Aqueous Extraction—An Alternative Oilseed Milling Process¹

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ABSTRACT

Oil can be removed from oilseed materials by a process which consists of an aqueous extraction of the comminuted seed, followed by a centrifugal separation which divides the aqueous extract into oil, solid, and aqueous phases. The protein may be recovered in the solids or aqueous phase, depending upon the conditions selected. Unit operations of this process are grinding, solid-liquid separation, centrifugation, demulsification, and drying of products. Aqueous extraction has been applied, to date, to coconuts and peanuts. For coconuts, a procedure has been developed to recover 93% of the oil and 91% of the protein. The major protein product is 25% protein and, when reconstituted in water, forms an acceptable beverage. The estimated production cost of this product is \$.24/lb. For peanuts, the recovery of oil was 89% and protein 92% for the concentrate procedure, whereas the corresponding values for the isolate procedure were 86% and 89%, respectively. The costs of production were estimated as \$.17/lb of concentrate (67% protein) and \$.28/lb of isolate (89% protein). Aqueous extraction offers several advantages over conventional solvent extraction-less initial capital investments, safer operation, capability of discontinuous operation, and production of a variety of products. Another advantage of aqueous processing is the capability for utilization of certain chemicals to remove or inactivate undesirable substances. In the case of peanuts, hydrogen peroxide - and sodium hypochlorite have proven to be effective for destruction of aflatoxins. Aqueous processing has the potential for application to a variety of other oilseeds.

INTRODUCTION

Modern oil milling technology is considered to date from the invention of the hydraulic press by Bramah in the late 18th century. This process consists of applying hydraulic pressure to oil bearing materials contained in bags, cages, or cloths. The process is comparatively efficient, but it is a batch-type operation and is labor intensive. Consequently, to a considerable extent, it has been superseded by continuous pressing using a screw press or expeller, or by solvent extraction, or by a combination of these two methods. It is these types of processes which are mainly used in those countries which have modern oil milling industries today.

All the processes mentioned above are primarily intended to produce edible oil. As a result, very little, or no, attention is given to the quality of the protein residues. The residue (or flakes) from the solvent extraction process must be desolventized, whereas the cake from the expeller process must be ground before either product can be used in the manufacture of compound feed stuffs. In recent years, however, increasing awareness of the need and importance of proteins for human nutrition has provided motivation for the beginning of significant changes in oil milling processes and practices. These changes fall into two broad categories: (A) those arising from the fact that the requirements for food-grade products are different than for feed-grade and (B) those concerned with more fully exploiting the potential nutritive value of the oilseed proteins. The first involves matters of sanitation and the presence in the end products of materials, such as fiber and other indigestible matter. The second involves avoiding damage to the proteins of the seed through overly severe processing conditions, such as excessive heat and, also, inactivation or removal of antinutritional factors which are present in some oilseeds and which can reduce the nutritive value of their proteins or which may be toxic to humans.

During the last two decades, several new processes for producing protein products from oilseeds have been suggested (1-8) as alternatives to conventional oil milling procedures. The new processes differ radically from standard oil milling processes in that they all employ an aqueous system of some kind to extract oil and proteins. All involve removal of at least part of the oil content of the oilseed; and, thus, they are more akin to oil milling than to the processes designed to produce full-fat products. These processes have been reviewed and compared by several workers (9-12); but, with the exception of two processes (5, 7), they have never been utilized for commercial production. Even these two (CFTRI and CHAYEN) are only in limited use today.

The aqueous extraction process developed in the Food Protein Research and Development Center, Texas A&M University, is designed for the simultaneous recovery of oil and protein concentrates or isolates from fresh coconuts (13) and peanuts (14, 15) utilizing the same basic concept.

The objectives of this paper are: (A) to discuss unit operations involved in the aqueous process; (B) to introduce the techniques which have been applied to coconuts and peanuts; (C) to show material balances of various products based upon pilot plant trials; and (D) to discuss economics involved in manufacturing protein products. In addition, the advantages and disadvantages of these processes and their potential applicability to other oilseeds are discussed briefly.

UNIT OPERATIONS

Oil and protein can be recovered simultaneously from oilseed materials by a process which consists of an aqueous extraction of the comminuted seed, followed by a centrifugal separation of the slurry into oil, solid, and aqueous phases. The proteins, either as concentrates or isolates, can be recovered in either the solid or aqueous phase, depending upon the conditions selected. The efficiency of the process is critically dependent upon several unit operations: grinding, solid-liquid separation, centrifugation, demulsification, and drying of products. Specific parameters utilized in each of the unit operations for processing the various oilseeds differ to some extent and are governed by differences in chemical compositions and physical structures. The following is a general description of each unit operation.

Grinding: The first critical step in the process is the grinding of the oil bearing materials. Grinding presents a problem since the seed cells must be ruptured: to release their constituents and to increase the efficiency of extraction of the oil and proteins. However, excessive grinding

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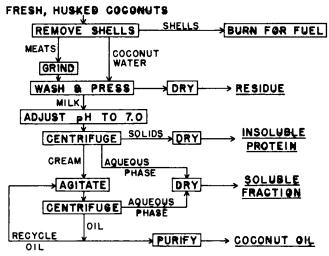


FIG. 1. Simplified flow chart for the aqueous processing of coconuts.

can produce smaller oil globules which make demulsification more difficult. Insufficient grinding, on the other hand, results in intolerable losses of oil in the residue.

The grinding may be carried out either wet or dry depending upon the oilseed to be processed. However, the choice between wet or dry grinding would be governed by the initial moisture content, the chemical composition, and physical structure of the oilseeds. For instance, coconuts, which have an initial moisture content of ca. 50%, can more economically be ground wet to avoid a costly drying step. It is generally considered that wet grinding may be somewhat superior to dry grinding in rupturing cells because the water softens the cell walls. However, with peanuts, wet grinding has been observed to produce a stable emulsion which has to be broken to recover oil at a later step. Since peanuts have a low initial moisture content of ca. 5%, they are best ground dry. Therefore, the selection of proper grinding methods and conditions and degree of grinding is considered critical to the efficiency of the overall operation.

Extraction: Basically the extraction step is carried out by dispersing the ground seeds in water and then agitating the dispersion to enhance the extraction of seed constituents. Factors which influence the efficiency of extraction include: solid-to-water ratio, kinds of salts and their concentrations, pH of the dispersion, extraction time and temperature, and degree of agitation.

Solid-liquid separation: Because of the rather high percentage of solids in the dispersion, removal of the major portion of the undissolved solids has been found to be necessary with coconuts and peanuts to make possible the

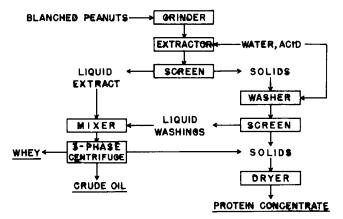


FIG. 2. Simplified flow chart for the aqueous processing of peanuts (concentrate procedure).

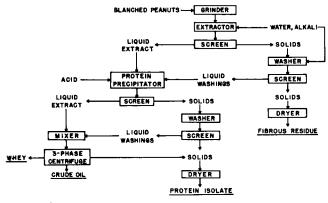


FIG. 3. Simplified flow chart for the aqueous processing of peanuts (isolate procedure).

efficient recovery of oil by centrifugation. The solids are mainly fibrous material, undissolved carbohydrates and proteins, depending upon the pH employed in the extraction step. Filtration through vibrating or pressing-type screens or through a clarifying centrifuge can be used for this purpose. The selection of a particular method will be determined by the physical nature of the material in the dispersion and also by operating costs.

Centrifugation: One of the key steps in the aqueous process is the three phase centrifugation in which the liquid containing both dissolved and undissolved matter is separated into oil, solid, and aqueous phases. Depending upon the material and the conditions selected, the oil can be recovered as free oil or as an oil-in-water emulsion.

Demulsification: If an emulsion has been formed, it can be broken using the phase-inversion technique described by Sugarman (2) in which clear oil is added to the emulsion. With the aid of shear force at an elevated temperature, nearly all of the oil can be freed by this procedure. This technique requires control of the emulsion pH and also reduction of the moisture content below a certain critical level, above which the emulsion cannot be broken. In either case, oil recovered from the first centrifugation or after breaking the emulsion is a high quality, water-washed product which requires little, if any, further treatment, except possibly removal of water by recentrifugation or vacuum drying.

Drying: The removal of water from protein products, usually achieved by spray-drying, is the most expensive step of aqueous processing. Spray-drying of high protein products, such as the peanut concentrates and isolates, presents no major technical problems since the proteins are relatively heat stable and the products are not hygroscopic. However, spray-drying of the whey-type coconut products has been difficult because of the lower protein content and the hygroscopic nature of the other constituents which are principally sugars and salts.

SPECIAL ASPECTS OF AQUEOUS PROCESSING OF COCONUTS AND PEANUTS

Ceconuts

The problem of coping with the large amount of fibrous material and low protein content is particularly important in the aqueous processing of coconuts. Coconut meats contain ca. 2.5% crude fiber and 3.1% crude protein (%N x 5.30).

The aqueous processing scheme developed for coconuts is shown in Figure 1. Grinding of the meats must be thorough to allow efficient extraction of the oil from the fiber. Since the meats are initially high in moisture (50%moisture and 35% oil), the grinding produces a rather stable emulsion which is separated with some difficulty from the fiber. However, extracting and pressing three times can accomplish efficient extaction.

The emulsion is broken by inversion, after reducing the proportion of moisture through addition of recycled oil. Inversion is accomplished by means of agitation, which, in practice, is achieved with a high velocity centrifugal pump (Tri-Clover, 3 horsepower, 3805 rpm).

Because the soluble fraction contains the soluble sugars and salts initially present in the coconut, the product is quite hygroscopic and difficult to dry.

Peanuts

Two processes, one for the production of a protein concentrate (Fig. 2) and the other for an isolate (Fig. 3), have been developed for the aqueous extraction of peanuts. Unlike coconuts, peanuts contain a relatively large amount (27-29%) of protein (N x 5.46), as well as oil (46-49%), but are low in initial moisture content (5%). The differences in composition from coconuts bring a different kind of problem. Here the problem is how to cope with a large amount of solids in the aqueous dispersion.

Grinding the peanuts to a peanut butter consistency or flaking to a thickness of 0.1 mm or less resulted in efficient extraction of both oil and protein.

The major difference between the isolate and concentrate processes for peanuts lies in the initial pH of extraction: isoelectric pH (4.0) for concentrates and alkaline pH (8.0) for isolates. The concentrate procedure is a process in which the acid soluble proteins, carbohydrates, oil, and other constituents are extracted leaving the fiber and acid insoluble substances in the protein concentrate. The isolate procedure, on the other hand, is a process which separates alkaline soluble substances from fiber and other insoluble materials prior to the recovery of oil and proteins.

It may be noted that, in both of the peanut processes, there is a whey or waste water product stream. Another product composed of low mol wt, nonprecipitable proteins,

TABLE I

Material Balance of Various Products from the Aqueous Processing of Coconuts

Product	Dry wt (kg)	Oil (kg)	Crude protein (kg) (N x 5.30)		
1000 kg fresh,					
husked coconuts	235 ^a	153	14.3		
Oil	140	140	0		
Soluble fraction	45	2.2	10.0		
Residue	44	10	1.3		
Insoluble protein	5.7	0.7	3.1		

^aIncludes meats and coconut water but not the shells. The coconut water contributes 14.5 kg of solids. The coconuts are, on a wet basis, 44% meats, 31% coconut water, and 25% shells.

sugars, and salts may be recovered from this waste water or whey by the techniques of ultrafiltration and reverse osmosis. These materials have unique properties in terms of solubility over a wide pH range, whippability, and low viscosity. This procedure has the added advantage of removing the pollution potential of the waste water.

MATERIAL BALANCE

The distribution of the components of the starting material and of the products from the aqueous processing of coconuts is shown in Table I. The recovered oil represents 90% of the initial oil content. The soluble fraction which is the major protein product of this process contains 5.1% oil and 27% protein. The protein content represents ca. 70% of the total protein of the fresh coconut. Additional data indicates that the soluble fraction contains 9% ash and 45% soluble carbohydrates.

In Table II are summarized the results obtained during the aqueous processing of peanuts for the production of

Ν	Material Balance of Various Products from the Aqueous Processing of Peanuts ^{a,b}						
Product	Сопсе	entrate pr	ocedure	Isolate procedure			
	Dry wt	Oil	Protein	Dry wt	Oil	Protein	
Oil	40.4	40.4	0	39.2	39.2	0	
Concentrate	38.6	3.2	25.1				
Isolate				27.2	2.5	24.2	
Fibrous residue			-	12.1	2.1	0.6	
Whey solids	7.2	1.1	1.6	8.4	0.4	2.1	

TABLE II

^aBased on 4.5% moisture content; 100 kg blanched peanuts contain 45.4 kg oil and 27.2 kg protein (N x 5.46).

^bColumns expressed as kg.

TABLE III

Estimated	Incomes	from Oi	l and I	Protein	from	Six	Oilseeds ^a
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Oilseed	Moisture ^b (%)	Oil ^b (%)	Oil ^c price/lb	Protein (%)	Income ^d from	
					Oil	Protein
Soybean	10	18	.13	40	\$23	\$160
Peanuts	5	48	18	28	86	112
Cottonseed	8	33	15	38	50	152
Coconut	51	35	14	зb	49	16
Sunflower seed	5	47		24b		96
Sesame	6	53	39	25	206	100

^aBased on dehulled kernels.

^bFor edible portion of oilseeds (ref. 16). Rounded to nearest percent.

^cFor crude oil, bulk, fob crushing site (ref. 17). Rounded to nearest cent. Sesame oil (refined) is New York price.

^dIncome/1000 lb of edible portion of oilseeds, assuming 100% recovery and \$40/lb of protein. Rounded to nearest dollar.

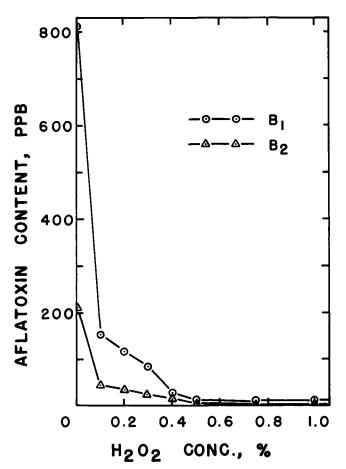


FIG. 4. Effect of hydrogen peroxide upon the detoxidation of aflatoxins during the aqueous processing of peanuts.

protein concentrates and isolates. The recoveries of free oil and protein in the concentrates are respectively 89% and 92% of the quantities originally present. The concentrate contains 65% protein ($\%N \times 5.46$) and 8% oil. For the isolate procedure, the recovery of free oil is 86% and protein in the isolates 89%. The isolate contains 89%protein and 9% oil. In either product, three washings will reduce the content of oil, sugars, and salts, as well as some proteins which are soluble at that pH, resulting in an increased percentage of protein in the remaining product.

ECONOMICS OF AQUEOUS PROCESS

If the aqueous processing of oilseeds is considered primarily as a means of recovering oil, then the efficiency of oil recovery is of primary importance. If, however, the objective is to recover both oil and protein, then the efficiency of recovery of protein also must be considered. In general, the income from the aqueous processing plant will be equal to the amount of oil produced multiplied by the price of oil plus the amount of protein product produced times the price of the protein product. The relative importance of oil and protein recoveries will, therefore, be dependent upon the relative prices of the oil and protein products and the relative amounts of each.

In Table III are given the oil and protein contents of several important oilseeds and an estimate of the potential income from the sale of the recovered oil and protein products. Price fluctuations in oil make the estimated income from oil somewhat uncertain. Furthermore the figure of \$.40/lb assumed for a protein price is speculative, inasmuch as protein obtained from different sources probably will command considerably different prices.

Nevertheless, the data indicate that, in the processing of soybeans, peanuts, and cottonseeds, the income from

protein could be more than the income from oil. Consequently, in the aqueous processing of these oilseeds for food products, the efficiency of protein recovery would become more important than that of oil recovery to the overall economics of the process.

Some preliminary estimates were made of the manufacturing costs of the soluble protein fraction from coconuts and peanuts protein concentrates and isolates. The estimates were based upon the oil and protein recovery data obtained from the pilot plant operation and other available information regarding the price of raw material, labor, equipment, interest, maintenance, administration, and miscellaneous expenses. The manufacturing costs do not include the expenses for marketing or profit.

In making the cost estimate for coconuts, oil and the soluble fraction were considered to be the main products of the aqueous process, and were assigned all costs. Based upon the 90% recovery of oil and 69% recovery of protein, it is estimated that the manufacturing costs of the soluble coconut fraction would be ca. \$.24/lb. It should be noted that any income from other products, which are anticipated as marketable commodities, would reduce the estimated cost of the soluble fraction.

For peanuts, oil and protein concentrate or isolate were considered as the main products. Based upon the price of oil stock peanuts (\$.085/lb, shelled) and 85% recovery of both oil and protein, the manufacturing costs are ca. \$.17/lb of concentrate and \$.28/lb of isolate.

ADVANTAGES AND DISADVANTAGES OF THE AQUEOUS PROCESS

At this particular point in the development of the aqueous processing technology, overall advantages and disadvantages of this alternative process over conventional oil milling cannot be assessed with certainty. Probably, the choice will often depend upon the particular properties and characteristics of the oilseed in question. Some of the potential advantages and disadvantages of the aqueous process may be summarized as follows.

Advantages

Capital investment: Preliminary estimates suggest that the smallest economic aqueous processing plant probably will be of smaller capacity than the smallest economic solvent extraction plant and may, consequently, require less initial capital investment.

Safety: Since a flammable solvent is not being used in the process, there is less fire hazard, less operational danger, and no air pollution from solvent losses.

Simplicity: Since the aqueous process is designed for simultaneous separation of oil and protein there are fewer processing steps than in conventional isolate production, i.e. solvent extraction and desolventization are eliminated, and, also, the capability of discontinuous operation.

Another advantage of aqueous processing as compared to solvent extraction, is the capability for utilization of certain chemicals to remove or inactivate undesirable substances. In the case of peanuts, hydrogen peroxide and sodium hypochlorite have proven to be effective for destruction of aflatoxins (18). The application of hydrogen peroxide at concentrations of 0.5% reduced the aflatoxin content in the concentrates and isolates to below 25 ppb from ca. 1000 ppb (Fig. 4), and the use of 0.2% sodium hypochlorite reduced aflatoxins in the products to below detectable levels (Fig. 5). Sreenivasamurthy, et al., (19) and Fischbach and Campbell (20) have demonstrated, by biological tests, the destruction of aflatoxins by hydrogen peroxide and sodium hypochlorite, respectively.

Disadvantages

The aqueous process, by nature, has some inherent

disadvantages also. Among them are: somewhat lower efficiency of oil extraction and recovery-the aqueous process recovers only ca. 95% as much oil as conventional processes-higher oil content of protein products which can cause storage stability problems, necessity of demulsification to recover oil in case of emulsion formation, and increased potential for microbial contamination because the materials undergo more processing steps while wet.

APPLICATION TO OTHER OILSEEDS

Application of the aqueous process to other oilseeds is of particular interest when evidence indicates that the solvent-extracted oilseed flour is not an acceptable food and, especially, when the chemistry of the oilseed in question seems to demand some aqueous treatment.

Two oilseeds which seem particularly good candidates for aqueous processing are sunflower seed and rapeseed. Sunflower seed meal contains sufficient caffeic and chlorogenic acids to cause it to turn dark green or brown when wetted. It has been demonstrated that these phenolic acids can be removed by aqueous extraction (21). Therefore, it ought to be possible to recover the oil and separate the protein simultaneously from the phenolic acids which cause the color problems.

Rapeseed contain toxic sulfur compounds (goitrogens) which need to be removed before the protein can be considered for food. Sosulski, et al., (22) demonstrated the successful removal of these toxic sulfur compounds by aqueous extraction. Simultaneous recovery of oil and separation of toxic sulfur compounds from protein suggests the possibility of aqueous processing being applied advantageously to rapeseed.

The application of aqueous processing to cottonseed is also an interesting possibility. Since glanded cottonseed contains pigment glands which rupture in aqueous media and react with the proteins, the application of the aqueous process seems impractical. However, the new varieties of the glandless type which are free of gossypol might be processed successfully by this method.

It also is believed that the application of aqueous processing to that king of the oilseeds, the soybean, might provide an opportunity for the removal of sugars and flavor factors which cause the problems of flatulence and offflavor which still plague this major source of plant proteins.

ACKNOWLEDGMENTS

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REFERENCES

- 1. Chayen, I.H., and D.R. Ashworth, J. Appl. Chem. 3:529 (1953).
- Sugarman, N., U.S. Pat. 2,762,820 (1956).
- Dangoumau, A., Rev. Franc. Corps Gras 5:254 (1958).
- Subrahmanyan, J., D.S. Bhatia, S.S. Kalbag, and N. Subra-4. manian, JAOCS 36:66 (1959).

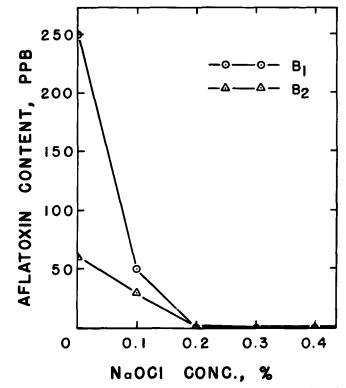


FIG. 5. Effect of sodium hypochlorite upon the detoxication of aflatoxins during the aqueous processing of peanuts.

- 5. Chayen, I.H., U.S. Pat. 2,928,821 (1960).
- 6. Rajasekharan, N., "Chemical and Technological Investigations on Coconut Products," Thesis, Banaras Hindu University, Utter Pradash, India, 1964.
- Bhatia, D.S., M.A.B. Parpia, and B.P. Baliga, J. Food Sci. Technol. (India) 3:2 (1966).
- Eapan, K.E., S.S. Kalbag, and V. Subrahmanyan, J. Amer. Chem. Soc. 43:585 (1966). 8.
- Rajasekharan, N., and A. Sreenivasan, J. Food Sci. Technol.
- (India) 4:59 (1967). Seconivasan. A., "Nutrition Document," R9/Add 5 PAG, Sreenivasan, A., 10. WHO-FAO-UNICEF, Geneva, 1963.
- 11. Smith, R.H., Adv. Chem. Series 57:133 (1966). 12. Orr, E., and D. Adair, "Tropical Products Institute Report," G-31, Ministry of Overseas Development, Great Britain, June 1967.
- 13. Hagenmaier, R.D., C.M. Cater, and K.F. Mattil, J. Food Sci. 38:516 (1973).
- 14
- Rhee, K.C., C.M. Cater, and K.F. Mattil, Ibid. 37:90 (1972). Rhee, K.C., C.M. Cater, and K.F. Mattil, Ibid. 38:126 (1973). Watt, B.K., and A.L. Merrill, "Agricultural Handbook," No. 1.5. 16. 8, Agricultural Research Service, U.S. Department of Agriculture, 1963.
- 17. U.S. Department of Agriculture, "Agricultural Statistics," U.S.
- Department of Agriculture, 1972.
 18. Rhee, K.C., K.R. Natarajan, C.M. Cater, and K.F. Mattil, J. Amer. Peanut Res. Education Assoc. 4:215 (1972).
 19. Sreenivasamurthy, V., H.A.B. Parpia, S. Srikanta, and A.S. Murti, J. Assoc. Offic. Agr. Chemists 50:350 (1967).
 20. Eventscheld Matter and Computer With a 49:00 (1967).
- Fischbach, H., and A.D. Campbell, Ibid. 48:28 (1965) 20.
- Sosulski, F.W., and C.W. McCleary, J. Food Sci. 37:253 (1972). Sosulski, F.W., F.S. Soliman, and R.S. Bhatty, Can. Inst. Food 22.
- Technol. J. 5:101 (1972).

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