Extrusion pretreatment to enhance soybean oil extraction

by

Yongsoo Chung

A Thesis Submitted to the

Graduate Faculty in Partial Fulfillment of the

Requirements for the Degree of

MASTER OF SCIENCE

Major: Food Technology

Signatures have been redacted for privacy

.

Iowa State University Ames, Iowa

TABLE OF CONTENTS

	pag	;e
INTRODUCTION	1	•
LITERATURE REVIEW		•
Soybean Oil	2	2
Preparation of Soybeans for Solven	t Extraction 4	٢
ALCON Process	8	}
Refining	14	ł
Chemical refining	14	ł
Physical refining		3
Characteristics of Phosphatides .	21	L
Extrusion		5
MATERIALS AND METHODS		3
Soybean and Solvent Source		3
Soybeans		3
Solvent		3
Preparation of Samples		3
Conventional method of making	flakes	3
ALCON process		9
Extrusion		L
Determination of moisture con	tent	1
Extraction Characteristics	31	-
Extraction rate	31	-
	•••••••••••••••••••••••••••••••••••••••	-

Residual oil content	
Solvent hold-up	
Bulk density	
Percolation rate	
Phosphatide Content	
Degumming with Water	
Statistical Analysis	
RESULTS AND DISCUSSION	
Extraction Rate	
Phosphatide Content	
Extraction Capacity	
Bulk density and solvent hold-up	
Percolation rate	
Residual oil content	
SUMMARY AND CONCLUSION	
BIBLIOGRAPHY	
ACKNOWLEDGMENTS	

iii

LIST OF TABLES

Table 1.	Composition and price for edible vegetable oils	3
Table 2.	Differences between crude, degummed and refined oils from the ALCON and conventional processes	12
Table 3.	Typical compositions of crude and refined soybean oils	15
Table 4.	Phosphorus and trace metal contents of crude and physically refined soybean oils	21
Table 5.	Working conditions for making soybean flakes	38
Table 6.	Phosphatide contents of the oils obtained from extruded pellets, conventional flakes and simulated ALCON agglomerates before and after degumming	48
Table 7.	Phosphatide content of the oils extracted from the extruded pellets produced from flaked, cracked and whole soybeans	51
Table 8.	Comparisons of bulk density and solvent hold-up	52
Table 9.	Percolation rates at different bed depths of various soy preparations	53
Table 10.	Residual oil contents of extracted meals	55

Page

LIST OF FIGURES

Figure	1.	Comparison of preparation steps in the conventional and ALCON processes	10
Figure	2.	Comparison of the steps involved in alkali refining and physical refining	13
Figure	3.	Common phosphatides in soybean lecithin	23
Figure	4.	Stirring apparatus used in the simulation of the ALCON process	30
Figure	5.	Flow scheme of the laboratory extractor-simulator .	33
Figure	6.	Assembly for comparing percolation rates	35
Figure	7.	Extraction characteristics of the extruded pellets, conventional flakes and ALCON agglomerates	40
Figure	8.	Percentage unextracted oil in the extracted meal samples at each stage of extraction	41
Figure	9.	Extraction curves for extruded pellets, conventional flakes and simulated ALCON agglomerates	43
Figure	10.	Extraction characteristics of the extruded pellets obtained from flaked, cracked and whole soybeans	46
Figure	11.	Phosphatide contents in crude soybean oils extracted from soybeans subjected to different pretreatments .	47
Figure	12.	Percolation rate of solvent for extraction of soybean oil	54

INTRODUCTION

The extraction of oil from soybeans is a major industry in the United States producing large amounts of edible oil. Many methods have attempted to obtain greater extraction yields and to produce better quality oils. Physical refining is favored over caustic refining because the later entails high losses of neutral oil, involves numerous complex steps and requires disposal of caustic refining wastes. The phosphatide content is one critical characteristic of a crude soybean oil which limits the application of physical refining. By appropriate pretreatment of soy flakes prior to extraction, it is possible to produce degummed oils with very low phosphatide contents which should be suitable for physical refining. The ALCON process, which is now practiced in Europe, is an example of one commercial alternative pretreatment process producing oil suitable for physical refining.

In this study, extrusion pretreatment of soybeans was explored as a means to obtain crude soybean oil suitable for physical refining. Industrial claims indicate that this treatment also increases the solvent extraction rate and, consequently, extractor capacity. Comparisons were made among extruded soybeans, flakes prepared by conventional practice and agglomerates obtained from simulation of the ALCON process. Phosphatide contents, extraction rates and extraction efficiencies were determined for each process.

LITERATURE REVIEW

Soybean Oil

Soybeans have played an important role in the diet of oriental civilizations for many centuries. They were first introduced into the United States in 1804 and were grown predominantly in the southeastern parts of the country (Burlison, 1936). Extensive soybean plantings in the United States began in the mid-1920s, largely for animal feed and silage. Presently, about 5% of the crop is retained for seed and, of the remainder, about two thirds is used for oil extraction and one third for export.

The soybeans are usually harvested with 15% or less moisture and are dried, if stored for long periods, to 12% or less for storage. The bean contains 35-45% protein (dry basis), 18-25% fat, 25-30% carbohydrate, 5% fiber and 5% ash. The amounts of these constituents vary with climate, variety and other factors.

Soybean oil is the major edible oil in the United States. According to the American Soybean Association (Serrato, 1981), soybean oil is used in the United States for 80% of the salad oils, 78% of the margarines, 75% of the cooking oils and 59% of the manufactured shortening. Soybean oil is less expensive than other vegetable oils, such as corn, sunflower and safflower oils, and it has many desirable characteristics of these so-called premium vegetable oils (Table 1). Soybean oil has a high

	Comp	osition, g/100	g oil	
Vegetable oil	Linoleic acid	Total poly- unsaturated	Total saturated	Crude oil price, cents per lb
Corn	57	58	13	30.7
Cottonseed	50	51	26	24.3
Oil palm	9	. 9	48	26.7
Peanuts	31	32	17	29.8
Rapeseed	22	34	7	39.0
Safflower	73	74	9	46.2
Soybean	51	58	15	23.8
Sunflower	64	64	10	34.5

Table 1. Composition and price for edible vegetable oils^a

^aBrignoli et al., 1976.

linoleic acid content and a low saturated fatty acid content making it desirable nutritionally.

According to Minshew (1977), soybean oil has a number of advantages and some disadvantages compared to other vegetable oils:

The advantages:

- (1) A high level of unsaturation is present.
- (2) The oil remains liquid over a relatively wide temperature range.
- (3) The oil can be hydrogenated under highly selective conditions for blending with semisolid or liquid oils.

- (4) The partially hydrogenated oil can be used as a pourable, semisolid oil because of a relatively low level of palmitic acid.
- (5) The oil can be processed readily to remove phosphatides, trace metals and soaps, thereby improving the oil's stability.
- (6) Naturally occurring antioxidants (tocopherols) are present.
- The disadvantages:
- The phosphatides, present in relatively large amounts (about 2%), must be removed by processing. However, the recovered gums are the source of commercial lecithin.
- (2) The oil contains 7-8% linolenic acid which is responsible for the flavor and odor defects in consumer products. However, partial and selective hydrogenation can be carried out easily to lower the linolenic acid content to below 3% which greatly improves the stability of the processed oil to reversion and autoxidation.

Preparation of Soybeans for Solvent Extraction

<u>Storage</u>

Soybeans often have to be stored for periods before they are used for processing. The moisture level is probably the most important parameter for the successful storage of soybeans. In general, 12% moisture is the maximum level consistent with long term storage (Markley, 1950). Soybeans containing 12% moisture have been stored for as long as three years without germination or substantive increases in free fatty acids. Soybeans stored with a moisture content greater than 13% may germinate which may generate sufficient heat to damage the protein and oil (Barger, 1981).

Seasonal migration of moisture within a storage bin is probably the greatest problem in stored beans. Soybeans stored at 12% average moisture in early fall may have pockets with moisture as great as 18-20% within a few months (Markley, 1950). Natural or forced air circulation systems may be used to control moisture accumulation and migration in stored soybeans, not only on the farm, but also at the industrial elevator.

Bean drying

For proper bean cracking and dehulling, a moisture content of 10-11% is required. Drying also loosens the cotyledon-to-hull adhesion and allows easier removal of the hull from the cotyledon. The dried beans are stored for about 10 to 30 days (the conditioning time), which allows bean moisture to equalize. Large moisture variations will cause operational variations in bean cracking, dehulling and flaking (Fawbush, 1977).

Bean cracking

The dried beans at a moisture content of 10% or lower are cracked between two corrugated rolls of a roller mill. This practice allows separation of the hulls from the cotyledons and breaks the cotyledons

into appropriately sized pieces, called meats, for the extraction operation. The degree of bean cracking can be from 50% halves and the remainder quarters, to all eighths. Coarse cracking will increase the power consumption of the flaking rolls and produce larger flakes. Fine cracking will decrease the power consumption of the flaking rolls and produce small flakes with more potential of generating dust. Good (1970) presented data showing that as the particle size of cracked beans going to flaking roll increased, oil extraction rates increased and oil residuals decreased at any given flake thickness. Cell distortion was explained on the basis of larger particles more distorted during flaking. The most desirable size of the cracked beans for optimum extraction varies with the type of extractor (Fawbush, 1977). According to Barger (1981), cracking soybeans into eighths yields the desirable screen profile: on 6-mesh, 10-15%; on 10-mesh, 60-70%; on 20-mesh, 5-15%; through 20-mesh, 0-3%.

Dehulling

In the dehulling step, loose hulls are separated from the cracked beans by aspiration. Dehulling increases yield since the hulls contain only about 0.6% of oil compared to 18-22% for the whole seed (Becker, 1966). The hulls, which make up about 7% of the entire seed, are high in fiber content and the separation of the hulls reduces the fiber content of the extracted meal and affects the nutritional value of soybean meal.

<u>Bean heating</u>

The dehulled bean particles are heated to increase pliability in flaking. The beans are normally heated to 65-71°C (150-160°F) for 30 min. The target moisture content produced by this conditioning operation varies from plant to plant, but in general it is between 9 and 11% (Markley, 1950). Becker (1966) mentions 10-11% being the desired range.

Bean flaking

The hot, cracked beans are next sent to a bank of flaking rolls stands. The capacity of the flaking rolls will depend on the size of the cracked bean particles, the temperature and moisture of the bean particles, and the flake thickness desired. The moisture level is critical for several reasons. If the moisture content of cracked beans is too low, the flakes that are produced tend to be fragile and create excessive amounts of fines. These fines cause difficulties during extraction, reducing the rate and efficiency by plugging the flake bed. On the other hand, if the moisture content is too high (above 12%), large flakes are produced, their higher moisture contents act as a barrier to solvent penetration and the rate of extraction again decreases. The moisture content is important also in relation to the temperature employed in flaking. Enzyme action can occur at a moisture level of 10% and at 65-71°C (150-160°F). Although the temperature of the flakes is relatively high, it is not high enough to destroy enzymes and prevent catabolysis. Particularly important in this regard are the lipid

degrading enzymes: lipases, which hydrolyze the neutral lipids, and lipoxygenase, which oxidizes polyunsaturated fatty acids. Becker (1966) noted that deterioration occurred more rapidly after flaking. Recently, processes have been developed for treating flakes with heat and moisture before extraction. It is claimed that such processes produce degummed oil with very low phosphatide contents which make them suitable for physical refining (Kock, 1983).

Flaking represents one of the most important operations in the processing plant. The proper flake thickness has a major impact not only on oil extraction efficiency, but also on desolventizing efficiency. Fawbush (1981) has shown that the optimum flake thickness will vary from plant to plant but is usually in the range of 0.254 mm (0.010 in) to 0.330 mm (0.013 in) for a shallow bed extractor and 0.305-0.406 mm (0.012-0.016 in) for a deep bed extractor. The optimum flake thickness is that thickness which gives the least residual oil in the extracted flakes. The purpose of flaking is not only to produce a thin flake but to disrupt the cellular organization of the meat which enables the oil and solvent to come into contact more rapidly and completely during the extraction process.

ALCON Process

The ALCON process, which is often used in Europe and Taiwan, is a process for conditioning soybean flakes. It has been claimed that the process improves extraction efficiency and quality of oil. The ALCON

process inserts an additional stage between the preparation and the extraction stages in conventional soybean milling. The purpose of this additional stage is to eliminate enzyme activity by moisture and heat treatment (Figure 1). Enzyme elimination yields a crude oil in which phosphatides can be efficiently removed by water degumming (Haraldsson, 1983). Also, because lipoxygenase is inactivated easily in processing the oil is believed to have greater oxidative stability. The ALCON process involves a three step conditioning treatment. In the first step, moisture, usually in the form of steam, is added until the moisture content of the flakes is in the 15-20% range. In the second step, the moistened meats are held for 15 min at 100°C (212°F). By stirring, during this stage, an agglomeration of the flakes occurs, leading to an increase in the bulk density and rupturing cell walls. In the third step, the flakes are dried and cooled. Precise operating conditions in the ALCON process have been tightly held trade secrets and actual conditions were not known until late in this study.

Flakes treated in this manner have a completely different granular structure from conventional flakes which is demonstrated not only in the altered bulk weight but also in the chemical composition. According to Penk (1985), the ALCON process has the following advantages compared to the conventional method:

- 1. The bulk density increases from about 93 kg/m³ (5.8 lb/ft³) to 141 kg/m³ (8.8 lb/ft³).
- Increasing the bulk density results in at least 35% greater through-put in the extractors.

Conventional preparation of soybean



ALCON process



Figure 1. Comparison of preparation steps in the conventional and ALCON processes

- 3. The percolation of the solvent or miscella increases from 175-261 1/m² min (4.3-6.4 gal/ft² min) for flakes to 419-900 1/m² min (10.3-22.1 gal/ft² min) for granules.
- Hexane retention in the extracted flakes before toasting normally amounts to 30-35%. Retention decreases in the ALCON process to less than 25%.
- 5. The phosphatides present in crude oil can be more completely hydrated and centrifugally removed. An important part of the ALCON process is that the formation of nonhydratable phosphatides is nearly eliminated (Penk, 1985).

Crude oils produced by the ALCON process have an extraordinary low phosphatide contents after water degumming with values ranging between 0.03 and 0.05% (equivalent to 10-17 ppm of phosphorus). This crude oil can be used directly for physical refining, which has lower neutral oil loss than conventional alkali refining. The differences in the phosphorus contents of crude, degummed and refined soybean oils between the ALCON process and conventional extraction are shown in Table 2. The essential prerequisite for application of the physical refining is a phosphatide content less than 20 ppm in the degummed oil.

Penk (1985) concluded that physically refined oils from the ALCON process were equal to or better than alkali refined oils with regard to their oxidation and flavor stabilities. Figure 2 illustrates the stages in conventional alkali refining that are eliminated in physical refining: acid degumming, neutralization, washing, soapstock splitting and sour water neutralization.

		 ħ	
	A/P ^a	A/A ^D	C/A ^C
Crude oils			
Acid value	2.0	2.1	1.7
Phosphatide, %	4.0	4.12	2.3
Color, Lovibond yellow/red	50/4.6	50/4.7	50/4.7
Degummed oils			
Acid value	0.93	1.03	1.19
Phosphorus, ppm	12	16	134
Color, Lovibond yellow/red	34/4.2	43/4.2	43/4.2
Peroxide value, meq/kg	1.39	1.21	1.05
Refined oils			
Free fatty acid, %	0.04	0.04	0.04
Phosphorus, ppm	2.0	2.0	3.9
Color, Lovibond yellow/red	3.3/0.8	3.8/0.7	3.8/0.7
Peroxide value, meq/kg	0	0	0

Table 2. Differences between crude, degummed and refined oils from the ALCON and conventional processes (Penk, 1985)

^aA/P = Alcon / Physical.

 b A/A = Alcon / Alkali.

 c C/A = Conventional / Alkali.

Alkali refining of crude soybean oil produced in accordance with the conventional extraction process



Physical refining of crude soybean oil produced in accordance with the ALCON process



Comparison of the steps involved in alkali refining and physical refining Figure 2.

Refining

Crude soybean oil contains relatively small and variable amounts of nonglyceride impurities which must be removed from the oil to make it suitable for human consumption. Removal of all kinds of impurities is done in a series of processes which collectively are called refining. These impurities influence the response to various processing steps and the yield of finished oil. The impurities are of two general types, oilinsoluble and oil-soluble. The insoluble impurities consist of seed fragments and excess moisture. A waxy fraction also can precipitate from some refrigerated oils and make them turbid. Impurities, such as seed fragments and meal fines, usually are removed readily by filtration. The oil-soluble impurities are more difficult to remove. The soluble impurities in crude soybean oil are gums or sludge which consists of phospholipids and their metal complexes (notably those of iron, calcium and magnesium), free fatty acids (FFA), peroxides and their breakdown products, and pigments.

The aim of the refining process is to remove these objectionable impurities with the least detrimental effect on finished oil quality and with minimum loss of triglyceride. Typical compositions of crude and refined soybean oils are given in Table 3.

<u>Chemical</u> refining

Chemical refining of edible oils almost always consists of four steps: degumming, deacidification or neutralization, bleaching and

	Crude oil	Refined oil
Triglycerides, %	95-97	>99
Phosphatides, %	1.5-2.5	0.003-0.045 ^b
Unsaponifiable matter, %	1.6	0.3
Plant sterols, %	0.33	0.13
Tocopherols, %	0.15-0.21	0.11-0.18
Hydrocarbons (Squalene), %	0.014	0.01
Free fatty acids, %	0.3-0.7	<0.05
Trace metals		
Iron, ppm	1-3	0.1-0.3
Copper, ppm	0.03-0.05	0.02-0.06

Table 3. Typical compositions of crude and refined soybean oils^a

^aPryde, 1980.

^bCorresponding to 1-15 ppm of phosphorus.

deodorizing. Since neutralization is done with caustic, the process is widely known as caustic, alkali or chemical refining.

<u>Degumming</u> Degumming of crude soybean oil is accomplished by the addition of water to the crude oil. The water hydrates most of the phosphatides and gums present in the crude oil making them insoluble in the oil and capable of being separated by centrifugation (Bell, 1985). In any event, virtually all of the gums must be removed from the crude soybean oil, either by degumming or during alkali refining, to produce a quality edible oil. Residual phosphorus in the refined oil poisons catalysts used in hydrogenation and adversely affects color, flavor and stability in the finished product.

Hydration-water is added to the crude oil based on its gum content. A typical crude soybean oil will contain between 2% and 2.5% gums. The water and crude oil mixture are gently agitated for 30-60 min at about 70°C (158°F) to hydrate the phosphatides and other colloidal impurities. The hydrated phosphatides and gums can be separated from the oil by settling, filtering or centrifuging. The oil-water mixture generally is heated to 50-70°C (122-158°F) to help break any emulsions that forms and to lower the oil viscosity before it is centrifuged (Carr, 1976).

Braae (1976) states that the pretreatment of solvent-extracted oils with acid ahead of the caustic refining step is a standard practice in Europe. Pretreatment with phosphoric acid precipitates the natural calcium and magnesium in the oil as insoluble phosphates with a high specific gravity. Addition of 0.02-1% (usually about 0.2%) of concentrated (85%) phosphoric acid to the oil at 70-90°C (158-194°F) is the usual practice. The phosphoric acid pretreatment also seemingly alters deleterious iron compounds so that they are more completely removed in the subsequent steps of caustic refining and bleaching (List et al., 1978a). The phosphoric acid pretreatment produces a darker soybean lecithin than that obtained by water degumming.

<u>Neutralization</u> Practically all edible soybean oil is refined with some type of alkali, usually caustic soda, in a continuous

processing system. The addition of an alkali solution to crude or degummed soybean oil results in chemical reactions and physical changes. The alkali combines with the free fatty acids present to form soaps; the phosphatides and gums absorb alkali and are hydrated and made insoluble; much of the coloring matter is degraded, absorbed by the gums, or made water-soluble by the alkali; and the insoluble matter is entrained with the gums and/or soapstock.

The concentration and amount of caustic used for refining crude oil varies with the free fatty acid content of the oil. Most soybean oils are refined with a 0.10-0.13% excess of caustic on a dry basis. Caustic strengths of 17-18°Baume (12-13% sodium hydroxide content) are used with soybean oil. The caustic solution is added to the warm (32°C) oil stream with good mixing and intimate contact with oil is maintained for at least 5-10 min in mixing vessels. The emulsion is thermally shocked by heating to about 75°C (167°F) to break out the soapstock which is then centrifugally separated from the neutral oil.

At the next step, the refined oil is mixed with hot, soft water and again centrifugally separated to remove small amounts of residual soap. One must use softened water to avoid formation of insoluble or unwashable calcium and magnesium soaps (Khoo et al., 1979). Sodium soaps from the refining step are readily removed from the neutral oil with water. Two things that water washing will not do, however, are to remove nonhydratable phosphatides left in the oil from the refining stage and to remove unwashable soaps.

<u>Bleaching</u> The water-washed, refined oil is continuously dried in a vacuum dryer and then is ready for bleaching. The primary function of the bleaching process is to remove color pigments. Secondarily, the process cleans up traces of soap and phosphatides from the refining step.

The basic kinds of materials used in edible oil bleaching are neutral clays, activated earths, synthetic silicates and activated carbon. For soybean oil processing, only acid-activated bleaching earths are usually used. Under atmospheric bleaching conditions, the clay should be added to dry, refined oil at 80°C (180°F), rapidly brought to a temperature of 100-110°C (212-230°F), and held at that temperature for 15-20 min to drive off moisture. Vacuum bleaching, batch or continuous, is somewhat more effective than atmospheric bleaching. Once adequately bleached, the oil should be guarded against thermal and oxidative abuses with the judicious use of heat exchangers and bottom filling tank practices (Burkhalter, 1976).

<u>Deodorization</u> The operation, sometimes known as vacuum steam distillation, was designed to remove all oxidative cleavage products that impart an odor or flavor to the oil. It may also remove tocopherols, sterols and other minor constituents. It will also remove substantial amounts of free fatty acids lowering their contents to 0.02-0.05%.

Physical refining

Physical refining of edible oils has been developed in the fats and oils industry during the past few years. One problem of the chemical

refining process is the production of soapstock, a mixture of sodium salts of fatty acids, neutral oil, water, unused caustic and other compounds resulting from the reaction of the caustic with the various impurities in the oil. The disposal of this soapstock has become very expensive due to governmental regulations (Tandy and Mcpherson, 1984). Another problem associated with chemical treatment is a significant loss of neutral oil.

Physical refining removes all of the free fatty acids, as well as the unsaponifiables and other impurities by steam stripping, thereby eliminating the production of soapstock and keeping neutral oil loss to a minimum. The physical or steam refining process has the advantages of a shorter processing time, higher yields of neutral oil and elimination of soapstock disposal. Further, the fatty acids obtained by physical refining can be marketed directly without further purification (Kanani et al., 1984). However, any phosphatides present in the feedstock will char at the high temperatures used for steam refining-deodorizing and impart dark colors to the oil. It has been claimed that a crude soybean oil suitable for steam refining-deodorizing can be prepared by water degumming followed by phosphoric acid treatment. Degumming and pretreatment of crude oils with phosphoric acid are currently recommended prior to deacidification-deodorization by steam refining (Sullivan, 1976). The purpose of the pretreatment in a physical refining system is to remove those impurities which will darken and cause a poor quality product when the oil is heated to the temperature required in the steam

refining-deodorizing unit. The pretreatment is normally a two-step operation involving the addition of a chemical to remove any trace quantities of gums remaining after water degumming and bleaching for color reduction. A water-degummed oil is mixed with 0.1-0.5% by weight of phosphoric acid at 95°C (203°F). The oil-acid mixture is then pumped to a slurry tank where 1-2% activated earth and filter aid are added.

Following pretreatment, the free fatty acid and other trace impurities are removed by steam distillation. Some of the pigments, such as the carotenoids, are converted to a colorless form by the high temperature treatment. Finally, the oil is deodorized by multistage countercurrent contact with steam (Gavin, 1978). In the chemical refining system, such a unit is called a deodorizer since it removes only trace of volatile flavor components, but in physical refining the unit is called a steam refining deodorizer since it removes all the free fatty acids and impurities. A deodorizer and steam refining deodorizer must be constructed differently because of their different functions and stripping efficiencies.

The quality of physically refined soybean oils which have been treated with phosphoric acid, degummed, steam refined and deodorized is equal in flavor to chemically refined oils both fresh and after storage (List et al., 1978b). The physical refining system has an inherently lower installation cost compared to chemical refining. Physical refining also entails lower steam, water and power requirements. Physical refining eliminates the handling of soapstock and associated waste

treatment problems that occur with caustic refining. The level of phosphorus and trace metals in crude and physically refined soybean oil are shown in Table 4.

		Trace met	als (mg/kg)
	Phosphorus,ppm	Iron	Copper
Crude oil	140	3.3	0.08
Water degummed oil, physically refined at 240°C for 2 hr	55	0.90	0.08
Phosphoric acid degummed oil, physically refined at 240°C for 2 hr	23	0.50	0.05
Phosphoric acid degummed and bleached oil, physically refined at 240°C for 2 hr	20	0.35	0.05
Normally alkali refined and deodorized oil	1-15	0.1-0.3	0.02-0.06

Table 4. Phosphorus and trace metal contents of crude and physically refined soybean oils^a

^aJawad et al., 1983.

Characteristics of Phosphatides

The soybean phospholipids in crude oil chiefly consist of phosphatidyl choline, phosphatidyl ethanolamine and phosphatidyl inositol. These phosphatides normally are hydratable with water, i.e., they swell and form gels which precipitate and are easily separated by centrifugation. Water hydration normally removes about 80-95% of the phospholipids. The residual phospholipids that are not removed by this treatment are products of hydrolysis, chiefly phosphatidic acid and lysophosphatidic acids, present as magnesium or calcium salts (Hvolby, 1971).

Phospholipids are also the main component of soybean lecithin, which is obtained from degumming sludge. The specific phospholipid that formerly was called lecithin is now referred to as phosphatidyl choline. The main phospholipids of soy lecithin are phosphatidyl choline and phosphatidyl ethanolamine (Figure 3). The position of this phosphoric acid radical is important (Carr, 1976). The phosphoric acid position shown in Figure 3 is termed the sn-3 position. Such phosphatides interact easily with water and hydrate. If the phosphate is found in the 2 position and calcium or magnesium ions are present, the phosphatide is not water precipitable without pretreatment to remove the metal ions. Such phosphatides have been identified as non- or difficult-hydratable phosphatides (Wiedermann, 1981).

Phospholipids are removed from the oil by degumming and alkali refining to avoid the problems of deposit formation during storage, oxidation and polymerization, extensive oil losses due to the emulsifying properties of phospholipids and darkening of the oil in the deodorizer (Braae, 1976). Braae et al. (1957) reported that soybean oil contains











Phosphatidyl Serine







Phosphatidyl Inositol



some phosphatides that are not removed by alkali neutralization and washing. These were referred to as nonhydratable phosphatides (NHP). Under certain conditions of bean storage, handling or processing, it is believed that enzymatic processes degrade soybean phospholipids and make them nonhydratable (Nakayama et al., 1981; Evans et al., 1974; Mounts et al., 1979). The total amount of nonhydratable phosphatides in the oil depends on the treatment of the beans at the oilseeds processing plant during conditioning and extraction. The degree of extraction to which flakes are subjected also affects the amount of nonhydratable phosphatides in the crude oil (Kock, 1983). More extensive extraction removes more phosphatides. Phospholipase D has been reported to bring about the hydrolysis of naturally occurring phospholipids in soybeans and cause the formation of nonhydratable phosphatides (Hvolby, 1971).

The nonhydratable phosphatides, which are highly unsaturated, can have a deleterious effect on oil quality. Formation of fishy odors and flavors is more likely to occur during the storage of refined oil when residual phosphatides are present. Residual phosphatides in refined oil also cause poor hydrogenation, and, by acting as sequestering agents, the residual phosphatides transfer metal ions, such as iron and copper, from water into oil during caustic washing. Such metal ion contamination contributes to reduced oil stability (Sleeter, 1981). Removal of both iron and phosphorus, which are usually present at elevated levels in oil from damaged beans, possess difficult processing problems. The nonhydratable phosphatides cause the formation of stable emulsions during

degumming and high refining losses.

Various practices have been studied to remove nonhydratable phosphatides. Such phosphatides can be removed more completely by acidulant treatment, which dissociates the calcium and magnesium phosphatide salts so that the phosphatidic acid moves from the oil phase into the water phase (Hvolby, 1971). List et al. (1978a) stated that phosphoric acid pretreatment prior to degumming did not reduce residual phosphorus in the degummed oil, but such acidification considerably reduced phosphorus content in subsequent processing steps.

Extrusion

Extrusion preparation of oilseeds for direct solvent extraction is utilized in several countries including the United States, but most extensively in Brazil. Extrusion is being promoted as a means to increase the solvent extraction rate and the extractor capacity. The conventional method of soybean preparation produces flakes suitable for extraction with hexane. In extrusion pretreatment, the explosive vaporization of water ruptures all oil cells in the oilseed, leaving the oil free in a very permeable matrix.

Rolled and properly conditioned soybean meats are introduced into the extruder. Moisture in the beans is completely diffused into the extruded mass because of the pressure and temperature developed inside the extruder. The material is forced out of a die under high pressure

forming a pellet. As these pellets leave the die and the pressure is released, the sudden vaporization of the water ruptures almost all oil spherosomes and cell walls. The oil, which is released from the meats through extrusion, is then absorbed into the pellet matrix. The pellets are quite porous and permeable. The porosity of extruded material can be changed substantially by changing the operating speeds, the type of die used and the moisture content.

Several operators in the United States are trying to determine the optimum operating condition in the extruder. Farnsworth et al. (1986) have reported the following results:

- After extraction, the extruded sample contained lower residual oils than extracted conventional flakes.
- Moisture contents below 12% resulted in pellets which were very extractable.
- Die exit temperatures between 88°C (190°) and 110°C (230°F) produced pellets that were more easily extracted than higher temperatures.
- Increasing the bulk density resulted in a 30-60% increase in extractor capacity.

According to Rittner (1984), the advantages of the extrusion process are: 1) elimination of dust problems, 2) higher extraction rates than with optimally produced flakes, 3) higher percolation and draining rates, 4) easier desolventization because of lower solvent hold-up, 5) higher capacity of available extractors because of higher extraction and percolation rates, 6) improved oil quality and meal quality (because of lower processing temperatures and heating times required to desolventize meal) and 7) extrusion conditioning of finely ground soybeans reportedly eliminated the need for the cracking, heating and flaking steps used in conventional processing (Rittner, 1984). Extrusion reportedly requires only an adequate communition of particles to pass a US #14 sieve.

Lusas et al. (1988) have studied on extrusion enhancement of oilseeds for solvent extraction. They mentioned some advantages claimed for extrusion of oilseeds especially focusing on economics. The savings to a soybean or cottonseed oil mill that installs an expander accrue from higher solvent extractor throughput; higher concentrations of oil in the miscella with reduced concentration costs for subsequent miscella refining or degumming; reduced energy costs to dry desolventized meal; and reduced capital, maintenance and energy costs when the expander replaces an expeller in a prepress-solvent extraction plant.

MATERIALS AND METHODS

Soybean and Solvent Source

Soybeans

One variety of soybean, Hack, was used in this research. The beans were certified seed obtained from Strayer Seed Farms, Inc., Hudson, IA.

<u>Solvent</u>

The solvent used to extract oil from the soybean was Skellysolve B, which is a petroleum ether fraction boiling in the range 60-70°C (140-158°F). It consists primarily of hexane isomers.

Preparation of Samples

Conventional method of making flakes

The moisture content of the original soybeans was about 7%. For the primary study, the calculated amount of distilled water required to bring the beans to 12% moisture was sprayed into 10-15 lb of soybeans. The moistened beans were mixed well and kept for 4-5 days in a refrigerator (4°C) for moisture equilibration. Beans with moisture contents of 11.3% were prepared similarly.

<u>Drying and cracking</u> Soybeans were dried in a drum drierconditioner for 2.5 hr to loosen the hulls. The drier was equipped with a fan to circulate air over the beans and keep the temperature near 30°C (86°F). The bean temperature and blower speed were controlled with a voltage Variac. The dried soybeans were cracked into 6-8 pieces each with corrugated roller mill (Ferrell-Ross, Oklahoma City, OK).

Dehulling, heating and flaking Hull removal was accomplished by passing the cracked beans through a Kice Aspirator (Kice Metal Products Co. Inc., Wichita, KS). The cracked, dehulled beans were conditioned by heating at 65-70°C (149-158°F) for 30 min to impart pliability. After conditioning, the meats were immediately flaked in a Roskamp roller mill (Roskamp Mfg. Inc., Waterloo, IA) adjusted to produce flakes with 0.254-0.305 mm (0.010-0.012 in) thickness and checked using a micrometer.

ALCON process

To simulate the ALCON process, 250 g of soybean flakes were spread uniformly on a plate to a depth of 0.5 cm. Steam was sprayed into the flakes for about 1 min at atmospheric pressure to obtain a 15-20% moisture content. The temperature of the flakes was around 85°C (185°F). Next, the flakes were brought immediately into the temperature-controlled stirring apparatus shown in Figure 4. The temperature of the soybean flakes was held at about 60°C (140°F) and the flakes were mixed thoroughly for 15 min. The agglomerated flakes were dried in an oven at 85°C (185°F) for 15-20 min in order to reduce the moisture to that of the original flakes. Late in the study, the trade secret temperature for tempering the flakes was discovered to be 100°C (212°F). Other attempts to simulate this process should be done at 100°C (212°F) for tempering.



Figure 4. Stirring apparatus used in the simulation of the ALCON process

Extrusion

Extrusion was carried out with a single-screw Insta-Pro Model 600 extruder (Triple "F" Feeds, Inc., Des Moines, IA). Soybean flakes, prepared without the conditioning step and at a moisture content of 9-10%, were fed to the extruder. All four screw segments were single flight screws. Two kinds of steamlocks were used: steamlock #8 with a diameter of 9.21 cm (3 5/8 in) and steamlock #5 with a diameter of 9.53 - cm (3 3/4 in). The steamlocks were placed between the screw segments in the order of 8-5-5-5 beginning at the feed inlet. Extruded pellets exited the die at a temperature of 112-117°C (235-242°F), were cooled at room temperature and stored at 4°C (-15°F) until extracted. Whole soybeans (11.3% moisture) and cracked beans (8.9% moisture) were also extruded.

Determination of moisture content

A sample of about 5 g was weighed into an aluminum weighing dish and was dried in a vacuum oven at 98°C (208°F) for 5 hr under 26 mmHg vacuum. After cooling in a desiccator, the sample was weighed again and the loss in weight was reported as the moisture content.

Extraction Characteristics

Extraction rate

The oil extraction rates of the various soybean preparations were measured with a laboratory glass extractor-simulator. The flow scheme of

the extractor is shown in Figure 5. This apparatus simulates extraction in a percolation extractor. The extractor was charged with 200 g of the sample, and the sample was extracted in six stages. In the first stage, 400 g of solvent was pumped over the samples. Percolation was simulated by spraying the solvent on the bed of flakes, allowing it to drain through the flakes and recycling the miscella back to the top of the flakes. This cycle was continued for 6 min. At the end of the extraction period, the bed of flakes was allowed to drain for 3 min. After draining, the volume of miscella was measured and an equivalent amount of fresh solvent was used in the next stage. Extraction temperature was maintained at 56-58°C (133-137°F). The extraction process was repeated six times using fresh solvent for each stage. Crude oils were obtained from miscella by evaporating the solvent using a rotary evaporator (Buchi model RE121, Brinkmann Inc., Vendor, NY). The oil was stored under nitrogen at -20°C in screw cap culture tubes.

<u>Residual oil content</u>

Residual oil content was determined by the acid hydrolysis method, AACC Method 30-10.

Solvent hold-up

After draining the solvent at the last stage, the extractor contents were weighed. The solvent present was evaporated by air drying for 48 hr. Solvent hold-up was calculated as the weight loss due to ambient evaporation divided by the dried weight.



Figure 5. Flow scheme of the laboratory extractor-simulator

Bulk density

Samples were placed in a beaker up to the level of one liter and the contents were weighed. Bulk density was calculated by dividing the weight by the volume.

Percolation rate

Percolation rate was determined using the assembly shown in Figure 6. A constant volume of sample was placed in a 1.5 m (5 ft) tall cylinder which had a diameter of 7 cm (2.74 in). A screen used in this study was US #18. Fresh solvent was added to a depth of 45 cm (1.5 ft) above the layer of the bed and the time required for the solvent level to drop from 30 cm (1 ft) above the bed to the top of the bed was measured. No data were taken until the solvent completely wetted the bed and began to flow from the test cell. The percolation rates were compared among samples at four different bed depths of the soybean preparations and were reported as the volume of solvent percolated per unit area per unit time.

Phosphatide Content

The phosphorus contents in both the crude and degummed oils were determined by modifications of the procedure of Rouser et al. (1966). The amount of oil obtained from each stage was not enough to use the American Oil Chemists' Society's standard method; therefore, this microanalytical procedure was adopted for a phosphorus analysis. Small



Figure 6. Assembly for comparing percolation rates

aliquots of oil were dropped into 30-ml Kjeldahl digestion flasks. After adding 0.9 ml of 72% perchloric acid, digestion was carried out on an electrically heated Kjeldahl rack for about 20 min. After digestion, the sides of the flask were rinsed with 5 ml of distilled water $(PO_4^{-3} \text{ free})$, and 1 ml of 2.5% ammonium molybdate solution was added. The flask was swirled and 1 ml of freshly-prepared 10% ascorbic acid solution and 2 ml of water $(PO_4^{-3} \text{ free})$ were added. The solution was transferred to a test tube and heated in a boiling water bath for 5 min. After cooling, the optical density was measured at 820 nm with a Beckman spectrophotometer, and the phosphorus content was calculated using a standard curve. The phosphatide content was calculated by multiplying the phosphorus content by 30.

Degumming with Water

Water was added to the crude oil in an amount about equal to the weight of gums in the oil and gently mixed for 20-30 min. Centrifugation at 260 x g for 15 min was used to completely separate the hydrated phosphatides from the oil.

Statistical Analysis

A Statistical Analysis System (SAS) computer package was used to perform an analysis of variance to determine if differences existed among treatments. Duncan's multiple range test or the least significant difference (LSD) procedure was used to determine differences between the experimental treatments.

RESULTS AND DISCUSSION

Extraction Rate

Working conditions for making flakes in a commercial plant were compared with our pilot plant conditions in Table 5.

Condition	Plant scale ^a	Pilot plant scale			
Moisture content of whole soybeans, %	10.0	11.6			
Temperature of whole soybeans before cracking, °C (°F)	32 (91)	30-32 (86-90)			
Moisture content of cracked beans, %	8.8	9.3			
Temperature of cracked beans, °C (°F)	60 (136)	60 (136)			
Seive analysis of cracked beans #8 seive, % #10 seive, % #20 seive, % pan, %	88.5 3.0 7.5 1.0	83.2 7.4 8.8 0.6			
Flake thickness, mm (in)	0.305-0.381 (0.012-0.015)	0.229-0.356 (0.009-0.014)			

Table 5. Working conditions for making soybean flakes

^aAG Processing, Inc., Eagle Grove, IA.

The extraction characteristics of extruded soybean pellets were compared with those of conventional soybean flakes and agglomerates from simulation of the ALCON process in Figure 7. The percentage of oil extracted was calculated by measuring the oil obtained at each stage divided by the amount of oil obtained before extraction by the acid hydrolysis method. The extruded pellets extracted faster and more completely than conventional flakes or ALCON agglomerates. After six stages, 93.3% of the oil was extracted from extruded pellets compared to 90.6% from flaked soybeans. The sixth stage of the extraction of flakes was not quite as complete as that achieved at the third stage of the extraction of extruded pellets. At 6 min per stage, this suggests extraction efficiency equivalent to that obtained in 36 min with flakes could be achieved within 18 min with extruded pellets. These results suggest extrusion would increase extraction capacity by 100% and achieve the same residual oil content in the meal by merely making the material more extractable. The agglomerates obtained from the ALCON process showed a higher oil extraction rate than flakes from the conventional method, but the ALCON agglomerates gave lower oil recoveries than the extruded pellets. Perhaps the ALCON process converts some free fat to bound fat making it unextractable with hexane.

Residual oil contents in the extracted samples at each stage are shown in Figure 8. Through three stages, there were significant differences in the residual oil content of the meal between extrusion preparation and that from the conventional pretreatment method. The



Figure 7. Extraction characteristics of the extruded pellets, conventional flakes and ALCON agglomerates (LSD was 4.14)



Figure 8. Percentage unextracted oil in the extracted meal samples at each stage of extraction

residual oil content, which depends upon the thickness of the flake and the extraction time, was lower for extruded pellets than conventional flakes.

In order to study the extraction rates among soy flakes, Norris (1982) introduced the following equation for comparing of extraction rates. It assumes a simple diffusion model:

$$E = \frac{8}{2} \sum_{n=0}^{n=\infty} \frac{1}{(2n+1)^2} \exp ((2n+1)^2 \left(\frac{\pi}{2}\right)^2 \left(\frac{D\theta}{R^2}\right)$$

where E is the fraction of the total oil unextracted at the end of time θ (in hours), R is one-half the flake thickness (in feet) and D is the diffusion coefficient (in square feet per hour). The preceding equation takes the form:

$$E = \frac{8}{\pi^2} \exp -\frac{\pi^2 D\theta}{4R^2}$$

or

$$\log_{10}E = -0.091 - 1.07 \frac{D\theta}{R^2}$$

Our data through three stages were fitted to the model and a plot of $\log_{10}E$ against θ (Figure 9) gave a straight line whose slope was dependent on the diffusion coefficient and the flake thickness (average 0.0292 mm). The slope for conventional flakes was 3.03 hr⁻¹. For extruded pellets and ALCON agglomerates, however, the data could not be



Figure 9. Extraction curves for extruded pellets, conventional flakes and simulated ALCON agglomerates

applied to the model due to their shapes. When assuming that the extruded pellets and ALCON agglomerates have constant thicknesses like the conventional flakes, slopes of 3.67 and 3.13 hr⁻¹ were obtained, respectively. An effective particle thickness was obtained from the slopes and the extraction equations. When a diffusion coefficient (6.50 x 10^{-7} ft²/hr) determined experimentally for conventional flakes was applied to the extruded pellets and the ALCON agglomerates, the "hypothetical" thicknesses of the extruded pellets were 0.264 mm (0.0104 in) and those of the agglomerates were 0.287 mm (0.0113 in).

We also calculated equivalent extraction times using the hypothetical thicknesses and the following empirical relationship (Becker, 1978):

$$\frac{\underline{Y}\underline{a}}{\underline{Y}\underline{b}} = \left(\frac{\underline{X}\underline{a}}{\underline{X}\underline{b}}\right)^{2.2}$$

Ya = minutes extraction with sample a Yb = minutes extraction with sample b Xa = inch flake thickness of sample a Xb = inch flake thickness of sample b

extraction of the conventional flake required about 1.25 longer time to attain the same residual oil level as extruded pellets and 1.04 times greater than simulated ALCON agglomerates. Extruded samples also were prepared from whole soybeans with 11.3% moisture and from cracked soybeans with 8.9% moisture. The pellets extruded from whole soybeans contained some fairly large pieces of soybeans. For the cracked beans, there were only a few large fragments. The yields of oil compared among those extruded pellets are shown in Figure 10. The yield of oil extracted from the pellets of whole soybeans was lower than those from the other two types of pellets. Probably, this was because the large bean pieces in this extruded product contained a large proportion of intact, undistorted cells which were resistant to extraction. The pellets from the cracked beans yield more oil on extraction than conventional flakes. Thus, one can get a higher yield of oil from soybeans without making flakes before extrusion simply by breaking the whole beans to smaller fragments or by fine grinding.

Phosphatide Content

The values of corrected optical densities from the phosphorus analysis were converted to micrograms of phosphorus using a factor derived from a standard curve prepared using disodium phosphate. The standard curve was linear over the concentration range from 0-10 μ g. The factor was 14.3 for these standard amounts. The correlation coefficient for the relationship of absorbance and concentration was 0.9994.

Figure 11 shows the phosphatide contents of the crude oils extracted from extruded pellets, conventional flakes and ALCON agglomerates. As the number of stages increased, the amount of phosphatides in the crude



Figure 10. Extraction characteristics of the extruded pellets obtained from flaked, cracked and whole soybeans



Figure 11. Phosphatide contents in crude soybean oils extracted from soybeans subjected to different pretreatments

oils increased for conventional flakes and ALCON agglomerates. These data suggest that a portion of slowly extracted oil was associated with phosphatides which are important constituents of cell membranes. For extruded pellets, however, the phosphatide content was relatively constant at each stage. This observation suggests that distortion of the cell structure of soybeans was more extensive in extruded samples so that the phospholipids and neutral lipids are extracted at nearly equivalent rates. Table 6 compares the total phosphatide contents before and after degumming the oils extracted from various samples. In our experiments,

Table 6. Phosphatide contents of the oils obtained from extruded pellets, conventional flakes and simulated ALCON agglomerates before and after degumming

	Extruded	Pellets	Conventi	onal Flakes	ALCON Agglomerates		
	Before	After	Before	After	Before	After	
	Degumming		Degumming		Degumming		
Phosphorus,	855 ^a	17 ^a	535 ^b	44 ^b	598 ^b	63 ^c	
Phosphatide content, %	2.57 ^a	0.05 ^a	1.60 ^b	0.13 ^b	1.80 ^b	0.19 ^c	

¹Means in the same horizontal row for 'before' and 'after' degumming bearing different letters differ significantly at the 5% level.

the amounts of oil obtained after the second stage were too small to carry out the degumming step under the same conditions used for the first two stages. It is known that the conditions for degumming, such as the amount of oil, the percentage of water added, mixing speed and mixing time, affect the amount of phosphatides remaining in the oil (Wiedermann, 1981). Therefore, the total amount of phosphatides after degumming, which was nonhydratable, was calculated on the basis of observations on the first two stages.

The total content of phosphatides before degumming was higher in the oil extracted from extruded pellets than oils from conventional flakes or ALCON agglomerates. The difference in total phosphatide contents of crude oils from extruded pellets and conventional flakes was statistically significant (P > 5%).

The total amount of phosphatides in the oils after degumming shows that most of the phosphatides extracted from the extruded pellets were hydratable. There was a 99.95% decrease in phosphorus content after water-degumming in oil extracted from extruded pellets. The degummed oils from extruded pellets had much lower phosphorus contents (17 ppm) than those from conventional flakes (44 ppm) despite the fact that crude oil from extruded pellets before degumming has the highest phosphatide content. These values for phosphatides in degummed oil from conventional flakes were lower than the usual values for degummed soybean oil (Table 2). This difference indicated less formation of nonhydratable phosphatides occurred during the course of preparation and extraction of our soybean flakes than might be typical in a commercial operation or with poorer quality raw beans. Kock (1983) illustrated that the content

of nonhydratable phosphatides increased in the degummed soybean oil with increasing moisture content of the beans and higher extraction temperatures because of greater phospholipase activity. Phospholipase will be inactivated by suitable thermal processing with dry heat or steam. Extrusion preparation evidently brought about the same effects as conventional thermal processing.

For the ALCON process, we obtained higher levels of phosphatides in degummed oil than we expected. Following completion of this experiment, it was learned that the holding temperature in the conditioning step of the ALCON process was not enough high to inactivate phospholipases (60°C verses 100°C). Because the temperatures used in the ALCON process have been closely guarded industrial secret and information to simulate this process has been difficult to obtain, conditions used in this study may have actually increased the amount of nonhydratable phosphatides. This result may be because we had insufficient information about the process conditions or because we were unable to truly simulate the process on a laboratory scale.

Table 7 shows that extruded pellets obtained from whole soybean yielded crude oils that were lower in total phosphatides than that of the oils extracted from extruded pellets obtained from flakes. Extruded whole beans contain a number of large pieces in which the cell walls are not completely ruptured. In such material more phospholipids would be incompletely extracted and remain in the meals.

Food	Phosphatic	de content, %
reeu	Before degumming	After degumming
Flaked beans	2.57	0.05
Cracked beans	2.34	0.09
Whole beans	1.42	0.02

Table 7.	Phosphati	de conte	nt of	the	oils	extract	ted	from	the	extruded
	pellets p	roduced	from	flake	d, ci	cacked a	and	whole	soy	vbeans

Extraction Capacity

Bulk density and solvent hold-up

Bulk density and solvent hold-up for each sample are presented in Table 8. The bulk densities of the extruded pellets were significantly different from those of conventional flakes and simulated ALCON agglomerates. The bulk density of the extruded pellets was about 450 kg/m^3 (28 lb/ft³) compared to 320 kg/m^3 (20 lb/ft³) for flakes. This difference alone could result in about a 40% increase in additional extractor capacity. Our results agree with the data of Farnsworth et al. (1986) who observed that extruded pellets had a higher bulk density than flakes. The ALCON agglomerates, which were shown to be extracted more readily than flakes, also had a higher bulk density than flakes but lower than extruded pellets.

Solvent hold-up is the amount of solvent that does not drain from spent flakes and must be recovered by evaporation. The amount of solvent Table 8. Comparisons of bulk density and solvent hold-up

	Bulk ₃ density,3 kg/m (lb/ft ³)	Solvent hold-up, %
Extruded pellets	451.7 ^a (28.2)	14.4 ^a
Conventional flakes	322.0 ^b (20.1)	45.2 ^b
ALCON agglomerates	406.9 ^c (25.4)	35.3 ^c

¹Means in the same column bearing different letters differ significantly at the 5% level.

left in the extracted pellets was significantly different from the amounts left in the spent flakes and agglomerates. Solvent hold-up in extracted pellets was 15% compared to 45% for conventional flakes. This means that 67% less solvent needs to be evaporated after gravity drainage from the extruded pellets. Because of the lower solvent hold-up, the steam consumption in desolventizing extruded pellet would be substantially lower than that involved in desolventizing flakes.

Percolation rate

Good percolation (flow of solvent through the bed) is necessary for efficient extraction. Percolation rates were determined using fresh solvent in our experiment because oil in the miscella greatly affected solvent flow. Percolation rates for each sample at different bed depths are presented in Table 9 and Figure 12. Percolation rates at each bed depth were significantly different among samples. Percolation rates in

Bed depth, ft	Extruded pellets	Flakes • min, gal/ft• m	ALCON agglomerates
1	1800, 44.2 ^a	370, 9.1 ^b	794, 19.5 [°]
2	1555, 38.2 ^a	297, 7.3 ^b	639, 15.7 ^c
3	1543, 37.9 ^a	277, 6.8 ^b	533, 13.1 ^c
4	1380, 33.9 ^a	244, 6.0 ^b	478, 11.0 ^c

Table 9. Percolation rates at different bed depths of various soy preparations

¹Means in the same row for bearing different letters differ significantly at the 5% level.

shallow beds were about 50% higher than in deep beds for flakes. Similar trends were noted for the percolation rates in both extruded pellets and ALCON agglomerates. Penk (1985) reported that one of the advantages of the ALCON process was that ALCON agglomerates gave higher solvent percolation rate. Our data were very close to his and showed percolation rates for ALCON agglomerates significantly higher than those for flakes. Percolation rates in extraction of extruded pellets ranged from 1380 1/m². min (34 gal/ft². min) to 1800 1/m². min (44 gal/ft². min) which was about two-fold greater than the percolation rate through agglomerates and about five-fold greater than the percolation rate through flakes.

<u>Residual oil content</u>

Another significant factor in extraction efficiencies was the residual oil contents of the extracted meals. Data on residual oil



Figure 12. Percolation rate of solvent for extraction of soybean oil

contents in our experiments is given in Table 10. The meals of extruded pellets exhibited lower residual oils than those obtained from flakes. Most oil exists in spherosomes in cells, but a smaller portion, termed "bound fat," may be complexed with protein and not be extractable without hydrolysis. Farnsworth et al. (1986) also examined residual oil contents of extracted meals of extruded pellets. They showed that extrusion of soybeans did not appear to increase bound fat. Our data, obtained from an acid hydrolysis method, agreed with their data.

	Extruded pellets	Flakes	ALCON agglomerates
Initial	23.8	26.5	26.4
After 6 stages	1.9	2.5	3.2

Table 10. Residual oil contents of extracted meals^a

^a% oil on moisture-free basis.

SUMMARY AND CONCLUSION

The purpose of this study was to compare the oil quality, extraction rate and extraction efficiency of soybeans prepared by extrusion, conventional flaking and the simulated ALCON process. A degummed oil with a very low phosphorus content (17 ppm) was obtained using extrusion preparation of flaked soybeans. This phosphorus content was low enough to allow the use of physical refining. The phosphorus content in degummed oil from extruded pellet was better than from the ALCON process.

Faster and more complete extraction of oil was accomplished with extruded pellets. Percolation rates for extruded pellets were about five-fold greater than the percolation rates for conventional flakes due to large differences in porosity and permeability. The residual oil content in the meal of extruded pellets was lower than that of conventional flakes or ALCON agglomerates. The increased bulk density from extrusion preparation resulted in about a 40% increase in capacity, and the 67% less solvent hold-up than in conventional flakes reducing steam consumption in the desolventizing process. Extrusion pretreatment can give rise to greater extractor capacity, increased extraction efficiency and improved oil quality.

BIBLIOGRAPHY

- Barger, W. M. 1981. Handling, transport and preparation of soybeans. J. Am. Oil Chem. Soc. 58:154-156.
- Becker, K. W. 1966. Advances in solvent extraction: Its meaning to the food industry. Chem. Eng. Progress Symposium Series, No. 86 64:60-73.
- Becker, K. W. 1978. Solvent extraction of soybeans. J. Am. Oil Chem. Soc. 55:754-761.
- Bell, W. J. 1985. Degumming soybean oil. Oil Mill Gaz., May 1985:45-47.
- Braae, B. 1976. Degumming and refining practices in Europe. J. Am. Oil Chem. Soc. 53:353-357.
- Braae, B., U. Brimberg, and M. Nyman. 1957. Some problems involved in the water wash of neutralized vegetable oils. J. Am. Oil Chem. Soc. 34:293-299.
- Brignoli, C. A., J. E. Kinsella, and J. L. Weihrauch. 1976. Comprehensive evaluation of fatty acids in foods. J. Am. Diet. Assoc. 68:224-229.
- Burkhalter, J. P. 1976. Crude oil handling and storage. J. Am. Oil Chem. Soc. 53:332-333.
- Burlison, W. L. 1936. The soybean: A plant immigrant makes good. Ind. and Eng. Chem. 28:772-777.
- Carr, R. A. 1976. Degumming and refining practices in the United States. J. Am. Oil Chem. Soc. 53:347-352.
- Evans, C. D., G. R. List, R. E. Beal, and L. T. Black. 1974. Iron and phosphorus content of soybean oil from normal and demaged beans. J. Am. Oil Chem. Soc. 51:444-448.
- Farnsworth, J. T., L. A. Johnson, J. P. Wagner, L. R. Watkins, and E. W. Lusas. 1986. Enhancing direct solvent extraction of oil seeds by extrusion preparation. Oil Mill Gaz., Nov. 1986:30-35.
- Fawbush, J. G. 1981. How to condition and flake to produce good quality. Oil Mill Gaz., May 1981:39-42.

- Fawbush, J. G. 1977. Soybean preparation. J. Am. Oil Chem. Soc. 55:474A-476A.
- Gavin, A. M. 1978. Edible oil deodorization. J. Am. Oil Chem. Soc. 55:783-791.
- Good, R. D. 1970. Theory of soybean extraction. Oil Mill Gaz., Sep. 1970:14-17.
- Haraldsson, G. 1983. Degumming, dewaxing and refining. J. Am. Oil Chem. Soc. 60:203A-208A.
- Hvolby, A. 1971. Removal of nonhydratable phospholipids from soybean oil. J. Am. Oil Chem. Soc. 48:503-509.
- Jawad, I. M., S. P. Kochhar, and B. J. F. Hudson. 1983. Quality characteristics of physically refined soybean oil: Effect of pre-treatment and processing time and temperature. J. Food Tech. 18:353-360.
- Kanani, M. K., D. J. Zuilichem, J. P. Roozen, and W. Pilnik. 1984. A modified procedure for low temperature infrared radiation of soybeans. Lebensm.-Wiss. U.-Tech. 17:39-41
- Khoo, L. E., F. Morsingh, and K. Y. Liew. 1979. The adsorption of beta-carotene 1. by bleaching earths. J. Am. Oil Chem. Soc. 56:672-675.
- Kock, M. 1983. Oil seed pretreatment in connection with physical refining. J. Am. Oil Chem. Soc. 60:150A-154A.
- List, G. R., T. L. Mounts, and A. J. Heakin. 1978a. Steam-refined soybean oil: Effect of degumming methods on removal of prooxidants and phospholipids. J. Am. Oil Chem. Soc. 55:280-284.
- List, G. R., T. L. Mounts, K. Warner, and A. J. Heakin. 1978b. Steamrefined soybean oil: Effect of refining and degumming methods on oil quality. J. Am. Oil Chem. Soc. 55:277-279.
- Lusas, E. W., L. R. Watkins, and L. A. Johnson. 1988. Extrusion for solvent extraction. J. Am. Oil Chem. Soc. 65:1109-1114.
- Markley, K. S. 1950. Soybean and soybean products. Interscience Publishers, New York.
- Minshew, B. 1977. Soybean Dig. 37:16h.

- Mounts, T. L., G. L. List, and A. J. Heakin. 1979. Postharvest handling of soybeans: Effects on oil quality. J. Am. Oil Chem. Soc. 56:883 -886.
- Nakayama, K., K. Saio, and M. Kito. 1981. Decomposition of phospholipids in soybeans during storage. Cereal Chem. 58:260-264.
- Norris, F. A. 1982. Extraction of fats and oils. Pages 175-251 <u>in</u> D. Swern, eds. Bailey's industrial oil and fat products. vol. 2 John Wiley & Sons, Inc., New York.
- Penk, G. 1985. Practical experience with ALCON process. Presented at the Am. Oil Chem. Soc. Congress, Cannes, France, Nov. 3-8, 1985.
- Pryde, E. H. 1980. Composition of soybean oil. Pages 13-31 <u>in</u> Handbook of soy oil processing and utilization. D. R., Erickson,
 E. H. Pryde, O. L. Brekke, T. L. Mounts, and R. A. Flab, eds. American Soybean Association, St. Louis, MO.
- Rittner, H. 1984. Conditioning of oil-bearing materials for solvent extraction by extrusion. J. Am. Oil Chem. Soc. 61:1200-1203.
- Rouser, G., A. N. Siakotos, and S. Fleischer. 1966. Quantitative analysis of phospholipids by thin-layer chromatography and phosphorus analysis of spots. Lipids 1:85-86.
- Serrato, A. G. 1981. Extraction of oil from soybeans. J. Am. Oil Chem. Soc. 58:157-159.
- Sleeter, R. T. 1981. Effect of processing on quality of soybean oil. J. Am. Oil Chem. Soc. 58:239-247.
- Sullivan, F. E. 1976. Steam refining. J. Am. Oil Chem. Soc. 53:358-360.
- Tandy, D. C., and W. J. Mcpherson. 1984. Physical refining of edible oil. J. Am. Oil Chem. Soc. 61:1253-1258.
- Wiedermann, L. H. 1981. Degumming, refining and bleaching soybean oil. J. Am. Oil Chem. Soc. 58:159-166.

ACKNOWLEDGMENTS

I express my sincere gratitude to my advisor and major professor, Dr. Mark H. Love, for his guidance, patience and assistance throughout the entire course of my graduate education. I wish to thank the committee members, Dr. Lawrence Johnson and Dr. Earl Hammond, for their advice relating to this study.

Thanks are also extended to all my graduate colleagues, especially, Chuljai Kim and Inmok Lee, who assisted in this study. I also wish to express thanks to my parents and Junghee Lee for their encouragement and inspiration.