Kinetics of Oil Extraction from Corn Germ

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This paper describes the effect of temperature and moisture content on the kinetics of oil extraction from corn germ flakes prepared by a dry degermination process. The experiments were carried out according to the factorial design 3^2. The moisture content in the extracted material was varied in the range 8%–12%, whereas the extraction temperature varied in the range 52.5°C–57.5°C. From the method of response surfaces, a functional dependence was established between the extraction rate, the moisture content in the material and the temperature of extraction. On the basis of this dependence, it was concluded that moisture content had a crucial effect on the rate of oil extraction. A decrease in moisture from 12% to 8% yielded a doubling of the extraction rate. On the other hand, temperature variations in the given range had no practical effect on the course of the extraction. Kinetics of oil extraction was determined according to the method developed in the Leningrad Institute of Oils and Fats (VNIIZh-method), modified to the extent described in the paper.

KEY WORDS: Corn germ, dry degermination process, kinetics of oil extraction, moisture content of flakes, oil extraction conditions, temperature of extraction.

Kinetics of oil extraction from oilseeds and by-products from corn-using industries (corn germ) is dependent on a number of factors (1,2). They include the composition and morphology of the raw material, structural and mechanical properties of the flakes after their hydrothermal treatment and milling before preparation of the material for extraction (3–6). During the extraction itself, essential conditions are the temperature and duration of extraction, as well as the polarity of the solvent used for extraction (7,8).

The composition of corn germ obtained by dry milling can vary widely. According to Leibovitz and Ruckenstein (3) corn germ contains from 10% to 24% oil. Our previous investigations have shown that the quality of corn germ may vary even within a degermination plant (4). The most probable reason for this is inappropriate treatment of maize before degermination. The germ with a lower content of oil contains more endosperm, which, if the moisture content during milling is low, causes the germ to break easily and form substantial amounts of fines. A higher content of fines in the material worsens the drainage properties of the extraction bed. For this reason, Leibovitz and Ruckenstein (3) proposed that material with higher fines content should be pelletized before its extraction. The material thus obtained possesses improved filtration properties. Besides, the authors proposed to use the TOM extractor (H.L.S. Industrial Company, Petah Tikva, Israel) in which the material is turned over at its halfway point inside the extractor with the aim of destroying the impermeable layer formed by the fines on top of the material bed.

The kinetics of oil extraction from oilseed flakes is usually determined on a laboratory apparatus (5,7–12). On the basis of our experience we have concluded that the most complete method used for this purpose is the one developed in the Leningrad Institute of Oils and Fats (VNIIZh-method) (9). One of the objectives of the present work is the modification of the VNIIZh method for the determination of oil extraction kinetics, with the aim to make it simpler and more rapid.

MATERIALS AND METHODS

Materials. The experiments were carried out on corn germ obtained by dry milling on Beall's degerminator (MIAG, Braunsweig, Germany). The oil content was 15.0%, calculated on dry matter content. Soybean flakes, serving as reference material, were obtained by standard plant procedure. The oil content was 23.3%. For extraction, commercial hexane was used.

Gravimetric method (VNIIZh method). Kinetics of oil extraction was determined with the aid of the laboratory tube extractor presented in Figure 1.

After adjusting to the appropriate temperature with a thermostat, the extractor was filled with 25 g of material prepared for the extraction. By opening valves 14 and 7, the solvent was allowed to pass through the material, taking care that the level in flask 9 was constant. The flow rate of miscella (15 cm^3/min) was regulated with valve 7. The total extraction time was 60 min, and miscella samples were collected in previously weighed flasks after 5, 10, 20, 30, 40, 50 and 60 min of extraction.

The amount of oil extracted in a time interval was determined gravimetrically by measuring the mass of the residue in the flask after removal of the solvent by distillation.

The content of oil remaining in the material after a given time of extraction, calculated on absolutely dry matter, was obtained from the relation (Equation 1).

$$M_n = \frac{M_c\,[P\,(100-V) - 100\sum_{1}^{7} m] + 10000\sum_{n+1}^{7} m}{P\,(100-V) - 100\sum_{1}^{n} m} \quad [1]$$

where M_n = content of oil in the material after the nth time interval from the beginning of the extraction (n = 1,2,...6), % calculated on absolutely dry matter; M_c = content of oil in the meal after 7th time interval (*i.e.*, 60 min extraction), % calculated on absolutely dry matter; P = amount of material used for extraction, g; V = moisture content in the starting material, %; $\sum_{1}^{7} m$ = total amount of oil from all 7 flasks, g; $\sum_{n+1}^{7} m$ = total amount of oil from the (n + 1)th to the 7th flask (n = 1,2,...6), g; $\sum_{1}^{n}$ = total amount of oil extracted to the nth time interval (n = 1,2,..6), g.

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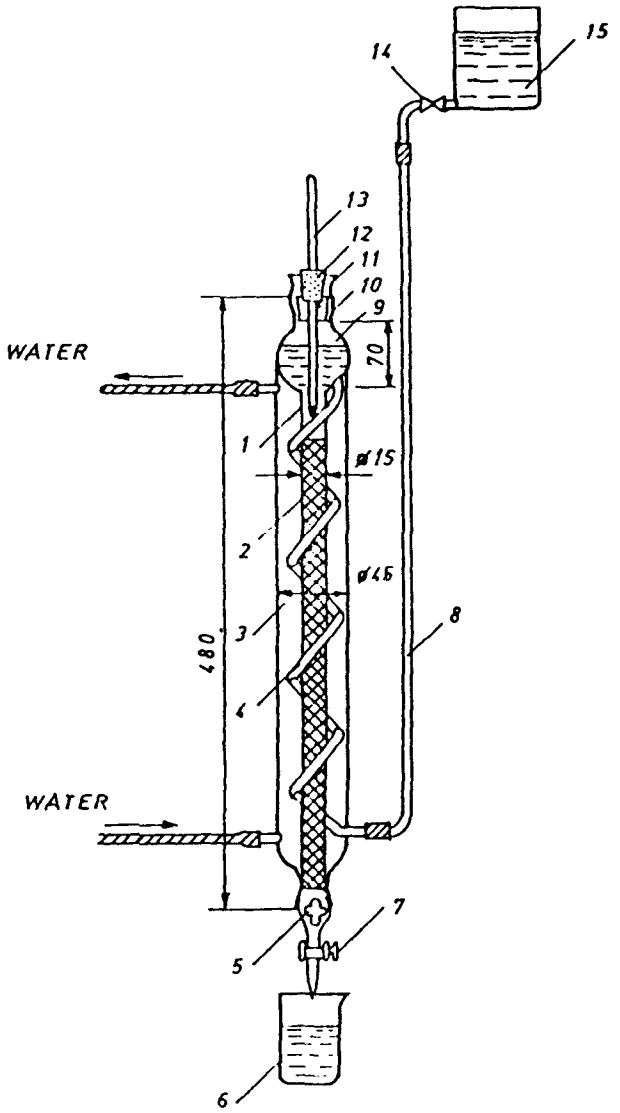

FIG. 1. Laboratory tube extractor for determining kinetics of oil extraction: 1 = extraction tube, 2 = raw material, 3 = heating jacket, 4 = spiral solvent preheater, 6 = miscella receiver, 7,14 = valves, 8 = solvent feed tube, 9 = bulb, 10 = safety opening, 12 = stopper, 13 = thermometer, 15 = solvent storage tank.

Modification of the VNIIZh method. The extraction procedure employed in the present work was almost identical to that given in the original VNIIZh method. The only modification introduced was concerned with the way of determining the amount of oil extracted during a particular time interval. The mass of the extracted oil was calculated on the basis of the volume and concentration of miscella for the given time interval from the equation (Equation 2):

$$Q_i = V_i c_i \qquad (i = 1,2,...7) \qquad [2]$$

where Q_i = mass of extracted oil, g; V_i = volume of miscella, cm^3; c_i = concentration of oil in miscella for a given time interval of extraction, g/cm^3.

The concentration of oil in miscella was calculated by measuring the miscella density and using a calibration plot obtained for a series of standard solutions of corn oil and soybean oil in commercial hexane. The concentrations of the standard solutions were chosen to cover the whole range of miscella concentrations obtained in the laboratory oil extraction. On the basis of experimental data for density and concentration of oil in the solvent, the least squares method was used to determine the slope and intercept of the straight lines representing best the dependence of the oil concentration in miscella on its density (Fig. 2 and 3).

Before measuring their volume and density, the miscella samples were dried over anhydrous sodium sulfate and thermostated at 25°C. The density measurements were carried out on an automatic densitometer, type DMA 46, manufactured by Anton Paar KG., Graz, Austria.

Experimental plan and preparation of samples. To study the effects of moisture content in the material and temperature of extraction on the kinetics of oil extraction from corn germ flakes, a series of experiments was carried out according to the factorial design 3^2 (2 factors at 3 levels) after Box and Behnke (13), as shown in Table 1.

The starting samples of corn germ were made to have moisture contents of 8%, 10% and 12% by using a laboratory sprayer. To obtain a uniform distribution of moisture, the material was conditioned at room temperature in a closed vessel for 4 days and shaken occasionally. After that, the germ samples were ground on a laboratory mill with slightly corrugated rolls (MIAG mill for determining quality of wheat – ZELLENY-test). The average thickness of the flakes was about 0.30 mm.

The soybean flakes used as reference material were taken directly from the flaking mills and stored in sealed vessels up to the moment of their investigation. The average thickness of the soybean flakes was about 0.27 mm.

All extraction experiments were carried out in duplicate and averages are presented throughout the tables.

Statistical treatment of the results. In determining the oil extraction kinetics, we started from a practical assumption that extraction to the range of about 5.0–0.5% residual oil (on the basis of dry, solvent-free meal) is so slow that it actually controls the overall extraction rate and extractor design (1). The extraction kinetics for this range is usually presented in semi-logarithmic or logarithmic coordinates (1,9).

Our previous investigations (14) showed that the part of the curve representing the extraction kinetics for the oil content in the material below 5% can be described by the following exponential equation (Equation 3):

$$C = b\, e^{a\tau} \qquad [3]$$

where C = content of oil in the material, % on absolutely dry matter; a and b = constants; τ = time of extraction, min.

From regression analysis, the coefficients can be calculated, while the semi-log plot gives a straight line whose slope represents the rate of extraction. Besides, this plot offers the possibility to interpolate or extrapolate the extraction time for a given oil content in the meal. In our experiments we adopted Becker's criterion (15), *i.e.*, for each sample we calculated the extraction time needed to decrease the oil content in the meal to 0.5%.

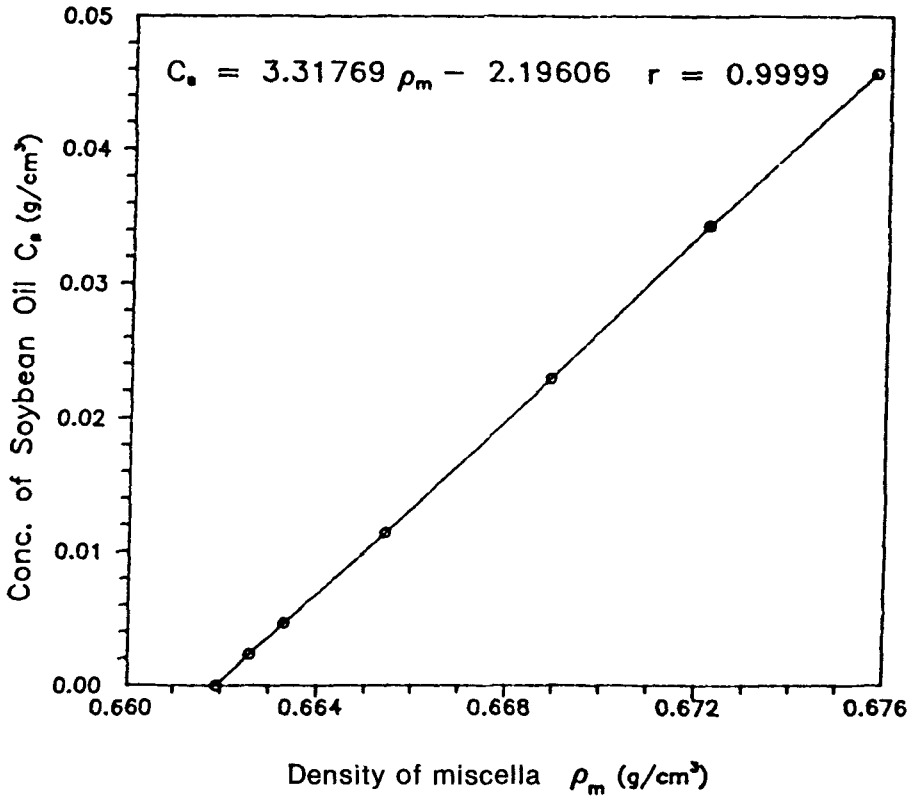

FIG. 2. Dependence of soybean oil concentration on density of miscella.

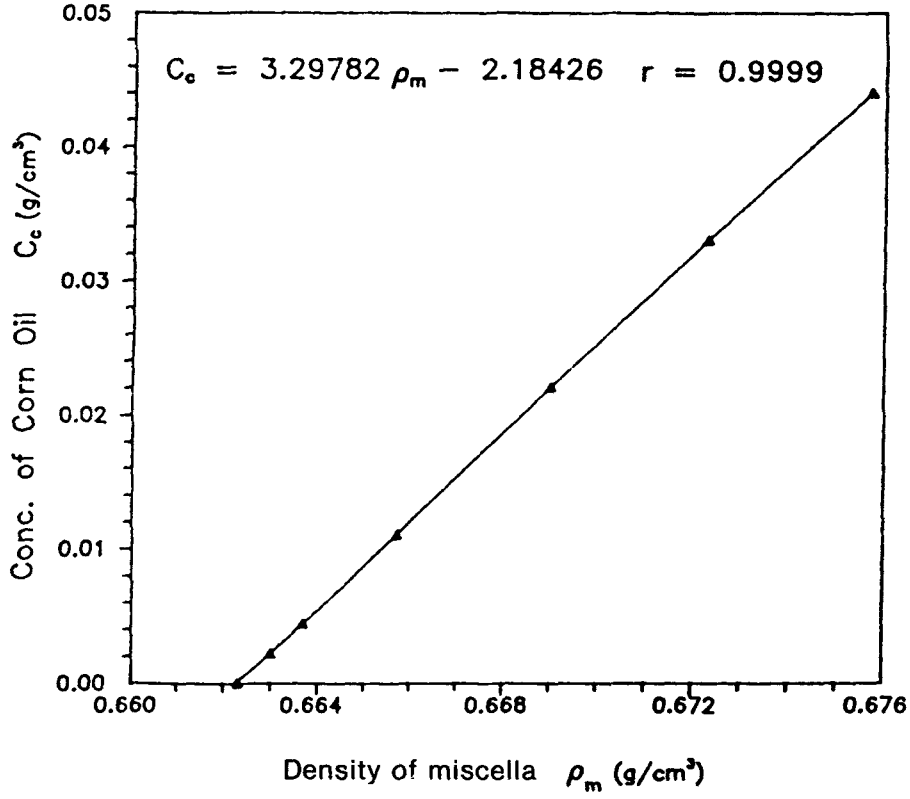

FIG. 3. Dependence of corn germ oil concentration on density of miscella.

TABLE 1

Experimental Conditions Selected for 3^2 Factorial Design[a]

Independent variables (factor: T, V)	Coded	Level +1	Level 0	Level −1	Interval of variation	Dimension
Temperature of extraction	X1	57.5	55.0	52.5	2.5	°C
Corn germ flakes moisture	X2	12	10	8	2	%

[a]Response functions (Y): Extraction rate = calculated time needed to decrease oil content in the material to 0.5% (min).

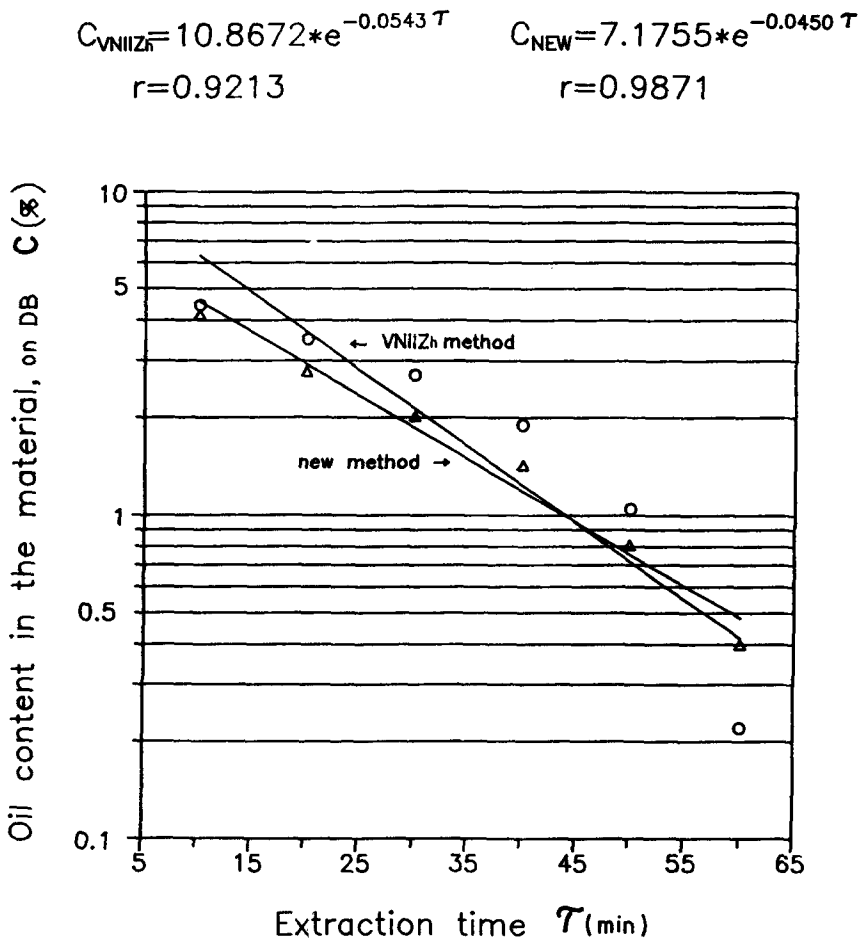

FIG. 4. Comparative results of determining the rate of oil extraction from soybean flakes by the classical VNIIZh method and the modified method.

For treatment of experimental results and calculation of the functional dependence describing the part of the extraction curve for the oil content in the material below 5%, use was made of an original program developed by Karlovic and Füstös (16). This program, on the basis of the obtained exponential equation, calculates the extraction time (τ) needed to lower the oil content in the meal to 0.5%.

The results of the measurements according to the factorial design were subjected to the RSM-2 program developed by Walker and Parkhurst (17), while the drawing of the response surfaces was carried out with the aid of the commercial program Statgraphics v.2.1 (18).

RESULTS AND DISCUSSION

Comparison of the VNIIZh method and modified method. In order to verify that the modified method, *i.e.*, the measurement of miscella density, can give satisfactorily reliable results, a series of experiments was carried out

TABLE 2

Variation of Oil Extraction Rate from Corn Germ, with Temperature (T) and Moisture Content (V)

Experiment number	Coded factors		Natural scale factors		Rate of Extraction[a] (min)
	X1	X2	T(C)	V(%)	Y
1	−1	−1	52.5	8	98
2	−1	0	52.5	10	140
3	−1	+1	52.5	12	229
4	0	−1	55.0	8	96
5	0	0	55.0	10	148
6	0	+1	55.0	12	220
7	+1	−1	57.5	8	95
8	+1	0	57.5	10	158
9	+1	+1	57.5	12	213

[a]Calculated time needed to decrease oil content in the material to 0.5%.

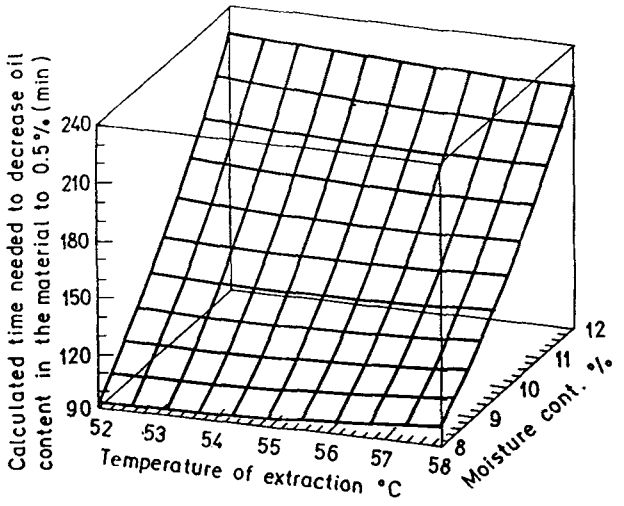

FIG. 5. 3D plot of the dependence of oil extraction rate on temperature and moisture content of corn germ.

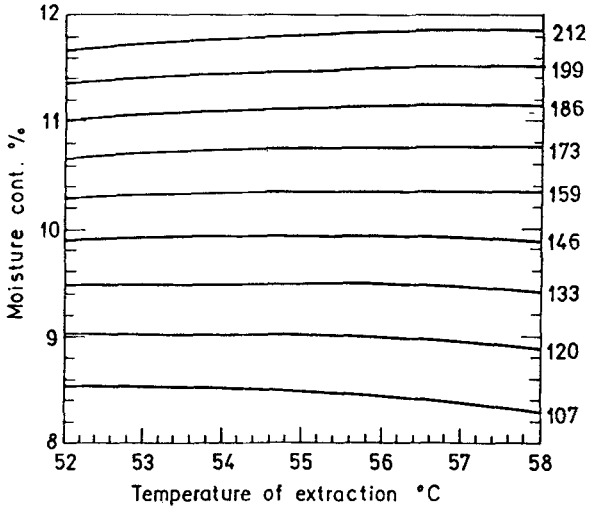

FIG. 6. Contour lines obtained from 3D plot in Figure 5.

with soybean flakes samples by the classical VNIIZh method and our modified method. The results obtained in these parallel experiments are presented in Figure 4.

The material balances made for the methods showed good agreement. Namely, from 25.0000 g soybean flakes after 60 min of extraction we obtained 4.9356 g and 4.9358 g of oil by the VNIIZh method and modified method, respectively. From these results, as well as from the approximately equal slopes of the straight lines obtained for the measurements in the range when the oil content was lowered below 5%, it was concluded that the modified method could be used for practical work. In this way, the kinetics of oil extraction could be determined in a much shorter time.

Effect of temperature and moisture content in the corn germ on the rate of extraction. Table 2 presents the results of determining the rate of extraction of oil from corn germ as a function of temperature and moisture content in the material.

By statistical treatment of the data, a functional dependence was established between the rate of extraction and temperature (T) and moisture content (V). A second-order polynom was obtained of the form (Equation 4):

$$Y = 164.14 - 9.40\ T + 17.71\ V - 0.65\ T\ V + 0.14\ T^2 + 2.46\ V^2 \quad [4]$$

where the coefficient of determination $r^2 = 0.9894$, the coefficient of multiple correlation = 0.9947, and the standard error of estimate = 9.17.

In Figures 5 and 6, this relation is presented graphically in the form of a three-dimensional diagram and a diagram with calculated contour lines respectively. The response surfaces thus obtained indicate that, in the range investigated, the temperature variation has no significant effect on the rate of oil extraction from corn germ. In contrast to this, the moisture content exhibits a substantial effect: an increase in the moisture content causes an exponential decrease of the extraction rate.

As can be seen, the moisture content in the extraction material is of crucial importance for the extraction process. A decrease of moisture content from 12.0% to 8.0% causes a doubling of the extraction rate. On the other

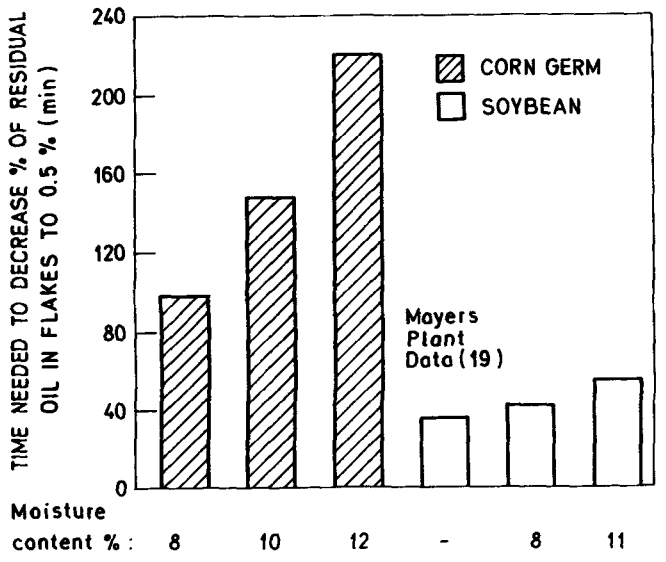

FIG. 7. Comparison of the rate of oil extraction from corn germ and soybean flakes with literature data.

hand, temperature variations in the limits ±2.5°C have no practical effect on the course of oil extraction from corn germ. These findings are in agreement with those of Minasyan *et al.* (6).

It is well known that oil contained in soybean flakes can be easily extracted. From the results presented by Mayers (19), it is possible to calculate that, in an industrial extractor, a decrease of oil content in soybean meal to 0.5% can be achieved in 36 min. These results agree well with the results of this study obtained for the extraction of the same material under laboratory conditions. In Figure 7 are presented the results of our comparative investigation of the rate of oil extraction from corn germ and soybean flakes, carried out at 55°C.

The results presented clearly show that for a temperature of 55°C and a moisture content of 8%, the extraction of oil from corn germ flakes is 2.3 times slower than from soybean flakes. For higher moisture contents, this ratio is even less favorable for corn germ flakes.

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