Fate of Jimsonweed Seed Alkaloids in Soybean Processing

G.R. LIST and **G.F. SPENCER**, Northern Regional Research Center, ARS, USDA, Peoria, Illinois 61604

ABSTRACT

The fate of Jimsonweed seed alkaloids (atropine and scopolamine) during solvent extraction of contaminated soybeans and alkali refining of crude oil was investigated. Extraction of a 50:50 mixture of soybeans and Jimsonweed seeds with petroleum ether yielded meal and crude oil fractions, in which chemical analyses showed that vitrually all the atropine and scopolamine remained in the meal. Alkali refining effectively removed atropine added to crude soybean oil.

INTRODUCTION

Jimsonweed (*Datura stramonium* L.) contains the alkaloids atropine and scopolamine, both of which are toxic to man and domestic animals. Animals are rarely poisoned from eating the green plant because of strong odor and unpleasant taste. However, eating hay containing large quantities of the dried plant does poison livestock (1). Although rare, ingestion of Jimsonweed seeds can be fatal to humans (2).

Jimsonweed plants prefer rich soil and sometimes grow in cultivated fields, barnyards, and hog lots (1). Compared to soybeans, Jimsonweed seeds are small and are easily separated by mechanical screening at the elevator. Furthermore, firsthand observations at domestic oil mills indicate that Jimsonweed contaimination is extremely unlikely in the United States because of the rigorous screening of dirt and small stones required to avoid damage to cracking and flaking rolls.

Nonetheless, over the past few years, the Northern Laboratory has had numerous inquiries about the fate of Jimsonweed alkaloids during processing of soybeans into edible oil and meal. We report here some preliminary observations on the distribution of Jimsonweed seed alkaloids during simulated processing conditions.

EXPERIMENTAL PROCEDURES

Jimsonweed seeds and soybeans (Amsoy Certified Seeds, 1971 crop year) came from reliable sources. Jimsonweed seeds (Exp 1) were finely ground through a Wiley mill. The soybeans were dehulled and flaked in pilot-plant equipment. A mixture of equal portions (10.0 g each of the prepared seed meal was extracted with petroleum ether (30-60 C fraction) for 6 hr in a small Soxhlet apparatus. Removal of the solvent on a rotating evaporator yielded a crude oil fraction weighing 4.68 g; the meal weighed 14.43 g after drying overnight at room temperature.

Recovery of atropine (Exp 2) from crude oil was determined by analysis of spiked samples prepared as follows: Atropine (100.1 mg, K and K Laboratories) was dissolved in 100 ml diethyl ether. From 1 to 10 ml of the atropine stock solution was added to 10-g portions of a commercially extracted crude soybean oil. Before analysis, the solvent was removed on a rotating evaporator.

Another portion (200 g) of the commercial crude oil was spiked with atropine (200 mg in diethyl ether). After removal of the solvent, the oil was separated into equal 100-g lots. One lot was reserved for determination of atropine. The other was refined with 14° Bé alkali at an excess of 0.5% (3). After removal of the gums and soapstock by centrifugation, the oil was washed with water (20% by wt) at room temperature. The refined and washed oil was recovered by centrifugation and dried on a rotating evaporator.

Analyses for atropine and scopolamine in meal were conducted as follows. The meal (14.43 g) from Exp 1 was wrapped in filter paper and soaked overnight (room temperature) in 100 ml of a mixture of diethyl ether, ethanol, and ammonium hydroxide (20:10:8). The meal and the solution were transferred to a Butt apparatus and extracted 6 hr with additional diethyl ether. The resulting solution was placed in a separatory funnel and extracted five times with 25 ml 5% aqueous tartaric acid. The combined tartaric acid extracts were adjusted to pH 10 with ammonium hydroxide and then extracted with five 25-ml portions of dichloromethane. After the combined dichloromethane extracts were brought to 10 ml in a volumetric flask, 2 ml was removed and an internal standard (1 mg/ml ethyl arachidate in dichloromethane) was added. The volume was reduced under nitrogen to ca. 0.25 ml. Approximately 200 µl of Regisil RC-1 [bis(trimethylsilyl)-trifluoracetamide] was added and the solution allowed to stand at ca. 50 C for 15 min before analysis. The silylated samples were analyzed by gas liquid chromatography (GLC) on a 4 ft x 1/4 in. glass column packed with 5% Apiezon L on Chromosorb W. The column oven was temperature programmed from 100 to 200 C at 5 C/min. The percentage of alkaloids was obtained by the internal standard method (4) directly from a program written for an 1800 IBM computer connected on line to the GLC instrument.

Alkaloids were determined in oils (Exp 2) by dissolving the oils in diethyl ether and extracting with tartaric acid. The remainder of the procedure was identical to the one described for analysis of the meal alkaloids.

RESULTS AND DISCUSSION

Exp 1 was intended to ascertain whether the initial soybean processing step—namely, extraction of the meal with solvent—would carry the alkaloids into the crude oil fraction or would they reside in the defatted meal (Table I). The whole Jimsonweed seed contained 0.31% atropine and 0.11% scopolamine. Theoretically, a 10 g sample of Jimsonweed seeds would yield 42 mg of alkaloids available for distribution within the oil and meal fractions, but only 37.4 mg of alkaloids were accounted for in defatted Jimsonweed-soybean meal (Table I), whereas virtually no alkaloids were found in the crude soybean-Jimsonweed oil. It is estimated that the error involved in these determinations is ±2 mg. Thus, the recovery of alkaloids is of the order of 90%.

Exp 2 was performed to determine the effect of alkali refining on traces of atropine carried into the crude oil during solvent extraction (Table I). Studies made on samples spiked with atropine showed excellent recoveries and attest to the reliability of the analytical method. More importantly, > 93% of the atropine was removed by alkali refining and washing. Lack of a reliable sample of scopolamine prevented similar studies with this alkaloid, but there is no reason to believe it would not be removed during alkali refining. The removal of atropine from crude oil by alkali refining is not surprising since atropine and scopolamine undergo hydrolysis in the presence of a base (5). Because alkaloid hydrolysis products are apparently more soluble in water than in oil, they are removed during refining and washing.

Admittedly, Exp 1 and 2 do not entirely simulate com-

TABLE I

Distribution of Alkaloids in Jimsonweed and Soybean Products

Product and weight (g)	Material balance			
	Atropine (mg)			Scopolamine (mg)
	Experiment 1			
Whole Jimsonweed seed	10.00	31.0		11.0
50:50 Mixture soybean and Jimsonweed meal–defatted	14.43	28.2		9.2
Crude soybean and Jimsonweed oil	4.68	0.04		Trace
	Experiment 2			
		Atropine (mg)		
		Theory	Found	% Removed
Crude oil spiked with:				
Atropine	2.00	2.02	2.10	---
Atropine	4.1	0.41	0.48	---
Atropine	2.00	2.00	1.95	---
Atropine–alkali refined and washed	3.00	3.00	0.20	93.3

mercial processing since the Jimsonweed seeds were neither flaked nor dehulled. Some variation might be expected depending on how the Jimsonweed seeds were ground. Furthermore, extraction in a Soxhlet apparatus does not entirely simulate the extraction of soybean flakes in a commercial oil processing plant.

Unfortunately, the sensitivity of the analytical method hampered the conduct of "real life" experiments involving the distribution of alkaloids at lower levels of Jimsonweed contamination. Much of the experimental design was dictated by the limited quantities of Jimsonweed seed available and the inherent difficulties in determining milligram amounts of alkaloids in plant materials. Nonetheless, these preliminary studies have indicated that the Jimsonweed problem, if any, resides with the meal and not with the oil fraction.

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REFERENCES

1. Morton, J.F., "Plants Poisonous to People in Florida and Other Warm Areas," Hurricane House, Miami, FL, 1971, pp. 56-57.
2. Kingsbury, John M., "Poisonous Plants of the United States and Canada," Prentice Hall, Inc., Englewood Cliffs, NJ, 1964, p. 278.
3. Bailey, A.E., "Industrial Oil and Fat Products," 3rd Edition, Interscience Publishers, New York, HY, 1964, p. 740.
4. Dal Nogare, S., and R.S. Juvet, Jr., "Gas-Liquid Chromatography," Interscience Publishers, New York, NY, 1965, p. 256.
5. Polesuk, J., and T. Sima, Mikrochim. Acta [Wien] 1973:393.

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