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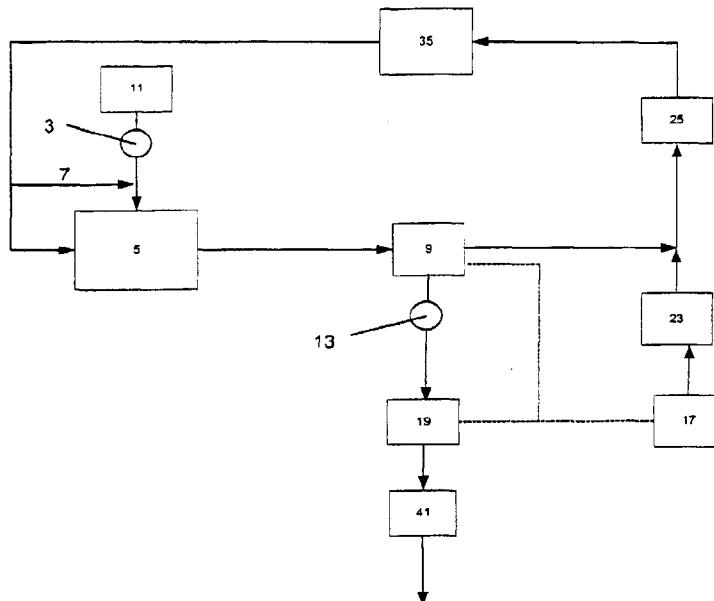
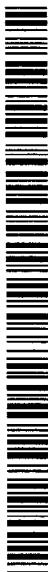
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(54) Title: FOOD MATERIALS WITH IMPROVED FLAVOR AND FUNCTIONALITY DUE TO SIZE REDUCTION IN A MODIFIED ATMOSPHERE



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(57) Abstract: Off-notes normally associated with chocolates made from ingredients containing dairy powders can be avoided using a dry mill in connection with a modified atmosphere to reduce the particle sizes of the ingredients. The modified atmosphere may be air at a temperature of about 65 °C to about 120 °C, or an atmosphere having less than about 5.0 percent oxygen.



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**FOOD MATERIALS WITH IMPROVED  
FLAVOR AND FUNCTIONALITY DUE TO  
SIZE REDUCTION IN A MODIFIED ATMOSPHERE**

5

**BACKGROUND OF THE INVENTION**

**Field of the Invention**

10

The invention relates to food processing and to methods and systems for particle size reduction of edible starting materials. Specifically, the invention utilizes dry milling for the particle size reduction 15 with either an unreactive ambient atmosphere circulating in a closed loop, or an increased temperature atmosphere. The methods and systems of the present invention may be used to produce novel chocolate products free of off-notes.

20

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#### Description of the Related Art

The smooth mouthfeel of chocolate depends to a large extent on the size of the chocolate solids contained in 5 a recipe. Large particles, exceeding 50 to 60 microns, for example, impart a gritty texture not normally viewed as acceptable. This requirement makes particle size reduction a critical step in chocolate making.

- 10 The traditional method of making chocolate employs a 2, 3, or 5 roll refiner for reducing the size of a paste containing sugar, milk, chocolate liquor and fat. Cocoa butter, milk fat, or a combination of these, may be used depending on the desired recipe. Agitated ball 15 mills, stone mills and refiner conches have also been used. All of these wet grinding apparatus are known to be labor intensive to operate and expensive to maintain.
- 20 In recent years, as consumers have demanded lower fat content chocolates, the roll refiner has met with limitations. International Application WO 99/45789, incorporated by reference herein in its entirety, discloses using particle size control to obtain 25 chocolates having fat content below about 25 weight percent.

Dry milling of chocolate ingredients in an air-jet mill, air classifying mill, hammer mill, or the like, 30 permits improved particle size control. Even for conventional recipes having greater than 25 percent fat, dry milling provides rheological advantages, as disclosed in V. Eynck, "Dry Grinding and Mixing of

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Compound Coatings," 43<sup>rd</sup> P.M.C.A. Production Conference (1989), pp. 35-38.

In view of the foregoing, it would be desirable to move 5 toward dry milling of chocolate and chocolate precursors from the standpoint of efficiency, cost and finished product quality.

However, dry milling has been found to lead to loss of 10 desirable flavors and in many cases to the development of off-notes. These off-notes are flavors not normally found in chocolate and generally seen as undesirable. The inventors have discovered several off notes arising 15 in the dry milling of chocolate ingredients, including "metallic," "fishy," "cardboard," "wet-dog" and "rancid" notes that can carry over to chocolate made from these ingredients. The extent of the off-note intensity has been linked to the source of the raw materials. Dairy powders derived from milk of pasture-fed cattle appear to have a greater propensity to 20 develop these undesirable flavors. This has severely limited the use of dry milling for widespread chocolate manufacture. Without wishing to be bound by any theory of what is responsible for these phenomena, it is believed that exposing nonfat milk, whole milk, lactose, sugar (sucrose), cocoa, flavorings and combinations thereof to attrition in combination with 25 high volumes of air is sufficient to create the objectionable off-notes.

30

The solution to the problem of off-notes has proven to be elusive. Addition of very effective natural and synthetic anti-oxidants including TBHQ, BHT, BHA, and

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vitamin E to a dry mix of powders prior to milling has no significant impact on the off-note generation.

International Application WO 93/19629 discloses a 5 method of preserving foods using noble gases. With regard to chocolate, the method entails contact, and preferably the saturation of, chocolate with a noble gas such as argon, at any stage; and preferably at every stage of chocolate production. The above 10 International Application does not disclose or suggest dry milling of chocolate ingredients.

#### SUMMARY OF THE INVENTION

15 This invention is directed to methods and systems for manufacturing powdered edibles free of off-notes. The invention allows for the integration of dry milling into the production of chocolate in a continuous process without using a roll refiner.

20 A method according to one embodiment of the invention comprises feeding an edible composition comprising milk powder and containing less than about 15 percent fat through a dry mill in continuous contact with air

25 having a temperature between about 65°C and about 120°C to produce reduced-size particles, and recovering the reduced-size particles. In a more preferred embodiment, the exit temperature of the dry mill (dry mill exit temperature) is between about 68°C and about

30 85°C. In the most preferred embodiment, the dry mill exit temperature is between about 70°C and about 75°C.

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In a preferred embodiment, the dry mill used with the system is an air classified mill which reduces the particle size of the starting material to between about 10 and about 60 microns. After particle size reduction, the particles may be combined with chocolate liquor, cocoa butter and other materials to make chocolate. When referring to particle size herein, the measurements were taken using a micrometer.

5

10 In preferred chocolate-manufacturing embodiments, the starting material comprises a milk powder and contains between about 0.08 weight percent and about 12 weight percent fat, and the particles obtained are free of off-notes. In preferred embodiments the starting material contains between about 10 and about 12 percent fat.

15

In another embodiment, the system of this invention includes a closed gas circulation loop containing a 20 circulating gas; a fan to circulate gas continuously throughout the closed gas circulation loop; a source of substantially unreactive gas operatively connected to the gas circulation loop; a damper to control the addition of the substantially unreactive gas to the 25 circulating gas; a source of edible starting material; a dry mill operatively connected to the source of edible starting material to reduce the particle size of the edible starting material to form product particles; and a gas-solid separator to recover the product 30 particles from the circulating gas. In a preferred embodiment, the edible starting material and the product particles are continuously contacted with said circulating gas.

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A preferred method of making chocolate using the above-described closed system comprises providing a substantially unreactive gas to the closed system, feeding a composition comprising chocolate liquor, 5 powdered milk product and sugar and containing less than about 15 percent fat through a dry mill in contact with the circulating gas to produce particles, recovering the particles from the circulating gas, and combining the particles with cocoa butter, additional 10 chocolate liquor and other ingredients to make chocolate.

**BRIEF DESCRIPTION OF THE DRAWINGS**

15 Figure 1 is a diagram of an open-loop system according to the invention.

Figure 2 is a diagram of a closed-loop system according to the invention.

20

**DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

"Air classified mill" and "classified mill" are used interchangeably herein to refer to a particle size reduction unit in which particles are borne by air or other gas, and in which sorting by particle size occurs in the particle size reduction unit as a result of gravitational forces.

30 The term "chocolate" is intended to refer to all chocolate or chocolate-like compositions with a fat phase or fat-like composition. In the United States, chocolate is subject to a standard of identity (SOI)

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established by the U.S. Food and Drug Administration under the Federal Food, Drug and Cosmetic Act.

Definitions and standards for the various types of chocolate are well established in the U.S.

- 5 Nonstandardized chocolates are those chocolates which have compositions which fall outside the specified ranges of the standardized chocolates. The term "chocolate," as used herein, is intended to include standardized and non-standardized chocolates, i.e.,
- 10 including chocolates with compositions conforming to the SOI and compositions not conforming to the SOI, including dark chocolate, baking chocolate, milk chocolate, sweet chocolate, semi-sweet chocolate, buttermilk chocolate, skim-milk chocolate, mixed dairy
- 15 product chocolate, low fat chocolate, white chocolate, aerated chocolates, compound coatings, non-standardized chocolates and chocolate-like compositions. Chocolate also includes products containing crumb solids or solids fully or partially made by a crumb process.
- 20 Nonstandardized chocolates result when, for example, the nutritive carbohydrate sweetener is replaced partially or completely; or when the cocoa butter or milkfat are replaced partially or completely; or when components that have flavors that imitate milk, butter
- 25 or chocolate are added or other additions or deletions in formula are made outside the FDA standards of identity of chocolate or combinations thereof. For the chocolate made according to the invention the only critical characteristic is that it be made with a dry
- 30 milled precursor having a fat content less than about 15 percent, and that it is substantially free of off notes.

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"Chocolate precursor" as used herein means a dry milled powder that can be combined with other ingredients to make chocolate.

- 5 "Closed loop system" as used herein means a system substantially closed to outside air. It will be understood that some air enters with the feed, and from other incidental sources.
- 10 "Dry milling," as used herein, means particle size reduction of materials having a fat content less than 15 percent, in which the particles are surrounded by a modified atmosphere during the particle size reduction. In dry milling, as used herein, particles are entrained 15 in a flow of the modified atmosphere during one or more stages of the particle size reduction, requiring separation of the particles from the flow. Dry milling apparatus that may be used in the step of dry milling according to the invention include, but are not limited 20 to: air classified mills; hammer mills, with or without screens or other internal classification; pin mills; media mills; fluid energy mills; disk mills; and stone mills. A preferred dry milling apparatus for use with the present invention is an air classified mill.
- 25 "Milk powder," as used herein, includes skim milk powder, whole milk powder, blends, buttermilk powder, whey powder and lactose enhanced powders and enzymatically modified milk powders.
- 30 "Modified atmosphere" as used herein means either an atmosphere having an oxygen content of less than about

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5 percent, and/or an atmosphere which has a temperature greater than about 65°C.

"Open system" or "open loop system" simply means that 5 air from outside the system is used as a source of atmosphere in the dry mill. Of course, to produce an atmosphere having a temperature between about 65°C and about 120°C around the particles being milled, the air has to be heated. Therefore, the atmosphere is 10 modified even though the outside air is used as a source of atmosphere.

It has surprisingly been found that a relationship exists between the temperature of the atmosphere 15 surrounding edible particles subjected to dry milling, the oxygen content of the atmosphere and off-notes generated in the milled edible powders. On one hand, the generation of off-notes can be prevented by providing a reduced-oxygen ambient atmosphere. At low 20 temperatures, between about 10°C and about 25°C, an oxygen content in the atmosphere less than about 1 percent will prevent the generation of off-notes. At an intermediate temperature, between about 50°C and about 60°C, off-notes can be prevented if the oxygen 25 content of the atmosphere is maintained between about 2 percent and about 5 percent. In general, such reduced-oxygen atmospheres (less than 5 percent oxygen) are provided by continuously adding a make-up amount of substantially unreactive gas to a closed system having 30 an atmosphere of predetermined composition, as will be described in greater detail below.

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It has also been found that by operating at elevated temperatures (between about 65°C and about 120°C), edible particles can be dry milled in air (which has an oxygen content of about 21 percent) without off-notes 5 being generated. At these elevated temperatures, an open system can be used without generating off-notes. The discovered relationship of temperature, oxygen content and off-notes is in some sense counterintuitive, as an increase in temperature would 10 generally be associated with an increase in the reactions thought to generate off-notes in the first place.

Between about 60°C and 70°C, it has been observed that 15 the occurrence of off-notes is suppressed. It has been noted that this corresponds with the glass transition temperature of the sugars commonly included in the edible starting materials. Without intending to be bound by theory, there may be an association between 20 these two events. A practical upper limit is placed on the temperature used in the open system by the browning or other adverse color changes that may occur at temperatures above about 120°C. The upper limit of temperature at which milling may take place in an open 25 system without inducing browning is time-dependent. Browning may occur at lower temperatures if the residence time in the mill is longer. Therefore, the operational range is considered to be between about 65°C and about 120°C. A preferred operating range is 30 between about 65°C and about 85°C. A most preferred range is between about 70°C and 75°C.

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The invention as embodied in an open loop system will now be described in connection with Figure 1.

Figure 1 is a flow diagram of an open system according 5 to a preferred embodiment of the invention. Edible starting material is fed from hopper 21 to dry mill 25. Air is also provided in the dry mill from source 33. It will be understood that the air is simply drawn from outside the system, and the atmosphere source 33 may 10 represent air conditioned by heating, humidifying or dehumidifying, as desired. It should be understood that hot air can be obtained by selecting a location where the air temperature is consistently elevated.

15 The edible starting material may comprise, for example, a confectionary ingredient, including sugar (sucrose), lactose, milk powder, cocoa, nutmeat, flavoring and combinations thereof, and included fats, such as cocoa butter, milk fat or other oils, and flavorings and 20 could also include crumb. However, the starting material should not contain more than about 15 percent fat. The starting material preferably contains between about 10 percent and about 12 percent fat. In some embodiments, for example when skim milk powder is used, 25 the fat content of the starting material can be as low as 0.08 weight percent.

Any edible starting material that might produce an off-note due to attrition and exposure to high volumes of 30 air is suited for use with the invention. Preferably, the foodstuff is a constituent used in the manufacture of chocolate. Such constituents typically comprise sugar, such as sucrose, lactose, or a combination

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thereof, and whole milk or skim milk powders. The constituent may also contain chocolate liquor, oils, cocoa butter, salt, vanillin and other ingredients commonly found in chocolate. In a preferred 5 embodiment, the methods and systems of the invention are advantageously applied to food products that contain non-U.S. origin milk products. These products are believed to be implicated in the formation of off-notes. Sources of non-U.S. milk powders include but 10 are not limited to: Argentina, Australia, Canada, China, France, Ireland, New Zealand, Russia, and the United Kingdom.

A preferred dry mill according to both the open and 15 closed loop embodiments according to the invention is an air classified mill. Although not customarily used in this context in chocolate manufacture, the basic design of an air classified mill is known in the art. An air classified mill typically constitutes a lower 20 part and an upper part. The lower part comprises a grinding chamber with a rotor and hammers which contact and comminute the particles fed from the hopper. In the open system, conditioned air enters through an annular space in the lower part of the air classified 25 mill and travels upward, carrying the particles into contact with the rotor and hammers of the grinding chamber and also the walls of the mill. The ground particles are sorted by size in the classifier vanes in the upper part of the air classified mill. Only the 30 particles having the desired particle size continue on to the solid-gas separation stage 27. Larger particles remain in the mill as a result of the gravitational

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forces acting thereon, which is an advantage of the classified mill technology.

Preferably, the particle size of the powders leaving  
5 the air classified mill for use in chocolate  
compositions is in a range of about 10-60 microns.  
Most preferably the particle size of the powders  
leaving the air classified mill is between about 20 and  
about 40 microns. Particle size may be controlled by a  
10 fan determining the velocity of the air traveling  
through the mill. Alternatively, the speed of the  
grinding rotor or the classifier vanes can be modified  
to obtain the desired particle size. Suitable air  
classified mills for use with the present invention are  
15 available for example from Hosokawa Micron Powders  
Systems, Summit, New Jersey. The selection of an  
appropriate, commercially available model and its  
operating parameters would be within the skill of one  
of ordinary skill in the art.

20 Generally there is a temperature rise across a dry mill  
due to the mechanical action taking place therein. In  
the case of an air classified mill, the rise in  
temperature can range between about 15°C and about  
25 35°C, depending on the flow rate and particle size in  
the mill. In order to maintain an exit temperature  
between 65°C and 120°C, the air entering the dry mill  
from source 33 should have a temperature in the range  
of about 30°C to about 105°C. The relative humidity of  
30 the air entering the dry mill from source 33 is  
generally in the range of about 0 percent to about 25  
percent. When a temperature of the modified atmosphere

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is specified herein, that is understood to refer to the exit temperature.

Recovery of the solid particles is accomplished in a 5 gas-solid separator 27. If the dry mill is an air classified mill, a dust collector (baghouse) is advantageously used as the gas-solid separator. However, alternative gas-solid separators are known, and the selection, design, and operation of appropriate 10 gas-solid separators would be readily apparent to one of ordinary skill in the art. At the pilot scale, recovery of the solid particles has been accomplished with a cyclone separator, which removes 90-95 percent of the particles, followed by a dust collector.

15

Chocolate precursor particles recovered from the gas-solid separator may be processed into chocolate by forwarding the precursor particles to a conche stage 31. In conche stage 31, the precursor particles are 20 combined with a source of additional chocolate liquor and cocoa butter 28 and slowly kneaded at elevated temperatures. The time and temperature of the conche is dictated by the type of chocolate being produced.

25 Additional fat-containing ingredients include, without limitation, cocoa butter, chocolate liquor, milk fat, vegetable fat and fat substitutes. Preferably, the additional fat-containing ingredients are added within one to three hours of recovering the particles, more 30 preferably within 15 minutes of recovering the particles.

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Figure 2 depicts a particle size reduction system according to a closed-loop embodiment of the invention. In the closed loop, a circulating gas having a substantially constant composition continuously 5 circulates through the system. Preferably, to eliminate off notes, the circulating gas contains less than about 5 percent oxygen. Depending on the temperature, the circulating gas may contain less than 2 percent oxygen or less than 1 percent oxygen.

10

Edible starting material is fed from hopper 11 through rotary gate-lock 3 to a dry mill 5 to prevent outside air from infiltrating the closed loop system. Other means of feeding edible starting material to the mill 15 that will inhibit the entry of outside air into the closed loop system, may also be used. For example, a screw feeder, or other alternatives known to those of ordinary skill in the art, may also be used.

20 A certain amount of air enters the closed gas-circulation system through the rotary gate-lock with the edible starting material. This addition affects the composition of the circulating gas, and is compensated for and controlled by the addition of a 25 substantially unreactive gas, as set forth below. In preferred embodiments where the mill is an air classified mill, a side-stream of gas 7 may be diverted to entrain edible starting material as it is fed to the classified mill.

30

The selection and operation of an appropriate dry mill is described above in connection with the open loop system, except of course that a continuously

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circulating gas is used. Particles leaving the mill are directed to gas-solid separator 9 where they are recovered.

5 In a preferred embodiment, where the dry mill is an air classified mill, product particles borne from the top part of the mill are recovered in product collection area 19 at the bottom of the gas-solid separator through a gate-lock 13, which prevents the entry of  
10 outside air into the closed gas-circulation system.

Substantially unreactive gas from source 17 may be provided to the product collection area 19 to ensure that the product particles are contacted by an  
15 unreactive environment throughout the process. In preferred embodiments, where the product particles comprise the constituent ingredients of chocolate, the product particles are incorporated with other ingredients to make chocolate precursor in a conching  
20 stage, preferably, within about 1 to about 3 hours of the particle size reduction in the dry mill.

Circulating gas exits the top of the dust collector (or other gas-solid separator) 9 and proceeds through fan  
25 25. The damper 23 controls the composition of the circulating gas determining how much substantially unreactive gas is added to the closed gas-circulating system. The circulating gas preferably has an oxygen content less than 5 percent, less than 2 percent, or  
30 less than 1 percent. As noted above, there is a relationship between the operating temperature of the closed loop system, the oxygen content of the circulating gas, and the generation of notes. The cost

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of operating at lower oxygen levels must be assessed against the desirability of operating in a given temperature range.

5 The composition of the circulating gas is maintained by adding a substantially unreactive gas to the closed loop system. Substantially unreactive gas includes, without limitation, nitrogen, carbon dioxide, and noble gases, such as argon and xenon. Nitrogen is a  
10 preferred substantially unreactive gas.

If the substantially unreactive gas is nitrogen, any convenient source may be employed, such as a source of liquid nitrogen coupled with an evaporator. In larger  
15 scale facilities, nitrogen is preferably provided by a commercially available nitrogen generator using membrane or pressure swing adsorption technology. The nitrogen source used in the present invention is used for the purpose of controlling the ambient atmosphere  
20 to which the particles are exposed, and it is not necessary that the nitrogen provide cooling to the system. Nitrogen from source 17 may be routed to the top of the dust collector, the bottom of the dust collector, the product collection area 19 and to the  
25 inlet of damper 23, as desired.

Preferably, the closed gas circulation system is used in connection with the manufacture of chocolate, and when the product particles contain powdered milk  
30 products, the oxygen content of the system is below about 5.0 percent. Most preferably, under these circumstances, the oxygen content of the circulating gas stream is between about 0.1 percent and about 2.0

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percent. Where possible, nitrogen addition to the system will be minimized, nitrogen being an expense. The nitrogen make-up requirements to maintain a given oxygen concentration in a closed gas-circulation system 5 were observed to be generally about 0.7-1.0 percent nitrogen added to the circulating gas to maintain an oxygen concentration of 1.5 percent; about 1.5 percent nitrogen added to maintain an oxygen concentration of 1.0 percent; and about 2.5-3.0 percent nitrogen to 10 maintain an oxygen concentration of 0.5 percent, where the percentages of added nitrogen given are with respect to the circulating gas flowrate. Of course, these requirements will differ depending on the design of the particular closed loop system.

15

Circulating gas, which will experience pressure drop and temperature rise through the mill 5, gas-solid separator 9, and fan unit 25, will be conditioned in gas conditioner 35 which may include a cooling coil 20 and/or dehumidifier, as necessary. A pressure relief valve (not shown) may also be provided as an additional means of controlling the pressure in the system.

The closed loop gas circulation system is substantially 25 sealed from the outside air and may have either a slightly positive or slightly negative pressure overall. Preferably, the entire system operates in the range of about 0 psi to about 1 psi (gauge), and is not expected to run at extremes of either positive or 30 negative pressure. In operation, the pressure at the outlet of the fan may be slightly positive, while the pressure at the inlet of the classified mill to the inlet of the fan may be slightly negative. If the

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pressure in the system is slightly positive overall, outside air is prevented from leaking into the system. On the other hand, positive pressure inside the system may cause dusting as particles are entrained in 5 escaping gas, and the escaping gas itself may present problems of safety and expense. As described above, dampers at the exit of the classified mill and at the exit of the fan control the pressure at those locations. Because nitrogen (or other inert gas) is an 10 expense attendant with the methods and systems of the invention, care must be taken to prevent nitrogen leaks or ingress of outside gas into the system, where possible. The leakage areas of particular importance are the fan seal, dust collector, doors on the dust 15 collector, and the pressure relief valve.

Prior to operating the closed loop system with a reduced-oxygen ambient atmosphere, the system is purged of air. The time necessary to purge a closed loop 20 system has manufacturing implications. The rate of purging is limited by the volume of gas delivery and pressure rating for a given system, which in turn is usually dictated by the explosion diaphragm in the dust collector. A 1-5 psi burst pressure is typical.

25 Closed loop milling inherently has the potential to recycle flavors, as well as moisture, with freshly milled product. Flashing of small levels of moisture is believed to occur when an air classified mill is run 30 on an open loop, (i.e. when an unreactive gas is not supplied, and the mill is run with a continuous supply of air.) If this moisture were observed to accumulate in the product in a closed-loop system, a drier/cool

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could be added to the system as part of gas conditioner 35.

The inventors have found that closed-loop milling in a 5 reduced-oxygen ambient atmosphere, below about 5.0 percent oxygen, eliminates off-notes in the milled powders. Nevertheless, certain undesirable flavors or notes may develop over time in edibles processed in the closed-loop classified mill particle size reduction 10 system of the invention. The inventors have found that these late-developing notes can be avoided if they are processed into chocolate within about one to about 3 hours of particle size reduction.

15 Product collected at the base of the gas-solid separator may then be further processed to make foods such as chocolate. In some embodiments, the milled material is combined with cocoa butter, chocolate liquor and lecithin in conche stage 41. This 20 combination may be performed in a pre-mixer, or the materials may be added directly to a conching stage. In either case, the refining step of the prior art, conventionally carried out in a roll refiner, is avoided using the dry milling apparatus according to 25 the invention. After conching, the product may be tempered and crystallized to produce finished product, as would be apparent to one of ordinary skill in the art.

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EXAMPLE 1

To demonstrate an open-loop system according to the invention, a chocolate precursor powder having the following composition was prepared by mixing the 5 following ingredients in a D-300 mixer available from Hobart for 10 minutes at speed #1:

Table 1

Ingredient	Mass (kg)
Sugar	8.0
Chocolate Liquor	0.9
Whole Milk Powder	3.0
Lactose	1.0
Vanillin	0.0116

15

The above composition was milled in a 10 ACM air classified mill, commercially available from Blue Tech, Inc., Hickory, North Carolina. The mill was operated with a classifier speed of 2200 RPM, a rotor speed of 20 3800 RPM and a feed rate of 35 kg/hr to obtain particles having a nominal particle size of 40 microns. Air entering the classified mill had an inlet temperature of 75°C, and a relative humidity (before heating, at 10-15°C) of 8 percent. Due to the small 25 scale of the experiment, the inlet temperature was taken to be approximately equal to the exit temperature.

Milled powders were recovered in a gas-solid separation 30 system and combined within fifteen minutes with further ingredients to make a chocolate recipe having the following composition:

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Table 2

Ingredient	Mass (kg)
Milled Powder (from Table 1)	3.0
Chocolate Liquor	0.9
Cocoa Butter	0.5
Lecithin	0.023

Conching was performed in a nominal 10# Teledyne Readco 10 sigma blade mixer for a 1.5 hour dry conche, followed by a 1 hour wet conche at a jacket temperature of 60°C. Chocolates were tempered in tablet form and stored at ambient temperature (20°C).

15 At one week of age, the chocolates were melted and evaluated for the presence or absence of a fishy off-note. The results are tabulated below.

COMPARATIVE EXAMPLES 1-3

20 Chocolate powders were prepared as in Example 1, except that the inlet temperature to the air classified mill was set to 53°C, 60°C, and 67°C for three different batches. The resulting chocolates were evaluated for 25 the presence of off-notes. The results are tabulated below:

Table 3

Example	Temperature (°C)	Off-note present
Ex. 1	75	No
Comp. 1	53	Yes
Comp. 2	60	Yes
Comp. 3	67	Slight

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It has been determined that because of the larger particle flow rates and lower volumes (proportionally) of air in contact with the particles experienced at the production scale, 65°C is an appropriate exit 5 temperature to avoid off-notes. A preferred dry mill exit temperature to avoid off notes is between about 68°C and about 85°C.

To determine the flavor effect on a chocolate 10 formulation using a variety of milk powders from U.S. and non-U.S. sources milled in a low oxygen environment using inert gas, chocolates were prepared using whole milk powders from U.S., Canadian, Australian, and Argentinian sources.

15 Samples of chocolates using these milk powders were prepared using "open loop" milling (Comparative Example 4) and "closed loop" milling (Example 2) for particle size reduction. In both cases, a pilot scale 20 air classifier mill was used. In the "open loop" method, air freely passes through the entire milling system. In the closed loop system, the entire system is closed and purged with nitrogen until the oxygen level is reduced to about 1.0 percent. The particles 25 are fluidized in the circulating nitrogen gas in the mill.

#### EXAMPLE 2

Two batches of the dry ingredients (10 kg sucrose, 4 kg 30 whole milk powder (WMP), 2 kg lactose, and small amounts of salt and vanillin) were mixed in a Hobart model D-300 mixer at speed #1, 10 min., at ambient temperature.

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Prior to milling each sample 25kg of sugar is milled to insure that the mill is clean. After milling the sugar one of the batches is milled to further flush the mill. The second batch is then milled and the sample is taken 5 for chocolate making.

The milling conditions for each different milk powder (Examples 2(a) to 2(d)) are listed below.

Table 4

10	Classifier (rpm)	Rotor (rpm)	Feed Rate (kg/hr)	% O <sub>2</sub> in mill	ACM Inlet Temp (°C)
US WMP	2500	3500	35	1.1	16
Canadian	2100	3500	35	1.4	18
Argentina	2200	3500	35	1.0	18
Australia	2200	3500	35	1.3	18

15 A total of 2.5 kg of the milled ingredients are added to a Teledyne Readco 6 quart Sigma lab mixer. 0.5 kg of cocoa butter, 1.0 kg of chocolate liquor, and 0.1 kg of anhydrous milk fat are melted and added to the milled 20 material. The water jacket temperature remains at 50°C. This mixture is 'dry conched' at 50°C for 1.5 hrs. At the end of the dry conche cycle 20 g of lecithin is added, the mixture is then 'wet conched' for one hour. The finished chocolate is hand tempered and molded into 25 bars.

#### COMPARATIVE EXAMPLE 4

The dry ingredients (10 kg sucrose, 4 kg whole milk powder (WMP), 2 kg lactose, and small amounts of salt 30 and vanillin) were mixed in a Hobart model D-300 mixer

at speed #1 for 10 minutes at ambient temperature. The mixture was milled to 40  $\mu\text{m}$  using an air classified mill with air conditioned to 8 percent relative humidity and 15°C. The mill was set to operate using a classifier RPM of 2300, a rotor RPM of 4100 and a feed rate of 35 kg/hr. The milled ingredients were 5 processed into chocolate in the same manner as described in connection with the immediately preceding Example 2.

Each chocolate produced using ingredients milled on the closed loop process were described by an expert panel as having typical chocolate attributes while those chocolates milled with air contained undesirable "wet dog" or "fishy" 10 attributes, and possessed lower chocolate intensity, as shown in Table 5 below.

Table 5

Example	Oxygen Conc.	Off-note Present
Ex. 2 (a)	1.1	No
Ex. 2 (b)	1.4	No
Ex. 2 (c)	1.0	No
Ex. 2 (d)	1.3	No
Comp. Ex. 4	21% (Air)	Yes

15 Variations and modification of the disclosed invention will be apparent to those of ordinary skill in the art, and are within the scope of the invention as described in the appended claims.

**The claims defining the invention are as follows:**

1. A method of preparing edible particles free of off-notes including the steps of:
  - a) introducing an edible composition containing less than about 15 percent fat to a dry mill having an air atmosphere wherein said air has a dry mill exit temperature between about 65°C and about 120°C,
  - b) dry milling the composition to produce particles, and
  - c) recovering the particles.
2. The method of claim 1, wherein said dry mill is an air classified mill and said particles have a particle size between about 10 and 60 microns, and said recovering is performed in a dust collector.
3. The method of claim 2, wherein said edible composition includes milk powder and contains between about 0.08 percent and about 12 percent by weight of fat.
4. The method of claim 1 wherein said edible particles constitute a chocolate precursor.
5. The method of claim 1, wherein said edible composition includes a milk powder, and an ingredient selected from the group consisting of sucrose, lactose and combinations thereof.
6. The method of claim 5, wherein said air has a dry mill exit temperature between about 68°C and about 85°C.
7. A method of preparing chocolate including the steps of preparing edible particles according to the method of claim 4 and mixing the particles with

additional fat-containing ingredients and emulsifier in a conching stage to make chocolate.

8. A method of preparing edible particles for use in chocolate or a chocolate precursor, comprising:

a) providing a closed system in which a circulating gas containing less than 5.0 percent oxygen continuously circulates,

b) providing a substantially unreactive gas to said closed system,

c) feeding an edible starting material containing less than about 15 percent fat through a dry mill in contact with the circulating gas to produce edible particles, and

d) recovering the edible particles from the circulating gas.

9. The method of claim 8, wherein the dry mill is an air classified mill and the edible starting material is fluidized in a stream of the circulating gas in the air classified mill.

10. A method according to claim 9, wherein the edible starting material includes a milk powder.

11. A method according to claim 10, further including a step of adding chocolate liquor and cocoa butter to said edible particles within about 3 hours of recovering said edible particles from said circulating gas.

12. A method according to claim 8, wherein the circulating gas contains less than 2 percent oxygen.

13. A method according to claim 8, wherein the substantially unreactive gas comprises nitrogen.

14. A method according to claim 8, wherein the edible particles have a particle size from about 10 microns to about 60 microns.

15. The method according to claim 10, wherein said edible particles are substantially free of off-notes.

16. A method of preparing chocolate including the steps of,

- a) providing a closed system in which a circulating gas continuously circulates,
- b) providing a gas containing less than about 5.0 percent oxygen to said closed system,
- c) feeding a composition including chocolate liquor, a powdered milk product, and sugar, and containing between about 0.08 percent and about 12 percent fat, through a classified mill in contact with said circulating gas to produce particles,
- d) recovering said particles from said circulating gas, and
- e) combining said particles with additional fat-containing ingredients in a conche stage to produce chocolate.

17. The method according to claim 16, further including tempering and crystallizing the chocolate after said step of combining to obtain a chocolate product free of off-notes.

18. A system for the manufacture of edible particles including:

- a) a closed gas circulation loop containing a circulating gas;
- b) a fan to circulate said circulating gas continuously throughout the closed gas circulation loop;

c) a source of substantially unreactive gas operatively connected to said gas circulation loop;

d) a damper to control the addition of said substantially unreactive gas to said circulating gas;

e) a source of edible starting material;

f) a classified mill operatively connected to said source of edible starting material to reduce the particle size of said edible starting material to form edible particles; and

g) a gas-solid separator to recover said edible particles from said circulating gas;

wherein said edible starting material and said product particles are continuously contacted with said circulating gas.

19. A chocolate product substantially free of an off-note, wherein the chocolate is prepared with a constituent powder having less than about 15 percent fat that has been reduced in particle size in a reduced-oxygen ambient atmosphere.

20. A chocolate product substantially free of off-notes made by the process of:

dry milling edible starting material containing a milk powder and having a fat content less than about 15 percent by weight in a modified atmosphere to obtain a milled chocolate precursor and adding chocolate liquor and cocoa butter to said milled chocolate precursor within about three hours of obtaining said milled chocolate precursor.

21. The chocolate product of claim 20, wherein the modified atmosphere is air having a temperature between about 65°C and 85°C.

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22. The chocolate product of claim 21, wherein the modified atmosphere is a continuously circulating atmosphere having an oxygen content less than about 5.0 percent.

23. A chocolate precursor including chocolate liquor, a powdered milk product, and sugar, and including between about 0.08 percent and about 12 percent by weight fat, having particles with sizes between about 10 and about 60 microns, wherein said particles are formed in a modified atmosphere.

**DATED** this 11 day of January 2006  
**MARS, INCORPORATED**

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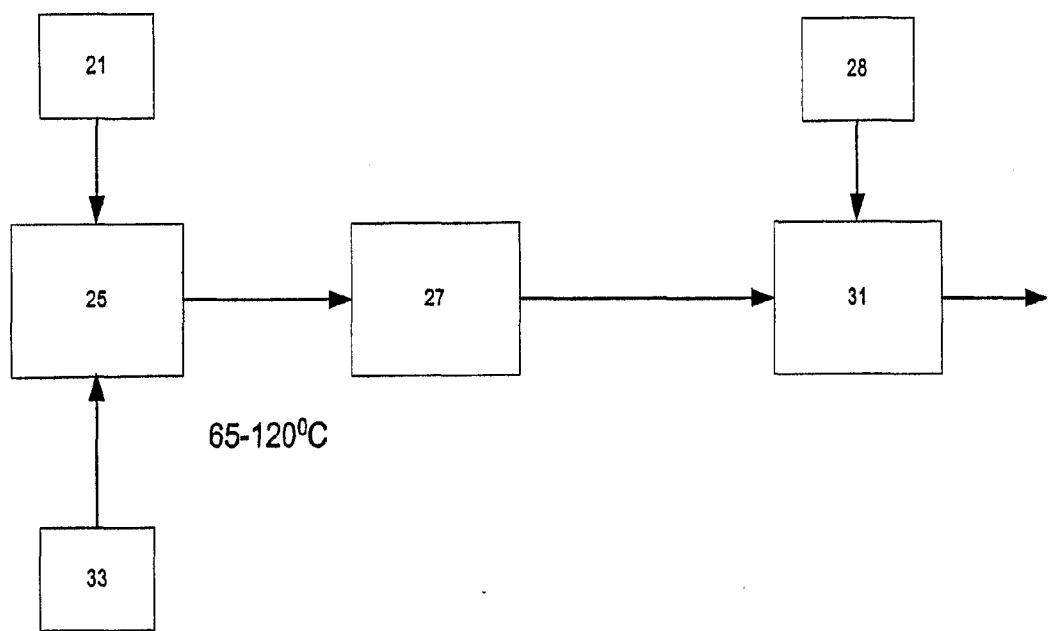


Figure 1

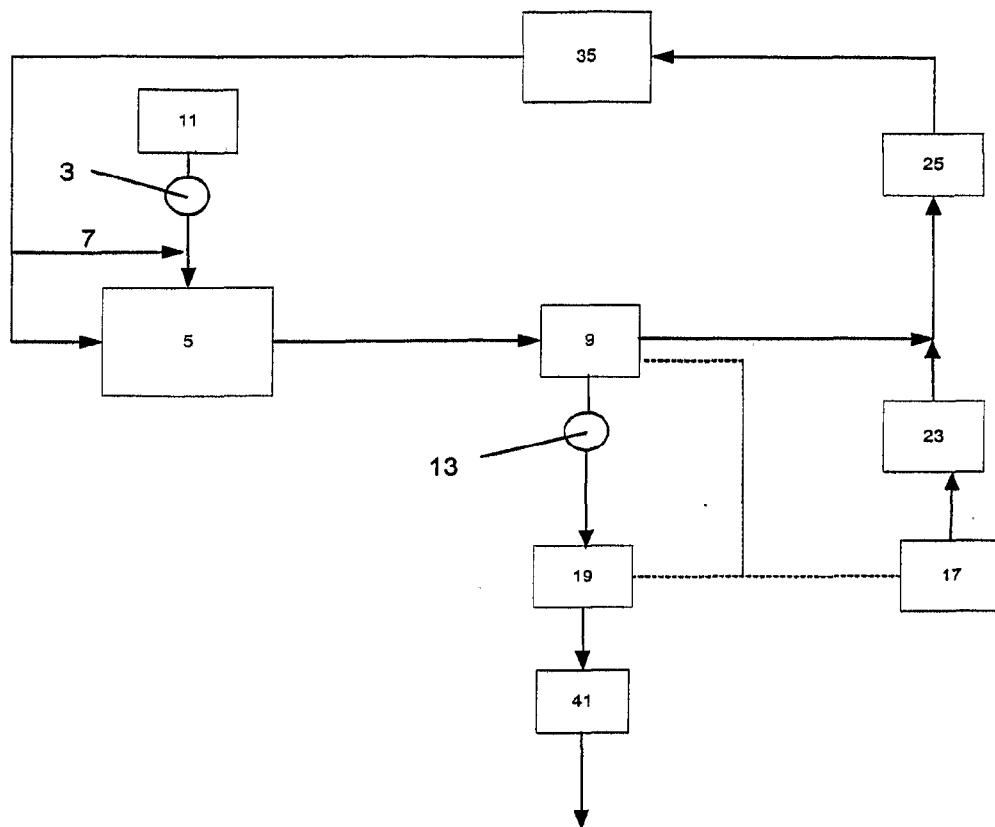


Figure 2