expressed as means ± standard deviation for all rapid methods and standard methods. The data were subjected to the analysis of variance (ANOVA) in the general linear models (GLM) or *t*-test using SAS Statistical Computer Package V.9.1 (SAS Institute, Cary, NC). Pearson correlation coefficients among the oil quality testing methods were calculated. Duncan's multiple range tests were used for comparing the differences between group means. The level of significance associated to the statistical test was 0.05.

RESULTS AND DISCUSSION

I. *The Content of Total Polar Compounds and Acid Value in Fresh Oils*

Table 1 lists the content of total polar compounds and

acid value in fresh soybean oil and palm olein, respectively, determined by standard methods and rapid methods. From the results of standard methods, soybean oil contained 2.3% of total polar compounds and 0.03 mg KOH/g of acid value, while palm olein contained higher content of total polar compounds (6.2%) and acid value (0.071 mg KOH/g). Other studies also showed that the content of total polar compounds and acid value in palm olein were higher than those in soybean oil [1,22,23].

Palm oil and palm olein typically contained higher amounts of diglycerides (6 - 8%) than soybean oil (2 - 3%)[1,24]. Bansal *et al.*[25] reported that palm olein with 8.93% of total polar compounds contained 6.46% of diglycerides. However, in most cases, FFA is seldom found in fresh oil[26,27]. In this study, the higher content of total polar compounds in fresh palm olein compared to soybean oil might be due to higher content of diglycerides. On the other hand, the lipase in palm fruit might decompose the oil to produce FFA[26]. This might explain the higher contents of acid value and total polar compounds in palm olein. The acid values in the two fresh oils used in this study were below the limit of refined oil (0.1 mg KOH/g)[26].

The content of total polar compounds in both soybean oil and palm olein obtained from Testo 270 were higher compared with those from the standard method (Table 1). The content of total polar compounds obtained from Ebro FOM 310 in the semiliquid mode (Ebro-SL) and liquid mode (Ebro-L) were lower than that obtained from Testo 270. The distinct formula in the device set up by different companies might lead to different readings of the content of total polar compounds.

The initial value of total polar compounds of Ebro FOM 310 was set to 2% in the liquid mode (Ebro-L) and 4% in the semiliquid mode (Ebro-SL) (Ebro 2005). This can be the reason why a higher content of total polar compounds was obtained from the Ebro-SL. In comparison to the standard method, the initial value of total polar compounds for soybean oil was closer to the value from Ebro-L, and the value for palm olein was closer to the value from Ebro-SL. It could

Table 1. Total polar compounds (%) and acid value (mg KOH/g) tested by standard and rapid methods in fresh soybean oil and palm olein[a]

Methods	Soybean oil	Palm olein
Total polar compounds		
Standard method	2.3 ± 0.3[A]	6.2 ± 0.4[B]
Testo 270	12.8 ± 0.4[B]	11.5 ± 0.0[A]
FOM 310 liquid mode	2.0 ± 0.7[A]	2.8 ± 0.4[A]
FOM 310 semiliquid mode	5.3 ± 0.4[A]	4.5 ± 0.7[A]
Acid value		
Standard method	0.030 ± 0.007[A]	0.071 ± 0.002[B]
3M Shortening Monitor	ND[b]	ND

[a] Values with different superscript capital letters in a row are significantly different (*p* < 0.05).
[b] ND = Not detectable.

be presumed that the Ebro-L and Ebro-SL were suitable for testing soybean oil and palm olein, respectively.

The acid value of fresh oils measured by the 3M Shortening Monitor was not detectable (Table 1).

II. *Effects of Oil and Food Types on the Content of Total Polar Compounds and Acid Value*

Figure 1 illustrates the total polar compounds in oils increased linearly with frying time. Similar results were found in other studies[28-30]. The polar compounds accumulation in oil without food was slower than that in oil frying with food (Figure 1). Since the degradation of frying oil was greatly accelerated by foods, the frying condition should be controlled carefully.

The maximum level of total polar compounds at 25% was set for the regulation of deep frying oil in Taiwan. After 48 h of frying with foods, the contents of total polar compounds in both oils were shown to exceed the limit of 25% (Figure 1). Man *et al.*[31] reported that the content of total polar compounds in the oil used to fry potato chips reached 25% after 20 h.

The rates of total polar compounds formation in soybean oil frying with different foods for 48 h were similar (Figure 1A). Similar results were also found in palm olein frying with different foods (Figure 1B). The slope of regression line over time for soybean oil frying with pork chop was 0.58 and that for palm olein was 0.44, indicating that the rate of total polar compounds formation in palm olein frying with foods was lower than that of soybean oil.

Rapid measurements results of the content of total polar compounds in oils by using Testo 270 and Ebro FOM 310 were shown in Figure 2 and Figure 3, respectively. Comparing the results in Figure 1, the rate of total polar compounds formation in used palm olein obtained by using Testo 270 (Figure 2B) was similar to that obtained by the standard method (Figure 1B). Noticeably, Testo 270 tended to overestimate the content of total polar compounds in used soybean oil (Figure 2A).

The Ebro-L tended to give the content of total polar compounds in used soybean oil (Figure 3A) similar to that obtained by the standard method (Figure 1A), but Ebro-SL overestimated the content of total polar compounds in used soybean oil (Figure 3C). The content of total polar compounds
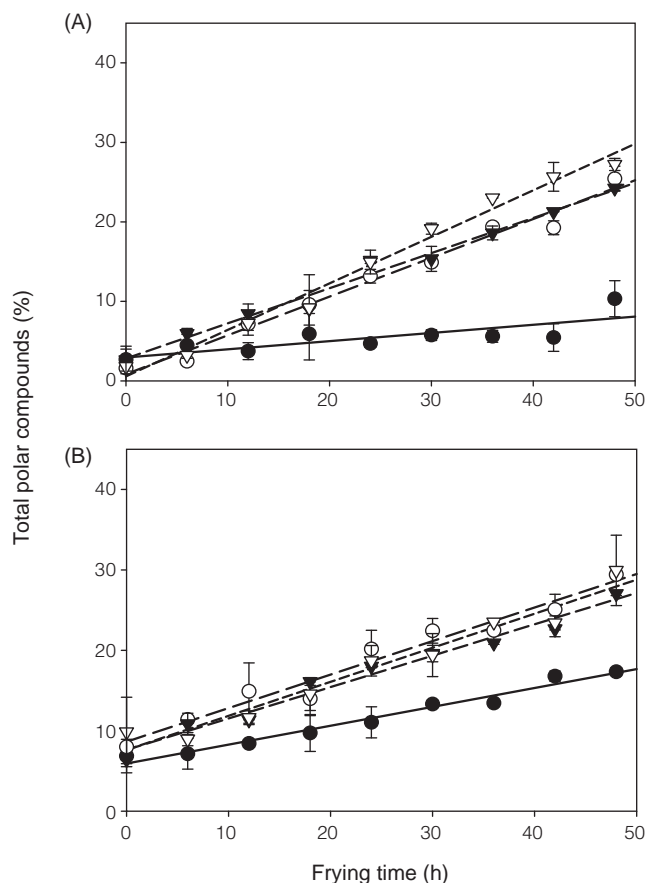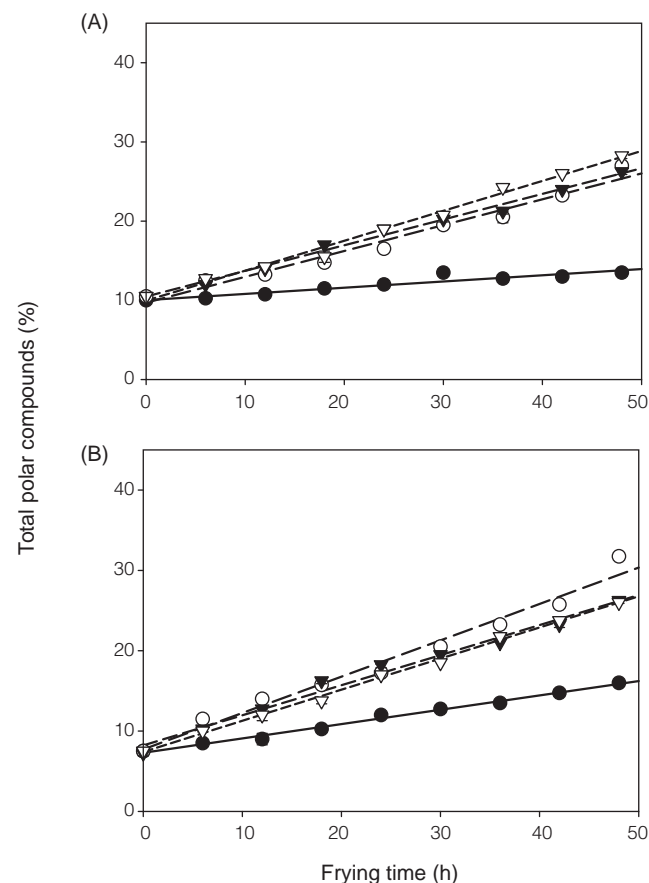
Figure 1. Formation of total polar compounds tested by the standard method in soybean oil (A) and palm olein (B) during the 48-h period of frying without food (●), with French fries (○), chicken leg fillet (▼), and pork chop (▽). The bars at each data point represent the standard deviation of duplicate measurement.

Figure 2. Formation of total polar compounds tested by Testo 270 Deep-frying Oil Tester in soybean oil (A) and palm olein (B) during the 48-h frying period without food (●), with French fries (○), chicken leg fillet (▼), and pork chop (▽). The bars at each data point represent the standard deviation of duplicate measurement.
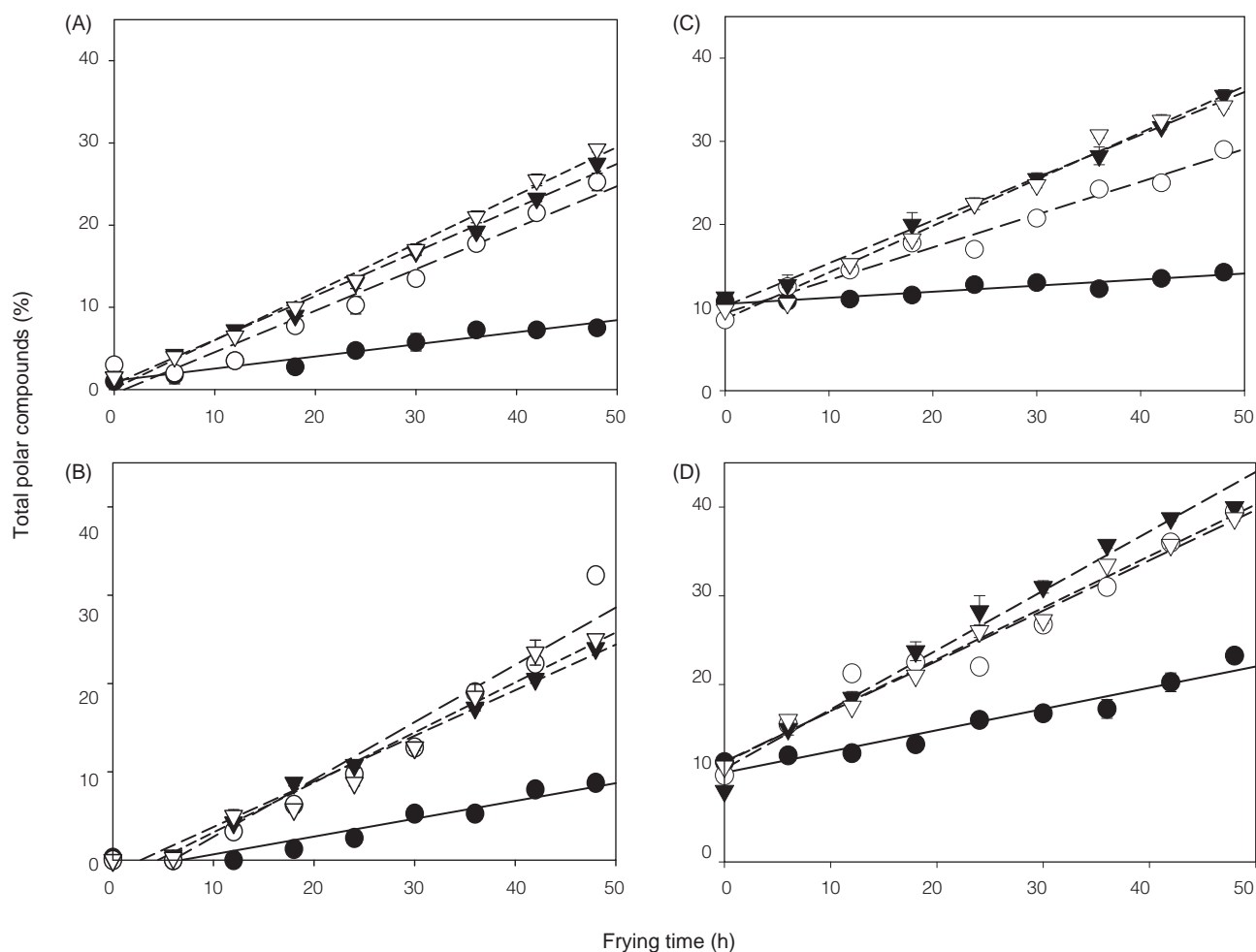
Figure 3. Formation of total polar compounds tested by Ebro FOM 310 liquid mode and semiliquid mode in soybean oil and palm olein during the 48-h frying period without food (●), with French fries (○), chicken leg fillet (▼), and pork chop (▽). The bars at each data point represent the standard deviation. (A) soybean oil-liquid mode, (B) palm olein-liquid mode, (C) soybean oil-semiliquid mode, (D) palm olein -semiliquid mode.

in used palm olein was underestimated by Ebro-L (Figure 3B *vs.* Figure 1B), but overestimated by Ebro-SL (Figure 3D). It can be concluded that Ebro FOM 310 in liquid mode was more suitable for measuring the quality of soybean oil in frying, while Testo 270 was a better choice for the quality evaluation of palm olein.

The changes of acid value in soybean oil and palm olein by the standard method and 3M Shortening Monitor (rapid method) for 48 h of frying with diverse foods were also observed in this study (data not shown). After 48 h of frying with foods, the acid value in soybean oil was still far below the limit of 2 mg KOH/g using the standard method, but an acid value that exceeded the limit was observed by the 3M Shortening Monitor after 36 - 40 h of frying.

In order to understand the effects of oil and food types on the content of total polar compounds and acid value in frying oils, a two-factor factorial design with repeated measurements was established. Since the repeated measurements of the content of total polar compounds and acid value within each oil and food type combination were significantly increased ($p < 0.01$) with frying time, the time was set as a

correlated random effect for statistical analysis.

The type of oil significantly affected the content of total polar compounds and acid value ($p < 0.001$) (Table 2), and this has also been found in many studies[14,33-35]. The result showed a pronounced increase of total polar compounds in soybean oil during frying (Figure 1). The relative autoxidation rate of linoleic acid was 40 to 50 times higher than that of oleic acid[13,26,36]. Xu *et al.*[35] mentioned that linolenic acid content was a critical factor affecting the quality of used oil during frying. Soybean oil has a greater amount of linoleic acid (51%) and linolenic acid (7%) in its fatty acid composition and is more liable to oxidation. Palm olein mainly consists of saturated fatty acids and monounsaturated fatty acids, and thus more stable. The polar compounds were derived from oxidation and thermal reaction of oil during frying. Therefore, more polar compounds in soybean oil was generated than in palm olein during frying because of the faster rate of oxidation.

The short chain and unsaturated fatty acids can facilitate hydrolysis and oxidation in frying oil. According to Erickson[26], soybean oil that predominantly contain

Table 2. Analysis of variance for the total polar compounds (%) and acid value (mg KOH/g) in used oils measured by standard and rapid methods

Methods	Independent variables[a]											
	FT			O			F			O×F		
	df	MS	P	df	MS	P	df	MS	P	df	MS	P
Total polar compounds												
Standard method	8	662.9	***	1	584.3	***	2	8.6	0.11	2	45.8	***
Testo 270	8	454.3	***	1	16.3	***	2	1.0	0.39	2	26.4	***
FOM 310 liquid mode	8	1034.2	***	1	94.5	***	2	8.6	*	2	27.3	***
FOM 310 semiliquid mode	8	975.6	***	1	526.7	***	2	66.7	***	2	21.2	**
Acid value												
Standard method	8	2.1	***	1	7.82	***	2	0.3	0.27	2	0.24	0.32

[a] FT = Frying time, O = Oil, F = Food, and O×F = Interaction between oil and food.
$* \, p < 0.05$, $** \, p < 0.01$, $*** \, p < 0.001$

unsaturated fatty acids should generate more FFA than palm olein during frying, but an opposite result was observed in our study. Other studies also confirmed this result that the rate of FFA formation in palm olein was higher than that in soybean oil during frying[14,31]. Since not only the fatty acid content but also the storage condition of oil might affect the rate of hydrolysis and oxidation during frying, different rates of FFA formation might be observed by different researchers.

Food type did not change the content of total polar compounds and acid value in the frying oils significantly ($p > 0.05$) in this study (Table 2). Water and fat in food released into the oil during frying will cause oil degradation. Based on the results from other studies[37,38], French fries, chicken leg fillet and pork chop contained roughly the same amount of moisture (70%) and fat (4.5%). As might be expected, the rates of hydrolysis were not significantly different. Furthermore, the influences of the fat leaching from the meat and of the starch from French fries or breading on oil deterioration were minor in the present study.

III. *Correlation between Standard and Rapid Methods*

Table 3 shows the measurements from all test methods were significantly correlated ($p < 0.05$). The standard methods moderately correlated with other methods ($r = 0.51$ - 0.64). The 3M Shortening Monitor had better correlations with the total polar compounds test methods ($r = 0.801$ - 0.888). In some studies[10,14,31,39], a poor relationship was observed between the acid value and the content of total polar compounds in used oil. However, Lee[12] pointed that the correlation between the content of total polar compounds and acid value exist only under the same oil, food and frying operation.

In this study, it was observed the discontinuous reading of the 3M Shortening Monitor. In addition, the color change in the bands of the 3M Shortening Monitor from blue to green or yellow was difficult to determine. Occasionally, it showed the color of blue-green or green-yellow, or showed the color

Table 3. Pearson correlation coefficient between standard and rapid methods for used oils

	ISO[a]	Testo	Ebro-L	Ebro-SL	CNS
Testo[b]	0.897*				
Ebro-L[c]	0.876*	0.983*			
Ebro-SL[d]	0.944*	0.933*	0.910*		
CNS[e]	0.576*	0.523*	0.518*	0.640*	
3M[f]	0.874*	0.826*	0.801*	0.888*	0.628*

For total polar compounds, the standard method was [a]ISO 8420 column chromatography; rapid methods were [b]Testo 270 Deep-frying Oil Tester, [c]Ebro FOM 310 in liquid mode, [d]Ebro FOM 310 in semi-liquid mode. For acid value, the standard method was [e]CNS 3647 titration; rapid method was [f]3M Shortening Monitor. The frying temperature was 180°C.
$* \, p < 0.05$.

at the edge of the band without a color in the center. When the color of the used oil was dark, the color of the band darkened and might cause a misreading. Other studies seemed to have the same problems during the determination of the band color[13,32].

The ISO 8420 column chromatography result was very close to that from Ebro-SL ($r = 0.944$) and was highly correlated ($r = 0.876$ - 0.897) with other rapid-measuring methods. Bansal *et al.*[13] also found that a moderate correlation ($r = 0.73$) between measurements from FOM 310 and the standard method of total polar compounds.

Since a high correlation exist between the standard and rapid methods, the total polar compounds measurement by the standard method can be estimated by rapid methods. Figure 4 shows the linear relationship of the total polar compounds measurements between the standard and rapid methods. The regression analysis was performed and the corresponding equations of regression line were given in Figure 4. The results were subjectively separated into oil types. A strong relationship was observed between the rapid and standard
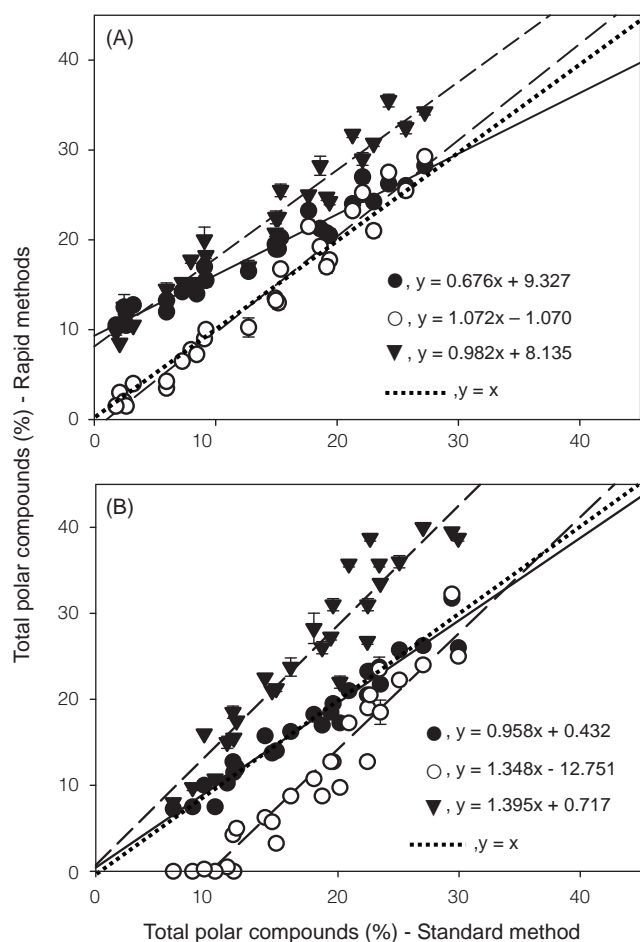
Figure 4. Comparison of the content of total polar compounds tested between the standard method and rapid methods in soybean oil and palm olein during the 48-h frying period with diverse foods. (A) soybean oil, (B) palm olein. Rapid methods were Testo 270 Deep-frying Oil Tester (●), Ebro Food Oil Monitor 310 in liquid mode (○) and semiliquid mode (▼).

methods ($R^2 = 0.905 - 0.961$). In general, the regression lines for Ebro-L and Ebro-SL were parallel and were different from the line for Testo 270. In soybean oil, Testo 270 overestimated the content of total polar compounds when the value was below 20% and underestimated it when the value was above 30% (Figure 4A). In palm olein, Ebro-SL estimated the content of total polar compounds properly at the very beginning, but then overestimated it when the value was above 10%. In comparison, Ebro-L underestimated the content of total polar compounds when the value was below 20% and estimated it better for values above 25%.

When soybean oil was used for frying, the slope of the regression line for Ebro FOM 310 was close to 1 (Figure 4A), indicating that Ebro FOM 310 well represents the standard method for monitoring the content of total polar compounds in used soybean oil. On the other hand, the slope of regression line for Testo 270 was close to 1 when the palm olein was used for frying (Figure 4B). Similar result for palm olein was reported in another study using Testo 265[13].

CONCLUSIONS

Oil type but not food type significantly affected the content of total polar compounds and acid value in used oil. The rate of total polar compounds generation in palm olein was slower than that in soybean oil during frying. However, the content of total polar compounds in palm olein reached the official limit of 25% faster than soybean oil because the fresh palm olein contained more total polar compounds than soybean oil. The measurements from all test methods were significantly correlated. Ebro FOM 310 was more suitable for monitoring the quality of soybean oil during frying, while Testo 270 was more suitable for palm olein. The 3M Shortening Monitor measured the acid value in frying oil inconsistently.

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