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D00375



Distr.  
LIMITED  
ID/WG.48/14  
6 January 1970  
ORIGINAL: ENGLISH

United Nations Industrial Development Organization

Joint UNIDO/FAO Expert Group Meeting on the  
Production of Fish Protein Concentrate

Rabat, Morocco, 8 - 12 December 1969

ISOBUTANOL AS SOLVENT FOR F.P.C. PRODUCTION <sup>1/</sup>

by

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id.70-020

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## INTRODUCTION.

Chile is one of the developing countries that has the problem of malnutrition. The diet of Chileans consists basically of carbohydrates and fats, with an evident deficiency of proteins.

It has been observed that the food shortage in the world cannot be solved through agricultural resources only, because of the limited arable lands available (Brody, 1965). For this reason an effort must be made to diversify the natural sources of proteins. This effort is being made throughout the world with an special emphasis on proteins of marine origin: Fish protein Concentrates (F.P.C.). The results of several of these efforts have been published by Levin (1959), Power (1962) Guttman et al. (1957), Pariser et al. (1963) and others.

Chile had a catch of 80.000 tons of hake in 1967 and 128.000 tons in 1968. Part of it was sold fresh, part frozen, and nearly half of it was reduced to fish meal for animal consumption (Servicio Agrícola y Ganadero, 1968, 1969). An attempt was made some time ago to

produce F.P.C. in an experimental plant using hake and ethanol and/or hexane as solvents (Yáñez et al. 1967a). At this time, the plant is closed and it will probably be dismantled.

The possibility of using isobutanol arose from the fact this solvent will be produced soon in Chile, while other more traditional solvents such as hexane, isopropanol, ethylene dichloride, etc., have to be imported. The present work was done only to study the possibility of using isobutanol in the production of F.P.C. and no at tempt was made to optimize the process.

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## EXPERIMENTAL.

A process was developed on a basis similar to the one given by Levin (1955, 1959). That is to say, the hake was extracted with solvent, and the water, the solvent and volatiles being continuously distilled at constant temperature. The distillate consisted of two immiscible phases: one rich in water, and the other in solvent, the latter being recirculated as reflux.

### Materials.

The raw material used was whole Chilean Hake (*Merluccius gayi*) processed no more than 20 hours after being caught. The mean composition of the hake is given in Table I. It must be pointed out that these composition showed a marked variation during the period of work (March to July). Special attention should be given to the fat content, which varied from 4% up to 22% dry basis. These figures may appear a bit high for a lean fish, but they agree with those given by Yáñez et al. (1967a).

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TABLE I

Composition of Hake (gr/100 gr. of dry material)

Batch	Protein* (%)	Ash (%)	Fat (%)
1	66.1	14.4	19.5
2	64.9	14.1	21.0
3-4	67.5	14.3	18.2
7-8	75.7	14.4	9.9

\* N<sub>x</sub>6.25

All analyses were made by AOAC methods.

When the supply of fresh fish was uncertain, the raw material was comminuted and then kept in isobutanol for not more than one week, a perfectly safe period according to the U.S. Department of Interior. It was observed that this previous treatment facilitated the extraction, preventing the formation of lumps.

The solvent used was Merck isobutanol (2-methyl-1-propanol) technical grade.



The isobutanol is partially miscible with water, a characteristic that appears favorable for several reasons:

- It permits the continuous elimination of approximately 50% of the water through distillation and decantation, saving energy and making the contact between solvent and fat progressively better.
- It permits a better cellular penetration than that obtained with completely immiscible solvents.
- It prevents loss of valuable solubles, as was pointed out by Levin (1955). Chromatographic analysis of the used solvent showed no trace of aminoacids.

Besides this property of partial miscibility, the isobutanol distills azeotropically with water at 89.2°C, considerably lower than 108°C, the boiling point of the pure solvent. This allows the extraction-distillation to be carry out at a nearly constant temperature, around 91°C.

Although 91°C may seem to be a much too high temperature for the purpose of preserving the nutritive value of the fish, studies made by Yñez et al. (1967b) showed that the nutritive value of hake was maintained, even after

being dried at 105° C.

Besides these considerations, the high boiling point of the pure solvent appears to be an advantage due to the difficulties of handling more volatile solvents.

Another point that deserves attention is the toxicity of isobutanol. Several authors have indicated that its toxicity is low (Kirk et al., 1948; Treon, 1963).

#### Procedure.

The process consists of six basic operations: washing, comminution, extraction, filtration, drying and grinding.

Approximately 2 Kgs. of fresh whole hake were washed with fresh water, then comminuted and homogenized in a 3<sup>1</sup>/<sub>4</sub> H.P. hobart comminuter-homogenizer for 5 minutes.

The fish, now in the form of a paste, was transferred to the extractor-distiller, which consisted of a 10 lt. glass flask with variable speed agitation, a reflux condenser, a distillate receiver externally cooled with water, and a 1000 Watt heating mantle with temperature regulator.

The first extraction was done at room temperature for 30 minutes and then for 4 hours at boiling temperature (89.2°-

91°C) using a ratio solvent: fish of 3:1 in weight. The extracted fish was then washed twice with cold solvent. The final fat content was 0.3% on a wet basis.

The next step was filtration. This was carried using an absolute pressure of 100 mm. Hg. and filtering through a bed of activated carbon. Drying was carried out in an agitated glass reactor externally heated with hot water at 60°-65°C and an absolute pressure of 25 mm. Hg. The operation was not very efficient and needed 18 hours to dry from 45% to 3-4%.

For the last operation, grinding, a Mikro Sampmill (hammer mill) was used.

The problem of solvent recovery was not studied in depth, but some experiments done showed that this operation is feasible. The solvent-fat solution was distilled in conventional laboratory glass equipment, using a 35 cm. height, 6 cm. diameter column packed with activated carbon. The carbon served both as an absorbent for odoriferous substances and as a packing to obtain a better rectification.

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## RESULTS.

Using the described method, a fine flour was obtained, with a light yellow-grey colour, no odor and only a slightly fishy taste.

This product showed a marked stability, therebeing no alterations after several months of storage at room temperature, packed in glass bottles with no special care. A sample that was stored for two months at 60°C in a flat dish placed in a forced circulation oven showed no change.

The process yield was 17% with no significant variations.

The composition of the F.P.C. obtained is given in Table II. A.C.A.C. (1965) methods of analysis were used. Results shown are mean figures for eight runs.

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TABLE II.

Composition of isobutanol F.P.C. (wet basis).

Protein	.....	80%
Ash	.....	16%
Fat	.....	0.3%
Volatiles	.....	4%

Biological quality was measured by the Protein Efficiency Ratio (P.E.R.), the pepsin digestibility, and the available lysine content.

P.E.R. Tests were performed following Chapman (1959), using 10 rats with a standard diet of casein as reference. Results are given in Table III, together with the digestibility in pepsin and the available lysine Carpenter's method (1960) was used for the latter.

TABLE III

Nutritive value of isobutanol F.P.C.

P.E.R.	casein	2.9
	F.P.C.	2.9
Pepsin digestibility		97.2%
Available lysine		7.5%

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**DISCUSSION.**

As a whole, it can be said that the result of this experience was positive. Isobutanol showed good properties for defatting and deodorizing, giving a product of good organoleptic and nutritive properties.

The fact that isobutanol is only partially soluble in water and that it forms a minimum point azeotrope can be considered favorable for the process.

Its high boiling point appear to be an advantage compared with more volatile solvents.

The P.E.R. value for the F.P.C. obtained resulted equal to the value of the control test made with casein, situation that can be considered very satisfactory. The values of pepsin digestibility and available lysine are satisfactory too, and the three of them are similar to the figures given by other processes (Brody, 1965, U.S. Department of Interior, Power, 1964, V&Aez et al., 1967b), and higher than the minimum values recommended by F.A.O.'s Tentative Specification for F.P.C. (1961).

For a definitive evaluation of the proposed method, further work must be done in toxicological aspects, stability during storage and process optimization, including production costs on an industrial scale.

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