# Effects of Flour Lipids on Cookie Quality<sup>1</sup>

L. T. KISSELL, Y. POMERANZ, and W. T. YAMAZAKI<sup>2</sup>

### **ABSTRACT**

Flours of four wheat varieties, defatted with petroleum ether, produced smaller cookies with reduced top-grain definition than parent whole flours. Return of unfractionated free lipids to defatted flours at normal concentration restored original spread and top-grain quality. Polar and nonpolar lipid fractions alone were only partially effective in improving defatted flour; both were required for full restoration of quality. Thin-layer chromatography of lipid extracts revealed no detectable varietal differences. Flour-liqid interchange by variety produced no cookie quality differences owing to free lipid source. Cookie characteristics at normal lipid concentration were determined by varietal properties of defatted flour residues. Both whole (parent) and defatted flours increased progressively in cookie spread and top-grain score when treated with free lipids at 1.5X, 2X, and 3X normal levels.

The present study was designed to assess the contribution of flour lipid fractions to soft wheat baking quality, using the sugar-snap cookie test (1). Results of this work agree with the earlier observations of Cole et al. (2), and extend the scope to determine the effect of varietal source of lipids. In addition, the concentration effects of free lipids and of nonpolar and polar fractions on both defatted and whole (parent) flours were studied by a simplified flour-lipid recombination technique. Within the context of this study, "free" lipids are defined as those compounds extractable in petroleum ether.

The recognition of wheat-flour lipids as a quality factor in hard-wheat bread-baking dates from the work of Sullivan et al. (3,4) and Cookson et al. (5,6). More recently, Pomeranz and co-workers (7,8,9,10,11), using column and thin-layer chromatography, fractionated and analyzed lipid extracts and determined functional properties by relating the fractions to loaf-volume responses. The effects of lipid materials on a soft wheat product, the sugar-snap cookie, were first reported by Cole et al. (2). Of the many research papers on lipids since 1960, few refer directly to soft wheat quality.

## **MATERIALS AND METHODS**

Wheats of four varieties-Purkof, Thorne, Avon, and Blackhawk-grown at

<sup>&</sup>lt;sup>1</sup>Presented in part at the 55th Annual AACC Meeting, Minneapolis, October 1970. Cooperative investigation of the Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture; the Department of Agronomy, Ohio Agricultural Research and Development Center; and the Department of Grain Science and Industry, Kansas State University.

<sup>&</sup>lt;sup>2</sup>Research Chemist, Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture, Soft Wheat Quality Laboratory, Ohio Agricultural Research and Development Center, Wooster, Ohio 44691; Research Chemist, Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture, Manhattan, Kansas 66502. (Present address: Barley and Malt Laboratory, Agricultural Research Service, U.S. Department of Agriculture, Wisconsin Agricultural Experiment Station, Madison, Wis. 53705); and Research Chemist, Plant Science Research Division, Agricultural Research Service, U.S. Department of Agriculture, Soft Wheat Quality Laboratory, Ohio Agricultural Research and Development Center, Wooster, Ohio 44691.

Copyright © 1971 American Association of Cereal Chemists, Inc., 3340 Pilot Knob Road, St. Paul, Minnesota 55121. All rights reserved.

Wooster, Ohio, in 1966, were milled to straight-grade flours. Purkof is a semihard red wheat, Thorne and Blackhawk are soft reds, and Avon is soft white. A portion of each flour was batch-refluxed in a Soxhlet apparatus with petroleum ether (b.p. 35° to 60°C.). After extraction for 8 hr., each residue was aerated, remixed, and refluxed for 8 additional hr. Extracted flours were spread thinly and equilibrated with the atmosphere. Extracts were pooled by variety, clarified by centrifugation, and concentrated by solvent evaporation under vacuum below 35°C. Fractionation of petroleum-ether extracts was achieved on a silicic acid column by elution with chloroform to separate nonpolar fats, followed by methanol to remove polar materials (11). Eluates were cleared and concentrated.

Qualitative separations of nonpolar components were made by thin-layer chromatography (TLC) by using chloroform as the solvent. Polar materials were separated with a 65:25:4 (v.:v.:v.) mixture of chloroform-methanol-water. Plates were charred with a potassium dichromate-70% sulfuric acid spray and photographed under ultraviolet light.

In the baking studies, recombination of lipids and extracted flours were accomplished in two ways. Initially, lipids were returned by contact-wetting of defatted flour with 2.0% solutions of test lipid in petroleum ether. Flours were stirred constantly during the addition of lipid solutions for uniform distribution of the deposited material. Treatment concentrations were calculated as fractions or multiples of the yield of lipid actually found for the specific flour. Flours were aerated overnight to remove traces of petroleum ether.

Experience with fat additives in the cookie test (12) suggested an alternative method of restoring fatty materials directly to the sugar-shortening creamed mass in the baking procedure. Baking performance of extracted flour treated with lipids by contact-wetting was compared with results from the simplified direct-addition procedure. Subsequent study of the lipid-concentration effects were carried out by the direct-addition method only.

All lipid samples were stored at 4°C. when not in use. With the exception of the lipid additives, the Wooster cookie test (Micro Method III) of Finney et al. (1) was used without further modification.

### **RESULTS AND DISCUSSION**

The ranges of protein and ash contents were normal for flours from wheats which had been grown near Wooster (Table I). Free lipids varied from 0.81% for

Free Lipids Ratio Nonpolar Flour Protein Ash Total Nonpolar Polar to Polar % % % % Purkof 8.9 .37 .89 67.3 32.7 2.06:1 Thorne 8.4 .36 .95 69.1 30.9 2.24:1 .81 28.8 2.47:1 Avon 7.3 38 71.2 Blackhawk .93 68.7 31.3 2.19:1

TABLE I. ANALYSES OF FLOURS AND LIPID EXTRACTS<sup>a</sup>

<sup>&</sup>lt;sup>a</sup>All data on a 14% moisture basis.

Avon to 0.95% for Thorne; these tabular values were used in calculating reconstitution treatments. Thus, the 2X application to Thorne, by either procedure, contained 1.90% lipid by weight, or 0.76 g. per 40 g. of test flour. The proportions of polar and nonpolar fractions varied over a small range, with the lowest ratio of nonpolar to polar contained in Purkof and the highest in Avon.

The physical appearance of defatted flour differed markedly from its control. Removal of pigmentation, along with lipids, by the solvent increased the whiteness. Lipid-free particles settled and packed more efficiently in containers; bulk density increased 20% for Purkof and about 35% for the soft varieties. Defatted flours, when poured, were extremely dusty and difficult to handle. Reconstitution by contact-wetting with lipid solutions appeared to return defatted flours to their original physical state.

Data from experimental baking were obtained as the mean diameter of two cookies from each dough and as a relative top-grain score (ranging from 0 for no top definition to 9 for maximum uniform breakup). The standard deviation for cookie

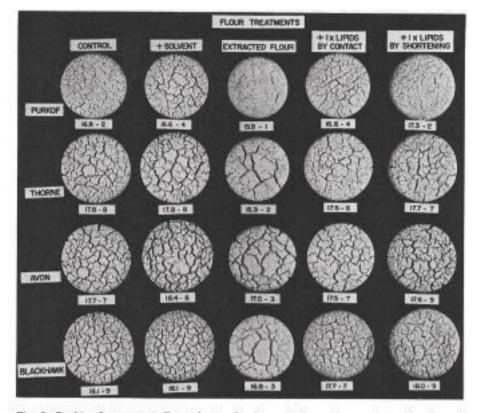


Fig. 1. Cookies from parent flours (control), after petroleum ether contact (+ solvent), following total free-lipid removal (extracted flour), and lipid restoration by contact-wetting and by addition to shortening in cookie dough. Values below cookies are mean diameters of two cookies (in cm.) and top-grain scores (range 0 to 9).

diameter was  $\pm 0.23$  cm. (LSD = 0.45 cm.) and for top-grain was  $\pm 1.3$  units (LSD = 2.5 units) for duplicate determinations.

Figure 1 summarizes the effects on the four control flours of solvent contact (+ solvent), removal of lipids (extracted flour), and return of the normal level of lipid (+1X) by contact-wetting with lipid solution and by direct addition of lipid to the cookie dough via the shortening-sugar creamed mass. Solvent effects on the parent flours were minor, with a tendency toward increased diameters and top-grain definition. The improving effects on top-grain of petroleum-ether wetting of whole flour (without removal of lipids) was noted earlier (12). The solvent-contact response may be attributed to a relocation of lipids to the flour surface by partial extraction and evaporation.

Cookies from extracted flours (Fig. 1) were significantly smaller in diameter and poorer in appearance than their respective controls. Extracted flours produced cookies of lighter than normal color; yellow hue was increased as the concentration of added lipid increased. Restoration of normal levels of free lipids by contact-wetting of defatted flour returned most of the baking potential to the varieties studied. However, the method was laborious, time-consuming, and inflexible. It was found that simple, direct application of a weighed quantity of

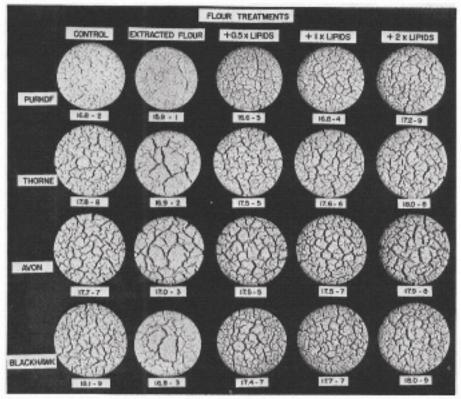


Fig. 2. Cookies from parent flours (control), following total free-lipid removal (extracted flour), and addition of 0.5-, 1-, and 2-times extracted lipids by the contact-wetting method.

lipid to the creamed sugar-shortening resulted in cookies bearing closer resemblance to the controls. The direct-addition method of lipid restoration was used for subsequent work.

The effects of lipid concentration on cookies are summarized in Fig. 2. For each variety, the addition of one-half the normal level (+0.5X) of free lipid to the extracted flour produced improvement in baking performance. Lipid treatments at normal (+1X) and twice-normal (+2X) levels resulted in cookies equal to or better than the control flours. For each variety, increased lipid applied to defatted flour gave larger cookie diameter, greater top-grain definition, and more intense cookie color.

In Fig. 3 a thin-layer chromatogram is shown of the polar and nonpolar fractions of free lipid extracts from the four varietal flours. Two hundred micrograms of each nonpolar lipid, on the left, were developed by chloroform. The spots are identified as (A) hydrocarbons and similar components, (B) triglycerides, (C) diglycerides, (D) free fatty acids, and (E) monoglycerides. Polar-lipid fractions (F) were unresolved in this solvent.

Figure 4 illustrates the TLC separation of the same lipid fractions by the polar mixed-solvent system (chloroform:methanol:water). In this case, (A) nonpolar lipids and (B) free fatty acids plus monogalactosyldiglyceride moved rapidly with the solvent front. Nonpolar materials were only partially resolved by this relatively polar solvent system. For the polar fraction, the major spot (C) is identified

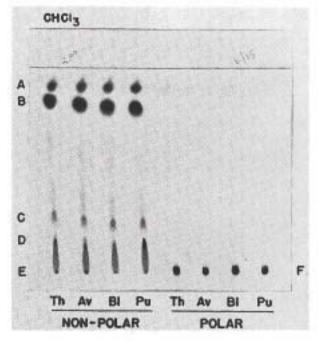


Fig. 3. Thin-layer chromatograms of nonpolar and polar fractions of petroleum-ether extracts from Thorne (Th), Avon (Av), Blackhawk (BI), and Purkof (Pu) flours. Chromatograms were developed in CHCl<sub>3</sub>. See text for identification of spots.

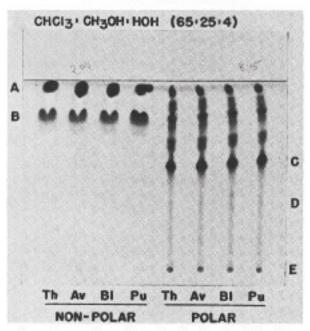


Fig. 4. Thin-layer chromatograms of nonpoler and poler fractions of petroleum ether extracts from Thorne (Th), Avon (Av), Blackhawk (BI), and Purkof (Pu) flours. Chromatograms were developed in CHCl<sub>3</sub>:CH<sub>3</sub>OH:HOH (65:25:4, v.:v.:v.). See text for identification of spots.

tentatively as digalactosyldiglyceride, the trace (D) as phosphatidyl choline, and (E) as highly polar components, including lipoprotein. Varietal differences were not shown by separations on either of these plates.

Table II presents results of an interchange of lipids in the four varietal extracted flours at the normal (+1×) level. All reconstitutions with Purkof defatted flour, regardless of lipid source, produced cookies with Purkof-type characteristics. Similar results were found for the remaining three varieties, although the 1× level of lipid was not sufficient to restore the full diameter to Blackhawk. In the foregoing data it appeared that some level of lipid was necessary to achieve cookie quality, but quality (varietal) differences resided in the base flour. These results were predicted by Cole et al. (2) from a single interchange of lipids from soft and hard wheat sources.

Polar and nonpolar fractions of free-lipid extracts from the varietal flours were returned by direct addition to doughs from their respective defatted flours at 1X, 1.5X, and 2X levels. Treatments combining nonpolar plus polar fractions (2:1 ratio) were also included at those concentrations (Table III). Additions of 1X polar or 1X nonpolar fractions alone were only partially effective in restoring the cookie-baking potential of extracted control flours. Cookie spread was essentially equal with either material, but top-grain was noticeably better with polar lipid. In every case the combination of lipid fractions resulted in cookies nearly identical to the original control flours.

After the initial improvement by +1X additions, Purkof was unresponsive to

TABLE II. RESULTS OF INTERCHANGE BY VARIETY OF FREE LIPIDS WITH EXTRACTED FLOURS<sup>a</sup>

	Control Flour		Extracted Flour		Variety Source of Lipid								
Flour					Purkof		Thorn		Avon		Blackhawk		
Variety	D	TG	D	TG	D	TG	D	TG	D	TG	D	TG	
	cm.		cm.		cm.		cm.		cm.		cm.		
Purkof	16.8	2	15.9	1	16.6	2	17.0	3	16.8	2	16.9	2	
Thorne	17.8	8	16.9	2	17.7	4	17.8	7	17.6	5	17.6	5	
Avon	17.7	7	17.0	3	17.9	8	17.7	6	17.6	7	17.7	7	
Blackhawk	18.1	9	16.8	3	17.5	8	17.5	9	17.6	8	17.7	8	

<sup>&</sup>lt;sup>a</sup>Lipids added by contact-wetting with 2% solution in petroleum ether at (1X) normal concentration. D = Diameter of two cookies, in cm.; TG = top-grain score.

TABLE III. RESPONSES OF DEFATTED AND CONTROL FLOURS TO CONCENTRATION OF NONPOLAR AND POLAR LIPIDS ALONE AND IN COMBINATION, APPLIED BY DIRECT ADDITION TO COOKIE DOUGHS

	Free	Variety Flour <sup>b</sup>							
	Lipid <sup>a</sup>	Purkof		Thorne		Avon		Blackhawk	
Flour Treatment	Level	D	TG	D	TG	D	TG	D	TG
	%	cm.		cm.		cm.		cm.	
Extracted flour	0.00	15.9	1	16.9	2	17.0	3	16.8	3
+ 1X Nonpolar lipid	0.60	16.3	3	17.4	3	17.6	4	17.4	4
+ 1.5X Nonpolar lipid	0.90	16.3	1	17.3	3	17.7	5	17.0	4
+ 2X Nonpolar lipid	1.20	16.2	2	17.8	5	17.9	6	17.3	4
+ 1X Polar lipid	0.30	16.4	3	17.2	4	17.4	4	17.4	5
+ 1.5X Polar lipid	0.45	16.4	2	17.4	6	17.6	6	17.7	7
+ 2X Polar lipid	0.60	16.4	2	17.6	7	17.9	8	17.8	8
+ 1X (Nonpolar and polar)	0.90	17.2	7	18.0	9	17.8	7	17.8	8
+ 1.5X (Nonpolar and polar)	1.35	17.3	9	18.0	9	18.1	9	18.2	9
+ 2 $ imes$ (Nonpolar and polar)	1.80	17.2	9	18.0	9	18.3	9	18.4	9
Control flour	0.90	16.8	2	17.8	8	17.7	7	18.1	9
+ 0.5X Free lipid	1.35	17.4	4	18.0	7	18.0	7	18.4	8
+ 1.0X Free lipid	1.80	17.2	8	18.2	8	18.3	8	18.2	8
+ 2.0X Free lipid	2.70	17.5	9	18.3	9	18.4	9	18.5	9
+ 3.0X Free lipid	3.60	17.6	9	18.7	9	18.5	9	18.4	9
+ 1X Nonpolar	1.50	17.1	6	18.0	8	17.6	5	18.2	8
+ 1X Polar	1.20	16.9	6	18.2	6	18.0	9	17.7	8
+ 1 $\times$ (Nonpolar and polar)	1.80	17.3	5	18.2	8	18.1	9	18.3	8
+ 2X Nonpolar	2.10	17.1	5	17.9	5	18.0	6	c	
+ 2X Polar	1.50	17.0	6	17.9	7	18.1	8	18.2	9
+ 2X (Nonpolar and polar)	2.70	•••		18.5	9				

<sup>&</sup>lt;sup>a</sup>Average quantity of free lipids present or added to flour. Exact quantity varied slightly with variety.

<sup>&</sup>lt;sup>b</sup>D = Mean diameter of two cookies, in cm.; TG = top-grain score.

<sup>&</sup>lt;sup>C</sup>Samples of lipid fractions exhausted.

higher levels of both polar and nonpolar extracts alone. The combination of lipids, however, continued to produce improvement in top-grain as lipid level was increased. Improvements in Thorne were noted with 2X additions of polar and nonpolar fats. Avon responded with increased spread and boldness of top-grain at higher levels of all lipid fractions used in the experiment. Blackhawk improved significantly with higher levels of polar lipids and with the combination treatments.

Baking responses for lipid additions to varietal control flours are also given in Table III. Both cookie diameter and top-grain score increased markedly with increased lipid at concentrations up to four times the level occurring naturally in the flour. Twice and three-times normal levels of nonpolar or polar lipid alone produced only small improvements over performance of the control flour. These data indicate that the combination of polar plus nonpolar lipids (corresponding to the original ratio of lipid fractions) is more effective than either fraction alone in improving the cookie spread of whole flours. Baking performance of a poor-quality flour can be improved significantly by additions of free flour lipid.

## Acknowledgments

The authors are indebted to R. L. Clements and H. W. Scott of the Soft Wheat Quality Laboratory for column-chromatographic fractionation of lipid extracts from the varietal flours. The assistance to Ben D. Marshall in the recombination and baking studies is also gratefully acknowledged.

#### Literature Cited

- 1.FINNEY, K. F., MORRIS, V. H., and YAMAZAKI, W. T. Micro versus macro cookie baking procedures for evaluating the cookie quality of wheat varieties. Cereal Chem. 27: 42 (1950).
- COLE, E. W., MECHAM, D. K., and PENCE, J. W. Effect of flour lipids and some lipid derivatives on cookie-baking characteristics of lipid-free flours. Cereal Chem. 37: 109 (1960).
- 3. SULLIVAN, BETTY C., and NEAR, CLEO. Chemical constituents which influence gluten quality. Ind. Eng. Chem. 19: 159 (1927).
- 4. SULLIVAN, BETTY C., NEAR, CLEO, and FOLEY, G. H. The role of the lipids in relation to flour quality. Cereal Chem. 13: 318 (1936).
- COOKSON, M. A., and COPPOCK, J. B. M. The role of lipids in baking. III. Some breadmaking and other properties of defatted flours and of flour lipids. J. Sci. Food Agr. 7: 72 (1956).
- COOKSON, M. A., RITCHIE, M. L., and COPPOCK, J. B. M. The role of lipids in baking. IV. Some further properties of flour lipids and defatted flours. J. Sci. Food Agr. 8: 105 (1957).
- 7.POMERANZ, Y. The role of the lipid fraction in growth of wheats, and in their storage and processing. Wallerstein Lab. Commun. 29: 17 (1966).
- 8. POMERANZ, Y., RUBENTHALER, G. L., DAFTARY, R. D., and FINNEY, K. F. Effects of lipids on bread baked from flours varying widely in break-making potentialities. Food Technol. (Chicago) 20: 131 (1966).
- POMERANZ, Y. A review of recent studies of wheat flour lipid in breadmaking. Baker's Dig. 40: 44 (1966).
- 10. HOSENEY, R. C., FINNEY, K. F., POMERANZ, Y., and SHOGREN, M. D. Functional (breadbaking) and biochemical properties of wheat flour components. V. Role of total extractable lipids. Cereal Chem. 46: 606 (1969).
- DAFTARY, R. D., and POMERANZ, Y. Changes in lipid composition in maturing wheat. J. Food Sci. 30: 577 (1965).
- 12.KISSELL, L. T., and DONELSON, J. R. Soft wheat test baking in the presence of silicone grease contamination. Cereal Sci. Today 13: 7 (1968).

[Received April 22, 1971. Accepted June 10, 1971]