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IMPROVEMENT OF RAPESEED LECITHIN FOR EDIBLE PURPOSES

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Rapeseed oil is the only vegetable oil produced in Poland. During its refining process a by-product, the so-called lecithin, is obtained. It can find wide application in the production of margarine as well as for baking purposes and for the use in other branches of industry. However, in comparison with soybean lecithin, which has been used for those applications for a very long time, only rapeseed lecithin of the highest quality can be considered to be used.

A comparison of rapeseed lecithin (Table I) with soybean lecithin (Table II) shows several differences.

TABLE I

COMPOSITION OF COMMERCIAL RAPESEED LECITHIN[5]

Fraction	%
Rapeseed Oil	38.1
Sterol Glycosides	7.9
Phosphatidyl Ethanolamine	17.5
Phosphatidyl Inositol	7.6
Phosphatidyl Choline(a)	6.0
Lysophosphatidyl Ethanolamine	2.0
Phosphatidyl Choline(a)	10.2
Unidentified Compounds	4.9
Residue(b)	5.8

(a) Phosphatidyl Choline divides into 2 fractions.
(b) Sum of the compounds that remain in the Chromatographic column.

TABLE II

COMPOSITION OF COMMERCIAL SOYBEAN LECITHIN[6]

Fraction	%
Soybean Oil	33.0
Phosphatidyl Ethanolamine	8.0
Phosphatidyl Inositol	20.0
Phosphatidyl Choline	21.0
Other Phosphatides	11.0
Other Compounds(a)	7.0

(a) Mainly: Carbohydrates, Sterols.

Rapeseed lecithin is characterized by its high content of phosphatidyl ethanolamine and a lower content of phosphatidyl choline compared with soybean lecithin. Commercial lecithin always contains crude oil. It is an unfavourable feature that cannot be avoided.

The fatty acid composition of the phosphatides of commercial rapeseed lecithin differs from that of rapeseed oil. The phosphatides contain only about 10% of erucic acid, more than 30% of linoleic acid and about 10% of linolenic acid. The rest consists mainly of oleic and palmitic acids. The low content of erucic acid in rapeseed phosphatides is a favourable property from the dietetic point of view.

However, much of this lecithin obtained on an industrial scale is of an interior quality, and cannot be used for food, particularly because of its unpleasant colour, bitter flavour and burned odor. Consequently it becomes desirable to develop a procedure which eliminates these deficiencies and makes it possible to use all the lecithin obtained during the rapeseed oil production and refining.

According to established methods lecithin is obtained upon the hydration of solvent-extracted oil. The gums are then dried to a moisture content of less than 1 percent. Thus, there are two factors which have an influence on the quality of the product, namely the extracted oil itself, and the technology of isolating the lecithin.

Excellent lecithin can be obtained only from sound seeds. Frequently, however, the oil industry has to use seeds of lower quality. In addition, the tendency to extract the

maximum quantity of oil from the seeds is the cause of an inferior quality of the phospholipds.

Quite often lecithin undergoes deterioration due to mistakes in the course of drying the extracted gums. High temperatures are especially harmful.

Reports published recently in the USSR[1] indicate that the main chemical changes which take place in phosphatides during the oil extraction and refining processes are caused by hydrolytic cleavage and by the formation of melanophosphatides. There are oxidation processes which play a decisive role in all stages of oil extraction and, above all, during the treatment and storage of oil.

The oxidation of phosphatides has a protective effect on oil. If they are, however, contaminated with melanophosphatides, they have a detrimental effect.

Any increase of the melanophosphatide content is accompanied by an intensive disagreeable burned odor and a bitter taste.

Our work[2] has proved that the extracts obtained differ considerably from the average composition of the given oil in the final stage of industrial extraction. The results of studies on the composition of residual fat occurring in extracted meal are of special interest to industry. It contains five times more unsaponifiables, three times more sterols, nearly 20 times more phosphorus, and the green colour is 6 to 10 times more intensive. On the other hand, there are no pigments of the carotenoid group.

Regardless of the causes the organoleptic deficiencies of lecithin must be overcome, if it is to be used in the food industry.

Several methods can bring about this improvement, and we have investigated three approaches, viz. -

- Solvent refining,
- Separation into fractions which may be used as emulsifiers,
- Exchange of the constituent oil.

I. SOLVENT REFINING

There are two known lecithin refining methods. They have been proposed for the purification of soybean lecithin by B. Rewald[3] and H. Pardun.[4]

Both these methods have been used by us for the refining of 2nd class rapeseed lecithin. The characteristics of that lecithin are given in Table III and compared with those of the 1st class lecithin.

TABLE III

CHARACTERISTICS OF RAPESEED LECITHIN

Description	LECITHIN	
	1st Class	2nd Class
Content of Acetone-Insoluble Substances %	66.5	55.9
Content of Benzine-Insoluble Substances (Impurities) %	0.22	0.41
Volatile Matter at 105°C %	0.90	1.12
Ash Content %	5.58	6.65
Iodine Number	82.2	73.2
Peroxide Number	4.5	37.2
Saponification Number	189.9	188.2
Acid Number	37.9	57.8
Acetone Acid Number	7.2	17.5
Phosphorus Content %	2.34	2.41
Taste	Sweetish	Bitter
Smell	Slight burned smell	Slight smell of rancid fat
Colour	Not uniform in texture light-brown to dark brown	Uniform in the whole mass, dark-brown
Consistency	Solid, easy crushing product	Greasy

From the above comparison it follows that the 2nd class lecithin differs from that of the 1st class both, in its chemical composition and in its organoleptic properties. The chemical composition of 2nd class lecithin does not differ from that of the 1st class lecithin after a short period of storage.

At first both grades of lecithin differ above all in their organoleptic properties. It is only during the course of storage that the acid value, peroxide value and the content

of acetone-soluble substances become higher.

The content of erucic acid in the 1st as well as in the 2nd class lecithin was almost the same, namely 17.2 and 17.9 percent respectively. When considering that the lecithin contains nearly 30 percent of oil, the content of erucic acid in the phosphatide fraction itself is correspondingly lower.

A. REFINING WITH ACETONE

Technical, anhydrous acetone in the ratio of 4 to 6:1 was used to extract the lecithin according to the method of B. Rewald[3]. Fifty grams of powdered lecithin were used in these tests. The first extraction was carried out with 300 ml of acetone. Shaking was continued for 1 hour. Next the acetone-oil miscella was decanted. Further extractions were carried out with 200 ml of acetone each time. The miscella was evaporated in portions and the content of phosphorus determined in the residue (oil). The refining results are given in Table IV.

Figures I and II illustrate the data given in Table IV.

TABLE IV

ACETONE REFINING OF RAPESEED LECITHIN

Extraction No.	Acetone Soluble Substances (Oil)	Phosphorus in Oil	Phospholipids in Oil
	%	%	%
	1st Class Lecithin		
1	22.8	0.085	2.57
2	6.9	0.102	3.06
3	2.9	0.131	3.94
4	0.6	0.298	8.94
5	1.1	0.350	10.50
	2nd Class Lecithin		
1	26.7	0.097	2.91
2	9.8	0.152	4.56
3	1.8	0.240	7.20
4	0.8	0.382	11.46
5	0.8	0.403	12.04

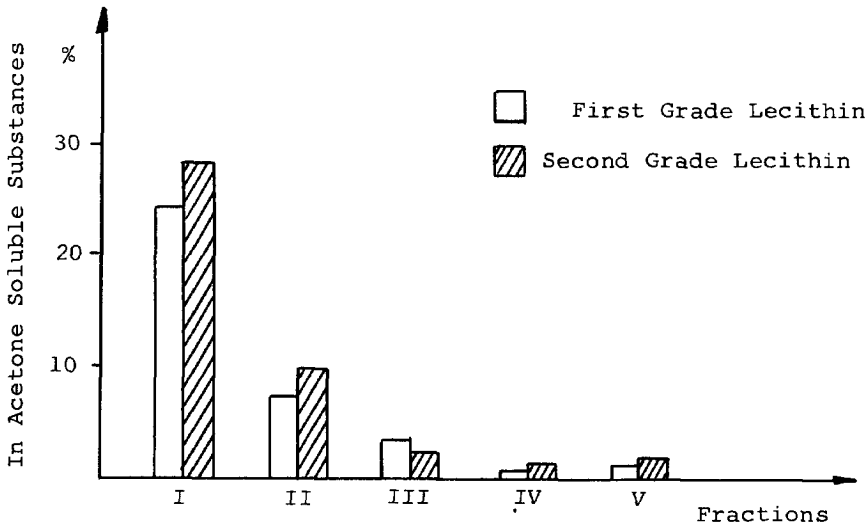


FIGURE I

THE DEPENDENCE OF THE PERCENTAGE OF THE ACETONE SOLUBLE SUBSTANCES ON THE EXTRACTION DEGREE DURING THE REFINING OF LECITHIN WITH ACETONE

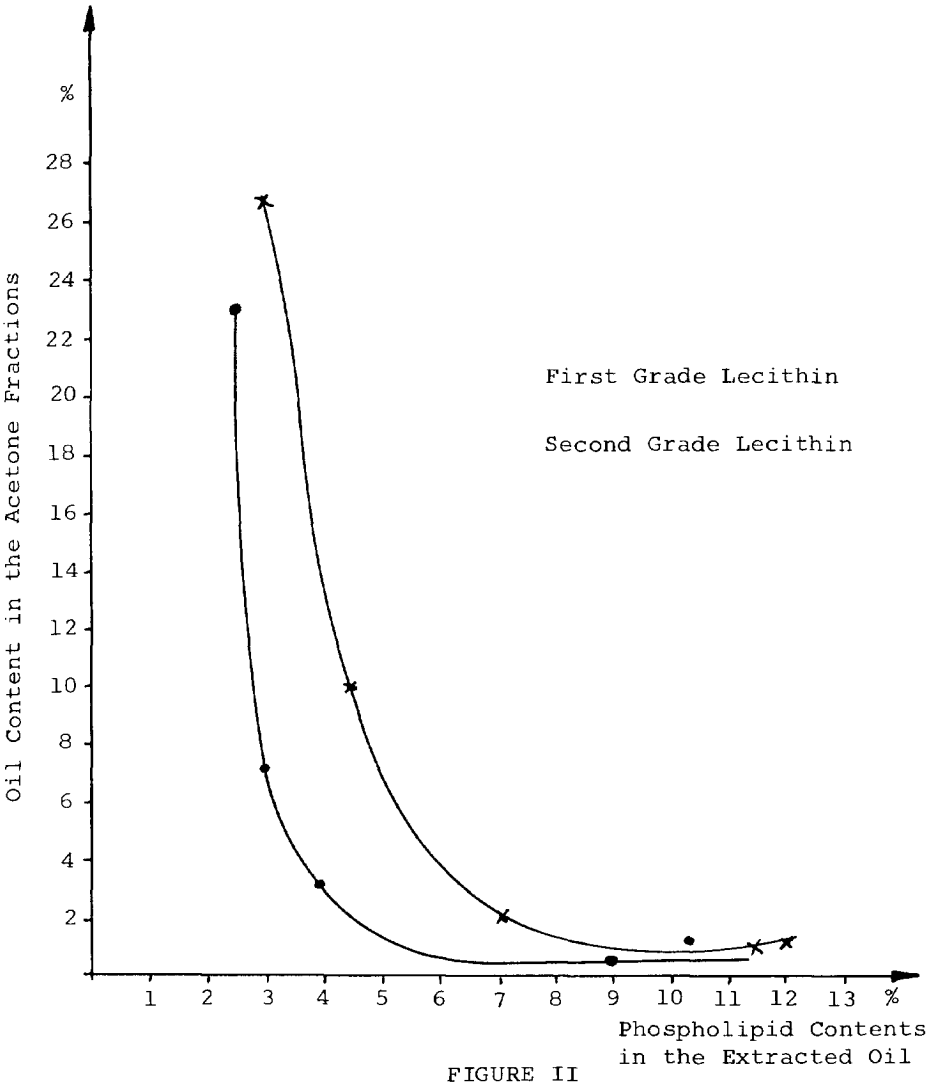


FIGURE II

THE DEPENDENCE OF THE ACETONE EXTRACT OIL CONCENTRATION ON THE PHOSPHOLIPID CONTENT IN THE EXTRACTED OIL

Table IV shows that the content of phospholipids in the oil increases as the yield of extracted oil decreases. Unfortunately, upon the first acetone treatment nearly half the phospholipids are found in the extracted oil. Although the phospholipid content of the oil of subsequent extractions is higher, the actual losses are very small.

It is easy to convert the 1st class lecithin into a powder through the oil extraction process. This, however, cannot be achieved as easily with the 2nd class lecithin, particularly when it has been stored for a long period of time. After the native oil has been separated, the lecithin was stabilized against autoxidation by adding refined oil.

The removal of the oil from the second class lecithin by acetone extraction improved its organoleptic properties. The odor of rancid fat was removed completely, but the bitter flavour was eliminated only in part. The 1st class lecithin, which had been subjected to oil separation with acetone, had a slight odor and a light colour.

As few as two extractions, which remove 70-80% of the oil, were sufficient for improving the organoleptic properties. The lecithin, however, had to be thoroughly ground by means of a disintegrator.

B. REFINING BY A MIXTURE OF SOLVENTS

In the first stage 320 ml of a mixture of 100 ml of benzine and 220 ml of acetone was used to refine 100 g of lecithin. When the product was completely dissolved the mixture was transferred to a separator, where it separated quickly into two layers. The lower benzine layer contained the phospholipids and the upper acetone layer contained the oil.

Next another portion of solvent was added to the lower layer. The extraction was carried out in four stages.

The results obtained with anhydrous acetone are given in Table V, and those with aqueous acetone in Table VI respectively.

When anhydrous acetone was used for the extraction, the two layers separated very slowly, because the benzine layer containing the phospholipids was very viscous. This affected the separation process. When aqueous acetone was used, separation took place very quickly due to the lower viscosity of the benzine layer.

TABLE V

BENZINE AND ANHYDROUS ACETONE REFINING OF RAPESEED LECITHIN

Extraction No.	Substances (Oil) Soluble in Acetone Phase	P in Oil	Phospholipids in Oil P x 30
	%	%	%
	1st Class Lecithin		
1	33.2	0.826	24.78
2	9.5	0.915	27.45
3	2.7	1.888	56.55
4	1.7	1.955	58.65
T O T A L	47.1	-	-
	2nd Class Lecithin		
1	26.7	0.305	9.15
2	11.4	0.369	11.07
3	7.6	1.550	46.50
4	2.2	0.885	26.55
T O T A L	47.9	-	-

The influence of water on the viscosity of the benzine layer was determined in an Engler viscosimeter.

Lecithin was dissolved in benzine in a ratio of 1:1 (V/V) and twice as much acetone as benzine was added. This changed the distribution of the water in that system. Figure III shows the viscosity changes of the benzine layer in response to the quantity of water added to the acetone.

As shown in Figure III the viscosity becomes much lower when water is added to the system under investigation. When the water content amounts to 5%, the viscosity is nearly one eighth that of a 2% water content. However, when the quantity of water, exceeds 5%, the layers cannot separate.

In our experiments, where successive extractions were applied, the addition of 2% water proved to be best for obtaining a proper separation at every stage of extraction. The higher yield of extracted oil by H. Pardun's method can be explained by the greater solubility of the phosphatides in the acetone

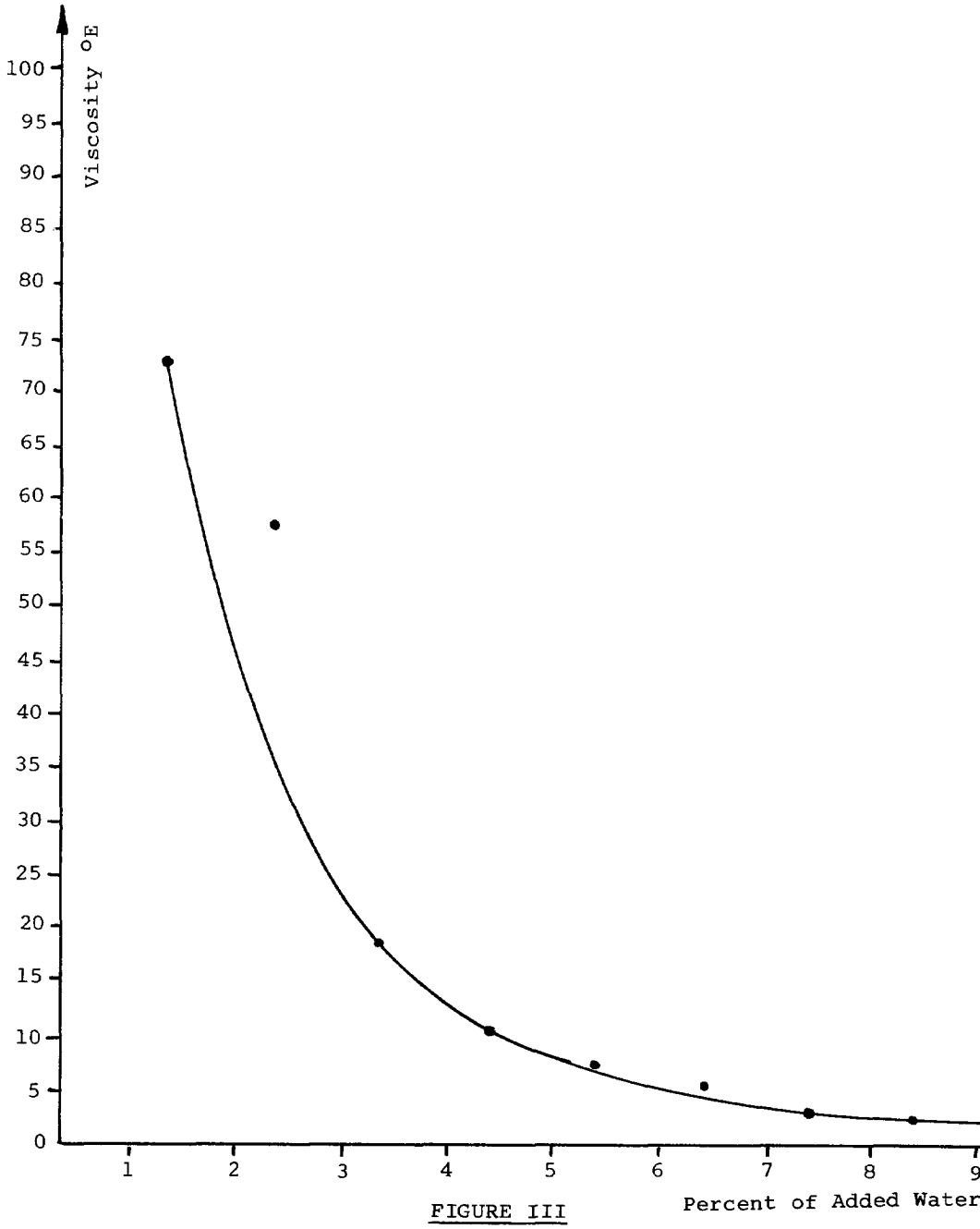


FIGURE III

THE DEPENDENCE OF BENZINE LAYER VISCOSITY ON THE WATER ADDED TO ACETONE

layer than when applying the method of B. Rewald. In the latter method only pure acetone is used, whilst in the former method the acetone layer contains benzine which is responsible for the greater solubility of phosphatides.

TABLE VI

BENZINE AND ACETONE REFINING OF RAPESEED LECITHIN WITH 2% ADDED WATER

Extraction No.	Acetone-Phase Substances Soluble in Acetone Phase	P in Oil	Phospholipids in Oil P x 30
	%	%	%
	1st Class Lecithin		
1	26.7	1.080	32.49
2	18.6	1.451	43.50
3	5.8	1.362	40.80
4	2.0	1.521	45.60
T O T A L	53.1	-	-
	2nd Class Lecithin		
1	28.3	0.768	23.04
2	13.2	0.763	22.89
3	7.1	1.195	35.85
4	7.0	1.690	50.70
T O T A L	55.6	-	-

Oil was separated from lecithin on a large laboratory scale according to the H. Pardun method with the aid of an LG 205 - type separator manufactured by Westfalia AG,Oelde. A single-stage process was applied. The results have been compiled in Table VII.

TABLE VII

REFINING OF 1st CLASS RAPESEED LECITHIN BY A MIXTURE OF BENZINE AND ACETONE WITH 2% ADDED WATER ON A LARGE LABORATORY SCALE

Residue	Extracted Substances	P in Oil	Phospholipids in Oil P x 30
	%	%	%
1	25.1	0.103	3.09
2	28.2	0.141	4.23

Solvent refining results in the improvement of the organoleptic properties of lecithin of inferior grade. It should be added, however, that acetone as well as acetone-benzine refining do not completely free the rapeseed lecithin from its unfavourable properties and above all not from those which are caused by the inferior quality of the raw material or by technological shortcomings. For instance, such properties as its burned odor can be removed only to a limited extent. This method of refining rapeseed lecithin is justified because of the possibility of replacing rapeseed oil by refined sunflower oil which will undoubtedly improve the nutritional value of the final product.

II. SEPARATION INTO FRACTIONS[11]

Another method of refining lecithin may be the separation of rapeseed phosphatides into fractions with specific emulsifying properties, i.e. into a lecithin fraction for the oil-in-water type emulsion and into a cephalin fraction for the water-in-oil type emulsion.

When choosing a suitable method, it is obviously necessary to consider the economic aspects because the emulsifiers will have to be competitive with soybean lecithin. On the other hand, the emulsifying action of pure lecithin or cephalin may be more effective in some cases than that of commercial lecithin where these two groups may act antagonistically.

A. SEPARATION OF DEFATTED PHOSPHATIDES IN A TWO-SOLVENT SYSTEM

Oil was removed from commercial rapeseed lecithin by the H. Pardun method in a benzine/acetone system with 2% of added water. Defatted phosphatides were obtained containing less than 2% acetone-soluble substances. Incomplete removal of oil is advantageous because the oil protects the phosphatides against quick oxidation.

I. RAW MATERIAL

The characteristics of commercial lecithin are given in Table VIII.

TABLE VIII

CHARACTERISTICS OF COMMERCIAL RAPESEED LECITHIN

Colour	Not uniform in texture, dark brown with light brown bands.
Odor	Characteristic smell of fresh lecithin with a slightly perceptible burned odor.
Consistency	Rather hard, clammy.
Flavor	Sweetish, slightly perceptible bitter and burned taste.

C O M P O S I T I O N	%
Acetone-Insoluble Substances	68.7
Acetone-Soluble Substances	31.1
Phosphorus	2.4

The separation of commercial lecithin and oil-free phosphatides, and next of the particular phosphatide fractions, was carried out by TLC on silica gel in a 65:25:4 chloroform:methanol: water system.

The TLC of commercial and defatted lecithin is given in Figure IV.

2. FRACTIONATION IN THE BENZINE/ETHANOL SYSTEM

The separation of rapeseed phosphatides into cephalin and lecithin fractions is most reasonable from the technological point of view. Owing to the fact that the separation takes place in a liquid medium, the whole process can be carried out with the use of separators.

In our experiments we determined the optimal phosphatide concentration in the initial benzine solution in relation to the concentration of ethyl alcohol. When it amounts to 96%, the optimal concentration of phosphatides is 0.030 g/ml. Further, the concentration of phosphatides was 0.150 g/ml and 0.073 g/ml respectively for 90% and 80% alcohol. The above values ensured an undisturbed separation of the benzine and ethanol layers at a maximum concentration of phosphatides.

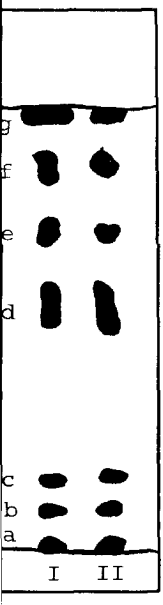
The results obtained on a laboratory scale permitted us to isolate 140 g of oil-free phosphatides. That quantity was dissolved in 2000 ml of benzine and extracted in two stages with the aid of 96% ethanol, of which 1000 ml were used in the first stage and 500 ml in the second stage respectively.

The recovered cephalin fraction amounted to 75 g and that of the lecithin fraction to 65 g.

The composition of both phosphatide fractions is given in Table IX.

The cephalin fraction had a characteristic neutral taste, a brown colour, a faint characteristic smell and was obtained as a powder.

On the other hand, the lecithin fraction had a sweet taste with a distinct bitter flavour, a light-brown colour, a characteristic smell and a clammy, slightly greasy, consistency.



a, b, c Unidentified compounds

d Phosphatidyl Choline

e Phosphatidyl Ethanolamine, Phosphatidyl Choline, Phosphatidyl Inositides

F Sterol Glycosides

G Neutral Oil

I Commercial Lecithin

II Defatted Lecithin

Eluent: Chloroform: Methanol: Water 65: 25:4

Detection: Saturated Solution of $K_2Cr_2O_7$ in 90% H_2SO_4

FIGURE IV

TLC OF THE COMMERCIAL AND DEFATTED LECITHINS

TABLE IX

COMPOSITION OF LECITHIN AND CEPHALIN FRACTIONS OBTAINED BY SEPARATION OF RAPESEED PHOSPHATIDES IN A BENZINE/96% ETHANOL SYSTEM

Lecithin Fraction	%
Unidentified Compounds	26.9
Phosphatidyl Choline	-
Phosphatidyl Inositol	48.8
Phosphatidyl Ethanolamine	-
Sterol Glycosides	8.1
Neutral Oil, Pigments	1.0

Cephalin Fraction	%
Unidentified Compounds	26.9
Phosphatidyl Inositol	-
Lysophosphatidyl Ethanolamine	47.7
Phosphatidyl Choline(x)	-
Phosphatidyl Ethanolamine	9.4
Sterol Glycosides	7.4
Neutral Oil, Pigments	3.0

(x) Trace Quantities.

Thus, the organoleptic properties of the cephalin fraction were better than those of the lecithin fraction. The problem of removing bitter substances and those responsible for the burned flavour is not a simple one. Attempts to completely remove the odor by vacuum treatment did not give satisfactory results. For that reason selective tests should be undertaken to remove those substances which are responsible for the bad taste and smell of the lecithin fraction.

3. FRACTIONATION OF OIL-FREE PHOSPHATIDES WITH ETHANOL OR METHANOL

a) Application of Ethanol(7)

This method requires a solid-liquid system. That is why suitable equipment was necessary for the dispersion of phosphatides in ethanol. That requirement was fulfilled by the application of a homogenizer.

A diagram showing the procedure is given in Figure V.

The composition of the lecithin and cephalin fractions obtained is given in Table X.

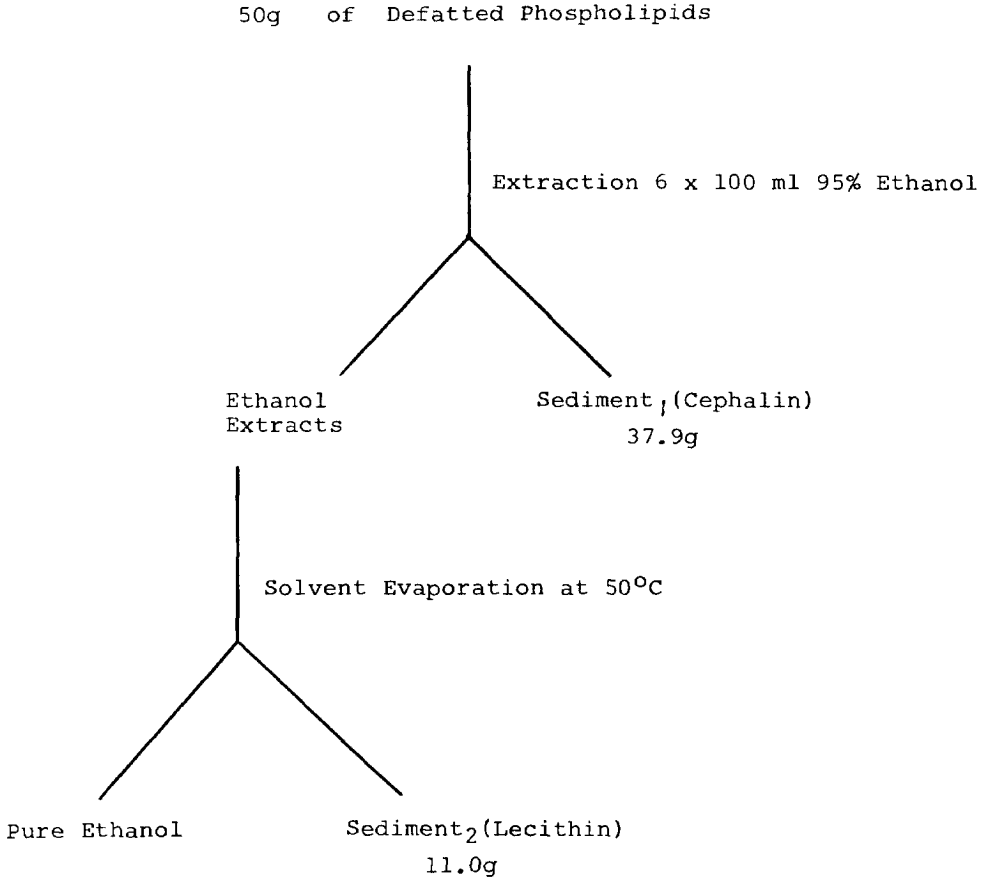


FIGURE V

ETHANOL FRACTIONATION OF PHOSPHOLIPIDS
AFTER C.R. SCHOLFIELD

TABLE X

COMPOSITION OF LECITHIN AND CEPHALIN FRACTIONS OBTAINED BY THE EXTRACTION OF RAPESEED PHOSPHOLIPIDS WITH 95% ETHANOL AT ROOM TERMPERATURE

Lecithin Fraction	%
Unidentified Compounds	27.0
Unidentified Compounds	8.5
Phosphatidyl Choline	-
Phosphatidyl Inositol(x)	48.8
Phosphatidyl Ethanolamine	3.4
Sterol Glycosides	8.1
Neutral Oil, Pigments	3.9
Cephalin Fraction	**%**
Unidentified Compounds	27.0
Phosphatidyl Inositol	-
Lysophosphatidyl Ethanolamine	47.7
Phosphatidyl Choline	9.4
Sterol Glycosides	7.4
Neutral Oil, Pigments	8.3

(x) Trace Quantities.

The cephalin fraction had a characteristic neutral flavor; a mild smell; a rather solid, slightly ductile consistency and a uniform brown colour. The lecithin fraction had a rather sharp taste with a bitter flavour, a "bland" odor, a greasy consistency and a light brown colour.

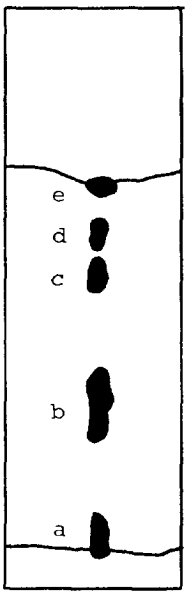
The thin-layer chromatograms of the cephalin and lecithin fractions are given in Figures VI and VII.

b) APPLICATION OF METHANOL

After subjecting a 50-gram sample of defatted phosphatides to an extraction with 98 percent methanol, the following fractions were obtained:

Cephalin 34.0 g
Lecithin 16.0 g

The characteristics of both these fractions were similar to those obtained in the case of the 95% ethanol extraction.



a Unidentified Compounds

b Lysophosphatidyl Ethanolamine,
Phosphatidyl Choline,
Phosphatidyl Inositides

c Phosphatidyl Ethanolamine

d Sterol Glycosides

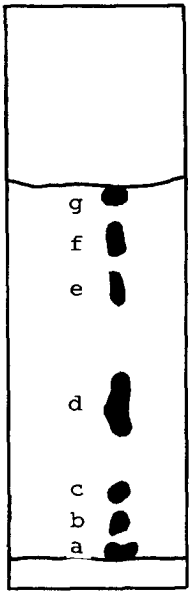
e Neutral Oil

Eluent:Chloroform:Methanol:Water 65:25:4

Detection:Saturated Solution of $K_2Cr_2O_7$ in 90% H_2SO_4

FIGURE VI

TLC OF THE CEPHALIN FRACTION FROM ETHANOL FRACTIONATION OF RAPESEED OIL PHOSPHATIDES



a,b,c Unidentified Compounds

d Phosphatidyl Choline, Phosphatidyl Inositides (traces)

e Phosphatidyl Ethanolamine

f Sterol Glycosides

g Neutral Oil

Eluent:Chloroform:Methanol:Water 65:25:4

Detection:Saturated Solution of $K_2Cr_2O_7$ in 90% H_2SO_4

FIGURE VII

TLC OF THE LECITHIN FRACTION FROM RAPESEED OIL PHOSPHATIDES ETHANOL FRACTIONATION

B. SEPARATION OF COMMERCIAL LECITHIN BY ETHANOL

Upon extracting 50 g of lecithin the following fractions were obtained:

Cephalin	38.1 g
Lecithin	10.5 g

In this case the cephalin fraction also contained neutral oil. The organoleptic characteristics of both fractions were similar to those of the previously mentioned fractions. The lecithin fraction, however, had a characteristic strong odor and a burned flavour. The reason for this was the fact that the substances responsible for the undesirable properties had not been removed even partially during the defatting of the commercial lecithin.

With regard to equipment, the extraction of commercial lecithin with ethanol is a simple process. The price of the cephalin and lecithin fractions should not be higher than that of lecithin defatted by acetone. Provided appropriate safety measures are observed, it is possible to use methanol instead of ethanol.

III. REPLACEMENT OF THE OIL NATURALLY ASSOCIATED WITH LECITHIN

Commercial lecithin contains a considerable quantity of crude oil in addition to the phosphatides and other hydratable compounds. This oil has a dark colour in accordance with an increased free fatty acid level. This is due to the presence of natural pigments belonging to the carotenoid and chlorophyll classes as well as to the presence of melanophosphatides.

Melanophosphatides are mixtures of condensation products of amino-alcohols and amino-acid phosphatides with reducing sugars as well as with oxymethylfurfurol and furfurol which appear as a result of the decomposition of pentoses and hexoses.

Thus, the intensity of the colour of the phosphatide concentrates depends first of all upon the colour of the oil from which the concentrates were obtained. Furthermore, the quality of commercial lecithin is greatly influenced by such technological factors as the temperature and length of heating of the seeds before pressing, by the distillation temperature of the miscella and by the intensity of the heat when drying the concentrate.

It is possible that extensive oxidation occurs during the industrial production of oil and lecithin, and this oxidation has an adverse effect on the quality of the product.

The oil occurring in crude lecithin is an integral part of the product which cannot be separated by mechanical methods. When adding refined oil to commercial lecithin and mixing it thoroughly, it replaces the original crude oil associated with the commercial lecithin.

The determination of the extent of such an exchange of oil in relation to the number of consecutive dilutions was the subject of our introductory investigations.

A. DISSOLVING COMMERCIAL LECITHIN IN REFINED SUNFLOWERSEED OIL

Tests were carried out, using commercial lecithin and refined sunflowerseed oil. This type of oil was used in order to determine the possibility of replacing crude oil by refined oil on the basis of the changes in the erucic acid content of the phosphatide fractions.

Commercial lecithin was dissolved in refined sunflowerseed oil at a temperature of 40°C. When reaching room temperature, the two layers were separated in a centrifuge at a speed of 6500 rpm. The phosphatide fraction obtained from the sediment was designated as phosph.fr.I, and the oil fraction as oil fr.I.

The phosphatide fraction I was then dissolved in sunflowerseed oil. After cooling and centrifuging, phosph.fr.II and oil fr.II were obtained. After carrying out consecutive dilutions of the phosphatide fractions in sunflowerseed oil, III, IV, V, VI, VII, phosphatide fractions and III, IV, V, VI, and VII oil fractions were obtained.

The commercial lecithin as well as the phosphatide fractions were subjected to saponification in a 1 n KOH-alcoholic solution. Methyl esters were then prepared from the extracted fatty acids. The methyl esters were subjected to GLC analysis. The results are given in Table XI.

The content of erucic acid in the constitutional oil of the phosphatide fractions and in the commercial lecithin was calculated on the basis of the percentage content of oil in those fractions and of the content of erucic acid determined in defatted phosphatides. The latter value amounts to 5%.

The data presented indicate that as early as following the first elution of the crude oil from the commercial lecithin by means of refined oil, the content of erucic acid diminishes considerably, namely from 43.3% to 14.1% in fr.I, i.e. 1/3 of the original quantity. It remains roughly at the same level in the consecutive fractions.

TABLE XI

CONTENT OF ERUCIC ACID IN PHOSPHATIDE FRACTIONS

Phosphatide Fraction No.	Erucic Acid in Oil Containing Phosphatides	Erucic Acid in Oil Contained in Phospholipid Fractions
0	16.5	43.3
I	9.4	14.1
II	8.4	12.3
III	7.5	11.3
IV	7.4	10.0
V	8.7	14.5
VI	8.2	13.6
VII	10.0	17.6

Thus, the mechanism of the replacement of the crude oil in commercial lecithin by refined oil seems to be as follows. When both components become thoroughly mixed, the original composition of the commercial lecithin is disturbed. The crude oil becomes thoroughly mixed with the refined oil. This follows from the composition of the oil fraction of the separated phosphatides. It corresponds to the ratio of both those oils, i.e. constitutional oil to oil used for the exchange.

Thus by consecutive elutions it is possible to approach a zero level of crude oil in the constitutional oil. It is most probable that similar results could be obtained, when a large excess of refined oil is used for a single operation.

B. DISSOLVING OF COMMERCIAL LECITHIN IN REFINED RAPESEED OIL

On the basis of the model tests explaining the replacement mechanism of the crude oil by refined oil, experiments were carried out to determine the refining parameters of commercial lecithin by means of refined rapeseed oil.

The process was carried out by eluting the crude oil with refined oil. It was being carried out at the following temperatures: 40°C, 60°C and 80°C. The following correlation of lecithin to oil was chosen: 1:1, 1:2, 1:4, 1:6, 1:8, 1:10, 1:15.

A sample of the lecithin was dissolved in oil at an appropriate temperature with intensive mixing. The mixture was then cooled down and the sediment separated from the oil fraction in a centrifuge at 6500 rpm. The resulting sediment was called "phosphatide fraction" and the liquid phase "oil fraction".

1) EXAMINATION OF THE PHOSPHATIDE FRACTIONS

(a) The oil content is within the range of 30 to 50%.

(b) In order to find out whether phosphatides undergo oxidation at increased temperatures, peroxide values were determined for fractions No. 3 and 5 at 40°C, 60°C and 80°C. The results correspond with the peroxide content of commercial lecithin.

(c) Nitrogen was determined by the Kjeldahl method. No changes have been found to take place in the nitrogen content in the particular phosphatide fractions at a given temperature or at different temperatures. The nitrogen content is within the range of 1.2 to 1.9%.

(d) The above conclusion also applies to the phosphorus content.

(e) From the relationship determined for the P/N ratio it follows that it does not change systematically, nor in dependence on the quantity of oil used for elution, nor with the temperature. This means, that the share of the particular phosphatide groups does not undergo any changes in the phosphatide fractions. The P/N ratio as determined in the phosphatide fractions corresponds with the P/N relation in commercial lecithin.

(f) The content of choline was determined by spectrophotometry(8). Its content in commercial lecithin and in all the phosphatide fractions was found to remain within the same limits. This is additional proof that the composition of the phosphatide fractions does not undergo any changes.

(g) The particular phosphatide groups were determined by TLC(9,10). Silica gel was used as the adsorbent. The chromatogram was developed by a mixture of chloroform, methanol and water. In order to identify the particular groups, a mixture of the following dyes was used: eosine, methylene blue and crystal violet. The spots were developed by a mixture of anisic aldehyde, glacial acetic acid

and conc. sulfuric acid. After a qualitative identification of the groups occurring in phosphatides, densitometry was applied for determining the percentage share of the particular compounds in those fractions. Defatted phosphatide fractions were subjected to these tests.

We have compared the results obtained for the particular groups of the following compounds: Unidentified substances, phosphatidyl choline, cephalin and sterol glycosides at various ratios of refined oil to commercial lecithin for the same as well as for different temperatures, have shown that the composition of the phosphatide fractions does not change. Thus, neither the quantity of oil nor the temperature have an influence on the selective elution of any of the groups of compounds that appear in commercial lecithin. These experiments have confirmed the conclusions based on data obtained from the calculation of the P/N ratio as well as of the choline content in phosphatide fractions.

(h) The colour of each of the phosphatide fractions is dependent mainly upon the colour of the crude oil which is present in the fraction. Oils obtained from each of the phosphatide fractions were investigated. It was found that the best bleaching effect was achieved by subjecting commercial lecithin to elution with refined oil in excess of ten times. At temperatures of 60°C and 80°C the greatest difference in colour took place when refining was carried out with oil at a ratio of 1:4. A further increase in the quantity of oil used does not result in a better bleaching effect so that the use of a 1:8 or 1:10 ratio instead of 1:4 is not justified.

The total bleaching effect, however, is small, because of the fact that melanophosphatides, which give the lecithin the dark brown colour, elute much more poorley than carotenoids.

2) EXAMINATION OF OIL FRACTIONS

(a) The peroxide content determined in oil fractions No. 3 and 5 showed the same level at three different temperatures (40, 60 and 80°C).

(b) The amount of hydratable compounds in the oil fractions was determined. It was found that the concentration of these substances remained the same regardless of the ratio of refined oil to commercial lecithin. This has also been confirmed at temperatures of 40, 60 and 80°C. Thus, it may be concluded that the total quantity of phosphatides which dissolves in oil fractions at a given temperature increases with the amount of oil used for their dissolution. At 60°C the degree of solution is 3 to 5 times higher than at 40°C. At 80°C, however, the degree of solution is only a little higher than at 60°C.

The organoleptic properties of the resulting phosphatide fractions showed a slight improvement. In conclusion it should be stressed that the best relation between commercial lecithin and refined oil is 1:8 and the best temperature for solution is 40°C.

IV. CONCLUSIONS

1) The use of solvents according to the Pardun or Rewald methods does not remove the unfavourable taste completely from rapeseed lecithin of inferior quality.

2) The separation into fractions makes it possible to obtain specifically acting emulsifiers, capable of stabilizing inversed types of emulsion.

3) As far as organoleptic properties are concerned, the lecithin fraction is worse than the cephalin fraction.

4) The replacement of the crude rapeseed oil by refined rapeseed oil improves the taste and smell. The colour, however, becomes only slightly better because it is caused by melanophosphatides, and those are only sparingly soluble in oils.

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SESSION IV

THE ORGANIZATION OF THE SWEDISH RAPESEED INDUSTRY

CHAIRMAN: Mr. A.M. Runciman,
President,
Rapeseed Association of Canada.