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Production of Fish Protein Concentrate
Rabat, Morocco, 3 - 12 December 1969

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by

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SUMMARY

THE HALIFAX ISOPROPANOL PROCESS FOR THE MANUFACTURE OF FISH PROTEIN CONCENTRATE [1]

by

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The Halifax process is based on the extraction of water, lipid and water solubles from oily and non-oily fish with isopropanol-water mixtures. The fish protein concentrate produced is light in color, from white to light tan, with negligible odor and flavor.

The product can contain up to 93% protein, depending upon the amount of bone allowed to remain in the product. Residual lipids are in the order of .01 to .06% for non-oily fish to 0.18% for oily fish, such as herring. The quality of the protein, as measured by the Protein Efficiency Ratio, is higher than casein and comparable with albumen.

A commercial plant to produce fish protein concentrate, using this process, is to be constructed at Canso, Nova Scotia, Canada.

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Work was started in the early 1950's at the Halifax Laboratory to develop a defatted and deodorized fish protein concentrate suitable for human consumption. Two different avenues were explored; enzymatic hydrolysis and solvent extraction. Studies on enzymatic hydrolysis lie outside the scope of this talk.

For our studies on solvent extracted fish protein concentrate it was decided to investigate the use of isopropyl alcohol. This solvent had shown promise in earlier studies on the extraction of fish roe. It has many characteristics which made it a logical choice for use in extraction of fat, water solubles, and water from fish. Its toxicity to humans is low, and it does not combine with the components in fish to form toxic compounds. Isopropanol is water soluble, readily available at relatively low cost, is easily handled, non-corrosive to equipment, and there are few government restrictions on its commercial use.

Studies were directed towards the extraction of filleting scrap to produce a tasteless and odorless product, and in the 1955-56 Annual Report of the Laboratory Dr. Guttmann described a procedure which is essentially the same as the one published in Progress Report No. 67 in 1957. The method was still essentially on a Laboratory scale.

The process was adapted to a pilot scale during 1957-1958. The essential features of the Guttmann-Vandenheuvel-Gunnarsson Process are

Slide 1 shown in Slide 1.

The FPC project was taken over by H.E. Power in 1958, and for the next two years the process was investigated in detail on a pilot plant scale. Considerable variability was experienced with the color of batches

Slide 2 of FPC (Slide 2). This variability seems to be related to season and

possibly reflected difficulty in reproducing the method with the same species at different times of the year (Slide 3).

Slide 3

The major problem lay in filtering or centrifuging the gelatinous mass which was formed when water and acid were added to the ground fish. The poor filtration characteristics of this blend were recognized by Gunnarsson but it was anticipated that centrifugation would solve the problem. Although satisfactory batches were prepared, frequent difficulties were encountered, e.g. some batches were not odorless, presumably a result of inadequate washing; processing of other batches had to be discontinued due to the centrifuge cake becoming completely impermeable to water.

Therefore, modifications to the process were indicated. In spite of the limitations of the process, quantities of quite good FPC were made available to many interested parties during this period.

The initial extraction with water was originally introduced to remove water soluble materials prior to the extraction of lipid with isopropanol. In another section of the Laboratory, Mr. Dambergs was interested in studying the protein of cod muscle as free as possible of other components. To achieve this goal he investigated various mixtures of isopropanol-water for the extraction of fat, water solubles and protein (Slide 4). It now became apparent that the isopropanol extracted the water soluble materials effectively if it contained 15-20% or more water. Fat was extracted optimally when the isopropanol contained 20-30% water. There was essentially no extraction of protein unless the water content exceeded 20% and very little up to 25-30%.

Slide 4

The solution to the problems presented by the water extraction was now apparent and from this point the first extraction was performed with 70% isopropanol. This procedure resulted in a porous mass resembling a

mixture of fine sawdust and water and completely eliminated the problems concerned with centrifugation and various odor characteristics. It also provided a means of preserving the raw material which was very susceptible to spoilage.

The Power-Dambergs improved method for preparing FPC from cod and related species is described in J. Fish. Res. Bd. Canada, 19: 1039-1045, 1962.

Slide 5

Step I: Fresh, skinned cod fillets (or other material) are ground to 1/4 inch size in a 1 1/2 hp meat grinder. Sufficient 99% isopropyl alcohol is added to give a 70:30 isopropyl alcohol-water ratio in the mixture making use of the water already contained in the muscle. This requires approximately 19 imp gal of 99% isopropyl alcohol per 100 lb of fillets. The mixture is stirred for 15 minutes in a stainless steel tank, during which time sufficient 20% phosphoric acid is added to bring the pH to 5.5. This partly hydrolyzes the connective tissue, making the collagen and gelatin more soluble. The flesh of this fillet is dehydrated and slightly denatured by the alcohol. The texture of the flesh changes from a soft paste to granular particles. This makes possible the use of a high speed comminutor for further size reduction. The alcohol-fillet mixture is then put through a Fitzpatrick comminutor fitted with a screen having 1/8 inch diameter holes.

Step II: The material is then put in a 30 gal reaction kettle and maintained at 178 to 180 F with constant agitation for 30 minutes. A glass reflux condenser is used to prevent loss of alcohol. The material is then pumped to the Bird Basket centrifuge and the liquid centrifuged off. The cake then contains 45 to 50% liquid. At this point approximately 94% of the fat and 72% of the water solubles which will be removed have been extracted. The cake is then broken up by passing it through the Fitzpatrick comminutor, this time fitted with a screen having 1/2 inch square openings.

Step III: The shredded cake is replaced in the reaction kettle with 10 gal of 70:30 isopropanol-distilled water mixture for each 100 lb of starting material. The temperature of the mixture is raised to 178 to 180 F and held there for 15 minutes with constant agitation. It is then pumped to the Bird centrifuge and the liquid removed. After this extraction approximately 97.5% of the fat and 98% of the water-soluble material which will be removed has been extracted. The cake is again shredded in the Fitzpatrick comminutor using the screen with 1/2 inch square holes.

Step IV: The material is then put in the reaction kettle with 99% isopropyl alcohol and again heated to 178 to 180 F for 15 minutes while being constantly agitated. In this extraction 10 gal of isopropyl alcohol are used per 100 lb of starting material. The slurry is then pumped to the centrifuge and the liquid centrifuged off. A further 4 gal of 99% isopropanol is then placed in the kettle and heated to 178 to 180 F and then pumped into the centrifuge in order to clear the last of the solids from the kettle, pump and lines and to wash the cake in the centrifuge. The cake is then broken up in the Fitzpatrick comminutor.

The fat content after the extraction will be less than .06% (usually from .016 to .04%) on a dry basis as measured by extraction with a methanol-chloroform mixture (Bligh and Dyer method). Extraction by ether in a Soxhlet apparatus shows a smaller fat content.

Step V: The ground cake is then dried in trays using a cabinet type dryer in which air at 100 to 110 F is blown over the trays of material. Drying takes from 24 to 48 hours depending on local weather conditions. The alcohol is removed and the moisture reduced to 4 to 6%. After drying, final grinding to flour size is performed in a Weits disintegrator using a .032 inch screen. The final product is then sealed in polyethylene bags.

The use of 99% isopropanol for the final extraction resulted in a cake which was easier to dry and eliminated the risk of spoilage when drying is done over an extended period. It is possible that acid could be eliminated providing very fresh starting material is used. However, experience suggests that the risk of odor in the finished product or subsequent flavor reversion is a very real one unless acid is used. When acid is used color and flavor have been consistently satisfactory.

The process has been modified for application to fatty species. In this instance, the isopropanol content is made as high as possible during the second extraction. Two extractions with herring reduced the fat content to 1%, and a third extraction brings this to less than 0.1%. This compares with 0.02-0.056% for lean fish. All these values are well below fat contents which have been recommended as satisfactory for the best quality FPC. FPC has now been prepared from cod fillets, filleting line scraps, cod filleting line scrap press cake, whole cod, whole eviscerated cod, eviscerated and headed cod, mature and immature herring, capelin, whole skate and whole dogfish. All these products are of satisfactory color, flavor and odor.

Air drying leaves a residual isopropyl alcohol content of approximately 1 to 1.2%; vacuum drying removes very little more of the residual alcohol. Steam stripping and redrying has been shown to reduce the level of residual solvent to 250 ppm or less.

Removal of all or part of the bone, in the raw material, before processing yields higher protein levels and low levels of fluoride in the final product.

Slide 6 The next slide shows the proximate analysis of fish protein concentrate made from various raw materials. The protein content is highest from

fillets where bone does not contribute significantly. Whole cod yields a concentrate containing 84.7% protein and trimmings contain 87%; whole herring results in 89.7% protein. The residual fat varies between 0.02% and 0.056% for lean fish to a high of only 0.18% for herring.

Slide 7

The nutritive value of the protein concentrate is high. The protein efficiency ratios (PER) of all samples are higher than the corresponding values for casein with the exception of the samples produced from press cake. Again the loss of proteinaceous material during pressing is the most probable cause of the decrease in quality of the protein of this material. The press cake cooked by the use of live steam, directly injected into the material, showed the lowest PER due to the extractive effect of the steam condensate. The highest PER's were given by the protein in the concentrate produced from cod fillets, whole herring, and whole cod. The PER's for these concentrates were 2.97, 2.74, and 2.64, respectively. Protein concentrate made from headed, eviscerated cod and cod trimmings gave intermediate values for the PER; 2.58, and 2.57, respectively. These can be compared with a value of 2.50 for casein. The samples of protein concentrate produced from cod trimming press cake gave the lowest values for the PER; 2.19 and 2.12 considerably lower than casein.

Slide 8

Available lysine is also very satisfactory. The values for available lysine are shown in the next slide (Slide 8). All samples show available lysine values of between 6.14% of the protein and 10.4% of the protein. With the exception of the protein prepared from mature female herring just prior to spawning, all values are above the minimum value of 6.5% recommended by F.A.O. in their Tentative Specifications of 1961.

The Fisheries Research Board and the Department of Trade and Industry have been co-operating to obtain the approval of the Food and Drug Directorate of fish protein concentrate as a food for human consumption in Canada. The chemical, nutritional and toxicological tests required by the Canadian Government before approval can be given have been successfully completed and a submission requesting approval has been submitted. It is expected that rulings will be given in the very near future. Fish protein concentrate has already received approval as a human food additive in the United States.

The present request for approval by the Canadian Food and Drug Directorate is based on fish protein concentrates made from four raw materials. These are: whole herring, whole capelin, and cod and haddock trimmings (that is, the remains of the eviscerated fish after the fillets have been removed. Approval will also be requested for species related to the foregoing and for fish protein concentrate made from hake on the basis of approval of this F.P.C. by the American authorities. In the future it is planned to obtain approval of fish protein concentrate made from a wider range of edible species if a species by species approval proves necessary. Some species being considered, which are not at the present time used in Canada for food purposes, are skate, dogfish, sand launce, argentines, flounder and many other underutilized species.

In view of the protein shortage evident in the world today, one facet of the adoption of fish protein concentrate as a human food is of particular importance; at the present time we are not using our marine resources in a very efficient manner. We bring large quantities of edible fish to the decks of our fishing vessels, select the species we can consume economically, and return the remainder, dead or dying, to the sea. With conversion to F.P.C. these presently unusable species can provide high quality protein which would otherwise be wasted. There are many species

of potentially edible fish we make little or no effort to catch; these include sand launce, argentines, hake and the elasmobranchs. If we are to increase the consumption of marine protein in future years, we will have to find means of using these species and other smaller fish which do not lend themselves economically to present processing practices. Exploration of new fish stocks is being carried out by Federal and Provincial Governments to determine what additional species are suitable for future exploitation.

Establishment of an F.P.C.-based industry would aid both the fisherman and the consumer. Fishing vessels bringing in both directly edible fish and fish for conversion to F.P.C. could obtain a full load more quickly, resulting in shorter trips with a consequent increase in quality of the fish caught during the early part of the voyage. As fewer boats would return with partially filled holds, the fisherman would also profit. Species presently landed for meal could also command a larger price if they could be made available in a form suitable for human consumption.

In the Federal Government there is an interdepartmental committee on fish protein concentrate to promote the effectiveness of various departments of the Government towards commercial application of the isopropanol process for making F.P.C. in Canada. Various sub-committees report to the committee in such important areas as research and marketing.

Last year, Cardinal Proteins Ltd., with head offices in Halifax, announced plans to proceed with the construction of a full scale commercial plant to manufacture fish protein concentrate at Canso, Nova Scotia. The Halifax isopropanol process will be used. It is planned that this plant will have the capacity initially to process 200 tons of

fresh fish per day; this will result in an output of 30 tons of protein concentrate per day. Raw material will be herring, cod and haddock trimmings, and edible species presently not used for human food. This plant, financed for $5 million dollars, is expected to go into production near the middle of 1970. The selling price of the product is expected to be 35 cents per pound. Scientists of the Fisheries Research Board of Canada are co-operating closely with company engineers in the design of this plant and are continuing research related to the process. It is expected that this prototype plant will be modified extensively as experience is gained.

Research is continuing on fish protein concentrate at the Halifax Laboratory. Methods have been developed for determining the fluoride remaining in F.P.C. and studies are underway to determine the fluoride content of various parts of the fish for a number of species. A gas chromatographic method has been developed to enable the residual alcohol content to be determined rapidly. Work on improving the efficiency of the extraction process is continuing and it has been shown that the isopropyl alcohol-water azeotrope, easily recovered by simple distillation, can be used effectively in the extraction process, even for fatty species such as herring. Another group is examining the possibility of producing fish protein concentrate with various physical characteristics such as water-binding and heat-coagulating ability; such an F.P.C. could be used in meat products. In fact, fish protein concentrate is not a product, but rather a variety of products, each modified to meet specific requirements. Jack Davis, the Federal Minister of Fisheries and Forestry, has compared F.P.C. to pulping in the forest industry.

A great advantage to properly packaged F.P.C. compared to conventional fish products lies in its almost indefinite shelf life under nearly any

environmental conditions. This is a very significant consideration in countries where refrigeration is at a premium. The stable nature of the product is also a significant plus feature when it comes to systematic marketing.

The plant to be constructed at Canso is expected to be the forerunner of many similar operations in Canada and in other parts of the world. In this way, our marine protein resources will be better able to make a significant contribution to solving the problems caused by the world-wide scarcity of high quality protein.