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Desolventizing-Toasting of Extracted Soybean Flakes: Development of Pilot Plant Equipment and Operational Procedure

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ABSTRACT

A pilot plant batch desolventizer-toaster (D-T) was designed and built with the intent of producing soybean meals of varied composition, as well as to simulate meals produced in a continuous commercial D-T unit. Trial runs were made first to determine workable loading levels, temperature control and sparge steam generation. Moisture levels after the steam sparge were influenced by the residual hexane content of defatted hexane-wet flakes reaching the D-T. Two moisture levels were used in testing the effectiveness of the toasting operations in producing flakes with low urease activity and trypsin inhibitor levels. The trial runs reported here also provide basic data for current work designed to optimize toasting procedures to produce suitable meals for ongoing animal nutrition studies.

INTRODUCTION

In the desolventizing-toasting process for extracted soybean flakes, the final removal of the last traces of solvent, inactivation of enzymes and destruction of antinutritional factors occur to produce an animal feed with good nutritional quality. The process has been adopted worldwide by most processors of soybeans. Design and operation of a desolventizer-toaster (D-T) have been reported by Kruse (1,2), Cravens and Sipos (3), Sipos and Witte (4) and Milligan (5). In a commercial D-T, hexane-wet defatted flakes are rapidly heated, both indirectly and by sparge steam, in the top section of the D-T where most of the hexane is evaporated and steam is partly condensed on the flakes. Steam sparging, in addition to adding moisture to the flakes, minimizes dust carryover to the condenser, and results in a combination of solvent removal and meal toasting. Meal toasting is continued on the lower trays of the D-T where meal moisture is reduced by indirect steam heating.

Here we report on a pilot plant batch D-T that was intended to prepare meals with varied composition as well as to simulate the conditions and product of a continuous commercial D-T. To determine the effectiveness of the equipment, range of parameters and mode of operation to produce the meals of varied quality, 51 pilot plant runs were made. Four typically significant runs from this series are reported. Since the control methods developed in this report were reliable, a study (6) involving 48 meals of varied predetermined composition was conducted relating these compositions to their nutritional qualities.

[1] Presented at the ISF-AOCS World Congress, New York City, April 1980.

EXPERIMENTAL

Materials and Methods

Beeson variety seed-grade soybeans (1978 crop) and Skellysolve B petroleum naptha (hexane) were used for all runs. To prepare full-fat flakes for extraction, soybeans were cracked, dehulled, heat-tempered and flaked in pilot plant equipment to produce full-fat, dehulled soy flakes of 0.2540-0.3048 mm thickness. The flakes were extracted in a Kennedy pilot-plant extractor with a hexane:flake ratio of 2:1. Defatted flakes were drained on a slotted inclined drag-line conveyor before being discharged from the extractor.

Solvent concentration of the hexane-wet defatted flakes was determined by weighing the flakes before and after solvent vaporization under ambient conditions until odorless. A Brabender Moisture Tester (Brabender Corp., Haake, Inc., Saddle Brook, NJ) was used to measure moisture of defatted hexane-free flakes and meal products by drying the samples at 120 C for 1 hr. Moisture of the wet flakes after sparging was determined by drying the flakes in a forced draft oven at 105 C according to AACC Method 44-15A (72 hr) (7). This method was more accurate than the Brabender Moisture Tester for the partially agglomerated flakes. Urease activity for processed meal at various stages of toasting, reported as pH increase, was determined by AOCS Method Ba 99-58 (8). Trypsin inhibitor values for the meal product were determined according to the procedure of Hamerstrand et al. (9).

Equipment

Typical commercial D-T. A diagrammatic sketch of a com-

mercial D-T is shown in Figure 1a and a typical time-temperature relationship for meal fed through a commercial D-T is shown in Figure 1b. Meal temperatures at various sections of a commercial D-T have been reported by several authors (4,5). Defatted soy flakes from the extractor enter the D-T at about 60 C; the flake temperature after steam sparge (section A) ranges from 88-99 C; the defatted desolventized flakes are further heated (sections B and C) to 104-110 C. Meal is cooled in section D before finishing and storing. Solvent vapor leaving section A reaches 77 ± 6 C.

Flake moistures of 15-20% at the end-of-sparge are required in a commercial D-T for adequate toasting (1,2,4) and the toasted meal discharges at ca. 12% moisture. Hexane in the hexane-wet flakes varies from 28 to nearly 40% to achieve the high end-of-sparge flake moisture.

Pilot plant D-T. The pilot plant D-T shown in Figure 2 is a cylindrical, jacketed 316 stainless-steel vessel with bottom-driven variable speed sweep. Inside dimensions are 76.2-cm diam. by 50.8-cm height. The access doors located on top (a), side (b) and discharge port (c) are vapor-tight. Solvent vapors and moisture exit through a 12.7-cm duct (d) fitted with a vent butterfly valve (3) and an exhaust fan (f). The vent, when closed, forms a nearly vapor-tight vessel. The sweep (g) is fitted with a 8.9-cm-high blade at 60° angle on the leading edge and perforated pipe for steam sparge on the trailing edge. The jacket (h) on sides and bottom can be either heated by process steam or cooled by water. Sparge steam, ranging from 3.52 kg/cm^2 to 7.03 kg/cm^2, enters the D-T chamber through a rotary joint (i) coaxially located on the sweep drive shaft.

Dial thermometers were located in the pilot plant D-T 10 cm from the bottom to measure the meal, vapor and jacket steam temperatures. Subsequently, iron-constantan thermocouples were substituted for the dial thermometers to measure the meal and jacket steam temperatures.

Procedure

Initial trials in the D-T were made without hexane to establish the meal capacity, sparge steam capacity and heating limits of the equipment. Workable batch size and sweep speed in the D-T were determined by observing the blending action with varying amounts of dry, defatted soy flakes at various sweep speeds.

Before introducing sparge steam to the flakes in the D-T, the sparge line to the D-T is heated and dried by purging with steam. A water separator, installed immediately upstream to the D-T, minimized the amount of entrained water in the sparge steam.

Hexane-wet runs. Runs were made to establish operational conditions and limits for processing hexane-wet soy flakes in the pilot plant D-T, including: handling hexane-flake mixtures, hexane-flake moisture relationships and minimization of meal agglomeration.

Handling of hexane-flake mixtures. Two methods were compared initially. One method was to discharge the hexane-wet flakes from the extractor into 55-gal drums and later transfer them into the D-T; the other method was to transfer the hexane-wet flakes directly from the extractor into the D-T.

Hexane-flake moisture relationship. Flake moisture at the end-of-sparge has been reported (1,2,4) to be important in the toasting efficiency of the desolventized meal. In our procedure, flake moisture was controlled by adjusting the hexane content in the hexane-wet flakes by the addition of a calculated amount of hexane to the batch while stirring. For >20% flake moisture at the end-of-sparge, it was necessary to pre-cool the flakes to 27 C before starting the run; for <20% flake moisture at the end-of-sparge, the flakes were preheated to 57 C before starting the run.

Minimization of meal agglomerates. It was generally considered good operational practice to minimize attrition of the flakes during flake preparation and to distribute dry sparge steam evenly throughout the flakes during desolventizing to minimal agglomeration of the flakes. Use of sieving screens and aspiration during the flake preparation minimized the amount of fines sent to the extractor. The separator, installed in the sparge steam line upstream to the D-T, eliminated flake agglomeration caused by slugs of condensate in the sparge steam. Superheated sparge steam was tried but did not reduce further the formation of agglomerates. The geometric arrangement of the sparge distributor holes in the sweep was modified to deliver more sparge steam to the bulk of the flakes, which are at the perimeter of the D-T.

RESULTS AND DISCUSSION

Batch Size and Sweep Speed

In commercial units, meal depths of 40.6-61.0-cm is considered ideal for adequate mixing during the desolventizing-toasting process. Many commercial units will, however, use much deeper beds (up to 150 cm) to improve contact with the live steam and use intermediate sweeps in the bed to provide additional blending. Best flake depth for the pilot plant D-T was found to be 23-25 cm. A sweep speed of 33

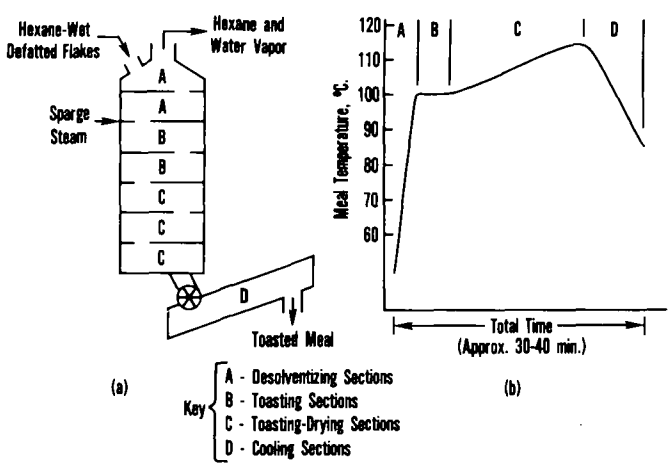

FIG. 1. (a) Diagram of processing sections in a commercial D-T. (b) Time-temperature heating curve in a D-T.

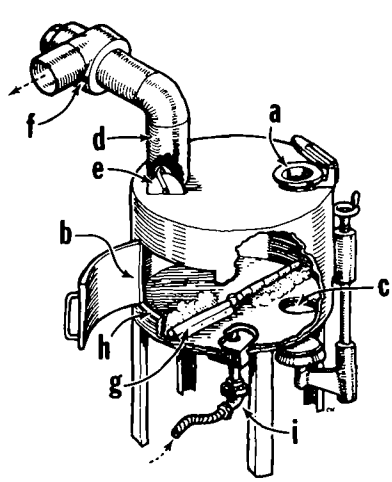

FIG. 2. Pilot plant (batch) desolventizer-toaster.

rpm effectively mixed the hexane-wet flakes, and 60 rpm was best for meal drying. Batch sizes of 31.7-36.3 kg desolventized soy flakes could be handled adequately in the pilot-plant operation.

Temperature Program

Jacket steam pressures between 1.05 kg/cm^2 and 2.81 kg/cm^2 had little effect on the meal temperature during the sparge period but, during the drying period, jacket steam pressure did influence the meal temperature. Jacket pressures of 1.41 kg/cm^2 and 2.81 kg/cm^2 were chosen to effect a significant spread in experimental drying temperature. It was necessary to cool the meal rapidly with jacket cooling water after drying to arrest further toasting of the meal.

Increasing Moisture by Sparge Steam

The amount of moisture added to the defatted soy flakes prior to toasting was dependent on condensate from sparge steam which cooled during vaporization of hexane from the hexane-wet flakes. The hexane concentration in the flakes was varied from 26 to 38%, which gave a broad range of end-of-sparge flake moistures. Hexane-wet flakes direct from the extractor contain about 26% hexane. Low-pressure process steam (4.71-7.03 kg/cm^2) yielded sufficient condensate to reach the upper meal moisture limit for toasting. Theoretically, based on the condensate from the sparge steam, if all the process steam at 7.03 kg/cm^2 was condensed on the flakes for 6 min, it would be possible to moisturize 39.9 kg defatted soy flakes to 37% moisture. However, equilibrium requirements dictate that a significant portion of the live steam must leave with the evaporated hexane vapors so this theoretical level of moisture is never reached.

Hexane-Flake Mixture Conditions

When hexane-wet flakes were discharged from the extractor into 55-gal drums, free hexane gravitated toward the bottom of the drum, and resulted in nonuniform hexane concentration in the flakes. It was found that hexane concentration in the flakes was much more uniform when flakes were charged directly to the D-T from the extractor. The hexane concentration in the flakes was measured just prior to each run, so that the proper amount of hexane could be added to the batch to ensure the designated level for the test.

Flake Moisture-Hexane Relationship

Kruse (1,2) and Sipos and Witte (4) reported that the complete moisturizing of the individual flake particle and cellular explosion within the flake were due to rapid steaming. The result of this rapid steaming was a product with good quality. The relationship between hexane concentration in the defatted hexane-wet flakes and flake moisture after rapid steaming is nearly linear. At 25 and 39% hexane, the end-of-sparge moisture was 16 and 22%, respectively. Sparge steam pressure had no effect on these values.

Minimization of Agglomeration

Although some agglomeration was prevalent in all D-T runs in the pilot plant (as it is in a commercial plant), the extent of agglomerization was minimized greatly by careful operational practices used. Trypsin inhibitor content of cross sectional samples of large agglomerates indicated that the trypsin inhibitor value at the center of the agglomerate was equal to that of nonagglomerated meal in the batch. The meal moisture was slightly more in the center of the large agglomerates.

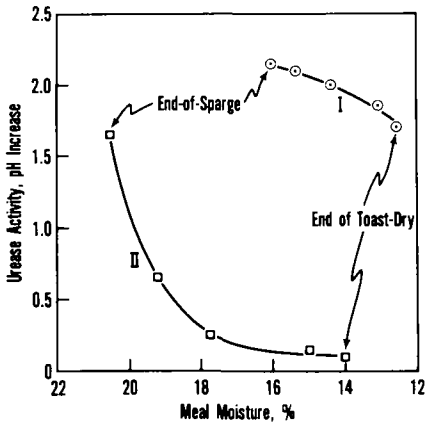

FIG. 3. Effect of flake moisture (end-of-sparge) on urease activity during drying period: I. end-of-sparge moisture—16%; II. end-of-sparge moisture—20.5%.

Effect of Toasting on Urease Activity and on Trypsin Inhibitors

Residual urease activity as a function of meal moisture during desolventizing and toasting of soybean meal is shown in Figure 3. At the end of the sparge, the flake moisture for run I was 16% and for run II was 20.5%. Both runs were dried at jacket temperatures of 123.3 C (35.58 kg/cm^2 pressure) for 40 min after sparging. Meal with a urease activity value of 0.05-0.12 is considered to be adequately toasted (10). Only meal II, with a final urease activity value of 0.1, was within these limits. Data for runs I and II indicate that, for an acceptable urease activity in the final product from our pilot-plant D-T, the end-of-sparge moisture content must be greater than 16% and near 20%. Kruse (1,2) reported the end-of-sparge flake moisture should be greater than 15% to produce a satisfactory meal product in the commercial D-T. Trypsin inhibitor inactivation was also a function of meal moisture level. Trypsin inhibitor destruction in end-of-run meal II was 87% and in end-of-run meal I was 76% of the trypsin inhibitor in the Beeson soybeans. Since one purpose of this study was to find the conditions for preparing meals of varied composition (meals with high and low urease and trypsin inhibitor) the methods for producing meals I and II satisfy this requirement.

Runs III and IV illustrate the effect of toasting time on urease activity at constant moisture and at elevated temperature. The results are shown in Figure 4. Both runs had an end-of-sparge moisture of 21% and were dried to final

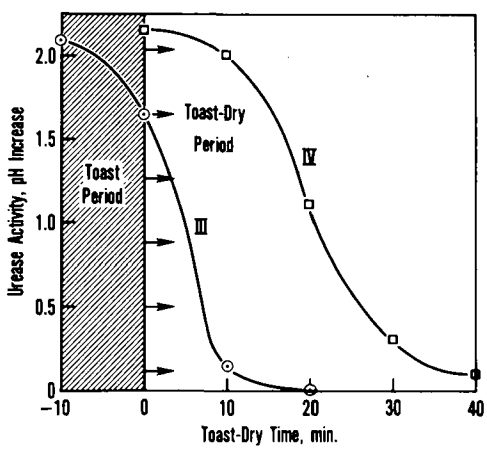

FIG. 4. Effect of an added toast period on urease activity level.

meal temperatures of 111.1 C. Run III was given a 10-min toast treatment at constant temperature with the vent closed and exhaust fan off before toast-drying, whereas for run IV, toast-drying was started immediately at the end-of-sparge. Although the urease activity declined rapidly in run III, the urease activity for the final meal product for both runs III and IV were at similar low levels, and the meals contained ca. 3 mg of trypsin inhibitor/g (92% trypsin inhibitor inactivation). The additional toasting period given for run III required to dry the meal to 12% moisture appeared to have little effect on final meal product quality. Sipos and Witte (4) also reported that toasting time at constant moisture was not critical to meal quality.

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Effect of Succinylation of Cottonseed Flour during Protein Extraction on the Yield and Some of the Properties of Protein Isolates

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ABSTRACT

A new method has been developed to improve extraction and recovery of protein isolates, which are highly water-soluble, low in sensitivity to calcium precipitation and light-colored, from defatted cottonseed flour. Optimal conditions include extraction of flour with acidified *n*-butanol (pH 4.5) to remove chromophores, succinylation of proteins at pH 8.5 using succinic anhydride at a concentration of 30% on protein basis, and precipitation of protein at pH 4.5. The resulting succinylated isolates contain more hydrophobic and neutral amino acids than untreated isolates. Gel filtration chromatography and polyacrylamide gel electrophoresis of protein isolates (water-, salt [4% NaCl]- and alkali [0.2% NaOH]-soluble), sequentially fractionated from succinylated and untreated flours, suggest that succinylation converts much of the salt- and alkali-soluble proteins to water-soluble forms. Succinylation increases emulsion stability of isolates. The 3 succinylated isolates showed similar chromatographic patterns on Sephadex G-100 columns and electrophoretic mobilities in polyacrylamide gels, whereas the corresponding 3 isolates from untreated flour had different gel patterns and mobilities. Mobilities of major protein components of the 3 isolates were increased by succinylation.

INTRODUCTION

Chemical modification of proteins induced by acylation with succinic, acetic and 3,3-dimethyl-glutaric anhydrides has been shown effective in improving properties of proteins in certain formulated foods (1-5). Reactions of proteins with these chemicals result in amino groups with different electrostatic charges than the original proteins. Succinic anhydride reacts with unprotonated free amino groups at high pH and changes distribution of charges in protein molecules (6). Moderate succinylation results in unfolding, expansion and increased negative charges in protein molecules, but extensive succinylation may cause the protein to dissociate into subunits (4,7). These types of molecular changes alter chemical and functional properties of proteins (8).

Acetic or succinic anhydride acylation is reported to alter functionality of glandless cottonseed flour (9,10). Succinylation of peanut flour increases nitrogen solubility at neutral pH, water absorption and retention capacities, emulsion capacity and viscosity (4). Final characteristics depend on the ratio of succinic anhydride to protein in the reaction mixture. Acetylation of soy protein isolate decreases water binding capacity and gel strength, but increases solubility in the pH range of 4.5 to 7.0 (11). Franzen and Kinsella (12) reported that succinylation of more than 90% of available amino groups shifts the isoelectric point of soy protein, and increases emulsifying and foaming capacities and stabilities. However, acetylation has little effect on functional properties of soy isolate.

Developments of glandless cottonseed varieties (13) and liquid cyclone processing techniques have resulted in cottonseed protein ingredients with very low gossypol contents (14), and have increased opportunities to incorporate cottonseed protein ingredients into, e.g., baked goods and meat analogs. The objectives of this research were to convert water-insoluble globular proteins of glandless cottonseed to water-soluble forms by succinylation, and to evaluate effects of succinylation on protein extractability, and on functional and physicochemical properties.

EXPERIMENTAL PROCEDURES

Materials

Glandless cottonseed flour defatted by hexane extraction at the Food Protein Research and Development Center at