bearing carboxyl groups (Steiner and Holtzem, 1955), it is suggested that the titrimetric method described could be used for the determination of saponins from other sources as well.

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PRO-XAN Process: Air Drying of Alfalfa Leaf Protein Concentrate

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Several methods of drying alfalfa leaf protein concentrate (LPC) for use as a pigmenter in poultry rations were investigated. Both a blend-back and a single pass feed system were developed for use with a pilot model of a commercially available rotary air drier. The dark green granular products retain most of the carotenes and xanthophylls originally present and are suitable for use as feed ingredients.

Although a number of methods for the preparation of leaf protein concentrate (LPC) have been described (Cowlishaw *et al.*, 1956; Morrison and Pirie, 1961), it has proven difficult to dry the resultant product economically and still preserve its nutrient content. Because of the need for minimum product alteration and convenience for overseas shipment, much of the experimental product prepared at Rothamsted has been freeze-dried (Pirie, 1969).

Subba Rau and Singh (1970) have observed no differences in the nutritional quality of LPC dried by a number of different methods, including air drying. Air drying is more economical (Arkcoll, 1969) but produces a hard granular material (Morrison and Pirie, 1961). Arkcoll (1969), in laboratory experiments, reported that LPC in the form of a fine powder suitable for food use can be produced by air drying in two steps, provided that a grinding step is inserted between them.

The PRO-XAN process was developed at this laboratory as a feasible commercial method for the production of alfalfa LPC for use in animal feeds (Booth *et al.*, 1972; Halloran, 1972; Kuzmicky *et al.*, 1972). Since LPC is high in xanthophyll and low in fiber, it is useful as a pigmenter in high energy poultry rations (Halloran *et al.*, 1971; Kohler *et al.*, 1968). Previous papers have described the crushing and expression of juice from fresh alfalfa (Knuckles *et al.*, 1970, 1972), the heat coagulation of the protein xanthophyll complex (Spencer *et al.*, 1970, 1971), and the separation of the coagulum from the alfalfa solubles (Lazar *et al.*, 1971). This paper compares several methods for drying LPC and evaluates the effects of drying on the carotenoid content of the products. It should be pointed out that an important difference between the products used in our work and some of the work cited in the literature is that our product contains substantial amounts of soluble solids. LPC designed for edible use is ordinarily washed to reduce the soluble solids content to 1% or less.

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Preparation of Coagulum. Alfalfa juice, prepared as described earlier (Knuckles *et al.*, 1970), was processed within 3 hr after rolling. Coagulation with steam followed the earlier process (Spencer *et al.*, 1971) except that no air was introduced. Wet coagulum collected immediately after separation from the brown juice contains about 85% water. Drained coagulum, containing about 80% water, was collected from the draining reel (Lazar *et al.*, 1971). Dejuiced coagulum was obtained by mechanical expression as described later, and contains about 55% water.

Freeze Drying. Wet coagulum was frozen in thin layers and dried in a RePP model 15 FFD sublimator at a shelf temperature of +75°F.

Spray Drying. A gas fired, cocurrent flow spray drier (Bowen Engineering Co.) was used. The outer conical shell of the drier was 2.5 ft at maximum diameter by 5.5 ft high. Prior to drying, the coagulum was put through a Waring Blendor and a Charlotte colloid mill to produce a smooth creamy liquid. The liquid was introduced into the drier through a two-fluid nozzle by a positive displacement gear pump at a rate of 120 ml/min. Air at 65 psig was used at the nozzle for atomization. The drier was operated at an inlet temperature of 450°F, and an outlet temperature of 210–240°F.

Drum Drying. A steam-heated Buflovak pilot plant model

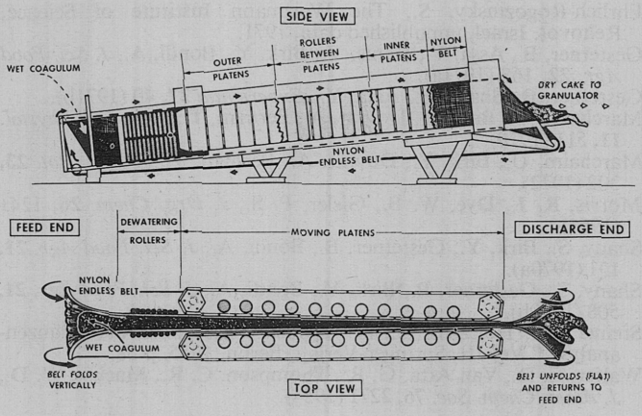

Figure 1. Schematic diagram of continuous belt press

containing two chrome-plated rolls 12 in. in diameter by 18 in. long was employed. Drum temperatures of 280–320°F, rotation times of 12–40 sec/rev, and gap settings of 0.010–0.012 in. were studied. Prior to drying, the coagulum was put through either a Waring Blendor or a high-speed disintegrator (Reitz or Fitzpatrick) to produce a smooth creamy liquid.

Screw Conveyor Drying. A small screw drier was designed and constructed to dry drained coagulum in a manner simulating continuous operation. It consisted of a half-pitch screw 4 in. in diameter by 15 in. long in a U-shaped trough with a perforated bottom (0.040 in. diameter holes, 30% open area). The bottom section was enclosed in a hot air jacket. Electrically heated air could be delivered to the jacket at rates to 50 cfm. This resulted in superficial air velocities of up to 120 fpm through the bed under the screw. The exhaust air path above the trough was enlarged in cross-sectional area to reduce the air velocity and trap small particles entrained in the air stream. A cheesecloth filter was placed across the top of the final exhaust area. The dust traps were essential only during the final stage of drying.

In operation, the drier was fed uniformly and continuously by hand from a large batch of drained coagulum. After the entire batch was fed through the drier, the product was cycled for additional passes to simulate staged drying. An inlet air temperature of 285°F and a product retention of 6 min/pass were used for the drying operation.

Vibrating-Fluidized Bed Drying. The vibrating-fluidized bed drier (W. R. Witte and Co.) consisted of a vibrating conveyor trough with a 125-mesh screen bottom, a plenum chamber for passing heated air up through the screen bottom, and a hood for collecting dust and exhausting air. The trough was approximately 10 ft long by 10 in. wide by 2 in. deep.

Drained coagulum was dried using two procedures. In the first method, drying was accomplished by successive passes through the drier. In the second method, a blend-back system was adopted. One part by weight dried curd from the first method was blended with two parts wet coagulum to reduce the moisture of the incoming feed. The resulting product was passed through the drier several times. A drier inlet temperature of 200°F was used. The temperature drop across the bed was 50°F.

Rotary Air Drying, Blend-Back Method. Experiments were conducted with a gas fired triple-pass pilot model Arnold dehydrator (The Heil Company Model S45-12). The dimensions of the drum are 4.5 ft o.d. × 12 ft in length. Product is fed into the high-temperature end and exits at the low-temperature end after making two reversals in direction within the drum. Material is advanced through the drum by the current of hot air drawn through the drier by a suction fan. The drier is rated at an evaporative capacity of 1000 lb H_2O/hr for product feed with moisture contents greater than 65%. Maximum recommended drier heat input is 2 million BTU/hr. Under normal operating conditions the desired drier outlet temperature is set by the operator and maintained by an automatic control system which regulates the flow of gas to the burner.

Feed for the drier was prepared by blending equal volumes of drained coagulum and dried product which has been ground through $^1/_{16}$ in. screen openings. After blending in a screw conveyor, the mix was passed through a curd mill with $^1/_4$ in. screen openings. The drier was fed continuously using a flighted drag conveyor system. Experiments were conducted over a range of outlet temperatures from 170 to 225°F and inlet temperatures of 400 to 1100°F.

Coagulum Dejuicing. Laboratory experiments were conducted using a hand press designed to exert 7 to 26 psi on a sample of drained coagulum. Pressure, adjusted by sliding a weight along a lever, was applied to the drained coagulum sample, and the brown juice volume expressed was measured as a function of time. In the pilot plant, dejuicing was accomplished either in a batch fashion using an hydraulic cider press (Palmer Cider Supplies Inc., are a 2.9 ft², 75 psi max) or in a continuous process with a demonstration model platen-belt press (Willmes "CONTIPAK").

In the continuous belt press (Figure 1), coagulum is placed on an endless multifilament nylon cloth. In operation, the cloth folds vertically and enters a series of dewatering rollers where preliminary squeezing is accomplished. The cloth then enters a space between two vertical traveling platen belts. The spacing between the belts decreases from the feed end to the discharge end and, as the platen-propelled cloth proceeds, pressure on the coagulum is correspondingly increased. At the discharge end the dejuiced cake separates from the cloth as it opens horizontally. The cloth then turns under the machine to return to the feed end. The platen belt spacing and speed are variable. Coagulum feed rates to the press ranged from 270 to 1080 lb/hr.

Rotary Air Drying, Single Pass. The pilot model Arnold dehydrator was able to dry dejuiced coagulum in a single pass through the dehydrator. Prior to drying, the dejuiced coagulum cake (~55% H_2O) was passed through a Stokes granulator with $^3/_{16}$ in. screen openings. The drier was continuously fed by a Syntron vibratory feeder. The coagulum was dried at an outlet temperature of 180°F and an inlet temperature range of 700 to 1200°F. The outlet temperature rose to 215°F as the final product emptied from the drier.

Carotene and Xanthophyll Determination. Dried coagulum samples were analyzed for total carotene and xanthophyll by the method of Knuckles *et al.* (1971), using an Evelyn Photoelectric Colorimeter.

Moisture Determination. Moisture contents were measured by drying samples in a forced draft oven at 110° for 2 hr.

RESULTS AND DISCUSSION

The wet coagulum prepared from whole alfalfa juice may be dried in a number of ways. Freeze drying produces a product with minimum nutritional deterioration and the freeze-dried coagulum is easily reconstituted to a smooth paste (Morrison and Pirie, 1961). Although freeze drying would be most desirable for products intended for food use, it is too expensive for large-scale production of an animal feed

Table I. Carotene–Xanthophyll Losses During Drum Drying[a]

Temperature, °F	Retention time on drum, sec	Moisture, %	Loss: Carotene, %	Loss: Xanthophyll, %
280	8	26	3	14
300	8	15	8	14
320	8	6	2	14
310	8	20	8	5
310	13	15	7	5
310	26	5	1	4

[a] Gap 0.010 in. first three values, 0.012 in. last three values.

Table II. Screw Conveyor Drying of Coagulum[a]

Pass	Cumulative drying time, min	Feed rate, g/min	Average temperature drop through bed,[b] °F	Water evaporation rate, g/min	Product moisture content, %
1	6	125	180	38	66
2	12	150	180	38	54
3	18	150	142	30	43
4	24	165	137	29	30
5	30	165	128	27	17

[a] Feed coagulum 75% moisture. [b] Inlet air 285°F.

Table III. Rotary Air Drying Using Blended Feed

Outlet, °F	Inlet, °F	Sample moisture, %	Carotenoid loss during drying: Carotene, %	Carotenoid loss during drying: Xanthophyll, %
170–180	400–500	9.8	3	6
170–180	800–900	8.5	3	7
170–180	900–1100	8.9	5	8
195	400–425	8.0	11	11
225	800–900	2.0	15	26

Table IV. Rotary Air Drying[a] of Dejuiced Coagulum

Particle description	Particle sieve size, U. S. series mesh no.	Particle fraction weight, %	Carotene,[b] mg/lb	Xanthophyll,[b] mg/lb
Very coarse	+ 12	9.8	255	400
Coarse	−12 + 20	28.2	250	399
Fine	−20 + 40	29.4	241	404
Very fine	−40	32.6	211[c]	368
		100.0	235[d]	390[d]

[a] Outlet temperature 170–215°F. [b] Average carotene and xanthophyll values of initial freeze-dried samples were 260 mg/lb and 443 mg/lb, respectively. [c] Judged significantly different from other carotene values ($p < 0.05$) by the Duncan's Multiple range test. [d] Weighted average.

supplement. Since freeze-dried coagulum consistently gave higher carotene and xanthophyll values than the same coagulum dried by other means, the carotenoid content obtained by freeze drying was used as the reference level for calculating the losses obtained by other drying methods.

Hartman *et al.* (1967) reported that spray drying of the whole juice was an excellent method of preserving vitamins and xanthophyll. They also reported that it produced no deleterious effect on the nutritional value as determined enzymatically. However, Subba Rau *et al.* (1969) found that spray-dried whole juice had a much lower protein efficiency ratio (PER) than washed and oven-dried LPC prepared from steam coagulated alfalfa juice. They attributed the reduced nutritional value of spray-dried whole juice to the remaining water-soluble constituents.

More recently, Subba Rau and Singh (1970) spray dried a thin slurry of heat-coagulated and water-washed LPC and found no significant difference in PER between samples that had been freeze dried, spray dried, or air dried at 40 to 60° in a cross-flow oven. No data on carotenoid stability was reported. We have found that spray drying of the wet coagulum resulted in carotene and xanthophyll losses of 5 and 12%, respectively. Our spray-dried product was a fine bright green powder, similar in appearance to that obtained by freeze drying.

When we attempted to drum dry the coagulum immediately after separation from the brown juice, damp flakes formed. No operating conditions were found that would produce a dry product without scorching and burning. However, blending the coagulum to a smooth paste before drum drying assured an even coating on the drums and substantially reduced the incidence of scorching and burning. This is in accord with previous observations (Cowlishaw *et al.*, 1956; Spencer *et al.*, 1970; Subba Rau and Singh, 1969). The resulting dark green flaky product was easily crumbled into a free-flowing powder.

Over a wide range of drum temperatures and retention times, carotenoid losses were not severe and did not exceed 14% (Table I). Attempts to drum dry coagulum on a commercial size drier (Model F, Buflovak) were largely unsuccessful.

A number of alternative methods of forced air drying were studied. The performance of a bench model screw conveyor air drier is presented in Table II. In this case the drained coagulum was dried in stages. The particles of product shrank as the moisture content was reduced, requiring an increase in the feed rate in successive passes to maintain a full layer over the surface of screen bed.

Drained coagulum could also be dried by repeated passes through a vibrating-fluidized bed drier. At high moisture levels, the coagulum was very difficult to feed. However, when the moisture level was reduced to 60%, the coagulum could be crumbled readily and fed easily to the drier. Therefore, a blend-back step was employed in preparing the drier feed to facilitate handling of the coagulum. Carotenoid losses were less than 8% after 22 passes through the drier, including three blend-back cycles.

The product from these air driers was dark olive green in color, and hard and granular in texture, but suitable for introduction into a basic animal feed with little additional processing. These promising results with small-scale laboratory equipment prompted the study of a more practical type of air drier. The rotary air drier, already widely used for producing dehydrated alfalfa meal, operates at high inlet temperatures, is highly efficient, and has a large throughput.

It was found in preliminary work with the Arnold rotary dehydrator that wet coagulum (80 to 85% moisture) plugged the feed inlet. However, no plugging occurred when a 50% moisture premix, prepared by blending wet and dry coagulum, was used as the feed. Both the feed material and the dried product has to be ground through screens to prevent the buildup of large lumps. These lumps were formed by addition of successive layers of wet coagulum on the dry granules during blending. Carotenoid losses remained below 8% when the outlet temperature did not exceed 180°F (Table III). The dried coagulum was similar in appearance and texture to that obtained from the screw and vibrating-fluidized bed driers.

The blend-back process could be eliminated if the wet coagulum could be reduced to 60% moisture. Laboratory

Table V. Compositional Comparison of Drained and Dejuiced Coagulum[a]

	Water,[b] %	Soluble solids, %	Crude protein,[c] %	Fat, %	Fiber, %	Ash, %	Carotene, mg/lb	Xanthophyll, mg/lb
Coagulum, drained	81.0	23.3	53.2	6.3	1.3	14.3	175	388
Coagulum, dejuiced	59.8	8.1	62.4	8.0	1.4	12.8	238	493
Brown juice	94.5	100	23.7			18.8		

[a] Dry basis analysis (data from Kuzmicky, 1972). [b] Wet basis. [c] Kjeldahl N × 6.25.

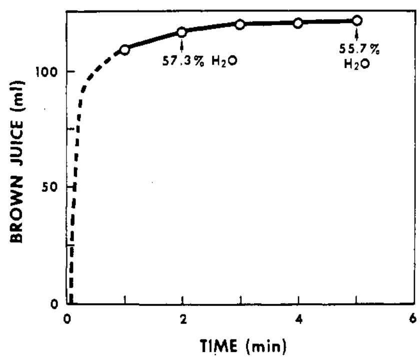

Figure 2. Mechanical expression (19 psi) of brown juice from 200 g of coagulum (80% H_2O, 175°F)

experiments demonstrated that this could be accomplished by subjecting the coagulum to a brief pressing (Figure 2). The resulting dejuiced coagulum crumbled easily and was found suitable for direct passage through the rotary air drier. The brown juice could be expressed most easily from freshly prepared coagulum, maintained at a temperature of about 175°F.

Larger quantities of feed material for pilot plant operation were obtained by pressing wet coagulum in either the hydraulic cider press or the continuous Willmes platen-belt press. The dried product had the same texture as the coagulum dried in the Arnold drier using the blend-back method but was decidedly greener in color. Losses of carotene and xanthophyll were low, averaging 10 and 12%, respectively. Carotenoid analysis of sieved fractions from a run producing 400 lb of dried product is presented in Table IV.

The single pass method using dejuiced coagulum results in a product with higher protein, carotene, and xanthophyll contents (Table V). The lower soluble solids content should result in a higher PER value than that obtained with the unpressed coagulum (Subba Rau and Singh, 1970). The production of finished product from dejuiced feed at 60% moisture would be 2.8 times that amount produced using 60% moisture blend-back feed with the same drier operating conditions.

The brown juice from the coagulum separation and dejuicing operations can be cycled back onto the alfalfa press cake prior to dehydration or processed into a separate product by vacuum concentration. If mixed with the press cake, the added brown juice reduces the level of fiber and contributes minerals, amino acids, and other nutrients to the alfalfa meal. This is the simplest method of processing the brown juice. For a brown juice concentrate, current foreseeable uses are as a liquid feed supplement or a culture medium for single-celled organisms. Vacuum concentration, although requiring additional equipment, is a more efficient method of removing water.

We have demonstrated that rotary air drying is a satisfactory commercial procedure for preparing dried LPC for use as an animal feed ingredient. Both the blend-back and single pass procedures yield an acceptable product (Booth *et al.*, 1972; Halloran, 1972; Kuzmicky *et al.*, 1972). The choice of one method over the other will depend on the desired product composition, size of the operation, amount of capital available, and other factors which can only be evaluated by the individual processor.

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