

## Isolation and Detection of Alkaline Contaminant Materials (ACM) in Used Frying Oils

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Alkaline contaminant materials are formed in frying oils during cooking. The ACM can be eluted from the same International Union of Pure and Applied Chemists-Association of Official Analytical Chemists (IUPAC-AOAC) silica gel column used to determine polar materials in frying oils. The ACM are eluted with methanol after "non-polar" and "polar" fractions already have been eluted from the column. A residue of silica gel in the methanol eluate must be insolubilized before the ACM can be identified and quantitated. IR was used to identify sodium oleate as the major constituent of ACM from a set of restaurant generated frying oil samples.

A previous paper (1) described a quick test solution for determining the amount of alkaline contaminant materials (ACM) in used frying oils. ACM include soaps and other highly surfactant materials. These ACM are formed during use of a frying oil along with other polar chemicals. Unlike fatty acids, such alkaline materials are expected to lower the interfacial tension around frying food (2). A decrease in interfacial tension between oil and food corresponds to an increase in the amount of pickup of heated oil on fried food.

The column chromatography method of Guhr and Weibel (3) has been adopted as an internationally used test (4) to determine the amount of polar materials formed in frying oils as a result of cooking. Polar materials are retained on a silica gel column while non-polar materials are eluted. The difference between the weight of applied oil and the weight of eluted non-polar materials is used to determine the amount of polar materials in the applied oil for regulatory purposes. For research purposes, the polar materials can be eluted from the column to determine the efficiency of the technique.

The more severely degraded oils deposit 1% to 2% non-elutable polar materials on the column (5). While the column chromatography method for determining polar materials in used frying oils is not perfect (6), it has been accepted by some regulatory agencies to judge the degree of use and abuse of frying oils.

Elutable non-polar and polar fractions of both fresh and used vegetable oil samples (Mel-Fry, Durkee) were examined. It was found that neither type of fraction reacted with the ACM Quick Test Solution (1), even though the whole oil, before chromatography, did react to indicate the presence of alkaline materials. This suggested that ACM are among the non-elutable polar materials which remain on the silica gel column after the recommended procedures are followed. We then developed a method for isolating and identifying ACM from the "non-elutable" polar materials left on the silica gel column.

### EXPERIMENTAL

*Sample preparation.* A premium vegetable oil consisting of partially hydrogenated soybean oil, TBHQ and

dimethyl silicone (Mel-Fry, Durkee) was heated in a 20-l capacity electric fryer. The fryer was operated at  $180 \pm 5$  C for 12 hr/day, seven days/week at a fast food restaurant. Foods fried were potatoes (75%) and breaded chicken, fish, clams and vegetables (25%), for a total of about 50 kg daily. Fresh oil was added to the fryer periodically during the day to make up for oil carried out on food.

Laboratory samples were of 50-ml volume per day taken after the hot oil was filtered through a MirOil (Allentown, Pennsylvania) plastic oil filter assembly. Polypropylene containers were used to hold/store the oil samples.

*Column chromatography.* The official IUPAC-AOAC method (4) was used to prepare a 5% hydrate silica gel chromatography column. The non-polar and polar fractions were eluted from the applied oil sample. For one gram of applied frying oil, 150 ml of petroleum ether-ether was used to elute the non-polars and 150 ml of ether to elute the polars from the silica gel column as specified. The polar fraction contains free fatty acids, among other materials.

*Isolation of ACM.* Next, 150 ml of ACM-free ACS Reagent grade methyl alcohol was passed through the still wet column to remove some of the otherwise "non-elutable" highly polar materials, including ACM, from the column. Elution of the ACM from the column with methanol leaches out relatively large amounts of alkaline silica gel solids which contaminate the eluate. The column leachings in this third (methanol) fraction were rendered insoluble by gently evaporating the eluted methyl alcohol from the round-bottom collection flask.

The dried column-residue solids from the methanol eluate are not soluble in 50 ml of phenol- and alkali-free acetone applied in 10-ml aliquots. However, ACM are soluble in the acetone, and when the ACM-containing acetone wash is mixed 1:1 with the quick test solution, green to blue colors are noted, depending on concentration of the ACM. (Acetone washings from the dried column-residue of "blank" test columns show no color development when mixed 1:1 with the ACM Quick Test Solution.)

*Identification of ACM.* Acetone washes were used to selectively solubilize the ACM from dried methanol residue. The quantitative amount of ACM in the acetone washings can be obtained by use of reference solutions made of sodium oleate in ACM test solutions (1).

The acetone washings were collected from the dried methanol fractionations of about 20 different columns. These were evaporated to about 20 ml in a rotary evaporator. Drops of the concentrated acetone solution were evaporated directly on a KBr plate to permit IR examination. This established that a significant component of the isolated ACM is a soap such as sodium oleate.

### RESULTS AND DISCUSSION

By extending the IUPAC-AOAC method for polar com-

TABLE 1  
ACM in Vegetable Frying Oil From Restaurant After Filtration

Day of use	ppm ACM found (as sodium oleate)
0 (fresh)	7.5
1	15
2	18
3	37
4 (discard)	43

ponents in frying fats, it is possible to isolate and determine the presence of ACM in fresh and used frying oils. The amount of detectable ACM isolatable from the polar materials in the restaurant generated vegetable frying oil samples described in this paper was proportional to the amount of use of the frying oil (Table 1).

The fresh Mel-Fry oil did not originally give a greenish tinge to the ACM test kit solution (1). Our experience with different brands of fresh oils is that ACM materials in the oil may be sufficiently masked by manufacture so that ACM solution will not change from its initial yellow color

except on allowing solutions to stand for a prolonged time (hours to days). However, ACM developed by the frying process always show up clearly with the ACM test solution and are readily confirmed by the above extension to the IUPAC-AOAC method.

#### ACKNOWLEDGMENTS

This work was funded by a grant from the MirOil Filter Division of Oil Process Systems, Inc., Allentown, Pennsylvania.

#### REFERENCES

1. Blumenthal, M.M., J.R. Stockler and P.J. Summers, *J. Am. Oil Chem. Soc.* 62:1373 (1985).
2. Fisher, L.R., E.E. Mitchell and N.S. Parker, *J. Food Sci.* 50:1201 (1985).
3. Guhr, G., and J. Waibel, *Fette, Seifen, Anstrichm.* 80:106 (1978).
4. Association of Official Analytical Chemists, *Official Methods of Analysis*, 14th edn., 1984, Polar Components in Frying Fats 18.074.
5. Walkling, A.E., and H. Wessels, *J. Assoc. Off. Anal. Chem.* 64:1329-1330 (1981).
6. Grandgirard, A., and F. Julliard, *Fette, Seifen, Anstrichm.* 86:98 (1984).

[Received August 7, 1985]