SPRAY-DRYING OF NATURAL CHEESE

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This is to certify that the

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Charles M Stine
Major professor

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ABSTRACT

SPRAY-DRYING OF NATURAL CHEESE

by Robert L. Bradley, Jr.

A study has been made of the processes that contribute to good flavor and shelf-life in the manufacture of spray-dried Cheddar cheese powder. Addition of sodium citrate, sodium chloride and water to comminuted, high score Cheddar cheese and heating produces a smooth, fluid slurry that can be homogenized and spray-dried. An adequate preheat treatment retards the onset of stale and oxidized flavors in the powders. High pressure injection of inert gas into the slurry prior to atomization causes puffing of the particles during drying and favors the retention of flavor volatiles. Manipulation of the air temperature in the drier and the use of nozzles with large orifice diameters to minimize heat damage and to obtain a large average particle size also produces increased retention of volatiles. Nitrogen-packing and blends of antioxidants have been shown to extend the shelf-life of the spray-dried cheese powder; however, off-flavors in the powders may result from using the maximum effective concentration of some antioxidants.

Separation of the volatiles from natural Cheddar cheese, cheese slurries and powders was accomplished by gas chromatography using a 0.02 inch i.d. x 150 foot capillary column and a hydrogen flame detector. Losses of volatiles as well as development of flavor compounds occurred during preparation of the slurries and during spray-drying. Analysis of the chromatograms indicated that preheating the cheese slurries to 180 F for 10 min and foam spray-drying at a low exit air temperature results in a cheese powder with highly desirable flavor and storage properties.

SPRAY-DRYING OF NATURAL CHEESE

by

Robert L. Bradley, Jr.

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NOTE: The term "flavor" in the legend and on Figures 5 through 11 refers to flavor volatiles collected by high vacuum distillation.

INTRODUCTION

Convenience foods are making inroads on the buying habits of shoppers today. These foods reflect new processing and storage problems that are involved with these commodities. Dehydrated cheese is such a food and is especially useful for food items that can be stored without refrigeration. However, the shelf-life of dried cheese is enhanced by refrigerated storage in nitrogen-packed cans. Dehydrated cheese is different from natural cheese in the respect that no molding, oiling-off or proteolysis is exhibited during storage even at room temperature. Dried cheese is ideally suited for uses in such preparations as macaroni and cheese dinners, chip dip bases, bakery products and garnishings for soups and vegetables.

This project was undertaken to evaluate the factors involved in the spray-drying of natural cheese, to study the keeping quality of the dehydrated cheese and to determine the possible loss of volatile flavor constituents occurring during the various processing steps in the manufacture of spray-dried cheese.

REVIEW OF LITERATURE

The use of dehydration to improve the storage properties of dairy products has been practiced for decades. Products containing high percentages of fat require the added protection of an atmosphere essentially devoid of oxygen during storage. These two procedures allow long term storage without refrigeration and provide an excellent possibility for preparation of many convenience food items.

Cheese has been widely accepted in the diets of people in the United States. With the present trend toward "instant" types of foods, manufacturers have shown great interest in the dehydration of the cheese required in the formulation of some of these convenience foods.

Dehydration of Cheese

Freeze-drying has been suggested by Evstrateva (28) as a means of preserving two varieties of surplus lactic-coagulated cheese in Russia. The dried cheese contained 2.9 to 4.0 per cent moisture after drying 18 hours at 0.5 to 0.8 mm pressure. When reconstituted, the cheeses contained 17.5 per cent fat or were fat-free, depending upon type of cheese and were given "high grades by many experts". The daily output of the Russian lyophilizer was 21 to 74.2 kg of dried product. A German dairy has set up equipment to freeze-dry quarg produced from 44,000 pounds of milk per working shift. The finished product was packed in plastic bags and was readily soluble in cold water (5).

Meyer and Jokay (56) and Jokay and Meyer (42) investigated the application of lyophilization and the characteristics and stability of many cheese products after lyophilization. Among these products were creamed Cottage, Cheddar, Brick, Muenster, Blue and Cream cheeses. After reconstitution, these cheeses had a soft, natural consistency with a mild flavor.

Saunders (3,4,70) reported the technique of dehydrating the hard varieties of cheese. Patents were subsequently granted in the United States and Great Britain for this method (71, 72). The grated cheese was partially dried at room temperature in moving air. This partial drying case-hardened the shreds and prevented the exudation of fat during the final drying at a maximum temperature of 145 F. The dried cheese flakes contained 3 per cent moisture. Several other researchers investigated this method of air-drying. The hard, grating style of cheese such as Romano and Parmesan have customarily been air-dried before grating. Olsansky and Schmidl (61) studied the effects of bright and dark infra-red lights during drying of Parmesan cheese. The use of dark infra-red lamps with filters to exclude visible light resulted in more efficient drying, whereas, the use of bright infra-red lamps induced adverse effects in the dried cheese.

The application of the air-drying method developed by Czulak, Hammond and Forss (21) resulted in a dried cheese-type product that exhibited no free fat or particle clumping. The observation was made that the addition of roller or spray-dried skimmilk or whey during grating and after drying was essential to keep the product particulate. The flavor of the product was "weak cheesy" since most of the flavor volatiles were lost during drying. A French patent was issued to Riesen (66) for a method of manufacturing cheese in powder or chip form. The cheese curd precipitated with calcium chloride was drained, hooped, brined in saturated salt solution, ripened at low temperature and finally grated and dehydrated. Yakhontov (90) described a process of air-drying cheese that resulted in a partially dried product containing 10 per cent moisture. This dried cheese was refrigerated to prevent spoilage. Kiermeier and Wildbrett (44) noted that dried Emmental cheese remained edible for several months in water-tight wrappers providing that the cheese had been dried to less than 6 per cent moisture.

The utilization of roller and spray-drying has been considered for manufacturing a dried cheese product. Traisman and Kurtzholts (85) were granted patent rights for a method of producing dried grated cheese. This method involved finely slicing and air-drying one portion of the cheese to 10 to 12 per cent moisture. Another portion of the cheese was melted with 1 per cent disodium phosphate, heated to 200 F and spray-dried. The spray-dried powder was mixed with an equivalent amount of air-dried cheese, milled, tempered, sieved and packaged. In another patent, Silberman (74) spray-dried Cottage cheese curd after preheating the curd to 130 F. Woud (89) obtained a British patent for the process and equipment to manufacture dried cheese products. The original cheese was pulverized, raised to a pH of 6.5 to 7.0 with citric acid and sodium carbonate, heated, diluted with water and roller-dried. Jordan, Clickner and Erekson (43) patented a method of preparing cheese dried on a roller-drier. This powder was intended for use in cheese spray mixtures for applying to crackers and popcorn. In a French patent, Giraud (29) described a process to spray-dry cheese that previously had been mixed with 10 to 20 per cent water and heated to 80 C prior to dehydrating.

Makar'in (50) developed a process for the manufacture of a dried cheese powder. This process involved melting the cheese, adding phosphates, sodium citrate and other emulsifiers, diluting with water to approximately 35 per cent total solids and spray-drying the product. Krasheninin, Shubin and Markhinin (46) used this procedure of preparing the cheese to manufacture dried processed cheese. Roundy (68) and Roundy and Ormond (69) were issued patents for methods to rapidly produce Baker's, Neufchatel and Cream type cheeses. These cheeses were spray-dried to increase shelf-life and to improve baking characteristics. Bauman, MacMillan and Stein (7) patented a process for the rapid manufacture of spray-dried products having characteristic cheese flavoring. A milk product containing casein was used as the fermentable base. Vegetable or butter oil with an emulsifier and no more than 2.14 per cent Cheddar cheese were added. Flavors resulting from the bacterial fermentation of milk were developed during 12 hours incubation at 75 F prior to spray-drying.

Boháč (11) roller-dried cheese mixtures that had been emulsified with tetrasodium pyrophosphate and the ammonium salt of "polymeric" phosphate. The fact that the roller drier was contaminated with dried skimmilk prevented accurate evaluation of these results. However, the results of the trials in which mixtures of cheese were spray-dried showed possibilities for special products for the food industry. This cheese powder was stored in air-tight wrappers or pressed into cubes. Rogers (67) was granted a patent for a method of manufacturing dehydrated powdered cheese. As an object of the invention Rogers avoided subjecting the cheese during dehydration "to any temperature high enough to cause material escape of the volatile flavors." In prior art, temperatures from 180 to 190 F had been used for dehydration. Mixtures of cheese and water containing 35 to 42 per cent total solids were heated during constant agitation to 160 F, cooled to 140 F, homogenized at 2500 to 5000 psig and spray-dried. A low drying temperature (165 F or less) was employed. Noznick and Bernardoni (59) patented a similar process for the manufacture of Blue or Cheddar cheese powder. The novel part of this patent was the mixing of the broken cheese with hot water.

Irvine et al. (40) spray-dried a "low fat" Cheddar cheese that was emulsified previously with disodium phosphate and diluted with water. The resulting powder containing 12 per cent fat and 3.2 per cent moisture stored well up to 6 months at 50 F. Subsequently, Bullock, Hamilton and Irvine (14) reported that spray-drying of Cheddar cheese containing 48 to 49 per cent fat on a dry basis was commercially feasible.

Foam spray-drying is a variation of conventional spray-drying in which a gas is introduced into the liquid either before or after the high pressure pump. The gas causes puffing of the droplets formed during atomization. Several patents have been issued to cover the novel aspects of this procedure. Heath and Washburn (37) saturated concentrated milk with carbon dioxide for the purpose of reducing the bacterial content and to produce a better product with a more agreeable flavor. Reich and Johnston (65) showed that bulk density, color and free-flowing characteristics of coffee and tea powders could be controlled and improved by the use of air, carbon dioxide and nitrogen that was metered into the concentrate under pressure. A process for the carbonation of coffee extract for manufacturing instant coffee was patented by Chase and Laursen (16). Oakes et al. (60) described a similar process for the manufacture of instant non-fat dry milk. After injection of nitrogen or nitrous oxide, the concentrate was whipped under pressure to a stable foam and spray-dried. Products that are difficult to dry by conventional spray-drying can be dried to a free flowing state with the injection of gas into the liquid material. Hanrahan and Webb (33) demonstrated that Cottage cheese whey could be dried by this technique. Subsequently, Hanrahan et al. (32) applied foam spray-drying to the production of dried whole milk using nitrogen as the foaming agent. The results of these trials showed that the powders had increased solubility in water.

Shelf-life of Dried Cheese

Numerous reports indicate that the exclusion of atmospheric oxygen and the presence of an inert atmosphere are necessary to maintain the palatability of dried products that contain high concentrations of fat. Lea, Moran and Smith (47) discussed many factors that affected the oxidation of milk fat or tallowiness in whole milk powder. Among these factors are storage temperature, duration of exposure to ultraviolet light and the concentrations of oxygen, copper and iron. Reduction of the quantity of oxygen in the headspace gas to a concentration below 2 per cent, improved the stability of spray-dried whole milk powder. Coulter (18) and Coulter, Jenness and Crowe (20) concluded that less than 1 per cent oxygen in the headspace gas was necessary to prevent oxidation of dry whole milk. Statistical analysis of data by Tamsma et al. (81) showed that 0.1 per cent oxygen in the headspace gas contributed to a more stable powder than powders containing higher concentrations of oxygen in the headspace gas. "Burned" or "stale" off-flavors appear to be associated with the protein in the powder. Development of stale flavor is reduced by low concentrations of oxygen in the stored containers. Henry et al. (38) showed that stale flavors are attributable to Maillard type browning. To protect against gas leakage through the rolled seams of the cans during storage, Lea, Moran and Smith (47) used molten Wood's metal layered with lactic acid as a dip. The lactic acid functions as a flux and is removed from the cans by washing with water.

Since the concentration of oxygen in the headspace is critical, an accurate measurement would be an obvious quality control measure. Many manufacturers of dry whole milk still use the Orsat gas analyser; however, the accuracy of this instrument is poor particularly with small volumes of gas. One modification of the Orsat analyzer was developed by Thiel, Loftus-Hills and Scharp (84). This complex apparatus was constructed to give reproducible results with small samples of gas.

Recently, gas chromatography has been used as a means of quantitatively determining the composition of gases. Murakami (58) showed that mixtures of oxygen, nitrogen, methane, carbon dioxide and carbon monoxide were quantitatively separable by using two columns containing silica gel and 5 A molecular sieve. Brenner and Cieplinski (13) developed a parallel column device to permit the simultaneous use of two columns. Carbon dioxide is irreversibly absorbed on the molecular sieve; therefore, a second column is required to separate this gas.

Stahl, Voelker and Sullivan (77) modified a Zahm Air Tester into a piercing device that was adaptable to any size of can or flexible package. A sample of headspace gas could be removed with an air-tight syringe. However, this gaseous sample is removed from inside the piercing apparatus and possibly the composition of the gas is different from that in the container. Vosti, Hernandez and Strand (86) developed a method for the complete removal of the headspace gases in a container. Hydrostatic pressure was used to force the gas from the container into a water-filled, inverted funnel. Beckman Instruments (8) also market a headspace sampler connected to a Beckman Oxygen Analyser. Aside from the expense of this equipment, the instrument provides means for accurate and rapid determination of oxygen.

Evaluation of the Volatile Flavors in Cheese

Investigation of the composition of complex mixtures of organic compounds and particularly, the mixtures of flavor constituents in foods requires exacting analytical techniques. Until the development of gas liquid partition chromatography (GLC) by James and Martin (41), many of the compounds that existed in trace quantities in foods were impossible to isolate and identify. The gas chromatograph is an analytical tool used to separate these volatile flavor materials. Harley, Nel and Pretorius (35) and McWilliam and Dewar (54) later described a flame ionization detector that had greater sensitivity for organic compounds than the thermal conductivity detector used prior to 1958. The combination of a flame ionization detector and a column packed with a solid support coated with the appropriate liquid phase permitted detection and separation of trace quantities of volatile flavor materials.

Often the individual flavor constituents in foods are similar or structural isomers in chemical configuration. Therefore, these constituents have boiling points that differ only by one degree Centigrade or less. Separation of these compounds is difficult, especially when the compounds are aggregated as a complex food flavor. Capillary tubes coated on the inside wall with a liquid phase can be used to obtain high separating efficiency. Moreover, recent improvements in the sensitivity of detectors have made possible the separation and elucidation of compounds with concentrations in the range of parts per billion.

The qualitative and quantitative evaluations of food flavor is a complex area of science. Patton and Josephson (62) indicated the need for objective research in this area to determine the spectrum of flavor constituents and those compounds which are present in concentrations above the taste threshold. Harper (36) reviewed the chemistry of cheese flavors and stated that after vacuum distillation the cheese residue retains a cheese flavor. This residue contained relatively non-volatile material such as lactic acid, amino acids, keto acids, certain amines and salt, whereas, the distillate that was collected possessed a typical cheese aroma and was composed of fatty acids, aldehydes, ketones, alcohols, amines, esters and sulfides.

Several researchers have used methods that involve GLC in the analysis of cheese flavors. Patton, Wong and Forss (63) used GLC and mass spectrometry to conclusively identify dimethyl sulfide, ethanol, acetone and diacetyl in Cheddar cheese. Scarpellino and Kosikowski (73) isolated acetic and butyric acids, 2-butanol, butanone, acetoin, ethanol and diacetyl from Cheddar cheese using GLC and a thermal conductivity detector. McGugan and Howsam (53) tentatively identified 42 volatile compounds in Cheddar cheese through the use of GLC, a capillary column and a hydrogen flame detector. Libbey, Bills and Day (48) demonstrated a method of low temperature high vacuum distillation that gave more complete removal of the volatile materials from Cheddar cheese. When a sample of the distillate was analysed by GLC, 29 components were separated and 19 of these components were identified. Day (22) reported the separation and identification of 128 neutral flavor constituents from Cheddar cheese using GLC with a hydrogen flame detector and a capillary column. Additional evidence concerning the identity of the compounds was obtained with a time-of-flight mass spectrometer.

Identification of the materials that comprise the volatile fraction of the flavor is an important aspect of any flavor research. The mass spectrometer has been used by many researchers. Gohlke (30) showed that if the mass spectrometer was a time-of-flight instrument with a high rate of scan, then a direct connection can be made to a gas chromatograph at the point where the column effluent discharges. This method provides a means of identifying samples of complex flavor compounds that otherwise are virtually impossible to characterize. Modifications to improve the recording and sensitivity of the mass spectrometer were described by Ebert (26). Subsequently, McFadden et al. (52) analysed several flavor extracts using GLC and a capillary column connected to a mass spectrograph. Teranishi et al. (83) calculated the efficiencies of capillary columns used in GLC with several organic compounds to determine the performance under the vacuum normally encountered in a mass spectrometer.

The lack of the necessary instrumentation to assist in identifying the compounds elucidiated by GLC requires the use of other methodology. Miwa (57) listed the methods of identifying peaks found in the GLC of organic compounds:

1) Identification of peaks by means of retention data from known compounds.

2) The use of auxiliary detection systems, such as, mass spectrometry or electron capture detectors.

3) The collection of the effluent as the individual peaks emerge from the column and identification of the compound by chemical and physical methods.

Some of the classical methods of organic chemistry can be applied to many of the flavor components to elucidate chemical structure. Libbey and Day (49) used 2,4-dinitrofluorobenzene to isolate methyl mercaptan as the dinitrophenyl-sulfide derivative from Cheddar cheese. Several researchers (23, 63, 87) have studied the volatile carbonyls from Cheddar cheese by complexing these aldehydes and ketones with 2,4-dinitrophenylhydrazine. Bassette, Özeris and Whitnah (6) showed that headspace gases were essentially free of carbonyls, esters and carbonyls, and sulfides when the liquid sample was treated with acidic hydroxylamine, basic hydroxylamine and mercuric chloride, respectively. Hoff and Feit (39) determined the functional groups of many organic compounds by means of reactions conducted in the syringe prior to injection of the liquid sample into the gas chromatograph. This technique assisted in the identification of alcohols, aldehydes, ketones, esters, ethers, alkenes, alkanes and cyclic compounds. Beroza and Acree (10) described a method whereby the compound in question is altered to the parent hydrocarbon and possibly the next lower homolog. The position of any functional group can be determined from the fragmentation pattern obtained by GLC. Walsh and Merritt (88) listed many reagents that can be used for functional group analysis.

The usual method of tentative identification of organic compounds is by means of retention volumes. Merritt and Walsh (55) showed that identification of the unknown can be established with columns coated with liquid phases of opposite polarity and plots of carbon number against the logarithm of retention time.

EXPERIMENTAL PROCEDURES

The purpose of these experiments was threefold:

1) to investigate the conditions that are optimum for spray-drying natural cheese,

2) to study the keeping quality of spray-dried natural cheese held under various conditions of storage, and

3) to determine the loss of volatile flavor constituents during preparation of the spray-dried cheese powder.

Selection of Cheese and Preparation of the Samples

Cheddar cheese, ripened to various ages, was selected to ascertain the optimum age for dispersing with water. For economical operation of a commercial spray drier, the total solids concentration of the liquid feed should be greater than 36 per cent. However, viscosity of the fluid to be pumped is a limiting factor. Consequently in this research, a concentration of 40 per cent total solids was arbitrarily selected as an optimum dilution. When the slurries of Cheddar cheese were prepared, heated and homogenized, a smooth liquid resulted that was suitable for pumping to the drier.

Eight pound lots of cheese were comminuted, slurried with tap water to 40 per cent total solids and heated to 120 F. Other slurries of Cheddar cheese were prepared with 2 per cent sodium citrate and 0.5 per cent sodium chloride added to permit the slurries to be heated in excess of 125 F for determination of an optimum preheat treatment. Later trials involved preparing slurries from 93 score medium aged Cheddar cheese, adding 2 per cent sodium citrate and 0.5 per cent sodium chloride, heating the emulsified slurries to 180 F, and holding 10 min. All slurries were homogenized at 2000 psig—two stage with a Manton-Gaulin homogenizer.

Spray-drying of Natural Cheese

The slurries prepared from Cheddar cheese were temperature conditioned to 120 F before feeding to the Manton-Gaulin high pressure pump. Subsequent trials which involved slurries preheated above 140 F, were temperature conditioned to 140 F prior to pumping. The slurries were atomized with Spraying Systems nozzles into a Rogers co-current inverted tear-drop drier. Two types of nozzles were used during these trials—an SX high pressure nozzle and an SBC low pressure nozzle.

A commercial operation was simulated by in-flight collection of the samples of spray-dried Cheddar cheese in wooden boxes 6 x 12 x 24 inches lined with Kraft paper. These boxes were placed on the catwalk in the drier. The dried particles dropped from the air stream into these boxes and were protected from the detrimental effects of the heated atmosphere encountered by the particles adhering to the walls of the drier. The latter powders were collected by sweeping the walls of the drier at the conclusion of each trial. All powders were stored in polyethylene bags at 40 F until analysed or canned.

The slurries that were foam spray-dried were sparged with water-pumped nitrogen injected at the rate of 1 ft^3/min into the mixing cylinder. At this rate of feed, 2 ft^3 of nitrogen were mixed with each gallon of concentrate. The mixing cylinder was located between the high pressure pump and the atomizing nozzle.

In order to conduct drying trials using the nitrogen sparging technique, several modifications of the standard equipment existing in the M.S.U. Dairy Plant were necessary. A Cherry-Burrell Super Homogenizer was used as a high pressure pump and was equipped with a variable speed motor to permit variation of the rate of pumping. The mixing cylinder was fabricated from type 304 stainless steel tubing 2 x 12 inches with a 1/4 inch wall thickness. The ends of the cylinder were threaded (Acme - 8 threads/inch) and fitted with 1 1/2 inch female caps (Cherry-Burrell no. 16 AI-151), teflon gaskets (Cherry-Burrell no. 40-IT) and nickel alloy nuts (Cherry-Burrell no. 13-H). The caps were drilled and tapped on center 1/4 inch National Pipe Thread (NPT). The gas inlet in the mixing cylinder was drilled and tapped 1/4 inch NPT and located 2 inches above the base of the cylinder. A 1/4 x 3 inch type 304 stainless steel pipe nipple with a stainless steel check valve (Matheson Company no. 401) was threaded in this inlet. The gas pressure was regulated with a Victor high pressure gas-dome regulator, and the rate of gas flow was determined with a Brooks high pressure rotameter. The check valve located between the rotameter and mixing cylinder was used to prevent backflow of the liquid product into the gas regulating equipment. Figure 1 shows the apparatus that was used for foam spray-drying.

Preparation for Storage

No. 211 x 400 cans were packed with the samples of cheese powder and sealed on an Automatic Can-Sealer. These cans were dipped in a Wood's metal and lactic acid bath to protect againstpossible leackage of atmospheric oxygen through the rolled seams. Some cans were air-packed while the other cans were punctured, exhausted under 200 μ pressure for 30 minutes, flooded with water-pumped nitrogen and resealed with solder. After 48 hours this process of gas packing was repeated to permit the removal of any interstitially trapped oxygen that had equilibrated with the essentially inert atmosphere. All canned samples were stored at room temperature.

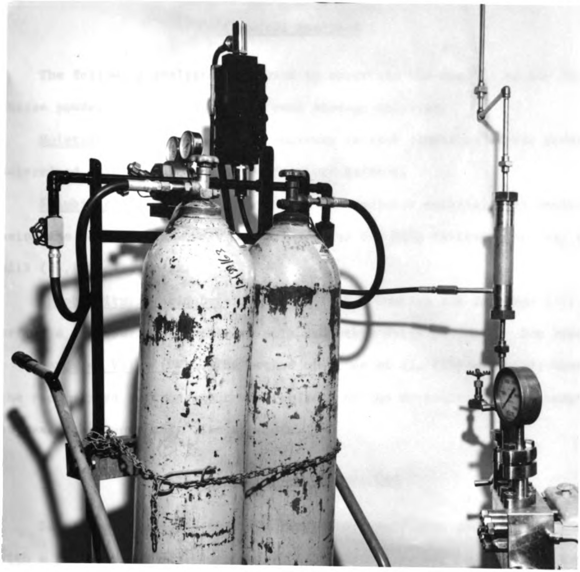

Figure 1. Equipment used for high pressure gas injection during spray-drying.

ANALYTICAL PROCEDURES

Chemical Analyses

The following analyses were used to ascertain the quality of the Cheddar cheese powder initially and after each storage interval.

Moisture. The percentage of moisture in each sample of cheese powder was determined with a Cenco Infra-red Moisture Balance.

Solubility Index. The milliliters of insoluble material were measured using the method recommended by the American Dry Milk Institute for dry whole milk (2). See appendix.

Thiobarbituric Acid Determination (TBA). Dunkley and Jennings (25) described a procedure applicable to milk and other dairy products. See appendix.

Peroxide Value (PV). The method of Stine et al. (78) was used; however, the recommended surface agent was replaced by the de-emulsification reagent suggested by Pont (64). See appendix.

Analysis of the Headspace Gas

Samples of headspace gas were removed directly from the sealed containers with a gas sampling device, a 5 inch 27 gauge hypodermic needle and a Hamilton gas-tight syringe. Two rinsings of the syringe were necessary to give reproducible results. The gas sampling device used for this analysis was described by Bradley and Stine (12) and is shown in Figure 2. With the can held firmly in the device, a vacuum was drawn with a vacuum pump between the can top and the valve. The valve was closed and the piercing tip forced into the top of the can. The needle on the syringe was inserted into the can through the internally contained hypodermic needle. The oxygen and nitrogen content of each gaseous sample was determined using a Perkin-Elmer model 154-B gas

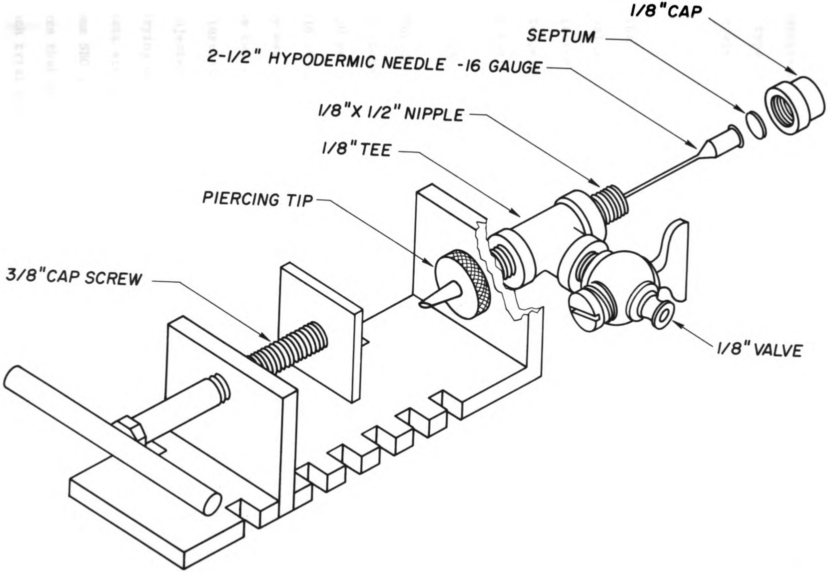

Figure 2. A detailed drawing of gas sampling device

chromatograph and a 1/4 inch x 2 m copper column packed with 30 to 60 mesh Linde type 13 A molecular sieve. Standard curves were prepared using atmospheric air as the reference gas.

Flavor Evaluation

The flavor quality of each sample of spray-dried Cheddar cheese powder was evaluated concomitant with the chemical analyses. The powders were reconstituted to 40 per cent total solids with tap water, allowed to stand at room temperature for at least 30 minutes and then judged for organoleptic flavor quality by qualified individuals.

Gas Chromatographic Analysis of Samples of Cheddar Cheese

The Cheddar cheese slurries sampled during manufacture of the cheese powder contained 40 per cent total solids, 0.5 per cent sodium chloride and 2.0 per cent sodium citrate. These slurries were heated to 120 and 180 F, held 10 min at these respective temperatures and homogenized. Each slurry was divided into two lots before spray-drying. One lot from each of the two slurries was spray-dried by conventional means and the remaining two slurries were foam spray-dried. During foam spray-drying nitrogen gas was injected at the rate of 2 ft^3/gal of concentrate. In all trials during the drying of these samples, the operating conditions were held constant. The exit air temperature in the drier was maintained at 160 F and a Spraying Systems SBC low pressure nozzle was used to atomize each slurry. The powders that were collected by sweeping the walls of the drier at the end of each trial were bagged and immediately refrigerated at 40 F.

Natural Cheddar cheese and the samples of Cheddar powder were diluted to 40 per cent total solids with distilled water and mixed to a homogeneous slurry in a Waring blendor. A 391 g sample of each of these slurries and the slurries prepared for drying were weighed into a 2 liter vacuum flask.

The vacuum flask containing the cheese slurry was attached to a modified Buchler Rotary Evaporator. Figure 3 shows a picture of the high vacuum apparatus used in the study. A vacuum of 10^{-3} Torr measured with a Pirani gauge was attained in this sealed system, using a mechanical vacuum pump and a mercury vapor pump. The cheese mixture contained in the flask was warmed for 4 hours in a water bath at 150 F during the vacuum distillation. Vapors evolved from the cheese slurries were collected in 3 traps. Water was removed from the distillate by means of a trap immersed in ethanol and dry ice, while the flavor volatiles were primarily condensed in the first of two cold finger traps immersed in liquid nitrogen. The second cold finger was used to prevent back-streaming of mercury from the mercury vapor pump.

At the end of each sampling interval, the vacuum was released and the first trap immersed in liquid nitrogen was stoppered and warmed to 70 F in a water bath. A 7.5 μ-liter aliquot of the fluid contents in the trap was withdrawn with a Hamilton microliter syringe and injected into the F and M (model 500) gas chromatograph equipped with a hydrogen flame detector (model 1609). A 150 foot stainless steel capillary column (inside diameter-0.02 inches) coated with Apiezon L grease was used in this study. Figure 4 shows the F and M instrumentation used to elucidate the volatile flavor materials in the cheese. The conditions of operation of the gas chromatograph were as follows:

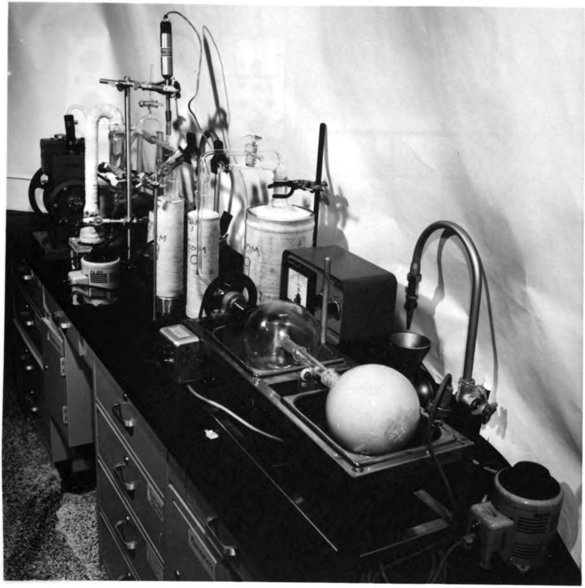

Figure 3. Apparatus used for high vacuum distillation of cheese volatiles.

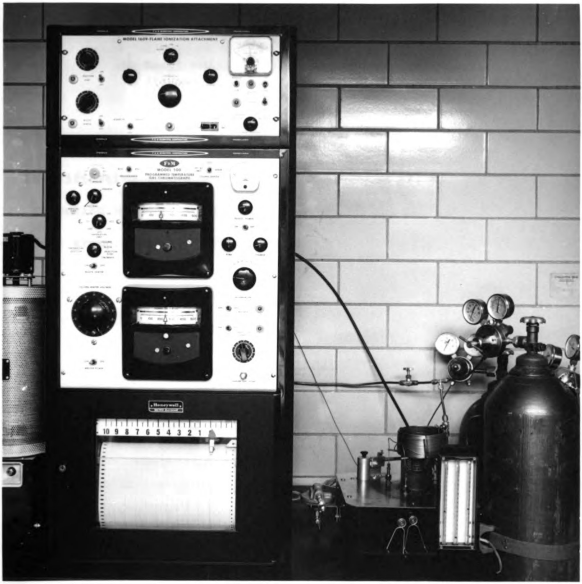

Figure 4. F and M gas chromatograph with flame ionization attachment.

Injection port temperature, C	150
Detector block temperature, C	275
Helium flow, ml/min	4
Hydrogen flow, ml/min	37
Air flow, ml/min	80
Range setting	1
Attenuation	16
Column temperature	
5 min hold at 35 C then programmed 35 to 185 C at 2.9 C/min	

Identification of the peaks shown in each chromatogram was accomplished using retention volumes of known volatile compounds and the technique of functional group analysis proposed by Bassette, Özeris and Whitnah (6).

RESULTS

Conventional Spray-Drying of Natural Cheese

The effect of inert additives. Inert additives such as corn starch, crystalline cellulose, soybean protein hydrolysate, Cheddar cheese whey and food grade lactose were added during the preparation of the cheese slurries in an attempt to protect the fat in the cheese powder from the deteriorative effects of oxidation. The slurries were spray-dried and the results of the analyses on these powders (Table 1) show that none of these additives contributed beneficially to the final quality of the dried products. The sizable increase in the PV of samples containing additives was generally reflected in the flavor criticisms. After 2 months storage in sealed cans that were air-packed or double gassed with nitrogen, the PV and TBA values increased. Furthermore, 1.5 per cent soybean protein hydrolysate contributed a noticeable off-flavor to the cheese powder. The results in Tables 1 and 2 indicate that the control samples were also poor and unacceptable in each trial after 2 months storage. The addition of 10 per cent gum arabic to the Cheddar slurries (Table 2) caused no improvement in the quality of the powder, whereas, 5 per cent starch and 5 per cent crystalline cellulose added to the cheese slurry imparted some protective influence on flavor quality. Whether the starch and cellulose actually protected the fat from oxidation or diluted the stale flavor materials in the powder is debatable.

The dry blending of crystalline cellulose and corn starch with Cheddar cheese resulted in a free-flowing product, but these products molded readily on storage because of high moisture content. The flavor was poor and the powder produced objectionable tactual sensations in the mouth. The poor flavor was attributed in part to the dilution of the cheese volatiles by the inert additive.

Table 1. The effect of additives on the quality of spray-dried cheese

Analyses	Control	Additives: 1.5% soybean protein hydrolysate	Additives: Cheddar whey (2% lactose)	Additives: 2% lactose
	Initial Analyses			
Moisture, %	2.4	2.3	2.3	2.3
TBA[a]	0.03	0.05	0.03	0.04
PV[b]	0.68	1.0	1.4	1.3
Flavor criticism	sl. stale	foreign	sl. stale	sl. stale
	2 months, nitrogen-packed			
Moisture, %	2.4	2.3	2.3	2.3
TBA[a]	0.06	0.10	0.08	0.07
PV[b]	0.71	0.85	9.0	6.3
Flavor criticism	v. sl. stale	v. sl. stale	sl. stale	stale
	2 months, air-packed			
Moisture, %	2.4	2.3	2.3	2.3
TBA[a]	0.06	0.09	0.07	0.06
PV[b]	0.86	0.86	4.7	7.4
Flavor criticism	stale	stale, soya flavor	stale, musty	stale
	Drying Conditions			
Inlet air temperature, F	215	215	214	212
Exit air temperature, F	160	160	160	160
Nozzle	SX	SX	SX	SX
Core	20	20	20	20
Orifice diameter, inch	0.038	0.038	0.038	0.038
Atomization pressure, psig	2500	2500	2500	2500

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

Table 2. The effect of additives on the quality of spray-dried Cheddar cheese.

Analyses	Control	10% Gum Arabic	Control	5% starch 5% crystalline cellulose
	Initial Analyses			
Moisture %	2.2	2.8	2.2	2.6
Solubility index, ml	4.0	4.2	4.8	6.0
TBA[a]	0.08	0.12	0.07	0.10
PV[b]	0.28	0.39	0.46	0.58
Flavor criticism	sl. stale sl. astringent	sl. stale sl. astringent sharper	sl. stale	sl. stale
	Nitrogen-Packed			
	2 months		3 months	
Moisture, %	2.4	2.9	2.7	3.0
Solubility index, ml	4.6	4.5	5.1	5.9
TBA[a]	0.12	0.16	1.3	1.2
PV[b]	1.5	4.0	0.55	0.32
Flavor criticism	sl. stale fruity	sl. stale, flat	sl. stale	v. sl. stale
	Air-Packed			
	2 months		3 months	
Moisture, %	2.5	2.9	2.2	2.2
Solubility index, ml	4.5	4.5	5.1	6.0
TBA[a]	0.21	0.26	1.2	1.1
PV[b]	1.2	1.7	0.86	0.49
Flavor criticism	sl. stale oxidized	sl. stale flat	stale, poor, fishy	sl. stale
	Drying Conditions			
Inlet air temperature, F	190	190	190	190
Exit air temperature, F	160	160	160	160
Nozzle	SX	SX	SBC	SBC
Core	17	17	standard	standard
Orifice diameter	0.040	0.040	0.040	0.040
Atomization pressure, psig	1500	1500	1000	1000

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

The Effect of Antioxidants. Commercial solutions of butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PG), citric acid (CA) and nordihydroguaiaretic acid (NDGA) (see appendix) were added in several combinations to Cheddar cheese slurries to retard the extensive lipid oxidation that occurred during spray-drying and subsequently in storage (Tables 3 and 4). Initially, the samples of cheese powder showed no increase in oxidative stability with the addition of 0.06 per cent primary antioxidant to the fat. During storage for 4 months in nitrogen-packed cans, the powders containing added antioxidant remained stable as indicated by the PV determination. The TBA values on these powders increased substantially from the results observed initially. The samples of cheese powder that were air-packed and stored 4 months at room temperature demonstrated the protective influence of the mixtures of BHA, BHT, PG and CA; BHA, PG and CA; and NDGA. The foreign off-flavor observed in the nitrogen-packed samples containing NDGA was not perceptible in the air-packed samples. Limited protection was afforded by the blends of PG and CA, and BHA and BHT that were added to the cheese slurries during manufacture.

The Effect of Particle Size. Cheese slurries were prepared, heated to 120 F, homogenized and atomized through SX nozzles with orifice inserts of different diameters. This procedure was employed to determine if any improvement in the flavor and storage quality of the powder was obtained when conditions of atomization were adjusted to favor formation of larger particles. The pressure used to atomize each cheese slurry was adjusted with each orifice diameter to give equivalent flow rates in gallons per hour. The initial flavor scores (Tables 5 and 6) indicated that the powders possessing the larger particle sizes were superior to cheese powders with smaller particle size. The PV and TBA values show poor correlation between particle size

Table 3. The effect of antioxidants on the quality of spray-dried Cheddar cheese.

Antioxidants	PV[a]	TBA[b]	Flavor criticism
	Initial analyses		
Control	0.56	0.02	stale, chalky
PG and CA	0.61	0.02	sl. stale
BHA and BHT	0.84	0.03	stale
BHA, BHT, PG and CA	0.74	0.03	sl. stale
BHA, PG and CA	0.51	0.03	sl. stale
NDGA	0.56	0.03	sl. stale
	4 months, Nitrogen-packed		
Control	1.8	0.09	stale
PG and CA	0.69	0.07	fruity, sl. stale
BHA and BHT	0.72	0.05	sl. stale
BHA, BHT, PG and CA	0.70	0.05	sl. stale
BHA, PG and CA	0.71	0.07	sl. stale
NDGA	0.54	0.05	sl. stale, foreign
	4 months, Air-packed		
Control	1.6	0.06	stale, poor
PG and CA	1.0	0.05	sl. stale
BHA and BHT	1.2	0.04	sl. stale
BHA, BHT, PG and CA	0.41	0.05	sl. stale
BHA, PG and CA	0.71	0.05	sl. stale
NDGA	0.31	0.05	sl. stale
	Drying Conditions		
Inlet air temperature, F	215-210		
Exit air temperature, F	160		
Nozzle type	SX		
Core	20		
Orifice diameter, inch	0.038		

[a] expressed as meq O_2/kg fat
[b] expressed as absorbancy

Table 4. The effect of antioxidants on the quality of spray-dried Cheddar cheese.

Antioxidants	PV[a]	TBA[b]	Flavor criticism
	Initial analyses		
Control	0.67	0.04	sl. stale
PG and CA	0.97	0.04	v. sl. stale
BHA and BHT	0.89	0.04	v. sl. stale
BHA, BHT, PG and CA	0.90	0.03	sl. stale
BHA, PG and CA	0.81	0.04	v. sl. stale
NDGA	0.93	0.04	v. sl. stale
	3 months, nitrogen-packed		
Control	0.79	0.08	chalky, stale
PG and CA	0.83	0.08	sl. stale, flat
BHA and BHT	0.64	0.06	sl. stale
BHA, BHT, PG and CA	0.53	0.07	sl. stale
BHA, PG and CA	0.58	0.06	sl. stale
NDGA	0.63	0.10	foreign, sl. stale
	3 months, air-packed		
Control	1.2	0.09	oxidized, stale
PG and CA	1.1	0.07	sl. stale
BHA and BHT	0.86	0.06	sl. stale
BHA, BHT, PG and CA	0.57	0.07	sl. stale
BHA, PG and CA	0.47	0.07	sl. stale
NDGA	0.59	0.07	sl. stale
	Drying conditions		
Inlet air temperature, F	215-210		
Exit air temperature, F	160		
Nozzle	SX		
Core	20		
Orifice diameter, inch	0.038		

[a] expressed as meq O_2/kg fat
[b] expressed as absorbancy

Table 5. The effect of orifice diameter on the quality of spray-dried Cheddar cheese.

Analyses	Orifice diameter, inch			
	0.038	0.040	0.052	0.055
	In-flight			
Moisture, %	2.6	2.5	3.0	2.8
TBA[a]	0.04	0.04	0.04	0.05
PV[b]	0.28	0.48	0.30	0.65
Flavor	sl. chalky	sl. chalky	fair	fair
	Swept			
Moisture, %	2.5	2.7	2.5	2.8
TBA[a]	0.05	0.04	0.04	0.05
PV[b]	0.95	1.0	0.86	1.2
Flavor	poor,	stale	sl. stale	sl. stale
	2 months, nitrogen-packed			
TBA[a]	0.09	0.08	0.08	0.07
PV[b]	1.2	1.0	0.70	0.41
Flavor	sl. oxidized sl. stale	sl. oxidized sl. stale	sl. bitter sl. stale	sl. bitter sl. stale
	2 months, air-packed			
TBA[a]	0.08	0.09	0.08	0.10
PV[b]	1.1	0.90	1.4	1.5
Flavor	sl. bitter sl. stale	sl. bitter sl. stale	sl. bitter sl. stale	sl. bitter sl. stale
	Drying Conditions			
Inlet air temperature, F	205	198	196	195
Exit air temperature, F	160	160	160	160
Nozzle	SX	SX	SX	SX
Core	20	17	17	17
Atomization pressure, psig	2500	1500	1000	1000

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

Table 6. The effect of orifice diameter on the quality of spray-dried Cheddar cheese.[a]

Analyses	Orifice diameter, inch			
	0.038	0.040	0.052	0.055
Moisture, %	2.6	2.8	2.9	2.9
TBA[b]	0.03	0.03	0.03	0.03
PV[c]	0.71	0.84	0.88	0.84
Flavor	sl. stale	sl. stale	v. sl. stale	v. sl. stale
Drying Conditions				
Inlet air temperature, F	220	220	220	220
Exit air temperature, F	160	160	160	160
Nozzle	SX	SX	SX	SX
Core	20	17	17	17
Atomization pressure, psig	2500	1700	1500	1300

[a] initial analyses
[b] expressed as absorbancy
[c] expressed as meq O_2/kg fat

and flavor score initially and also after a 2 month storage period (Table 5). The effect of the stale flavor appears to mask any benefit derived from the larger particle sizes.

The effect of reducing the percentage of fine particles. Two types of atomizing nozzles obtained from Spraying Systems, Inc. (an SX nozzle and an SBC nozzle) were used to demonstrate the effect of fine particles on increasing the amount of stale flavor in the cheese powders. The pump pressure was adjusted to give an equivalent flow rate with the SBC low pressure nozzle since particle size was the only variable desired in this experiment. The results in Table 7 indicate that the powder obtained by atomizing the cheese slurry through an SBC nozzle, which minimizes the proportion of fine particles, was superior initially in comparison to the powder obtained by atomizing through the SX nozzle. Apparently, the reduction in surface area of the powder containing the low percentage of fine particles reduced the amount of flavor volatiles lost during drying, and resulted in powder of higher moisture content and lower PV. After storage for 2 months in nitrogen and air-packed cans, the cheese powders that were double gassed with nitrogen retained good flavor and oxidative stability, while the powders that were air-packed deteriorated considerably. The extent of lipid oxidation in the cheese powders is indicated by the relatively high TBA values. Low correlation again existed between the flavor of the powders and the PV and TBA values. Generally, a high TBA or PV in a food containing fat would be indicative of perceptible oxidation that was demonstrated in these storage trials.

The effect of exit air temperature. Slurries of Cheddar cheese were prepared, heated to 120 F, homogenized and spray-dried at various exit air temperatures to determine the effect of different drying temperatures on the quality of the cheese powders. The results of the analyses show that the

Table 7. The effect of a reduction in the percentage of fine particles on the quality of spray-dried Cheddar cheese.

Analyses	Type of nozzle: SX	SBC
In-flight, initial		
Moisture, %	2.4	3.2
Solubility index, ml	4.0	4.0
TBA[a]	0.18	0.16
PV[b]	0.16	0.12
Flavor	sl. stale	v. sl. stale
Swept, initial		
Moisture, %	2.2	2.8
Solubility index, ml	4.0	4.1
TBA[a]	0.08	0.09
PV[b]	0.28	0.21
Flavor	sl. stale	v. sl. stale
2 months, air-packed		
Moisture, %	2.5	2.9
Solubility index, ml	4.5	4.1
TBA[a]	0.21	0.28
PV[b]	1.2	3.4
Flavor	sl. stale, oxidized	sl. stale, astringent
2 months, nitrogen-packed		
Moisture, %	2.8	3.2
Solubility index, ml	4.6	4.1
TBA[a]	0.12	0.14
PV[b]	1.5	1.1
Flavor	sl. stale, fruity	sl. stale
Drying Conditions		
Inlet air temperature, F	192	190
Exit air temperature, F	160	160
Nozzle	SX	SBC
Core	17	standard
Orifice diameter	0.040	0.040
Pump pressure, psig	1500	800

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

degree of heat damage increases with exit air temperature (Table 8). The flavor of these samples varied from "very slightly stale" and "fair" in the samples dried at an exit air temperature of 150 and 160 F, respectively, to "stale" in the powder spray-dried at an exit air temperature of 190 F. The lowest feasible exit air temperature that gave a dry product, limited adhering of particles to the wall of the drier, and the least amount of staleness was 160 F. The PV of the samples both initially and after 3 months storage increased with the temperature of the exit air. The sample dried at an exit air temperature of 150 F had high PV and TBA values that were attributable to the fact that this sample of powder adhered to the walls of the drier and received excessive exposure to the hot drying air.

The effect of preheat treatment. Cheese slurries containing 40 per cent total solids, 0.5 per cent sodium chloride and 2.0 per cent sodium citrate were heated to temperatures of 120, 150, 170, 180 and 190 F to ascertain an optimum preheat temperature. Each slurry was held at the respective temperature for 10 min, homogenized and spray-dried at an exit air temperature of 160 F through the Spraying Systems SBC nozzle. The results are shown in Table 9. The powder prepared by preheating the slurry to 180 F for 10 min possessed a lower PV initially and throughout the 5 months storage interval than any of the other samples of cheese powder in this trial. Moreover, this sample had more Cheddar flavor and less staleness perceptible than the other samples. The TBA value of the sample of cheese powder manufactured from a slurry preheated to 180 F was higher throughout storage than the TBA value of the samples preheated during manufacture to 120 and 170 F except in the air-packed samples. The flavor scores and the TBA values showed poor correlation. Since a preheat of 180 F for 10 min appeared optimum, all subsequent slurries were given this treatment prior to spray-drying.

Table 8. The effect of exit air temperature on the quality of spray-dried Cheddar cheese.

Analyses	Exit air temperature, F				
	150	160	170	180	190
			Initial		
Moisture, %	2.8	2.5	2.4	2.3	2.3
TBA[a]	0.03	0.03	0.03	0.03	0.03
PV[b]	0.67	0.24	0.57	0.69	0.62
Flavor	v. sl. stale	fair	v. sl. stale	v. sl. stale	stale
			3 months, nitrogen-packed		
TBA[a]	0.08	0.08	0.08	0.06	0.08
PV[b]	0.65	0.64	0.74	0.70	0.67
Flavor	sl. stale	sl. stale	sl. stale	sl. stale	sl. stale
			3 months, air-packed		
TBA[a]	0.08	0.07	0.08	0.09	0.08
PV[b]	0.71	0.62	0.66	0.70	0.53
Flavor	sl. stale	sl. stale	sl. stale	sl. stale	sl. stale
			Drying Conditions		
Inlet air temperature, F	210	215	220	228	236
Nozzle	SX	SX	SX	SX	SX
Core	20	20	20	20	20
Atomization pressure, psig	2500	2500	2500	2500	2500

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

Table 9. The effect of heating Cheddar cheese slurries to various temperatures on the quality of spray-dried Cheddar cheese.

Analyses	Heat treatment, F 120	170	180	190
	In-flight, initial			
Moisture, %	2.4	2.5	2.7	2.8
Solubility index, ml	4.3	5.0	6.0	7.8
TBA[a]	0.10	0.13	0.14	0.14
PV[b]	0.50	0.50	0.43	0.51
Flavor	fair, sl. stale	fair	fair, more Cheddar flavor	fair, sl. grainy
	Swept, initial			
Moisture, %	1.9	2.1	2.4	2.6
Solubility index, ml	4.0	5.0	5.8	7.3
TBA[a]	0.09	0.08	0.12	0.13
PV[b]	1.0	0.85	0.75	0.86
Flavor	sl. stale	v. sl. stale	fair, v. sl. stale	fair, sl. grainy
	5 months, nitrogen-packed			
Moisture, %	3.0	3.2	3.3	3.2
Solubility index, ml	4.0	4.8	6.0	7.0
TBA[a]	0.09	0.10	0.12	0.06
PV[b]	0.53	0.45	0.39	0.51
Flavor	fair, v. sl. stale	fair, v. sl. stale	good	fair, v. sl. stale
	5 months, air packed			
Moisture, %	3.0	3.1	3.2	3.2
Solubility index, ml	4.3	4.5	6.5	7.5
TBA[a]	0.11	0.13	0.10	0.10
PV[b]	1.0	0.79	0.67	0.81
Flavor	fair	fair	fair	fair
	Drying Conditions			
Inlet air temperature, F	215	215	212	212
Exit air temperature, F	160	160	160	160
Nozzle	SBC	SBC	SBC	SBC
Core	standard	standard	standard	standard
Orifice diameter, inch	0.040	0.040	0.040	0.040

[a] expressed as absorbancy

[b] expressed as meq O_2/kg fat

The results of the preheat treatments (Table 10) support the evidence shown in Table 9 in the respect that flavor and oxidative stability increase with higher preheat temperatures. The solubility index (Tables 9 and 10) increased with each increase in preheat treatment.

Foam Spray-Drying of Natural Cheese

The effect of the type of spray-drying. Previous results (Tables 5, 6 and 7) demonstrate that the quality of spray-dried cheese was improved by spray-drying to obtain a larger average size of particles. To determine if an additional increase in particle diameter would improve the quality of the cheese powder, slurries were prepared, preheated to 180 F for 10 min, homogenized and spray-dried by conventional means and with 2 ft^3/min of nitrogen injected prior to atomization. The exit air temperature in this trial was 160 F. The analyses performed on these powders (Table 11) show that the foam spray-dried powder has increased flavor retention and a lower moisture content than observed in the powders that were dried by conventional means. The foam spray-dried powder exhibited none of the stale flavor that commonly masked the typical Cheddar flavor in cheese powders spray-dried conventionally. The bulk density of the foam-dried powder was approximately 50 per cent of that for the powder dried conventionally. This difference in bulk density is attributed to the fact that the nitrogen puffed the particles after atomization. The foam spray-dried powder was free flowing and exhibited no lumping. When these two cheese powders were sieved to determine the range of particle sizes, 77 per cent of the particles in the sample of foam spray-dried powder were collected in the 297 μ sieve, whereas, only 43 per cent of the particles from the cheese powder dried conventionally remained in the 297 μ sieve.

The effect of relative particle size. Slurries of Cheddar cheese were

Table 10. The effect of heating Cheddar cheese slurries to various temperatures on the quality of spray-dried Cheddar cheese.[a]

Analyses	Heat treatment, F		
	120	150	170
	In-flight		
Moisture, %	3.4	2.6	2.9
Solubility index, ml	4.3	2.8	6.3
TBA[b]	0.11	0.11	0.06
PV[c]	0.63	0.43	0.32
Flavor	sl. stale	fair	good
	Swept		
Moisture, %	3.0	2.2	3.0
Solubility index	4.3	4.5	5.0
TBA[b]	0.07	0.06	0.06
PV[c]	0.81	0.84	0.69
Flavor	sl. stale	fair	good
	Drying Conditions		
Inlet air temperature, F	190	190	185
Exit air temperature, F	160	160	160
Nozzle	SBC	SBC	SBC
Orifice diameter, inch	0.040	0.040	0.040
Atomization pressure, psig	1000	1000	1000

[a] initial analyses
[b] expressed as absorbancy
[c] expressed as meq O_2/kg fat

Table 11. The effect of the type of spray-drying on the quality of Cheddar cheese powder.[a]

Analyses	Type of spray-drying: Conventional	Foam
Moisture, %	3.4	3.1
Solubility index, ml	4.3	4.5
TBA[b]	0.11	0.13
PV[c]	0.63	0.53
Bulk density, g/ml	0.47	0.22
Flavor	good	good, more Cheddar flavor
Drying Conditions		
Inlet air temperature, F	190	190
Exit air temperature, F	160	160
Nozzle	SBC	SBC
Orifice diameter, inch	0.040	0.040

[a] initial analyses
[b] expressed as absorbancy
[c] expressed as meq O_2/kg fat

prepared, heated, homogenized and foam spray-dried using an SBC nozzle with orifice insert diameters of 0.040 and 0.047 inches to determine if the flavor and quality of the Cheddar cheese powder could be improved further by increasing the particle size. These powders were collected in-flight and by sweeping the walls of the drier at the end of the drying trials. The data (Table 12) show no significant differences in the results of analyses on the swept and in-flight samples other than slight differences in moisture content; therefore, in-flight collection was terminated. Initially, these samples had a good Cheddar flavor that was characteristic of foam spray-dried cheese powder. No improvement in flavor quality was observed due to the increase in particle size. The powder atomized through the larger orifice showed greater PV and TBA values due presumably to retention of particles on walls of the drier.

These powders were air-packed and sealed under nitrogen and analysed at intervals of 3 months over a 9 month period. The flavor of these cheese powders during storage showed no apparent deterioration. In fact, no difference was observed organoleptically in the air and gas-packed samples stored 3 months. The PV and TBA values in both of the samples show a gradual decrease during the 9 month storage interval while the percentage of moisture, solubility index and the oxygen concentration remained relatively constant.

The effect of antioxidants. Previous results (Tables 3 and 4) showed that antioxidants particularly NDGA and the mixture of BHA, BHT, PG and CA (Tenox 6) improved the storage stability of conventionally spray-dried Cheddar cheese powder. This trial was conducted to determine if the shelf-life of foam spray-dried Cheddar cheese also could be extended by the use of antioxidants. The addition of 0.06 per cent of primary antioxidant was used for observation of the effectiveness of this maximal concentration. These slurries were foam spray-dried and collected by sweeping the drier.

Table 12. The effect of relative size of particles on the quality of foam spray-dried Cheddar cheese

Analyses	Orifice diameter, inch 0.040	0.047
	In-flight	
Moisture, %	3.5	3.7
Solubility index, ml	4.8	4.9
TBA[a]	0.15	0.14
PV[b]	0.53	0.79
Flavor	good	good, sl. fruity
	Swept	
Moisture, %	3.0	3.4
Solubility index, ml	4.5	4.5
TBA[a]	0.13	0.14
PV[b]	0.53	0.70
Flavor	good	good
	3 months, nitrogen-packed	
Moisture, %	3.2	3.4
Solubility index, ml	5.0	5.0
TBA[a]	0.11	0.10
PV[b]	0.27	0.31
Headspace, % O_2	2.0	2.5
Flavor	good	good
	3 months, air-packed	
Moisture, %	2.8	3.0
Solubility index, ml	5.0	5.0
TBA[a]	0.13	0.12
PV[b]	0.48	0.46
Flavor	good	good
	6 months, nitrogen-packed	
Moisture, %	3.3	3.4
Solubility index, ml	5.0	5.0
TBA[a]	0.10	0.10
PV[b]	0.22	0.31
Headspace, % O_2	2.1	2.1
Flavor	good	good
	9 months, nitrogen-packed	
Moisture, %	3.3	3.4
Solubility index, ml	4.5	5.0
TBA[a]	0.08	0.08
PV[b]	0.18	0.17
Headspace, % O_2	2.1	2.1
Flavor	good	good
	Drying Conditions	
Inlet air temperature, F	210	210
Exit air temperature, F	160	160
Nozzle	SBC	SBC
Orifice diameter, inch	0.040	0.047

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

The data in Table 13 show no improvement initially from the effect of the added antioxidants. In fact, the sample containing the added BHA, BHT, PG and CA showed a greater initial PV than the other samples in the trial.

After storage for 3 and 6 month intervals at room temperature in nitrogen and air-packed cans, these powders were analysed again. The results of the analyses performed on these powders at the end of 3 months show that nitrogen packaging very slightly improved the storage stability. A moderate protective effect from the antioxidants is apparent since the PV and TBA values are somewhat lower for the samples containing added antioxidants than the control cheese powder that contained no added antioxidant. After 6 months storage the samples with antioxidants showed some off-flavor while the control sample maintained excellent flavor during this storage interval.

The effect of additional ripening of the cheese. Some cheese manufactures have stated that additional ripening of Cheddar cheese at room temperature before blending the cheese into a slurry will improve the flavor quality of the spray-dried powder. Two varieties of Cheddar cheese were selected (uncolored New York and colored Wisconsin). The blocks of cheese were cut and wrapped in foil. One portion of each block was refrigerated and the remaining portion of Cheddar cheese was conditioned at room temperature for 24 hours. The results of the analyses on these powders are shown in Table 14. The additional ripening at room temperature contributed to increases in the PV and TBA values and the solubility indexes of the cheese powders. The flavor evaluations indicated that the powder from the conditioned Wisconsin Cheddar had more flavor than the powder from the refrigerated sample. However, no distinction was made between the two samples of New York Cheddar powder.

Table 13. The effect of antioxidants on the oxidative stability of foam spray-dried Cheddar cheese.

Analyses	Control	Antioxidants added: BHA, BHT, PG and CA	Antioxidants added: NDGA
	Initial		
Moisture, %	3.3	3.4	3.4
Solubility index, ml	5.0	5.5	5.5
TBA[a]	0.15	0.13	0.14
PV[b]	0.57	0.81	0.56
Flavor	good	good	good, sl. sharpness
	3 month, air-packed		
Moisture, %	3.4	3.2	3.3
Solubility index, ml	4.5	5.5	4.8
TBA[a]	0.13	0.13	0.13
PV[b]	0.47	0.47	0.35
Flavor	good	good	good, sl. sharpness
	3 month, nitrogen-packed		
Moisture, %	3.4	3.3	3.4
Solubility index, ml	4.5	5.3	5.0
TBA[a]	0.13	0.12	0.12
PV[b]	0.42	0.36	0.31
Headspace, % O_2	2.1	2.1	2.1
Flavor	good	good	good, sl. foreign
	6 month, nitrogen-packed		
Moisture, %	3.4	3.4	3.3
Solubility index, ml	4.0	4.5	4.8
TBA[a]	0.15	0.14	0.13
PV[b]	0.33	0.26	0.23
Headspace, % O_2	2.1	2.2	2.1
Flavor	good	flat	good, sl. foreign
	Drying Conditions		
Inlet air temperature, F	200	200	200
Exit air temperature, F	160	160	160
Nozzle	SBC	SBC	SBC
Orifice diameter, inch	0.040	0.040	0.040

[a] expressed as absorbancy
[b] expressed as meq O_2/kg fat

Table 14. The effect of conditioning at room temperature on the quality of foam spray-dried Cheddar cheese.[a]

	Varieties of Cheddar cheese			
	New York		Wisconsin	
Analyses	Refrigerated	Conditioned	Refrigerated	Conditioned
Moisture, %	2.4	2.4	2.8	2.7
Solubility index, ml	7.0	9.5	4.8	6.0
TBA[b]	0.06	0.06	0.12	0.14
PV[c]	0.60	0.97	0.55	0.78
Flavor	*	*	good	**
	Drying Conditions			
Inlet air temperature, F	210	210	208	206
Exit air temperature, F	160	160	160	160
Nozzle	SBC	SBC	SBC	SBC
Orifice diameter	0.040	0.040	0.040	0.040

[a] initial analyses
[b] expressed as absorbancy
[c] expressed as meq O_2/kg fat

* - lacks Cheddar flavor, bland, v. sl. stale
** - good, sl. better than refrigerated

Gas Chromatographic Analysis of Cheddar Cheese Volatiles

Natural Cheddar cheese. The volatile flavor materials that were collected by high vacuum distillation from natural Cheddar cheese were separated by GLC using a capillary column and a hydrogen flame detector. The chromatogram of the volatiles collected is shown in Figure 5. A total of 44 peaks was elucidated. The concentration of low boiling compounds represented by peaks 5 through 18 and denoted by increased attenuation values shown under the number corresponding to the peak, is particularly apparent in the chromatogram. This chromatogram is used as a reference pattern when a comparison is made of the relative loss of volatile material observed in the chromatograms of the cheese slurries and powders. The upward slope of the baseline at higher temperatures is a function of programming at high sensitivity. The slope could be eliminated by using a dual flame detector. The data in Table 15 show the relative peak height from each of 7 chromatograms. Where no known compound corresponded to the functional group and retention data, then only an indication of the loss of the constituent can be obtained.

Cheese slurries. Samples of two Cheddar cheese slurries heated to 120 and 180 F for 10 min were submitted to vacuum distillation and a portion of the distillate collected in the liquid nitrogen trap was examined by GLC. Figures 6 and 7 show the results of these trials. The preheat treatment caused liberation of additional sulfide containing material shown by peaks 28, 39, 47 and 52. This process also contributed to the development of additional flavor materials that were not observed in the flavor spectrum from the natural cheese. Peaks 1, 3, 39, 47 and 51 represent new materials formed during heating. The total concentration of volatile constituents in the slurries was lower than that observed in the chromatograms of natural cheese.

Table 15. Relative peak height from gas chromatograms of Cheddar slurries, powder and natural cheese, cm

Peak No.	Retention time, min.	Identity of volatile	Natural cheese	Preheat treatment: 120 F for 10 min Slurry	FSD	CSD	Preheat treatment: 180 F for 10 min Slurry	FSD	CSD
1	2.7	?		0.2		0.5	0.1		
2	3.5	acetaldehyde	0.2	0.2	0.2	0.2	0.1		
3	4.3	ethyl formate or acetone		0.5	0.3	0.7	0.3		0.5
4	4.6	butanone or butanal	3.0	52.8	3.6	3.8	16.4	8.6	3.2
5	5.1	methyl propionate	166.0	11.1	13.8	13.2	67.6	23.6	11.7
6	5.3	?				13.5		17.2	12.2
7	5.5	?	112.0			19.4		14.0	10.0
8	5.7	n-butanol	16.0	16.2	8.0	8.0		14.1	
9	5.9	S-S	46.0	17.2	10.8	10.5		10.1	7.8
10	6.0	?	43.2					9.0	
11	6.2	ethyl acetate	44.0					11.6	
12	6.4	methyl butyrate	56.0					12.5	
13	6.5	S-S	163.0	32.0	17.2	16.0		21.0	15.4
14	7.3	?	20.4	13.6	10.6				
15	8.0	C=O	23.6	8.0	11.5	5.6	7.6		3.9
16	9.6	hexanal	7.2						
17	10.8	ethyl butyrate	16.6	5.5	7.0	4.5	5.4		
18	11.9	?	17.4		8.7	4.2	3.8	9.2	4.0
19	13.2	S-S, ester	6.8	2.9	3.5	4.4	2.5	3.2	3.6
20	14.6	propionic acid		1.8	2.0			1.5	
21	15.2	?		1.6	1.7			2.3	
22	15.7	heptanone	5.3		14.5		2.5	8.9	
23	16.2	?	4.1		9.9			12.3	
24	16.9	heptanal	3.2		3.3				
25	18.9	acetoin			2.1				
26	20.3	?			1.5				
27	20.5	?	2.9		2.6				
28	21.2	S-S	33.4	87.2	14.4		47.2		
29	21.5	?			10.1				2.9
30	23.0	?			8.8	3.2		10.0	5.9
31	23.8	?	5.9	5.3	6.5			6.3	4.4
32	24.7	octanone	5.4	3.9					
33	26.4	?	5.4	4.6	5.8	2.8	4.0	4.6	3.2
34	26.9	octanal	6.7	5.0	6.9	2.8	4.3	4.5	3.1
35	27.4	?	7.0		6.8				
36	28.4	?	7.6	5.2		3.2	4.4	5.1	3.2
37	29.1	?	9.2		7.3	3.1	5.1		3.4
38	29.4	ester			7.3	3.7		5.7	3.5
39	30.1	S-S		182.0	8.3	4.3	69.6		3.6
40	30.5	diacetyl	10.5	11.8	8.4	4.5	5.0	6.4	3.7
41	30.9	?	1.0	2.8	1.2	0.4		0.2	1.1
42	31.6	?	1.3	3.3	1.4	0.7		0.6	1.3
43	32.3	?	1.1	1.6	1.2	0.8			
44	33.2	S-S	33.2	2.2	7.1	2.1	1.0	7.3	1.2
45	34.4	?	1.7	1.6	1.6	1.0			
46	35.2	methyl caprylate	1.1						
47	36.2	S-S		60.8	12.7	4.3	39.6	8.7	4.0
48	37.6	?	0.7	1.2	1.8		1.2		1.1
49	39.8	?		1.3	0.6	1.0			
50	40.3	S-S	0.6	1.0	1.5		1.2	1.2	
51	40.9	?		1.2	0.9	1.3	0.8	0.7	
52	42.6	S-S	13.2	9.3	2.5	1.9	14.6	3.8	1.4
53	42.9	?	1.4	2.4					
54	43.5	undecanone		1.2	0.6	1.0		0.7	
55	44.1	?			0.5	0.9			0.7
56	44.6	?	1.5		0.3	0.8			
57	46.0	?			0.2				
58	46.5	S-S	0.9		1.5	1.4	1.1	0.7	1.6
59	49.2	?			0.2				0.5
60	49.8	?			0.4	0.6			
61	50.4	?			0.3	0.6			
62	52.2	?			0.2				
63	52.9	dodecanal			0.1				
64	54.6	?		0.2	0.4			0.3	
65	55.3	S-S	0.1	0.3	0.9		0.3	0.6	0.2
66	56.3	?			0.3				
67	60.5	?	0.5		0.2	0.2		0.6	0.2
68	63.7	?		0.2	0.1	0.1			

Abbreviations used: FSD, foam spray-dried; CSD, conventional spray-dried

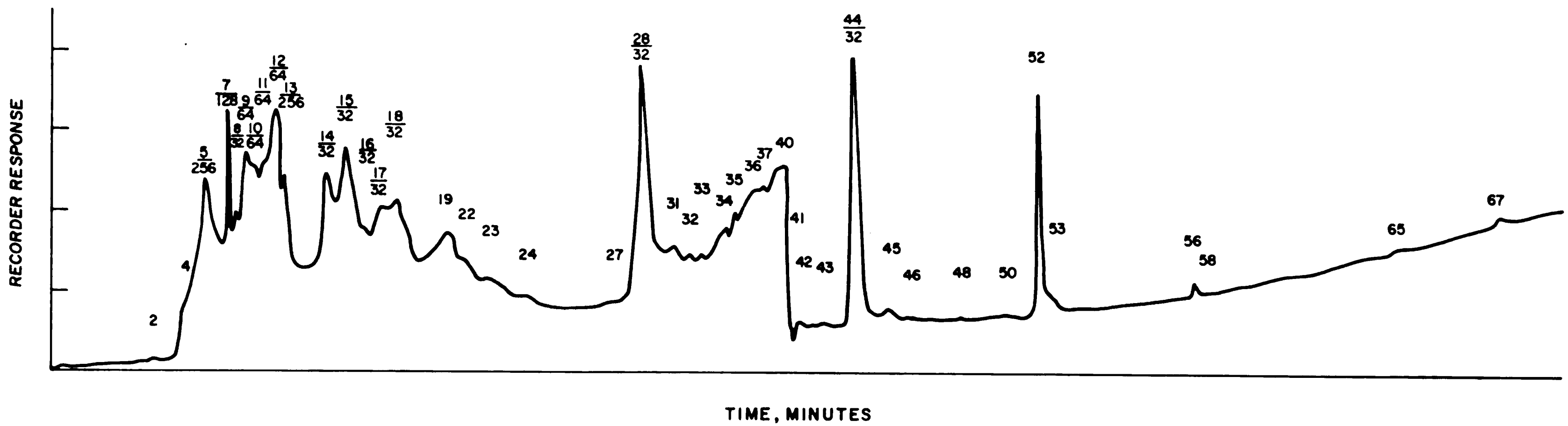

Figure 5. A gas chromatogram of the flavor from natural Cheddar cheese

(The term "flavor" used in the titles of Figures 5 through 11 refers to flavor volatiles collected by high vacuum distillation).

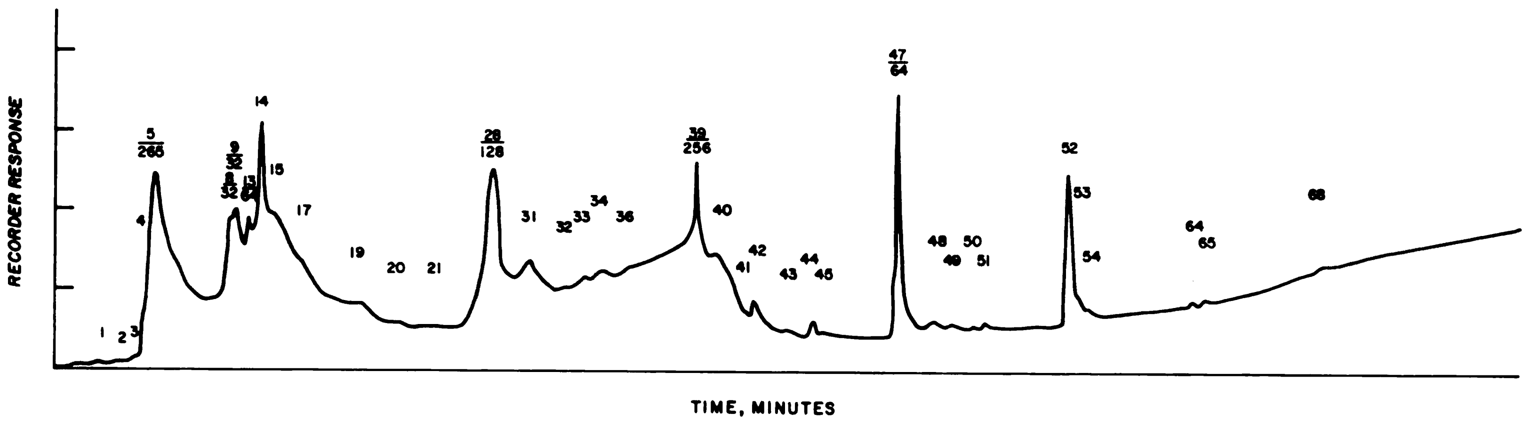

Figure 6. A gas chromatogram of the flavor from a Cheddar cheese slurry heated to 120 F.

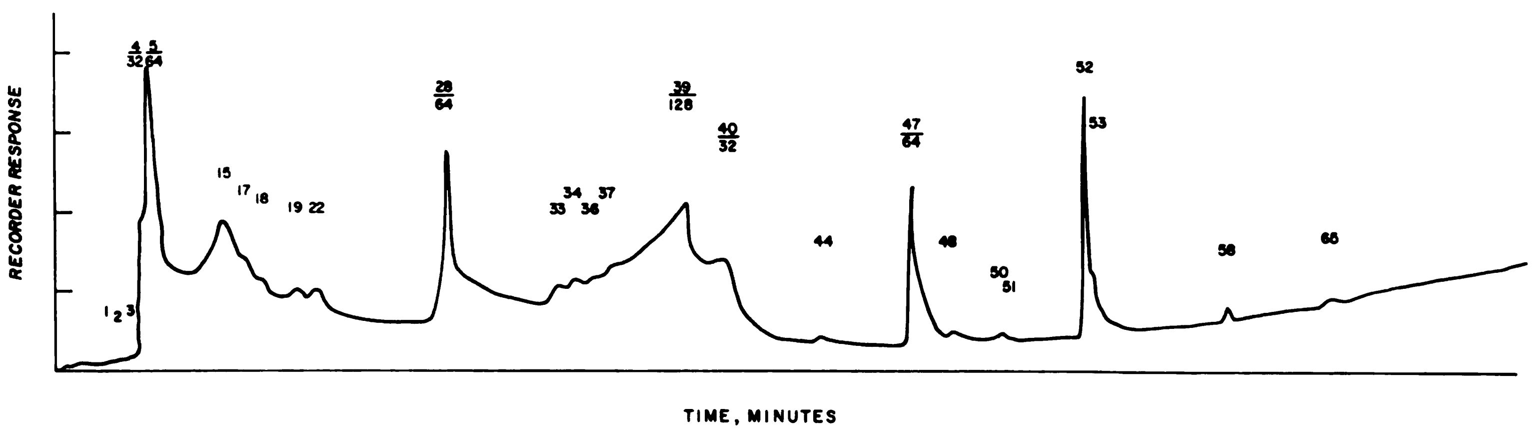

Figure 7. A gas chromatogram of the flavor from a Cheddar cheese slurry heated to 180 F.

Cheddar cheese powders. Figures 8 and 9 are the chromatograms of the vacuum distilled flavors from the Cheddar cheese powders preheated during preparation to 180 F and spray-dried by two methods. The chromatogram of the flavor in the cheese powder obtained by foam spray-drying shows a broad spectrum of flavor volatiles, most of which appear in much smaller concentrations in the chromatogram of the flavor in the powder spray-dried conventionally. The foam spray-dried powder contains a greater quantity of low boiling flavor constituents than observed with conventional powders.

A similar comparison of the flavor from Cheddar cheese powders is shown in Figures 10 and 11. These powders were manufactured by preheating the slurries to 120 F and spray-dried by two different techniques. These chromatograms also indicated that the foam spray-dried Cheddar cheese possessed a larger concentration of volatile flavor compounds. A comparison of the areas occupied by the low boiling constituents in the chromatograms shows a greater concentration of volatile material in the foam spray-dried powder than in the cheese powder dried conventionally.

A comparison of the chromatograms in Figures 9 and 11 shows some differences in the concentrations of volatiles. The chromatogram of the foam spray-dried cheese powder prepared by preheating the slurry to 180 F (Figure 9) shows a greater concentration of low boiling compounds, whereas, the chromatogram of the foam spray-dried powder preheated during manufacture to 120 F shows more volatile material in the area of peaks 37 to 47.

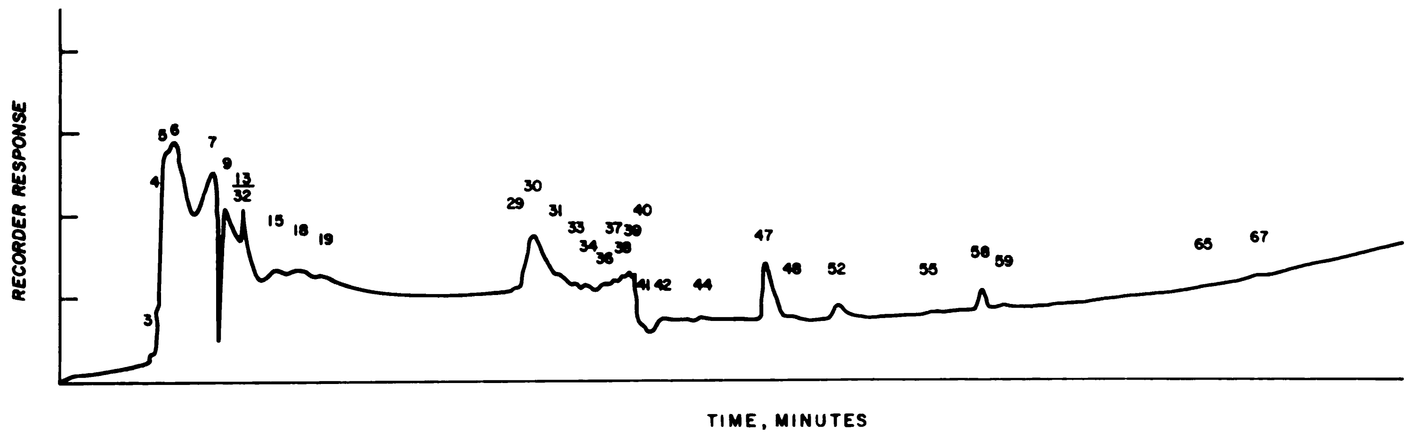

Figure 8. A gas chromatogram of the flavor frɒm spray-dried Cheddar cheese preheated to 180 F.

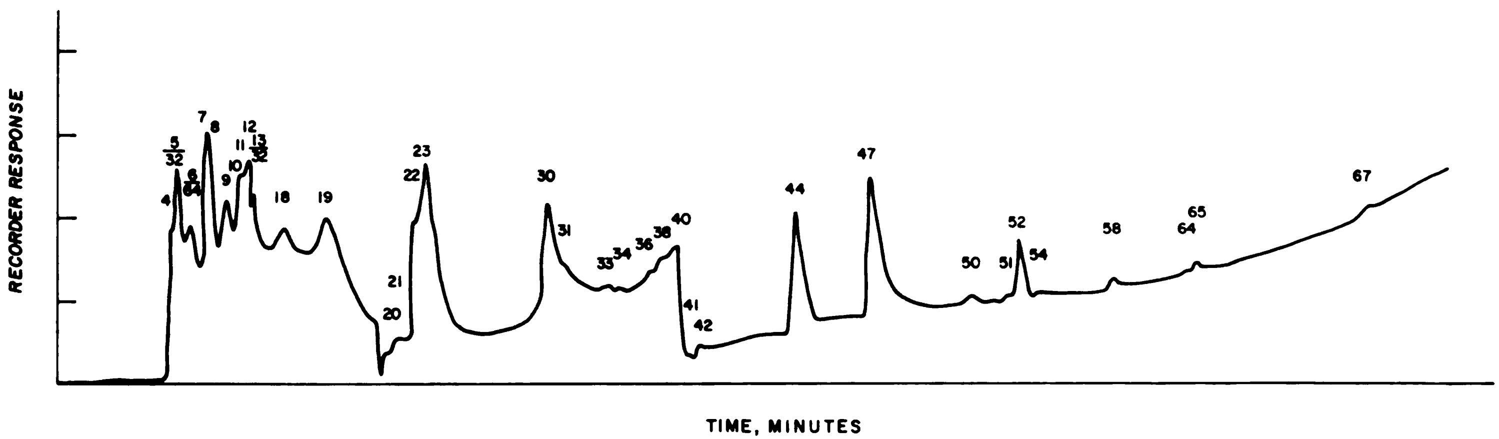

Figure 9. A gas chromatogram of the flavor from foam spray-dried Cheddar cheese preheated to 180 F.

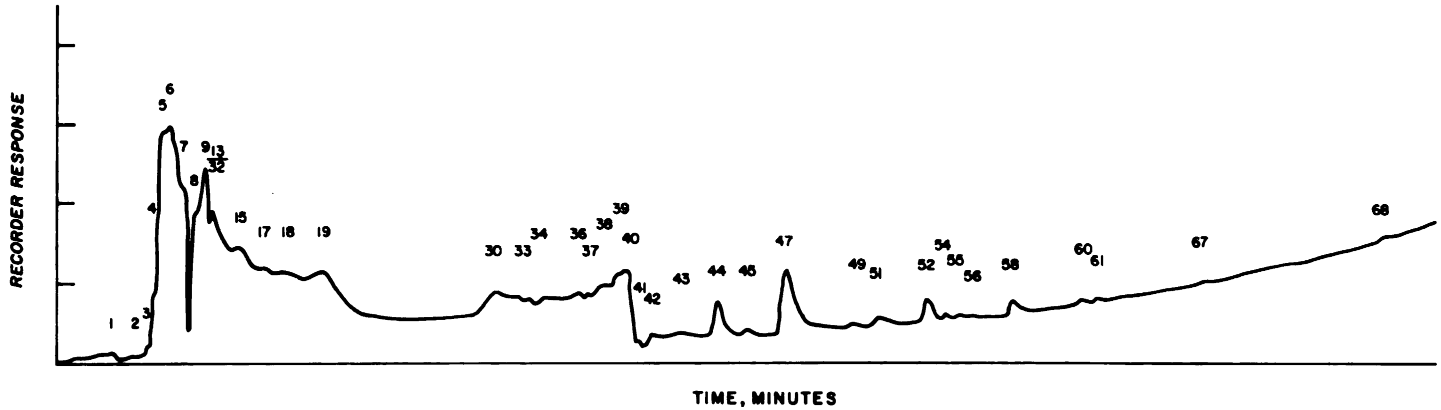

Figure 10. A gas chromatogram of the flavor from spray-dried Cheddar cheese preheated to 120 F.

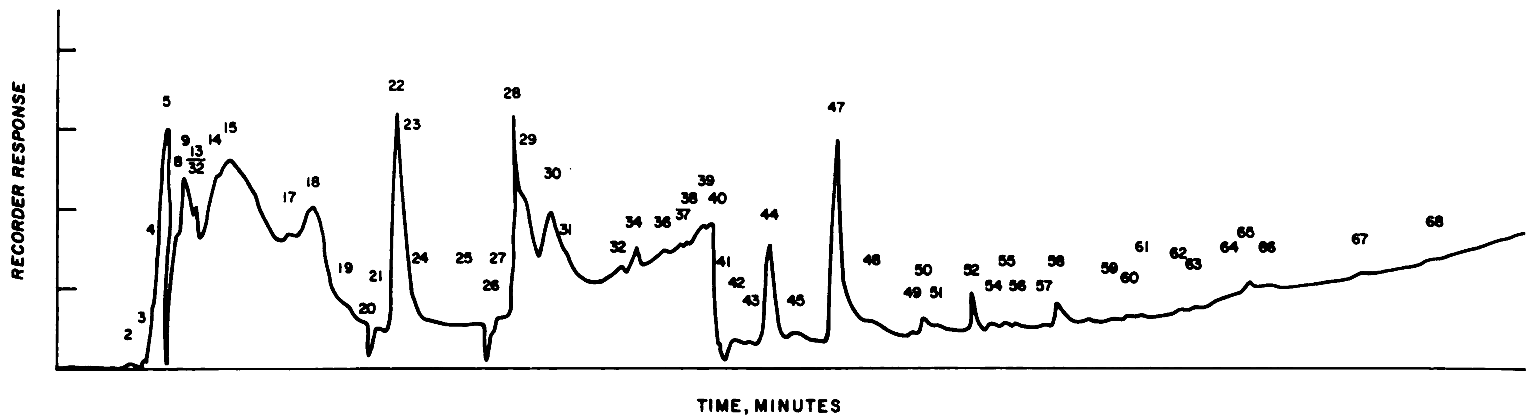

Figure 11. A gas chromatogram of the flavor from foam spray-dried Cheddar cheese preheated to 120 F.

DISCUSSION

The fact is well documented that the keeping quality of dehydrated whole milk products is enhanced by adequate preheat treatments (17, 24, 34, 51). During the trials to develop a Cheddar cheese powder that resisted oxidation and staling, several preheat treatments were evaluated (Tables 9 and 10). These results indicated that a preheat treatment of 180 F for 10 min was optimum. Manus and Ashworth (51) showed that whole milk powder prepared from milk which had been preheated to 180 F for 10 min or 170 F for 30 min possessed slightly better palatability over a 10 month storage period even in air-packed cans. Furthermore, the activation of the sulfhydryl groups during preheating served to retard the development of oxidation. Throughout the storage of cheese powders, the PV of the samples preheated to 180 F (Table 9) and 170 F (Table 10) was lower than other samples in these trials. The lower the PV, the longer the induction period before an oxidized flavor is evident. The sample of Cheddar powder that had received a heat treatment of 180 during manufacture illustrated this longer induction period.

Preheating the cheese slurries to 180 F for 10 min also appeared to retard the stale off-flavor that often overshadowed the typical cheese flavor of some cheese powders. Tarassuk and Jack (82) proved that heating whole milk to 190 to 195 F before drying results in a much superior powder than powders exposed to other preheat temperatures. Moreover, adequate preheat temperatures are essential for reducing the potential for staling. The cheese powders that received the higher preheat treatments during manufacture showed less stale flavor than the other cheese powders in these trials.

Since the flavor of Cheddar cheese depends on the presence of volatile organic materials (36), a low exit air temperature consistent with a low moisture content in the powder is mandatory to retain these volatiles. In the patent issued to Rogers (67), an exit air temperature of less than 165 F is specified. Chase and Laursen (16) claim that an exit air temperature of 110 F could be used in foam spray-drying coffee extract. Roundy and Ormond (69) stated that an exit air temperature of 138 to 155 F would result in a desirable cheese product containing 3 per cent moisture. The results in Table 8 show that the optimum exit air temperature with the operating parameters and the drier used in these trials is 160 F. A lower temperature of the exit air contributed to some adhering of particles to the walls of the drier. These adhering particles were exposed to the detrimental effects of the heated atmosphere for a prolonged period. The PV and TBA values of the powder dried at an exit air temperature of 150 are higher than the values for the powder dried at an exit air temperature of 160 F and show the effect of this excessive exposure to the drying atmosphere.

Another important factor to consider when products with highly volatile flavorings are dried is the size of the dried particle. Sivetz (75) indicated that instant coffee manufacturers spray dry to obtain large particles since these particles have the greatest flavor. The larger the particle, the greater the concentration of volatiles trapped within. This fact has been observed in the spray-drying trials involving Cheddar cheese. The flavor criticism in Tables 5 and 6 show that the powders obtained by atomizing the slurries through nozzles of large orifice diameters had more residual Cheddar flavor than the cheese powders obtained by atomizing through smaller orifice diameters. After storage for 2 months in nitrogen-packed cans, the powders with the larger particle size demonstrated greater resistance to the formation of peroxides.

Examination of the results shown in Table 7 further support the premise that a large particle retains more flavor than a small particle. The low pressure SBC nozzle is designed to limit the range of particle sizes; therefore, a comparison of two nozzles using equivalent flow rates would indicate the deleterious effect of the fine particles on the quality and stability of the resulting powders. The cheese powder obtained by spray-drying using the SBC nozzle indicated a decided flavor superiority over the cheese powders having a greater percentage of fine particles and spray-dried through the SX nozzle. However, on some occasions, the stale flavor which developed in the storage trials complicated the flavor judgments.

The apparent improvement of the Cheddar cheese powders was reconsidered during the trials involving foam spray-drying. Sivetz and Foote (76) indicated that instant coffee powders are hollow, partially from expansion of entrapped carbon dioxide and partially due to formation of vapors during spray-drying. The particles dried in this hollow form are much more flavorful than particle fragments or solid particles obtained by degassing the coffee extract. Normally, dried particles of dairy products are not hollow. Sparging of the liquid concentrate with gas is necessary to induce hollowness. Hanrahan and Webb (33) showed that high pressure gas injection permitted drying of products that were impossible to dry by other means. Several patents (16, 60, 65) have been issued to cover the use of high pressure sparging as a means to facilitate the drying of heat sensitive products. The application of foam spray-drying to the manufacture of Cheddar cheese powder has been successful because these cheese powders retained more volatile flavor than found in cheese powders dried conventionally. These powders exhibited no stale flavor. When a nozzle designed to limit the percentage of fine particles is used in conjunction with foam spray-drying a powder with good shelf-life and excellent flavor resulted (Table 12). The injected gas contributed to increased surface area with no increase in the average weight of the particles. Comparison of the flavor in the cheese powders from the two methods of spray-drying (Table 11) indicated more Cheddar flavor in the foam spray-dried cheese.

Under magnification the structure of these particles appears sponge-like and hollow (9, 32). The puffing of the cheese particles attributable to the entrapped gas increases the relative surface area. Evaporation occurs at a faster rate; therefore, the particles remain cooler during the falling rate drying period. Apparently, cheese particles that remain cooler during drying retain more flavor. This increased flavor was observed in the powders that were foam spray-dried. Similarly, cooler particles are less subject to the deteriorative effects of the heated atmosphere encountered in the drier, as shown by the PV of the foam spray-dried cheese powder which is less than that for the powder dried conventionally (Table 11).

Inert additives have been recommended and used in many dehydrated foods. The manufacturers claim that the use of these additives enhances flavors, improves flow properties and body characteristics and reduces lipid oxidation by encapsulating the particles during drying. The results (Table 1 and 2) demonstrate that these additives contributed no beneficial quality to the dried cheese products except in the trial involving corn starch and crystalline cellulose. Comparison of the PV and TBA values (Table 1) also shows that the control sample had better oxidative stability than the cheese samples containing the additives. Even under an atmosphere of nitrogen no advantage was observed from using most additives. Lactose as a dry crystalline powder or in Cheddar whey accelerated the appearance of staleness and possibly contributed to Maillard browning.

Antioxidants have been used in many food products that contain fat to improve shelf-life and stability. The use of antioxidants in most milk products is prohibited by law; however, no standards of identity have been established for dried cheese. The data in Tables 3 and 4 show that antioxidant solutions, particularly the mixtures of BHA, BHT, PG and CA; BHA, PG, and CA; and NDGA contributed the most stabilizing influence in the Cheddar cheese powders. Abbot and Waite (1) reported that PG and NDGA afforded good protection in dry whole milk, while BHA showed poor ability to prevent oxidation. Several authors (15, 79, 80) have observed that citric acid functions as a synergist; therefore, the activity of antioxidants is improved. The presence of CA in the antioxidant blends (Tables 3 and 4) increased the efficiency of these mixtures. Stull, Herreid and Tracy (79) gave no report of an off-flavor with as much as 0.1 per cent NDGA in dry whole milk, whereas, objectionable off-flavors were observed with greater than 0.04 per cent NDGA in spray-dried ice cream mix containing 12 per cent fat. El-Negoumy and Hammond (27) reported an objectionable bitter off-flavor from 0.01 per cent NDGA in butter. The results also indicated no benefit from the use of antioxidants in butter. The results from the drying trials in Tables 3 and 4 show that the antioxidant mixtures functioned to protect the powders from oxidation only after 4 and 3 months storage, respectively. In these trials where the overall quality of the cheese powder was fair, antioxidants served to extend shelf-life and retard oxidation. The samples containing 0.06 per cent NDGA possessed a noticeable off-flavor after storage in nitrogen-packed cans.

The flavor scores on these foam spray-dried cheese powders (Table 13) indicate that the antioxidants served no beneficial purpose. In this trial, preheating the Cheddar cheese powders to 180 F for 10 min and foam spray-drying contributed to the overall quality of the powder. The PV and TBA values indicate some stabilizing activity from these antioxidants. Apparently, the concentration of any stale flavor compounds in the foam spray-dried cheese powders is below the threshold value since all foam spray-dried Cheddar cheese powders exhibited no perceptible stale flavor. The evaporation of moisture from the increased surface of the puffed particles keeps these particles cooler during drying and these particles are less subject to the oxidative effects of the heated atmosphere.

The shelf-life of the powders containing high concentrations of fat can be extended by sealing the containers in an atmosphere of nitrogen (31). Coulter and Jenness (19) showed that double gassing within a 24 hour interval was necessary to remove the traces of oxygen trapped interstitially in the particles of dry whole milk. Double gassing was used on all samples of Cheddar cheese powder during preparation for storage. As anticipated, the results of the storage trials indicate that a nitrogenous atmosphere reduces the rate of oxidation. Foam spray-dried cheese powders probably require only one gassing and the resulting powders would exhibit shelf-life similar to powders that were double gassed. Since nitrogen is used as the sparging gas, the interstices of the particles are presumably filled with nitrogen. A second gassing of the container after an equilibration period would not be expected to remove significant amounts of oxygen.

The PV and TBA values shown in the data from each trial often indicate little correlation with the organoleptic evaluations on the cheese powders. The poor correlation of these analytical values with flavor has been observed by others. El-Negoumy and Hammond (27) stated that the PV and TBA tests measure the extent of oxidation occurring under certain given conditions and these values may or may not correspond closely to flavor evaluations under all conditions. Kliman, Tamsma and Pallansch (45) found no correlation between PV and flavor scores. The PV and TBA values recorded for the trials involving Cheddar cheese are useful for the specific trial in question. PV and TBA values differ between lots of Cheddar cheese as well as between wheels of cheese. These differences make correlations between the samples in different trials unreliable.

Gas chromatography has proved to be a very useful tool to establish the flavor profile of Cheddar cheese powders. GLC is particularly useful to correlate flavor spectra with organoleptic flavor evaluations. Comparison of the chromatograms (Figures 8, 9, 10 and 11) shows that the cheese powders spray-dried conventionally possessed less overall Cheddar flavor than the powders that were foam spray-dried. This evidence substantiates the results of the organoleptic judgments. The observation was made previously that increases in retention of volatiles accompanied increases in particle size. The chromatograms in Figures 9 and 11 and the data in Table 15 show a greater concentration of volatile material for these foam dried cheese samples than shown in the samples represented by Figures 8 and 10 that were spray-dried conventionally.

The heat treatments to which the slurries were subjected served to develop more flavor and to retard the onset of staling and oxidation. The liberation of additional sulfide-containing material (peaks 28, 39, 47 and 52) from those shown in the chromatogram of the natural cheese (Figure 5) may supply the resistance to oxidation and staling. According to the analyses for functional groups, these compounds are disulfides. Evidence was presented in Table 14 to indicate that mild oxidation constantly occurs in natural cheese. The rate of oxidation is accelerated by increasing the storage temperature. Some of the oxygen sensitive sulfhydryl compounds exposed during preheating are lost in this manner while the remainder may be complexed with the mercuric chloride during analysis for the functional groups.

The slurry that was heated to 180 F for 10 min lost more volatile constituents than the slurry heated to 120 F for 10 min. This loss was expected since the flavor materials would be more volatile at elevated temperatures. However, the slurry heated to 180 F was cooled at room temperature to 140 F before spray-drying. Perhaps this cooling treatment explains the increase in low boiling compounds shown in the chromatogram of the foam spray-dried cheese powder (Figure 9).

The slurries that were heated to 120 F before homogenization were spray-dried immediately. In addition to the sulfide compounds, other compounds were formed during processing (peaks 1, 3, 39, 47, 51) most of which appear in greater concentrations in the slurry heated to 120 F. The chromatograms of these two powders (Figures 10 and 11) show more flavor material in the region of peaks 22 to 40 than is shown in Figures 8 and 9. The volatile material in the powders preheated to 120 F may have been fractionated further by the higher heat treatment. The foam spray-dried powder that was preheated to 180 F possessed more highly volatile material in the region of peaks 4-19. The presence of this volatile material apparently accounts for the preference of this powder over powders resulting from other variations in manufacturing technique.

SUMMARY AND CONCLUSIONS

1. Cheddar cheese slurries prepared by diluting the comminuted cheese with water to 40 per cent total solids, adding 2.0 per cent sodium citrate and 0.5 per cent sodium chloride and heating to 180 F for 10 min contributed to good flavor quality and shelf-life in the dried cheese powder.
2. Injection of an inert gas under pressure into the cheese slurry and spray-drying this foam at a low exit air temperature consistent with proper operation of the drier and a low moisture content in the powder resulted in a cheese powder with no stale flavor and good retention of volatiles.
3. Spray-drying the cheese slurries through an orifice of large diameter in a nozzle designed to limit the percentage of fine particles maximizes the retention of flavor volatiles and minimizes the detrimental effects of the heated atmosphere in the drier on the cheese particles.
4. Rapid cooling of the powders after collection followed by packaging in containers sealed under nitrogen provides for a long shelf-life.
5. Addition of inert materials to improve the quality of the dried cheese powders appeared unnecessary. The cheese powders containing additives that were examined in this study generally were inferior to the control samples.
6. The use of maximum effective concentrations of antioxidant mixtures improved the oxidative stability of the cheese powders. However, some off-flavors were observed particularly in the samples containing NDGA. The addition of antioxidants to the cheese slurries that were foam spray-dried showed no improvement in shelf-life.
7. High vacuum distillation and gas chromatography were used to isolate the flavor volatiles in natural Cheddar cheese, in the cheese slurries sub-

jected to two different heat treatments and in the cheese powders resulting from spray-drying portions of each slurry using two different techniques. The cheese slurries lost some flavor volatiles and several sulfide compounds were developed during preheating. The foam spray-dried powders showed much higher concentrations of flavor material than the cheese powders dried conventionally. The foam spray-dried cheese powder that was preheated to 180 F during manufacture possessed more low boiling flavor constituents which apparently contributed to the preference of cheese powders prepared in this manner.

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APPENDIX

Solubility Index

A 13 g sample of cheese powder was mixed for 90 sec with 100 ml distilled water at 75 F in a Cenco Solubility Index Mixer. The mixed liquid was transferred to a beaker and the foam was spooned off after a quiescent period of standing that was no longer than 15 min. The sample was mixed for 5 sec in the beaker and 50 ml was poured into a conical centrifuge tube. The tube was centrifuged at 848 rpm for 5 min. The supernatant was siphoned off without disturbing the sediment and distilled water at 75 F was added to the 50 ml mark. The contents of the tube were mixed and the tube was recentrifuged for 5 min. The amount of sediment in the tube was measured in milliliters.

Thiobarbituric Acid Determination

Reagents:

2-thiobarbituric acid solution - 0.025 M 2-TBA in 1M H_3PO_4 prepared before use by mixing equal volumes of 0.05 M 2-TBA with 2M H_3PO_4.

Extraction mixture - 2:1 isoamyl alcohol to pyridine.

A 2 g sample of cheese powder was mixed with 10 ml distilled water in a conical 50 ml centrifuge tube. A 5 ml aliquot of 2-thiobarbituric acid solution was added and the contents of the tube mixed well. After stoppering, the tubes were placed in a boiling water bath for 10 minutes. The tubes were cooled immediately in a cold water bath. Exactly 15 ml of the extraction mixture was added and mixed thoroughly for 30 sec with the heated material. The tubes were centrifuged for 5 min at 3000 rpm. The solvent layer was removed with a pipet and the adsorbancy determined at 535 mμ on a Coleman Universal Spectrophotometer.

Peroxide Value

Reagents:

De-emulsification reagent - 50 g sodium citrate and 50 g sodium salicylate were dissolved in water, 86 ml redistilled n-butanol was added and the total volume was made up to 450 ml with water.

Benzene-Methanol solvent - 70 volumes of benzene (thiophene free) and 30 volumes of anhydrous methanol. Benzene was redistilled and the methanol was refluxed for 4 hours with magnesium ribbon (5 g/liter) before redistilling.

Ferrous chloride - 0.4 g hydrated barium chloride dissolved in 50 ml water was added slowly with constant stirring to a solution of 0.5 g hydrated ferrous sulfate in 50 ml water. Two milliliters 10N hydrochloric acid were added. The precipitate was allowed to settle; the supernatant was filtered and stored in a refrigerator until used.

Ammonium thiocyanate - 30 g ammonium thiocyanate was dissolved in water and the volume made up to 100 ml with water.

A 30 ml aliquot was taken from a mixture of 8 g cheese powder mixed with 100 ml distilled water and added to a Babcock 9 g cream bottle. A 15 ml aliquot of de-emulsification reagent was added. The bottles were placed in a Cherry-Burrell Babcock bottle shaker for 5 minutes and then in a water bath at 70 C for 10 min. The bottles were removed from the water bath and centrifuged in a Babcock centrifuge for 1 min. Hot distilled water was added to bring the fat layer into the upper portion of the neck of the bottles. The bottles were centrifuged for another minute, removed from the centrifuge and placed in a water bath at 45 C to temper the contents of the bottles. A 0.5 ml portion of the fat is removed with an Ostwald-Folin pipet and placed in a 10 ml standard taper volumetric flask. The fat in the volumetric flask was made up to 10 ml with benzene-methanol solution, stoppered and inverted to disperse the fat. One drop each of the ferrous chloride and ammonium thiocyanate was added. The flasks were shaken vigorously and placed in a water bath at 50 C for exactly 2 min to develop the color. The flasks were removed immediately and placed in an ice water bath for 2 min. The solutions in the flasks were placed in cuvettes and the transmission was read at 505 mμ. The reagent blank (with no fat added) was used as a check for deterioration of the reagents. A fat blank (with no ferrous chloride added) was also used to determine erroneous transmittancy. The sum of these blanks, calculated as μg Fe was subtracted from sample readings in determining the peroxide value. The spectrophotometer was balanced at 100% transmittancy against the benzene-methanol solvent.

Antioxidant Solutions Used

Tenox S-1	20% propyl gallate
	10% citric acid (anhydrous)
Tenox 4	20% butylated hydroxyanisole
	20% butylated hydroxytoluene
Tenox 6	10% butylated hydroxyanisole
	10% butylated hydroxytoluene
	6% propyl gallate
	6% citric acid
Tenox 7	28% butylated hydroxyanisole
	12% propyl gallate
	6% citric acid (anhydrous)
NDGA	10% NDGA

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TECHNICAL NOTES

SIMPLE DEVICE FOR OBTAINING SAMPLES OF HEADSPACE GAS DIRECTLY FROM SEALED CONTAINERS FOR ANALYSIS BY GAS CHROMATOGRAPHY [1]

The determination of oxygen in the headspace gas of sealed containers of food products has been a commonly used quality control measure for many years. The Orsat method which is often employed generally requires large volumes of gas and is time-consuming. Instruments are available for detecting a gas by measuring its paramagnetic susceptibility, but such devices cannot yield a complete analysis of mixtures of gases. Several methods (3, 4) have been described for the removal of gas mixtures from the headspace of sealed containers and procedures have been developed (1, 2) for the separation and identification of microquantities of gases by gas chromatography.

A simple, inexpensive device (Figure 1) has

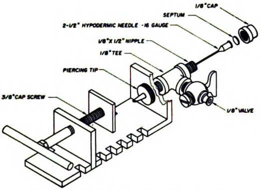

FIG. 1. Detail of gas sampling device.

been developed in this laboratory to permit the removal of headspace gas from sealed cans without contamination of the sample from an outside atmosphere. The materials used in construction of the device were:

1. Base Plate—2″ × 14″ × 3/16″ mild steel with a 90° bend 2″ from end
2. End Plate—2″ × 2″ × 3/16″ mild steel
3. Screw—3/8″ × 4″ hex head cap screw
4. Screw Plate—1″ × 1″ × 3/16″ mild steel
5. Piercing tip with rubber stopper
6. 2—1/8″ × 1/2″ pipe nipples
7. 1/8″ × 1/8″ × 1/8″ pipe tee
8. 1/8″ pipe cap with a 1/8″ drilled hole
9. 1/8″ valve
10. Hypodermic needle, 16 ga., 4″
11. Rubber injection gasket, 3/8″ dia.

Adjustments made by moving the end plate of this device adapt the base to any size container. The can is first held loosely in the apparatus and the screw is tightened until a seal is accomplished between the rubber stopper and the container. The system can then be purged by attaching a cylinder of inert gas and flushing or, alternatively, the contaminating atmospheric gases in the unit may be removed with a vacuum pump. The screw is tightened further, compressing the rubber stopper (not shown in the diagram) until the can is punctured by the piercing tip. A gas-tight syringe with a 4-inch hypodermic needle (23 to 27 ga.) is inserted through the rubber septum. An internally contained hypodermic needle (16 ga.) provides a passageway to guide the inserted needle through the can piercing tip and into the can. Without this internally contained hypodermic needle, probing is necessary to find the hole in the piercing tip. Consequently, time is wasted and the needle is dulled after only a few uses. Two rinsings of the syringe with the headspace gas are necessary to obtain reproducible results when the gas sample is injected into a gas chromatograph. The hypodermic needle on the air-tight syringe may be withdrawn and inserted many times without danger of contaminating the internally contained gases.

R. L. BRADLEY, JR.
AND
C. M. STINE
Department of Food Science
Michigan State University
East Lansing

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COMPARISON OF 305- AND 365-DAY PRODUCTION RECORDS UNDER STRATIFIED LEVELS OF HERD PRODUCTION [1, 2]

The relative merit of lactation records of varying duration has been studied by numerous research workers. Thoele (5), in a review of the literature dealing with 305- and 365-day records, generally concluded that for purposes of selection the 305-day record is at least as useful as the 365-day record. The increased influence of calving interval, age, and season of calving on records longer than ten months is apparent (2-5). This, plus the knowledge that variation in feeding and management have greater influence on later than on early stage of lactation, has stimulated recent research dealing with the usefulness of records of shorter duration than ten months.

The issue of 305- vs. 365-day records has been renewed, by dairymen, as a result of the rapid growth of Dairy Herd Improvement Registry (DHIR) through which 305-day and completed lactation records but not 365-day records are provided as a result of routine Dairy Herd Improvement Association (DHIA) procedures. The question raised by dairymen is whether 365-day records are more useful for selection purposes in high-producing herds under Advanced Registry (AR), Herd Improvement Registry (HIR), and DHIR conditions. This study was made in an attempt to appraise the relative merits of 305- and 365-day records for selection purposes in stratified levels of AR and HIR herd production.

[1] Supported in part by Wisconsin's AI Cooperatives.

[2] Approved for publication by the Director of the Experiment Station, University of Wisconsin.

Production data were provided by the Holstein-Friesian Association of America and consisted of 81,226 records made by 15,892 cows in 76 AR and HIR herds. The herds used in this study were selected on the basis of having been on test for 15 or more consecutive years and for variation in level of herd fat production (low = 400-449 lb; medium = 450-499 lb; high = 500-550 lb) The data included actual lactations of 306 to 365 days and both M.E. and actual 305-day or less records completed through 1959. A total of 15,430 records represented 3× milkings in 17 herds.

The general description of the data with respect to five length-of-lactation groups is as follows:

Lactation days	Herd groups			
	Low 2× (%)	Medium 2× (%)	High 2× (%)	3× (%)
<305	21.9	23.5	17.8	19.1
305	39.7	38.7	41.5	41.0
306–335	13.4	14.8	14.3	11.4
336–364	9.3	8.5	9.8	8.4
365	15.8	14.5	16.7	20.1

There appeared to be no important trends in the distribution of length of lactation groups over the three levels of 2× herds. However, in 3× herds a somewhat greater percentage of 365-day records was noted. The average age at calving, length of lactation, and number of records per cow were very similar and in most cases identical over the four herd groups. The

TABLE 1

Repeatability estimates of first and second available 305- and 365-day actual and M.E. production

Herd group	No. herds	No. cows	1st 305-day fat		Repeatability estimates					
					1st vs. 2nd 305			1st vs. 2nd 365		
			$\bar{x}$	s	Milk	Fat	% Fat	Milk	Fat	% Fat
2×, M.E.										
Low	24	767	471	84	.421	.375	.658	.417	.376	.690
Medium	22	602	516	79	.417	.348	.732	.437	.390	.763
High	27	1,050	582	89	.496	.494	.748	.496	.501	.770
Over-all	73	2,419	530	85	.453	.429	.719	.458	.442	.746
2×, Actual										
Low	24	767	402	80	.420	.383		.428	.390	
Medium	22	602	440	80	.408	.363		.427	.381	
High	27	1,050	501	94	.456	.474		.476	.501	
Over-all	73	2,419	454	87	.443	.424		.451	.444	
3× on M.E., 2× basis										
	17	426	502	70	.514	.535	.750	.506	.530	.781
3× actual										
	17	426	507	81	.504	.510		.503	.512	

SUMMARY

A study has been made of conditions suitable for spray-drying natural cheese. Blending comminuted cheese with water, salt and sodium citrate and heating results in a smooth dispersion which can be homogenized and spray-dried. Manipulation of the drying procedure to minimize heat damage in the drier and to obtain a large particle size enables the production of a cheese powder of good flavor and reconstitutability.

SPRAY - DRYING OF NATURAL CHEESE[1,2]

By

R. L. Bradley, jr.,[3] and C. M. Stine

Department of Food Science

Michigan State University, East Lansing, Michigan

Dehydrated cheese has been merchandised for many years. This product is well suited to foods that require no refrigeration and is widely used in chip dip bases, macaroni and cheese dinners, special bakery products and directly as a free-flowing powder on prepared foods.

Most of the recent research reported on dried cheese has been concerned with dehydration of shredded or grated flakes in an air stream ranging in temperatures from 72 degrees F. to 290 degrees F. Sanders[15, 16] showed that shredded cheese, case-hardened by drying at room temperature, could then be dried further at elevated temperatures without undesirable exudation of fat. Czulak et al.[4] elaborated on this technique and claimed that addition of roller or spray-dried skimmilk or whey to the cheese during and following grating resulted in a dried cheese-type product that exhibited no visible free fat or particle clumping. Bohac[3] roller and spray-dried various cheese mixtures and reported actual flavor improvement by such processing treatments. In one trial, Bohac dried a mixture of cheddar, "Dutch-type" cheese, low fat "loaf," cottage cheese, water "emulsifying salt G," alkaline polyphosphate and potassium polyphosphate. In another experiment, Roquefort cheese, Moravian cheese loaf, water, "emulsifying salts G and B," "polymeric phosphate" and ammonium polyphosphate were blended and spray-dried. The observation that the drier was heavily contaminated with dried skimmilk limited an interpretation of the data from these roller-drying experiments.

The hard, grating type cheeses such as Romano and Parmesan have customarily been air-dried before grating. Research comparing the relative effectiveness of bright and dark infra-red lamps and warm air currents[11] indicated that the use of dark infra-red lamps with filters to exclude visible light yielded the best quality dried cheese.

Various U. S. patents have been granted on the drying of cheese. An early patent[8] covers vacuum roller-drying of natural cheese and preparation of cheese emulsions for spraying on foods, such as, crackers or popcorn. Patents issued to Roundy[13, 14] pertain to prepa-

[1]Supported by a grant from the American Dairy Association.

[2]Published with the approval of the Director of the Michigan Agricultural Experiment Station as Journal Article No. 3074.

[3]This article is part of a dissertation to be presented to the Faculty of the Graduate School of Michigan State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

TABLE 1. The effect of exit air temperature on the quality of spray-dried Cheddar cheese.

Analyses	Exit air temperature, F.			
	160	170	180	190
Moisture, %	2.5	2.4	2.3	2.3
TBA[a]	0.026	0.032	0.034	0.031
PV[b]	0.241	0.572	0.691	0.618
Flavor	fair	v. sl. stale	v. sl. stale	stale
Drying conditions				
Nozzle	SX	SX	SX	SX
Orifice diameter, in	0.038	0.038	0.038	0.038
Pump pressure, psig	2500	2500	2500	2500

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

TABLE 2. The effect of orifice diameter on the quality of spray-dried Cheddar cheese.

Analyses	Orifice diameter, in			
	0.038	0.040	0.052	0.055
In-flight				
Moisture, %	2.6	2.5	3.0	2.8
TBA[a]	0.043	0.044	0.040	0.051
PV[b]	0.280	0.478	0.301	0.648
Flavor	sl. chalky	sl. chalky	fair	fair
Swept				
Moisture, %	2.5	2.7	2.5	2.8
TBA[a]	0.050	0.044	0.042	0.051
PV[b]	0.952	1.042	0.858	1.221
Flavor	poor, stale	stale	sl. stale	sl. stale
Drying conditions				
Nozzle	SX	SX	SX	SX
Core	20	17	17	17
Pump pressure, psig	2500	1500	1000	1000
Exit air, F.	160	160	160	160

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

TABLE 3. The effect of a reduction in the percentage of fine particles on the quality of spray-dried Cheddar cheese.

Analyses	Type of nozzle used	
	SX	SBC
In-flight		
Moisture, %	2.4	3.2
Solubility index, ml	4.0	4.0
TBA[a]	0.176	0.159
PV[b]	0.164	0.124
Flavor	sl. stale	v. sl. stale
Swept		
Moisture, %	2.2	2.8
Solubility index, ml	4.0	4.1
TBA[a]	0.079	0.088
PV[b]	0.282	0.207
Flavor	sl. stale	v. sl. stale
Drying conditions		
Nozzle	SX	SBC-6
Orifice diameter, in	0.040	0.040
Core	17	standard
Exit air, F.	160	160
Pump pressure, psig	1500	800

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

TABLE 4. The effect of a reduction in the percentage of fine particles on the quality of spray-dried Cheddar cheese after storage for 2 months.

Analyses	Type of nozzle used	
	SX	SBC
Nitrogen-packed		
Moisture, %	2.8	3.2
Solubility index, ml	4.6	4.1
TBA[a]	0.121	0.138
PV[b]	1.512	1.075
Flavor	sl. stale, fruity	sl. stale
Air-packed		
Moisture, %	2.5	2.9
Solubility index, ml	4.5	4.1
TBA[a]	0.214	0.284
PV[b]	1.214	3.376
Flavor	sl. stale, oxidized	sl. stale, astringent

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

ration and spray-drying of Baker's and Neufchatel type products containing natural or synthetic stabilizers. The manufacture of spray-dried, cheese-flavored products from blends of skimmilk, nonfat dry milk, vegetable oil, emulsifier and as little as 1.2-2.1% natural cheese is described in a patent by Bauman.[3] In some examples, the blends were also treated with enzymes or subjected to bacterial fermentation prior to drying. In a brief report with no data recorded, Makar'in[8] mentioned the techniques of spray-drying cheese emulsions, packaging the resulting cheese powders and their possible use in the manufacture of processed cheese.

Freeze-drying or lyophilization of cheese has also been investigated. Evstrat'eva et al.[6] suggested that Russian seasonal surpluses of lactic coagulated cheese might be preserved by freeze-drying. Their pilot plant had a daily output of 163 lb. of dry curd containing 2.9-4.0% moisture. The highly porous curd was claimed to reconstitute readily with warm water. Natural cheeses such as cheddar, brick, Muenster, blue, cream and cottage have been freeze-dehydrated by Meyer and Jokay.[7,10] These dried cheeses could be slowly rehydrated to a soft but natural consistency and with a mild flavor.

The work reported herein was undertaken to determine which processing procedures would yield a dried cheese powder with flavor comparable to the original cheese and to obtain a powder that would retain desirable properties during a reasonably long storage period.

MATERIALS AND METHODS

Selection of cheese and sample preparation. Cheddar cheese ripened to various ages was selected to determine the optimum ages that would readily disperse with water. Eight pound samples of cheese were comminuted, slurried with tap water to 40% total solids and heated to 120 degrees F. Cheddar cheese less than 3 months old resisted slurring and subsequently caused the homogenizer valves to stick. Whereas, cheeses older than approximately 3 months dispersed readily in water and caused no valve sticking. Furthermore, cheeses that possessed a distinctly bitter flavor or were criticized as being "acid" or "bitter" would produce cheese powders of similar flavor quality.

Commercial spray-drying operations require a total solids concentration of at least 36 to 42% for good operating efficiency of a drier. Consequently, a concentration of 40% total solids was arbitrarily selected as an appropriate dilution for economical drying. When cheddar cheese was prepared to contain 40% total cheese solids, heated and homogenized in a Manton-Gaulin homogenizer at 2000 psig single-stage, a smooth liquid suitable for pumping to the drier resulted.

Spray-drying. The slurries, prepared from 8 lb. lots

TABLE 5. The effect of heating Cheddar cheese slurries to various temperatures on the quality of spray-dried Cheddar cheese.

Analyses	Heat treatment, F.			
	120	170	180	190
In-flight				
Moisture, %	2.4	2.5	2.7	2.8
Solubility index, ml	4.25	5.0	6.0	7.75
TBA[a]	0.103	0.127	0.140	0.143
PV[b]	0.500	0.503	0.425	0.507
Flavor	fair, sl. stale	fair	fair, more Cheddar flavor	fair, sl. grainy
Swept				
Moisture, %	1.9	2.1	2.4	2.6
Solubility index, ml	4.0	5.0	5.75	7.25
TBA[a]	0.089	0.080	0.121	0.133
PV[b]	0.998	0.848	0.752	0.863
Flavor	sl. stale	v. sl. stale	fair, v. sl. stale	fair, sl. grainy
Drying conditions				
Nozzle	SBC-6	SBC-6	SBC-6	SBC-6
Orifice diameter, in	0.040	0.040	0.040	0.040
Exit air, F.	160	160	160	160

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

TABLE 6. The effect of heating Cheddar cheese slurries to various temperatures on the quality of spray-dried Cheddar cheese after storage for 5 months.

Analyses	Heat treatment, F.			
	120	170	180	190
Nitrogen-packed				
Moisture, %	3.0	3.2	3.3	3.2
Solubility index, ml	4.0	4.75	6.0	7.0
TBA[a]	0.090	0.097	0.115	0.059
PV[b]	0.533	0.450	0.388	0.511
Flavor	fair, v. sl. stale	fair, v. sl. stale	good	fair, v. sl. stale
Air-packed				
Moisture, %	3.0	3.1	3.2	3.2
Solubility index, ml	4.25	4.5	6.5	7.5
TBA[a]	0.110	0.132	0.100	0.100
PV[b]	1.010	0.786	0.665	0.809
Flavor	fair	fair	fair	fair

[a]expressed as absorbancy
[b]expressed as meq O_2/kg fat

of 93 score cheddar cheese with or without added emulsifying salts and sodium chloride, were conditioned to 120 degrees F. before feeding to the Manton-Gaulin high-pressure pump. These slurries were atomized through Spraying Systems nozzles into a Rogers co-current, horizontal, inverted tear-drop drier. A commercial operation was simulated by "in-flight" collection of the powders in wooden boxes 6 x 12 x 24" lined with Kraft paper and placed on the cat walk in the drier. The dried particles of cheese dropped into these boxes and were protected from the detrimental effects of the heated atmosphere encountered by the particles adhering to the walls of the drier. The latter powder was collected by sweeping at the conclusion of each trial. Powders obtained by these two methods were compared for flavor quality and extent of heat damage occurring during drying.

Chemical Analyses. The following analyses were performed to determine the quality of the samples of spray-dried cheddar cheese powder initially and after a storage period.

a. Moisture — Infra-Red Moisture Balance (Cenco).
b. Solubility Index — The method for dry whole milk.[1]
c. Thiobarbituric Acid Determination (TBA) — The method described by Dunkley and Jennings.[5]

d. Peroxide Value (PV) — The procedure described by Pont.[13]

Flavor evaluation. The flavor quality of each sample of spray-dried cheddar cheese powder was evaluated concomitant with the chemical analyses. The powders were reconstituted with tap water to 40% total solids, allowed to stand at room temperature for at least 30 minutes and then sampled for organoleptic judgment by qualified individuals.

Preparation for storage. No. 211 x 400 cans were packed with cheese powder and sealed on an Automatic Can-Sealer. These cans were dipped in a Wood's metal and lactic acid bath to protect the contents from possible gas leakage through the rolled seams. Some cans were air-packed while others were punctured, exhausted under 200μ pressure for 30 minutes, flooded with water-pumped nitrogen and resealed with solder. After 48 hours, this evacuation process was repeated to allow removal of any interstitially trapped oxygen that had equilibrated with the essentially pure nitrogen atmosphere. All samples were stored at room temperature.

RESULTS

Many drying trials have been completed in duplicate to obtain a series of variables that when combined would yield a cheddar cheese powder of acceptable flavor quality and shelf-life. Typical trials have been selected and reported herein.

Slurries of cheddar cheese were prepared, heated to 120 degrees F., homogenized and spray-dried at exit air temperatures of 160, 170, 180 and 190 degrees F. to determine the effect of different drying temperatures on the cheese powders. The results of a typical trial (Table 1) show that the degree of heat damage increased with exit air temperature. The flavor of these samples varied from "fair" in the samples dried at an exit air temperature to 160 degrees F. to "stale" in the samples dried at an exit air temperature of 190 degrees F. Often, the flavor of the powders can be correlated with the PV and TBA values which in these trials increased with the exit air temperature.

Data from the analysis of powders obtained by atomizing cheese slurries through the same type of nozzle with various orifice diameters are shown in Table 2. Cheddar cheese was comminuted, heated to 120 degrees F., homogenized and spray-dried at an exit air temperature of 160 degrees F. The powders spray-dried with orifice diameters suitable to obtain larger particle sizes had more volatile retention, more cheddar flavor and slightly higher moisture content than the cheese powder resulting from spraying through nozzles with smaller orifice diameters. In each trial, the pump pressure was altered to give equivalent flow rates in gallons per hour.

Two types of nozzles with similar orifice diameters were used to correlate particle size in the dried powders with flavor and retention of volatiles. With each nozzle, the speed of the high pressure pump was altered to give equivalent flow rates in gallons per hour. Table 3 shows the results of a typical trial in which cheddar cheese slurries were spray-dried at an exit air temperature of 160 degrees F. The powder obtained by atomizing cheese slurries with an SBC type nozzle showed an improvement in flavor quality over that observed in the powder obtained from using the SX type nozzle. The decrease in relative surface area obtained by using the SBC nozzle apparently reduced the amount of lipid material directly exposed to the heated atmosphere. This assumption is also reflected in the lower PV and TBA values. However, the higher moisture content in the powder spray-dried through the SBC type nozzle complicates this observation since a powder of higher moisture content will often have received less heat damage in the drier.

The powders collected by sweeping were stored for two months in air and nitrogen-packed cans. The results of a typical storage trial are shown in Table 4. The quality of these samples indicated that the sample of cheese powder obtained from spray-drying with a SBC nozzle had more desirable flavor after this storage period than the other powders in this trial. The samples stored under an atmosphere of nitrogen showed lower PV and TBA values than the air-packed samples. Moreover, low correlation existed between the flavor of the powders and the values obtained to indicate oxidative deterioration. Poor correlation is often observed when attempting to compare these tests to the flavor of gas-packed powders. Generally, fat containing foods with a TBA or PV as high as shown with these samples would be perceptibly oxidized.

Other cheese slurries containing 40% total cheese solids, 0.5% sodium chloride and 2.0% sodium citrate were heated to temperatures of 120, 150, 170, 180 and 190 degrees F. to ascertain an optimum preheat temperature. The slurries were held at these temperatures for 10 min., homogenized and spray-dried at an exit air temperature of 160 degrees F. through a nozzle designed to produce a low percentage of fine particles. Typical results (Table 5) indicate that the sample heated at 180 degrees F. for 10 min. showed a somewhat lower PV, possessed more apparent cheddar flavor and less perceptible staleness than the other samples in this trial. This preheat treatment appears optimum for slurries prior to spray-drying because of the contribution to the overall flavor in the dried cheddar powder.

The cheese powders collected by sweeping and reported in Table 5 were sealed in air and nitrogen-packed cans and analyzed after a storage period of five months at room temperature. These results (Table 6) show that only slight oxidative deterioration occurred in the powders packed under nitrogen from that observed in the powders immediately after manufacture. The PV and TBA values indicate that more lipid oxidation occurred in the cheese powder stored in air than the nitrogen-packed samples.

DISCUSSION

Heating cheese slurries is also a critical step in the preparation of slurries for drying. Temperatures in excess of 125 degrees F. induce the curd to form a rubbery clot that subsequently settles to the bottom and resists redispersion. Sodium citrate, disodium phosphate and sodium potassium tartrate were used in this study to evaluate their emulsifying properties and to permit the use of higher preheat temperatures without encountering clotting of the cheese curd. Preliminary work indicated that disodium phosphate often contributed an unwanted increase in viscosity and a slight curdy appearance in the slurry. The addition of tartrate salts showed no improvement over the less viscous slurries containing sodium citrate. Therefore, cheddar cheese slurries were prepared with 2% sodium citrate and 0.5% sodium chloride added.

The powders collected in-flight were superior to the cheese powders collected by sweeping. The data (Tables 1, 2, 3 and 5) show a marked difference between these two methods of collection. Apparently, those particles that adhered to the walls of the drier were subjected to more of the deteriorative effectives of the hot air than the particles collected in-flight. This observation can be substantiated by comparison of the PV and flavor scores for the individual samples collected by these two methods.

Cheddar cheese powders containing large particle sizes showed a definite flavor superiority over powders having small particle sizes (Table 3). Some further improvement is realized when the atomizing nozzle is designed to limit the range of particle sizes. At 800 psig pump pressure, this SBC-nozzle with a 0.040" orifice produced 43% of the particles 297μ in diameter. However, after a two month storage period, the condition of these powders (Table 4) was generally poor. Subsequent trials, evaluating the effect of heat treatments during preparation of slurries (Table 5), showed that slurrying cheddar cheese with water to 40% total solids and heating to 180 degrees F. for 10 minutes in the presence of 0.5% sodium chloride and 2.0% sodium citrate contributed to the final flavor quality of the spray-dried powder. Moreover, the results of analyses performed on the stored samples (Table 6) indicated that pre-heating to 180 degrees F. with a 10 min. hold, induced more desirable effects in the dried cheese than other heat treatments used.

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Foam Spray Drying of Natural Cheese [1, 2, 3]

by
R. L. Bradley, jr.[4] and C. M. Stine
Department of Food Science
Michigan State University
East Lansing, Mich.

SUMMARY

Cheddar cheese powders that were foam spray-dried possessed improved flavor quality, larger particle diameter and somewhat greater shelf-life than cheese powders obtained by conventional spray-drying. Shelf-life was shown to be enhanced by nitrogen-packing.

LITERATURE REVIEW

Natural cheese has been dehydrated in the past by such methods as lyophilization, tray-drying and spray-drying. However, the lack of typical cheese flavor in the powders produced by these methods is due to some loss of volatile flavor constituents during dehydration. Foam spray-drying recently reported by the group at the USDA laboratories is a relatively new method of dehydration that has been successfully applied to products which are difficult to dry by conventional means. Hanrahan and Webb[6] showed that cottage cheese whey could be dried to a free-flowing, highly porous powder by this technique. Also, whole milk concentrates have been foam spray-dried[5]. The data show that free-fat and particle size increase directly with the volume of nitrogen injected into the concentrate.

The factors affecting the flavor quality and storage stability of cheese spray-dried conventionally were discussed in a previous paper.[4] The experiments reported herein describe the application of foam spray-drying to natural cheese.

1. Supported by a grant from the American Dairy Association.

2. Journal Article No. 3288 from the Michigan Agricultural Experiment Station.

3. This article is part of a dissertation to be presented to the Faculty of the Graduate School of Michigan State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

TABLE 1. The effect of foam spray-drying on the quality of dried cheddar cheese[a]

Analyses	Type of spray-drying: Foam	Conventional
Moisture, %	3.1	3.4
Solubility index, ml	4.5	4.3
TBA[b]	0.126	0.112
PV[c]	0.532	0.628
Flavor	good, more cheddar flavor	good
Drying conditions		
Nozzle type	SBC-6	SBC-6
Orifice diameter, in	0.040	0.040
Exit air, F	160	160

a. initial analyses
b. expressed as absorbancy
c. expressed as meq O_2/kg fat

TABLE 2. The effect of orifice diameter on the quality of foam spray-dried cheddar cheese[a]

Analyses	Orifice diameter, in: 0.040	0.047
In-flight		
Moisture, %	3.5	3.7
Solubility index, ml	4.8	4.9
TBA[b]	0.147	0.139
PV[c]	0.526	0.794
Flavor	good	good, sl. fruity
Swept		
Moisture, %	3.1	3.4
Solubility index, ml	4.5	4.5
TBA[b]	0.126	0.143
PV[c]	0.532	0.703
Flavor	good	good
Drying conditions		
Nozzle type	SBC-6	SBC-8
Exit air, F	160	160
Inlet air, F	210	210

a. initial analyses
b. expressed as absorbancy
c. expressed as meq O_2/kg fat

MATERIALS AND METHODS

Preparation of samples. Eight pound lots of medium aged cheddar cheese selected for flavor quality, were comminuted and slurried with tap water to 40% total solids and 0.5% sodium chloride and 2.0% sodium citrate were added. The slurries were heated to 180° F., held for 10 min. and homogenized at 2000 psig. two stage with a Manton-Gaulin homogenizer. These cheese slurries were delivered to the high pressure pump at 140° F. Water-pump nitrogen was injected into the slurries at a rate of 1 ft.^{3}/min. in a mixing cylinder located between the high pressure pump and the atomizing nozzle. This foamed mixture was spray-dried at an exit air temperature of 160° F. through a Spraying Systems type SBC nozzle. At the conclusion of each drying trial, the drier was cooled and the powder sample collected by sweeping. Each sample was placed in a polyethylene bag and stored immediately in a refrigerator until analyzed. These drying trials were repeated and typical results are reported.

Several changes were necessary to permit foam spray-drying with the equipment existing in this laboratory. A Cherry-Burrell Super-Homogenizer used as a high pressure pump, was equipped with a variable drive motor to control the flow rate of the product. A cylinder to facilitate product-gas mixing was designed 1½" i.d.x12" with ¼" wall thickness — type 304 stainless steel tubing, threaded and fitted with 1½" female caps, Teflon gaskets and 1½" nickle alloy nuts. These caps were drilled and tapped on center ¼" NPT. The gas inlet, drilled and tapped ¼" NPT, is located 2" from the base of the tube. This inlet is fitted with a ¼"x3" stainless steel pipe nipple and a Matheson ¼" NPT Check Valve.

A Victor high pressure gas-dome regulator controls the nitrogen discharge and the rate of nitrogen flow is determined by a Brooks high pressure rotameter. The check valve located between the mixing cylinder and the rotameter prevents back flow of the product into the gas regulating equipment.

Headspace analysis. Samples of headspace gas were drawn directly from the cans of cheese powder using a Hamilton air-tight syringe and the sampling device designed by Bradley and Stine[2]. Two rinsings of the syringe with the headspace gases were necessary to give reproducible results. The oxygen and nitrogen content of each sample of gas was determined on a Perkin-Elmer model 154-B Vapor Fractometer and a ¼" x2m. copper column packed with 30-60 mesh Linde type 13A Molecular Sieve.

Chemical Analyses. The following analyses were performed to determine the quality of the samples of spray-dried cheddar cheese powder initially and after a storage period.

a. Moisture — Infra-Red Moisture Balance (Cenco).

b. Solubility Index — The method for dry whole milk.[1]

c. Thiobarbituric Acid Determination (TBA) — the method described by Dunkley and Jennings.[4]

d. Peroxide Value (PV) — The procedure described by Pont.[7]

Flavor evaluation. The flavor quality of each sample of spray-dried cheddar cheese powder was evaluated concomitant with the chemical analyses. The powders were reconstituted with tap water to 40% total solids, allowed to stand at room temperature for at least 30 minutes and then sampled for organoleptic judgment by qualified individuals.

Preparation for storage. No. 211x 400 cans were packed with cheese powder and sealed on an Automatic Can-Sealer. These cans were dipped in a Wood's metal and lactic acid bath to protect the contents from possible gas leakage through the rolled seams. Some cans were air-packed while others were punctured, exhausted under 200μ pressure for 30 minutes, flooded with water-pumped nitrogen and resealed with solder. After 48 hours, this evacuation process was repeated to allow removal of any interstitially trapped oxygen that had equilibrated with the essentially pure nitrogen atmosphere. All samples were stored at room temperature.

TABLE 3. The stability of foam spray-dried cheddar cheese after storage for 9 months.

Analyses	Samples 1	Samples 2
3 months, air-packed		
Moisture, %	2.8	3.0
Solubility index, ml	5.0	5.0
TBA[a]	0.129	0.117
PV[b]	0.478	0.463
Flavor	good	good
3 months, nitrogen-packed		
Moisture, %	3.2	3.4
Solubility index, ml	5.0	5.0
TBA[a]	0.111	0.096
PV[b]	0.267	0.308
Flavor	good	good
6 months, nitrogen-packed		
Moisture, %	3.3	3.4
Solubility index, ml	5.0	5.0
TBA[a]	0.099	0.091
PV[b]	0.221	0.259
Headspace, % O_2	2.1	2.1
Flavor	good	good
9 months, nitrogen-packed		
Moisture, %	3.3	3.4
Solubility index, ml	4.5	5.0
TBA[a]	0.082	0.078
PV[b]	0.183	0.171
Headspace, % O_2	2.1	2.1
Flavor	good	good
Drying conditions		
Inlet air, F	210	210
Exit air, F	160	160
Nozzle type	SBC	SBC
Orifice diameter, in	0.040	0.040

a. expressed as absorbancy
b. expressed as meq O_2/kg fat

TABLE 4. The effect of antioxidants on the oxidative stability of foam spray-dried cheddar cheese.[a]

Analyses	Antioxidants added: Control	PG and CA BHA, BHT,	NDGA
Moisture, %	3.3	3.4	3.4
Solubility index, ml	5.0	5.5	5.5
TBA[b]	0.145	0.133	0.142
PV[c]	0.565	0.810	0.556
Flavor	good	good	good, sl. sharpness
Drying conditions			
Nozzle type	SBC-6	SBC-6	SBC-6
Orifice diameter, in	0.040	0.040	0.040
Exit air, F	160	160	160
Inlet air, F	210	210	210

a. initial analyses
b. expressed as absorbancy
c. expressed as meq O_2/kg fat

RESULTS

Representative results of a comparison of cheddar cheese powders obtained from 10 trials using the two methods of spray-drying are shown in Table 1. The flavor observations and peroxide values (PV) indicate that the foam spray-dried cheddar cheese possessed more flavor volatiles and less material that contributed to the PV than the cheese powder obtained from conventional spray-drying.

Cheddar cheese slurries were prepared and atomized through an SBC type nozzle using orifice inserts of two different diameters to determine if cheese powders with larger particle sizes would have better flavor and quality. These powders were collected in-flight and by sweeping the walls of the drier at the conclusion of each drying trial. Table 2 shows the results of a typical analysis. The PV of the samples atomized with the SBC nozzle and the 0.040" orifice was lower than the values ob-

TABLE 5. The effect of antioxidants on the quality of foam spray-dried cheddar cheese after 6 months of storage.

Analyses	Control	PG and CA BHA, BHT,	NDGA
	Antioxidants used		
3 months, air-packed			
Moisture, %	3.3	3.2	3.3
Solubility index, ml	4.5	5.5	4.75
TBA[a]	0.134	0.126	0.129
PV[b]	0.471	0.466	0.351
Flavor	good	good	good, sl. sharpness
3 months, nitrogen-packed			
Moisture, %	3.4	3.3	3.4
Solubility index, ml	4.5	5.25	5.0
TBA[a]	0.127	0.118	0.121
PV[b]	0.424	0.358	0.313
Headspace, % O_2	2.1	2.1	2.1
Flavor	good	good	good, sl. foreign
6 months, nitrogen-packed			
Moisture, %	3.4	3.4	3.3
Solubility index, ml	4.0	4.5	4.8
TBA[a]	0.149	0.140	0.126
PV[b]	0.329	0.264	0.232
Headspace, % O_2	2.1	2.2	2.1
Flavor	good	flat	good, sl. foreign
Drying conditions			
Inlet air, F	210	210	210
Exit air, F	160	160	160
Nozzle type	SBC	SBC	SBC
Orifice diameter, in	0.040	0.040	0.040

a. expressed as absorbancy
b. expressed as meq O_2/kg fat

tained from the samples atomized through the SBC nozzle with a 0.047" orifice.

These powders were stored at room temperature and analyzed after periods of 3, 6 and 9 months. These results are shown in Table 3. After 3 months the powders exhibited excellent flavor quality and a panel of judges could not distinguish between the cheese powders that were air and nitrogen-packed. The apparent decrease in the concentration of peroxides from the initial values (Table 2) was greater in the gas-packed samples than in the air-packed samples, as might be expected. Typical results representative of several trials are shown in Table 3. During storage for 6 and 9 months, these samples of cheddar cheese powder showed good flavor stability and reconstituted with ease. Organoleptic evaluation indicated no perceptible stale flavor even in the cheese powder stored for 9 months. Moreover, the PV of the cheese powder showed an apparent decrease from that observed in the control after 3 months storage in gas-packed cans. This decrease continued during 6 and 9 months storage.

Slurries of cheddar cheese were prepared and either of two types of antioxidants was added prior to homogenization to give 0.06% primary antioxidant in the fat to ascertain if this maximal effective concentration would contribute to improved shelf-life. These antioxidants were commercial blends containing 10% butylated hydroxytoluene (BHT), 10% butylated hydroxyanisole (BHA), 6% propyl gallate (PG) and 6% citric acid (CA) in a mixed lipid carrier (Tenox 6), and a solution of 10% nordihydroguaiaretic acid (NDGA) dissolved in propylene glycol. These slurries were foam spray-dried and collected by sweeping. The data from the analysis of the powders containing added antioxidants (Table 4) indicate no initial improvement over the control powders. In fact, the powders containing BHA, BHT, PG and CA showed a greater initial PV than the other samples in this trial. These powders were analyzed again after storage for 3 and 6 months at room temperature in nitrogen and air-packed cans. The PV and thiobarbituric acid (TBA) values (Table 5) indicate an apparent improvement in the oxidative stability of the samples containing the added antioxidants compared with the stability observed in the controls. The most evident stabilizing effect was contributed by NDGA. However, the flavor of this sample was noticeably "foreign" after 3 and 6 months storage in nitrogen-packed cans. The chemical tests for stability indicate that the powder containing BHA, BHT, PG and CA was effective in retarding oxidation as shown by lower PV and TBA values than recorded for the controls. The flavor of these samples containing BHA, BHT, PG and CA deteriorated during 6 months storage and were judged "flat." The control samples showed slightly higher PV and TBA values at each sampling interval; however, the flavor of these powders was more typical of cheddar cheese.

DISCUSSION

The data reported illustrate some of the controllable factors that influence the quality of foam spray-dried cheddar cheese.

An organoleptic comparison of many powders obtained by foam spray-drying showed that these powders were perceptibly free of the stale flavor typically present in cheese powders obtained by conventional spray-drying. Foam spray-drying produces a powder that has a relatively larger surface area exposed to the heated atmosphere of the drier. The presence of gases in the interstitial areas of the particle could serve to reduce the amount of volatilization of cheese flavors during spray-drying and thereby contribute to the overall improvement in the flavor retained in the dried cheese powder. Hanrahan et al.[5] noted that the individual particles of foam spray-dried whole milk possessed both small and large gas bubbles distributed throughout. These bubbles were the result of the expansion of dissolved and dispersed gasses during atomization. This gas contributed to the general increase in particle size and the relative surface area of the whole milk powder.

Foam spray-drying used in conjunction with a nozzle designed to limit the range of particle size was used in these trials. Results indicate that this combination produces a particle of large surface area and better flavor than a powder obtained by conventional spray-drying using the same SBC type nozzle. Apparently, the increase in relative surface area permits a more rapid rate of water evaporation from the particle surface during dehydration. This increased rate of evaporation results in a more pronounced cooling effect on the particles; therefore, the particles are less subject to the deteriorative effects of the heated atmosphere encountered in the drier. These observations are suggested by the following: a) foam spray-drying results in greater drier efficiency[5], b) foam spray-dried powders have lower moisture contents than conventional powders dried under similar conditions (Table 1)[6], and c) staleness present in cheddar cheese powders dried conventionally is not evident in the foam spray-dried cheddar cheese powder. Instant coffee manufacturers also feel that large dry coffee particles retain more flavor volatiles than fine particles[8].

The data (Tables 1 and 2) show that cheese powders obtained by foam spray-drying are superior to the cheese powders dried conventionally in that no staleness was perceptible in the foam spray-dried powders. Moreover, during storage for 9 months the foam spray-dried powders maintained their excellent quality.

The shelf-life of cheddar cheese powders can be extended by the addition of antioxidants prior to homogenization of the slurries (Tables 4 and 5); however, off-flavors were sometimes observed in the samples that contained added antioxidants. The samples that contained NDGA exhibited greater stability than those powders that contained the blend of antioxidants.

The commonly used indices of oxidative deterioration (PV and TBA) appear to be variable and of limited value in determining flavor quality or storage stability of a single sample. However, these tests are useful in following oxidation of series of samples in a particular experiment. The best criterion of quality appears to be the subjective organoleptic evaluation of the powders by a qualified taste panel.

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