## EXPRESSION OF OIL FROM DRIED FISH MEAL

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#### EXPRESSION OF OIL FROM DRIED FISH MEAL

#### CHAPTER I

#### INTRODUCTION

## A. Importance of the Problem

Expression of oil from fatty fish has been a process of commercial importance for more than a century, in the United States as well as in many other countries of the world. In the United States the Atlantic menhaden and Pacific pilchard are particularly used for this purpose, and the major part of the large catch of herring in Norway and Iceland is utilized for the manufacture of fish meal and oil.

By far the most common process of production is the one generally referred to as wet reduction. In this method the fish is cooked with steam in long cylinders through which the fish are passed continuously by a revolving screw. From the cooker the fish goes to a screw press which removes the press liquor, a mixture of oil and stick water. The latter, the aqueous fraction, contains considerable quantities of dissolved and suspended proteins, vitamins, and minerals.

The method has the advantage of being well suited for large scale production, but it has serious disadvantages. The most important one, from the nutritional as well as the economical standpoint is the loss of water soluble solids in the stick water, a loss of yield which amounts to some 20 per cent of the dry material of the fish (18, 40

p.220).

During recent years, a considerable interest has developed in improving the yield of fish meal and recovering the stick water solids. Due to advances in biochemistry and nutrition, fish meal is now recognized as an excellent source of amino acids, vitamins, and minerals, and it is used extensively in the feeding of domestic animals. At the same time the demand for marine oils has dropped, resulting in a greater effort from manufacturers to produce high quality meal and at the same time increase the yield.

Mr. S. Einarsson has a new method (18) consisting of evaporating most of the water in a specially designed evaporator as a first step in the process. The oil is removed from the meal by solvent extraction. H. Asgeirsson has suggested a use of a similar evaporator on fishing trawlers to make use of the commonly rejected fish and fish wastes which sometimes amounts to 60 or 70 per cent of the catch (2). The difficulty in utilizing these is a lack of space in most of the fishing boats, the conventional equipment is too bulky. In the above cases the oil can be removed by solvent extraction or possibly by the dry press method studied here.

A method generally known as dry rendering is frequently used for the reduction of non-fatty fish. The fish is disintegrated and dried. There is no stick water, and the meal contains essentially all the constituents of the fish with the exception of water. The method is particularly used for reduction of fillet waste from cod and haddock.

A modification of this method involves a new drier which the

Seafoods Laboratory in Astoria, Oregon, has recently been experimenting with. The drier is described in Chapter III of this thesis, "Description of Apparatus." The meal has been used in the hatchery fish-feeding experiments now being carried on by the laboratory, and has proved to be exceptionally good, as measured by weight gain of the fish being fed and low mortality.

When the dry rendering method is used, the percentage oil in the meal will be approximately four times that of the original fish, but meal which is high in oil deteriorates rapidly. It has a disagreeable odor and many animals do not eat it. Hence, when the method of dry rendering is used for fatty or semi-fatty fish, it becomes essential to lower the oil content of the dried meal. This work was done to investigate the removal of the residual oil by subjecting the dried oily meal to pressure in a hydraulic press. This would be of particular value for small plants, since solvent extraction equipment and equipment for recovering the solvent are expensive and call for a large scale operation.

# B. Object of the Investigation

It was the purpose of this work to investigate the efficiency of oil removal from dried fish meal by expression in a hydraulic press, and to study some of the variables which govern the press efficiency, and particularly the following: moisture content, applied pressure, time of exposure to the pressure (dwell time), thickness of the meal cake, temperature during expression, and meal age.

It is realized that the method of dry rendering of fatty fish is not practiced on a large scale, but it is hoped that these experiments might help by pointing to some of the difficulties in order that they might be solved in the future.

#### CHAPTER II

#### REVIEW OF LITERATURE

# A. Nutritional value of fish meal. Wet-pressed versus dry-pressed meal.

Some 25 million tons of fish are caught annually in the world, of which about ten per cent is used for the production of meal and oil (37). Fish meal forms a very valuable addition to livestock rations, and is used extensively for the feeding of domestic animals. Thus the United States used 318 thousand tons of fish meal in 1951 of which 128 thousand tons were imported. (31, p.1)

Fish meal has been found particularly valuable as a supplement in poultry feed and discoveries of vitamin  $B_{12}$  and other recent growth factors have been of special interest to fish meal manufacturers, since fish meal and fish solubles have been found to be a good source of these (7, 9, 21). These and other constituents are discussed in some detail below.

Amino acids in fish by-products. Pottinger, Harrison, and Anderson (35) in an early investigation of fish meal proteins studied the effect of manufacture on the composition of haddock fish meal proteins, and particularly on the content of tyrosine, tryptophan, and cystine, since these were thought to be most likely to be affected. The water-soluble proteins (stick water proteins) were found to be relatively devoid of these three amino acids, whereas the dry meal (press cake

meal) was higher in these amino acids. Similar data are reported by Deas and Tarr (14,15) and by Lassen (26).

Insen and associates (26) report amino acid composition of whale and sardine solubles. Thirteen amino acids were determined by a microbiological assay. The solubles were especially low in cystine, and also quite low in tryptophan and tyrosine.

The principal value of animal protein, including fish meal, is in the amino acid distribution. The critical amino acids in poultry nutrition are lysine and methionine (22). Soybean meal is low in methionine (36), and most cereals are low in lysine (bh). Tarr and associates (bh) have found that the following amino acids appear to show best correlation with chick growth: lysine, methionine, cystine, and tryptophan. All these are usually found in fish meal, whereas stick water is apt to be lacing in cystine and tryptophan as indicated above. There is some indication that high temperature drying of fish meal causes diminution of cystine. For example, Pottinger (35, p. 9-11, lh) reports slight diminution of cystine at a temperature above 100 F. Tryptophan was affected to a lesser extent, but tyrosine not at all.

It was mentioned above that soybean protein is particularly low in methionine. Ringrose and associates (36) used methionine, vitamin  $B_{12}$ , and fish meal to supplement soybean meal. The experiment was replicated five times using a variety of combinations. Growth was significantly improved with fish meal, but not with methionine or  $B_{12}$ . (See further vitamins below).

A comparison between amino-acid content of fish meal protein and stick-water protein is reported by Deas and Tarr (13). The same workers (15) compared fish meals, stick waters, condensed fish solubles, eggs, and milts for essential amino acids (arginine, histidine, iso-leucine, leucine, lysine, methionine, phenylalanine, threonine, valine, tryptophan, and tyrosine) and concluded that of these sources, fish meal was by far the best over-all source of essential amino acids.

Several sources state that fish meal protein promotes a better growth for laboratory animals than does casein of milk. Deas and Tarr (13) tell of an experiment at the Pacific station by Dr. Beveridge. White rats gained more weight per unit of feed consumed when fed on a diet containing dried fat-free flesh of various species of fish than when dried beef-flesh, casein, or egg-albumin were fed. The differences in weight gain were statistically significant. The presence of smaller amount of less digestible connective tissue in fish-flesh than in beef was suggested to be the reason for the higher nutritive value of the former, since no large differences in amino-acid content were noted. H. J. Deuel and associates at the University of Southern California (16) found that white rats gained more weight when fed dried fat-free flesh of mackerel, sardine, and tuna than when the protein was provided by comparable amounts of casein.

Vitamins. From the above discussion, it is seen that based on the amino-acid composition, stick water and condensed fish solubles are of a lower nutritive value than fish meal. However, the B-vitamins of the fish tend to be concentrated in the stick water, which is known to

be a source of thiamin, riboflavin, nicotinic acid, pantothenic acid, pyridoxin, biotin, vitamin  $B_{12}$ , and possibly one or more unidentified vitamins which are at least required by poultry (see below).

Experiments by Bakken (5) showed that "whole meal," herring meal produced by including the stick water in the press-cake, contained 10-12 micrograms per gram of ribeflavin, against 5-6 micrograms per gram in common herring meal.

The Seattle, Washington, laboratory of the U. S. Fish and Wildlife Service has analyzed many types of fish meal and condensed fish solubles for vitamins. Menhaden was found to contain 50 to 70 micrograms per gram of nicotinic acid, based on a water and oil-free basis (51). Riboflavin and nicotinic acid content of products from pilchard from a commercial plant using an airlift drier were determined by the laboratory (49, 11). On a water and oil-free basis the stick water contained 245 and 8 micrograms per gram of nicotinic acid and riboflavin respectively, whereas the meal contained only 42 and 2.6 micrograms per gram of these vitamins. A commercial meal from a plant using a flame drier (49) showed 691 and 23.7 micrograms per gram of nicotinic acid and riboflavin respectively in the stick water on oil and water-free basis. The meal contained 66 and 4.4 micrograms per gram of these vitamins. There was little or no loss of riboflavin due to flame drying of the meal but considerable loss of nicotinic acid. For example, the press-cake contained 90 micrograms per gram of nicotinic acid, but the meal 66 on a dry basis, that is approximately one-fourth of the nicotinic acid was destroyed in the flame-drying

procedure.

The VioBin process, a recent method which does not result in any stick water (27, 41) has been shown by the Seattle Laboratories of the U.S. Fish and Wildlife Service to result in considerable loss of nicotinic acid (49). The loss amounted to 40 and 46 per cent for pilchard meal and menhaden meal respectively. The process showed no destruction in riboflavin and only minor destruction in vitamin B<sub>12</sub>. The B<sub>12</sub> content was 0.75 micrograms per gram for pilchard meal, 0.46 for menhaden meal, and 0.26 for tuna meal, all based on a microbiological assay and computed to oil and water-free basis.

Two samples of commercial herring meal analyzed by Tarr and associates (43) contained 0.39 and 0.46 mg of vitamin  $B_{12}$  per pound.

A recent experiment in Norway (28) showed that 40 to 60 per cent of the total amount of vitamin  $B_{12}$ , riboflavin, nicotinic acid, and pantothenic acid was in the stick water. The importance of producing whole meal was clearly indicated by the results as shown in the following table:

Material	Weight gm	Vitamin B <sub>12</sub> mg	Riboflavin ng	Nicotinic Acid mg	Pantothenic Acid Eg
Herring Press-cake Stick water	1000 437 456	0.11 0.07 0.04	2.13 1.20 0.90	29.8 14.0 17.1	10.2 3.5 6.1
o/o vitamin in stick water		36 <b>.3</b>	lı2.lı	57.2	60.0

Tarr and associates (43) have pointed out after small scale laboratory tests that the B<sub>12</sub> content of stick water can be increased at least 15 times by growing organisms, such as <u>Streptomyces griseus</u> on the stick water. The organisms grew readily on the stick water without anything being added except a trace of a cobalt salt.

Folic acid in herring meal is reported by Biely and Tarr (8) and found to vary from 0,27 to 4.7 micrograms per gram for commercially flame-dried and low-temperature dried meal respectively. The high temperature employed in the flame-drying procedure was claimed responsible for the low folic acid content. Low growth rate in chicks occurred when commercial fish meal was the source of folic acid, whereas good growth occurred with the low temperature dried meal.

Within the recent months, a considerable interest has developed in a new vitamin or vitamins, which are particularly found in fish meal and fish solubles and required at least by poultry. R. J. Lillie and associates of the U. S. Bureau of Animal Industries announced a factor, found in fish solubles, fish meal, and meat meal (32). A concentrate was found to promote a rapid growth in young growing chicks even when fed all the known nutrients. Biely, March, and Tarr (9) report a factor, which is needed for chicks and poultry, and which is present in herring meal, but not present to any appreciable extent at least in liver meal, dried distiller's solubles or dried brewer's yeast. The factor was not identified with any of the known vitamins. The laboratories of the U. S. Fish and Wildlife Service are currently investigating these or other unknown factors in fish meal (51) as well as many other laboratories such as the Pacific Coast Stations of Canada (9).

The importance of including the stick water in the meal (obtained for instance by dry-pressing) is clearly illustrated by the following data reported by Lassen and associates (26), on vitamin content of condensed sardine solubles determined by a microbiological assay. All values are in micrograms per gram and are for solubles of 50 per cent water: thiamin 4, riboflavin 20, nicotinic acid 325, calcium pantothenate 40, folic acid (whale solubles) 0.14. The folic acid was not reported for sardine solubles. In addition 6. Sand (40, p.214) reports biotin 0.14 and pyridoxin 12.5 from an American source not stated. The B<sub>12</sub> content and newer vitamins are discussed above.

Minerals. Whole fish meal contains all the mineral content of the fish and is particularly rich in calcium and phosphorus, since these are in large quantities in bones.

When the wet-press method is used, some minerals are removed by the stick water. Lassen (26) gives the following mineral content of condensed sardine solubles at 50 per cent water basis: potassium 1.93 per cent, sodium 1.87 per cent, iron 0.025 per cent, copper 0.007 per cent, manganese 0.0004 per cent, calcium 0.087 per cent, phosphorus 0.85 per cent, magnesium 0.016 per cent, and aluminum 0.005 per cent.

There has been some question whether calcium from bones is utilized by animals. Drake and associates made comparison between utilization by humans of calcium of skim milk powder and of bone meal (17). The availability of calcium in bone meal for humans was in the same range as that of milk. For rats, the retention of calcium from bone meal was approximately 90 per cent of the retention from

whole dried milk.

General. The nutrients of fish meal are stressed above. It is particularly pointed out that the conventional method of reduction, the wet-press method, results in a great loss of nutrients, as well as yield loss which amounts to about 20 per cent. This fact has resulted in a search for different methods of making fish meal, methods which permit the removal of oil without losing important constituents. Some of these methods will now be discussed.

# B. Methods of Overcoming Loss Due to Stick Water

The above discussion of nutrients in fish meal indicates clearly the important disadvantage of the wet-press method, mainly loss of valuable nutrients. Another disadvantage is that fatty meal frequently occurs with some type of fish, when pressed wet. For example, Tornes (40, p.220) reports considerable difficulty with Norwegian summer herring.

The wet-press method is described in many books and publications (48, p.469-482; 25, p.112-120; 4, p.199-210; 10, p.3-6). In this method the fish is cooked with steam under low pressure (5 to 10 psig). The cooked mass is pressed, separating the fish into two fractions, presscake and press-liquor. The press-cake is disintegrated, dried, and sold as fish meal. The press-liquor is separated into two fractions, oil and stick water. The stick water is usually thrown away, but sometimes concentrated and marketed as condensed fish solubles, most frequently at a concentration of about 50 per cent.

The chief advantage of the method is its suitability for a large scale continuous production. Consequently it is almost solely used for large plants, such as for the reduction of the Atlantic herring of Norway, and Iceland, the large menhaden industry of the Atlantic states, the pilchard and Pacific herring of the West Coast and Japan, and the South African pilchard. The U. S. government classifies the product as by-product, although meal and oil are the only products of many of the plants, and the entire catches of the boats frequently go for reduction. In many cases it is, however, a true by-product of canneries and freezing plants.

An important advantage of the method is that it is old and well established. Manufacturers and operators are familiar with it and equipment is well standardized and easy to obtain in many countries.

The industry is said to have started in the United States by
Barker and Tallman in 1811 at Black Point Wharf, Portsmouth, Rhode
Island (19). This was the first serious effort to separate oil from
flesh. The fish was boiled in water in two iron pots until fleshy
tissues broke down and oil was released. This boiled mass was poured
into containers and weighted down by rocks placed on boards. When oil
floated it was skimmed and barreled for shipment.

The first factory to use steam was built in 1841 by John Tallman at Portsmouth, Rhode Island (19). During the last hundred years, equipment has been improved but the basic process has remained the same.

Realization of the nutritive value of stick water as well as the

fact that it contained considerable quantity of solids which could be sold for profit led to the process of condensation of stick water. At least 20 per cent of the solid content of the fish is removed by the stick water, (18 and 40, p.220). A method for the recovery of stick water, which has been used extensively, is the Sharples-Lassen process (48, p.483). The stick water is collected in a receiving tank and acidified with sulfuric acid to a pH of 4.5. It is heated to 150 F for 30 minutes to coagulate proteins and precipitate enzymes. It contains approximately five per cent solids at this stage. It is centrifuged to remove the precipitated solids and evaporated under vacuum, usually in triple effect evaporators, to a solid content of approximately 50 per cent, the volume hence reduced 90 per cent. It is marketed as condensed fish solubles in the liquid form.

condensation of stick water permits the recovery of stick water solids in a plant already equipped with the conventional wet reduction process. There are, however, important disadvantages, such as being a liquid high in water, shipping costs are high and containers expensive (18). It is very hygroscopic and most producers do not find it profitable to concentrate it more than to 50 per cent. It is also pointed out in part A above, that stick water protein is not a complete protein since it is low in some important amino acids, such as cystine, tryptophan, and tyrosine.

Feed manufacturers thus prefer to get the stick water proteins and vitamins mixed with the meal. Thus in Norway a process of mixing the stick water with the press-cake and drying them together has

recently been developed (40, p.222). This method also provides a way to recover the stick water solids in a plant already using the wet press procedure but it can hardly be considered a final solution because of high cost. Consequently, research within recent years has concentrated on an entirely new approach, that is leaving out the conventional wet press.

Sveinn S. Einarsson of the Kveldulfur Corporation in Iceland has developed a new process (18, 33). A flow sheet is reported by Einarsson (18). The method involves mixing of the raw fish with fish oil in a specially designed evaporator where most of the water is removed. The oil acts as a convection medium for the fish. Part of the oil is removed from the concentrate of meal and oil by centrifugation. The rest of the oil is extracted out of the meal by solvent, and equipment is provided for the recovery of the solvent. There is one plant in Reykjavik, Iceland, using this process. Advantages of the method are listed as well suited for large scale operation, no offensive odors from drier-vapors, and no stick water, which frequently is a nuisance, when dumped into public wastes.

The VioBin Corporation in Monticello, Illinois, has developed a solvent rendering process (27, 41). The method is based on azeotropic distillation of two immiscible liquids (27). A solvent, immiscible with water, is mixed with the ground-up fish in a special solvent cooker where solvent and water vapor are evaporated. Since the solvent is immiscible with water, the temperature is that where the sum of the vapor pressures equals the pressure of the surroundings. The miscella,

a mixture of oil and solvent is drawn from the cooker and separated. The solids are discharged, drained free of solvent, and dried in a continuous drier. Advantages of the method are listed by Levin and Lerman (27) as elimination of obnoxious odors, coagulation of ground tissue to reduce dust and fines, no loss due to stick water, nor a pollution problem. The U. S. Fish and Wildlife laboratories have shown that the method results in a considerable loss of nicotinic acid (49), a loss which amounts to some 40 per cent of the original nicotinic acid in the fish.

The Sharples Corporation has developed a centrifuge to perform the same function as the wet press, but using centrifugal force instead of pressure (4, p.211; 40, p.221-222). A liquid of oil and water is separated from the solids of the fish by centrifugation. In actual operation considerable difficulty has been experienced in using this centrifuge in place of a press (4, p.21) and it does not solve the stick water problem.

Fish meal manufacturers have for years been interested in the dry rendering method, since it enables utilization of all the dry materials in the fish. Anderson, Harrison, and Pottinger carried on extensive investigation of this method in 1935 (1). The materials used were cod and haddock waste since these were low in oil and did not need to be pressed. The formation of cake on the drier walls was particularly troublesome, since the water soluble parts contributed greatly to the stickiness (hence stick water). The investigators were able to control this by regulation of steam pressure and vacuum; high steam pressure

and high vacuum resulted in the greatest amount of cake.

Improved methods of drying have solved these problems and today the method of dry rendering is used extensively for producing fish meal from non-oily fish and fish waste, such as fillet waste from freezing plants. This is at least true for Iceland, with which the author is familiar.

A question rises whether the method can be used for oily fish, that is whether the oil can be pressed out after most of the water has been evaporated off. S. Einarsson (18) reports that he knew of Norwegian experiments after World War I on dry-pressing. The Norwegians had hopes to be able to produce meal with approximately five per cent oil.

One company in Norway uses the Norwegian Notevarp method (40, p.228). The fish is dried to 45-55 per cent water before pressing. The stick water contains 15-17 per cent solids.

Tressler (48, p.469) reports that dry-pressed oil is more highly oxidized and in general brings a lower price than oil produced by the wet reduction method.

It appears to be possible to overcome the difficulty of overheating the oil during the drying operation, by using a well designed drier such as the double drum drier described in Chapter III, of this thesis.

This work was undertaken to try to evaluate some of the factors governing the efficiency of pressing dried fish meal. It is realized that the method has not been found practical on a large scale, but it

is hoped that the results of these experiments might point to some of the difficulties in order that they may be overcome in the future.

# C. Factors of Quality for Fish Meal and Oil

In the early days, fish meal was used as a fertilizer only, but since then it has come a long way and is today an important ingredient in animal feeds (see part A).

Analysis of fish meal usually involves the following: crude protein, fat, moisture, and ash and it is sold primarily on the basis of its protein content as based on nitrogen determination and multiplied by the factor 6.25, (3, p.8-9; 47; 55; 40, p.17). In terms of the present knowledge of fish meal this is entirely unsatisfactory, but progress is handicapped by the present complexity of nutritional research and a lack of a quick practical method to determine its quality.

Realizing this, the California Hay, Grain, and Feed Association worked out a code for fish meal, the use of which is voluntary (47, 42). The code specifies: I. Texture and composition in terms of uniform grind, protein content, particle size, moisture, and fat; II. Specifications for drying; and III. Packing and shipping. The code was a timely attempt to standardize the product but is not a solution to the existing problem of determining the nutritive quality.

Bakken (5) points out a method to determine whether herring meal is "whole meal" or wet-pressed meal. This method involves determination of the content of water soluble proteins in the meal which was

found to be 17-24 gm per 100 gm of protein in whole meal as against 6-7 gm per 100 gm of common herring meal. Chemical determination of riboflavin was also found to be useful. Whole meal was found to contain 10-12 micrograms per gram as against 5-6 micrograms per gram of common herring meal.

The Pacific Fisheries Experimental Station in Vancouver, British Columbia, under the leadership of H. L. A. Tarr and the Poultry Nutrition Laboratory of the University of British Columbia, under the leadership of Jacob Biely are currently investigating the nutritive value of fish meal and solubles. Much of the work involves feeding experiments with chicks, and has been reported regularly in Progress Reports of the Pacific Coast Stations (9, k3, kk, k5). It is hoped that the work will result in a clarification of the problem.

This thesis makes no attempt to solve the existing problem of analyzing fish meal for quality, but the above discussion serves to point out the existing difficulty.

For fish oils (body-oils) there are two analyses which the producer usually performs. These are the free fatty acid content, reported as cleic, and the amount of water in the oil. G. Sand points out (40, p.16) that the buyer of oil for industrial use does not like over six per cent free fatty acid, and usually a sharp price reduction occurs at this limit. He also points out that water in the oil enhances rate of oxidation and claims 0.3 per cent water to be the critical limit.

Iodine value is frequently reported, and varies with species of

fish. For instance pilchard oil has an iodine value of 175-185 indicating a higher degree of unsaturation than herring oil with an iodine value of 120-150 (10).

There are several other analytical values which are frequently reported. Among these are (4, p.360-374) color, saponification value, ester value, unsaponifiable matter, specific gravity, acetyl value, etc.

# D. Uses of Fish Meal and Fish Oils

Fish meal is now classified as animal feed rather than as a fertilizer, although fish meal is undoubtedly still used to some extent as a fertilizer.

The value of fish meal for animal feeding is discussed above. Today it is an important constituent in the rations of chickens, turkeys, and pigs, and is used for feeding practically all domestic animals. It is usually mixed with other feed materials, such as alfalfa meal, bran, soybean meal, or other vegetable matter (48, p.487).

The Seafoods Laboratory in Astoria has found by unpublished experiments that fish meal is an excellent food for hatchery fish.

This appears to be a wide open field, when it is considered that in 1947, the hatcheries in Washington and Oregon required over 10 million pounds of food (38, p.8) and today the figure is probably much higher.

Another wide open field for fish meal appears to be as a food for mink and other fur bearing animals which are now fed essentially

on fish and fish scrap. Storage and transportation problems would be minimized by dehydrating the fish.

Fish oils have been used for a wide variety of purposes. Bailey, Carter, and Swain (4, p.273-312) give the following uses for fish oils:

I. In nutrition; (a) Human foods including canning oils, margarine, cooking fats, and shortening; (b) Medicinal oils (liver oils); (c) Feeding oils (animal feeding); II. In industry; soaps and glycerine, paints and varnishes, linoleum (floor coverings), oiled fabrics, printing inks, core oils in the steel industry, rubber manufacture, lubricants, metal-treating oils, insecticides, leather, alkyd resins, cosmetics, polishing compounds, and other miscellaneous uses.

The above list serves to illustrate the great variety of uses to which fish oils have been applied. It is thus not surprising to find that in the early days of the industry, fish meal was considered merely a by-product of the fish oil industry.

## E. The Theory of Expression

Expression is a special case of filtration (20) but where filtration is the term used for the separation of relatively thin or pumpable slurries, expression is used for mixtures which are too thick to flow readily. This may be the case of a liquid of high viscosity, low ratio of liquid to solid, or when the liquid phase is not continuous.

Expression may be defined as follows (34, p.1072):

Expression is the separation of liquid from a twophase solid-liquid system by compression of the system under conditions that permit the liquid to escape while the solid is retained between the compressing surfaces. Expression is distinguished from filtration in that the pressure is applied by movement of the retaining walls instead of by pumping the material into a fixed space.

The theory of expression is far from complete, and most of the experimental work has been with a particular material and has led to the development of empirical equations without general application (34, p.1074).

Gurnham and Mason in an attempt to fill the gap in the theory of expression as a separate unit operation (20) stated that in actual expression there are two phases to be considered:

- (1) Condition of a system at equilibrium in which there is no flow of liquid or any change with passage of time;
- (2) The condition of a system not at equilibrium, in which a flow of liquid or a decrease in volume, or both, are occurring under the influence of the applied pressure.

The above workers, considered the first condition, that is the equilibrium condition, for which the following general equation was derived:

$$\frac{\mathrm{dP}}{\mathrm{P}} = \mathrm{K} \; \left( \frac{\mathrm{d1}}{\mathrm{V}} \right)$$

which can be integrated to

$$\log P = a + \frac{b}{v}$$

where a, b, and K are constants, P pressure, and V volume.

Koo worked with the expression of vegetable oils (24). His work was with conditions not at equilibrium, that is case (2) above. Based

on the expression of seven kinds of oil seeds the following empirical formula was found to hold on a dry basis:

$$\mathbf{v} = \mathbf{c} \, \mathbf{w}_0 + \frac{\mathbf{p} \cdot \mathbf{q}_0}{\mathbf{p}^2}$$

where W is the oil yield, per cent,  $W_0$  initial oil content of material, per cent, P pressure applied,  $\theta$  pressing time, V kinematic viscosity of the oil at press temperature, z and C are constants depending on the vegetable oil tested.

The present work is related to that of Koo, in that equilibrium conditions were not considered. It is shown later in this thesis (Figures 1 and 2) that even after eight hours of pressing equilibrium was not reached.

The author has been unable to find a report on a similar work on fish meal, but various workers have reported on the expression of vegetable oils.

Beisler (6) used hydraulic presses and the Anderson Expeller for tung oil. About five per cent of the oil was carried by the cake.

Woolrich and Carpenter (55) report that hydraulic pressing, expelling by pressure expellers, solvent extraction, and releasing the oil by bacterial fermentation are the four available methods for the production of vegetable oils.

There appears to be an optimum moisture content for pressing vegetable material. Taylor (46) claims this to be seven to eight per cent for cottonseed. (Koc states (24) that the optimum varies from six to 13 per cent for various vegetable oils.

#### CHAPTER III

#### DESCRIPTION OF APPARATUS

A Carver Laboratory Press (12) was employed for the pressing of the dried fish meal. The press was capable of producing a hydraulic pressure of 16,000 pounds per square inch, obtained by a hand-operated oil-pump. The pressing surface was six by six inches.

Electric hotplates, six by six inches, were provided for heating, and were equipped with thermostatic temperature control. Mercury-inglass thermometers were located in holes in the hotplates for temperature indication.

Two pressure gauges were used, one for low pressures, for hydraulic pressure from 0 to 5000 psi, and the other for high pressures, from 0 to 16,000 psi.

Standard test cylinders from the Fred S. Carver Company (12) were used for confining the meal samples. There were three cylinders, 1-1/8 inch, 2-1/4 inch, and 3-1/2 inch in diameter, corresponding to cross sectional areas of one square inch, four square inches, and 9.6 square inches, respectively. The fish meal to be pressed was confined between the plunger and the base. Filter pads and an oil pan were used to recover the oil.

The depth of the two smallest cylinders was three inches, whereas the largest one was 7-1/2 inches deep and had numerous vertical slots on the inside giving free flow of oil to the outer grooves.

Most of the fish meal used for pressing was dried on a double-drum,

pilot-plant drier, available at the Seafoods Laboratory of the Food Technology Department at Astoria. The meal was dried on the outside of the two steam-heated drums. A short description of this drier follows.

The two drums were 8-1/2 inches in outside diameter, and 11-3/8 inches long, the outside heating area thus being 4.22 square feet. The distance between the drums was adjustable, as well as the speed of rotation.

The drums were heated on the inside by 80 psig steam, thus giving a large temperature difference between the drums and the fish meal, and leading to short-time drying.

The heat transfer coefficient was determined for the drier, using various fish meals, and at various speeds, it was found to range from 30 to 57 Btu/hr/°F/ft<sup>2</sup>. The steam economy for the drier ranged from 1.3 to 1.5 pounds of steam per pound of water evaporated.

A small rotating-drum vacuum drier was available at the laboratory. A sample of shad meal, dried in that drier, was pressed.

A few samples of commercial fish meals were also pressed. They had previously been wet-pressed, and dried in commercial hot-air driers.

For the analytical work, common laboratory equipment was used.

This included glassware, analytical balance, hot-air oven, vacuum oven for moisture determinations, Kjeldahl equipment for nitrogen determinations, centrifuge, etc.

A cylinder of dry, compressed nitrogen was on hand and nitrogen

was bubbled through the solvent-oil mixture during the evaporation of the solvent in the oil determinations.

Viscosity data were obtained on the expressed turbot oil at various temperatures. For this purpose a Stormer Viscosity Meter was used, which measures the viscosity in terms of the time it takes an immersed metal cylinder to make 100 revolutions in the solution under test, when a weight is connected to the cylinder through a gear and pulley system. The Stormer data are convertible to absolute units (poises).

#### CHAPTER IV

#### METHODS OF PROCEDURE

# A. Preparation of Samples

Various types of fish meals were processed, some of which were manufactured in the Scafoods Laboratory, using the pilot-plant equipment available, and others were obtained from the fish meal plants in the Astoria-Warrenton area.

Detailed runs were made with meal obtained from whole turbot. The fish was finely ground, first in a meat chopper and then in a disintegrator, and afterwards dried on the double-drum drier described in Chapter III, Description of Apparatus. The dried meal, which came off the drier in flakes, was run through the disintegrator, put into cans, vacuum sealed, and stored in a cool room at 33 F, samples being taken out only when needed for pressing. This meal will, on the following pages, be referred to as fresh turbot meal, and since the majority of the experimental data was obtained using this meal, it is described in somewhat more detail than the rest of the samples.

Analysis of the meal showed the following composition:

The color of the meal was relatively light, and it had a fresh fishy odor. Oil ran out of it freely immediately upon applying pressure to it. This oil had a light, almost clear color, and was quite thin

(viscosity relatively low). This oil turned brown on standing on the oil pan during the pressing.

A few expression runs were made using "four months old" turbot meal which had been prepared as follows for the purpose under study. Fresh turbot was frozen whole in September 1951. It was glazed with a coat of ice and stored at 0 F for five months or until February 1952 when it was chopped up and dried on the double-drum drier the same way as the fresh turbot described above. The dried meal was stored at room temperature in a covered container for four months (until June), when it was pressed. The expelled oil was considerably darker in color and more viscous than the one from fresh turbot previously described, but the pressed meal was only slightly darker in color than the fresh meal.

The rockfish meal, which was pressed, had been dried on the doubledrum drier and stored in an open container at room temperature for four months.

The dover sole was dried on the double-drum drier and pressed immediately after drying.

The shad meal had been dried in a pilot-plant rotary-vacuum drum drier available at the laboratory.

All the samples mentioned above were prepared in the Seafoods
Laboratory of the Food Technology Department at Astoria, Oregon. Other
samples were obtained from the fish meal plants in the AstoriaWarrenton area. These were commercially manufactured fish meals, made
by the conventional cooker and wet-press method and dried in a rotary
air drier.

The oil content of the commercial meals varied from 8.0 to 13.6 per cent. Included were scrap meal from tuma and bottom fish, scrap meal from shad and bottom fish, and sucker meal.

All the samples were finely ground in a mortar before they were pressed.

In cases where the pressings were done in the four square-inch press cylinder, the weight of the sample was 50 grams, which amounts to four pounds of meal per square foot of pressing area. This was used all through the experiment except in cases where the effect of sample size was under investigation, in that case the weight of the sample was varied.

When the effect of cylinder size was being studied, the four pounds of meal per square foot of pressing area was kept constant, and the weight of the sample was varied accordingly.

## B. Expression

As mentioned above, the four square-inch press cylinder was used for most of the work. The finely ground meal was put into the cylinder and confined between the base and the plunger. Filter pads were used to aid in removal of the oil.

The electric plates were heated until a constant temperature reading was reached. The setting of the thermostat had previously been fixed to give the desired temperature.

By pumping the press slightly, the lower plate was lifted until

the plunger touched the upper plate, and this position was held until a uniform temperature reading was secured on both plates. This usually required 10 to 15 minutes, after which the pressure was applied gradually until the desired pressure was reached as indicated by the pressure gauge. The oil pump had to be pumped intermittently during the run in order to keep the pressure at the desired level.

When the desired time interval was over, the pressure was released and the press-cake removed. The excess oil on the surface of the press-cake was wiped off with a paper towel, and the cake thickness was measured in each case.

Where the time of applied pressure was under investigation, 50 gm samples were pressed in the four square-inch press cylinder at a temperature of 78 C, and the time was varied from 10 minutes to 480 minutes (eight hours).

Where the temperature was the variable, 50 gm were pressed for three hours in the four square-inch press cylinder at a constant pressure of 1500 pounds per square inch.

For the study of the effect of moisture, the following procedure was followed. The fresh turbot meal, which had been dried to 8.6 per cent water, was adjusted to the desired water content. Different quantities of water were added to several batches to increase the water content. In order to obtain meal lower in water than the original, some meal was dried in a vacuum oven and different quantities of water added to the dry meal. Fifty gram samples were then pressed in the four square-inch press cylinder for three hours.

The influence of pressure on expression of oil was similarly investigated by subjecting the fish meal to various pressures at a constant temperature.

Variation with sample size was studied by pressing, in the four square-inch press cylinder, samples of two, four, six, and eight pounds of fresh turbot meal per square foot of pressing area.

The difference between various types of fish meals was studied by pressing 50 gm samples in the four square-inch press cylinder for three hours at 78 C.

For some indication of the effect of storage of the meal before pressing, a control experiment was run, where the canned turbot meal, described above was subjected to the same type of pressing after zero, four, 13, and 27 days of storage in the cool room.

# C. Analytical

The pressed fish meal was analyzed for oil by using a slightly modified method of the Vitamin Oil Producers Institute (53, p.11). Essentially the method was as follows:

The press-cake was broken up, finely ground in a mortar and in a Waring blender, and thoroughly mixed.

Approximately 10 gm samples were weighed to the nearest 0.01 gram into a small beaker.

An excess of pulverized, anhydrous sodium sulphate, and approximately one teaspoonful of diatomaceous earth were added, and the mass thoroughly mixed.

Four extractions were made with 30 to 40 cc of petroleum ether (Skellysolve F), stirring thoroughly and decanting after each extraction into a 250 ml volumetric flask.

Treatment was continued by transferring the residue from previous extraction to a Waring blendor and extracting four additional times with petroleum ether, grinding thoroughly each time, and decanting to the flash. The extract was then made to volume and mixed.

The extract was centrifuged to rid it of fines, small particles of meal dispersed in the solvent. In a few determinations this was accomplished by having the flasks stand over night and letting the particles settle.

A 50 ml aliquot was pipetted into a tared flask, and rendered free of solvent by heating on a steam-bath until boiling ceased. Dry nitrogen was bubbled through the solution during the evaporation of the solvent to minimize exidation of the oil. The flasks were dried in an air-oven for a few minutes, cooled in a desiccator, and weighed on an analytical balance.

The oil content was computed as per cent oil in the sample, that is.

Wt of oil in aliquot 
$$\times 250 \times 100$$
  
Wt of sample 50

All moisture determinations were done by heating in a vacuum oven for six hours and measuring the loss in weight.

The amounts of free fatty acids as oleic (FFA) in the fish oils were determined for some of the samples by using a modified method of the

American Oil Chemists' Society (30,pCa-5a-40). The samples of the oil from the meals were obtained as follows:

For the original unpressed meal, the oil was extracted out of the meal by using the same solvent and same method of extraction as for the oil determination (see above), and the FFA determined for this oil.

As mentioned above, the percentage of oil in the pressed meal was determined by weighing a small quantity of oil extracted out of a sample of meal. The FFA was then determined by titrating the residual oil in the oil flasks.

A sample of the expressed oil was taken from the oil pan (see description of apparatus) and titrated for free fatty acids. The result was compared with the FFA obtained by titrating the residual oil left in the meal after completion of the expression, as well as to that of the original unpressed meal.

Since the oil samples were very small in most cases, a five ml microburette was used for the titration. The Oil Chemists' Society suggests the use of a much larger sample. A large enough sample was in most cases unavailable, but it is believed that a suitable accuracy was obtained by using the microburette.

The indicator used for the titration was thymolphalein, since it was impossible to see the color of the phenolphthalein in the dark fish oil. A Beckman pH-meter was also used during the titration as a further aid in determining the end-point, by titrating to a pH of 9.5.

## D. Viscosity

For measuring the viscosity of the fish oil, a Stormer Viscosimeter was used (see Chapter III, Description of Apparatus).

The principle of this viscosimeter is the timing of a revolving cylinder in the solution being tested. The time it takes the cylinder to make 100 revolutions in the solution when a weight is connected to it through a system of gears and pulleys is a measure of the viscosity of the solution. Then by running a standard solution which has similar timing and known viscosity, the viscosity of the unknown oil can be found.

A 60 per cent sucrose solution was found to give similar timing as the fish oil. The temperature-viscosity relationship for the syrup was obtained from the Handbook of Chemistry and Physics (23, p.1741), and by plotting these data, along with temperature-Stormer viscosity for the syrup, the curve of viscosity versus temperature for the fish oil could be obtained.

### CHAPTER V

### RESULTS

The results of the various tests are presented in seven graphs and ten tables in this section of the thesis. They are somewhat self-explanatory, but will be discussed separately.

The term press efficiency or efficiency of oil removal is defined as the per cent of the original oil which is removed by the press, that is:

$$E = \frac{\frac{F_o}{100 - F_o} - \frac{F_1}{100 - F_1}}{\frac{F_o}{100 - F_o}} \times 100$$
 (1)

where E is the press efficiency, per cent;  $F_0$  is the oil content of the meal before pressing, per cent; and  $F_1$  is the oil content of the meal after pressing; per cent.

## A. Time

The relationship between the dwell time (time of applied pressure) and the efficiency of oil removal was investigated. The results are tabulated in Table I and plotted in Figures 1 and 2.

The first section of Table I shows the relationship for freshly dried turbot meal. This meal was easily pressed, it had a light color, and the expelled oil analyzed 5.4 per cent free fatty acids as oleic.

After three hours of pressing the very fresh meal (Table VI), the oil

TABLE I

EFFECT OF DUELL TIME ON THE EFFICIENCY OF
OIL EXPRESSION FROM FISH MEALS

Type of Meal Pressed	Time Minutes	Pressed Meal % Oil	Efficiency of Oil Removal %	Oil Expelled gn per 100 gn Meal
2 weeks old Turbot Meal Moisture 8.6%	0 30 90 180	37.5 8.97 6.59 5.92	0.0 83.6 88.2 89.5	0.0 31.3 33.0 33.5
h months old Turbot Meal Moisture 8.3%	0 10 30 60 120 180 480	35.7 20.4 18.7 17.2 13.5 12.9 10.6	0.0 54.0 58.9 62.8 72.0 72.4 78.7	0.0 19.3 21.0 22.4 25.7 25.8 28.1
4 months old Rockfish Meal Moisture 7.9%	0 30 60 120 180 240 480	20.4 17.2 14.9 13.7 13.1 12.6 11.3	0.0 19.1 31.7 38.0 41.3 43.9 50.3	0.0 3.9 6.46 7.74 8.32 8.96 10.25

Pressure 1500 psi; Temperature 78 C; & square inch press-cylinder

content of the meal was down to 5.4 per cent, corresponding to a press efficiency of 90.4 per cent.

The second part of this table shows the same kind of data for the four months old turbot meal, described in section IV, Methods of Procedure. The oil was expressed out of this meal with much more difficulty than in case of the fresh meal, possibly due to exidation or some other chemical changes in the meal or oil. The free fatty acid of the expelled oil from this meal was 8.95 per cent.

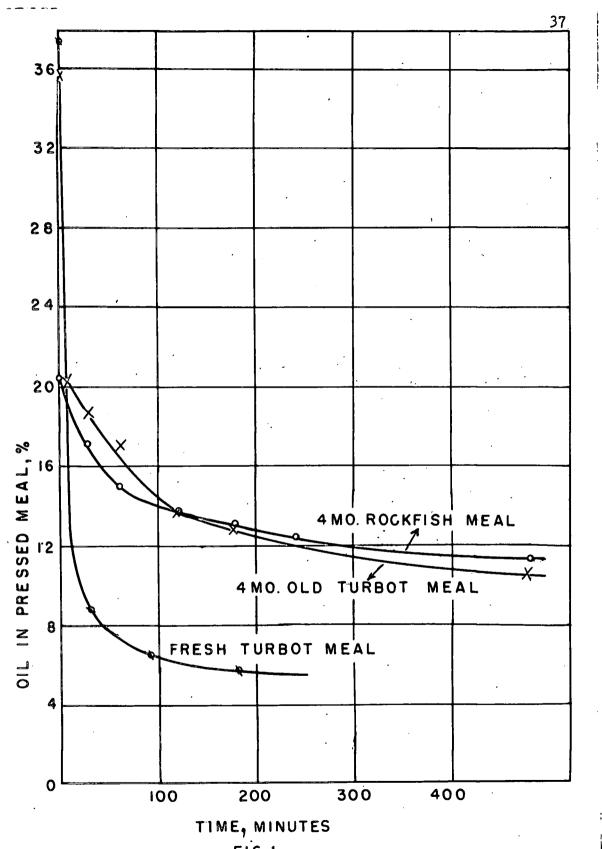


FIG.1
EFFECT OF DWELL TIME ON THE RESIDUAL OIL CONTENT OF GAKE

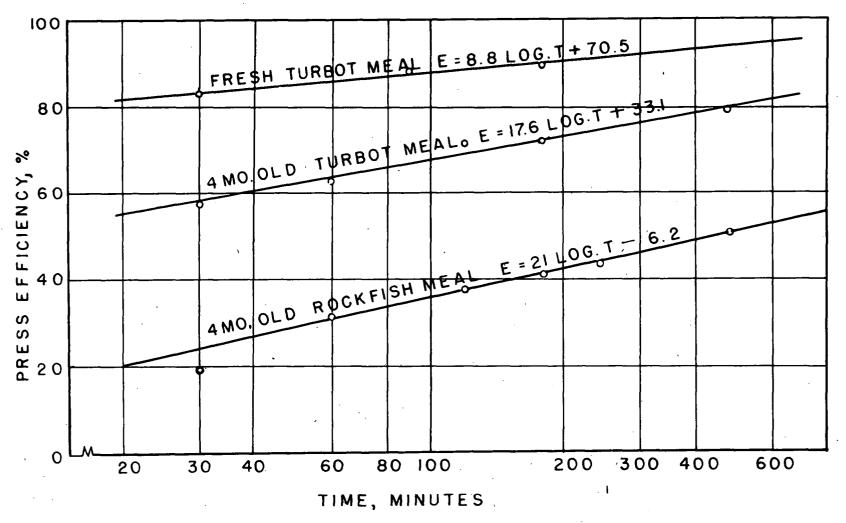


FIG. 2
SEMI-LOGARITHMIC GRAPH OF THE EFFECT OF DWELL TIME ON EFFICIENCY OF OIL EXPRESSION

Rockfish meal, four months old, was also studied for the effect of time. The residual cil content of the pressed meal followed that of the four months old turbot meal very closely. This is particularly noticeable in Figure 1, the curves for the old turbot and rockfish meals run close together, whereas the fresh turbot meal runs much lower in residual oil. It is also noted from this figure that in case of the fresh turbot most of the oil which will express out is removed in the first two or three hours, whereas in the case of the older meal the equilibrium is reached at a slower rate.

This fact is even better illustrated in Figure 2, where the press efficiency is plotted against the time on semi-logarithmic paper. The curve for the fresh turbot does not slope as much as the other two.

From the straight line functions shown in Figure 2, the equations for the press efficiency can be computed. In these equations, E represents the press efficiency, per cent, and T the dwell time in minutes. The equations thus computed are:

For the fresh turbot meal,

$$E = 8.8 \log T + 70.5$$
 (2)

For the four months old turbot meal,

$$E = 17.6 \log T + 33.1$$
 (3)

For the four months old rockfish meal,

$$E = 21.0 \log T - 6.2$$
 (4)

Since the final oil content of the four months old rockfish and turbot meals are about the same (Figure 1), the reason for the lower efficiency of rockfish meal (Figure 2) is the lower oil content of the original meal.

It should be pointed out at this point that these curves and equations (Figure 2) should only be applied within the experimental limits of this work. Thus 100 per cent efficiency can never be obtained even at infinite time, as there will always be some oil left in the meal. Similarly at zero time, the efficiency is zero, and not that indicated by equations (2), (3), and (4) above.

It is very possible that there is a limit at about five per cent oil in the press cake. Koo has shown (24) that this is approximately the case for vegetable oils. However, since feeders require fish meal to contain between five and ten per cent oil (42, 47) this is no objection.

It was attempted to apply Koo's type of equation to the data (24), but it was found not to hold as well as the type of equations shown here, particularly for short dwell times.

## B. Temperature and Viscosity

The relationship between the temperature of freshly prepared turbot meal under pressure and press efficiency was tested. The data are tabulated in Table II. It is observed that as the temperature goes up, the residual oil in the pressed meal goes down. This relationship is shown in Figure 3.

It was attempted to establish a relationship between temperature and press efficiency but no direct relationship could be established. However, the main effect of increasing temperature is to lower the viscosity of the oil. Thus in Koo's equation (24, see also review of

TABLE II

EFFECT OF TEMPERATURE ON THE EFFICIENCY OF
OIL EXPRESSION FROM TURBOT MEAL

Temperature o <sub>C</sub>	Pressed Meal & Oil	Efficiency of Oil Removal %	Oil Expelled gm per 100 gm Meal	Viscosity of Oil Centipoises
30	8.76	84.1	31.5	30.5
35	8.00	85.6	32.1	25.2
56	6.61	88.3	33.0	11.8
<b>7</b> 9	5.92	89.5	33.7	6.0
102	5.46	90.3	33.8	3.9
12h	5.24	90.9	34.1	2.9

Initial Oil 37.5%; Pressed 3 Hours; Pressure 1500 psi; h square-inch press-cylinder

literature, E. Theory of Expression), viscosity, but not temperature is a factor in press efficiency. Hence, the viscosity of the turbot oil was determined at various temperatures, and the data is tabulated in Table II.

Press efficiency versus viscosity is plotted in Figure 4 and shows a linear relationship on the semi-logarithmic paper. For this particular batch of turbot meal the relationship is as follows,

$$E = 92.5 - 5.33 \log \mu$$
 (5)

where E represents the press efficiency, and u the viscosity of the oil in centipoises. Again it is stressed that an equation of this type can safely be applied only between the experimental limits of the test.

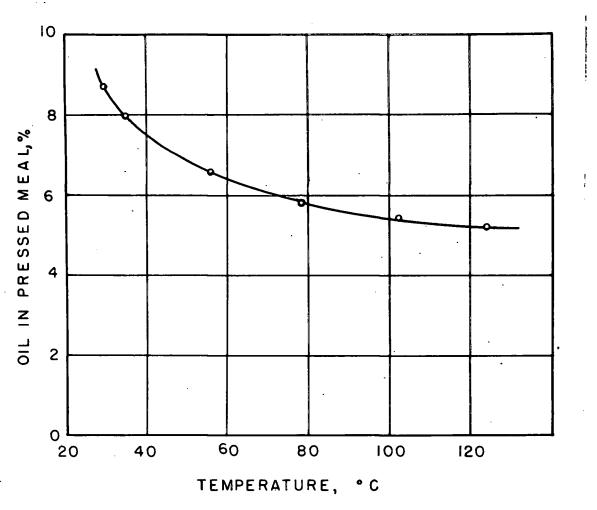


FIG. 3
EFFECT OF TEMPERATURE ON RESIDUAL OIL CONTENT OF PRESS-CAKE

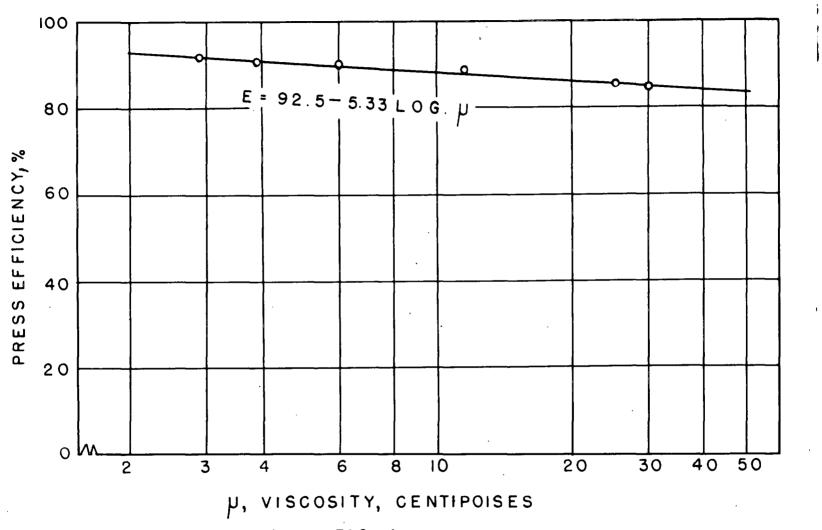


FIG. 4
SEMI-LOGARITHMIC GRAPH OF EFFECT OF OIL VISCOSITY ON EFFICIENCY OF OIL EXPRESSION FROM TURBOT MEAL

## C. Pressure

Considerable experimentation was carried out in order to attempt to obtain a relationship between the pressure applied and the press efficiency. The experimental data is reported in Table III.

Run I for freshly prepared turbot meal, run II for the same meal after two weeks of storage, and run V for four months old rockfish meal seem to indicate that increasing the pressure above 500 pounds per square inch does not result in increased press efficiency. However, run III for another batch of freshly prepared turbot meal, and run IV for a still different batch of fresh turbot meal indicate that increased pressure does result in increased press efficiency, particularly in the case of run III.

The reasons for these differences are not definitely known but several possibilities were suggested and these hypotheses tested. Run V for rockfish meal was the first experiment to be done and was followed by run I for freshly prepared turbot meal. It was thought to be a possibility that the high pressure was applied too quickly, thus restricting the flow of oil out of the meal by building up a dense press-cake too early. Run II was done to test this hypotheses. A low pressure of 500 psi was first applied for 30 minutes and then the final pressure was applied for two and one-half hours. The meal was the same as in run I but two weeks older. A pressure of 1500 psi appeared to be better than 3000 and 3500, but 3500 was better than 3000. The hypothesis was hence rejected, the effect does not seem to be a critical factor.

TABLE III

EFFECT OF PRESSURE ON THE EFFICIENCY OF
OIL EXPRESSION FOR FISH MEALS

Run	Description of Meal Pressed	Pressure on Meal Psi	Pressed Meal	Efficiency of Oil Removal \$
I	Fish Turbot 0il 37.5% N20 8.6% Pressed 3 hours in a 4 sq in. cylinder 50 gm samples	500 1000 1500 2000 3000 4000	5.53 5.73 5.41 5.61 6.85 7.25	90.3 89.9 90.4 90.0 89.0
II	Meal same as No. I 2 weeks older	1500 3000 3500	5.92 6.36 6.21	89.5 88.6 89.0
III	Fresh Turbot H2O 3.0% Oil 32.4% 60 gm samples Pressed 2 hours in a 9.6 sq in. cylinder	125 1250 2500 3750	31.01 16.55 9.35 7.87	6.67 58.8 78.7 82.4
IA	H <sub>2</sub> O 6.53% Oil 31.3% Otherwise as in III	250 1250 3750	10.23 9.82 9.13	76.կ 77.կ 79 <b>.</b> 2
V	4 months old Rockfish meal 011 20.4% H_0 7.9% Fressed 3 hours 50 gm samples 4 sq in. cylinder	125 375 750 1250 1500 3000 4000	12.05 11.85 13.37 12.80 12.92 13.10 13.30	46.6 47.7 40.0 42.7 42.3 41.3

A possibility was considered that the narrow (2-1/4 inch diameter) and solid press cylinder restricted the flow of oil out of the meal.

In order to test this hypothesis, the larger, 3-1/2 inch diameter,

press cylinder was used for runs III and IV. This cylinder had numerous slots in it giving more free flow of oil (see Chapter III, Description of Apparatus). In this test higher pressures did result in higher press efficiency.

It is thus suggested that the failure to obtain good results with high pressure in the former cases was due to large wall effects of the smaller press cylinder or te restrictions of oil-flow due to lack of slots in the wall of the smaller cylinder or both of these. However, the over-all results with pressure also indicate that the effect of pressure above a certain limit such as 1500 psi, is not as critical as the effect of some other factors such as time and temperature, particularly for meal close to its optimum water content (see below on "Effect of Moisture").

An interesting incidental fact was observed in these two runs. Run III was with meal which had been dried to three per cent water, run IV to 6.5 per cent water. The meal with higher water content showed higher press efficiency, particularly at low pressure. The effect of moisture was further tested in another experiment and is discussed in the next section.

These two runs, III and IV, are plotted in Figure 5, which is a semi-logarithmic graph of press efficiency versus pressure. The equations for press efficiency are as follows:

For run III, fresh turbot meal of three per cent water content:

$$E = 51.2 \log P - 100.4$$
 (6)

and for run IV of 6.5 per cent water

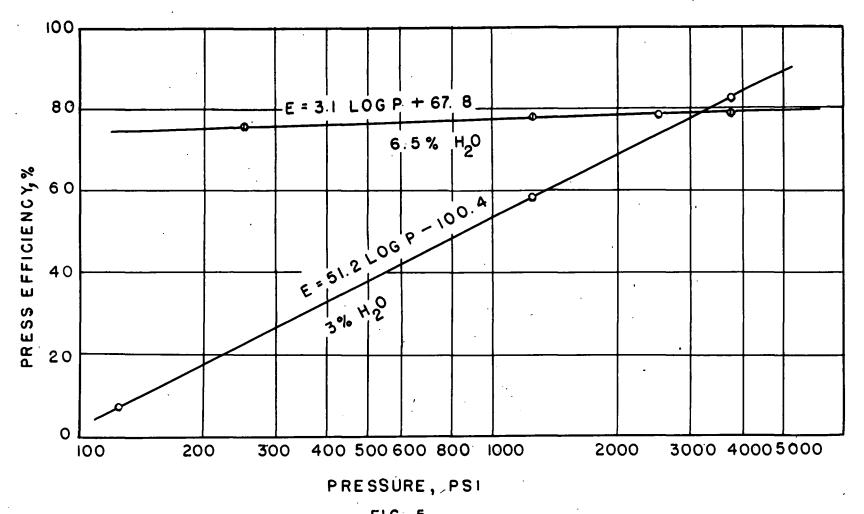


FIG. 5.

SEMI-LOGARITHMIC GRAPH OF EFFECT OF PRESSURE ON EFFICIENCY OF OIL EXPRESSION FROM TURBOT MEAL

(7)

where E is the press efficiency, per cent, and P is the pressure, pounds per square inch.

## D. Moisture

It was mentioned earlier (Review of Literature, E. Theory of Expression) that Koo (24) and Taylor (46) found that there was an optimum water content for pressing plant material for oil production. This varied from six to 13 per cent for different materials. It was also noted and discussed in connection with the tests on pressure above that the meal which had been dried to 6.5 per cent water pressed better than the meal which had been dried to 3.0 per cent water, especially at low pressures.

The effect of moisture was further tested in a separate experiment on freshly prepared turbot meal. The data are shown in Table IV. The residual oil content of the meal was computed to 8.5 per cent water, which was the water content of the original meal. These values are also tabulated in Table IV and are plotted in Figure 6, which is a plot of the residual oil in pressed meal versus water content of meal. It is observed that at low moisture low oil yield is obtained, indicating that for dry-pressing of fish meal, the meal should not be dried below five per cent water. This is particularly the case if low pressure is used (see Figure 5).

EFFECT OF MOISTURE ON THE EFFICIENCY OF OIL EXPRESSION FROM TURBOT MEAL

Moisture	Pressed Meal	Presses Meal % Oil Computed	Efficiency of	Oil Expelled gm per
B	% Oil		Oil Removal \$	100 gm of Meal
0.68	21.55	19.85	60.0	22.5
5.46	7.07	6.83	87.8	32.9
8.6	5.41	5.41	90.4	33.9
8.6	5.92	5.92	89.5	33.6
10.0	6.09	6.19	88.9	33.3
12.0	6.18	6.43	88.5	33.2
21.0	7.06	8.17	85.9	32.2

Initial Oil 37.5%; Pressed three hours; Sample of 50 grams

Temperature 78 C; Pressure 1500 psi; Pressed in a h square inch cylinder

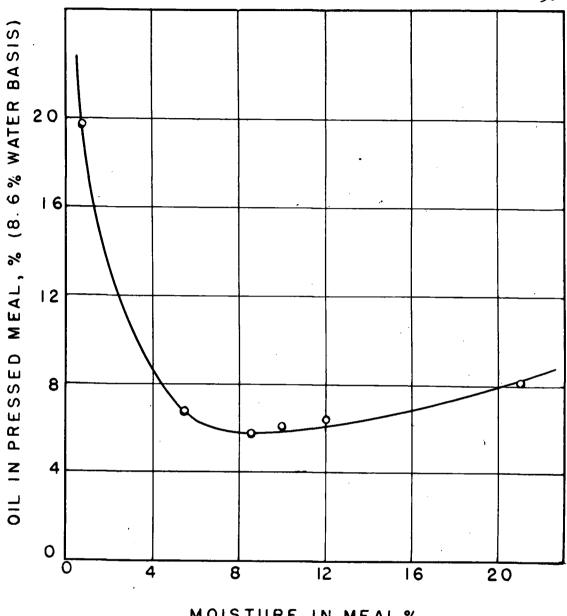
# E. Sample Size

An hypothesis, that the thinner the press-cake, the more the yield, was tested. The data are reported in Table V. Press efficiency versus sample size for the turbot meal tested is plotted in Figure 7. There appears to be a straight line relationship of the following equation,

$$E = 93.5 - 0.98 S$$
 (8)

where E is the press efficiency, per cent, and S the quantity of meal put in the press, pounds per square foot of pressing area.





MOISTURE IN MEAL,%
FIG. 6

EFFECT OF MOISTURE ON RESIDUAL OIL CONTENT OF PRESS CAKE

TABLE V

EFFECT OF SAMPLE SIZE ON THE EFFICIENCY OF OIL EXPRESSION FROM TURBOT NEAL

Sample	Sample lb/sq ft of Pressing Area	Pressed Meal % Oil	Efficiency of Oil Removal \$	Oil Expelled gm per 100 gm of Meal
25	2.0	5.03	91.3	34 <b>.1</b>
50	4.0	5.41	90.4	33.8
50	4.0	5.92	89.5	33.6
75	6.0	7.83	85.8	32 <b>.1</b>
100	8. <b>0</b>	8.01	8 <b>5.</b> 4	32 <b>.</b> 0

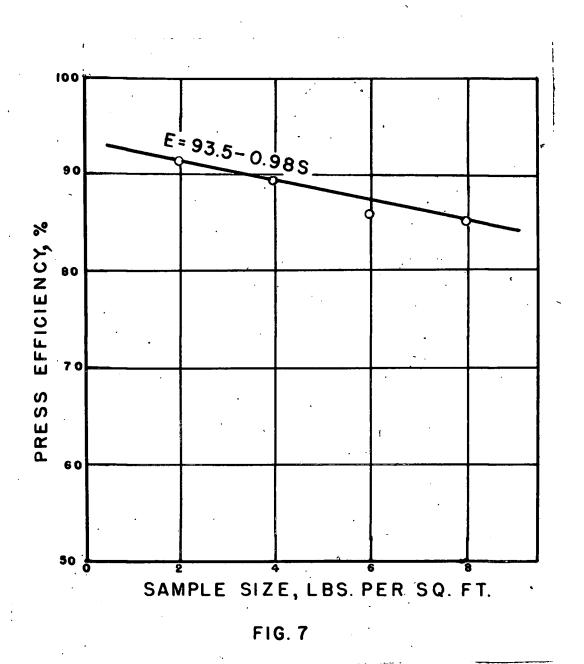
Pressure 1500 psi; Temperature 78 C; Pressing time three hours; h square inch cylinder; Initial oil 37.5%; Water 8.6%

## F. Age of the Meal

The data for the control run of "fresh" turbot meal are reported in Table VI. It is observed that as the meal ages, it becomes considerably more difficult to express the oil out of it, even if it is stored under vacuum in sealed cans in a cool room, as was the case for run I.

Run II shows the press efficiency for the four months old turbot meal, and this appears to be considerably less than that of the fresh meal in run I.

Thus it can be concluded that for best results fish meal should be pressed immediately after it is prepared.



EFFECT OF SAMPLE SIZE
ON THE EFFICIENCY OF OIL EXPRESSION FROM TURBOT MEAL

TABLE VI

EFFECT OF MEAL AGE ON THE EFFICIENCY OF
OIL EXPRESSION FROM TURBOT MEAL

<u> </u>				Oil Expelled
Run	Age of Meal	Pressed Meal	Efficiency of	gm per
- Control of the Cont	Days at 33 F	<u> % Oil</u>	Oil Removal A	100 gm of Meal
Ĭ	<u>L</u>	5.41	90.4	33.9
_	13	5.92	89.5	33.5
	27	6.45	88.4	33.1
II	4 months at			
	room temp.	12.9	72.4	25.8

Pressure 1500 psi; Sample 50 gm; 4 square inch press cylinder; Temperature 78 C; Pressed three hours;

- I. 0il 37.5%; Water 8.6%;
- II. 0il 35.7%; Water 8.3%

# G. Size of the Press Cylinder. Wall-Effect

Wall-effect in case of a small cylinder was mentioned by Gurnham and Mason (20). It was also noted during the experimentation with pressure (see above) that at high pressure the oil failed to express as well as it did at low pressure when the 2-1/4 inch press cylinder was used. A special investigation of this factor (Table VII) showed that better results are obtained with a press cylinder of large diameter. Reasons for this could be adhesion of the oil to the cylinder wall or restrictions of flow of oil out of the meal.

TABLE VII

EFFECT OF SIZE OF THE PRESS CYLINDER (WALL-EFFECT) ON THE

EFFICIENCY OF OIL EXPRESSION FOR TURBOT MEAL

Diameter of Press Cylinder Inches	Cross-Sectional Area Square Inches	Pressed Meal % Oil	Efficiency of Oil Removal %
1-1/8	1.0	12.82	<b>73.8</b>
2-1/l <sub>4</sub>	4.0	5.92	89.5
3-1/2	9.6	5.38	90.5

Temperature 78 C; Dwell time 3 hours; Pressure 1500 psi;

Original oil 37.5% Original H<sub>2</sub>O 8.6%

Sample size 4 pounds per square foot of pressing area

# H. Various Types of Fish Meal

It was thought that the type of fish used as well as the method of reduction, particularly the drying procedure, might be a factor in press efficiency. To test this, various types of fish meals were pressed, which included both laboratory prepared meals and commercially manufactured ones.

The experimental data is presented in Table VIII. The column headed pressed meal, per cent oil, probably shows best the efficiency of oil removal. It is noted that the freshly prepared turbot meal and dover sole meal which both were dried on the double drum drier, pressed well. The vacuum dried shad meal also pressed well and the commercial meal from tuna and bottom fish waste were similar, as well as the commercial meal from shad and bottom fish waste. However, the

TABLE VIII

EXPRESSION OF OIL FROM VARIOUS TYPES
OF FISH MEAL

			Original	'Original	Pressed	Efficiency
1		Type of	Moisture	Meal	Meal	of Oil
		Meal.	58	8 011	% Oil	Removal %
1 1	Dried on louble Drum	Turbot Fresh Rockfish L mo. old Dover Sole	8.6 7.9	37.5 20.4	6.36 13.1	88.6 40.7
	D D	Dover Sole Fresh	9.1	24.6	6,92	77.2
PRESSURE	Vacuum Drier	Shad Vacuum Dried	5.3	20,6	5.27	78•5
	l	Turbot	8,6	37.5	5.41	90,4
H	ble Dr Drier	Fresh Turbot 4 mo. old Rockfish 4 mo. old	8,3	35.7	12.9	72 <b>.</b> L
8	Dow	Rockfish 4 mo. old	7.9	20 <b>.</b> lı	12.92	L12.3
JRE 1500	ctally ctured	Tuna and Bottom Fish <u>a</u>	6.74	8.03	4.70	43.4
PRESSURE		Bottom Fish a Tuna and Bottom Fish b	6.57	8.27	4.58	46.8
	Commer	Shad and Bottom Fish	5 <b>.7</b> 9	12.54	5.67	58.1
		Sucker	. 7.56	, 13.6	12.72	7.24

Pressing Time 3 hours; Temperature 78 C; 4 sq in. press cylinder

commercially manufactured sucker meal showed poor results, resulting in 12.7 per cent oil in the residual meal. From these data it cannot be concluded that the method of manufacture or of drying is a factor in

press efficiency.

It is very possible that the type of fish used is a factor in press efficiency, but such a conclusion cannot be drawn from these experiments.

The effect of meal age is discussed earlier. This is very well illustrated in Table VIII.

### I. Free Fatty Acids

An hypothesis, that the oil which remained in the meal after pressing was higher in free fatty acids than the oil which was expressed, was tested. Table IX shows data for this test along with statistical analysis of the results. The oil of the original freshly prepared turbot meal contained 5.36 per cent free fatty acids. The expelled oil had a mean value of 4.99 per cent free fatty acids, and the residual oil 5.85 per cent. The difference is statistically significant at the one per cent level. Thus it is concluded that the free fatty acid content of the oil remaining is higher than that of the expelled oil. This indicates that "rancid" oil will not press out well.

It is also noted from Table IX that the free fatty acid of the expressed oil as well as that of the residual oil is well below the six per cent limit for good grade industrial oil (40, p.16). Tressler states, however, that oil thus produced is of lower quality than wetpressed oil (48, p.469).

TABLE IX
FREE FATTY ACID CONTENT OF TURBOT OIL

	FFA in Expelled Oil	FFA in Oil Left in Heal \$	
	5.26 4.89 4.31 5.43 4.95 4.75 5.37	5.33 6.39 6.39 5.90 5.27 5.97 5.96 5.59	This was from freshly dried meal prepared at 78 C for 2 hours in a 9.6 sq in. cylinder. Oil in original meal 32.4% FFA in the oil 5.35%
Sum Mean	34.96 4.99	46.80 5.85	

## ANALYSIS OF VARIANCE

Variance Due to	Sum of Squares	Degrees of Freedom	Mean Square	F
Column	2.7335	<b>1</b>	2.7335	15.94
Error	2.2292	13	0.1714769	-
Total	4.9627	14	, ., .	

F = 15.94 with 1 and 13 degrees of freedom.

This is significant at the 1% level.

Conclusion: The free fatty acid content of the expelled oil is lower than that of the oil which remains in the meal.

From Tables IX and X it is computed that there was not an increase in the free fatty acid of the oil during the pressing procedure, and in some cases there appears to be a small decrease, easily explained human errors in analysis of oil content and free fatty acids.

TABLE X

EXPRESSION OF OIL FROM COMMERCIAL WET-PRESSED FISH MEALS (CANNERY WASTE)

Meal No.	Fish	Protein %	Oil %	Water %	Ash %
	Tuna and Bottom fish				
I	waste	55.7	8.03	6.74	23.45
11	Same as I	57.4	8.27	6.57	23.85
	Shad and Bottom fish				
III	waste	55.2	12.54	5.79	22.85

B.	EXPRESSION DATA	1500 psi f	or 3 hours at	78 C	
Mea. No		Efficiency of Oil Re- moval \$	FFA of Oil in Original Meal %	FFA in Expelled Oil %	FFA of Oil in Residual Meal %
I		43.6 46.7 58.1	14.30 13.10 4.96	9.66 8.57 4.27	18.68 17.07 6.66

It is thus concluded that there was no increase in free fatty acid content of the oil during the pressing procedure of three hours at 78 C.

# J. Wet-Pressed Meal

It is interesting to note that considerable yield of oil was obtained by dry-pressing the commercially manufactured meals, which

already had been wet-pressed. The data are reported in Table VIII and in Table X, part B. The oil content of one of the commercial samples was lowered from 12.54 per cent to 5.67 per cent. The lowest value obtained in the whole experimentation was 4.58 per cent oil in the case of commercial tuna and bottom fish. The original meal contained 8.03 per cent oil.

#### CHAPTER VI

## . SUMMARY AND CONCLUSIONS

It was the object of this work to investigate oil removal from dried fish meal by expression in a hydraulic press, and to study some of the variables which govern the efficiency of oil removal.

Fish meal has been produced for over a hundred years, mostly using a process known as wet reduction. The method is well suited for large scale operation but results in a loss of nutrients as well as yield loss which amounts to about 20 per cent. Being an important ingredient in the feeding of domestic animals, it is important to include all the nutrients of the fish in the fish meal. Thus it has become important to recover the stick water solids which are a good source of nutrients, particularly vitamins.

Recent methods to avoid stick water losses involve condensation of stick water (48, p.483), mixing stick water with press-cake before drying (40, p.223), evaporation and solvent extraction (18), and a solvent cooker method (27). The method of dry rendering is commonly used for non-oily fish and fish waste, but expression of oil out of oily meal has not been practiced on a large scale.

A Carver laboratory press was employed for the pressing of dried fish meals, and standard press cylinders were used for confining the samples. Major part of the work was with laboratory-prepared whole turbot meal, but other samples were also pressed including commercially prepared wet-pressed meals. The following variables were studied:

pressure, moisture, dwell time, temperature (and oil-viscosity), meal age, and quantity of meal in the press. In addition some observations were done on the free fatty acid content, and the effect of size of press cylinder. Press efficiency was computed as the percentage of original oil which was removed by the press.

All the above variables were found to influence the press efficiency. The effect of dwell time and pressure was conveniently expressed in terms of an equation of the following type:

$$E = a \log x + b$$
,

where E is per cent press efficiency, x the independent variable (temperature or pressure), and a and b are constants, depending among other things on the condition of the meal and its original oil content. The effect of temperature was more conveniently expressed in terms of oil viscosity, and followed a relationship of the following kind:

$$E = b - a \log \mu$$

where p is the oil viscosity.

### Conclusions

- 1. Oil can be expressed out of freshly dried fish meal prepared from oily fish to give a residual fish meal which is below six per cent in oil.
- 2. A definite relationship exists between the time in which the pressure is applied and the efficiency of oil removal (press effi-

ciency). For the meals investigated this relation was of the following type:

$$E = a \log T + b$$
.

where E is per cent press efficiency, T dwell time in minutes, and a and b constants, depending among other things on the type of meal, its condition and original oil content. For fresh turbot meal a = 8.8, b = 70.5. For four months old turbot meal, a = 17.6, and b = 33.1. For four months old rockfish meal a = 21, and b = -6.2.

- 3. Increased temperature results in more yield of oil. This may be conveniently expressed in terms of oil-viscosity. The relation between oil viscosity and press efficiency for freshly prepared turbot meal was  $E = 92.5 5.3 \log \mu$ , where E is per cent press efficiency, and  $\mu$  viscosity in centipoises.
- 4. Increased pressure results in increased oil yield but only if the press cylinder is large enough to minimize wall effects.

  Moisture content of the meal also functions in the pressure-effect.

  For freshly prepared turbot meal in a 3-1/2 inch press cylinder the relation was: For meal of three per cent moisture, E= 51.2 100.4.

  For meal of 6.5 per cent moisture, E = 3.1 log P 67.8.
- 5. For good results fish meal should not be pressed when the water content is below five per cent.
- 6. The less the quantity of meal in the press, the more the oil yield. For the freshly prepared turbot meal under the conditions of this test, the relationship was E = 93.5 S, where E is press efficiency and S is pounds of fish meal per square foot of pressing

area.

- 7. The older the meal, the less the efficiency of oil removal.

  This is true for meal stored under vacuum at 33 F as well as that

  stored at room temperature with access to air.
- 8. The smaller the diameter of the press cylinder used, the less the press efficiency. This is at least true for the three press cylinders used in these tests.
- 9. Oil which is expressed out of meal is lower in free fatty acids than the residual oil, suggesting that "rancid" oil does not press out well.
- 10. There was no increase in free fatty acid content of the oil during the pressing procedure of three hours at 78 C. The oil was over five per cent in free fatty acids.

## CHAPTER VII

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