Gülbaran extractor is that the environment and solvent temperatures can be adjusted as desired. Owing to this property, it is advantageous in extraction procedures which should be done at low temperatures in research laboratories. In addition to giving more accurate results, the Gülbaran extractor can reduce personnel expenditures in research laboratories and lower cost in technology, product and quality control laboratories of oil factories. The latter effects are attributable to shortened extraction times.

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The Flash Devolatilization of Cocoa Butter

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A water-induced flash devolatilization process has been developed for the deodorization of cocoa butter. Per flash devolatilization efficiencies of 65-75% are high enough to give commercial quality products in a single flash.

Cocoa butter is an important ingredient in the production of chocolate. It is obtained from the cocoa bean and gives chocolate its characteristic taste, odor and consistency. Unfortunately, cocoa butter is of little value in its raw form because it contains many impurities which adversely affect its quality. A variety of refining processes have been developed to remove these impurities and improve uniformity.

An important step in the refining of cocoa butter is the deodorization process. Deodorization removes low molecular weight fatty acids and other volatile impurities from the butter. It is performed after the butter has been washed and filtered to remove bulkier impurities. Presently, the method most commonly used for deodorizing cocoa butter is steam distillation. In this process, hot, liquid cocoa butter is passed through a distillation column countercurrently to steam. The volatile compounds diffuse out of the cocoa butter into the water vapor and are removed with this water at the top of the column. Steam distillation may be performed in batch, semi-continuous or continuous modes and produces a highly deodorized butter (1). However, steam requirements are high because the interfacial contact with the cocoa butter is quite poor.

Other deodorization processes, such as chemical treatment, have been used in cocoa butter refining, but have been shown less efficient than steam distillation.

Flash devolatilization is a recently developed method for cocoa butter deodorization. It is a more efficient and economical process than steam distillation because it promotes a very large interfacial contact area between the cocoa butter and the devolatilizing medium. Flash devolatilization is a single stage process. Therefore, it requires relatively little monitoring. In addition, it requires a fairly low residence time to produce a highly devolatilized butter.

EXPERIMENTAL

Process. Flash devolatilization is a rather simple physical separation process, conceptually consisting of a single equilibrium stage. The necessary raw materials are prewashed butter and deionized, deaerated water.

The devolatilization process begins with the formation of a dispersion of about 2 wt.% water in cocoa butter. A relatively stable dispersion is achieved, without the use of emulsifying agents, when the mean particle size is less than 1 μm.

After the dispersion is formed, the mixture is quickly heated to 100-150 C under sufficient pressure to prevent boiling, e.g., 50 psi. The dispersion then undergoes a flash in which the pressure is dropped suddenly to approximately 5 torr. This pressure drop causes the water droplets to become gaseous. Because of this

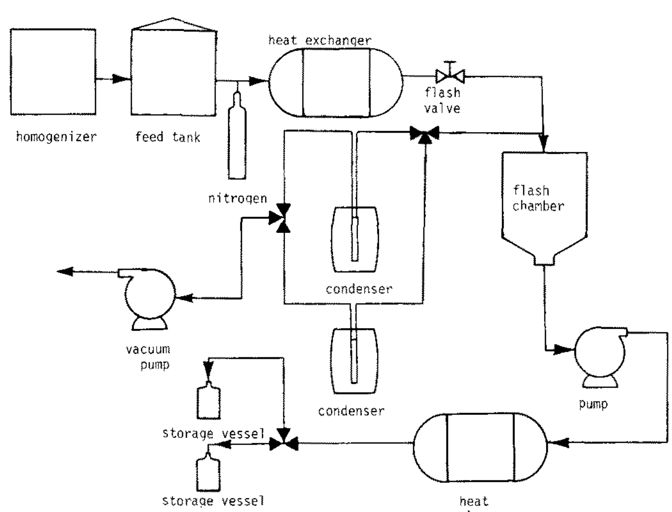

FIG. 1. Flash devolatilization process.

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change from the liquid to the gaseous state, the bubbles begin to expand and to coalesce. The water phase becomes continuous because the void fraction after the flash is approximately 0.999. The mixture is then composed of cocoa butter droplets dispersed in water vapor. The cocoa butter droplets are very fine, which implies that the interfacial area between the cocoa butter and the water vapor is large. Thus, the path length for the diffusion of volatiles is short, and a close approach to equilibrium can be achieved with minimum residence time in the vapor phase.

Equipment. A pilot plant was constructed to test the feasibility of flash devolatilization as a means for deodorizing cocoa butter. A schematic drawing of this pilot plant is given in Figure 1. The plant was designed to produce 2 lb/hr of devolatilized cocoa butter.

At the start of the devolatilization process, a water dispersion in cocoa butter was formed in a homogenizer. For convenience, this was done in batch mode although continuous operation is clearly possible. The homogenizer was a mechanical pump which forced both the cocoa butter and the water through a small (1 mm in diameter) orifice. The pressure drop across this orifice was typically 3000 psi. Because of the magnitude of the pressure drop, a stable dispersion of very fine water droplets in the cocoa butter was formed. Thus, it was unnecessary to use an emulsifying agent, such as lecithin, to stabilize the dispersion.

The cocoa butter had to be handled carefully to avoid contamination because the devolatilized samples usually underwent a taste test. For this reason, the raw butter was melted in a sterile environment. The water, which was to be dispersed in the butter, was deionized and deaerated to prevent it from degrading the butter's triglycerides through oxidation and dehydrogenation. The triglycerides are the major components of cocoa butter. In addition, the dispersion was not allowed to recycle to the homogenizer too many times because over-homogenization would cause burning. This was due to the large inputs of mechanical energy to the butter.

After its formation in the homogenizer, the dispersion was passed to a feed tank where it was stored until the run was to begin. The feed tank also eliminated any fluctuations in the feed rate.

Nitrogen under a pressure of 50 psi was used to pump the dispersion through a single-tube heat exchanger which heated the butter to the desired preflash temperature. The heat exchanger was composed of a 1/8″ tube which was surrounded by a lead casting. Heat was provided to the exchanger by electrical tape which was wrapped around the lead. The layer of lead gave the exchanger a constant wall temperature and provided uniform heating.

After the heat exchanger, the cocoa butter-water dispersion was flushed across a back pressure valve, the pressure being reduced from 50 psi to the desired flash pressure (typically 5 torr). The two-phase mixture then entered a flash chamber (Fig. 2). The cocoa butter droplets were disentrained from the vapor and fell to the bottom of the chamber, where they were removed with a small gear pump. The volatiles and water were removed through the top of the chamber and condensed and frozen in an acetone/dry ice condenser. The frozen volatiles were stored under refrigeration for subsequent analysis.

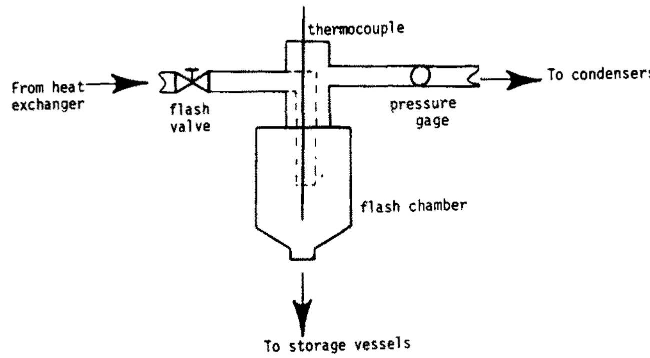

FIG. 2. Flash system.

The devolatilized butter was pumped to sterile storage vessels. These vessels were sealed and refrigerated while awaiting analysis.

Cocoa butter melts at 40 C. As an operating convenience, all process piping was contained inside a polyethylene tent with an internal temperature of about 45 C.

RESULTS AND DISCUSSION

Runs. Five sets of experiments were done to determine optimum operating conditions. In all cases, a constant flow rate of 2 lb/hr of cocoa butter and 2% by weight water was used.

In the first set of experiments, the preflash temperature was varied from 45 to 150 C, with the flash pressure held at a constant 5 torr. In the second set, the flash pressure was varied from 5 torr to 500 torr with a constant preflash temperature of 100 C.

The third set of experiments was performed to determine the effect of varying the devolatilization agent. Both water and potable ethanol were used. For these experiments, preflash temperatures of 100 and 150 C and a flash pressure of 5 torr were used.

The fourth set of experiments was performed on cocoa butter which was spiked with a high molecular weight volatile material, tetramethylpyrazine (TMP). Although this material is considered volatile, it has a rather high boiling point in comparison with most other volatile materials. Therefore, if the concentration of TMP is significantly reduced, it is assumed that the other volatile materials are reduced to an even greater extent.

The final set of experiments was performed on cocoa butter which was spiked with butyric acid. Butyric acid was chosen as a representative of lower molecular weight contaminants. It was quite easy to detect in a gas chromatograph. In addition, butryic acid is quite odoriferous, and its concentration could easily be analyzed in a qualitative sense by smelling the butter. A multipass run was performed on butyric acid spiked butter in which the butter was sent through the devolatilization process several times.

Analysis. Several types of analyses were performed on the devolatilized samples obtained in the above runs to determine their volatile concentration. The most useful were a standard taste test and a head space gas chromatographic analysis, both performed by Cadbury-Schweppes, Inc.

Cadbury-Schweppes' taste test is a combination of several standard taste analyses. A panel of professional tasters who have been properly prepared for tasting cocoa butter are given a set of reference samples as a basis for comparison. They are then given the sample to be evaluated and are instructed to compare the sample to the reference samples in terms of certain key flavor descriptions on a scale of 0 to 9, with 0 being the most devolatilized and 9 the least devolatilized.

A summary of Cadbury's taste analysis of the runs performed in the flash devolatilization pilot plant is given in Tables 1 and 2. The values presented in these tables were obtained by averaging the scores which the butter received for various taste descriptions, such as salty, cocoa and bitter. Note that even the standard has a low flavor score because a mild butter was used. The taste score for commercially devolatilized butter typically ranges from 0.3 to 0.5. Note that the samples prepared at the highest preflash temperature (150 C) and the lowest flash pressure (5 torr) were devolatilized to the greatest extent. The analysis of the runs in which the flash pressure was varied at a constant preflash temperature indicates that the lowest possible flash pressure is optimal. However, flash pressures that are significantly below 5 torr are not economically attractive on an industrial scale. Thus, the optimum flash pressure is appproximately 5 torr.

No significant difference was seen between the runs using water and ethanol as the devolatilizing agent. Therefore, the logical choice is water because it is much less expensive.

Although process feasibility was established using Cadbury-Schweppes' taste test, it was desirable to obtain a more quantitative measure of volatile concentrations. Gas chromatographic, head space analysis is a standard tool for analyzing volatiles in cocoa butter. A sample analysis, as performed by Cadbury-Sceweppes, is shown in Figure 3 and indicates the very complex nature of cocoa butter volatiles.

A simpler analysis, based on key components such as TMP and butyric acid, was useful for design and scaleup purposes. Head space analysis of the TMP spiked samples showed little difference between those prepared at a preflash temperature of 100 C and those prepared at 150 C. This indicates that the devolatilization is mass transfer controlled (i.e., diffusion limited) at preflash temperatures above 100 C. Further increases in the level of devolatilization must be achieved through the use of a higher residence time in the flash chamber.

The samples which were spiked with 1% by weight butyric acid were used to explore a multipass devolatilization. They were analyzed in a Perkin-Elmer Sigma 500 gas chromatograph with a 3′ long, 1/8″ ID Supelco glass column packed with Chromosorb W. A head space analysis was performed on the samples by heating them in a sealed sceptum bottle to 100 C and allowing the butter to equilibrate with its vapor head space. The vapor was removed with a syringe and injected directly into the GC.

Results showed that after one pass, the butyric acid concentration was decreased to 14% of its initial value while it was reduced to 10% and 5% of its initial value after the second and third passes, respectively.

The devolatilization efficiency can be defined as:

$$\mathrm{Eff} = 1 - \frac{C_{out} - C^*}{C_{in} - C^*} = \frac{C_{in} - C_{out}}{C_{in} - C^*}$$

where C* is the equilibrium concentration (here assumed to be zero) and where C_{in} and C_{out} are concentrations of the key component before and after the flash. Thus the devolatilization efficiency declined from 86% in the first pass to 30-50% in the second and third passes. This decrease in efficiency suggests that the butyric acid was not completely mixed in the cocoa butter prior to

TABLE 1

Taste Test Results—Constant Flash Pressure

Run	Temperature		
	45 C	100 C	150 C
Water medium	0.622	0.520	0.504
Ethanol medium	—	0.530	0.510
Tetramethylpyrazine	—	0.280	0.440
Standard	0.708	0.708	0.708

Flash pressure, 7 torr.

TABLE 2

Taste Test Results—Constant Preflash Temperature

Run	Pressure		
	5 Torr	50 Torr	500 Torr
Pressure variable	0.504	0.580	0.544
Standard	0.708	0.708	0.708

Preflash temperature, 100 C.

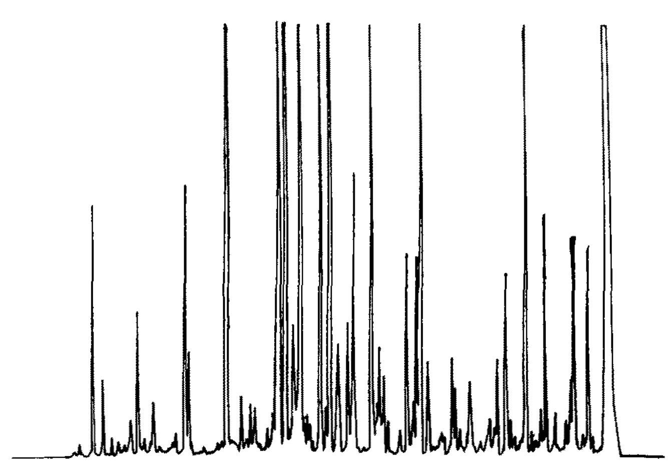

FIG. 3. Sample gas chromatographic analysis of raw cocoa butter volatiles.

devolatilization. This is a common problem when spiked samples are used in devolatilization experiments.

The chromatographic analysis for butyric acid also gave an approximate result for all other volatile components. These "total volatiles" were decreased to 27% of the initial value after one flash and to 4% after three flashes. These results indicate a preflash devolatilization efficiency of 65-75%. The taste panel results indicate that commercially acceptable cocoa butters are achieved after a single flash at this efficiency.

This study has shown flash devolatilization to be a viable method of cocoa butter deodorization. Hopefully, flash devolatilization will be further researched and refined to enable the chocolate industry to produce its product in the most economical fashion possible.

ACKNOWLEDGMENT

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