

COCOA ALKALIZATION PROCESSES

CROSS-REFERENCE TO RELATED APPLICATION

- 5 [0001] The present application claims the benefit of U.S. Provisional Patent Application No. 63/625,053, filed January 25, 2024, which is hereby incorporated by reference in its entirety.

TECHNICAL FIELD

- 10 [0002] In some aspects, this disclosure relates to processes for preparing an alkalized cocoa material. In some examples, the process includes mixing cocoa and a first alkalization agent together at an initial temperature and adding heat until a first elevated mixing temperature is reached, then adding a second alkalization agent, and mixing and heating
15 the cocoa until reaching a second elevated mixing temperature. Water be added with one or both of the alkalizing agents, or in some examples, alkalizing agents are added and water is later added at a first elevated temperature. The process may include mixing these components together at the second elevated mixing temperature for at least one hold period to provide a mixture, where conditions may change between any hold
20 periods when there are multiple such periods (*e.g.* a lower temperature and/or pressure is reached between period), and the process may include then drying the mixture after any hold period(s) to provide an alkalized cocoa material.

BACKGROUND

- 25 [0003] Cocoa material may be alkalized, resulting in a change in appearance or flavor. However, traditional methods for producing dark cocoa powders often require extended reaction times or considerable quantities of alkalizing agents to achieve such dark colors. The use of these alkalizing agents in the process needs to be reduced in view of
30 health and safety regulations becoming more stringent as well as updated food

regulations. New processes of alkalization may result in beneficial product characteristics. Thus, new processes for preparing alkalized cocoa material may be beneficial which make use of reduced amount of alkalizing agent. There is a need in the art for a more efficient and advanced cocoa material (or cake) alkalization process aimed at producing highly alkalized, dark cocoa powders, while at the same time with the aim of preserving the inherent properties and flavor of cocoa powder while considerably shortening the processing time and wherein the process has a greener footprint.

10 SUMMARY

[0004] It is an object of the present invention, amongst other objects, to address the above need in the art. The object of present invention, amongst other objects, is met by the present invention as outlined in the appended claims. Specifically, the above object, amongst other objects, is met, according to a first aspect, by the present invention a process for production of an alkalized cocoa material comprising the steps of mixing and heating in a reactor a cocoa raw material, a first alkalization agent, a second alkalization agent at an elevated mixing temperature for a first hold period to provide a first mixture; aerating of the first mixture, and; optionally, subsequently heating the first mixture to the elevated mixing temperature and mixing for at least one additional hold period providing a further mixture, wherein the further mixture is aerated between any one of the at least one additional hold period(s); drying the first mixture or further mixture to provide the alkalized cocoa material; wherein the elevated mixing temperature is between 100 to 160 °C, preferably 120 to 150 °C, more preferably 125 to 135 °C, wherein any one of the hold periods are 20 to 150 minutes, preferably 40 to 110 minutes, more preferably 50 to 90 minutes in length, wherein water is added to the cocoa raw material, the first alkalization agent and the second alkalization agent prior to the first hold period, wherein an elevated pressure is present during any one of the hold periods, preferably wherein the elevated pressure is between 1 to 8 bara, preferably 2 to 7 bara, more preferably 3 to 6 bara, most preferably 4 to 5 bara.

[0005] In some aspects, this disclosure relates to processes of, *inter alia*, preparing an alkalized cocoa material. In some examples, the disclosure relates to the process including the step of mixing cocoa and a first alkalization agent together at an initial temperature, such as ambient temperature or typical room temperature. The process may then include mixing and heating the cocoa and the first alkalization agent until reaching a first elevated mixing temperature (*e.g.* about 100-150 degrees Celsius). The process may include adding a second alkalization agent to the cocoa and the first alkalization agent. Then, the process may include mixing and heating the cocoa, the first alkalization agent, and the second alkalization agent until reaching a second elevated mixing temperature (*e.g.* about 100-160 degrees Celsius). In some examples the process may include mixing the cocoa, the first alkalization agent, and the second alkalization agent at the second elevated mixing temperature for at least one hold period to provide a mixture (*e.g.* one hold period, or between about one and ten or between about one and five hold periods). In some examples, the process may include aerating the mixture and drying the mixture to provide an alkalized cocoa material. The process of present invention preserves the inherent properties and flavor of cocoa powder while considerably shortening the processing time and reducing the total amount of alkalization agent being used. The cyclical nature of this process enhances air replenishment, resulting in improved oxidation and accelerated reactions, ultimately streamlining production while maintaining or even improving the cocoa powder product characteristics. While the cycle process of enhances reaction speed, it alone cannot does not attain the desired dark colors and corresponding flavor without an (increasing amount of) alkalizing agent; *i.e.* the more alkalizing agent is added, the darker the color will be. To address the need to reduce the alkalizing agent while obtaining the desired dark color, a production process is developed by including an air replacement step. The aerating step optimizes air exchange between the cocoa material and allows for oxidation and accelerated reaction to occur and to achieve the same rich color with a reduced amount of alkalizing agent (less than 7% in view of the prior art processes). This advancement aligns with the growing demand for clean-label products in both the European and American markets.

5 [0006] According to a preferred embodiment, the present invention relates to the process, wherein the total amount of alkalization agent in the first mixture is between 6 to 18 wt%, preferably between 7 to 16 wt%, more preferably between 8 to 15 wt%, most preferably 10 to 14 wt%, based on the total weight of the cocoa raw material.

10 [0007] According to another preferred embodiment, the present invention relates to the process, wherein the at least one additional hold period comprises between one to five additional hold periods, preferably two to four additional hold periods.

[0008] According to yet another preferred embodiment, the present invention relates to the process, wherein the first hold period is 75 to 90 minutes in length and the at least one additional hold period is shorter than the first hold period.

15 [0009] According to a preferred embodiment, the present invention relates to the process, wherein aeration results in cooling of the first mixture or further mixture to a temperature below the elevated mixing temperature, preferably to a temperature of 1 to 50 °C, preferably 5 to 25 °C, more preferably 10 to 15 °C below the elevated mixing temperature.

20 [0010] According to yet another preferred embodiment, the present invention relates to the process, wherein the aeration is done at an air flow rate of about 1.61 to 16.05 m³/hr, preferably 3.21 to 12.84 m³/hr, more preferably 4.82 to 9.63 m³/hr, even more preferably 6.42 to 8.03 m³/hr.

25 [0011] According to another preferred embodiment, the present invention relates to the process, wherein the cocoa raw material is one or more selected from the group consisting of cocoa beans, cocoa nibs, cocoa butter, cocoa cake, cocoa powder, cocoa liquor, and any source that comprises cocoa solids, preferably cocoa powder, cocoa
30 liquor or cocoa cake.

- 5 [0012] According to a preferred embodiment, the present invention relates to the process,
wherein total length of the hold periods in the process is between 20 to 400 minutes,
preferably 60 to 380 minutes, more preferably 120 to 350 minutes, most preferably
150 to 320 minutes.
- 10 [0013] According to another preferred embodiment, the present invention relates to the
process, wherein the moisture content of the cocoa raw material is between 15 to 60
wt%, preferably 20 to 50 wt%, more preferably 25 to 40 wt%, based on the total
weight of the cocoa raw material.
- 15 [0014] According to yet another preferred embodiment, the present invention relates to the
process, wherein the cocoa raw material has a fat content of between 6 to 55 wt%, 8 to
24 wt%, preferably between 10 to 20 wt%, more preferably between 12 to 18 wt%,
based on the total weight of the cocoa raw material.
- 20 [0015] According to a preferred embodiment, the present invention relates to the process,
wherein the water is added to the cocoa raw material, the first alkalization agent and
the second alkalization agent when reaching a temperature during heating of about 80
to 105 °C, preferably 85 to 95, more preferably 87 to 90 °C.
- 25 [0016] According to another preferred embodiment, the present invention relates to the
process, wherein the first alkalization agent and the second alkalization agent each
comprise or consist of one or more hydroxide materials, one or more bicarbonate
materials, one or more carbonate materials, or combinations thereof.
- [0017] According to a preferred embodiment, the present invention relates to the process,
wherein the first alkalization agent and the second alkalization agent are the same or
are different, preferably different.

- 5 [0018] According to another preferred embodiment, the present invention relates to the process, wherein the first alkalization agent and the second alkalization agent each comprise or consist of sodium hydroxide or ammonium carbonate or ammonium bicarbonate or potassium carbonate.
- 10 [0019] According to a preferred embodiment, the present invention relates to the process, wherein the first alkalization agent and the second alkalization agent each comprise or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide, magnesium oxide, sodium carbonate, potassium carbonate, ammonium carbonate or ammonium bicarbonate, calcium carbonate, or combinations thereof.
- 15 [0020] According to yet another preferred embodiment, the present invention relates to the process, wherein any mixing comprises agitating the materials being mixed, chopping the materials being mixed, or both.
- 20 [0021] According to another preferred embodiment, the present invention relates to the process, wherein any agitation is at a speed of 50-170 revolutions per minute (rpm), preferably 65 to 150 rpm, more preferably 70 to 100 rpm, and/or wherein any chopping is at a speed of 1000-3500 rpm, preferably 1400 to 2500 rpm, more preferably 1700 to 2000 rpm.
- 25 [0022] According to yet another preferred embodiment, the present invention relates to the process, wherein the elevated pressure is generated by the addition of heated air, steam, or a combination thereof.
- [0023] According to yet another preferred embodiment, the present invention relates to the process, wherein the mixture is dried by air drying, hot air drying, vacuum drying, or combinations thereof.

[0024] According to yet another preferred embodiment, the present invention relates to the process, wherein aeration is done by adding compressed air to the reactor.

- 5 [0025] According to yet another preferred embodiment, the present invention relates to the process, wherein any aeration is performed at an ambient pressure, more preferably at an atmospheric pressure of about 1 bar.

- 10 [0026] According to yet another preferred embodiment, the present invention relates to the process, wherein the aeration of the first mixture is performed at an elevated pressure of about 2 to 10 bara, preferably 3 to 8 bara, more preferably 4 to 6 bara.

- 15 [0027] The present invention, according to a further aspect, relates to an alkalized cocoa material obtained via or obtainable by the process of the present invention.

- [0028] According to a preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has a fat content of preferably 6 to 30 wt%, more preferably 7 to 25 wt%, even more preferably 8 to 18 wt%, most preferably 10 to 15 wt%, based on the total weight of the cocoa material.

- 20 [0029] According to another preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has a Na content of preferably 9000 to 24000 mg/kg of the cocoa material, more preferably 10000 to 20000 mg/kg, even more preferably 12500 to 18000 mg/kg, most preferably 15000 to 25 17500 mg/kg.

- [0030] According to yet another preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has a K content of

preferably 19000 to 24000 mg/kg of the cocoa material, more preferably 20000 to 23000 mg/kg, even more preferably 21000 to 22000 mg/kg.

5 [0031] According to a preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has an ash content of preferably 5 to 18 wt%, more preferably 7 to 16 wt%, even more preferably 8 to 14 wt%, most preferably 10 to 12 wt%, based on the total weight of the cocoa material.

10 [0032] According to yet another preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has an acrylamide content of at most 300 µg/kg of the cocoa material, preferably at most 200 µg/kg, more preferably at most 150 µg/kg, even more preferably at most 90 µg/kg, most preferably at most 30 µg/kg.

15 [0033] According to another preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has a dark color as based on a CIELAB value, wherein L is between 16 to 30, preferably 17 to 25, more preferably 18 to 22, C is between 5 to 9, preferably 5.5 to 8, more preferably between 6 to 7.5, and h is between 40 to 52, preferably between 42 to 50, more preferably
20 between 44 to 48, as determined by spectrophotometer.

[0034] According to yet another preferred embodiment, the present invention relates to the alkalized cocoa material, wherein the alkalized cocoa material has a pH of between 6.0 to 8.5, preferably between 6.2 to 8.2, more preferably 6.5 to 7.8, most preferably 6.8 to
25 7.5.

[0035] The present invention, according to a further aspect, relates to an edible composition comprising the alkalized cocoa material of present invention.

[0036] In some examples of the process, the first elevated mixing temperature may be about 100-175 degrees Celsius. In certain examples of the process, the second elevated mixing temperature may be about 100-200 degrees Celsius. In various examples of the process, the first alkalization agent and the second alkalization agent each include or consist of one or more hydroxide materials, one or more bicarbonate materials, one or more carbonate materials, or combinations thereof. In some examples, the first alkalization agent and the second alkalization agent each include or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide, sodium carbonate, potassium carbonate, ammonium carbonate or ammonium bicarbonate, calcium carbonate, or combinations thereof. In certain examples, the first alkalization agent and the second alkalization agent may be different materials.

[0037] In some examples, the mixing may include agitating the materials being mixed, chopping the materials being mixed, or both inside a reactor or chamber. Any agitation (with one or more mechanical operators, *e.g.* one or more plows and/or one or more scrapers and/or one or more stirrers) may be performed at a speed of about 50-170 revolutions per minute, *e.g.* about 100-125 rpm. Any chopping (with one or more mechanical operators, *e.g.* one or more blades) may be at a speed of about 750-5000 revolutions per minute (rpm), *e.g.* about 1000-3500, or about 3000-3500 rpm. A suitable reactor or chamber that may be used in the process of present invention may be a 130 L horizontal reactor including a plowshare mixer. The chopper uses a 7.5 kW motor with a standard speed of 3480 rpm. However, it is to be understood that while 3500 rpm for choppers is generally used due to motor limitations, the rpm for plow mixers depends on the size of the reactor. For example, with a larger reactor (for example a 300 L horizontal reactor) a lower rpm may be used to achieve a similar tangential velocity compared to smaller reactors at higher rpm values.

[0038] In some examples of the process, the at least one hold period may total a single hold period, while in some examples the at least one hold period may total two or more hold periods (*e.g.* two hold periods, three hold periods, four hold periods, five hold periods,

six hold periods, seven hold periods, or eight hold periods). In some examples, the cocoa, the first alkalization agent, and the second alkalization agent may be cooled between any hold periods.

5 **[0039]** In certain examples, the cocoa, the first alkalization agent, and the second alkalization agent may be subjected to a pressure during the at least one hold period. If there are multiple hold periods, at least one hold period may be at ambient pressure and at least one additional hold period may be at a non-ambient pressure, while in some examples all hold periods are at a relatively high pressure and any intermediate processing is at ambient pressure or a lower pressure than during the hold period. In some examples the cocoa, the first alkalization agent, and the second alkalization agent are subjected to a pressure of about 1-10 bara during the at least one hold period, or during at least one of the hold periods. In some examples, the pressure may be generated by the addition of heated air, steam, or a combination thereof.

15 **[0040]** In some examples, the obtained, alkalized mixture may be dried, for example by air drying, hot air drying, vacuum drying, or combinations thereof. In some examples of the process, the process may include adding water to at least the cocoa, and then mixing the water and at least the cocoa. In some examples of the process, the process may include adding compressed air to the cocoa, the first alkalization agent, and the second alkalization agent after at least one hold period to lower the temperature of the mixture.

25 **[0041]** In some examples, the cocoa raw material may include a moisture content of about 1-20%, by weight. In certain examples, the cocoa may include or entirely consist of cocoa cake. In certain examples, the cocoa may have a moisture content of 1-20%, by weight, before alkalization.

30 **[0042]** In certain examples, processes for forming dark cocoa or cocoa powder are provided. These processes may include any of the steps, conditions, or equipment described above and/or in processes for preparing other cocoa products (and vice versa). In certain examples, a process for forming dark cocoa includes the steps of mixing cocoa cake and

a first alkalization agent together at an initial temperature. The process may include mixing and heating the cocoa cake and the first alkalization agent until reaching a first elevated mixing temperature. The process may include adding a second alkalization agent to the cocoa cake and the first alkalization agent. The process may include mixing and heating the cocoa cake, the first alkalization agent, and the second alkalization agent until reaching a second elevated mixing temperature.

[0043] In some examples, the process may include mixing the cocoa, the first alkalization agent, and the second alkalization agent at the second elevated mixing temperature for at an initial hold period to provide a mixture, and then aerating (ambiently or under pressure) and optionally cooling the mixture. In certain examples, the process may include heating the mixture to a third elevated mixing temperature (which can be the same, or different, from the second elevated temperature) and mixing for at least one additional hold period. In some examples, the process may include drying the mixture to provide an alkalized dark cocoa material, such as an alkalized dark cocoa material.

[0044] In certain examples, all of the elevated mixing temperatures may be about 100-160 degrees Celsius. In some examples, any hold periods (initial or additional) may be about 20-150 minutes in length. In certain examples, the mixture may be cooled between any of the at least one additional hold periods (*e.g.* back to about 80 to 100 degrees Celsius, or about ambient temperature). In certain examples, water is added to at least the cocoa cake and mixed with at least the cocoa cake prior to the initial hold period (whether to the cake, or to the cake and at least some other material like one or two alkalizing agents).

[0045] In certain examples of the process, the first alkalization agent and the second alkalization agent each include or consist of one or more hydroxide materials, one or more bicarbonate materials, one or more carbonate materials, or combinations thereof. In some examples, the first alkalization agent and the second alkalization agent each include or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide,

sodium carbonate, potassium carbonate, ammonium carbonate or ammonium bicarbonate, calcium carbonate, or combinations thereof. In some examples, the first alkalization agent and the second alkalization agent are different materials. In various embodiments, the first alkalization agent and the second alkalization agent each include or consist of sodium hydroxide or ammonium carbonate or ammonium bicarbonate (*e.g.* one is sodium hydroxide and the other is ammonium carbonate or ammonium bicarbonate).

5 [0046] In certain examples, any mixing includes agitating the materials being mixed, chopping the materials being mixed, or both. In some examples, any agitation is at a speed of about 50-170 revolutions per minute, and wherein any chopping is at a speed of about 1000-3500 revolutions per minute. The use of choppers significantly improved mixing efficiency, resulting in smaller particle sizes. Choppers also drastically reduced lumping.

15 [0047] In some examples of the process, the at least one additional hold period includes at least two additional hold periods, and the mixture is aerated (and may be also cooled linked or due to the aeration) between the additional hold periods. In certain examples, the at least one additional hold period includes at least three additional hold periods, and the mixture is aerated between the additional hold periods.

25 [0048] In some examples of the process, the cocoa, the first alkalization agent, and the second alkalization agent are subjected to an elevated pressure during the initial hold period and the at least one additional hold period (*e.g.* about 2-8 bara, or about 4-6 bara, or about 4-8 bara). In some examples, the cocoa, the first alkalization agent, and the second alkalization agent are subjected to an elevated pressure of up to about 8 bara during the initial hold period and the at least one additional hold period. In certain examples, the pressure is generated by the addition of heated air, steam, or a combination thereof. In some examples, the mixture is dried by air drying, hot air drying, vacuum drying, or combinations thereof.

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- [0049] In some examples of the process, the process further includes adding compressed air to the cocoa, the first alkalization agent, and the second alkalization agent after the at least one additional hold period to lower the temperature of the mixture. In certain examples, the cocoa raw material includes a moisture content of about 1-20%, by weight. In some examples of the process, any aeration is performed at an ambient pressure. In certain examples of the process, any aeration is performed at an elevated pressure, preferably the first aeration of the first mixture is performed at an elevated pressure.
- [0050] In some examples, a process for forming an alkalized cocoa material, such as dark cocoa, is provided including the step of mixing and heating cocoa cake, a first alkalization agent, a second alkalization agent and water at a first elevated mixing temperature for at an initial hold period to provide a mixture, and then aerating and cooling the mixture. In certain examples, the process then includes heating the mixture to a second elevated mixing temperature and mixing for at least one additional hold period, and then drying the mixture to provide an alkalized dark cocoa material. In some examples, all of the elevated mixing temperatures are about 100-150 degrees Celsius. In some examples, any hold periods are about 30-150 minutes in length, and the mixture is aerated and/or cooled between any of the at least one additional hold periods. In certain examples, at least some aeration, or any aeration, is performed at an elevated pressure.
- [0051] In another aspect of the disclosure, edible compositions such as cookies or other desserts are provided. Example edible compositions include cookies, cakes, breads, beverages, ice cream, pies, or truffles. In some examples, an edible composition includes an alkalized cocoa material made from a process of this disclosure. In other examples, an edible composition includes two or more alkalized cocoa materials, each made from distinct processes of this disclosure.

[0052] These and other features, advantages, and objects of the present disclosure will be further understood and appreciated by those skilled in the art by reference to the following specification and claims.

5 BRIEF DESCRIPTION OF THE DRAWINGS

[0053] These and other features, aspects, and advantages of the present disclosure will become better understood with regard to the following description, claims, and drawings. The present disclosure is illustrated by way of example, and not limited by, the accompanying figures. A more complete understanding of the present disclosure and the advantages thereof may be acquired by referring to the following description in consideration of the accompanying drawings, which show example process steps and/or information, including:

[0054] Figure 1 shows a graph illustrating example temperature and time variations across an example process using multiple hold periods.

[0055] Figure 2 shows a process flow chart with example steps for an example implementation of the process.

DETAILED DESCRIPTION

[0055] The examples, materials and methods of described herein provide, *inter alia*, processes and systems for preparing alkalized cocoa materials. These and other aspects, features and advantages of the disclosure or of certain embodiments of the disclosure will be further understood by those skilled in the art from the following description of example embodiments. In the following description of various examples, reference is made to the accompanying drawings, which form a part hereof. It is to be understood that other modifications may be made from the specifically described methods and systems without departing from the scope of the present disclosure.

- [0056] In some aspects, this disclosure relates to processes of, *inter alia*, preparing an alkalized cocoa material. In certain examples, the process relates to preparing an alkalized dark cocoa material, such as dark, alkalized cocoa cake. The cocoa material to be alkalized may be cocoa beans, cocoa nibs, cocoa butter, cocoa cake, cocoa powder, cocoa liquor, or any other cocoa material, *e.g.* a combination of any cocoa source(s) that contain cocoa solids. The cocoa material may or may not have already been reacted with one or more alkalizing agents before undergoing additional alkalization process(es) as described herein.
- [0057] The Figures provide illustrations of concepts described in more detail below. For example, Figure 1 shows an example temperature profile 100 of an alkalization process over time (scaling is exemplary), where in an initial plateau components may be combined (*e.g.* cocoa material and alkalizing agent) and then during mixing the temperature starts to increase, where another alkalizing agent may be added at this time, or in the initial plateau. The materials are then mixed and held at an elevated temperature for a hold period, before conditions are relaxed (*e.g.* the reactor is opened, materials are cooled, air is replenished, and the like) before another period of being held under elevated reaction period occurs. In this example, there are three total hold periods at an elevated temperature (all equal here, but this is not always the case) with transition periods of relatively lower temperatures, followed by a final cooldown period. Figure 2 provides a process flow diagram of an example process 200 with example steps 202-214, including optional steps, *e.g.* step 208b. Various implementations of these steps are described in more detail below. Reactors suitable for performing example processes may be configured to provide a homogenous or essentially homogenous mixture of solids materials, capable of reducing particle sizes (*e.g.* reducing the particle size of cocoa cake), proper dispersion of solids when water is added, and provision of uninformed or essentially uniform heating without “cold spots” and/or material accumulation in a particular location of the reactor. For example, a plow mixers with a jacket capable of heating/cooling the drum may be used.

[0058] In some examples, the disclosure relates to the process including the step of mixing cocoa and a first alkalization agent together at an initial temperature, *e.g.* an ambient temperature or an elevated temperature above an ambient temperature (temperatures, unless specified for equipment, such as a reactor jacket, are for the materials being treated, *e.g.* the cocoa material). The process may then include mixing and heating the cocoa (*e.g.* by adding steam to the jacket of a reactor chamber) and the first alkalization agent until reaching a first elevated mixing temperature.

[0059] The elevated temperature or first elevated temperature may be, for example, about 100-160 degrees Celsius, about 125-150 degrees Celsius, about 130-145 degrees Celsius, about 135-140 degrees Celsius. The first elevated temperature may be, for example, about 160 degrees Celsius or less, or about 155 degrees Celsius or less, or about 150 degrees or less, or about 125 degrees Celsius or less. The first elevated temperature may be, for example, about 100 degrees Celsius or more, or about 125 degrees Celsius or more, or about 150 degrees Celsius or more, or about 155 degrees Celsius or more.

[0060] The process may include adding a second alkalization agent to the cocoa and the first alkalization agent (or, in some examples, both alkalization agents are added at an initial temperature and water is added at the first elevated temperature). Once the reactor is sealed and heating is underway, additional components like a second alkalization agent or water may be added through a port, or by otherwise reopening the reactor briefly. Then, the process may include mixing and heating the cocoa, the first alkalization agent, and the second alkalization agent until reaching a second of further elevated mixing temperature (*e.g.* about 100-160 degrees Celsius) where the materials may be held for a hold period to allow the reaction to proceed. The second elevated temperature may be equal, or less than the first elevated temperature (if still above ambient temperature, wherein ambient temperatures are generally considered to be within the range of 20-25°C). In certain examples of the process, the second elevated mixing temperature may be about 85-155 degrees Celsius, about 75-150 degrees Celsius, about 50-140 degrees

Celsius, about 100-150 degrees Celsius, about 75-120 degrees Celsius, about 90-110 degrees Celsius. For any elevated temperatures, heat/steam may be used to heat the reactor jacket to a higher temperature until the mixture reaches the desired temperature (e.g. a jacket temperature of 135 degrees is used until a desired mixture temperature of 110 degrees is reached). However, any type of heat transfer may be suitable for reaching the desired temperature.

[0061] In examples with additional hold periods, subsequent elevated temperatures, *e.g.* a third elevated temperature, may use any of these values/ranges. Subsequent elevated temperatures may be different from the second elevated temperature and/or each other, or the same as the second elevated temperature and/or each other.

[0062] In certain examples of the process, any alkalizing agents are added together to the initial cocoa material being alkalized, *e.g.* cocoa nibs or cocoa cake, for example before any temperature ramp-up. In some examples, any alkalizing agents are added together to the initial cocoa material being alkalized and then mixed until reaching a first elevated temperature, and then water is added at the first elevated temperature. In some embodiments, water and a first alkalizing agent are added to cocoa, and then mixing/temperature ramp up occur, and second alkalizing agent is added at a first elevated temperature. Any of the procedures, steps, and values described herein may be used for either method examples, *e.g.* regardless of whether the addition of water may be timed to occur initially or at a first elevated temperature, and/or whether any additional alkalizing agents (if used) may be added to a first alkalizing agent initially or at a later time such as the first elevated temperature, any described values for temperature, pressure, hold periods, etc. may be used.

[0063] In some examples the process may include mixing the cocoa, the first alkalization agent, and the second alkalization agent at the second elevated mixing temperature for at least one hold period to provide a mixture. In some examples, the at least one hold period

may total a single hold period, while in some examples the at least one hold period may total two or more hold periods (*e.g.* two hold periods, three hold periods, four hold periods, five hold periods, six hold periods, seven hold periods, or eight hold periods). In some examples, there may be only one hold period for mixing at temperature. In certain examples, there may be two hold periods or more, three hold periods or more, 5 four hold periods or more, five hold periods or more, six hold periods or more, or seven hold periods or more. In hold period(s), the temperature and/or pressure, and often both, may be elevated compared ambient conditions and held constant or relatively constant for an extended period of continued agitation to facilitate the cocoa alkalization reaction and processing. 10

[0064] The temperature and/or pressure may be lowered in between hold periods, *e.g.* through ambient heat loss or intentional efforts for example by opening a chamber containing the materials/depressurizing and/or adding air, such that in some examples, the cocoa, 15 the first alkalization agent, and the second alkalization agent may be cooled between any hold periods and/or after a hold period, for example through a cycling between the hold period temperature and a lower temperature than the second elevated temperature (*e.g.* a temperature at, above, or below the first elevated temperature, *e.g.* via heat loss to ambient air upon opening of the heated reactor and/or via blown air). In some 20 examples, there may be between about one and ten hold periods, between about five and ten hold periods, or between about one and five hold periods, or between about three and five hold periods, or between about six and eight hold periods. In some examples, there may be one hold period for mixing at temperature, or two, or three, or four, or five, or six, or seven hold periods.

25 [0065] In some examples of the process, any hold periods of the at least one hold period may be about 20 minutes or more in length, 25 minutes or more in length, about 30 minutes or more in length, or about 45 minutes or more in length, or about 60 minutes or more in length, or about 75 minutes or more in length, or about 90 minutes or more in length, 30 or about 120 minutes or more in length, or about 150 minutes or more in length. In some

examples, a total residence time at an elevated temperature (total length of the hold periods in the process, *e.g.* including a second of further elevated temperature) may be about 50 minutes or more, or about 150 minutes or more, or about 250 minutes or more, or about 300 minutes or more, or about 400 minutes. In some examples, a total residence time at the second elevated temperature may be about 400 or less, 250 minutes or less, or about 200 minutes or less, or about 150 minutes or less, or about 125 minutes or less, or about 100 minutes or less. In some examples, an initial hold period is used, then a transition period occurs (*e.g.* opening a reactor/venting, cooling the mixed materials through blown air) before subjecting the mixture to an additional hold period at an elevated temperature and/or pressure. This may replenish the air and oxygen in the reactor prior to additional processing in a subsequent hold period. In some examples, an initial hold period is used, then a transition period occurs (*e.g.* opening a reactor/venting, cooling the mixed materials through blown air, and providing air replenishment) before subjecting the mixture to an additional hold period at an elevated temperature and pressure, followed by an additional sequence or sequences of a transition period and another hold period. This multi-stage, repeated hold period process can advantageously enhance processing time and/or lower the amount of alkali material needed by increasing oxidation and accelerating the desired reaction rate. The number of transitions/air replenishments may be about 1-8 times, about 2-5 times, about 1-5 times, or about 3-8 times, or 1 or more times, or 2 or more times, or 3 or more times, or 4 or more times, or 5 or more times, followed by an appropriate amount of hold periods.

[0066] In some examples, the same total residence times may include any temperature ramp up periods (including but not limited to the first elevated temperature) while in others additional time will be taken by any temperature ramp-up(s) and/or the first elevated temperature and residence time is based on the hold period time only. The hold periods may have minor variation in materials temperature based on heat application timing, mixing, and/or reaction process and thus may be at or around a particular temperature or temperature range, *e.g.* within five degrees of a target elevated temperature.

[0067] In some examples, an initial hold period is longer than any additional, subsequent hold periods, *e.g.* an initial hold period is about 20 minutes more than any subsequent hold periods, about 30 minutes more than any subsequent hold periods, about 40 minutes more than any subsequent hold periods, about 50 minutes more than any subsequent hold periods, or about 60 minutes more than any additional, subsequent hold periods, or is longer than at least one subsequent hold period by these or other amounts. In some examples, the initial hold period is about 30 minutes, about 45 minutes, about 60 minutes, about 75 minutes, or about 90 minutes, or about 60-90 minutes, or about 30-90 minutes, or about 90 minutes or less, or about 60 minutes or less, or about 30 minutes or more, or about 60 minutes or more. Subsequent hold periods may have shorter intervals, such as about 10 minutes, or about 20 minutes, or about 30 minutes, or about 10-45 minutes, or about 20-60 minutes, or about 10-30 minutes.

[0068] As noted above, if there are multiple hold periods, the temperature and/or pressure may be lowered in between hold periods, *e.g.* the pressure may return to ambient pressure or a pressure closer to ambient pressure, for example by opening or venting a reactor. Thus, there may be an intermediate temperature between any hold periods that is lower than the second elevated temperature, which may be higher than the ambient temperature, and higher, equal to, or lower than the first elevated temperature. The intermediate temperature may be, for example, about 100-160 degrees Celsius, about 105-155 degrees Celsius, about 110-120 degrees Celsius.

[0069] In various examples of the process, the first alkalization agent and the second alkalization agent each include or consist of one or more hydroxide materials, one or more bicarbonate materials, one or more carbonate materials, or combinations thereof. In some examples, the first alkalization agent and the second alkalization agent each include or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide, sodium carbonate, potassium carbonate, ammonium carbonate or ammonium bicarbonate, calcium carbonate, or combinations thereof. In certain examples, the first alkalization agent and the second alkalization agent may be different materials, while in

others they are the same material, or include the same material. For example, one agent may be calcium carbonate and/or ammonium carbonate or ammonium bicarbonate, while another may be magnesium hydroxide and/or sodium hydroxide. In examples where one or more ammonium materials are used, the process may include scrubbing
5 and/or treatment equipment to mitigate or substantially prevent any potential emissions.

[0070] In some examples, the mixing may include agitating the materials being mixed, chopping the materials being mixed, or both. Any agitation (with one or more mechanical operators, *e.g.* one or more plows and/or one or more scrapers) may be
10 performed at a speed of about 50-170 revolutions per minute, or about 70-100 revolutions per minute, or about 75-125 revolutions per minute, or about 120-170 revolutions per minute. Any chopping (with one or more mechanical operators, *e.g.* one or more blades) may be at a speed of about 1000-4000 revolutions per minute, or about 1500-3500 revolutions per minute, or about 1200-1800 revolutions per minute, or about
15 1600-1900 revolutions per minute, or about 2700-3400 revolutions per minute, or about 1000-3500 revolutions per minute, or about 2000-3500 revolutions per minute. Speeds may be selected based on the size of the reactor to ensure sufficient tangential velocity that can provide appropriate mixing/particle size reduction.

[0071] The materials being mixed may be agitated and/or chopping, preferably inside the reactor during mixing via components in the reactor chamber. For example, the one or more agitator(s) and/or chopper(s) may be static while present in a rotating container or drum, to further facilitate mixing, or vice versa (*i.e.* the drum is static and one or more agitator(s) and/or chopper(s) may be in motion). The agitation and/or chopping may
20 occur throughout the entire process, *e.g.* during temperature ramp up, hold period(s), and intermediate time between hold period(s), any aeration, and/or any drying.

[0072] In certain examples, the cocoa, the first alkalization agent, and the second alkalization agent may subjected to a pressure during the at least one hold period. If there are
30 multiple hold periods, at least one hold period may be at ambient pressure and at least

one additional hold period may be at a non-ambient pressure, *e.g.* an elevated pressure. In some examples the cocoa, the first alkalization agent, and the second alkalization agent are subjected to a pressure of about 1-10 bara (absolute, and in this disclosure all pressure values are absolute instead of gauge values unless otherwise specified, *e.g.* bar(a) rather than bar(g) values) during the at least one hold period, or during at least one of the hold periods, or about 1-5 bara, or about 2-4 bara, or about 3-6 bara, or about 3-7 bara, or about 4-7 bara, or about 4-8 bara, or about 6-7 bara, or about 6-8 bara, or about 5 bara or less, or about 10 bara or less, or about 7 bara or less, or about 3 bara or more, or about 2 bara or more, or about 5 bara or more. In some examples, the pressure of an initial hold period is between about 1-5 bara, or about 3-4 bara, or about 3-5 bara, while the pressure for any subsequent additional hold period(s) is higher, *e.g.* about 1-10 bara, about 2-8 bara, about 5-7 bara, or about 6-8 bara, or about 4-8 bara.

[0073] In some examples, adding heat/steam may be sufficient to reach a desired pressure by raising the temperature of the contents of the reactor, while in others, if the internal reactor pressure is lower than desired once the mixture has reached a desired temperature, the difference may be supplemented with air such as compressed air. In some examples, there may be an initial pressure value/range targeted, *e.g.* when all materials are added and first mixed, and then a different pressure value/range will be used during any subsequent hold periods, or may be set as a maximum pressure value (after which the system will vent, *e.g.* to drop about 0.5 bara in pressure) as the reaction may increase pressure as the materials react. Any of these values may be used for either set, *e.g.* an initial pressure of about 2-4 bara, or about 3-6 bara, or about 3-7 bara, or about 4-7 bara, or about 3 bara, or about 4 bara, or about 5 bara, or about 6 bara, is targeted in early mixing and then the hold period will either use the same pressure or a higher pressure than can occur through the reaction itself, *e.g.* about 3-6 bara, or about 3-7 bara, or about 4-7 bara, or about 5-8 bara, or about 4 bara, or about 5 bara, or about 6 bara, or about 7 bara, or about 8 bara.

- [0074] In some examples, the pressure may be generated by the addition of heated air, steam, or a combination thereof. In some examples, any increase in temperature may be generated by the addition of heated air, steam, or a combination thereof. Steam may be applied to the exterior of a drum or jacket or other container that contains the materials being mixed/heated, and/or may be added directly into a mixing area to more directly increase temperature and pressure, *e.g.* steam may be added externally until reaching the first elevated temperature, then after adding of addition component(s) steam is added directly into the chamber to reach a second elevated temperature, or vice versa. If a desired temperature is reached, air such as compressed air may be used to influence pressure, *e.g.* raise pressure further, to *e.g.* about two bara or about four bara for the hold period(s). For example, steam may be added until reaching a desired temperature and/or pressure to be used in a hold period and/or to maintain a desired temperature and/or pressure in a hold period, and then not applied after the hold period (*e.g.* at the end of treatment or between hold periods, if multiple).
- [0075] In some examples, the process may include aerating the mixture after processing at the hold period(s), for example through the addition of air, during and/or after the operation of mixing equipment. In some examples, the aeration is an ambient aeration, *e.g.* the reactor is opened and vented until reaching an ambient pressure, and then additional air is provided to aerate the material without subjecting it to relatively elevated pressures above ambient pressure (beyond minor changes in pressure from flowing air, *e.g.* about 4.82 m³/hr or 9.63 m³/hr of air, that can also escape the opened and vented reactor). In some examples, the aeration is a pressurized aeration, *e.g.* the reactor is closed and the vent valve is adjusted to maintain a pressure above ambient pressure while additional airflow is added, for example about 3.21 m³/hr or about 4.82 m³/hr of air is blown into the reactor and the vent valve is adjusted such that the inner pressure may be maintained during the addition of airflow at a pressure of about 4 bara, or about 5 bara, or about 6 bara, or about 7 bara, or some other pressure above ambient pressure. In some examples, an ambient aeration is performed, and then the reactor is sealed and a pressurized aeration is performed afterward. In certain examples of the process, at least some

aeration is pressurized aeration. In some examples, pressurized aeration is used instead of any depressurizing between hold periods, if multiple hold periods.

[0076] For example, air may be added (in an ambient aeration, a pressurized aeration, or both) during mixing after a hold period for a period of about one minute, about two minutes, about three minutes, about four minutes, about five minutes, about six minutes, about seven minutes, about eight minutes, about ten minutes, or about fifteen minutes, or about 30 minutes, or about 45 minutes, or about 60 minutes, or about 75 minutes, or about 90 minutes. In some examples, air may be added for about fifteen minutes or less, or about ten minutes or less, or about five minutes or less, or about one minute or more, or about three minutes or more, or about five minutes or more, or about ten minutes or more, or about 10-15 minutes, or about 5-10 minutes, or about 1-3 minutes, or about 3-5 minutes, or about 5-8 minutes, or about 1-5 minutes, or about 15-30 minutes, or about 15-45 minutes, or about 45-60 minutes, or about 45-75 minutes, or about 60-75 minutes). In some examples of pressurized aerations, air is added for at about 30 minutes or more, or about 45 minutes or more, or about 60 minutes or more, or about 75 minutes or more. Air may be added for a desired aeration period, and/or for whatever time is appropriate to reach a desired intermediate temperature for the materials being mixed, *e.g.* about 80 degrees Celsius or about 150 degrees Celsius or about 90 degrees Celsius or about 130 degrees Celsius, rather than a defined time period.

[0077] Air may be added, *e.g.* after venting and/or opening the chamber containing the materials being mixed until reaching a lower temperature and/or pressure in an ambient aeration, or into a closed or partially closed reactor in a pressurized aeration, to the mixing material at a rate of about 1.61 m³/hr, about 3.21 m³/hr, about 4.82 m³/hr, about 6.42 m³/hr, about 8.03 m³/hr, about 9.63 m³/hr, about 12.84 m³/hr, or about 16.05 m³/hr, or about 16.05 m³/hr or less, or about 8.03 m³/hr or less, or about 4.82 m³/hr or less, or about 1.61 m³/hr or more, about 3.21 m³/hr or more, about 4.82 m³/hr or more, about 8.03 m³/hr or more, or about 16.05 m³/hr or more, or about 1-8.03 m³/hr, about 5-16.05 m³/hr, or about 2-8.03 m³/hr, or about 5-12.84 m³/hr, or about 3-12.84

m³/hr. The number of aerations/air replenishments between hold periods may be 1 or more, 2 or more, 3 or more, 4 or more, 5 or more, 4 or less, 3 or less, 2 or less, 1 or less, or 1-10, 1-5, 3-8, 2-4, or 1-2.

5 **[0078]** In some examples, any alkalizing agents and any cocoa materials may be combined in a reactor or a chamber where the materials are mixed, *e.g.* under temperature and pressure and/or cycles of the same and then may be subjected to additional treatments in the chamber(s) such as the addition of air or imposition of vacuum. For example, the materials may undergo mixing at a second elevated temperature for a hold period inside
10 a reactor or chamber, which is then opened to allow aeration/cooling/depressurizing for an aeration period (*e.g.* 15 minutes or less) to an intermediate elevated temperature (lower than the second elevated temperature but higher than ambient) and then the reactor is closed to again raise the temperature/pressure for another hold period, all while mixing or other mechanical action is ongoing.

15 **[0079]** In some examples, the process includes drying the obtained mixture to provide an alkalized cocoa material. In some examples, the obtained, alkalized mixture may be dried, for example by air drying, hot air drying, vacuum drying, or combinations thereof. In some examples, the alkalized mixture may be dried multiple ways, *e.g.* some is air
20 dried and some is vacuum dried, for example under reaching a desired moisture content such as about 10% or less, by weight, or about 7% or less, or about 5% or less. In some example, some or all of the mixture is dried by hot air, *e.g.* by blowing air at a temperature equal to the first and/or second elevated temperature. In some examples, the alkalized mixture is dried via vacuum drying for a period of about 15-75 minutes,
25 or about 30-60 minutes.

30 **[0080]** As noted above, in some examples of the process, the process may include adding water to at least the cocoa (potentially with one or more alkalizing agents present and/or added later) and then mixing the water and at least the cocoa. For example, water may be added to a combination of cocoa and the first alkalization agent, or after both

alkalization agents are added but before an initial hold period of processing. The water may be added at a selected elevated temperature, after already added ingredients have been heated to a desired level such as 90-115 degrees Celsius, or about 90-100 degrees Celsius, or about 120-150 degrees Celsius, *e.g.* during a ramp up to a desired elevated temperature later achieved, such as through the continued addition of steam. In some examples, the amount of water added is equal to about 20%, by weight, of the amount of cocoa material, *e.g.* if 1 kg of cocoa material is being alkalized, 200 grams of water is added. In some examples, the amount of water is equal to about 10%, by weight, of the amount of cocoa material, or about 15%, or about 20%, or about 25%, or about 30%, or about 35%, or about 40%, or about 45%, or about 50%, or about 60%, or about 10-60%, or about 20-40%, or about 30-50%, or about 10-25%, or about 50% or less, or about 40% or less, or about 30% or less, or about 20% or less, or about 10% or less, or about 10% or more, or about 15% or more, or about 20% or more, or about 30% or more, or about 50% or more. Water may be added to provide an overall moisture content, by weight of the overall mixture, of about 20% or more, 30% or more, 50% or more, 40% or less, 50% or less, or 20% or less, or about 10-60%, about 20-50%, about 10-40%, about 25-55%, about 20-30%, about 30-40%, or about 10-15%, or about 10-30%.

[0081] In some examples, the amount of the first alkalizing agent added is equal to about 1%, by weight, of the amount of cocoa material, *e.g.* if 1 kg of cocoa material is being alkalized, 10 grams of the first alkalizing agent (*e.g.* MgOH or NaOH) is added. In some examples, the amount of the first alkalizing agent is equal to about 2%, by weight, of the amount of cocoa material, or about 3%, or about 4%, or about 5%, or about 7%, or about 10%, or about 12%, or about 15%, or about 1-15%, or about 1-4%, or about 4-10%, or about 2-4%, or about 4-8%, or about 5-10% or about 4-20%, or about 5-18%, or about 7-14%, or about 15% or less, or about 10% or less, or about 8% or less, or about 4% or less, or about 3% or less, or about 2% or less, or about 1% or less, or about 1% or more, or about 2% or more, or about 3% or more, or about 5% or more, or about 8% or more.

[0082] In some examples, the amount of the second alkalizing agent added is equal to about 1%, by weight, of the amount of cocoa material, *e.g.* if 1 kg of cocoa material is being alkalized, 10 grams of the second alkalizing agent (*e.g.* calcium carbonate or ammonium carbonate or ammonium bicarbonate) is added. In some examples, the amount of the second alkalizing agent is equal to about 2%, by weight, of the amount of cocoa material, or about 3%, or about 4%, or about 5%, or about 7%, or about 10%, or about 12%, or about 15%, or about 1-15%, or about 1-4%, or about 4-10%, or about 2-4%, or about 4-8%, or about 15% or less, or about 10% or less, or about 8% or less, or about 4% or less, or about 3% or less, or about 2% or less, or about 1% or less, or about 1% or more, or about 2% or more, or about 3% or more, or about 5% or more, or about 8% or more. As shown above by the illustrative examples, the molar amounts of the alkalizing agents may differ and the first agent may be present in a higher molar amount than the second, *e.g.* if the first agent is a hydroxide compound and the second agent is a carbonate compound, or vice versa, *e.g.* if the first agent is a carbonate compound and the second agent is a hydroxide compound.

[0083] The alkalizing agents may be added in solid or liquid form. If added in liquid form, the concentration of agents may be about 25% solution or about a 50% solution (with the active concentration used to determine the amount of agent to add, *i.e.* the diluent/water of the solution is not used when determining the amount of agent, by weight, that should be present), or about 25-75%, or about 25-50%, or about 15-40%. In some examples, the total amount of alkalizing agents is about 1-20%, by weight, of the amount of cocoa material, or about 3-18%, or about 5-14%, or about 6-18%, or about 7-12%, or about 5-18%, or about 11-22%, or about 13-17% or about 8-10%, or about 18% or less, or about 15% or less, or about 14% or less, or about 12% or less, or about 10% or less, or about 8% or less, or about 7% or less, or about 6% or less, or about 4% or more, or about 5% or more, or about 7% or more, or about 9% or more, or about 12% or more. In some examples, the inventive processes surprisingly result in cocoa materials including cocoa

cake that are alkalized to a dark, dark cocoa material using a total amount of alkalizing agents less than 7% by weight (*e.g.* 6.9%). If a diluted liquid form is used, the amount of water added via the alkalizing agent(s) may be subtracted from the amount of additional water added to the reactor, if any.

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[0084] In some examples, the ratio of the two alkalizing agents to each other are about equal, *i.e.* each provides about 50% of the total amount of alkali added, by weight, or in a ratio of about 45% to 55%, or a ratio of about 40% to 60%, or a ratio of about 35% to 65%, or a ratio of about 30% to 70%. In some examples, a third agent may be used, where
10 amounts may be about equal or within about 10% of each other, based on the total amount of alkali added, *e.g.* all are within 23%-43% by weight of the total alkali and total 100%. In certain examples, an alkalizing agent including ammonium is present in a relatively smaller amount than an alkalizing agent that does not include ammonium. In certain examples, a hydroxide alkalizing agent is present in a higher amount, by
15 weight, than the second agent. In other examples, an ammonium agent is present in a higher amount, by weight, than a hydroxide agent.

[0085] In some examples, no solvents are used in the preparation of the alkalized cocoa material. In some examples of the process, the process may include adding compressed
20 air to the cocoa, the first alkalization agent, and the second alkalization agent after at least one hold period, for example to lower the temperature of the mixture, and/or to aerate the material(s), either at the end of the process and/or between hold periods, if multiple. As discussed herein, the procedures may involve adding cocoa, alkalizing agents, and water together, and mixing under heat for a hold period, then
25 depressurizing/aerating the mixture while continuing to mix before repeating a higher temperature/pressurized hold period, such that cycles of hold periods and depressurized aeration may be used before final drying step(s) after the final hold period.

[0086] In some examples, the alkalized cocoa material may include a moisture content of about
30 1-20%, by weight, or about 10-50%, or about 20-40%, or about 10-30%, or about 20-

50%, e.g. about 35% or less, about 25% or less, about 15% or less, about 13% or less, about 10% or less, about 8% or less, about 5% or less, or about 3% or less, or about 1% or less, or about 3-7%, about 2-8%, about 8-14%, or about 8-18%, or about 6-14%. In certain examples, the alkalized material is dried via vacuum to a moisture content of about 8% or less, or about 5% or less. The vacuum process may not commence until the material is cooled below the final hold period elevated temperature, e.g. to a temperature of about 80-120 degrees Celsius, or about 100-115 degrees Celsius. The vacuum pressure may be about 22-36 mm Hg, or about 26-30 mm Hg, or about 30-36 mm Hg. Moisture content is as determined by the oven drying method, ISO 2291.

[0087] In certain examples, the cocoa being alkalized may include or entirely consist of cocoa cake. In certain examples, the cocoa being alkalized has a fat content of about 60% or less, about 30% or less, about 40% or less, about 30% or less, about 25% or less about 15% or less, about 12% or less, about 11% or less, or about 10% or less, preferably about 6-30%, more preferably about 7-25%, even more preferably about 8-14%, or about 5-30%, or about 8-24%, or about 10-16%. In certain examples, the cocoa material being alkalized may have a moisture content of about 1-20%, by weight, before alkalization, about 15% or less, about 13% or less, about 10% or less, about 8% or less, about 5% or less, or about 3% or less, about 1-6%, about 2-5%, or about 1-10%. Fat content is as determined by the Soxhlet method, IOCCC method NR.115 – 1990.

[0088] In some examples, the alkalized cocoa material is a dark cocoa material. In some examples, the alkalized cocoa material is a dark cocoa material. In certain examples, the alkalized cocoa material is a dark cocoa material. The alkalized cocoa material may be water soluble. In some examples, the obtained material is ground or milled into smaller pieces, e.g. after any drying, for example may be made into ground alkalized cocoa powder.

[0089] As discussed and illustrated herein, these cake alkalization processes can produce highly alkalized dark cocoa materials such as dark cocoa powders. Color may be measured

using the following parameters: Lightness (L), which is grey scale value or the light or dark aspect of a color, where the lower the L-value, the darker the cocoa powder will appear; Chroma (C), which is the intensity of a color by which one distinguishes a bright or gray color, where the higher the C-value, the brighter the powder will be; and Hue (H), which is referring to color in daily speech, such as red, yellow, or blue, where for cocoa products, a low H value indicates a red color and a high H-value indicates a brown color; the A or a* parameter represents the range from green to red, and the B or b* parameter, which represents the range from blue to yellow.

10 [0090] In certain examples, the alkalized cocoa material has color values in the following ranges. For intrinsic color analysis (methodology provided below), example compositions have an L value of about 0.2-6, a C value of about 0.5-6, and H value of about 35-45, an A value of about 0.3-4.5, and a B value of about 0.2-4.5. For white diluent color analysis (methodology provided below), example compositions have an L value of about 16-30, a C value of about 3-9, and H value of about 40-55, an A value of about 2-6, and a B value of about 1.5-8.

20 [0091] Color values are as measured by a Datacolor Spectraflash Color Spectrophotometer (models SF450X, 500, 700 or 800), with the following settings, Illuminant D65, standard observer 10°, color in L*a*b*C*h or L*C*h according to CIELAB, measuring geometry d/8, aperture plate LAV (30mm illuminated, 26mm measured), specular reflection excluded, and UV-FILTER 0%. After an initial calibration (confirmed by green tile measurement and/or a control sample with known color reference values, which is prepared and tested the same way as the test sample), a test cocoa sample is prepared and tested to obtain intrinsic color values. About 7.5 grams of test cocoa is added to 100 mL of water and stirred to remove lumps and ensure consistency, then another 50 mL of water is added and the suspension is pumped through a quartz flow cuvette while stirring and the color values are recorded using the spectrophotometer. For the white diluent color analysis values, the procedure is the same as the intrinsic

color analysis described above, except that a calcium carbonate/water medium having a L value of 82-89 is prepared and used instead of pure water.

5 [0092] In some examples, the alkalized material at the end of the process may have a low ash content, such as about 12% or less (as determined by IOCCC Method 16) by weight, or about 11% or less, or about 10% or less, or about 9% or less, or about 8% or less, or about 8-12%, about 8-10%, or about 10-12%. In some examples, the alkalized material at the end of the process may have a low sodium content, such as about 20,000 or less (as determined by NEN-EN 15510 ICP-AES method) or about 15,000 or less, or about 10 12,000 or less, or about 12,000-24,000, or about 10,000-12,000, or about 20,000-24,000 mg/kg. In some examples, the alkalized material at the end of the process may have a low acrylamide content, such as about 150 micrograms per kilogram or less (as determined by CHE587W-HPLC-MS/MS method) or about 100 or less, or about 50 or less, or about 30 or less.

15 [0093] In an example of the process, dark cocoa cake is made by mixing (*e.g.* chopping and plowing, with agitation at a speed of 50-170 revolutions per minute chopping at a speed of 1000-3500 revolutions per minute) cocoa cake and a first alkalization agent together at an initial temperature of 100-160 degrees Celsius, *e.g.* 90-110, then mixing and heating the cocoa cake and the first alkalization agent, *e.g.* a hydroxide material like sodium hydroxide, until reaching a first elevated mixing temperature of 100-150 degrees Celsius, *e.g.* 100-130 degrees, and an elevated pressure of about 2-8 bara, *e.g.* 4-8 bara or 6-8 bara, then adding a second alkalization agent, *e.g.* an ammonium material like ammonium carbonate or ammonium bicarbonate, to the cocoa cake and the first 20 alkalization agent, and then adding water and mixing. The total amount of alkalizing material is about 15% or less, by weight, *e.g.* about 14.5%, about 12%, about 8%, or about 6.9%. In this example, the process then includes mixing and heating the materials until reaching a second elevated mixing temperature of 100-150 degrees Celsius, *e.g.* 120-150 degrees, and an elevated pressure of about 4-8 bara, *e.g.* 6-8 bara, and then 25 mixing materials at the second elevated mixing temperature for at an initial hold period 30

of 30-150 minutes in length, *e.g.* 90 minutes, or 60 minutes, to provide a mixture. In this example, the process then includes aerating and cooling the mixture, *e.g.* via an ambient aeration (but a pressurized aeration, *e.g.* using 4.82 to 9.63 m³/hr of air and a maximum pressure of 4-6 bara, is also possible), and then heating the mixture to a third elevated mixing temperature of 75-150 degrees Celsius, *e.g.* 120-150 degrees, and an elevated pressure of about 4-8 bara, *e.g.* 6-8 bara, and mixing for at least one additional hold period of 30-150 minutes in length, *e.g.* 20 minutes each, *e.g.* two additional hold periods or three additional hold periods (with cooling/depressurizing/aerating in between additional hold periods). In this example, the pressure is increased by adding steam. The mixture was then dried with vacuum drying, until then alkalized cocoa material has a moisture content of 1-20%, by weight, *e.g.* 5-15%. The resulting product may have a moisture content of less than 10%, *e.g.* 4-8%. The resulting product may have an ash content of about 15% or less, *e.g.* about 8-12%. The resulting product may have a sodium content of about 25,000 mg/kg or less, *e.g.* about 10,000-20,000 or about 10,000-12,000. The resulting product may have a potassium content of about 25,000 mg/kg or less. The resulting product may have an acrylamide content of about 150 micrograms/kg or less, *e.g.* about or less than 30 micrograms/kg. The resulting product may have, for intrinsic color analysis (CIELAB), an L value of about 0.2-6, a C value of about 0.5-6, and H value of about 35-45, an A value of about 0.3-4.5, and a B value of about 0.2-4.5., and for white diluent color analysis, an L value of about 16-30, a C value of about 3-9, and H value of about 40-55, an A value of about 2-6, and a B value of about 1.5-8.

[0094] In an example of the process, dark cocoa cake is made by mixing (*e.g.* chopping and plowing, with agitation at a speed of 50-170 revolutions per minute and chopping at a speed of 1000-3500 revolutions per minute) the cocoa cake, a first alkalization agent, *e.g.* a hydroxide material like sodium hydroxide, and a second alkalization agent, *e.g.* an ammonium material like ammonium carbonate or ammonium bicarbonate, and then adding water and mixing. The total amount of alkalizing material is about 15% or less, by weight, *e.g.* about 14.5%, about 12%, about 8%, or about 6.9%. In this example, the

process then includes mixing and heating the materials until reaching a first elevated mixing temperature of 75-150 degrees Celsius, *e.g.* 120-150 degrees, and an elevated pressure of about 4-8 bara, *e.g.* 6-8 bara, and then mixing materials at the second elevated mixing temperature for at an initial hold period of 30-150 minutes in length, *e.g.* 90 minutes, or 60 minutes, to provide a mixture. In this example, the process then includes aerating and cooling the mixture, *e.g.* via an ambient aeration (but a pressurized aeration, *e.g.* using 3- 4.82 to 9.63 m³/hr of air and a maximum pressure of 4-6 bara, is also possible), and then heating the mixture to a third elevated mixing temperature of 75-150 degrees Celsius, *e.g.* 120-150 degrees, and an elevated pressure of about 4-8 bara, *e.g.* 6-8 bara, and mixing for at least one additional hold period of 30-150 minutes in length, *e.g.* 20 minutes each, *e.g.* two additional hold periods or three additional hold periods (with cooling/depressurizing/aerating in between additional hold periods). In this example, the pressure is increased by adding steam. The mixture was then dried with vacuum drying, until then alkalized cocoa material has a moisture content of 1-20%, by weight, *e.g.* 5-15%. The resulting product may have a moisture content of less than 10%, *e.g.* 4-8%. The resulting product may have an ash content of about 15% or less, *e.g.* about 8-12%. The resulting product may have a sodium content of about 25,000 mg/kg or less, *e.g.* about 10,000-20,000 or about 10,000-12,000. The resulting product may have a potassium content of about 25,000 mg/kg or less. The resulting product may have an acrylamide content of about 150 micrograms/kg or less, *e.g.* less than 30.

[0095] According to another preferred embodiment, the present invention relates to the process (or method), wherein the alkalized cocoa material has a dark (dark brown or even black) colour based on the CIELAB value, wherein L is between 16 to 30, preferably 17 to 25, mor preferably 18 to 22, a* is between 1 to 7, preferably 2 to 6, more preferably between 3 to 5, and b* is between 1 to 10 , preferably between 1.5 to 8 , more preferably between 2 to 7, as determined by spectrophotometer using the white diluent method (CIELAB). Suitable spectrophotometers are for example Datacolor Spectraflash color spectrophotometer (SF450X, 500, 700 or 800), according to the Datacolor DC series

5 userguide, using settings; Illuminant D65, standard observer 10°, geometry d/8°, aperture plate LAV (30mm illuminated, 26mm measured). The CIELAB values of cocoa powder can be used for quality control to ensure consistency in the color of their cocoa powder products across batches and for food scientists and product developers to adjust the color of cocoa powder formulations to meet specific requirements or to match the color of existing products improving product's appearance. Specifically, the L value represents the lightness or darkness of the cocoa powder, with 0 being black and 100 being white. The a* and b* values represent the color components along the green-red and blue-yellow axes, respectively.

10 [0096] In another aspect of the disclosure, edible compositions such as cookies or other desserts are provided. Example edible compositions include cookies, cakes, breads, beverages, ice cream, pies, or truffles. In some examples, an edible composition includes an alkalized cocoa material made from a process of this disclosure, such as a dark cocoa material. Edible compositions may include alkalized cocoa material with any of the
15 property characteristics discussed herein, including the properties identified in Tables 1-2 below. E.g. a cookie may include a dark alkalized cocoa powder with an intrinsic color L value between 0.2-6. In other examples, an edible composition includes two or more alkalized cocoa materials, each made from distinct processes of this disclosure.

20 [0097] Compositions made from the methods of this disclosed may have the following properties provided across Tables 1-3.

TABLE 1

Intrinsic Color Analysis					
	<u>L</u>	<u>C</u>	<u>H</u>	<u>A</u>	<u>B</u>
<u>Minimum</u>	0.2	0.5	35	0.3	0.2
<u>Maximum</u>	6	6	45	4.5	4.5

TABLE 2

White Diluent Color Analysis					
	<u>L</u>	<u>C</u>	<u>H</u>	<u>A</u>	<u>B</u>
<u>Minimum</u>	16	3	40	2	1.5
<u>Maximum</u>	30	9	55	6	8

TABLE 3

Other Properties						
	Fat Content (%)	pH	Na (mg/kg)	K (mg/kg)	Ash (%)	Acrylamide (%)
<u>Minimum</u>	8	6.3	9,000	20,000	7.0	<30
<u>Maximum</u>	24	8.25	24,000	23,000	16	300

5 EXAMPLES

[0098] Example processes according to present invention are below to further illustrate the content of this disclosure.

- 10 [0099] In a first working example, about 90 kg of cocoa cake (11% fat) was combined with about 3.8 kg sodium hydroxide and about 2.5 kg ammonium bicarbonate. The materials were combined and mixed (plow speed 70 rpm and chopper speed 1740 rpm) while steam was applied to the reactor jacket until reaching a temperature of 90 degrees Celsius, at which point about 27 kg of water was added via injection using the pressure
- 15 pot, and mixing was increased (105 rpm for plow and 2610 rpm for chopper). The mixture was heated by the application of steam until reaching a mixture temperature of about 130 degrees Celsius, and a pressure of about 4 bara, where the reactor was completely sealed. The components were then mixed under these conditions for about 90 minutes, where the pressure increased as the reaction proceeded, and the reactor in

this stage was set to have a maximum pressure of about 6 bara and to drop to about 5.5 bara if a reduction in pressure was needed. The reactor was then opened and the mixture was aerated using about 9.63 m³/hr blown air for about five minutes to (cool the mixture and) replenish air. Then the reactor was sealed and the mixture underwent three additional, similar heated and pressurized mixing cycles where the additional cycles each had a hold period of about 20 minutes. Total holding time in the process (product residence time at elevated temperature/pressure) was about 150 minutes. Then mixture was then dried via vacuum drying for 15 minutes. Property information for the prepared material is provided in the table below.

[00100] In a second working example, about 90 kg of cocoa cake (11% fat) was combined with about 1.73 kg sodium hydroxide and about 1.13 kg ammonium bicarbonate. The materials were combined and mixed (plow speed 105 rpm and chopper speed 3480 rpm) while steam was applied to the reactor jacket until reaching a temperature of 90 degrees Celsius, at which point about 27 kg of water was added via injection using the pressure pot. The mixture was heated by the application of steam until reaching a mixture temperature of about 130 degrees Celsius, and a pressure of about 5 bara, where the reactor was completely sealed. The components were then mixed under these conditions for about 90 minutes, where the pressure increased as the reaction proceeded, and the reactor in this stage was set to have a maximum pressure of about 7 bara and to drop to about 6.5 bara if a reduction in pressure was needed. The reactor was then opened and the mixture was aerated using about 9.63 m³/hr blown air for about five minutes to replenish air, and was then further aerated using a pressurized aeration, with a closed/appropriately vented reactor heated to about 125 degrees Celsius using about 4.82 m³/hr blown air to provide a pressure of about 7 bara, with these conditions held for about 60 minutes. The mixture then underwent three additional, heated and pressurized mixing hold cycles under similar conditions as the initial 90 minute hold period, with each additional hold in the fully sealed reactor totaling 60 minutes. The mixture was then dried via vacuum drying for 15 minutes. The total

holding time in the process was about 270 minutes. Property information for the prepared material is provided in the table below.

- 5 **[00101]** In a related, third example (prophetic), the fourth example is repeated, but the first hold period is thirty minutes in length and, prior to vacuum drying but after the pressurized aeration, the reactor is opened/depressurized, then re-sealed and the mixture undergoes 2 additional, similar heated and pressurized mixing cycles under conditions of the first hold period, where the additional cycles each have a hold period of about 30 minutes.
- 10 **[00102]** In a fourth example (prophetic), about 90 kg of cocoa cake (11% fat) is combined with a total amount of alkalizing agents equivalent to 6.9% wt. of the cocoa material, where 60% of the total alkalizing agent amount is sodium hydroxide and 40% is ammonium carbonate or ammonium bicarbonate. The materials are combined and
- 15 mixed (plow speed 113 rpm and chopper speed 3480 rpm) while steam is applied to the reactor jacket until reaching a temperature of 90 degrees Celsius, at which point about 27 kg of water is added via injection using the pressure pot. The mixture is heated by the application of steam until reaching a mixture temperature of about 150 degrees Celsius, and a pressure of about 5 bara, where the reactor is completely sealed. The
- 20 components are then mixed under these conditions for about 90 minutes, where the pressure increases as the reaction proceeded, and the reactor in this stage is set to have a maximum pressure of about 8 bara and to drop to about 7.5 bara if a reduction in pressure was needed. The reactor is then opened and the mixture is aerated using about 9.63 m³/hr blown air for about five minutes to cool the mixture and replenish air, and
- 25 is then further aerated using a pressurized aeration, with a closed/appropriately vented reactor heated to about 125 degrees Celsius using about 9.63 m³/hr blown air to provide a pressure of about 8 bara, with these conditions held for about 90 minutes. The mixture is then dried via vacuum drying for 30 minutes.

- 5 **[00103]** In a fifth example (prophetic), the sixth example is repeated, but prior to vacuum drying, and after the pressurized aeration, the reactor is opened/depressurized, then re-sealed and the mixture undergoes one additional, similar heated and pressurized mixing cycle under conditions of the first hold period, where the additional cycle has a hold period of about 45 minutes.
- 10 **[00104]** In a reference example 1, 3,500 kg of cocoa powder was added to a horizontal reactor, without chopping, and about 105 kg of ammonium bicarbonate and about 98 kg of sodium hydroxide were combined at ambient temperature and atmospheric pressure, heated to about 90 degrees Celsius, and about 1,225 kg of water was added. The mixture was heated to a temperature of about 115 degrees, and a stirrer was operated at about 125 rpm. Then direct steam injection was used to bring the product temperature to 110-120 °C and reactor inner pressure at 1.6 bar. The mixture was kept for 90 minutes of the conditions where the materials were at 110-120 degrees Celsius and under a pressure of 1.4-1.6 bar. Then the reactor was opened, pressure was released to atmospheric pressure in 15 minutes, further cooled with blown air for 180 minutes, and the obtained mixture was dried via vacuum drying. The total process time was about 620 minutes. Property information for the prepared material is provided in the table below.
- 20 **[00105]** In a reference example 2, about 3,500 kg of cocoa powder (10.9% fat content) and about 225 kg of ammonium bicarbonate and about 200 kg of sodium hydroxide were combined and charged to a reactor, where they were mixed, heated to about 90 °C, and then about 1,250 kg of water was added. The mixture was heated, until reaching a temperature of about 120 degrees Celsius and pressure of about 3.5 bar. After venting, 25 the mixture was kept at a temperature of about 125-130 degrees Celsius and the pressure fell from about 3.5 to 2.4 bar from venting during the mixing during the cycle, where mixing was maintained under these conditions for 60 minutes. Next, the reactor was depressurized, opening the chimney valve from 0% to 100% in 10 minutes. Product temperature fell down from 133 degrees Celsius to 111 degrees Celsius and pressure 30 dropped from 2.4 bar to 1.1 bar. Next, the temperature/pressure cycle was repeated 3

times, with 60 minutes of reaction at a temperature of about 135 degrees Celsius and a pressure of about 3.4 bar for each 60 minute cycle. Then the reactor was opened, and the obtained mixture was dried via vacuum drying. The total process time was 702 minutes. Property information for the prepared material is provided in the table below.

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[00106] Color measurements are performed of the alkalized cocoa material produced in the above examples to characterize the products. Color measurements on the alkalized cocoa material of the above examples were performed using a Spectraflash 500 spectrophotometer (Datacolor), according to manufacturer's instructions. The color measurements were registered per extract according to the CIELAB color parameters, based on using the intrinsic color measurement or the white diluent measurement. The CIELAB is a color model used to describe the colors in three dimensions: lightness (L), chroma (a*, representing the green-red axis), and hue (b*, representing the blue-yellow axis). It is a standardized color space defined by the International Commission on Illumination (CIE). When it comes to cocoa powder, the CIELAB value provides information about its color characteristics. Specifically, the L value represents the lightness or darkness of the cocoa powder, with 0 being black and 100 being white. The a* and b* values represent the color components along the green-red and blue-yellow axes, respectively. From the a* and b* values, the C and h values are calculated according to the CIELAB color model. Furthermore, the product characteristics were measured per alkalized cocoa material produced in the examples, including moisture content (MC), pH, fat content, Na and K, Ash and acrylamide content (via HPLC-MS or MS), see Table 4.

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TABLE 4. Characteristics of the alkalized cocoa material of the examples.

	Ex 1	Ex 2	Ref 1	Ref 2
Moisture Content (wt%)	6.2	3.8	2.78	4.15
Fat Content (wt%)	9.0	11.1	-	-
Sodium Content (mg/kg)	23,000	11,000	-	-

Potassium Content (mg/kg)	21,000	22,000	-	-
Ash Content (wt%)	10.9	8.6	-	-
Acrylamide Content (µg/kg)	<30	149	-	-
pH	7.4	6.1	8.2	8.3
L (intrinsic)	2.4	2.5	10.1	4.1
C (intrinsic)	1.6	2.5	4.4	5.5
h (intrinsic)	40.4	38.4	21.1	34.2
L (white diluent)	21.6	21.4	38.7	26.35
C (white diluent)	6.8	7.0	20.0	12.2
h (white diluent)	47.6	46.3	52.1	49.5

RESULTS

5 **[00107]** In view of the alkalized cocoa product obtained via the process of the reference example, the alkalized cocoa products obtain according to the process of the examples, have a darker color, while using equal or less alkaline solution and obtained via a much more efficient and significantly faster process (for example the holding time is significantly shorter). In part, this was due to higher processing pressure, choppers at high speed for breaking lumps ultimately resulting in more surface area for reaction, processing sequence and opex efficiency. Furthermore, it was observed that when using higher temperatures and pressure the darker the cocoa is obtained, however going outside the indicated ranges of the process of the invention the flavor is affected and results in (over)cooking and/or burning of the product.

15 **[00108]** These materials, systems and process descriptions are merely examples. In certain embodiments, the processes include additional combinations and/or substitutions of some or all of steps and/or use of the components or conditions

described above. Moreover, additional and alternative suitable variations, forms and steps will be recognized by those skilled in the art given the benefit of this disclosure. Finally, any of the features discussed in the example embodiments of the processes and/or systems may be features of embodiments of the resulting materials, and vice versa.

[00109] The disclosure further comprises the following clauses;

1. A multi-stage process for forming an alkalized cocoa cake material comprising the steps of:

mixing cocoa cake and a first alkalization agent together at an initial temperature, the cocoa cake comprising a fat content of 8-24%, by weight, and a moisture content of 1-7%, by weight;

mixing and heating the cocoa cake and the first alkalization agent until reaching a first elevated mixing temperature;

adding a second alkalization agent to the cocoa cake and the first alkalization agent;

mixing and heating the cocoa cake, the first alkalization agent, and the second alkalization agent through the applications of external heat until reaching a second elevated mixing temperature;

mixing the cocoa, the first alkalization agent, and the second alkalization agent at the second elevated mixing temperature for at an initial hold period to provide a mixture;

aerating and cooling the mixture;

heating the mixture to a third elevated mixing temperature and mixing for at least one additional hold period; and

drying the mixture to provide an alkalized cocoa material;

wherein all of the elevated mixing temperatures are 75-150 degrees Celsius;

wherein any hold periods are 10-150 minutes in length, and wherein a total residence time of the hold periods is 100-350 minutes;

wherein the mixture is aerated and cooled between any of the at least one additional hold periods; and

5 wherein water is added to at least the cocoa cake and mixed with at least the cocoa cake prior to the initial hold period.

2. The process of clause 1, wherein the first alkalization agent and the second alkalization agent each comprise or consist of one or more hydroxide materials, one or more bicarbonate materials, one or more carbonate materials, one or more alkaline oxide materials,
10 or combinations thereof.

3. The process of clause 2, wherein the first alkalization agent and the second alkalization agent each comprise or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide, sodium carbonate, potassium carbonate, ammonium carbonate, ammonium
15 bicarbonate, calcium carbonate, magnesium oxide, or combinations thereof.

4. The process of clause 1, wherein any mixing comprises agitating the materials being mixed, chopping the materials being mixed, or both.

20 5. The process of clause 1, wherein the first alkalization agent and the second alkalization agent are each solid alkalization agent or a liquid alkalization agent with a concentration of 25-75%, by weight, of the active alkalization agent.

6. The process of clause 1, wherein the first alkalization agent and the second
25 alkalization agent each comprise or consist of sodium hydroxide, ammonium bicarbonate, ammonium carbonate, or ammonium hydroxide.

7. The process of clause 1, wherein the at least one additional hold period includes at least two additional hold periods, and wherein the mixture is cooled and aerated between the additional hold periods.

5 8. The process of clause 1, wherein the at least one additional hold period includes at least three additional hold periods, and wherein the mixture is cooled and aerated between the additional hold periods.

9. The process of clause 1, wherein the cocoa, the first alkalization agent, and the
10 second alkalization agent are subjected to an elevated pressure above 2 bars during the initial hold period and the at least one additional hold period.

10. The process of clause 8, wherein the cocoa, the first alkalization agent, and the
15 second alkalization agent are subjected to an elevated pressure of up to 8 bars during the initial hold period and the at least one additional hold period.

11. The process of clause 10, wherein the pressure is generated by the addition of heated air, steam, or a combination thereof.

20 12. The process of clause 1, wherein the mixture is dried by air drying, hot air drying, vacuum drying, or combinations thereof.

13. The process of clause 1, further comprising adding compressed air to the cocoa,
the first alkalization agent, and the second alkalization agent after at least one additional hold
25 period to lower the temperature of the mixture.

14. The process of clause 1, wherein the total amount of the first and second alkalization agents is equal to 3-18%, by weight, of the amount of cocoa material.

15. The process of clause 1, wherein the alkalized cocoa material comprises a moisture content of 1-20%, by weight.

16. The process of clause 1, wherein any aeration is performed at an ambient
5 pressure.

17. The process of clause 1, wherein at least some aeration is performed at an elevated pressure.

10 18. A multi-stage process for forming an alkalized cocoa material comprising the steps of:

mixing and heating cocoa cake, a first alkalization agent, a second alkalization agent and water at a first elevated mixing temperature for at an initial hold period to provide a mixture;

aerating the mixture;

15 heating the mixture to a second elevated mixing temperature through the applications of external heat and mixing for at least one additional hold period; and

drying the mixture to provide an alkalized cocoa material;

wherein all of the elevated mixing temperatures are 75-150 degrees Celsius;

20 wherein the cocoa cake, the first alkalization agent, and the second alkalization agent are subjected to an elevated pressure above 2 bars during the initial hold period and the at least one additional hold period;

wherein any hold periods are 10-150 minutes in length, and wherein a total residence time of the hold periods is 100-350 minutes; and

wherein the mixture is aerated between any of the at least one additional hold periods.

19. The process of clause 18, wherein at least some aeration is performed at an elevated pressure.

20. An edible composition comprising an alkalized cocoa material made from the
5 process of clause 18.

CLAIMS

1. A process for production of an alkalized cocoa material comprising the steps of:
- mixing and heating in a reactor a cocoa raw material, a first alkalization agent, a second
5 alkalization agent at an elevated mixing temperature for a first hold period to provide a first mixture;
- aerating of the first mixture, and;
- optionally, subsequently heating the first mixture to the elevated mixing temperature
and mixing for at least one additional hold period providing a further mixture, wherein the
10 further mixture is aerated between any one of the at least one additional hold period(s);
- drying the first mixture or further mixture to provide the alkalized cocoa material;
- wherein the elevated mixing temperature is between 100 to 160 °C, preferably 120 to
150 °C, more preferably 125 to 135 °C,
- wherein any one of the hold periods are 20 to 150 minutes, preferably 40 to 110 minutes,
15 more preferably 50 to 90 minutes in length,
- wherein water is added to the cocoa raw material, the first alkalization agent and the
second alkalization agent prior to the first hold period,
- wherein an elevated pressure is present during any one of the hold periods, preferably
wherein the elevated pressure is between 1 to 8 bara, preferably 2 to 7 bara, more preferably 3
20 to 6 bara, most preferably 4 to 5 bara.
2. Process according to claim 1, wherein the total amount of alkalization agent in
the first mixture is between 6 to 18 wt%, preferably between 7 to 16 wt%, more preferably
between 8 to 15 wt%, most preferably 10 to 14 wt%, based on the total weight of the cocoa raw
25 material.

3. Process according to claims 1 or 2, wherein the at least one additional hold period comprises between one to five additional hold periods, preferably two to four additional hold periods.

5 4. Process according to any one of the preceding claims, wherein the first hold period is 75 to 90 minutes in length and the at least one additional hold period is shorter than the first hold period.

5. Process according to any one of the preceding claims, wherein aeration results in cooling of the first mixture or further mixture to a temperature below the elevated mixing temperature, preferably to a temperature of 1 to 50 °C, preferably 5 to 25 °C, more preferably
10 10 to 15 °C below the elevated mixing temperature.

6. Process according to any one of the preceding claims, wherein the aeration is done at an air flow rate of about 1.61 to 16.05 m³/hr, preferably 3.21 to 12.84 m³/hr, more preferably 4.82 to 9.63 m³/hr, even more preferably 6.42 to 8.03 m³/hr.

7. Process according to any one of the preceding claims, wherein the cocoa raw
15 material is one or more selected from the group consisting of cocoa beans, cocoa nibs, cocoa butter, cocoa cake, cocoa powder, cocoa liquor, and any source that comprises cocoa solids, preferably cocoa powder, cocoa liquor or cocoa cake.

8. Process according to any one of the preceding claims, wherein total length of the hold periods in the process is between 20 to 400 minutes, preferably 60 to 380 minutes, more
20 preferably 120 to 350 minutes, most preferably 150 to 320 minutes.

9. Process according to any one of the preceding claims, wherein the moisture content of the cocoa raw material is between 15 to 60 wt%, preferably 20 to 50 wt%, more preferably 25 to 40 wt%, based on the total weight of the cocoa raw material.

10. Process according to any one of the preceding claims, wherein the cocoa raw
25 material has a fat content of between 6 to 55 wt%, 8 to 24 wt%, preferably between 10 to 20 wt%, more preferably between 12 to 18 wt%, based on the total weight of the cocoa raw material.

11. Process according to any one of the preceding claims, wherein the water is added to the cocoa raw material, the first alkalization agent and the second alkalization agent when reaching a temperature during heating of about 80 to 105 °C, preferably 85 to 95, more preferably 87 to 90 °C.

12. The process of any one of the preceding claims, wherein the first alkalization agent and the second alkalization agent each comprise or consist of one or more hydroxide materials, one or more bicarbonate materials, one or more carbonate materials, or combinations thereof.

13. The process of any one of the preceding claims, wherein the first alkalization agent and the second alkalization agent are the same or are different, preferably different.

14. The process of any one of the preceding claims, wherein the first alkalization agent and the second alkalization agent each comprise or consist of sodium hydroxide or ammonium carbonate or ammonium bicarbonate or potassium carbonate.

15. The process of any one of the preceding claims, wherein the first alkalization agent and the second alkalization agent each comprise or consist of magnesium hydroxide, sodium hydroxide, calcium hydroxide, magnesium oxide, sodium carbonate, potassium carbonate, ammonium carbonate or ammonium bicarbonate, calcium carbonate, or combinations thereof.

16. The process of any one of the preceding claims, wherein any mixing comprises agitating the materials being mixed, chopping the materials being mixed, or both.

17. The process of claim 16, wherein any agitation is at a speed of 50-170 revolutions per minute (rpm), preferably 65 to 150 rpm, more preferably 70 to 100 rpm, and/or wherein any chopping is at a speed of 1000-3500 rpm, preferably 1400 to 2500 rpm, more preferably 1700 to 2000 rpm.

18. The process to any one of the preceding claims, wherein the elevated pressure is generated by the addition of heated air, steam, or a combination thereof.

5 19. The process of any one of the preceding claims, wherein the mixture is dried by air drying, hot air drying, vacuum drying, or combinations thereof.

20. The process of any one of the preceding claims, wherein aeration is done by adding compressed air to the reactor.

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21. The process of any one of the preceding claims, wherein any aeration is performed at an ambient pressure, more preferably at an atmospheric pressure of about 1 bar.

15 22. The process of any one of the preceding claims, wherein the aeration of the first mixture is performed at an elevated pressure of about 2 to 10 bara, preferably 3 to 8 bara, more preferably 4 to 6 bara.

20 23. An alkalized cocoa material obtained via or obtainable by the process of any one of the preceding claims.

24. Alkalized cocoa material according to claim 23, wherein the alkalized cocoa material has a fat content of preferably 6 to 30 wt%, more preferably 7 to 25 wt%, even more preferably 8 to 18 wt%, most preferably 10 to 15 wt%, based on the total weight of the cocoa material.

25 25. Alkalized cocoa material according to claim 23 or 24, wherein the alkalized cocoa material has a Na content of preferably 9000 to 24000 mg/kg of the cocoa material, more preferably 10000 to 20000 mg/kg, even more preferably 12500 to 18000 mg/kg, most preferably 15000 to 17500 mg/kg.

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26. Alkalized cocoa material according to any one of the claims 23 to 25, wherein the alkalized cocoa material has a K content of preferably 19000 to 24000 mg/kg of the cocoa material, more preferably 20000 to 23000 mg/kg, even more preferably 21000 to 22000 mg/kg.

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27. Alkalized cocoa material according to any one of the claims 23 to 26, wherein the alkalized cocoa material has an ash content of preferably 5 to 18 wt%, more preferably 7 to 16 wt%, even more preferably 8 to 14 wt%, most preferably 10 to 12 wt%, based on the total weight of the cocoa material.

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28. Alkalized cocoa material according to any one of the claims 23 to 27, wherein the alkalized cocoa material has an acrylamide content of at most 300 µg/kg of the cocoa material, preferably at most 200 µg/kg, more preferably at most 150 µg/kg, even more preferably at most 90 µg/kg, most preferably at most 30 µg/kg.

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29. Alkalized cocoa material according to any one of the claims 23 to 28, wherein the alkalized cocoa material has a dark color as based on a CIELAB value, wherein L is between 16 to 30, preferably 17 to 25, more preferably 18 to 22, C is between 5 to 9, preferably 5.5 to 8, more preferably between 6 to 7.5, and h is between 40 to 52, preferably between 42 to 50, more preferably between 44 to 48, as determined by spectrophotometer.

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30. Alkalized cocoa material according to any one of the claims 23 to 29, wherein the alkalized cocoa material has a pH of between 6.0 to 8.5, preferably between 6.2 to 8.2, more preferably 6.5 to 7.8, most preferably 6.8 to 7.5.

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31. An edible composition comprising an alkalized cocoa material according to any one of the claims 23 to 30.