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- (72) **Inventor; and**
- (71) **Applicant :** ZUMBE, Albert [GB/GB]; 23 Glendon Way, Dorridge, Solihull B93 8SY (GB).
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(54) **Title:** PROCESS FOR THE MANUFACTURE OF TEMPERED COCOA BUTTER POWDER AND OTHER POWDERED PRODUCTS WHICH CONTAIN COCOA BUTTER

(57) **Abstract:** Cocoa butter or a cocoa containing composition including cocoa liquor or chocolate is tempered and then cryogenically milled to form a tempered powder which if stored correctly will retain its free flowing properties and will not clump or bloom.

## Technical Field of Invention

This invention relates to the process of the cryogenic milling of tempered cocoa butter and cocoa butter containing compositions that retain their tempered properties.

Cocoa butter powder, cocoa liquor powder and full fat chocolate powder already exist and are manufactured using various technologies. They all involve processes that generate heat or involve heating the chocolate to above the melting point of the cocoa butter, which will de-temper the cocoa butter. Upon uncontrolled cooling the cocoa butter crystals may not form in the required crystalline format thus causing the powder to be more unstable than properly tempered powder.

If these cocoa butter powder or cocoa butter powder compositions are not stored properly at the correct temperature and relative humidity the powder particles will clump much more easily to form a solid mass. Tempered powder will be more resilient; retain better free flow properties and void of clumps.

Properly tempered cocoa butter powder and cocoa butter containing powder compositions such as cocoa liquor and chocolate can be used as a "seed" to temper liquid chocolate mass.

This technology is not relevant for cocoa butter powder compositions of less than 15% total fat content because at this level the powder composition is pretty stable against clumping and then compacting into a solid mass upon storage and can be stored at higher temperatures for prolonged periods of up to 20 C without any surface fat melting and bridging to create a solid block. Indeed cocoa powder with 12% fat, which is essentially non tempered, is extremely resistant to compacting into a solid mass. Cocoa powder with 22% fat, also essentially non tempered, is not so resilient and has to be handled more carefully, stored at cooler temperatures and avoiding even short exposures of warm temperature.

Cocoa butter melts at 17-36C depending on the crystalline format and there are considered to be six crystalline formats. The most stable form is Type 5 and this is the required format for tempered cocoa butter. The other formats 1-4 melt more easily and indeed the melting point of Type 1 is 17C. Type 5 melts at 34C and Type 6 at 36C.

Chocolate powder compositions can be made up in water or milk for chocolate beverages or chocolate cake mixes.

Cocoa liquor powder and chocolate powders are convenient for use in industry as they do not need further processing for their addition and mixing into other food mixtures; for example into milk powder or liquid milk, for the manufacture of chocolate flavoured ice cream, for chocolate flour mixes for the manufacture of cakes or biscuits, chocolate sugar confectionery and/or for chocolate syrups. Free flowing powders free of clumped matter is much easier to handle.

This invention also covers ideal storage conditions of the cocoa butter powder and cocoa butter compositions so that they retain their free flowing properties and are more resilient to clumping.

For the purpose of this document the definitions of cocoa butter, cocoa liquor and chocolate are those recognised by the CODEX standards and the European Union regulations. It also includes milk and white chocolate compositions with 15% cocoa butter or more total fat.

### **Description of the prior art**

It is simply not possible to mill large quantities of kibbled solid cocoa butter pieces or pieces of cocoa butter containing compositions such as chocolate or cocoa liquor of more than about 15 % fat without the friction of the moving parts in the mill heating up and melting some of the surface cocoa butter. Using cooled air will limit some of the melting but invariably the mill will block up with melted chocolate building up on the metal surfaces in the mill. For this reason conventional milling cannot be used for the industrial production of cocoa butter powders, cocoa liquor powders and chocolate compositions.

A technology that has been described in patent application literature has been to heat solid cocoa butter, solid cocoa liquor or a solid chocolate composition to form a pump able liquid mass and spray it thorough nozzle into a cryogenic chamber. Whilst the liquid mass is clearly de-tempered it is my understanding that the thermal cryogenic shock of the droplets in the cryogenic chamber puts the cocoa butter into the Type 5 crystalline format. There are a number of these industrial installations that make use of this technology.

A limitation of the spray crystalline technology is that lower fat compositions, for example chocolate compositions of less than about 25% total fat are very viscous and difficult or impossible to pump up from the holding tank to and through the nozzle head.

Another technology is cryogenic milling which as a technology has been available for years to stop solid materials at room temperature from melting during the conventional milling process. It has not been reported to date as being used for cocoa butter, cocoa liquor or full fat chocolate compositions above 18% total fat.

For cocoa butter and cocoa butter compositions no attention has been given to the necessity of using tempered material in the Type 5 crystalline format and the necessity to maintain a predominant amount of this crystalline format through out the milling process in order to create a more stable powder with good free flowing properties.

The production of reduced fat cocoa butter compositions has been reported by mixing full fat chocolate powder with other chocolate making ingredients such as sugar or reduced fat cocoa ( WO 2006 1 20380 ), but as the resulting total fat content has been much lower with consequently less free surface fat there has not been a necessity to keep the cocoa butter predominantly in the Type 5 format.

This is also the case for reduced fat milled cocoa and chocolate compositions which have had fat partially removed with an expeller or hydraulic press. There is no need to have tempered cocoa butter as the fat content is too low to cause clumping of the particles especially with compositions of less than 18% total fat.

Cocoa butter containing powders, which of course have a much higher surface area than the equivalent weight of solid pieces or chips, which are higher for example above about 18% total fat are particularly susceptible to fat bloom if the process and storage temperatures are not ideal, and also to sugar bloom if the moisture levels are too high. It is well known that storage of tempered chocolate above 20C will start to de-temper and cause fat blooming. Exposure of these chocolate powders to excessive moisture will provoke sugar bloom causing re crystallisation of the sugar in the chocolate. Excessive pressure caused by large unit quantities of powder or sacks stacked upon one another will cause compacting and should be avoided. Light and air is also known to cause oxidation of the fat and modify the organoleptic quality of the product. Non fat cocoa containing compositions for example cocoa liquor and plain chocolate will be more resistant to oxidation they contain high levels of cocoa polyphenols.

### **Detailed description of the invention.**

Example 1:

1 kg of tempered plain chocolate pieces of 2.5 cm diameter and 1 cm thick are pre-cooled to minus 30C with liquid nitrogen and then pulverised into a powder in a kitchen blender fitted with blades to form a powder. The chocolate in the blender was controlled so that it never attained a temperature above 5C throughout the whole pulverisation process. A similar operation was performed with tempered milk chocolate and tempered white chocolate chunks of similar dimension. All these powders when allowed to settle at room temperature ( of less than 17C ) in an atmosphere of less than 70 relative humidity were free flowing powders void of clumps.

**Example 2:**

As per example 1 although the substrate material was tempered cocoa liquor drops of 1 cm diameter that that were formed by depositing on a flat belt, passed though a traditional chocolate cooling tunnel and stored at 15C for 2 weeks prior to pre-cooling the cocoa liquor drops with liquid nitrogen.

**Example 3:**

As per example 1 although the substrate material was tempered cocoa butter drops of 1 cm diameter that were formed by depositing on a flat belt, passed through a traditional chocolate cooling tunnel and stored at 15C for 1 week. The drops were pre-cooled with liquid nitrogen.

**Example 4:**

As per examples 1-3 although 1 kg of cocoa butter , cocoa liquor and/or chocolate compositions were melted to a temperature of 45 C, stirred gently and then covered with a cryogenic liquid for example liquid nitrogen so that the substrate material is subject to a tremendous thermal shock and virtually instantly solidifies. The solid materials are then mechanically broken into small pieces and pulverised in a kitchen blender to form a powder. The substrate material whilst broken up into pieces or during blending was controlled so that it never reached a temperature above 5C ( bearing in mind that some heat can be generated by the friction of the blender whilst in operation for prolonged periods). This powder was allowed to settle to 15C ( room temperature at the time) with a maximum 70% relative humidity. The powder was free flowing and void of clumps.

**Example 5:**

Tempered plain chocolate drops were pre-cooled with liquid nitrogen to minus 30 C. The feeding of the drops was controlled by use of a funnel and rpm-controlled airlock. Grinding of the of the cooled chocolate drops was performed within a Hosokawa Classifier mill type ACM 10, with the grinding disk at 5800 rpm and classifier at 2000 rpm. During grinding liquid nitrogen was injected into the air feed of the mill. Both a cyclone and filter were used to separate the gas from the powder product. The feed capacity was 50 kg/h. This was also performed with tempered milk chocolate drops , tempered white chocolate drops .tempered cocoa liquor drops and cocoa butter drops. The powder was allowed to settle to 15C with a controlled humidity environment of maximum 70% relative humidity. The powder was free flowing and void of lumps.

**Example 6:**

Tempered plain chocolate drops were introduced into the mill as in example 1 and without cryogenic treatment. The temperature of the chocolate was 15C. The process conditions of the classifier mill were also identical as in example 1 and during grinding liquid nitrogen was injected into the air feed of the mill. The power was allowed to settle as described in example 5. This was repeated for tempered milk chocolate drops, tempered white chocolate drops, tempered cocoa liquor drops and tempered cocoa butter drops. The powder were free flowing and void of clumps.

**Example 7:**

40 kg of plain chocolate was fed into the mill with 20 kg dry ice ( carbon dioxide pellets at minus 78C) with a combined mix temperature of about minus 65C. The feeding of the mix was controlled by funnel and a rpm air lock. Grinding was performed with a Hosokawa classifier mill type ACMIO with the grinding disc at 6500 rpm and the classifier set at 300 rpm. During grinding nitrogen was injected into the air feed of the mill. The cyclone and filter were used to separate the gas from the powder product. The feed capacity was 25 kg/h. The powder was free flowing and void of clumps.

**Example 8:** Chocolate powders, cocoa liquor powder or cocoa butter powder as made in the above examples was stored in 10 kg sealed polythene lined bags in a rigid cardboard box, at 12-20C for prolonged periods of over 18 months. The powders remained free flowing and void of clumps. The packaging had added food grade humidifying sachets to ensure the powders did not pick up extra moisture.

## Claims

1. The process of cryogenic milling of solid pieces of tempered cocoa butter to form a tempered, free flowing powder and relatively free of clumped particles. The process makes use of a cryogenic material such as liquid nitrogen or dry ice.
2. The process of cryogenic milling of solid pieces of tempered cocoa liquor to form a tempered, free flowing powder and relatively free of clumped particles. The process makes use of a cryogenic material such as liquid nitrogen or dry ice.
3. The process of cryogenic milling of solid pieces of tempered cocoa butter containing chocolate compositions to form a tempered free flowing powder and relatively free of clumped particles. The process makes use of a cryogenic material such as liquid nitrogen or dry ice.
4. As per claim 1-3 above, with the additional process step of further maintaining the milled particles in a controlled environment preferably at less than 70% relative humidity to enable the cold powder to settle at the room temperature of less than 17 C for prior to packing preferably in rigid containers to minimise that the powders are compacted due to excessive weight.
5. As in claim 1-4 except that the starting solid substrate materials containing cocoa butter are non tempered. These materials are re melted to above 36 C, but preferably above 45C to completely melt all the fat crystals, and frozen with an intense thermal shock with a cryogenic material ( i.e. liquid or chips) The cold solid material is then broken up, kibbled then cryogenically milled.
6. Chocolate that has suffered heat damaged, and shows sign of fat blooming is re-melted and processed as in claim 5.
7. As in claim 1-6 to include cocoa butter compositions which contain minimum of 15 % of cocoa butter. Non cocoa butter components can include sugar(s), polypols, starch, fibre, protein, peptides, amino acids and/or flavouring(s) either alone or in combination of one or more of these components.
8. The cryogenic material in claims 1-7 can be liquid nitrogen, liquid carbon dioxide and combinations of any thereof. Other cryogenic materials can include dry ice (carbon dioxide chips) and liquid argon.

9. As per claim 2, 4 & 5 the cryogenic milling of kibbled solid pieces or chips of reduced fat cocoa liquor of more than 24 % total fat to produce a tempered, free flowing powder.

10. As per previous claims and in which the substrate materials are first pre-cooled with a cryogenic material, kibbled and milled in a conventional mill. The substrate is kept under 15C at all times during the kibbling and milling process or pulverisation process. Cold de-humidified air is optionally passed through the mill.

11. Tempered cocoa butter powder and cocoa butter containing powder compositions manufactured with cryogenic milling processes as described in previous claims can be used as a "seed" to temper liquid chocolate.

12. Cocoa butter and cocoa butter containing compositions can be tempered by pre-heating to above 36C and preferably above 45C and then exposed to thermal shock with a cryogenic material such as liquid nitrogen or dry ice.

13. As per previous claims 1-5, 9 & 10 the free flowing properties of cryogenic milled powders containing cocoa butter are maintained better when they are stored in a dry place away from light and air, at an ideal temperature of between 12C to 20 C and at a maximum relative humidity of 70C.

14. As per previous claims 1-5, 9 & 10 the free flowing properties of cryogenic milled powders are best protected by physical compacting ( by pressure) by storing in rigid containers of about 10 kg maximum or ideally less. Large unit quantities of powder will tend to compact under their own weight.

18. As in claims 1-7 , 9 & 10 a classifier mill is employed to manufacture the powder although other types of equipment that pulverise the substrate material can also be employed. A cryogenic material is always used in the process, either to pre-cool the substrate material and/or during the milling process itself.

19. A product produced as in claims 1-7, 9 & 10

20. The product of claims 1-7, 9 & 10 having a fineness whereby 99% of the particles are of less than 75 micrometers.

21. The product of claims 1-7 ,9 & 10 having a mean particle size of less than 40 micrometers in diameter

22. The non cocoa solids, sugar and other non fat materials in the cocoa liquor, chocolate combinations and other combinations listed in claims 2-7.9 & 10 can be pre-refined to 25 micrometers in a 3 and/ or 5 cylinder roller or a combination of both.

**INTERNATIONAL SEARCH REPORT**

International application No  
PCT/GB201Q/002144

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> INV. A23G1/Q0 A23P1/00 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) A23G A23P		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal , FSTA, BIOSIS, MEDLINE, WPI Data		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	wo 96/19923 AI (CADBURY SCHWEPES PLC [GB] ; ZUMBE ALBERT [GB] ; SANDERS NIGEL [GB] ) 4 July 1996 (1996-07-04) the whole document -----	1-22
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<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C.		<input checked="" type="checkbox"/> See patent family annex.
* Special categories of cited documents :		
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed		"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family
Date of the actual completion of the international search 18 February 2011		Date of mailing of the international search report 28/02/2011
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Vernier, Frederic

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>HACHIYA I ET AL: "SEEDING EFFECTS ON SOLIDIFICATION BEHAVIOR OF COCOA BUTTER AND DARK CHOCOLATE. I. KINETICS OF SOLIDIFICATION", JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY, SPRINGER, BERLIN, DE, vol . 66, no. 12, 1 December 1989 (1989 - 12-01 ), pages 1757 - 1762 , XP0020615 19, ISSN: 0003 -02 1X, DOI: DOI:10.1007/B F02660743 the whole document</p> <p align="center">-----</p>	1-22
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Information on patent family members

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