

Membrane Processing of Used Frying Oils

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ABSTRACT: Studies were conducted with used frying oils in a flat membrane batch cell using five different types of polymeric membranes to decrease the soluble degradation products. During membrane processing, triglycerides permeated preferentially compared to the majority of the polar compounds including oxidation products, polymers, and color compounds. Two of the composite membranes, NTGS-AX and NTGS-2200, selectively rejected polar compounds and oxidation products to the extent of 25–48% and 24–44%, respectively. The reduction in Lovibond color values (5R+Y) was in the range of 83–93%. The viscosity of the used frying oil was reduced to the extent of 22%. The composite membranes were effective in reducing the soluble impurities, as well as insoluble particulates, without causing any undesirable changes to the oil. The membrane process appears to improve the life of used frying oils and does not have the disadvantages associated with the active filtration systems, however, for commercial application the permeate flux needs to be improved considerably.

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Deep fat-fried foods have exquisite taste, good flavor, excellent mouthfeel, and a characteristic texture. The primary function of oils during frying is to serve as a heat-transfer medium. The oil used to transfer heat energy undergoes chemical and physical changes leading to the odors, flavors, and textures associated with fried foods. A myriad of less desirable products also are formed that affect heat transfer efficiency and also the nutritional value of the incorporated oil (1).

The degradation reactions occurring to the oil are the result of heat, air, and moisture, which lead to polymerization, oxidation, and hydrolysis (2). Heat not only serves as a catalyst accelerating the formation of free fatty acids during the frying process but is also responsible for the polymerization of the oil. Oxidation can occur during storage, frying, or in the package of finished product. The oxidation reaction is also greatly influenced by the presence of metal ions, especially copper and iron. Moisture is responsible for the hydrolysis of triglycerides resulting in the creation of free fatty acids,

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monoglycerides, diglycerides, and in some extreme cases, glycerol (3). Extraneous matter also contributes to oil breakdown (2). The buildup of cracklings increases the rate of development of free fatty acids (FFA) and, when they are left in the fryer for extended periods of time at elevated temperatures, will cause the oil to darken and develop off-flavors. To maintain consistency in the quality of the frying oils, both insoluble particulates and soluble components have to be removed periodically, thereby decreasing the overall rate of degradation. The quality of the oil as a frying medium directly influences the quality of the food fried in it (4). Oil passes through five phases during the frying process with heating time: break-in oil, fresh oil, optimal oil, degrading oil, and runaway oil.

Filtration using passive and active filters can extend the optimal frying region. The passive systems include metal screens, rolling paper filters, paper cones, plastic cloths, canvas on plate and frame systems, systems using diatomaceous earth, and leaf filters (1). They remove only the insoluble particulates of certain sizes and have little effect on the actual chemistry of the frying oil. On the other hand, the active filtration system uses adsorbents, which not only remove the particulates but also alter the chemistry of the frying oil by removing specific oil-soluble chemical compounds such as surfactants and precursors of polymers in the heated oil, thereby decreasing the overall rate of degradation (1). Active treatment materials include diatomaceous earth, magnesium and calcium silicates, rhyolitic and pumice materials, zeolites, and aluminas. These are directly added to the frying oil and filtered out. Active filtration offers many advantages apart from extending the optimal frying region, namely, reduced energy use, improved food quality, reduced oil usage, enhanced shelf life, reduced downtime, and reduced cleanup time and costs. Though active filtration systems have great potential for enhancing frying operations, there are some associated disadvantages: the initial presence of free-form foreign powders in oil or their leaching into it, leaching of metals into oils, lack of good filtration equipment, legal issues, and capital costs (1).

Among the other approaches to improve the optimal life of frying oils, a simple membrane process offers many advantages over the conventional processes, namely, low energy consumption, ambient temperature operation, no addition of

chemicals, and retention of nutrients and other desirable components. In the past, micelle-enhanced ultrafiltration was used for degumming of hexane–oil miscella, and also attempts were made to remove FFA from vegetable oils, as summarized in our earlier articles (5,6).

Recovery of frying oils for further use was explored with polymeric membranes (7). Our earlier studies on membrane processing of vegetable oils showed that nonporous composite polymeric membranes selectively rejected phospholipids, color compounds, and oxidation products while retaining tocopherols without any pretreatment or dilution of crude oil with organic solvent (5,6). Encouraged with the results of the above work, we undertook the present study to explore the suitability of membrane processing to enhance the life of used frying oils by removing oil-soluble impurities, thereby extending the optimal frying region of the quality curve. For this purpose, real systems, that is, used frying oils, were used in the study.

EXPERIMENTAL PROCEDURES

Materials. The departmental canteen of the Institute kindly provided fried sunflower oil samples (I and II). Commercial samples were also obtained from local restaurants (III and IV). Four different types of polymeric composite membranes, with silicon as active layer and polysulfone (NTGS-AX, NTGS-BX, NTGS-CX) and polyimide (NTGS-2200) as support layers, were donated by M/s Nitto Denko Corporation (Kusatsu, Japan). NTGS-2200 is a commercially available membrane, whereas NTGS-AX, NTGS-BX, and NTGS-CX membranes are laboratory-made membranes varying in the thickness of the silicon layer (NTGS-AX > NTGS-BX > NTGS-CX). M/s Tonen Chemical Corporation (Kawasaki, Japan) kindly supplied polyethylene (PE) microfiltration membranes. The membranes were cut into circular discs (diameter 7.5 cm and effective area 32 cm²) before fitting in the membrane cell.

Membrane apparatus. Experiments were conducted using a flat membrane test cell (model C40-B; Nitto Denko Corporation) under a nitrogen atmosphere at room temperature. The cell was placed on a magnetic stirrer, and the magnetic spin bar fitted into the cell provided the agitation. A schematic diagram of the experimental setup is given in Scheme 1. The required pressure was applied by adjusting the pressure regulator of the nitrogen cylinder, that is, 0.3 MPa for microfiltration membranes and 4.0 MPa for polymeric composite membranes. The stirrer spin bar speed was maintained at 600 rpm. The unit was operated in batch mode by charging the cell with 100 g of crude oil, and the experiment was stopped when permeate collection was approximately 30 g.

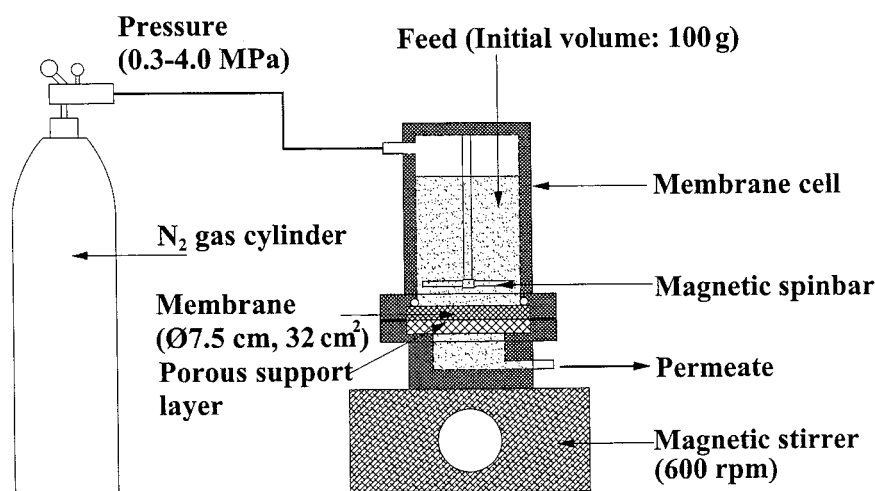
Analyses. Polar compounds in oil samples were determined by column chromatography according to AOCS official method, Cd 20-91 (8). The color was determined by Lovibond method of AOCS, Cc 13e-92, using a glass cell with an optical path length of 10 mm (8). FFA content, peroxide value, and *p*-anisidine value (*p*-A) were determined using AOCS methods, Ca 5a-40, Cd 8-53 and Cd 18-90, respectively (8). TOTOX values (an index of oil quality and stability) were obtained by the relation $TOTOX = p-A + 2 \times POV$ (8). Viscosity measurements were carried out at room temperature using a disc spindle measuring system in a digital viscometer (model DV-II+, Version 2.0; Brookfield Engineering Laboratories Inc., Stoughton, MA).

Performance parameters. The performance of the membrane process was expressed in terms of percent observed rejection (R_o) and percent reduction (PR) or increase of each component, and physical property of oil.

R_o was determined by assuming that it was constant during each batch of the experiment using the following equation (9):

$$R_o = \frac{100 \ln(C_{Rf}/C_{Ri})}{\ln(W_i/W_f)} \quad [1]$$

where C_{Ri} and C_{Rf} are the initial and final concentrations of



SCHEME 1

each component in the retentates (kg/kg-oil), and W_i and W_f are the initial and final weights of retentate (kg-oil), respectively.

PR was calculated using the following equation:

$$PR = \frac{100(C_F - C_P)}{C_F} \quad [2]$$

where C_F and C_P are the concentrations of each component or physical property in the crude and the processed oils (kg/kg-oil).

RESULTS AND DISCUSSION

Evaluation of degree of degradation. Total polar materials (TPM) is the chemical index used to determine the degree of cumulative degradation of oil (1). TPM is essentially the non-triglyceride fraction of the oil, which is broadly grouped into polymers and decomposition products based on molecular weight and polarity. The quality of the membrane-processed oil is assessed mainly in terms of TPM as recommended by the American Oil Chemists' Society (8). The values of TPM of used frying oil and membrane permeates are given in Table 1. The TPM of used frying oils varied between 12.4% and 17.0%. During membrane processing, composite polymeric membranes rejected TPM to the extent of 8.2–47.5%. NTGS-2200 membrane gave the highest rejection of 31.5% when tested for the same feed material (sample III). The degree of rejection by the remaining membranes increased with increasing thickness of the active layer. The rejection of NTGS-AX was between 24.6% and 47.5% for different feed materials. TPM is an excellent predictor of food quality for many operations (1). Regulations in a number of nations have limited the maximum permissible amount of TPM to 25% by mass. Blumenthal (4) suggested the model to describe oil degradation should be the reduction of the total amount of unaltered triglycerides, which is directly proportional to TPM

forming or accumulating in the oil. Oil absorption by fried food increases with the increase in oil deterioration (4), exacerbating the harmful effects of consuming deteriorated oil. In this context, the reduction of TPM to 40% in the membrane process is very significant for retaining fried-food quality and also for extending the life of frying oils.

Oil degradation markers. TPM in an oil contains all the compounds that influence the cooked quality of fried food and linearly relates to oil usage over time. The main concern in regulatory and quality operations is that the official method of estimating polar materials is time- and solvent-intensive. All other chemical and physical measurements determine "markers." There is no single marker compound or method which is a satisfactory index for all oils under all conditions of use in all ranges of use or abuse (1). However, in some instances or ranges, the rate of formation of specific marker compounds may coincide with the formation of polar materials. In our study, we estimated chemical markers such as POV, *p*-A value, FFA, and physical characteristics such as color values and viscosity of the feed and membrane permeates of used frying oils.

Oxidation products. POV represents peroxide and other similar oxidation products. *p*-A primarily measures secondary oxidation products, namely, α , β -unsaturated aldehydes. POV analysis is a standard test for fresh oils but has limited value for frying oils. However, POV in conjunction with *p*-A can be used for mapping past and future degradation profile. TOTOX is a single useful value for quantifying the oxygen-directed oil degradation.

The values of oxidation products of used frying oil and membrane permeates are given in Table 2. POV and *p*-A of used frying oil varied between 16.6–18.7 meq/kg and 54.8–109, respectively. Oxidation products such as peroxides, aldehydes, ketones, hydroperoxides, polymers, oxidized monomers, and the like can all cause harmful effects (10).

TABLE 1
Polar Compounds in Feed and Permeates of Used Frying Oils^a

| Sample description | Total polar materials | | |
|-----------------------|-----------------------|--------|-----------|
| | Contents (%) | PR (%) | R_o (%) |
| Used frying oil - I | 14.0 | | |
| Permeate of NTGS-AX | 8.4 | 40.0 | 47.5 |
| Used frying oil - II | 15.6 | | |
| Permeate of NTGS-AX | 10.7 | 31.4 | 37.1 |
| Used frying oil - III | 12.4 | | |
| Permeate of PE | — | | |
| Permeate of NTGS-CX | 11.6 | 6.5 | 8.2 |
| Permeate of NTGS-BX | 10.6 | 14.5 | 18.2 |
| Permeate of NTGS-AX | 9.7 | 21.8 | 24.6 |
| Permeate of NTGS-2200 | 9.0 | 27.4 | 31.5 |
| Used frying oil - IV | 17.0 | | |
| Permeate of NTGS-BX | 12.7 | 25.3 | 32.5 |
| Permeate of NTGS-AX | 11.5 | 32.4 | 37.0 |

^aPolysulfone NTGS-AX, NTGS-BX, and NTGS-CX and polyimide NTGS-2200 support layers (M/s Nitto Denko Corp., Kusatsu, Japan. PR, percent reduction; R_o , observed rejection; —, not determined.

TABLE 2
TOTOX Values of Feed and Permeates of Used Frying Oils^a

| Sample description | POV (meq/kg) | <i>p</i> -A | TOTOX | Oxidation products | |
|-----------------------|--------------|-------------|-------|--------------------|-----------|
| | | | | PR (%) | R_o (%) |
| Used frying oil - I | 18.7 | 96.6 | 134.0 | | |
| Permeate of NTGS-AX | 19.1 | 46.6 | 84.8 | 36.7 | 43.9 |
| Used frying oil - II | 16.6 | 96.4 | 129.6 | | |
| Permeate of NTGS-AX | 13.7 | 70.0 | 97.4 | 24.8 | 29.0 |
| Used frying oil - III | 16.7 | 109 | 142.4 | | |
| Permeate of PE | 14.1 | 111 | 139.2 | 2.2 | 2.9 |
| Permeate of NTGS-CX | 12.5 | 108 | 133.0 | 6.6 | 7.9 |
| Permeate of NTGS-BX | 15.0 | 99.3 | 129.3 | 9.2 | 11.0 |
| Permeate of NTGS-AX | 12.4 | 86.3 | 111.1 | 22.0 | 24.8 |
| Permeate of NTGS-2200 | 15.0 | 75.3 | 105.3 | 26.1 | 30.0 |
| Used frying oil - IV | 17.1 | 54.8 | 89.0 | | |
| Permeate of NTGS-BX | 16.4 | 50.9 | 83.7 | 6.0 | 8.3 |
| Permeate of NTGS-AX | 13.4 | 43.4 | 70.2 | 21.1 | 24.3 |

^aTOTOX, index of oil quality and stability; POV, peroxide value; *p*-A, *p*-anisidine value; PE, polyethylene microfiltration membrane (M/s Tonen Chemical Corporation, Kawasaki, Japan); for other abbreviations and manufacturer see Table 1.

Peroxide formation is particularly a major concern from a toxicological point of view. Vitamin E is destroyed in body membranes by peroxides or subsequent free radical reaction (10). Food lipid oxidation products such as peroxides, the free radicals involved in their formation and propagation, malonaldehyde, and several cholesterol oxidation products are also reported to promote atherosclerosis and coronary heart disease (11). NTGS-AX and NTGS-2200 membranes rejected oxidation products to a greater extent (24.3 to 43.9%) than did the other membranes tested, thus reducing the health risk of using membrane-processed used frying oil.

In membrane processing, mild operating conditions prevent oxidation changes. Further, from this study, it is clear that some of the peroxides and aldehydes present in the feed material were rejected significantly during the process, as indicated by the lower POV and *p*-A values in the permeates (Table 2). Freshly deodorized oil should have a POV of zero to be considered a good quality oil. TOTOX and *p*-A values of less than 4.0 and 2.0, respectively, indicate good stability. However, the TOTOX values of the permeates were much above the recommended values of fresh oils mainly due to the large amounts of oxidation products present in the feed material.

FFA. Determination of FFA is not a reliable measure of oil deterioration even though the regulations of a number of nations insist on estimating the total FFA (12). However, it is important to quantify FFA separately if they are formed in considerable quantities, although they are included in the decomposition product fraction. FFA in the oil on the surface of a fried food offers a "sink" for oxygen in a package and can be used to predict the shelf life of the food with respect to the development of rancidity (1). Greater amounts of FFA form if the initial levels of FFA present in the fresh oil are higher (3). The FFA content in the used frying oils varied between 0.14 and 0.91%. During membrane processing, FFA content did not change significantly.

Color compounds. The color measurements of used frying oil and membrane permeates were obtained using Lovibond Universal Tintometer (Table 3). The color increases dramatically during frying and is influenced by frying temperature, although the quantitative amount of chemicals causing the increase in color is very small (4). Carotenoids and xanthophylls are usually responsible for yellow color. Oxidation products, polymerization products, carbonization products, and chroma-5,6-quinones are responsible for the red color (12). In Lovibond scale, yellow color values were higher than red in the feed as well as permeated oils.

Although color should not be used as an absolute index for discarding oil, it is valuable to measure Lovibond color values when evaluating a frying system and developing quality standards (1). Regulations in a number of nations stipulate that color must be used as a sensory criterion for discarding frying oils (12). The rejections of color compounds by the membranes were very significant. Even a microfiltration membrane, which was not so effective in rejecting oxidation products, rejected color compounds to the extent of 20.8%. NTGS-AX and NTGS-2200 membranes, which showed

TABLE 3
Color Values of Feed and Permeates of Used Frying Oils^a

| Sample description | Lovibond color | | | PR of color (%) |
|-----------------------|----------------|-----|------|-----------------|
| | Yellow | Red | 5R+Y | |
| Used frying oil - I | 5.0 | 0.6 | 8.0 | |
| Permeate of NTGS-AX | 0.6 | 0.1 | 1.1 | 86.3 |
| Used frying oil - II | 7.0 | 2.0 | 17.0 | |
| Permeate of NTGS-AX | 0.7 | 0.1 | 1.2 | 92.9 |
| Used frying oil - III | 9.0 | 3.0 | 24.0 | |
| Permeate of PE | 9.0 | 2.0 | 19.0 | 20.8 |
| Permeate of NTGS-CX | 5.0 | 2.0 | 15.0 | 37.5 |
| Permeate of NTGS-BX | 2.0 | 0.7 | 5.5 | 77.1 |
| Permeate of NTGS-AX | 1.0 | 0.6 | 4.0 | 83.3 |
| Permeate of NTGS-2200 | 1.0 | 0.4 | 3.0 | 87.5 |
| Used frying oil - IV | 6.0 | 0.9 | 10.5 | |
| Permeate of NTGS-BX | 3.0 | 0.7 | 6.5 | 38.1 |
| Permeate of NTGS-AX | 0.4 | 0.2 | 1.4 | 86.7 |

^aFor abbreviations and manufacturers see Tables 1 and 2.

higher rejection of polar materials and oxidation products, also exhibited greater rejections of color compounds to the extent of over 83%. The reduction in yellow color was comparatively higher than red in the majority of the cases.

Viscosity. The viscosity values of used frying oil and membrane permeates are given in Table 4. Formation of polymers during frying is mainly responsible for the changes in the viscosity of the oil (12). Viscosity of oils increased during frying and was influenced by frying temperature rather than frying oil type. The viscosity of the used frying oil was reduced to the extent of 21.6% (from 55.2 to 43.3 mPa·s) using NTGS-AX membrane. Viscosity of the frying oil is an important factor determining the total volume of oil sticking in the large cavities in the crust of the food product (12). Higher viscosity resulted in a larger volume of oil in the fried food; oil absorption in potatoes increased during frying from 8.5% in fresh oil to 15% in degraded oil due to the increase in viscosity (12). Hence, reduced viscosity will result in less oil absorption in fried products. The reduction in viscosity indicates that polymers, which are the high molecular weight fraction of the degradation products, are reduced in the oil.

TABLE 4
Viscosity of Feed and Permeates of Used Frying Oils^a

| Sample description | Viscosity (mPa·s) | PR (%) |
|-----------------------|-------------------|--------|
| Used frying oil - I | 49.8 | |
| Permeate of NTGS-AX | 46.2 | 7.2 |
| Used frying oil - II | 57.2 | |
| Permeate of NTGS-AX | 48.0 | 16.1 |
| Used frying oil - III | 55.2 | |
| Permeate of PE | 54.6 | 1.1 |
| Permeate of NTGS-CX | 53.4 | 3.3 |
| Permeate of NTGS-BX | 48.1 | 12.9 |
| Permeate of NTGS-AX | 43.3 | 21.6 |
| Permeate of NTGS-2200 | — | |
| Used frying oil - IV | 57.3 | |
| Permeate of NTGS-BX | 49.8 | 13.1 |
| Permeate of NTGS-AX | 46.8 | 18.3 |

^aFor abbreviations and manufacturers see Tables 1 and 2.

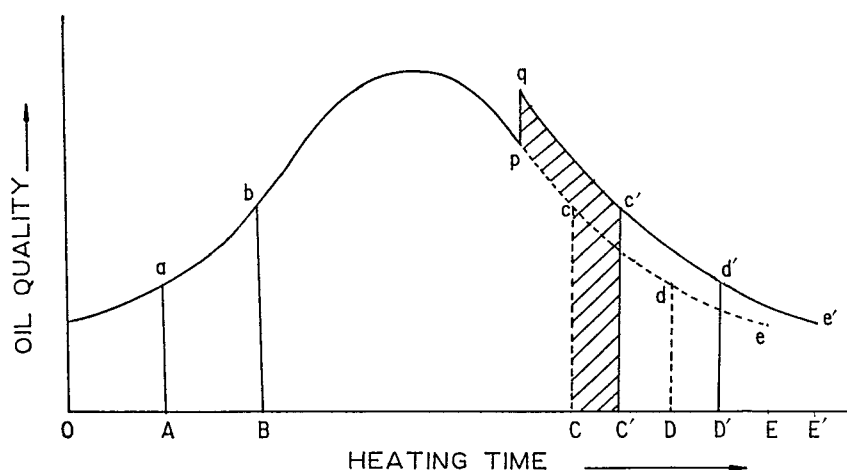


FIG. 1. Impact of membrane processing on used frying oil quality. For each of the following zones, the areas for normal oil (----) and membrane-processed oil (—) are, respectively, break-in, O-A, O-A; fresh, A-B, A-B; optimum, B-C, B-C'; degrading, C-D, C'-D'; runaway, D-E, D'-E'. C-C' represents the extended life of frying oil, p-q, membrane process, and the cross-hatched area is the increased area of the optimum zone.

Permeate flux. The flux values remained practically constant throughout the experiment. The polymeric composite membranes used in the present study gave permeate flux values in the range of 0.05–0.56 kg/(m²·h) at 4 MPa operating pressure and room temperature (less than 30°C). NTGS-AX and NTGS-2200 membranes, which showed higher rejection performance, gave flux values of 0.12 and 0.06 kg/(m²·h), respectively. These membranes gave flux values of 0.67 and 0.08 kg/(m²·h), respectively, with crude screw press-expressed groundnut oil at 3 MPa and 40°C (6). The normalized flux values obtained with used frying oils are lower compared to crude oil values, which may be due to the effect of operating temperature as well as lack of proper prefiltration of feed. The permeate flux needs to be improved considerably for industrial adoption.

Extended life of used frying oil. Blumenthal (4) proposed the frying oil quality curve and related it to fried food quality. During membrane processing of used frying oils, the quality of the oil is vastly improved as indicated by the PR in TPM (up to 40%). The quality curve comprises five distinct regions: break-in oil, fresh oil, optimal oil, degrading oil, and runaway oil. However, we felt that the optimal zone in that curve should extend beyond the peak and should not abruptly end at the peak. Hence, this feature was incorporated in the modified quality curve after consulting the original author (Blumenthal, M.M., personal communication). Oil quality is judged by the combined effects of heat transfer properties of the oil, its chemistry related to nutrition, and the sensory characteristics of the foods fried in it. However, the oil quality curve predominantly reflects the sensory characteristics of the fried foods. The impact of membrane processing on the quality of oil with heating time has been conceptually depicted on the modified oil quality curve (Fig. 1); it is a conceptual depiction, which may not be exact but approximates the true ef-

fect of the membrane process. The improvement in oil quality results in an extended life of used frying oils and consequently increases the area of the optimal-frying region.

At low surfactant concentrations, there is little cooking of the exterior or the interior of the food, whereas at high surfactant concentrations foods have overcooked exteriors and undercooked interiors. Oil quality increases along the curve up to the peak, owing to the increase in surfactant concentration, and then decreases because of the increase in the concentration of surfactants as well as other degradation products. The onset of membrane processes (line p-q in Fig. 1) could be anywhere toward the end of the optimal frying region, but the typical starting point is shown as p in Figure 1. During membrane processing, TPM are reduced and the oil quality further increases, thus increasing the total area of the optimal-frying region (hatched area in Fig. 1).

Performance of polymeric membranes. During the membrane process, triglycerides permeated preferentially, compared to the majority of the polar compounds including oxidation products, polymers, and color compounds, in polymeric composite membranes. This observed behavior could be mainly attributed to the solution-diffusion effect, that is, interactions between each solute and membrane (silicon active layer). The polymeric composite membranes were effective in reducing the soluble impurities as well as insoluble particulates without causing any undesirable changes in the oil. This approach appears to improve the life of used frying oils. The membrane process does not have the inherent disadvantages associated with the active filtration systems that were discussed earlier in this paper. Moreover, the reduction in viscosity of the used frying oils would result in reduction in oil absorption in the fried products. Besides, greater reduction in color compounds would increase the organoleptic acceptability of the oil. This study indicated that membrane pro-

cessing would extend the time that the oil stays in its optimal region/stage of oil quality, producing optimal quality fried foods. The membrane-processed oil obtained from this batch process is ready for reuse. Though the work was carried out on a batch mode, it can be employed on-line to suit continuous processing. The permeate flux would need to be improved for adoption in large-scale operations.

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