# Determination of Optimal Conditions for Selected Adsorbent Combinations to Recover Used Frying Oils

Song Lin, Casimir C. Akoh\*, and A. Estes Reynolds

University of Georgia, Department of Food Science & Technology, Athens, Georgia 30602-7610

ABSTRACT: The treatment of frying oils with adsorbents could practically extend the frying life of oils. Combined synthetic adsorbent treatment of used frying oils was studied the first time. The combinations of four commonly used filter aids: Britesorb (Br), Hubersorb 600 (HB), Frypowder (Fr), and Magnesol (Ma) were evaluated for frying oil recovery. AOCS official methods were used to evaluate their adsorptiveness, including free fatty acids (FFA), conjugated diene value, total polar components, oxidative stability index (OSI), and absorbance at 420 nm. The selected combinations HB+Ma+Fr and HB+Ma+Br exhibited consistent high recovery abilities on various used oil samples. A 3, 3, and 2% HB, Ma, and Fr, respectively, for the first combination (F), and 2, 3, and 2% HB, Ma, and Br, respectively, for the second combination (B) were the most effective. The optimal treatment duration was 6-9 min and 3-6 min for combinations F and B, respectively, which reduced FFA by 82.6-87.6%, absorbance by 26.8–32.6%, and Foodoil Sensor readings by 5.6-8.6%. Addition of antioxidant, such as 50 ppm butylated hydroxytoluene and 50 ppm propyl gallate, increased the OSI value by 48.9-80.8%. Such adsorbent combinations may be used in practical operation to extend frying life of frying oils and improve the healthy aspects of used frying oils.

Paper no. J9019 in JAOCS 76, 739–744 (June 1999).

**KEY WORDS:** Adsorbent, adsorbent combination, adsorption, antioxidant, butylated hydroxytoluene, filter aids, frying oil, oil recovery, propyl gallate.

Frequent filtration of frying medium with filter aids efficiently maintains the frying life of fats and improves the health aspect of finished products by controlling buildup of free fatty acids (FFA) without adverse effect on the flavor (1). The applications of adsorbents were demonstrated as having ability to control fat-soluble degradation products as well as to remove insoluble particles. Many types of filtration products were studied, including both natural and synthetic adsorbents. Usually, active forms of carbon, calcium, silica, alumina, and magnesia are major constituents of such products. Synthetic calcium silicate and synthetic magnesium silicate were used to reduce FFA and color, respectively (2). Magnesium silicate, among 11 commonly used adsorbents, was found to have the largest surface area and the most active sites, which were responsible for the adsorption of degradation materials, while alumina and silicates adsorbed color compounds (3,4).

During the past 10 years, more new synthetic filter aid products were studied. These are blends of silicates with magnesium and aluminum oxides, and various silicates formed by fusing lime, magnesium, and aluminum oxides with diatomaceous earth. Some of the filter aids were effective in recovering used frying oils. Regular treatment of used frying oil with Frypowder (MirOil, Allentown, PA) was found to be effective in reducing buildup of total polar compounds (TPC) and alkaline contaminant materials (ACM) (5), retarding color darkening, foaming tendency, dielectric constant changes (DCC), and reducing formation of conjugated dienes to twothirds of the values without treatment (6). However, Frypowder had no effect on reducing FFA due to acidic properties (Naylor, D., E. McGowan, P. Phengvath, and A.P. Handel, unpublished observation). Other researchers investigated mixtures of various compositions with the intention to improve the recovery ability of adsorbents. Three levels of activated carbon or silica adsorbents were blended and found to reduce FFA value, peroxide value, photometric color, polar compounds, and carbonyls by 28-59% (7). However, the researchers did not find any significant difference among the studied concentrations. Mancini-Filho et al. (8) compared the effects of bleaching clay, charcoal, magnesium oxide, Celite, and their mixtures. They concluded that DCC was improved by bleaching clay and charcoal, FFA was decreased by MgO, and color (absorbance at 420 nm) was effectively reduced by all treatments, including individual adsorbents and their mixtures. Treatment with 10% of adsorbent combination (4.5% clay, 0.5% charcoal, 2.5% MgO, and 2.5% Celite) demonstrated the highest effectiveness, which reduced DCC, color, and FFA by 18, 37, and 74%, respectively. In addition, Mancini-Filho et al. (8) added 1,000 ppm ascorbyl palmitate, 50 ppm butylated hydroxyanisole (BHA), and 10 ppm SAG100 dimethylpolysiloxane silicone antifoam into the treated oil to improve the oil stability and to prevent foaming.

The recovery abilities of nine adsorbent products and some of their combinations were compared in our previous work (9). Three combinations were found the most effective because they reduced FFA by 90.8–93.7%, improved oil stabil-

<sup>\*</sup>To whom correspondence should be addressed at Department of Food Science & Technology, Food Science Building, Room 211, The University of Georgia, Athens, GA 30602-7610. E-mail: cmscakoh@arches.uga.edu

ity by 23.4–24.7%, and decreased TPC by 6.0–17.8%. The current study was conducted to test the efficiency of selected combinations with four more used oil samples, which were subjected to a variety of foods and frying durations. The optimal conditions for desired adsorbent combinations were determined for possible use in practical frying operation.

## **EXPERIMENTAL PROCEDURES**

*Oil samples.* One fresh frying fat (F2) and five used frying fats (S1, S2, S3, S4, and S516) were obtained from a local processor. The fresh fat was a partially hydrogenated soybean oil (PHSBO) refined by Cargill Inc. (Minneapolis, MN). It was opaque at room temperature and turned pale when heated. The used fats were taken from commercial fryers after frying chicken breast, pork chop shapes, and rib meat at 360°F. The coating ingredient for the meat fried in S1, S2, and S516 was wheat flour, sugar, yellow corn flour, yeast, salt, caramel color, and oleoresin paprika, and fried continuously for 15 h. S4 was used to fry very spicy foods until acidity increased to 0.1%, while S3 was subjected to all kinds of foods with intermittent operation for 10 d.

Adsorbents. Britesorb (Br) (PQ Co., Valley Forge, PA), Hubersorb 600 (HB) (J.M. Huber Corporation, Havre de Grace, MD), Frypowder (Fr) (MirOil Company) and Magnesol (Ma) (Dallas Group of America, Inc., Jeffersonville, IN) were used in the study. We recently reported the composition and properties of these adsorbents (9). Briefly, Britsorb is a white amorphous, odorless powder composed of silicon dioxide, aluminum hydroxide, and water; Hubersorb 600 is a white amorphous odorless calcium silicate powder (6 microns); Frypowder is a white, granular, odorless powder composed of porous rhyolite, citric acid, and water; and Magnesol is a white, amorphous, odorless magnesium silicate powder (hydrous form).

Analysis tests. FFA content and TPC were analyzed with modified methods as reported previously (9). Conjugated diene value (CDV) was determined by AOCS Method Ti la-64 (10) with a W-64 Beckman Ultraviolet Photoelectric Spectrophotometer (Beckman, San Ramon, CA) by measuring the absorbance at 234 nm, oxidative stability index (OSI) by AOCS Method Cd 12b-92 with an Oxidative Stability Instrument (Omnion Inc., Rockland, MA), viscosity (V) by AOCS Method Ja 10-87 with an RV Brookfield Digital Viscometer (Brookfield Engineering Laboratories, Inc., Stoughton, MA). Absorbance at 420 nm was measured using 50% oil sample dissolved in isooctane (8) with a Spectronic 20+ spectrophotometer (Fisher Scientific, Pittsburgh, PA). Color was measured in Hunter Coordinates (L, a, and b) with a CS-5 Chroma Sensor<sup>®</sup> (Applied Colorsystems, Inc., Charlotte, NC), and a 4.0 mL (10×10×45 mm) cuvet (Fisher Scientific, Pittsburgh, PA). Foodoil Sensor (FOS) readings were taken with an NI-21B or NI-21C FOS Oil Quality Analyzer (Northern Instrument Co., Lino Lakes, MN).

*Fatty acid profile.* The fatty acid compositions of the oils were analyzed with an HP 5890 Series II gas chromatograph

JAOCS, Vol. 76, no. 6 (1999)

(Hewlett-Packard, Avondale, PA). A DB-225 fused-silica capillary column ( $30 \times 0.25$  mm i.d.) (J&W Scientific, Folsom, CA) and flame-ionization (FID) were employed. The injector and detector temperatures were 250 and 260°C, respectively. Helium was used as carrier gas with a total flow rate of 23 mL/min, and the isothermal analysis was carried out at 205°C for 15 min. The sample was methylated with 3 mL of 6% HCl in methanol at 75°C for 2 h before injection. The fatty acid methyl esters were quantitated with heptadecanoic acid (17:0) ester as an internal standard.

Adsorbent treatment. The adsorbent was added to oil at 150°C, stirred and filtered through a #41 Whatman filter paper (Whatman International Ltd., Maidstone, England). The filtrate (treated oil) was stored for further analysis in a sealed vessel at 4°C after flushing with nitrogen. The efficiency of three adsorbent combinations previously blended at 1:1(:1) by weight, HB+Ma, HB+Ma+Fr and HB+Ma+Br (9) were further evaluated using four used frying oils and five methods, namely: FFA, CDV, TPC, OSI and absorbance at 420 nm. The desired concentration of each component in selected adsorbent combinations (HB+Ma+Fr and HB+Ma+Br) was determined using three levels (1-3%), adopting Latin Square block experimental design. For example, a  $2 \times 3$  design (two used frying oils S1 and S3 and three concentrations of each adsorbent, levels 1, 2, and 3% by weight of oil as factors) was used to determine the optimal concentration of each adsorbent in the combination treatments. The order of treatments was randomly selected using a random number table. Then, the oil was subjected to the adsorbent combinations with appropriate concentrations during a time-course study from 0 to 15 min, and consequently, the optimal treatment time was determined. Finally, the oil was treated using desired combination(s) at proper time, followed by addition of 50 ppm antioxidant, butylated hydroxytoluene (BHT) and/or propyl gallate (Sigma Chemical Co., St. Louis, MO), into the treated oil to improve OSI. The OSI value is related to the resistance to oxidation and frying-life of recovered oils.

*Data analysis.* At least three replications for each combination and each oil sample were performed with each test. All the values were converted to percentage improvement before being subjected to statistical analysis. The averages and standard deviation were calculated, followed by statistical analysis using SAS program (11). The Duncan's multiple range test was used to determine the differences at 0.05 significance level.

## **RESULTS AND DISCUSSION**

Sample analysis. S1, S3, and S516 had relatively high values of FFA, TPC, and peroxide value (PV). S2 was high on CDV while S3 and S4 exhibited high absorbance at 420 nm (Table 1). All samples gave lower L values (lightness) and higher a and b values than fresh oil (F2), indicating that they were darker, more reddish and yellowish compared to the fresh oil. Frying with spicy ingredients resulted in a highly viscous S4, suggesting that serious polymerization occurred

TABLE 1	
Sample Parameters	5

	Fresh 2	Used 1	Used 2	Used 3	Used 4	Used 5
Samples	(F2) <sup>a</sup>	(S1)	(S2)	(S3)	(S4)	(\$516)
FFA <sup>b</sup> (%)	$0.06 \pm 0.01$	$0.66 \pm 0.02$	$0.20 \pm 0.01$	$0.76 \pm 0.09$	$0.32 \pm 0.02$	$0.69 \pm 0.03$
CDV (%)	$1.40 \pm 0.05$	$1.66 \pm 0.10$	$1.93 \pm 0.12$	$1.73 \pm 0.17$	$0.15 \pm 0.03$	$1.30 \pm 0.18$
TPC (%)	$4.0 \pm 1.0$	$8.4 \pm 0.5$	$5.9 \pm 0.2$	$10.1 \pm 1.0$	$2.5 \pm 0.5$	$9.9 \pm 2.2$
PV (meq/kg oil)	$1.38 \pm 0.09$	$2.05 \pm 0.06$	$1.57 \pm 0.05$	$2.64 \pm 0.04$	$1.06 \pm 0.04$	$2.55 \pm 0.16$
OSI (h)	$22.25 \pm 1.54$	$7.67 \pm 1.47$	$10.37 \pm 2.02$	$9.63 \pm 0.57$	NA <sup>c</sup>	$6.63 \pm 1.02$
Absorbance @ 420 nm	$0.07 \pm 0.01$	$0.36 \pm 0.01$	$0.19 \pm 0.01$	$0.87 \pm 0.01$	$0.79 \pm 0.06$	$0.45 \pm 0.03$
Color (Hunter Coordinates)						
L	$46.20 \pm 0.12$	$41.61 \pm 2.45$	$44.73 \pm 0.91$	$36.06 \pm 0.33$	$27.40 \pm 0.12$	$41.26 \pm 0.28$
a	$-1.26 \pm 0.02$	$0.91 \pm 0.09$	$-1.12 \pm 0.05$	$5.70 \pm 0.05$	$3.39 \pm 0.07$	$1.06 \pm 0.09$
b	$5.89 \pm 0.07$	$14.14 \pm 1.26$	$10.26 \pm 0.02$	$14.80 \pm 0.08$	$7.21 \pm 0.06$	$13.97 \pm 0.60$
FOS readings	0.00	$2.33 \pm 0.53$	$0.66 \pm 0.08$	$0.93 \pm 0.05$	NA	$0.94\pm0.06$
Viscosity (cP @ 25°C)	$91.5 \pm 0.5$	$102.1 \pm 0.5$	$93.1 \pm 0.6$	$89.7 \pm 0.8$	$118.7 \pm 0.1$	$89.9 \pm 0.1$
Fatty acid profile (wt%)						
16:0	$14.4 \pm 0.4$	$13.6 \pm 1.3$	$13.3 \pm 0.7$	$13.7 \pm 1.1$	NA	$13.4 \pm 0.9$
16:1n-7	Trace	Trace	Trace	$0.6 \pm 0.1$	NA	$0.5 \pm 0.1$
18:0	$4.5 \pm 0.2$	$4.5 \pm 1.0$	$4.3 \pm 0.8$	$3.0 \pm 0.5$	NA	$4.7 \pm 0.2$
18:1n-9	$44.1 \pm 0.2$	$42.6 \pm 1.8$	$41.9 \pm 1.7$	$44.9\pm0.8$	NA	$37.9 \pm 0.7$
Unknown	$1.6 \pm 0.5$	$2.3 \pm 0.5$	$2.7 \pm 0.2$	$1.7 \pm 0.9$	NA	$2.3 \pm 0.7$
18:2n-6	$32.9 \pm 1.0$	$32.5 \pm 0.7$	$34.0 \pm 1.1$	$32.3 \pm 0.5$	NA	$33.9 \pm 0.7$
18:3n-6	$0.3 \pm 0.1$	$3.3 \pm 1.9$	$1.2 \pm 0.7$	$1.3 \pm 0.5$	NA	$1.0 \pm 0.1$
18:3n-3	$2.4 \pm 0.4$	$2.9 \pm 0.8$	$2.7 \pm 0.5$	$3.2 \pm 0.7$	NA	$2.3 \pm 0.1$

<sup>a</sup>F2 is a fresh oil, while S1, S2, S3, S4, and S516 are the used frying oils.

<sup>b</sup>FFA = free fatty acids, CDV = conjugated diene value, TPC = total polar components, PV = peroxide value, OSI = oxidative stability index, and FOS = Foodoil Sensor.

<sup>c</sup>NA, Not applicable.

in this sample. The low values of TPC and PV in S4 were probably due to the severe secondary degradation caused by overcooking.

*Evaluation of adsorbent combinations.* The adsorbent combinations HB+Ma, HB+Ma+Fr, and HB+Ma+Br were most effective on recovery of a used frying oil (F516) in our previous work (9). These combinations were further tested in four used frying oils with varied degradation in the current study (Table 2). Generally, the acidity of all used oils was remarkably reduced by treatment with adsorbent combinations.

TABLE 2

Even though S2 was not severely abused, with a FFA of 0.20% (Table 1), its FFA values after treatment dropped by 25-50%. In terms of absorbance at 420 nm, the darker the color, the better the oil was recovered. For example, the color of S2 was not darkened much, resulting in absorbance decrease from 0.19 to only 0.14–0.18. On the other hand, the absorbance of much darker samples, S3 and S4, was regenerated to a high degree (39–42% and 79–85%, respectively). The means calculated for adsorbent combinations and oil treatability were compared as shown in Table 3. It was obvi-

The Farameter of Four Recovered Osed Hying Ons with Three Ausorbent Combinations									
	Filter	FFA	CDV	TPC	OSI	Absorbance			
Samples <sup>a</sup>	aid <sup>b</sup>	(%)	(%)	(%)	(h)	(@420 nm)			
S1	1	$0.18 \pm 0.02$	$1.73 \pm 0.13$	$7.16 \pm 0.94$	7.3 ± 1.8	$0.26 \pm 0.01$			
	2	$0.13 \pm 0.01$	$1.73 \pm 0.26$	$7.06 \pm 0.47$	$6.7 \pm 1.3$	$0.26 \pm 0.03$			
	3	$0.15 \pm 0.03$	$1.77 \pm 0.18$	$6.91 \pm 0.52$	$7.0 \pm 1.3$	$0.28 \pm 0.05$			
S2	1	$0.15 \pm 0.01$	$1.84 \pm 0.11$	$5.74 \pm 0.73$	$7.0 \pm 1.9$	$0.18 \pm 0.01$			
	2	$0.11 \pm 0.02$	$1.67 \pm 0.05$	$6.17 \pm 0.95$	$6.8 \pm 2.4$	$0.14 \pm 0.01$			
	3	$0.10 \pm 0.03$	$1.66 \pm 0.15$	$6.01 \pm 0.92$	$6.7 \pm 3.0$	$0.14 \pm 0.01$			
S3	1	$0.21 \pm 0.08$	$1.57 \pm 0.13$	$8.50 \pm 1.11$	$7.9 \pm 1.4$	$0.53 \pm 0.01$			
	2	$0.12 \pm 0.02$	$1.51 \pm 0.11$	$8.49 \pm 0.61$	$8.3 \pm 1.4$	$0.51 \pm 0.01$			
	3	$0.15 \pm 0.04$	$1.56 \pm 0.15$	$8.54 \pm 0.44$	$7.9 \pm 1.4$	$0.52 \pm 0.01$			
S4	1	$0.03 \pm 0.01$	$0.15 \pm 0.04$	$2.65 \pm 0.43$	NA	$0.12 \pm 0.02$			
	2	$0.03 \pm 0.01$	$0.10 \pm 0.02$	$1.51 \pm 0.70$	NA	$0.17 \pm 0.02$			
	3	$0.03 \pm 0.01$	$0.10 \pm 0.02$	$1.81 \pm 0.56$	NA	$0.13 \pm 0.01$			

The Parameter of Four Recovered Used Frying Oils with Three Adsorbent Combinations

<sup>a</sup>S1, S2, S3, and S4 are used frying oils as in Table 1.

<sup>b</sup>Adsorbent combinations: 1 = HB+Ma, 2 = HB+Ma+Fr, 3 = HB+Ma+Br, where HB = Hubersorb 600, Ma = Magnesol, Fr = Frypowder and Br = Britesorb. See Table 1 for other abbreviations.

 TABLE 3
 Summary of Improvement Ability<sup>a</sup> (%) of Adsorbent Combinations

Sample	FFA	CDV	TPC	OSI	Absorbance
S1	78.1 b <sup>b</sup>	2.2 с	15.8 a	-6.1 a	25.5 с
S2	40.9 c	4.4 bc	–2.1 b	–35.0 c	19.7 d
S3	79.5 b	10.3 b	15.6 a	–16.4 b	39.8 b
S4	89.8 a	24.3 a	17.1 a	NA	82.3 a
Filter aid <sup>c</sup>	FFA	CDV	TPC	OSI	Absorbance
1	65.5 b	3.2 b	5.8 a	–17.1 a	39.8 a
2	76.3 a	14.2 a	15.5 a	–19.9 a	42.9 a
3	74.4 a	13.6 a	13.6 a	–20.5 a	42.8 a
3.0001					

<sup>a</sup>The recovery efficiency of adsorbents on used frying oils were calculated as follows with data in Tables 1 and 2:

improvement percentage		value of untreated oil - means of treated oils
improvement percentage	=	value of untreated oil
For OSI,		
improvement percentage	_	means of treated oils - value of untreated oil
improvement percentage		value of untreated oil
by the state of th		

<sup>b</sup>Values in the same column with the same letter are not statistically different at 0.05 significance level. Values are comparisons of means for treatments and oils.

<sup>c</sup>Adsorbent combinations: 1 = HB+Ma, 2 = HB+Ma+Fr, and 3 = HB+Ma+Br, where, HB = Hubersorb 600, Ma = Magnesol, Fr = Frypowder, and Br = Britesorb. See Table 1 for other abbreviations.

ous from the samples that the improvement abilities of treatment were inversely proportional to the degree of abuse of used frying oils as indicated by the significantly high ( $P \le 0.05$ ) values for S4 and lowest values for S2.

With respect to adsorbent combinations, the significance existed in the binary combination, HB+Ma. Its treatment of used oils improved FFA and CDV removals at significantly lower levels (P = 0.0001 and 0.0006, respectively) than the two ternary combinations, HB+Ma+Fr and HB+Ma+Br. However, the improvement percentage values of OSI were always negative, indicating that the adsorbent treatment did not improve oil stability, which confirmed the report of Mancini-Filho *et al.* (8). Because the binary combination HB+Ma

 TABLE 4

 Concentration Test<sup>a</sup> for Individual Adsorbents in Two Adsorbent Combinations<sup>b</sup>

was not very effective overall, we decided to further deter-
mine the optimal conditions of combinations HB+Ma+Fr and
HB+Ma+Br to recover used frying oils S1 and S3.

Concentration determination. Although some filter media suppliers recommended between 0.1 to 2.0% for a single adsorbent (1), Mancini-Filho *et al.* (8) concluded that 10% of total adsorbents was the most efficient. Moreover, 9% charcoal combined with 6% silica was found to be the most effective on the reduction of acid value by McNeill *et al.* (7). Thus, we decided to test 1-3% concentration of each adsorbent composition. The values in Table 4 are comparisons of means for treatments and oils.

For the first adsorbent combination HB+Ma+Fr (Table 4), separated into individual concentration levels (1–3% each), the concentrations of HB 600 and Magnesol were significant  $(P \le 0.05)$  when the reduction of both FFA and absorbance at 420 nm were taken into account. HB 600 (calcium silicate) (3%) alone was consistently effective for both the FFA and absorbance reduction. As noted before (9), this is probably due to the calcium silicate content of HB 600 and large surface area, which gives it basic properties, enabling HB 600 to attract acids and polar compounds. Magnesol (magnesium silicate) (3%) alone was significantly efficient on the FFA removal (P = 0.0001) but was not significantly different from 2% Magnesol at 0.05 significance level when the absorbance were considered. Magnesol has high content of acidic and basic surface sites and has the ability to remove color bodies. The concentrations of the citric acid containing Frypowder were significant only for FFA reduction (P = 0.0019), but not at 2 and 3%. The FOS reduction was not significant in this case. Overall, 3% HB 600, 3% Magnesol, and 2% Frypowder were the desirable concentrations that can be used to recover the used frying oil.

For HB+Ma+Br (Table 4), 2% HB 600 was selected. Although 3% concentration removed more FFA than 2% HB 600, 2% HB 600 alone significantly (P = 0.017) reduced more color compounds than 3% concentration by 138%. Selected for Magnesol composition was 3% due to its improvement in removal of FFA and reduction of absorbance. The highest im-

concentration rest for individual Adsorbents in Two Adsorbent Combinations									
		FFA		Absorbance			FOS		
Adsorbent conc. (%)	HB	Ma	Fr	HB	Ma	Fr	HB	Ma	Fr
1	52.5 c <sup>c</sup>	55.4 c	59.5 b	7.0 c	6.0 b	11.1 a	1.29 a	0.94 a	–0.50 a
2	61.0 b	61.7 b	62.2 a	11.7 b	14.3 a	11.4 a	–0.38 a	0.43 a	–0.09 a
3	72.4 a	68.8 a	64.2 a	17.5 a	15.9 a	13.8 a	0.57 a	0.11 a	2.06 a
Adsorbent conc. (%)	HB	Ma	Br	НВ	Ma	Br	HB	Ma	Br
1	58.4 c	59.3 c	67.4 a	17.2 a	–0.2 b	8.1 b	–0.53 a	3.27 a	3.78 a
2	68.9 b	67.9 b	67.9 a	16.9 a	18.2 a	17.8 a	-1.01 a	1.14 ab	–0.52 ab
3	74.4 a	74.5 a	66.4 a	7.1 b	23.3 a	15.4 ab	3.26 a	–2.69 b	–1.55 b

<sup>a</sup>All data were converted into improvement percentage calculated same as in Table 3.

<sup>b</sup>Two adsorbent combinations previously used at 1:1:1 ratio by weight (10) were HB+Ma+Fr and HB+Ma+Br. See Table 1 for abbreviations.

Values in the same column with the same letter are not statistically different at 0.05 significance level. Values are comparisons of means for treatments and oils.

Time		Combination F			Combination B	
(min.)	FFA	Absorbance	FOS	FFA	Absorbance	FOS
3	$78.5 \pm 4.7 \text{ b}^{c}$	29.2 ± 2.9 bc	–2.1 ± 7.2 b	77.5 ± 1.2 b	29.9 ± 0.3 a	4.8 ± 3.2 a
6	87.6 ± 0.6 a	32.6 ± 1.1 a	8.6 ± 9.4 a	82.6 ± 1.7 a	26.8 ± 1.0 b	5.6 ± 1.8 a
9	88.7 ± 1.4 a	32.0 ± 1.8 a	-3.0 ± 7.1 b	79.1 ± 1.1 b	22.7 ± 0.7 c	6.6 ± 3.4 a
12	89.4 ± 1.2 a	31.8 ± 2.6 ab	–13.8 ± 6.0 c	77.3 ± 1.8 b	21.8 ± 0.3 d	7.7 ± 3.2 a
15	$89.7 \pm 0.7 a$	28.2 ± 2.8 c	–11.2 ± 8.1 bc	77.5 ± 1.1 b	$20.2 \pm 0.3 \text{ e}$	9.4 ± 4.3 a

 TABLE 5

 Treatment Duration Test<sup>a</sup> for Two Selected Adsorbent Combinations<sup>b</sup>

<sup>a</sup>All data were converted into improvement percentage calculated as in Table 3.

<sup>b</sup>Adsorbent combinations were: F = 3% HB + 3% Ma + 2% Fr, and B = 2% HB + 3% Ma + 2% Br. See Table 1 for abbreviations.

Values in the same column with the same letter are not statistically different at 0.05 significance level.

provement in FOS was just 3.27%. Britesorb (aluminum silicate) concentrations were not significant on FFA removal, but 2% was significant on reduction of both absorbance and FOS (P = 0.041 and 0.045, respectively). Overall, we recommend 2, 3, and 2% of HB 600, Magnesol, and Britesorb, respectively, for the HB+Ma+Br combination. This recommendation is based on comparisons of means across several different oils tested.

*Duration test.* Adsorbent suppliers and researchers suggest different treatment times for different adsorbent products. Britesorb and Purify were recommended to circulate or stir with oil for 2 min, but Naylor *et al.* (Naylor, D., E. McGowan, P. Phengvath, and A.P. Handel, unpublished observation) believed Frypowder should be allowed to be in contact with oil for 15–20 min before being filtered out of the oil. To determine the optimal treatment time, we kept adsorbent combinations in the frying oil at 150°C for 15 min, with sampling every 3 min. Data for used oils S1 and S3 treated with the same adsorbent combination were averaged and reported.

For the combination F, which contains 3% HB 600, 3% Magnesol and 2% Frypowder, the FFA values continuously decreased (Fig. 1). However, the statistical analysis (Table 5) indicated that it was significant (P = 0.0001) only at 3 min, suggesting that 6–15 min treatment with combination F can reduce FFA concentration in used frying oils. The absorbance at 420 nm (Fig. 1) dropped initially, kept decreasing slightly until 12 min, and then increased. Therefore, 6–9 min treatment was preferred for absorbance reduction, though it was insignificantly different from 12 min. The FOS reading was reduced at first and reached the lowest point at about 6 min, followed by a significant elevation thereafter (P = 0.0004). We concluded that 6 min was the optimal time to remove adsorbent combination F from the used frying oil.

The treatment duration for combination B (2% HB 600, 3% Magnesol and 2% Britesorb) was determined. Again, 6 min was the desired treatment time for acceptable FFA content and FOS values (Fig. 1). However, 3 min gave greater improvement in absorbance than 6 min (Table 5), which was significantly (P = 0.0001) better than 9–15 min. We recommend 3–6 min treatment with adsorbent combination B.

Antioxidant test. BHT was used to improve the stability of treated frying oils, due to its overall higher stability than ter-

tiary butylhydroxyquinone (TBHQ), butylated hydroxyanisole (BHA), and PG under frying temperature (12). According to Mancini-Filho *et al.* (8), 50 ppm BHT was efficient. We used 50 ppm in this study. As shown in Figure 2, the addition of BHT extended OSI values of treated oils by 0–32.8% and 12.7–41.4% with adsorbent combinations F and B, respectively. This implies that antioxidants can be added to treated frying oils to extend shelf life. Statistical analysis confirmed that the efficiency of BHT was significant at 0.017 level, regardless of the oil source and treatment with adsor-



**FIG. 1.** Changes in quality parameters during 3–15-min treatment of used oil with adsorbent combinations. A represents the trials with adsorbent combination F (3% HB 600 + 3% Magnesol + 2% Frypowder), and B represents combination B (2% HB 600 + 3% Magnesol + 2% Britesorb).  $\blacklozenge$  = free fatty acid (FFA),  $\blacksquare$  = absorbance at 420 nm, and  $\blacktriangle$  = Feedoil Sensor (FOS) readings.



**FIG. 2.** The effectiveness of the addition of antioxidants, 50 ppm butylated hydroxytoluene (BHT) and/or 50 ppm propyl gallate (PG). A is the oxidative stability index (OSI) changes of treated used frying oils (S1 and S3) with adsorbent combinations F (3% HB + 3% Ma + 2% Fr) and B (2% HB + 3% Ma + 2% Br), as affected by the addition of BHT. B is the OSI improvement of treated used oil (S516) with F and B combinations by addition of antioxidants BHT and/or PG. The initial number represents the used oil samples, such as 1 = S1, 2 = S2 and 516 = S516. The letters, F and B, are the adsorbent combinations subjected to the used oil.

bent combinations. However, in comparison to PG (Fig. 2), BHT was less effective in improving OSI values. The addition of combined BHT and PG significantly (P = 0.0001) increased OSI value more than BHT or PG alone. Therefore, the oil stability were improved by 48.9 and 80.8% for treated used frying oil (S516) by adsorbent combinations F and B, respectively.

The adsorbent combinations HB+Ma+Fr and HB+Ma+Br consistently demonstrated effectiveness on recovery of used frying oils when applied to various sources of oil samples. A 3, 3, and 2% HB 600, Magnesol and Frypowder, respectively,

and 2, 3, and 2% HB 600, Magnesol and Britesorb, respectively, were the most effective combinations. After treatment with combination F for 6–9 min, and addition of 50 ppm BHT and 50 ppm PG, the value of FFA, absorbance at 420 nm, and FOS reading were reduced by 87.6, 32.6, and 8.6%, respectively, and OSI values were extended by 48.9%. On the other hand, when treated with combination B for 3–6 min, the improvement percentage was 82.6, 26.8, 5.6 and 80.8% for FFA, absorbance, FOS and OSI, respectively. Therefore, such optimized adsorbent combinations could be used in practical operation to extend the life of frying oils.

#### ACKNOWLEDGMENTS

Research funded by the State of Georgia Governor's Food Processing Initiative research grant. Authors are grateful to MirOil Company, Dutch Quality Company, J.M. Huber Corporation, Filtration Sales and Services, Inc., and the suppliers of adsorbent materials for their support.

### REFERENCES

- Brooks, D.D., Some Perspectives on Deep-Fat Frying, Food Technol. 2:1091–1095 (1991).
- Jacobson, G.A., Quality Control of Commercial Deep-Fat Frying, *Ibid.* 21:43–48 (1967).
- 3. Yates, R.A., and J.D. Caldwell, Adsorptive Capacity of Active Filter Aids for Used Cooking Oil, *J. Am. Oil Chem. Soc.* 69:894–897 (1992).
- Yates, R.A., and J.D. Caldwell, Regeneration of Oils Used for Deep Frying: A Comparison of Active Filter Aids, *Ibid.* 70:507–511 (1993).
- 5. Booth, C., M. Wingett, and S. Koch, Trial of New Oil Treatment for Deep Fryers, *Food Australia* 46:372–374 (1994).
- Kim, C., I. Kim, and H. Shin, Effects of Composited Powder Treatment on the Increase of the Useful Lifetime of Frying Oil, *Korean J. Food Sci. Technol.* 20:637–643 (1988).
- 7. McNeill, J., Y. Kakuda, and B. Kamel, Improving the Quality of Used Frying Oils by Treatment with Activated Carbon and Silica, *J. Am. Oil Chem. Soc.* 63:1564–1567 (1986).
- Mancini-Filho, J., L.M. Smith, R.K. Creveling, and H.F. Al-Shaikh, Effects of Selected Chemical Treatments on Quality of Fats Used for Deep Frying, *Ibid.* 63:1452–1456 (1986).
- 9. Lin, S., C.C. Akoh, and A.E. Reynolds, The Recovery of Used Frying Oils with Various Adsorbents, *J. Food Lipids* 5:1–16 (1998).
- 10. AOCS, Official Methods and Recommended Practices of the American Oil Chemists' Society, 4th edn., American Oil Chemists' Society, Champaign, 1989.
- SAS, *The SAS System for Windows*, Release 6.11, TS040. SAS Institute, Inc., Cary, NC, 1989–1996.
- 12. Nawar, W.W., Lipids, in *Food Chemistry*, 3rd edn., edited by O.R. Fennema, Marcel Dekker, Inc., New York, 1996, pp. 225–319.

[Received September 14, 1998; accepted March 19, 1999]