to obtain maximum cyclization. In contrast, ethylene enters into the reaction and adds to the conjugated fatty acids forming a cyclized C_{20} molecule (5). Increasing ethylene pressure increases cyclic acid yield to 84.6 and decreases polymer yield slightly.

The highest cyclic acid yield, 84.6 g per 100 g linolenic acid, was obtained with 6:1 solvent ratio, 100% excess catalyst concentration, 295C, and 500 psi ethylene pressure. The cyclic acid content of the monomer from this test was 95%.

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Effect of Seed Preparation on Efficiency and Oil Quality in Filtration Extraction of Rapeseed[1]

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Abstract

The application of filtration extraction to rapeseed is discussed with particular emphasis on the effect of seed preparation on the hydrogenation characteristics of the oil. It was found that cooking the crushed seed without the addition of water, and at temperatures not exceeding 220F, produced a satisfactory oil. Under these conditions extraction efficiency was good and the resulting meal showed no harmful effects in feeding trials with mice.

Introduction

THERE HAVE been a number of reports and papers (1–7) on filtration extraction of a variety of oil seeds. However, we believe that we have overcome some unique problems associated with the extraction of rapeseed, and this paper discusses how various changes in the handling and cooking of rapeseed have a marked effect on plant performance and oil quality.

Rapeseed has a high oil content, 38–46%, and tends to disintegrate into fines when placed in hexane. Therefore, it is generally considered necessary to prepress prior to solvent extraction. The ability of the filtration extraction unit to handle both high oil content and fines was the basis of a decision to convert from expeller processing to straight solvent extraction.

Experimental

Figure 1 is a schematic diagram of the filtration extraction unit as used in rapeseed processing. The cleaned seed is rolled and then cooked in a five-high stack cooker. The cooked flakes are rapidly cooled in an evaporative step called "crisping." The material is re-rolled and conveyed to the solvent extraction section of the plant. The re-rolled seed is fed continuously into the extractor and conveyed down its length as a slurry with miscella and slowly agitated to accomplish maximum extraction of the oil with minimum disintegration of the flakes. The slurry is laid down on the rotating horizontal screen filter. The concentrated miscella drains through the filter, leaving the marc on the pan ca. 2 in. thick. As the filter rotates, the cake is washed: first with concentrated miscella to remove fines prior to stripping; then with two washes of decreasing strength miscella; and finally with pure solvent. The miscella from the second wash is fed to the extractor to make up the slurry. The marc is continuously removed and discharged into a conveyor to the desolventizer.

In the initial plant start-up we were concerned with two main points: 1) attaining low residual lipids in the meal at the rated capacity of the plant, and 2) obtaining oil suitable for the edible trade and meal acceptable for animal feeds. The latter was of prime concern because of problems attributed to sulfur-containing compounds, viz., isothiocyanates and thiooxazolidones, present in rapeseed but not present in most other oil seeds. Specifically, these problems are extraction of a portion of the sulfur compounds with the oil, resulting in catalyst poisoning during

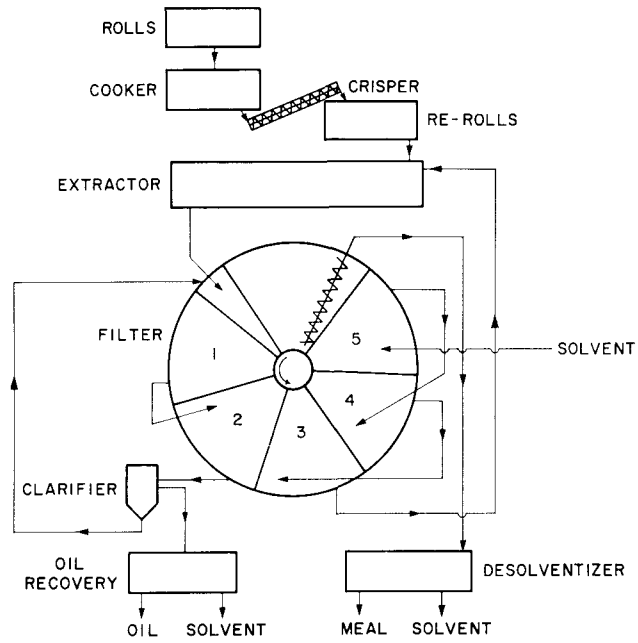

FIG. 1. Schematic diagram of filtration extraction unit as applied to rapeseed.

[1] Presented at AOCS meeting in Toronto, 1962. N.R.C. 7667.
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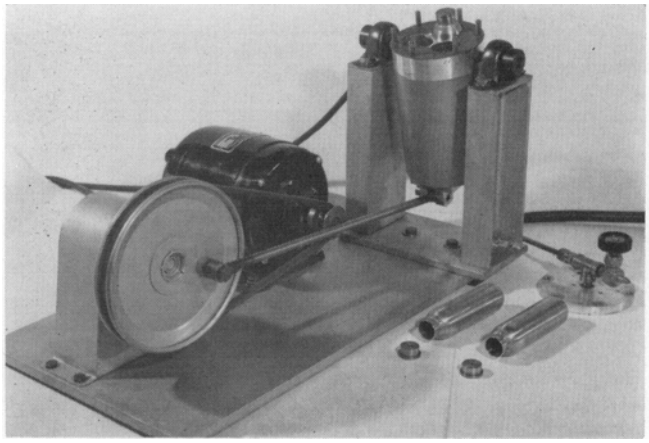


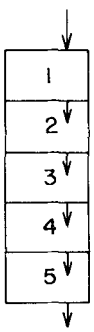
FIG. 2. Photograph of laboratory hydrogenator.

hydrogenation, and retention of some of the sulfur compounds in the meal in a form harmful to animals when a high level of the meal is fed.

Residual lipids and overall plant efficiency are readily assessed. Oil quality, with reference to hardening ability, could be assessed only by actual hydrogenation of the oil, and meal quality by feeding trials with animals. A shaking hydrogenator, similar to the Parr type, was constructed as shown in Figure 2 for use in a routine control test of hardening ability. A similar drive and shaking stand were utilized, but the glass hydrogenation tube was replaced by an electrically heated aluminium block. This block was drilled to take three stainless steel tubes so that three samples could be run simultaneously. Five ml of bleached refined oil and 0.1% commercial nickel catalyst were placed in each tube. The tubes were placed in the heated block, the cover attached, and the bomb swept first with nitrogen, then with hydrogen. Hydrogenation was carried out at 180C and 20 psig for 30 min. The drop in refractive index of the oil was used as a quality index for the oil. Oil samples for hydrogenation were refined and bleached in the laboratory. Refining was done with 3% of 14° Baumé NaOH at 65C for 15 min. The soaps were removed by centrifuging. Bleaching was done with 4% activated bleaching clay at 110C for 30 min.

Feeding trials of the meals obtained were carried out by J. M. Bell, University of Saskatchewan Animal Science Dept. He has been carrying on an active investigation of deleterious factors in rapeseed meals using mice as test animals (8,9,10).

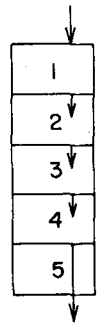
Initially we were guided by previously reported conditions for handling high oil content seeds by the filtration extraction process. This involved high moisture levels in the initial stages of cooking, followed by



	MOISTURE	TEMP °F	REFRACTIVE INDEX DROP x 10^4
1			
2	15.2	216	32
3	13.2	213	48
4	13.3	214	42
5	9.9	214	10
	7.4	219	3

RESIDUAL LIPIDS – 6.3%

FIG. 3. Original plant cooking conditions.



	MOISTURE	TEMP °F	REFRACTIVE INDEX DROP x 10^4
1			
2	9.5	100	
3	11.5	216	59
4	6.4	217	49
5			
	5.6	220	49

RESIDUAL LIPIDS – 7.2%

FIG. 4. Modified plant cooking conditions.

flashing off or crisping of the material prior to re-rolling and extraction. Under these conditions, residual lipids in the meal were high and the oil obtained would not hydrogenate satisfactorily. A number of variations in amounts of water added and cooking temperatures were tried. Some improvement was obtained with respect to residual lipids in the meal as a result of these changes, plus changes in the flaking and re-rolling procedures. Oil quality remained below standard with only minor variations.

Samples were then taken from the various kettles in the cooker, checked for moisture content and the oil extracted, refined, bleached, and hydrogenated. The results are shown in Figure 3. On the basis of hydrogenation of soybean oil under the conditions described above, a satisfactory figure for refractive index drop $\times 10^4$ for rapeseed oil would be in the range of 75–80. The results obtained are well below the acceptable value in all kettles, and drop rapidly in the latter stages of cooking.

In view of these results the length of cooking time was reduced by decreasing the depth of bed in each kettle, and later by eliminating kettle 5. Figure 4 shows the results of this modification. Although there was an improvement, it was not sufficient nor was it consistent in response.

To obtain a better understanding of the effect of various cooking factors, a small laboratory cooker was constructed as shown in Figure 5. It is a horizontal stainless steel cylinder, electrically heated and operated as a closed system to assure known moisture levels during cooking. Agitation was provided by a stainless steel paddle. The seed was rolled, the desired amount of water added, then well mixed in an open beaker. The moist flakes were placed in the preheated cylinder and cooked for a specified time. The cooked flakes were solvent extracted, the solvent distilled

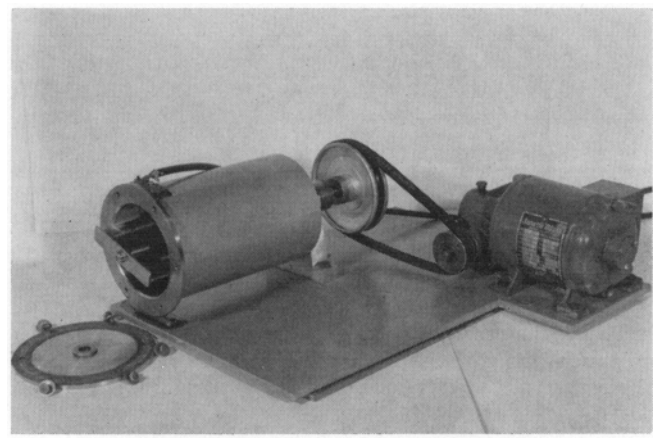


FIG. 5. Photograph of laboratory cooker.

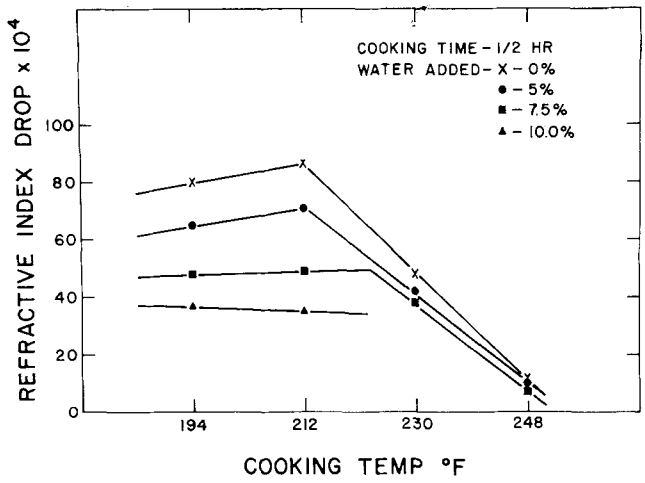


FIG. 6. Effect of moisture and temperature in laboratory cooker.

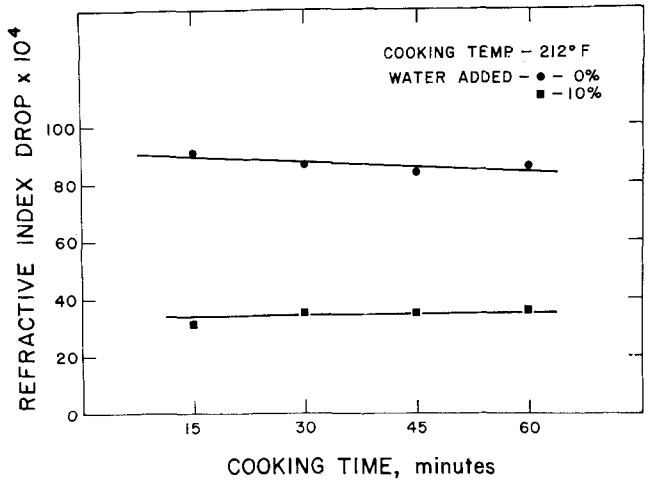


FIG. 7. Effect of time in laboratory cooker.

from the oil, and last traces of solvent removed by steam stripping under reduced pressure. The crude oil was refined, bleached, and hydrogenated as described above.

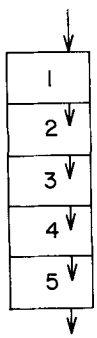
The effects of added moisture and temperature for a given cooking time are shown in Figure 6. Increasing moisture decreased the ease with which the oil could be hardened. Cooking temperatures exceeding 212F also caused a decrease. The time of cooking had very little effect, as shown in Figure 7.

After these results, addition of water to the plant stack cooker was discontinued and temperatures were maintained along a slow gradient to a maximum of 220F at the final kettle prior to crisping. Results from this type of operation are shown in Figure 8. Samples with a refractive index drop of greater than 90×10^{-4} were solid at the temperature of measurement, 55C, and are shown as 90+. The refractive index drop is of the same order as that obtained with the laboratory cooker and is in the acceptable range. Operating efficiency of the plant increased markedly, residual lipids in the meal decreasing to 1.5–1% level. This improved plant efficiency and oil quality has remained consistent over several months' operation.

Initial feed trials with meal from the plant, using mice as the test animals, showed no deleterious effects (11).

Discussion

This was an entirely empirical solution to the problem. In rapeseed the sulfur compounds are present in the form of thioglucosides along with an enzyme (myrosinase) capable of hydrolysing these glucosides. A possible explanation of the results obtained is that



	MOISTURE	TEMP °F	REFRACTIVE INDEX DROP x 10^4
2	9.2	114	90+
3	8.6	166	90+
4	8.8	186	90+
5	8.9	206	83
	6.8	220	81

RESIDUAL LIPIDS - 1.2%

FIG. 8. Final plant cooking conditions.

at the low moisture levels used, enzymatic activity is not appreciable before the enzymes are inactivated by heat. As a result, the sulfur compounds are not liberated in a form which is soluble in the miscella, but are left in the meal in a form which does not appear to be toxic to animals. This explanation would agree with results obtained for the analysis of sulfur compounds in the meal carried out by Wetter (12,13). Using the modified cooking procedure, all of the isothiocyanates and thiooxazolidones in the seed could be accounted for in the meal, whereas cooking with the addition of moisture resulted in a substantial drop in the amount of these sulfur components in the meal.

In recent papers by Mustakas et al. (6,7) on processing of mustard seed, the reverse of the present approach was taken in the cooking procedure. High levels of moisture were added and a conversion time introduced to give maximum enzyme activity. The volatile sulfur components released were then steam stripped from the crushed seed prior to extraction to give bland oil and meal. This process does not appear to be applicable to rapeseed since it contains sulfur compounds which are not steam-volatile after enzymic hydrolysis (thiooxazolidones), as well as the volatile components (isothiocyanates) similar to those present in mustard seed.

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