Chemistry of Lipids in Processing and Technology of Pasta Products[1]

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ABSTRACT

Investigations were made of changes undergone by lipids in the processing of pasta products from durum wheat semolina. Three varieties of pasta were made from each of four samples of durum wheat, by both extrusion and sheet-forming techniques. Lipid extracts of semolinas and respective pasta products were analyzed for lipids, sterols, and fatty acids, by infrared and gas-chromatographic analyses. Infrared analysis of lipids extracted with acetone indicated quantitative changes in triglyceride, phospholipid, and fatty acid content in extruded and sheet-formed pasta with respect to the corresponding semolina. The IR spectrum of the sterol fraction showed a decrease of absorption at the wave length of beta-sitosterol in pasta, with respect to the corresponding semolina. GLC analysis confirmed the quantitative changes noted above in sterol and fatty acid content. These data indicate that, in the transformation of semolina into pasta through technological processing, changes occur which result in a smaller percentage of extracted fats.

Studies of wheat lipids have been particularly numerous in recent years (1). Research has involved not only the total lipids of wheat but also derived products, in relation to variety, zone of cultivation, etc. Studies have also been undertaken on the transformations which these lipids undergo during technological processing (2)—e.g., milling, breadmaking, and duration and environmental conditions of storage; on the influence which such lipids have on the behavior of bread doughs, and on the qualitative characteristics of the finished bread product. This work has been concerned with soft, hard, and durum wheats. However, with regard to transformations which occur during various stages of technological processing, research has focused mainly on derived products of soft and hard wheats, and particularly on breadmaking. Although durum wheat and derived products also have been investigated, this research was concerned chiefly with methods of identification of the two types of wheat, and particularly with detection of the presence of mill products of soft or hard wheats in pasta which has been declared as a product of durum wheat semolina (3–8).

In carrying out extraction of lipids according to the method of Brogioni and Franconi (9), we have observed that qualitative and quantitative modifications of some components of lipids were verified, both during the stage of preparation of pasta from pure durum wheat semolina and in relation to various methods of processing.

Since these aspects of the problem do not appear to have been previously examined, we initiated a line of research to point out and possibly interpret such variations. Many factors can influence the interpretation of such phenomena, since the technological phases of the work are numerous, and still more are the possibilities of variation in all of those operations concerned with production. Therefore we have limited ourselves in this pre-

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liminary paper to outlining a program of work and to reporting some of the results in the initial phases of study.

MATERIALS AND METHODS

Apparatus

Infrared spectra reported were obtained with a Perkin-Elmer 521 spectrophotometer; a sodium chloride cell with a path length of 0.4 mm. was used.

Chromatograms were obtained with a Perkin-Elmer 881 gas chromatograph, with a flame ionization detector. For analysis of sterols, a glass column packed with 3% SE-30 on 80- to 100-mesh silanized Gas-Chrom P was used. Fatty acid analysis employed a steel column packed with 15% DEGS on 80- to 100-mesh Chrom-W.

Four samples of semolina from mixed varieties of durum wheat and their respective pasta products were studied. The semolinas were those used in commercial pasta manufacture and were chosen because they are representative of commercial practice.

The pasta products—spaghetti, farfalle, and pipe rigate—were made by the following techniques:

Spaghetti made from semolinas A, B, and C was extruded through a die having a relatively high die chamber at a pressure of 100 kg./cm.^{2} Drying was done in a CPL Braibanti line for 24 hr. at an internal temperature of 55°C.

Farfalle from semolinas A, B, and C was formed out of a dough sheet extruded at a pressure of 90 kg./cm.^{2} The circular die employed was 1.5 mm. thick. The pasta was dried at an internal temperature of 45°C. for 10 hr. in a continuous dryer.

Spaghetti and farfalle made from semolina D were processed by the same techniques employed for the above pasta products from semolinas A, B, and C, with only the extrusion pressure and drying temperatures differing (see Table I).

Pipe rigate from semolina D was produced by the same extrusion techniques used for spaghetti, but with an extrusion pressure of 80 kg./cm.^{2} and a drying temperature of 53°C.

With the above techniques (extrusion and sheet-formation), pasta was made from every sample of semolina; this enabled us to compare data of three pasta samples of each semolina with the semolina itself.

Extraction of Lipids

The method of Brogioni and Franconi (9) was used. Samples were milled on a Buhler laboratory mill to pass through a sieve having 60 mesh/cm.^{2} Lipids were extracted from a 30-g. sample with 150 ml. of anhydrous acetone. After addition of the acetone, solvent and sample were gently swirled and left to stand for 24 hr. at room temperature. After filtration, the extracts were evaporated under vacuum in a glass apparatus at 45°C. The residue was weighed and dissolved in carbon tetrachloride; the amount of carbon tetrachloride was calculated for each sample to obtain a final concentration of 2% by weight.

Extraction of Sterols

The procedure used followed that of Muntoni *et al.* (10). Semolina

(50 g.) was mixed with 50 g. of sodium sulfate and placed in a desiccator for 48 hr. Sterols were then extracted with anhydrous acetone in a Soxhlet extractor for 48 hr. The extract was evaporated, and after saponification of the dry residue with an 8% solution of alcoholic potassium hydroxide for 3 hr., the unsaponifiable fraction was extracted with petroleum ether (40°–60°). Beta-sitosterol and campesterol were extracted by this method.

Extraction and Methylation of Fatty Acids

The saponified residue, obtained as above, was treated with 6*N* hydrochloric acid to liberate the fatty acids. The free fatty acids were extracted with ethyl ether and then esterified with a mixture of methanol and sulfuric acid (4% sulfuric acid by weight) for 3 hr. under reflux. The methyl esters were then extracted with petroleum ether (40°–60°).

RESULTS AND DISCUSSION

In IR analysis of semolinas A, B, C, and their respective pastas, two bands of absorption appear (Figs. 1–3). The first (1,163 cm.$^{-1}$) is due to

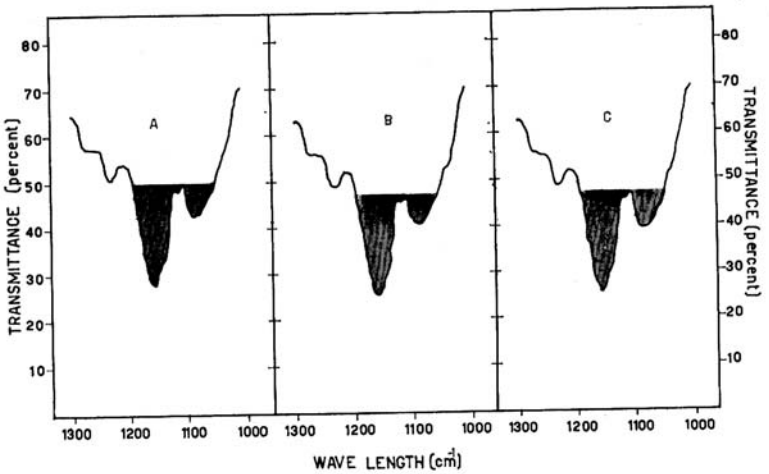

Fig. 1. IR spectra (1,300–1,000 cm.$^{-1}$) of lipids extracted from semolina A and derived products. A, semolina A; B, spaghetti; C, farfalle.

the characteristic –COOR group (ester group: triglycerides) and the second to the absorption of two characteristic groups: –COOR (triglycerides) at 1,100 cm.$^{-1}$, and P–O–C (phospholipids) at 1,070 cm.$^{-1}$

It appears from the figures that the extruded pasta shows a diminution of the band between 1,100 and 1,050 cm.$^{-1}$, with a consequent increase of the ratio of the areas of the two bands. Sheet-formed pasta, on the other hand, shows enlargement of the above-mentioned band, which results in a decrease of the area ratio.

The above variations become apparent on comparison of values obtained from the graphs of the pasta and the corresponding semolina.

According to the method of Brogioni and Franconi employed here (9), it is possible to establish a numerical index representing the ratio between the area of the second band, occurring between 1,124 and 1,219 cm.$^{-1}$ with

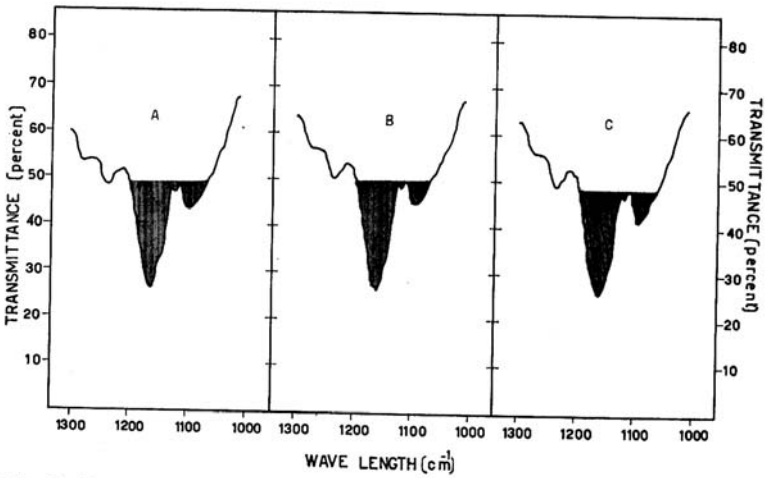

Fig. 2. IR spectra (1,300–1,000 cm.$^{-1}$) of lipids extracted from semolina B and derived products. A, semolina B; B, spaghetti; C, farfalle.

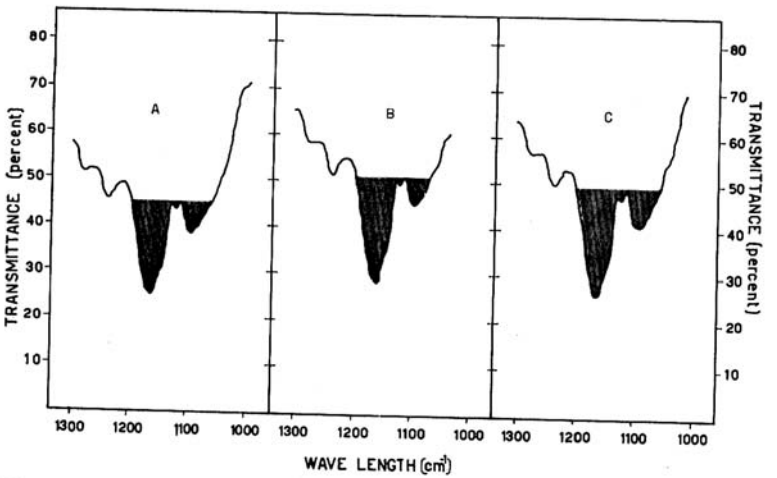

Fig. 3. IR spectra (1,300–1,000 cm.$^{-1}$) of lipids extracted from semolina C and derived pastas. A, semolina C; B, spaghetti; C, farfalle.

a maximum at 1,165 cm.$^{-1}$, and that of the first, which occurs between 1,053 and 1,111 cm.$^{-1}$, with the point of maximum absorbance about 1,099 cm.$^{-1}$. The areas of the absorption band referred to by the above authors are those bound by the absorption curve and a base line starting at a point of minimum absorption of 1,110 cm.$^{-1}$ and running parallel to the abscissa.

Experimental conditions observed in the course of preparation of the pasta are reported in Table I.

In scanning the spectra of the acetone extract (Fig. 4), another difference was noted at times. In the semolina extract, a peak of maximum absorbance appeared at 1,710 cm.$^{-1}$ which disappeared in the spectrum of the extract from the corresponding pasta. Such a peak is evidently due to

TABLE I

EXPERIMENTAL CONDITIONS IN PREPARATION OF PASTA

	EXTRACTED FATS[a]	AREA RATIO[b]	EXTRUSION PRESSURE	DRYING TEMPERATURE
	%		kg./cm.²	°C.
Semolina A	0.590	4.5	....	....
Spaghetti (long goods)	0.195	4.7	100	55
Farfalle (sheet-formed)	0.180	3.3	90	45
Semolina B	0.569	6.2	....	....
Spaghetti (long goods)	0.156	8.3	100	55
Farfalle (sheet-formed)	0.114	5.9	90	45
Semolina C	0.623	4.3	....	....
Spaghetti (long goods)	0.232	6.2	100	55
Farfalle (sheet-formed)	0.208	3.9	90	45
Semolina D	0.742	13.1	....	....
Spaghetti (long goods)	0.230	22.5	110	52
Pipe Rigate (short goods)	0.209	15.0	80	53
Farfalle (sheet-formed)	0.207	11.4	65	54

[a] According to Brogioni-Franconi (dry matter) (see ref. 9).
[b] H_1 (1,165 cm.$^{-1}$)/H_2 (1,100 cm.$^{-1}$).

TABLE II

DIFFERENCE IN DEGREE OF ACIDITY

SAMPLE	DEGREE OF ACIDITY	TRANSMITTANCE
	ml. Normal NaOH/100 g. dry matter	*% at 1,710 cm.*$^{-1}$
Semolina D	4.66	34 (peak)
Spaghetti (long goods)	2.01	58 (flex point)
Farfalle (sheet-formed)	2.18	55 (flex point)
Pipe rigate (short goods)	2.89	51 (flex point)

the presence of the –COOH group in the extract of the semolina. A difference in the degree of acidity, determined according to the official method and expressed as ml. of *N* NaOH solution per 100 g. dry substance, served to confirm the above (Table II).

To better interpret the variations observed in lipid content through IR spectrophotometric investigations, we initiated a study with gas chromatography. From the first results it appears that the methyl esters of the fatty acids of the pasta products of semolina D and the semolina itself differ in concentration. This difference is manifested by a variation in the intensity of recorder response (see Fig. 5).

A study of sterols present in wheat also was made. Figure 6 shows both spectra of beta-sitosterol standard and those of the sterol extracts obtained from samples of semolina and pasta. At the characteristic wave length of beta-sitosterol (11), a decrease of absorbance and consequently of the concentration of this substance can be noted in the extracts from pasta compared to that from the semolina.

Parallel investigations with gas chromatography confirmed the quantitative decrease noted with IR analysis and permitted good separation of the two peaks corresponding respectively to beta-sitosterol and campesterol

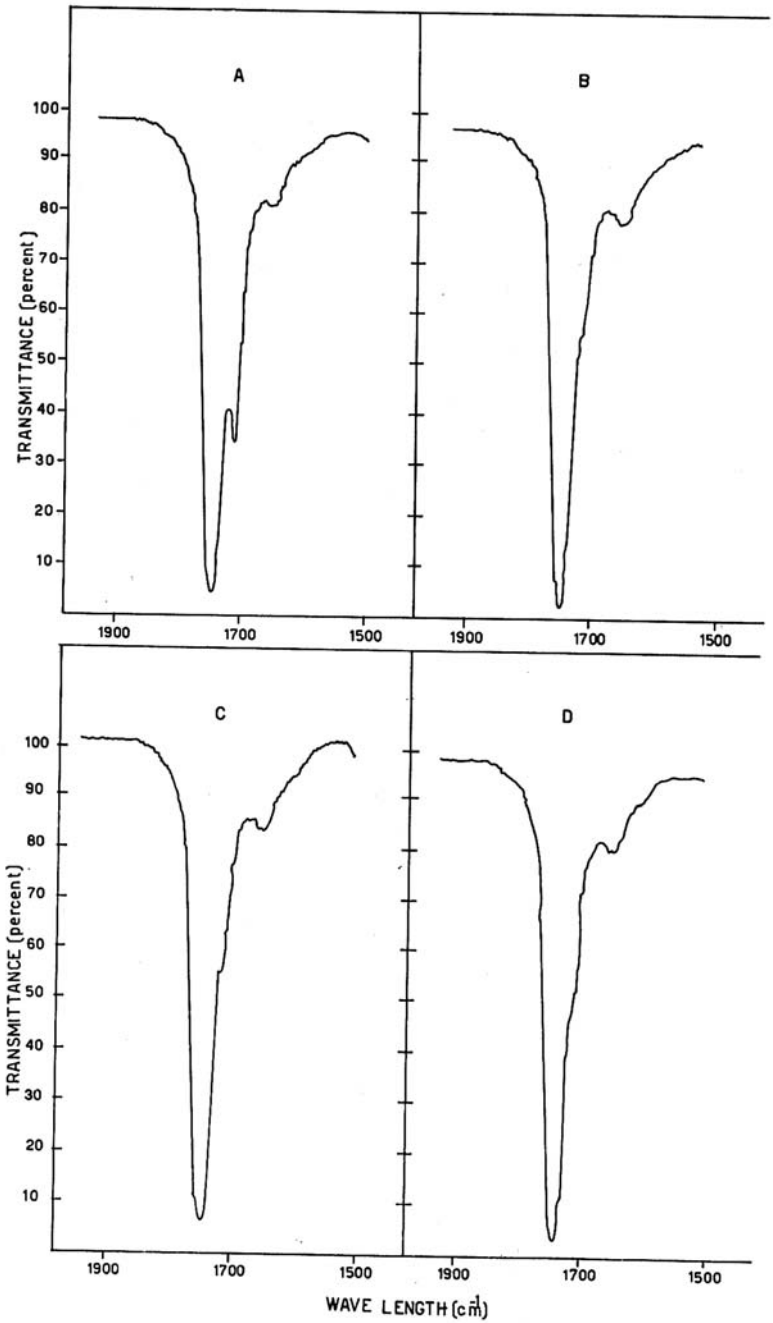

Fig. 4. IR spectra (1,800–1,600 cm.$^{-1}$) of lipids extracted from semolina D and derived pasta. A, semolina D; B, spaghetti; C, farfalle; D, pipe rigate.

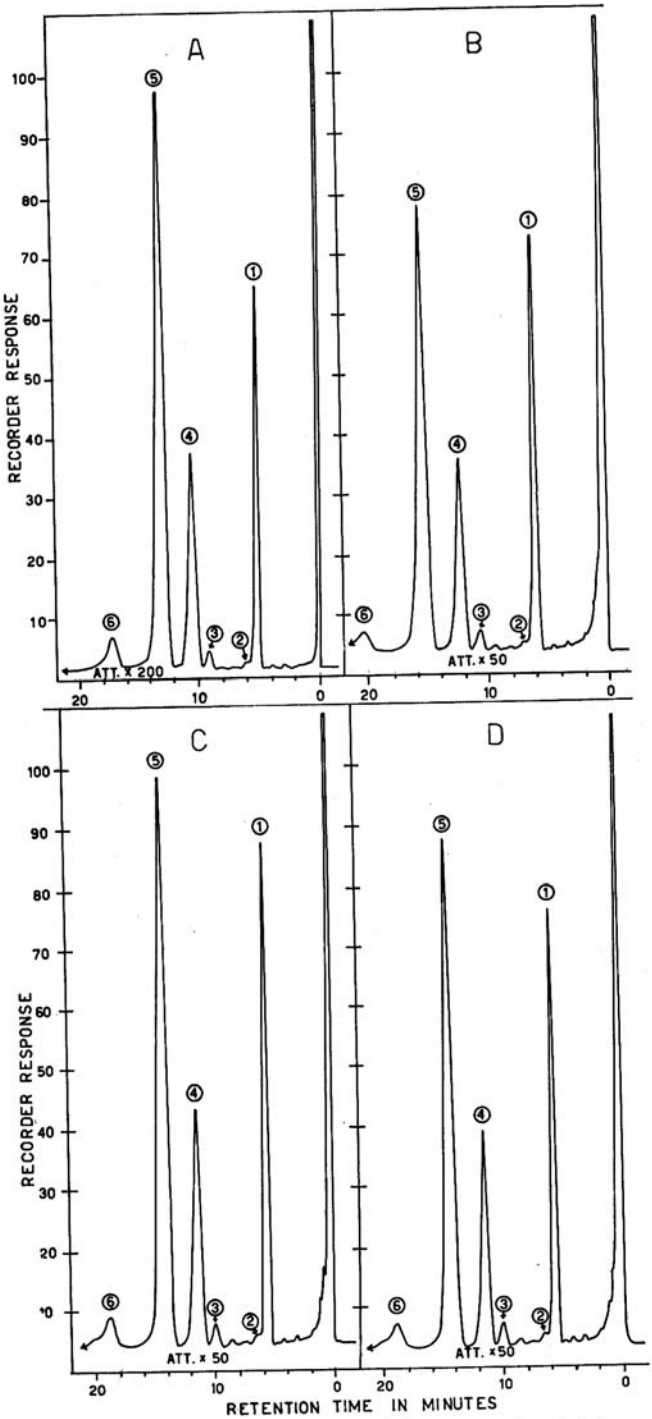

Fig. 5. GLC analysis of fatty acid methyl esters extracted from semolina D and derived pasta. A, semolina D; B, pipe rigate; C, farfalle; D, spaghetti; 1, palmitic; 2, palmitoleic; 3, stearic; 4, oleic; 5, linoleic; and 6, linolenic acid.

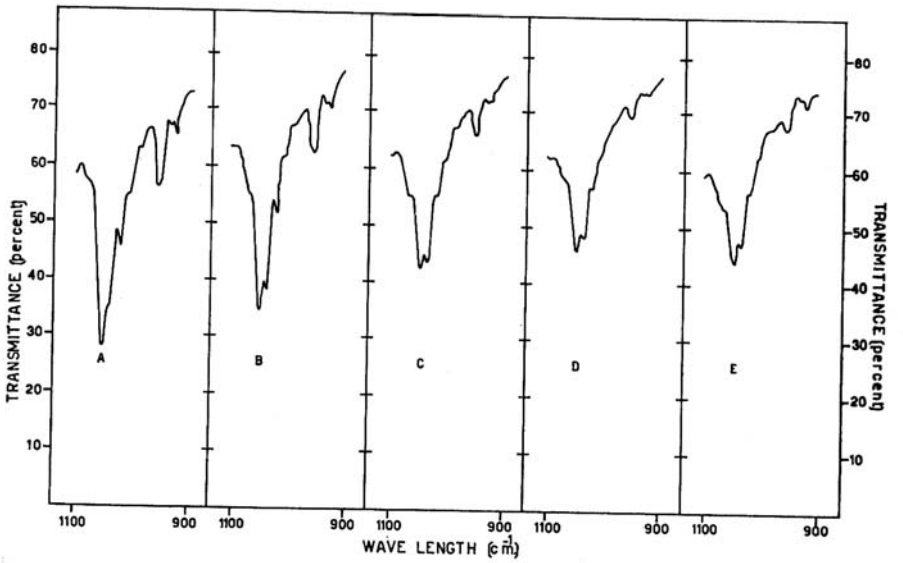

Fig. 6. IR spectra (1,100–900 cm.$^{-1}$) of steroids extracted from semolina D and derived pasta. A, beta-sitosterol; B, semolina D; C, spaghetti; D, farfalle; E, pipe rigate.

TABLE III
RELATIVE RETENTION TIMES AND AREA RATIOS

SAMPLE	RETENTION TIMES AT 225°C.		AREA RATIO: BETA-SITOSTEROL/ CAMPESTEROL
	Campesterol	Beta-Sitosterol	
	cm.	*cm.*	
Semolina D	5.8	7.2	2.3
Spaghetti (long goods)	5.7	7.2	2.2
Pipe rigate (short goods)	5.8	7.2	2.2
Farfalle (sheet-formed)	5.6	7.1	1.9

(Fig. 7). Relative retention times and area ratios of the two peaks are given in Table III.

From these data it may be concluded that, through transformation of semolina into pasta during technological processing, changes occur which result in a smaller percentage of extracted fats.

The cause of this difference in degree of extractability of wheat lipids before and after manufacture of pasta may lie in a chemical transformation undergone by the lipids when they are subjected to the screw press in the presence of the dough water, or in a binding or complexing of the lipid with some other component of the pasta. These two possibilities need not be opposed to one another; they could occur simultaneously.

Results obtained so far from analyses of the sterols have shown a quantitative decrease of the sterol extract of pasta samples, confirming the above.

These first results, which naturally cannot yet be considered conclusive, are nevertheless sufficient to warrant further research; it should include both

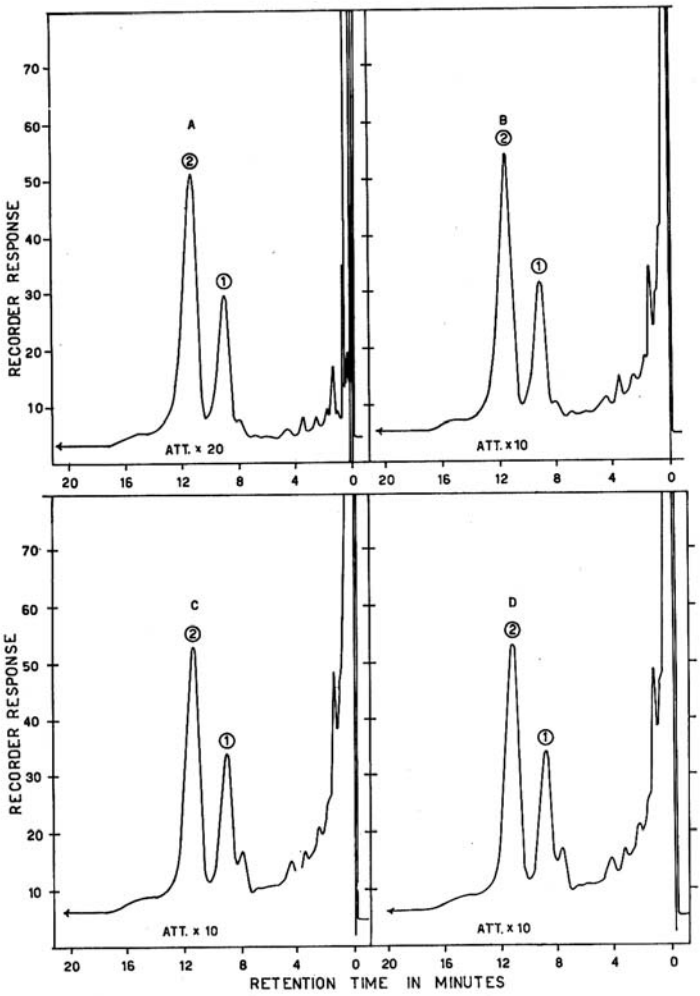

Fig. 7. GLC analysis of steroids extracted from semolina D and derived pasta. A, semolina D; B, pipe rigate; C, farfalle; D, spaghetti; 1, campesterol; 2, beta-sitosterol.

broader utilization of other methods of analysis—e.g., thin-layer chromatography for sterols (12–14)—and further development of methods already employed in studies of lipids (15,16).

Acknowledgment

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