PRO-XAN Process. Pilot Plant for Separation of Heat-Precipitated Leaf Protein from Residual Alfalfa Juice

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A new drag separator was developed and tested for continuous separation of protein-xanthophyll coagulum from the residual liquor after heat coagulation of the leaf protein in alfalfa juice (the PRO-XAN process). The wet coagulum was drained in a cloth-lined reel and the residual liquor was clarified in a settling tank. An average yield of 2.4 lb of wet coagulum (19% total solids), equivalent to 0.45 lb of PRO-XAN (10% moisture), was obtained per pound of raw juice solids. Performance test data are presented.

Two major assets of dried alfalfa as a feedstuff are its protein and xanthophyll content (Hunter, 1969). Normally, 15–17% protein is required in feedgrade dehydrated alfalfa but protein contents in alfalfa grown in some areas may be as high as 21% or more. Marketing the surplus protein and xanthophyll values for feeding monogastric animals appears to be well worthwhile, without significantly affecting the residual feed value for ruminants.

Procedures have been studied at this laboratory for preparing a protein-xanthophyll concentrate (PRO-XAN) for use as a poultry feed supplement. The basic process consists of several operations: treating freshly harvested chopped alfalfa with gaseous ammonia; pressing part of the juice (green juice) from the plant; heating this juice by direct injection of steam to coagulate protein and coprecipitate xanthophyll; separating the coagulum from the hot residual juice fraction (brown juice); drying the coagulum to 10% moisture (PRO-XAN); and concentrating the brown juice to 50% solids for use separately as a "molasses" feed supplement.

Separation of protein coagulum from brown juice after coagulation is a difficult operation. Variations in chemical and physical properties of alfalfa cause variations in the nature of coagulum obtained under constant conditions. Freshly precipitated coagulum may sink, float, or remain randomly suspended in the brown juice; it may be grainy and easy to handle or slimy and hard to dewater; it may coalesce into curds or remain fine. The separation process must be designed to handle all types with little or no change in operating procedure and with minimum labor and capital investment.

Conventional drag classifiers (Perry *et al.*, 1963) are not suitable because of the unpredictable characteristics of the coagulum. The objective of this work was to design and operate pilot plant equipment suitable for continuous separation of all types of coagulum from residual brown juice, and to predict how the methods could be scaled up to commercial size.

EXPERIMENTAL

The juice extraction rolls and steam injection coagulating system have been previously described (Knuckles *et al.*, 1970; Spencer *et al.*, 1970, 1971; Witt *et al.*, 1971). Ammonia treatment was maintained constant in all tests to obtain the optimum pH of 8.5 in the green juice. Juice was processed within 1 to 3 hr after pressing. A variable-speed positive delivery pump fed green juice to the coagulation system and reduced back-pressure fluctuations. The feed pump delivered 0.75 to 2 gpm to the coagulator. Coagulator temperature was automatically controlled at 195° F in all tests, and provided complete coagulation for our tests.

It was shown in preliminary bench tests that ammoniated green juice heated to 195° F needs 0.5 to 4 min hold time for coagulation and agglomeration of protein-xanthophyll fraction, plus additional time for separation from the brown juice. A 3-gal vessel was provided after the steam injection heater to give about 2 to 3.5 min hold time for the throughput used. This and additional time in the drag separator were more than adequate to obtain good separation in all tests.

The equipment was operated continuously for periods for 2.5 to 4.5 hr in each test (except test #5). Flow rates, yields, and samples were taken only after steady state was obtained, usually after 1 hr of operation.

PILOT PLANT EQUIPMENT AND OPERATION

Drag Separator. A trough-like tank with sloping bottom, perforated sheet metal sidewalls, and a drag conveyor with perforated metal flights was designed and constructed (Figure 1). The tank was about 40 in. long, 14.5 in. wide, and 10 in. deep at its deep end. The false walls created gutters about 1.5 in. wide. The drag flights were ell-shaped in cross-section, 1 in. by 0.75 in., and mounted 2.5 in. apart on an endless #40 chain traveling on two sprockets, 3.75 in. diameter at the shallow end, and 4.75 in. diameter at the deep end. Brown liquor strained through the false walls into side gutters and then to the settling tank. Coagulum was dragged up the inclined discharge ramp, allowing brown juice to drain back. The trough had a capacity of 3 to 4.5 gal, depending on the setting of the level control overflow tube. This volume provided to 2 to 4.5 min hold time with the flows used. The false walls and the drag flights were fabricated of 20-gauge perforated stainless steel ($^3/_{32}$-in. diameter perforations on $^5/_{32}$-in. centers, 32% open space). The flow velocity through the perforations in the false walls was only $^3/_{16}$ in. per sec at 1 gpm flow. The settling rate (or floating rate) of the bulk of the coagulum must be greater than the horizontal velocity of brown juice to escape being drawn into the screen openings. It is obviously impossible to prevent some of the coagulum from depositing on the screen or from passing through the false walls along with the brown juice. To prevent clogging of the screen, flexible silicone rubber blades were provided at each end of three of the drag flights, equally spaced, to wipe

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Table I. **Summary of Performance of Pilot Plant Units**

Run		Green juice		Coagulum			Brown juice			Coagulum yield
				Drag separator[a]	Reel		Reel	Settling tank		
No.	hr	lb/min	%t.s.	%t.s.	lb/min	%t.s.	lb/min	lb/min	%t.s.	wet[b]
1	2.5	7.14	7.75	13.7	1.07	20.3	0.71	6.29	4.69	2.06
2	4.5	7.14	7.89	15.9	1.33	19.7	0.90	4.62	4.40	2.45
3	3	7.82	8.10	13.1	1.83	18.2	1.23	5.14	4.74	2.76
4	3	7.80	8.90	16.8	1.73	18.0	0.54	5.50	5.70	2.36
5	1.25	12.50	9.07	11.4	2.73	18.4	3.07	7.31	5.71	2.32
6	2.5	9.90	7.70	14.7	1.88	18.1	1.08	7.79	5.00	2.35
7	2.5	9.90	9.80	16.6	2.21	20.0	0.72	8.20	6.20	2.40
									Avg	2.39

[a] Average of spot samples. [b] Pounds of coagulum (19% t.s.) per pound of green juice solids. To obtain pounds of coagulum (10%) moisture per pound of green juice solids, multiply values in last column by 0.21.

the screens continuously and remove the mat of coagulum which tended to form.

The flights of the drag separator were cleaned continuously on their return path by a curtain of fine jets of compressed air.

Drain Reel. A slowly rotating (1 rpm) perforated drum, 18 in. in diameter by 28 in. long, lined with a loose-fitting coarse cotton burlap-weave cloth (Figure 1) was provided after the drag separator to drain the remaining free-running brown juice from the coagulum. The drum was pitched 1 in. per ft to provide a gentle slope for the tumbling coagulum to move through the unit and discharge into the collector.

Settling Tank. A 25-gal settling tank was provided to remove sediment that escaped the drag separator. This tank (Figure 1) 19 in. in diameter and 22 in. deep, had a sleeve 7 in. in diameter and 9 in. long to direct the incoming brown juice down into the center of the body of liquid. An overflow pipe near the upper rim continuously drained off the clarified brown juice. At the end of the run the accumulated sediment was drained and measured.

DISCUSSION OF RESULTS

The results of seven test runs are summarized in Table I. All runs were 2.5 to 4.5 hr long except run 5. Data were taken after steady state was achieved; results shown are averages. Data for yield of wet coagulum (19% solids) are shown in the last column, averaging 2.39 lb per lb of raw juice solids. Using these factors, and assuming a specific heat of 1.00 for juice, equations may be established for calculation of coagulum and brown juice yields.

$$C = 0.454 \frac{FS_F}{S_c}$$

and

$$B = F\left(1 + \frac{t_c - t_F}{970} - 0.454 \frac{S_F}{S_c}\right)$$

where F = feed rate, green juice, lb/min; S_F = solids content in feed juice, %; S_c = solids content in coagulum, %; C = coagulum, lb/min at S_c; B = brown juice, lb/min, total from settling tank plus reel; t_c = coagulation temperature, °F; and t_F = green juice temperature, °F. Deviations of actual and calculated values are shown in Table II. Brown juice yield averaged about 92% of the calculated value, probably because of evaporative losses from the hot open system.

Table II. **Comparison of Calculated and Actual Values of Brown Juice and Coagulum**

Run no.	Brown juice, lb/min			Coagulum, lb/min		
	calcd	actual	%[a]	calcd	actual	%[a]
1	6.83	7.00	102	1.24	1.07	86
2	6.76	5.52	82	1.30	1.33	102
3	7.25	6.37	88	1.58	1.83	116
4	7.06	6.04	86	1.75	1.73	99
5	11.30	10.38	92	2.86	2.73	95
6	9.27	8.87	96	1.91	1.88	98
7	8.98	8.92	99	2.20	2.21	100

[a] % of calculated value.

Capacity of the pilot plant varied with the green juice characteristics, but our tests showed that up to 10 to 12.5 lb/min of green juice could be easily handled. One test (not shown) at 17 lb/min feed rate indicated that the equipment soon became overloaded.

Operation of the drag in the separator was optimum at 105 sec/cycle. Higher speeds reduced coagulum drainage time, and slower speeds also reduced drainage by accumulating too much coagulum on the drag flights.

A small amount of fine coagulum escaped through the sidewall screens with the brown juice but it was only 1.6 to

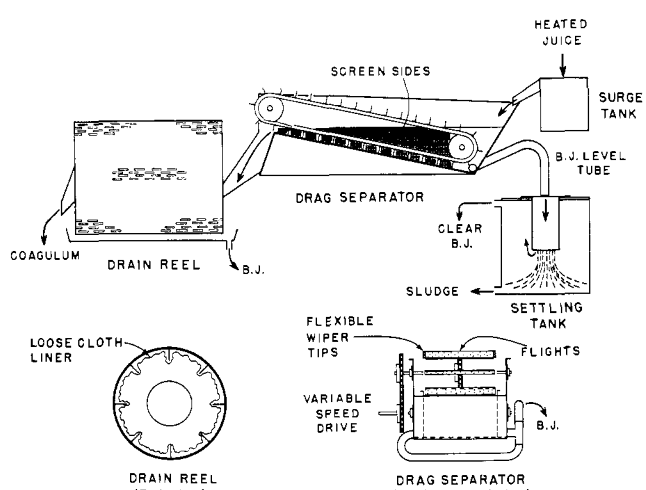

Figure 1. Pilot plant for separation of coagplum from brown juice in the PRO-XAN process

Table III. Performance of Settling Tank

Run no.	Green juice rate lb/min	Coagulum from reel lb/min[a]	Sediment in brown juice lb/min[a]	Sediment % of coagulum[b]
2	7.14	0.262	0.017	6.5
3	7.82	0.334	0.021	6.3
4	7.80	0.312	0.005	1.6

[a] Dry basis. [b] If sediment were not removed from the brown juice, it would amount to about 4% of the brown juice solids.

Table IV. Performance of Drain Reel

Run no.	Coagulum yield lb/hr[a]	Reel hold-up, lb at steady state[a]	Calcd retention time, min
2	80	40	30
4	104	30	18
7	133	48	22

[a] As-is basis.

6.5% of the total coagulum (see Table III) and was easily removed in the settling tank. When a finer screen (0.045-in. openings) with the same open area was tested, clogging was prohibitive.

The sediment passing through the screens with the brown juice was shown in bench tests to settle rapidly, in only 2 to 3 min. The settling tank provided perhaps five times the retention time needed but it was deliberately oversized to accumulate sediment for an entire day's run. It is common practice to pump off an underflow continuously from commercial settling tanks. In our plant the sludge could be returned to the drain reel.

At steady state in the tests the drain reel held different amounts of coagulum and, as a result, retention time varied (Table IV). This was attributed to different drainage characteristics of the coagulum produced on different days because of variations in alfalfa. Retention time was self-adjusting unless drain reel capacity is exceeded. The cloth liner did not clog if no more than $^1/_3$ of the coagulum discharged from the drag separator was free-running brown juice (about $^1/_8$ gpm). Heavier loads of brown juice caused by coagulum characteristics or by overloading the drag separator resulted in incomplete drainage, and wetter coagulum was discharged from the reel.

SCALE-UP OF EQUIPMENT

To scale-up pilot plant equipment to commercial size, only the drag separator and the drain reel are considered because scale-up of other units may be treated conventionally.

The pilot plant drag separator has a capacity of about 3.5 gal between screens when the liquid is 5 in. deep at the deep end. At 1 gal per min, retention time was 3.5 min with the overflow control tube set for 5-in. depth. To scale up to 50 gpm, for example, volume must be 175 gal to obtain the same retention time. Scaling-up the dimensions of the pilot plant unit by a factor of $(50)^{1/3}$, or 3.68, would give the required volume, retention time, and liquid flow rates through the screen. Similarly, to scale up to 100 gpm, the factor would be $(100)^{1/3}$ or 4.64. Screen openings must be retained close in size to those in the pilot plant model, and liquid through-flow velocity must not be exceeded.

To scale-up the drain reel, the same factors should be used. Also, it may be that the ratio of cloth drain area to volume of coagulum is important, a factor which is still to be investigated.

SUMMARY

Pilot plant equipment was developed to separate coagulum from residual liquor after heat precipitation of a protein-xanthophyll concentrate from alfalfa juice. In continuous tests lasting 2.25 to 4 hr, an average of 2.24 lb of wet coagulum (0.45 lb dry matter) was produced per lb of original raw juice solids. The coagulum (1.6 to 6.5% of the total) that escaped the drag separator into the residual liquor was easily recovered in a settling tank. Scale-up 50-fold would require units with dimensions 3.68× as large as the pilot plant units; for 100-fold scale-up the factor would be 4.64.

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LITERATURE CITED

Hunter, J. E., *Feedstuffs* **41,** 32 (1969).
Knuckles, B. E., Spencer, R. R., Lazar, M. E., Bickoff, E. M., Kohler, G. K., J. Agr. Food Chem. **18,** 1086 (1970).
Perry, R. H., Chilton, C. H., Kirkpatrick, S. D., "Chemical Engineers Handbook," 4th ed., McGraw-Hill, New York, N.Y., 1963.
Spencer, R. R., Mottola, A. C., Bickoff, E. M., Clark, P. J., Kohler, G. O., J. Agr. Food Chem. **19,** 504 (1971).
Spencer, R. R., Bickoff, E. M., Kohler, G. O., Witt, S. C., Knuckles, B. E., Mottola, A. C., *Trans. Amer. Soc. Agr. Eng.* **13,** 198 (1970).
Witt, S. C., Spencer, R. R., Bickoff, E. M., Kohler, G. K., J. Agr. Food Chem. **19,** 162 (1971).

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