EFFECT OF DRYING AND DESOLVENTIZING ON THE FUNCTIONAL PROPERTIES OF FISH PROTEIN CONCENTRATE (FPC)

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ABSTRACT

Experiments were performed to determine the effects of drying and steam desolventizing on the functional properties of fish protein concentrate (FPC). The FPC's were produced by a room temperature extraction of either red hake or menhaden with azeotropic isopropyl alcohol. FPC's thus produced contained about 36% soluble protein and, when dried at ambient temperature and pressure, showed very little loss in protein solubility. Drying the extracted wet solids at 40° to 50°, 60° to 70°, 90° to 100°, or 140° to 150°C for 30 or 120 min produced decreased protein solubility, i.e., 30.7% (40° to 50°C) to 12.5% (100° to 120°C). Emulsion stability of an FPC-water-oil system was satisfactory with all samples except those dried at 140° to 150°C.

Desolventizing dry solids or alcohol wet solids by steam stripping produced a dramatic loss in soluble protein and emulsion stability. There was also a significant darkening in color of the FPC's desolventized as wet solids as compared to FPC's desolventized as dry solids.

Food protein additives are used because of their nutritional and/or functional properties. Functional properties include solubility, dispersibility, water holding capacity, and emulsifying capacity (Johnson, 1969, 1970). FPC (fish protein concentrate) can have a range of functional properties depending upon the processing methods used. It is necessary, however, to control certain processing parameters in order to retain functionality.

Extraction of fish with IPA (isopropyl alcohol) at 20° to 30°C produces an FPC with better functional properties than extraction at 50°C (Dubrow, 1971). Similar results have been obtained by extracting chicken protein with IPA (Toledo, 1970).[2] Although low temperature extracted FPC retains a certain degree of protein solubility and emulsifying capacity, these properties may be lost during subsequent drying and desolventizing of the wet solids. Drying and desolventizing is necessary to reduce the residual IPA to 250 ppm to meet FDA (Food and Drug Administration) regulations (Federal Register, 1967). The purpose of the present studies, therefore, was to determine the effect of time and temperature of drying and desolventizing on the functional properties of FPC.

EXPERIMENT I: EFFECT OF TIME AND TEMPERATURE OF DRYING ON FUNCTIONAL PROPERTIES

MATERIALS AND METHODS

Preparation of Samples

Whole red hake (*Urophycis chuss*) were obtained from Block Island off the coast of Pt. Judith, R.I. They were iced on board the fishing vessel and then frozen at dockside. The extraction process consisted of a five-stage crosscurrent batch extraction at 22° to 27°C. The solvent to raw fish ratio was 2:1 w/w. Each

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[2] Toledo, R. T. 1970. Design data for a low temperature continuous countercurrent extraction process for protein concentrate production. Paper presented at the Institute of Food Technologists, 30th Annual Meeting, San Francisco, Calif.

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extraction stage was limited to 10 to 15 min followed by centrifugation of the solids from liquid. Under these conditions of extraction, the residual lipid in the FPC is reduced to less than 0.5%. Approximately 10 lb. from the last stage centrifuged wet solids were used for drying experiments.

To dry the wet solids, approximately 454 g of wet solids were placed in an aluminum foil dish and spread evenly to a depth of about 6.5 mm. Thermocouples were inserted into the bed of solids for temperature recording. The sample was then placed into a vacuum oven and subjected to drying temperatures of (1) 40° to 50°C, (2) 60° to 70°C, (3) 90° to 100°C, (4) 110° to 120°C, or (5) 140° to 150°C for either 30 or 120 min. Residence time was from the time the sample reached temperature. The sample, after drying, was milled in a Wiley mill[3] and passed through a 40-mesh screen. The samples were then placed into polyethylene bags for storage and subsequent analysis. A control was dried overnight at ambient temperature and pressure.

Methods of Analysis

The following properties were determined:

Salt soluble protein.—Two grams of FPC were added to 50 ml of cold 5% NaCl (in 0.02 M $NaHCO_3$) and magnetically stirred for 3 hr (Dubrow, 1971). The slurry was filtered through Whatman #1 filter paper. The filtrate was analyzed for nitrogen by Kjeldahl method (Horwitz, 1965). Protein was calculated as N × 6.25.

Emulsion stability.—Two grams of FPC were blended (Waring blender, Model #1083) in a pint-size jar with 20 ml of 5% NaCl (in 0.02 M $NaHCO_3$) for 3 min at low speed. Twenty ml of corn oil were added to the blender and the entire mixture blended for 1.5 min at low speed. Ten ml portions of the mix were then poured into three graduated test tubes. The tubes were placed in a water bath (at about 98°C) for 30 min and were then cooled in an ice water bath. Since FPC is more lipophylic than hydrophylic, measurements were taken of the volume of water separated. If oil separated at the same time, measurements were also taken of this phase. Emulsion stability was calculated as the percentage of water (total) that separated from the system.

Residual isopropyl alcohol.—Residual IPA was determined according to the method of Smith and Brown (1969).

Total volatiles.—Total volatiles were determined by placing a weighed sample in a 103°C oven overnight; cooling in a dessicator and reweighing.

RESULTS AND DISCUSSION

Table 1 shows the results of drying temperature and time upon the protein solubility of the FPC solids. The wet solids, prior to drying, had about 36.6% soluble protein. In comparison, wet solids produced by extraction at 70° to 80°C prior to drying contained only 3% soluble protein. Drying overnight under ambient conditions resulted in very little loss in solubility (36.4%). Vacuum drying at 40° to 50°C showed a 15-18% decrease in soluble protein over the ambient dried sample. Variable and unexplainable results were obtained by drying at 60° to 70°C: the soluble nitrogen was less after 30 min drying than after 120 min. Increasing the drying temperature to 90° to 100°C or to 110° to 120°C produced a further decrease in protein solubility. Drying at 140° to 150°C resulted in about a 65% decrease in solubility from the starting wet solids.

The emulsifying stability of the dried FPC's produced under the various conditions of drying, showed that all treatments, except the FPC's dried at 140° to 150°C formed stable oil:water emulsions (Table 1). Separation of oil and water occurred with the FPC's dried at 140° to 150°C.

[3] Reference to trade names does not imply endorsement by the National Marine Fisheries Service, NOAA.

TABLE 1.—Effect of drying temperature and time on the salt soluble protein and emulsifying capacity of FPC.

Temperature	Time	Kjeldahl soluble nitrogen[1]	Soluble protein (N × 6.25)		Emulsifying capacity
°C	hr	mg N/ml	% dry wt	$\bar{x}$	% water separated
Wet solids	--	1.17 ± 0.08	36.56	36.56	
Ambient	16.0	2.10 ± 0.04	36.43	36.43	0
40-50	0.5	1.80 ± 0.08	31.33		0
	2.0	1.76 ± 0.08	29.99	30.66	0
60-70	0.5	1.44 ± 0.03	24.06		0
	2.0	1.81 ± 0.11	31.46	27.76	0
90-100	0.5	1.76 ± 0.09	28.92		0
	2.0	1.62 ± 0.10	26.88	27.90	0
110-120	0.5	1.31 ± 0.04	21.64		0
	2.0	1.29 ± 0.03	21.42	21.53	--
140-150	0.5	0.84 ± 0.01	13.55		100
	1.0	0.77 ± 0.01	12.19		100
	2.0	0.73 ± 0.02	11.70	12.48	100

[1] Mean ± standard deviation.

The effects of drying times and temperatures on the residual IPA and total volatiles are shown in Table 2. The sample of FPC dried overnight, under ambient conditions, had a residual IPA content of 2.0%. The samples dried at 40° to 50°C averaged 2.76% IPA; 60° to 70°C averaged 2.62%; 90° to 100°C averaged 2.53%; 110° to 120°C averaged 2.42%; and 140° to 150° averaged 1.45%. Retention of alcohol residues of about 1 to 2% has been obtained even under prolonged drying for up to 4 hr at 70° to 80°C.

TABLE 2.—Effect of drying temperature and time on the total volatile and residual isopropyl alcohol contents of FPC.

Temperature	Time	Total volatiles	Residual isopropyl alcohol[1]	
°C	hr	%	%	
Wet solids		50.00		
Ambient	16	9.94	2.00 ± 0.09	
40-50	0.5	10.25	2.47 ± 0.09	2.76
	2.0	8.30	3.05 ± 0.17	
60-70	0.5	6.50	2.85 ± 0.13	2.62
	2.0	10.10	2.40 ± 0.14	
90-100	0.5	4.90	2.75 ± 0.19	2.53
	2.0	5.85	2.32 ± 0.13	
110-120	0.5	5.40	2.55 ± 0.06	2.42
	2.0	5.90	2.30 ± 0.14	
140-150	0.5	3.20	1.50 ± 0.14	1.45
	2.0	2.45	1.40 ± 0.18	

[1] Mean ± standard deviation and mean of the two groups combined.

In general, the total volatile content of the FPC's ranged from 5 to 10% except those dried at 140° to 150°C, which contained from 2.4 to 3.2% volatiles.

EXPERIMENT II: EFFECT OF DESOLVENTIZING ON FUNCTIONAL PROPERTIES

The results obtained from the drying tests were used to set up the second phase—desolventizing. Thus, the solids dried at ambient temperatures, which yielded the least loss in protein solubility, were used to steam desolventize. Also, the effect of desolventizing wet solids was determined. In this phase, whole menhaden (*Brevoortia tyrannus*) were used for solvent extraction because previous observations had shown this species to be subject to greater color differences than red hake.

MATERIALS AND METHODS

Preparation of Samples

Twenty pounds of whole menhaden were extracted with IPA by the same manner as described for hake in Experiment I. After extraction, one-half of the wet solids were dried at ambient temperature and pressure overnight

and were then steam desolventized. The other half was desolventized as wet solids.

Desolventizing consisted of placing approximately 454 g of either wet solids or dry solids in an autoclave and steaming (exhaust vent open) at 2 to 3 psi for 0, 5, or 10 min. Time of exposure was determined from the time when the autoclave reached pressure, which took about 2 min. After stripping, the sample was dried overnight at ambient conditions and then milled through a 40-mesh screen.

Methods of Analysis

Tests for soluble protein, emulsion stability, residual IPA, and total volatiles were as reported in the experiments on drying. Color of the samples was determined by reflectance analysis (Dubrow, 1971), using a Beckman DB Spectrophotometer with reflectance attachment. Reflectograms were obtained by scanning from 700 mμ to 390 mμ.. Analysis of color was also determined on a Hunter Color Meter, Model D25.

RESULTS AND DISCUSSION

Desolventizing Wet Solids

Table 3 shows the effect of steam desolventizing wet solids on the salt soluble proteins, emulsion stability, color, residual IPA, and total volatiles.

The protein solubility of FPC's decreased from 32.3% in the nonsteamed solids to between 8.6 and 10.5% in the steamed solids. The loss in solubility occurred during the come-up period as evidenced by the low soluble protein content of the solids from the 0 min treatment.

The loss in protein solubility was also accompanied by a loss in emulsion stability. All treatments resulted in a separation of phases; emulsion, water, and solids. About 20 to 25% of the water separated from the mixture.

The effect of steaming to remove IPA showed that after 5 min the residual alcohol was less than 250 ppm, decreasing from 30,000 ppm at 0 min to 200 ppm after 5 min and to 75.5 ppm after 10 min. The total volatiles of the FPC's were 7.8%, 5.3%, and 4.9%, respectively.

Steam desolventization of wet solids changed the color of the FPC's. A significant darkening, from a grayish tan to a grayish brown, occurred in those samples steamed for 5 and 10 min. A reflectance spectra of the 0-min and 10-min treatments is shown in Figure 1. Hunter L, a, and b values are presented in Table 3.

Desolventizing Dry Solids

Table 3 also shows the effects of steam desolventizing dry solids on protein solubility, emulsion stability, color, residual IPA, and total volatiles. The initial protein solubility, prior to treatment, was 28.2%. Although steaming the FPC for 0 min did not result in any great loss in protein solubility, the 5-min and 10-min steamed samples showed about a 57% loss in

TABLE 3.—Effect of steam desolventizing FPC wet solids and FPC dry solids on the salt soluble protein, emulsifying capacity, color, and volatile content.

Sample	Time	Salt soluble protein	Emulsifying capacity	Color			Residual isopropyl alcohol	Total volatiles
				L	a	b		
	min	*% of dry wt*	*% water separated*				*ppm*	*%*
Wet solids:								
Nondesolventized	--	32.29	--	--	--	--	--	--
	0	9.49	[1]20.0	62.02	+0.30	+11.72	30,000	7.76
	5	8.57	[1]24.3	52.94	+0.35	+11.76	200	5.29
	10	10.52	[1]20.7	50.83	+0.71	+11.22	75.5	4.91
Dry solids:								
Nondesolventized	--	28.23	0.0	--	--	--	55,000	11.44
	0	26.70	0.0	65.65	−0.60	+10.75	24,500	6.38
	5	12.71	[1] 5.0	62.12	−0.07	+12.15	1,000	5.35
	10	10.68	[1]15.0	61.71	−0.08	+12.50	367	4.91

[1] Separation into three phases: water, solids, emulsion.

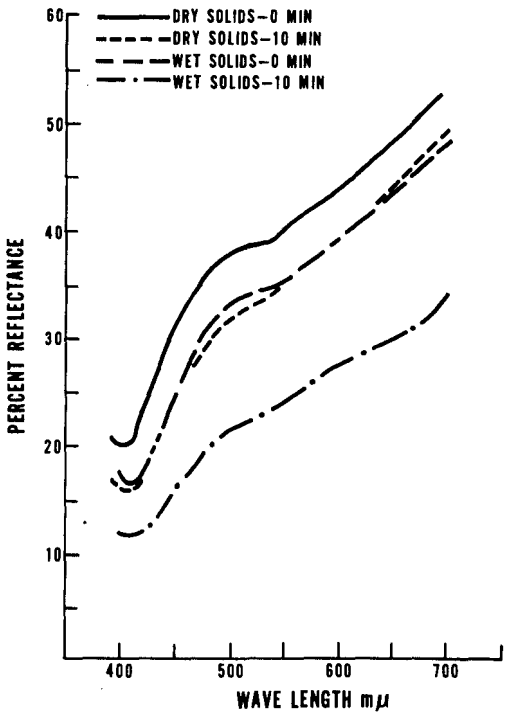

FIGURE 1.—Reflectance spectra of FPC's steam desolventized, as either dry solids or alcohol wet solids, at 2 to 3 psi for 0 or 10 min.

protein solubility as compared to the nonsteamed sample.

The emulsifying capacity and stability of the treated solids was affected in a manner similar to that for protein solubility. Both the nonsteamed and the 0-min treated samples of FPC's emulsified in oil and water systems. On the other hand, the solids steamed for 5 and 10 min showed a decrease in emulsion stability.

Steam desolventization of the dry solids reduced the residual IPA with each increment of exposure time. The initial residual content was 55,000 ppm, and after 10 min the level was found to be 367 ppm. The total volatiles of the treated solids ranged from 6.4% (0 min) to 4.9% (10 min).

The color of the FPC's after steaming showed only a slight darkening. The color changed from a grayish tan to a slight yellowish tan with respect to time of exposure. Hunter L, a, and b values are presented in Table 3. Figure 1 illustrates the reflectance spectra for the 0-min and 10-min FPC's and shows that the 10-min steamed dry solid sample was similar in its reflectance to the 0-min steamed wet solid sample; whereas the 0-min steamed dry solid was much lighter.

CONCLUSIONS

Two critical steps in the preparation of FPC by low temperature extraction with IPA are the drying and the desolventizing. Both stages involve heating: either dry or moist heat, or both. The results of this study showed that drying alcohol wet FPC solids at ambient conditions resulted in negligible loss in soluble protein and emulsifying capacity. Hot air drying of the wet solids, under vacuum, at temperatures ranging from 40° to 50°C to 110° to 120°C for 30 or 120 min resulted in a temperature dependent decrease in protein solubility. The dry FPC's, however, still retained emulsifying capacity. Under these drying conditions, the residual IPA was reduced to 2 to 3%. Higher drying temperatures of 140° to 150°C resulted in further loss of protein solubility and a complete loss in emulsifying capacity.

Removal of residual IPA, to a level of less than 250 ppm, by steam desolventization, was faster for wet solids than for dry solids. This procedure, however, brought about a 70% loss in protein solubility, a complete loss in emulsion stability, and a significant darkening of the product as compared to steam dry solids.

A similar loss in functionality, but at a slower rate and with less darkening of the FPC's, resulted from steaming dry solids.

Low temperature extraction coupled with low temperature drying produced FPC with greater functional properties than that produced by high temperature drying. To retain this functionality, methods other than steaming appear to be necessary.

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