Continuous Fractionation of Chilean Anchovy Oil With Furfural

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ABSTRACT

Useful data for liquid-liquid extraction were obtained by countercurrent fractionation of Chilean anchovy oil in an extraction column. Furfural was used as the extracting solvent while the oil was dissolved in petroleum naphtha. The data obtained by varying the velocity of agitation, the feed rate, the furfural oil ratio and the naphtha oil ratio are adequate for the design and operation of an extraction system.

INTRODUCTION

Furfural is the most practical and most commonly used of the many selective solvents which are suitable for the extraction of natural glyceride oils (1-3). Although there is an abundant bibliography on the liquid-liquid extraction of vegetable oils with furfural (4), there is little specific information on the extraction of fish oils with this solvent.

For this reason, the following four important variables in the separation were studied with the aim of obtaining a commercially valuable separation: the furfural-oil ratio in the range of 4:1 to 12:1, by weight; the reflux conditions, using naphtha in the range of 0:2 to 1:2 (naphtha-oil, by weight) as an alternative to reflux (5); the agitation in the column, as a means of obtaining a maximum efficiency, in the range of 500-900 rpm, and the feed rate in the range of 0.4-1.0 g/min of oil and naphtha combined.

Two product streams were obtained in these experiments. After distillation to remove the furfural, the bottoms extract had iodine numbers of 196-206. The overhead oil, after removal of the solvent, had iodine numbers of 105-128. This demonstrates the greater solubility of the unsaturated glycerides in the furfural stream and permits the potential use of this product in drying oils, plasticizers, paints, etc.

It was also observed that increasing the furfural-oil ratio also increased the efficiency of the extraction. At low ratios of furfural to oil, the ratio of naphtha to oil showed a marked influence in the efficiency of the extraction; the optimum ratio was 0.4:2, naphtha-oil, by weight. For higher ratios of furfural to oil, the use of naphtha lowered the efficiency of the extraction.

Varying the speed of agitation and the feed rate had no appreciable effect in the ranges studied.

EXPERIMENTAL PROCEDURES

The oil, obtained from the Compania de Industrias y Azucar de Vina del Mar, Chile (COIA), was Chilean anchovy oil that had been neutralized, bleached and winterized. Its properties are the following: 0.28% free fatty acids; trace humidity; an iodine number of 180 Wijs; a saponification number of 190 mg KOH; a density of 0.958 g/cc at 30 C; and a kinematic viscosity of 0.38 stokes at 30 C. Furfural, (Hopkin and Williams Ltd., Essex, England) was distilled at 15 mm Hg with CO_2 stripping and the 68-72 C fraction was used immediately to avoid contamination and darkening. The petroleum naphtha was 63-91 C cut. The properties of the solvent, furfural, are the following: a density of 1.159 at 20 C; a refraction index of 1.525 at 20 C; and a normal boiling point of 160-162 C.

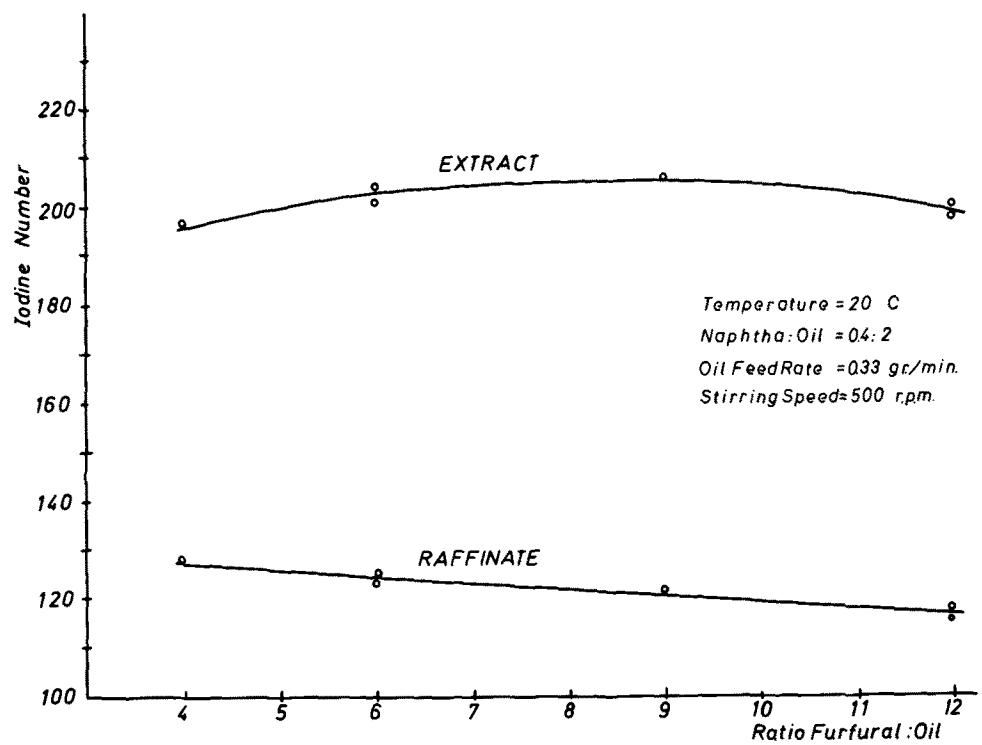

FIG. 1. Effect of furfural on extract and raffinate iodine numbers.

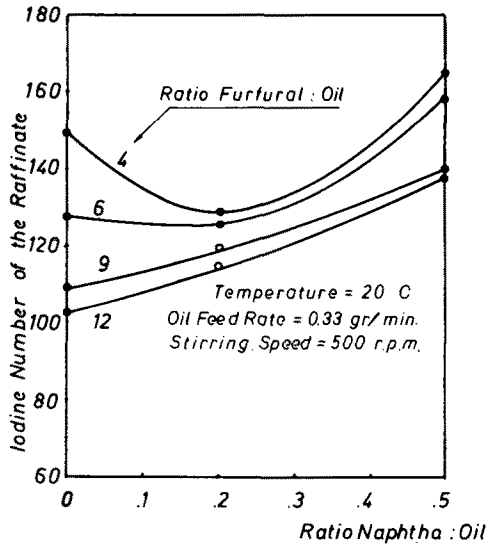

FIG. 2. Effect of both solvents on raffinate iodine number.

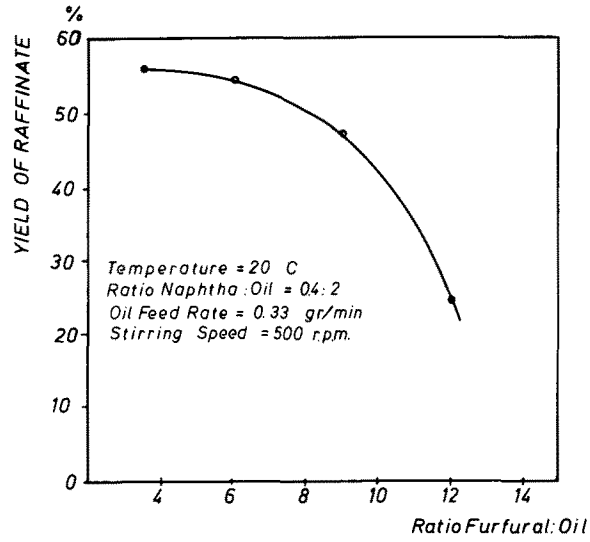

FIG. 3. Effect of furfural on raffinate yield.

Petroleum naphtha's properties are the following: a density of 68.4° A.P.I. at 20 C and a normal boiling point of 63-92 C.

The liquid-liquid extraction was carried out in an extraction apparatus model XA-1 of the York Process Equipment Corporation. The column had 11 stages and a nominal capacity of 0.6 gal/hr with an inside diameter of 1 in. and a length of 48 in. In this type of column, extraction is done in mixing zones where most of the mass transfer takes place, alternating with packed zones of calming where equilibrium is approached.

The mixing zones were 1/2 in. in height each and consisted of a stirrer connected to a central axis. The stirrer was actuated by a 1/8 HP, Duty Master monophase motor. The packed zone consisted of a stainless steel mesh and each section was 3 in. long. The feed to the column was measured by Brooks rotameters. The furfural was fed near the top of the column in ratios of 4 parts to 12 parts furfural, by weight, to 1 part oil. The oil entered near the bottom mixed with naphtha in ratios of zero to one part naphtha to two parts oil, by weight. This feed system was influenced by the two following important consideration: the phase of least velocity and highest viscosity (oil) was dispersed; attempts to disperse the furfural were unsuccessful. The oil was mixed with naphtha to provide an alternative to reflux. This increased the extraction efficiency for certain furfural-oil ratios (5).

The level of the interphase was controlled in the top of the column over the furfural feed point. The raffinate was taken from the overhead of the column and distilled at 15 mm Hg to remove the solvents. The extract at the bottom was also distilled at 15 mm Hg to remove the solvents. The distillations were done in 550 cc Quickfit laboratory equipment using CO_2 for stripping. Care was taken to keep the temperature under 100 C in the distillation process to avoid polymerization of the unsaturated compounds. This presented problems in the extract fraction where these compounds exist in greater quantities; at times a darkening of this fraction was observed.

The iodine number of the distilled fraction was measured using the Wijs method as recommended by the Association of Official Agricultural Chemists (AOAC) (6). The method was modified slightly, one-half of the quantity of each reagent recommended by the AOAC was used.

The furfural-oil ratio was studied under the following conditions: the feed rate was 0.4 g/min, oil+naphtha; the naphtha-oil ratio was 0.4:2; the temperature was 20 C; the agitation speed was 500 rpm.

RESULTS

Figure 1 presents the results of varying the furfural-oil ratio. It shows that an increasing ratio, up to 9:1, increases the extraction efficiency, i.e., increases the difference in iodine numbers of the two fractions from 68, for the 4:1 ratio, to 85, for the 9:1 through 11:1 ratios; a further increase shows an adverse effect.

Figure 2 shows the combined influence on the two solvents on the raffinate iodine number. It is observed that for low ratios of furfural to oil, there is a minimum for this iodine number, corresponding to approximately 15%, by weight, of naphtha in the feed. High furfural-oil ratios are economically undesireable because of the cost of purification of the furfural rich fraction.

Figure 3 shows the effect of the furfural in the yield of the low iodine number fraction.

Table I shows that the stirring speed and feed rate had little effect on the efficiency of extraction in the range studied. It is a summary of runs at room temperature, with ratios of furfural to oil of 6:1 and of naphtha to oil of 0.4:2.

TABLE I

Effect of Agitation Speed and Oil Feed Rate

Agitation speed, rpm	Oil+naphtha flow rate g/min	Raffinate yield, % weight	Iodine Number	
			Overhead	Bottoms
500	0.4	55.5	123	204
500	1.0	52.5	138	198
900	0.4	59.5	129	194
900	1.0	50.5	124	198

ACKNOWLEDGMENTS

Work was supported by a grant given by the Corporacion de Fomento de la Produccion. Cooperation was given by the Instituto de Fomento Pesquero.

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[Received August 25, 1970]