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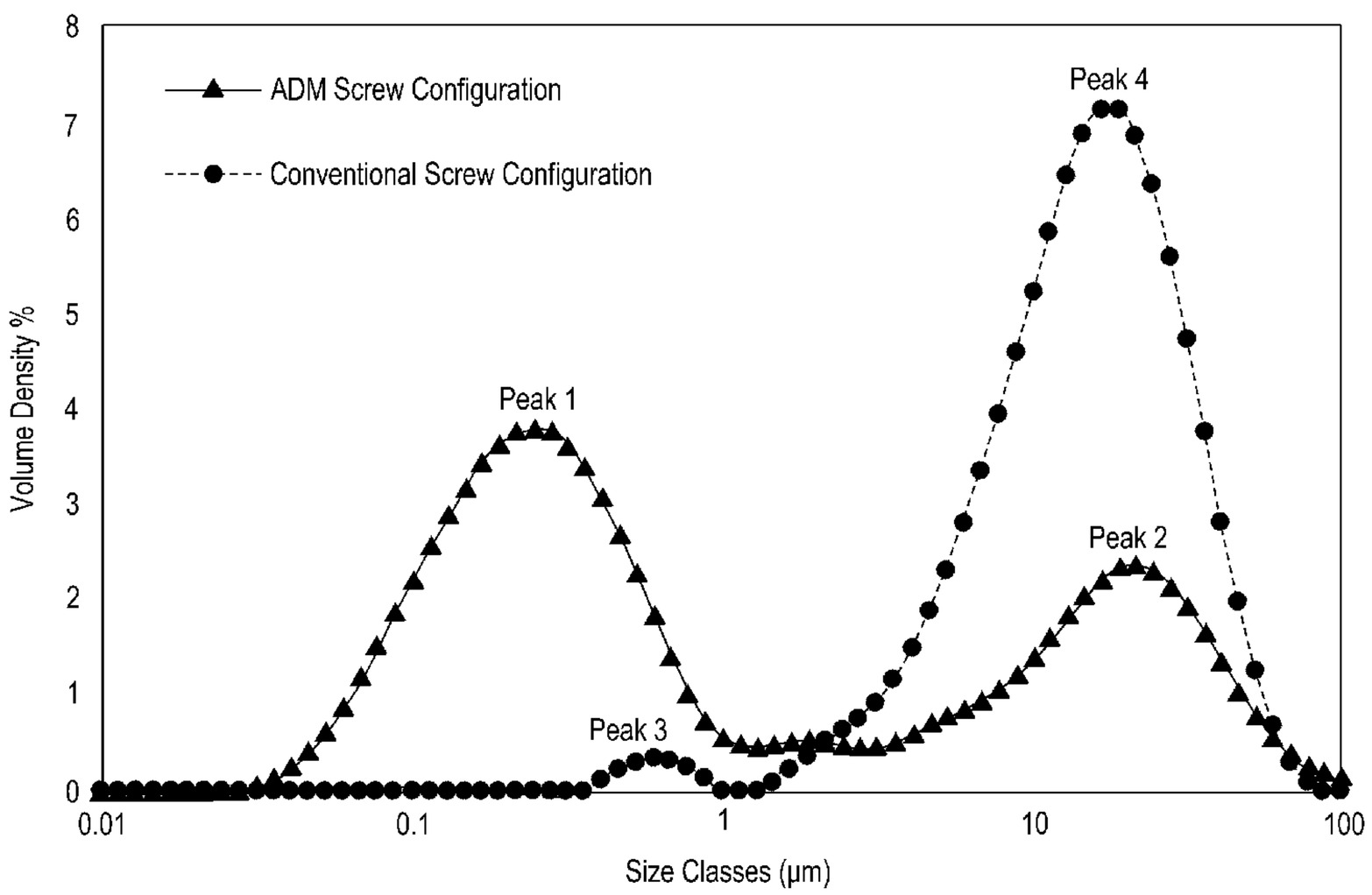

FIG. 8

(57) Abstract: A method of forming an ultra-fine starch/flour product comprises at least one of (a) or (b), wherein (a) comprises heating a mixture of water and native/modified starch/flour, and extruding the mixture with a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw to produce an extrudate. Step (b) comprises forming a mixture of water, a lipid, and native/modified starch/flour, and drying the mixture to produce a dried lipid starch/flour intermediate. The starting starch/flour may be milled prior or after steps (a) or (b). The ultra-fine starch/flour particle product has a higher water solubility as compared to a starch/flour particle product that is produced with a screw configuration devoid of a high shear mixing screw, or a starch/flour intermediate produced in (b) without a lipid. In an embodiment, the method is devoid of chemical or enzyme reaction.

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ULTRA-FINE STARCH OR GRAIN BASED FLOUR COMPOSITION AND RELATED METHODS

FIELD OF THE INVENTION

[01] The present invention relates to a starch or grain-based flour composition and related methods.

BACKGROUND OF THE INVENTION

[02] Starches and grain-based flours are natural occurring ingredients made from agricultural feedstocks. Starch has been refined in industry by grinding, sieving and drying. Native starches occur in crystalline microscopic granules held together by an association of molecules. These granules typically have poor solubility in cold water and high viscosity when gelatinized. These poor solubility and high viscosity characteristics limit the use of native starches and/or require further chemical modification. Because starch is environmentally friendly, starch particulates, and more specifically, starch particles have received commercial interest and have been suggested as a promising ingredient in a variety of fields including foods, beverages, coatings, cosmetics, and pharmaceuticals, as well as various composites as used in food and industrial applications.

[03] Various processes have been proposed for the manufacture of starch particles of submicron particle size. U.S. Patent No. 6,677,386 discloses a chemically reactive extrusion process for preparing biopolymer nanoparticles, in which the biopolymer is plasticized using shear forces and a cross-linking agent is added during processing. This patent discloses that exemplary cross-linking agents are dialdehydes and polyaldehydes, which reversibly form hemiacetals, acid anhydrides and mixed anhydrides (e.g. succinic and acetic anhydride) and the like. The patent discloses that suitable dialdehydes and polyaldehydes are glutaraldehyde, glyoxal, periodate-oxidised carbohydrates, and the like, and that glyoxal is a particularly suitable cross-linking agent. The patent describes ultra-fine starch particles, aqueous dispersions of said particles, and an extrudate prepared by the process.

[04] PCT International Patent Publication No. WO 00/40617 discloses a method for the preparation of starch particles utilizing a two-phase system, wherein the method comprises a) preparing a first phase comprising a dispersion of starch in water, b) preparing a dispersion or emulsion of the first phase in a second liquid phase, with the proviso that the second phase is not water, c) cross-linking of starch present in the first phase, d) separating the starch particles thus

formed. Disclosed examples of cross-linking agents include epichlorohydrin, glyoxal, trisodium trimetaphosphate, phosphoryl chloride or an anhydride of a dibasic or polybasic carboxylic acid.

[05] U.S. Patent No. 9,828,441 discloses a process for preparing an extruded pre-gelatinized, partially hydrolyzed starch utilizing acid in an aqueous environment.

[06] U.S. Patent 9,510,614 discloses a low shear process for processing soluble whole oat flour (whole grain). Enzyme-treated oat flour is prepared by combining a whole oat flour starting mixture and a suitable enzyme solution in a mixer (sometimes called a pre-conditioner) and then heat treating the mixture. The enzyme-treated mixture is then subjected to an extrusion process to gelatinize, hydrolyze, and cook the oat flour mixture. The patent discloses that low shear is applied to mixture in the extruder. The patent discloses that as the enzyme has preconditioned the starch, high shear is not required for the process. The patent discloses that high shear makes it difficult to control the degree of hydrolysis, and can also increase the dough temperature excessively, which can overcook it resulting in too much cooked grain flavour. The patent discloses that a low shear extrusion process is characterized relative to a high shear extrusion by high moisture and low shear screw design versus low moisture and high shear screw design, and that typical screw speeds for the low shear process are 200-350 rpm.

[07] CN102870853 discloses a soybean flour having a particle size of 6.5 μm $\leq$ D < 13 μm. The document states that soybean powder is obtained by pulverizing the soybean, and the ultra-fine soy flour is a soy product obtained by extracting soybean oil after the soybean meal is mainly obtained by pressing and extracting soybean meal, and then being pulverized by airflow. The document states that the ultra-fine soy flour has better solubility and is more easily absorbed and digested by the human body. The document discloses a bean nutrition substitute meal prepared by using ultra-fine soy flour as the main raw material, and that the raw materials are all food grade.

[08] An important limitation is that conventional methods of preparing starch particles for useful applications are complex and require toxic or harmful organic solvents. The products from such conventional processes are typically not perceived as label-friendly for FDA purposes, and typically cannot be characterized with a "clean" label in food and other industrial segments. Other limitations of conventional methods include expensive techniques, which often times require the use of large amounts of solvents and/or high energy. Conventional methods include acidified aqueous techniques that are difficult to control, and wherein the effect of temperature, time,

concentration, acid strength, procedures and devices influence the extent of starch particle modification. Treatment of starches with acid, in turn, necessitates the addition of large amounts of alkali for the neutralization, which, in itself, generates considerable disadvantages and difficulties. Furthermore, acidified methods are not applicable to flours due to the presence of other components such as protein, fiber, and ash. These components complicate the acid modification and negatively affect the quality of products, thus making them unsuitable for commercial applications.

[09] Conventional processes have produced products that lack suitable high stability and water solubility, and often results in phase separation.

[10] In addition to the above challenges faced by industry, there is now an increasing demand for non-chemically or non-enzymatically modified products. There is a need for simple and reliable methods for preparing non-chemically modified cereal/grain-based flours and starches, and in particular, ultra-fine particulates.

SUMMARY

[11] The present invention provides advantages over conventional methods and products. In an aspect, a method of forming ultra-fine (also called submicron) starch or flour particles, comprises mixing a starch or de-germed flour, or a combination thereof, with liquid water or steam, or combination thereof, thus producing a mixture. As used herein, ultra-fine or submicron is used to characterize particles having a diameter of less than one millionth of a meter.

[12] In an aspect, the method of forming an ultra-fine starch or flour product comprises at least one of steps (a) or (b). Step (a) comprises heating a mixture of water and native or modified starch or flour to a temperature in the range of 25° Celsius to less than 200° Celsius, and extruding the mixture with a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw to produce an extrudate. As used herein, modified starch or modified flour means a starch or flour derivative prepared by physically treating a native starch or flour to change its properties.

[13] Step (b) comprises forming a mixture of water, a lipid, and native or modified starch or flour, and drying the mixture of water, lipid, and native or modified starch or flour to produce a dried lipid starch intermediate or dried lipid flour intermediate.

[14] In an aspect, the method comprises at least one of steps (c) or (d). Step (c) comprises, prior to either steps (a) or (b), milling the native or modified starch or flour to reduce particle size of

the native or modified starch or flour. Step (d) comprises breaking apart the extrudate produced in (a), or breaking apart the dried lipid starch intermediate or flour intermediate produced in (b), thus producing an ultra-fine starch or flour particle product with high water solubility as compared to a starch or flour particle product that is produced wherein extruding of the mixture in (a) is with a screw configuration consisting of a low-shear forward conveying screw and devoid of a high-shear mixing screw, or a starch or flour intermediate produced in (b) without a lipid; wherein the method is devoid of chemical or enzyme reaction. In an embodiment, breaking apart the extrudate produced in (a), or breaking apart the dried lipid starch intermediate or flour intermediate produced in (b) is performed by roll pressing, grinding, or milling, and combinations thereof.

[15] In an aspect, the method comprises heating the mixture to a temperature of 25° Celsius to less than 200° Celsius, and extruding the mixture with a screw configuration, thus producing ultra-fine starch particles without chemical or enzyme reaction. In an aspect, the screw configuration comprises in series at least one low-shear forward conveying screw and at least one high-shear mixing screw, in series. In an embodiment, the ultra-fine (i.e., submicron) starch or flour particles have high water stability as compared to starch or flour particles extruded with a screw configuration consisting of a low-shear forward conveying screw and devoid of a high-shear mixing screw. In an aspect, the method is devoid of a pulverizing step.

[16] In an aspect, an apparatus comprises a source of heat and a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw portion, wherein the source of heat is configured to heat a mixture of a starch or de-germed flour or a combination thereof, with water to a temperature of 25° Celsius to less than 200° Celsius, wherein the screw configuration is configured to extrude the mixture to produce ultra-fine starch particles without chemical or enzyme reaction, as compared to starch or flour particles extruded with a screw configuration consisting of a low-shear forward conveying screw and devoid of a high-shear mixing screw. In an aspect, the apparatus is devoid of pulverizing apparatus.

[17] In an aspect, an ultra-fine starch or grain-based particle extruded product comprises ultra-fine starch or grain-based particles characterized by a peak size of around 0.12 μm at a volume density of about 4%, wherein the extruded product is preferably devoid of chemical or enzyme reactants.

[18] In an aspect, an ultra-fine starch or grain-based particle extruded product comprises ultra-fine starch or grain-based particles characterized by a percent solubility in water in the range of about 75 to 95% of up to at least 48 hours.

[19] In an aspect, a method comprises mixing the extruded ultra-fine starch particles with water to produce an aqueous solution having substantially no phase separation.

[20] In an aspect, a starch or grain-based flour comprises ultra-fine particles having high solubility and stability in aqueous solution.

[21] In an aspect, a starch or grain-based flour comprises ultra-fine particles having high solubility and stability in an oil solution.

[22] In an aspect, an aqueous solution comprises a starch or grain-based flour comprising ultra-fine particles devoid of chemical or enzyme reactants.

[23] In an aspect, a method comprises forming of particulate starch products by the use of mixing a starch with water so as to subject the non-chemically or non-enzymatically modified feed source to mechanical forces and shear. The present invention provides a process by conducting the extrusion at a temperature of 25° Celsius to less than 200° Celsius over the course of the processing, which surprisingly yields product exhibiting a high solubility and may be carried out without the use of any additives. In particular, the process need not be carried out under acid conditions or alkaline conditions or in the presence of chemical additives and/or enzymes.

[24] In an aspect, a method of forming an ultra-fine starch or flour product comprises (a) forming a mixture of water, a lipid, and native or modified starch or flour, and drying the mixture of water, lipid, and native or modified starch or flour to produce a dried lipid starch intermediate or dried lipid flour intermediate; and at least one of steps (b) or (c) wherein (b) is prior to step (a) and comprises milling the native or modified starch or flour to reduce particle size of the native or modified starch or flour; wherein (c) comprises breaking apart the dried lipid starch intermediate or flour intermediate produced in (a), thus producing an ultra-fine starch or flour particle product with high water solubility as compared to a starch or flour intermediate produced in (b) without a lipid. In an embodiment, the method is devoid of chemical or enzyme reaction.

[25] In an aspect, the present invention relates to a novel starch or grain-based flour composition consisting of a unique ultra-fine particulate matter with unique solubility and stability in aqueous systems. The design and use of the process parameters of the present disclosure enable the formation of new and unique starch-based particles. The processes disclosed here yield products and compositions that may be used in a variety of fields including the fields of drugs,

cosmetics, coatings, and polymeric compositions. In particular, the disclosed ultra-fine product compositions and subsequent powder properties can be used in certain food and beverage products with the following improvements and applications:

a. Improved sensory and organoleptic functionality in high moisture food systems.
b. Improved delivery of flavour, oil and micro/macro nutrients due to increased surface area and activity as needed in foods and feeds.
c. Improved texture in certain foods - bakery, crackers, bars, and gluten-free foods, etc., where high solubility and stability imparts to better adhesion and textural functionality.
d. Improved carbohydrate and protein solubility to deliver nutritional functionality in foods and feeds.
e. Improved particulate composition for coating application in paper and improved adhesion as required in products replacing latex and bio-adhesives.

[26] These and other aspects, embodiments, and associated advantages will become apparent from the following Detailed Description.

BRIEF DESCRIPTION OF THE DRAWINGS

[27] FIG. 1 shows a portion of a low shear conveying screw according to aspects of the present invention.

[28] FIG. 2 shows a portion of a high shear mixing screw according to aspects of the present invention.

[29] FIG. 3 shows a portion of two parallel triple flight cone screws according to aspects of the invention.

[30] FIG. 4 shows a portion of two parallel feed screws according to aspects of the invention.

[31] FIG. 5 shows a portion of a forward feeding lobed Shearlock screw according to aspects of the invention.

[32] FIG. 6 shows a portion of a reverse lobed Shearlock screw according to aspects of the invention.

[33] FIG. 7 shows a screw configuration according to aspects of the present invention.

[34] FIG. 8 is a graph of volume density (%) versus size classes (μm), which shows wet particle size distribution of starch particles produced in accordance with aspects of the present invention as compared to wet particle size distribution of native dent corn starch.

[35] FIG. 9 is a graph % solubility versus time, which shows stability by % solubility in real time (RT) for various particles produced in accordance with aspects of the present invention.

[36] FIG. 10 depicts a starch product produced in accordance with aspects of the present invention.

[37] FIG. 11 depicts a flour product produced in accordance with aspects of the present invention showing high solubility in aqueous solution as compared to a conventional flour product in aqueous solution.

[38] FIG. 12 depicts X-ray diffraction (XRD) patterns of corn starch samples made in accordance with aspects of the invention.

DETAILED DESCRIPTION

[39] FIG. 1 shows a portion of a low shear conveying screw 100 according to aspects of the present invention. Low shear conveying screw 100 is located within a tube or pipe (not shown). Low shear conveying screw 100 is used to move or convey materials through a tube or pipe. Low shear conveying screw 100 has a helical surface 102 surrounding a central shaft 104. Helical surface 102 comprises external screw threads 106. Screw threads 106 have equal dimensions and are aligned in the same manner as each adjacent screw thread 106. Shaft surfaces 108 are located between adjacent screw threads 106. As low shear conveying screw 100 is rotated around the axis of central shaft 104, material within the tube or pipe is moved through the tube or pipe by low shear conveying screw 100.

[40] FIG. 2 shows a portion of an exemplary high shear mixing screw 200 according to aspects of the present invention. High shear mixing screw 200 is located within the tube or pipe (not shown), and has asymmetric surface 202, surrounding a central shaft 204. Asymmetric surface 202 comprises screw threads 206, which are offset from each adjacent screw thread 206. As high shear mixing screw 200 is rotate around the axis of central shaft 204, material within the tube or pipe is mixed by high mixing screw 200. In FIG. 2, eight screw threads 206 are shown. However, more or less screw threads 206 may be used in embodiments of the invention.

[41] FIG. 3 shows a portion of two parallel triple flight cone screws 300 according to aspects of the invention.

[42] FIG. 4 shows a portion of two parallel feed screws in combination 400 according to aspects of the invention. FIG. 4 shows two low shear conveying screws depicted in FIG. 1, wherein the screws are aligned so that the threads 106 of one screw align with shaft surfaces 108 between two threads 106 of the other screw.

[43] FIG. 5 shows a portion of a forward feeding lobed Shearlock screw 500 according to aspects of the invention. While conveying threads 106 as shown in FIG. 1 put work into extrudate relatively slowly, paddles (Shearlocks) of lobed Shearlock screw 500 put work into the extrudate more rapidly. Paddle 502 is an oval piece that is a poor conveying element, even when configured as part of a group of elements set to "forward conveying." Forward conveying is lining paddles 502 up so the general direction of the progression of the longest dimension of the paddles (lobe) continue the direction of the conveying elements. Neutral conveying (not shown) is essentially setting the paddles so from one paddle to the next in the profile, the lobes are offset by 90 degrees.

[44] FIG. 6 shows a portion of a reverse feeding lobed Shearlock screw 600 according to aspects of the invention. Reverse conveying is essentially lining up the paddles 602 so the general direction of the progression of the lobes are opposite the direction of the conveying elements.

[45] Those skilled in the art having the benefit of the present disclosure will recognize that paddles 502 (shown in FIG. 5) and paddles 602 (shown in FIG. 6) may be built up in sets equal to 0.5 D in length. Generally, for 0.5 D in length, a block of elements may be offset by 90 degrees, so for the forward and reverse conveying paddles, each paddle may be offset by 30 degrees from the paddle upstream. Those skilled in the art will recognize that one way to look at the direction of conveying of the paddles is to look at the top or bottom of the group of paddles as they would rotate in the extruder. If the "wave" that comes around goes from left to right, the parts are forward conveying (direction of extrudate flow). If the "wave" that comes around is right to left, the parts are reverse conveying (opposite direction of extrudate flow).

[46] The present invention is more particularly illustrated by the examples and comparative examples which follow:

[47] EXAMPLES

[48] Materials

[49] The non-chemical and non-enzymatic modified process disclosed herein may be used to produce the unique ultra-fine starch particulates from starch or de-germed flour, or a combination thereof, and water or steam, or a combination thereof. An exemplary, but not limiting, dent corn starch is ADM 106 (Archer Daniels Midland). An exemplary de-germed flour is de-germed corn flour. The starch of de-germed flour may be derived from a plant source selected from the group consisting of corn, wheat, peas, rice, tapioca, potatoes and other cereal grains such as rye, barley, and oat as well as from certain legumes such as soybeans, peanuts, and combinations thereof.

[50] Mixing Process

[51] Native starch is subjected to mixing with water with sufficient shear and gentle heating to achieve a characteristic particle size distribution. The water may be added in the form of steam or liquid water. Over the course of the processing, the temperature is in the range of 25° Celsius (i.e., room temperature, "RT") to less than 200° Celsius, preferably in the range of in the range of 25° Celsius to less than 140° Celsius. The mixing process may be batch or continuous mixing. This starting mixing process preconditions the starch to achieve characteristics, such as moisture content, pH, and temperature, desirable for further processing of the starch.

[52] Example 1

[53] Extrusion Process. Starch particles demonstrating high solubility and stability were produced using a pilot scale TX-57 Magnum co-rotating two screw extruder system (Wenger Manufacturing, Sabetha, KS) that can be fitted with screw shafts and barrels of varying lengths and equipped with water cooling capability and steam heating. A two-screw configuration was used for a screw configuration identified as conventional screw configuration (conveying screws). A two-screw configuration was used for a novel ADM screw configuration identified as ADM screw configuration (a mix of forward and reverse feeding lobed shearlocks, forward cut flight screw, and a shallow flight, cut flight cone). FIG. 7 shows the novel ADM screw configuration that was used for the above extrusion process.

[54] Novel ADM screw configuration, shown as screw configuration 700 in FIG. 7, was used to investigate the effects of higher mechanical shear on the final properties of product. Those skilled in the art having the benefit of the present disclosure will recognize that suitable extruder systems useful for the present invention are not limited to a particular screw variety, and may also include, for example, single screw, ram, or other similar extrusion methods.

[55] As shown in FIG. 7, screw configuration 700 has two screws 702 and 704. Each screw 702, 704. Each screw 702, 704 has a respective first segment 706 comprising forward cut flight, 1d screws, i.e., screws having the screw configuration shown in FIG. 4. Each screw 702, 704 has a respective second segment 708 comprising 4 x 45° forward Shearlock screw configuration shown in FIG. 5. Each screw 702, 704 has a respective third segment 710 comprising forward cut flight, 1d screws, i.e., screws having the screw configuration shown in FIG. 4. cut flight, 1. Each screw 702, 704 has a respective fourth segment 712 comprising a 3 x 45° forward Shearlock screw configuration (similar to the configuration shown in FIG. 5, but with three Shearlocks or paddles instead of four Shearlocks or paddles shown in FIG. 5). Each screw 702, 704 has a respective fifth segment 714 comprising a 3 x 45° reverse Shearlock screw configuration (similar to the configuration shown in FIG. 6, but with three Shearlocks or paddles instead of four Shearlocks or paddles shown in FIG. 6). Each screw 702, 704 has a respective sixth segment 716 comprising a

2 x 45° reverse Shearlock screw configuration. Each screw 702, 704 has a respective seventh segment 718 comprising a 3 x 30° forward Shearlock screw configuration. Each screw 702, 704 has a respective sixth segment 716 comprising a 2 x 45° reverse Shearlock screw configuration. Each screw 702, 704 has a respective seventh segment 718 comprising a 3 x 30° forward Shearlock screw configuration. Each screw 702, 704 has a respective eighth segment 720 comprising a 3 x 45° forward Shearlock screw configuration. Each screw 702, 704 has a respective ninth segment 722 comprising a shallow flight cut flight cone configuration. Segment 722 has the same configuration as parallel tripole flight cone screws 300 as shown in FIG. 300. Zones 1, 2, 3, 4, and 5 (referred to as Barrels 1, 2, 3, 4, and 5 in FIG. 7) have extrusion temperatures as identified in FIG. 7.

[56] Example 2

[57] Formulations. Different formulations which were designed and prepared for the development and evaluation of the extruded flour and starches in accordance with aspects of this disclosure, and these formulations are summarized in Table 1. Trials were run at pH 6. ADM screw configuration listed in Table 1 is shown in FIG. 7 as screw configuration 700. The conventional screw configuration listed in Table 1 is a screw configuration consisting of only conventional conveying screws.

Table 1

Sample	Screw Configuration	Temperature (in degrees C)					% Water Added by weight of starch
		Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	
1	conventional	rt	100	100	120	120	40
2	ADM	rt	100	100	120	120	40
3	conventional	rt	100	100	120	120	20
4	ADM	rt	100	100	120	120	20

Zone 1 is room temperature (rt), i.e., 25°C.

[58] Results

[59] The starch particle distribution was determined for sample 4 (made using ADM screw configuration 700) and for sample 3 (made using a conventional screw configuration consisting of only conventional conveying screws). Sample 4 made in accordance with the present disclosure had a unique starch particle distribution, with >50% of particles in submicron range. See Table 2 below and FIG. 8. As shown in Table 2, sample 4 (made using ADM screw configuration 700) had 67% of its particles with a D90 (μm) of 0.591 (Peak 1), whereas sample 3 (made using a conventional screw configuration consisting of only conventional conveying screws), had only 1.72% of its particles with a D90 (μm) of 0.84 (Peak 3).

Table 2

	Screw Configuration	Frequency %	D90 (μm)
Peak 1	ADM	67	0.591
Peak 2	ADM	33	39.8
Peak 3	Conventional	1.72	0.84
Peak 4	Conventional	98.27	36.67
D90 represents the particle diameter corresponding to 90% cumulative (from 0 to 100%) undersize particle distribution.			

[60] Wet particle size distribution. FIG. 8 is a graph of volume density (%) versus size classes (μm), which shows wet particle size distribution of starch particles produced in accordance with aspects of the present invention (i.e., sample 4 of Table 1 with 20% water added, extruding with novel ADM screw configuration 700) as compared to wet particle size distribution of native dent

corn starch that is extruded using a conventional screw configuration consisting of only conventional conveying screws (i.e., sample 3 of Table 1 with 20% water added). As shown in FIG. 8, ultra-fine starch particles produced with novel ADM screw configuration 700, and devoid of chemical or enzyme reactants, are characterized by a peak size of around 0.12 μm at a volume density of about 4%. As shown in FIG. 8, particles made in accordance with aspects of the present invention, i.e., sample 4 of Table 1, have a much greater volume density % and at a smaller size class (see Peak 1) than that of native dent corn starch (i.e., sample 3 of Table 1) that is extruded using a conventional screw configuration consisting of only conventional conveying screws (Peak 3).

[61] Mixing with water and determining solubility. In a preferred embodiment, the products prepared in accordance with the invention will also be substantially completely cold-water soluble, i.e., soluble in water at 25° C (i.e., room temperature). A method for determining solubility is described below. In accordance with a preferred method for determining cold-water solubility, 4.0 g (dry basis) product is dispersed in 80.0 g of distilled water. After stirring for 10 minutes at 25° C, the slurry is transferred into a 100 mL graduated cylinder and diluted to volume. The graduated cylinder is inverted three times and allowed to sit at 25° C for 12 min. One 20 g aliquot of the supernatant is then transferred to a pre-weighed pan. The pan is then placed on a hot plate to be evaporated to dryness. The pan is then weighed and recorded as a dry sample weight. Solubility is calculated using the following formula: Solubility= [(dry sample weight)/0.8*100]. The product will be deemed high solubility if the solubility is of at least about 70%, and more preferably at least about 80%. The product prepared in accordance with the invention has excellent cold-water solubility and is particularly useful in connection with foods, coatings, cosmetics, pharmaceuticals as well as various composites.

[62] Percent solubility versus time. FIG. 9 is a graph % solubility versus time, which shows stability by % solubility in water at room temperature (RT), i.e., 25°C, for various particles produced in accordance with aspects of the present invention. As shown in FIG. 9, products made in accordance with the present disclosure (samples 2 and 4, i.e., extruding with novel ADM screw configuration 700, see Table 1) have % solubility in water over time that was much greater than corresponding products made using a conventional screw configuration consisting only of conventional conveying screws (samples 1 and 3, see Table 1). Sample 4 had % solubility of over 80% at about 2 hours, and greater than 75% at 48 hours, as compared to corresponding sample 3 that had % solubility of about 40% at about 2 hours, and about 10% at 48 hours. Sample 2 had % solubility of over 60% at about 2 hours, and about 43% at 48 hours, as compared to corresponding sample 1 that had % solubility of less than 30% at about 2 hours, and about 10% at 48 hours.

[63] Example 3

[64] High stability in aqueous solution of starch product. FIG. 10 is a photograph that depicts a starch product 1002 produced in accordance with aspects of the present invention (i.e., sample 4 of Table 1, extruding with novel ADM screw configuration 700) after being combined with water according to the process described above in the heading "mixing with water and determining solubility." As shown in FIG. 10, the starch product 1002 has high stability in aqueous solution, with no phase separation. The photograph of starch product 1002 in water shown in FIG. 10 was taken 24 hours after starch product 1002 was mixed with water.

[65] Example 4

[66] High stability in aqueous solution of flour product. FIG. 11 is a photograph that depicts a flour product 1102 produced in accordance with aspects of the present invention (i.e., extruded with novel ADM screw configuration 700) after being combined with water according to the process described above in the heading "mixing with water and determining solubility," showing high solubility in aqueous solution as compared to a conventional flour product 1104 in aqueous solution. As shown in FIG. 11, a flour product 1102 made in accordance with the present disclosure has high stability in an aqueous solution, with no phase separation, as compared with a conventional flour product 1104 that is extruded using a conventional screw configuration consisting of only conventional conveying screws, which has significant phase separation as depicted in bottom phase 1106 having more solids than upper phase 1108. The photograph of flour product 1102 in water and conventional flour product shown in FIG. 11 was taken 24 hours after each was mixed with water.

[67] Example 5

[68] Starch/Flour and lipid formulation. Aspects of the invention include a starch/flour mixture with lipid formulation.

[69] Composition of lipid formulations.

[70] Aspects of the process include lipid formulation preparation in accordance with the following:

[71] (1) Microemulsion (ME): 5 grams of monoglyceride was added to 5% glycerol solution in DI water and well mixed. 2 grams of soy lecithin was then added to the solution and well mixed. 12 grams of medium chain triglycerides ("MCT") was then added to the solution and well mixed.

[72] (2) Emulsifier Blend (EM): 12.6 grams of monoglyceride was added to 12% glycerol solution and well mixed. 5 grams of soy lecithin was then added to the solution and well mixed.

[73] (3) Palmitic Acid Formulation (PAF): 2.5 grams of monoglyceride was added in 15 grams of DI water and well mixed. 2.5 grams of palmitic acid was then added to the solution and well mixed.

[74] Table 3 identifies the composition of the lipid formulations in % by weight.

Table 3. Composition of lipid formulations

Composition of formulations		
Microemulsion (ME)	Emulsifier Blend (EM)	Palmitic Acid Formulation (PAF)
60% MCT	25% soy lecithin	12.5% palmitic acid
10% soy lecithin	63% monoglyceride	12.5% monoglyceride
25% monoglyceride	12% glycerol solution	75% water
5% glycerol solution		

[75] Mixing starch/flour with lipid formulation preparation. Aspects of the process include a starch/flour mixture with desired lipid formulation preparation in accordance with the following: (1) a 10% dry solids (DS) slurry was made by adding 30 grams DS of the desired starch/flour to deionized (DI) water; (2) for a sample in which a lipid formulation was added, then 6 grams of the desired lipid formulation was added to the slurry; (4) the slurry was dried using a Buchi B290 spray dryer with an inlet temperature of 100° Celsius, an outlet temperature of about 60° Celsius, and a pump speed of 1.1-1.4 mL/min. For spray drying, the liquid sample is pumped to the spray drying nozzle.

[76] Example 6

[77] Milling. A fluidized bed jet mill (Netzsch Condux CGS 10) was utilized to make ultra-fine starch or flours. The starch or flour was introduced into the mill by a volumetric feeder and milled by compressed gas supplied at 6 bars to three grinding nozzles. The particle size may be tuned by adjusting the rotational speed of an internal classifier. At a classifier speed of 14,000 rpm, starches or flours were produced with a D50 of 3-4 μm and a D90 of less than 10 μm (Table 4). The particle size may also be tuned by adjusting the milling time. Milled corn starch-1 and milled corn starch-2 have the same starting material, but milled corn starch-1 was milled for greater milling time than the milling time for milled corn starch-2.

[78] Particle size and surface area of powders. Particle size and surface area of powders were analyzed using Malvern Mastersizer 3000 dry module. Changes in the particle size and surface area of dry powders were monitored. Particle size and surface area are shown in Table 4. As shown in Table 4, the tuneable process technology of producing ultra-fine products in accordance with this disclosure provides increased surface area up to 3,278 m^2/kg by decreasing particle size

of D10 down to 1.42 µm compared to the base material from which it was derived having a surface area of 318 m^2/kg and a particle size D10 of 13.0 µm – see milled rice flour as compared to native rice flour. Ultra-fine products of the present invention have the following improved characteristics as compared to the base material from which it was derived: (i) milled corn starch-1, a decrease in particle size D10 of 79% (1.82/8.80), and an increase in surface area of 7.6 times (3073/401); (ii) milled corn starch-2, a decrease in particle size D10 of 80.7% (1.70/8.8), and an increase in surface area of 4.7 times (1892/401); (iii) milled modified tapioca starch, a decrease in particle size D10 of 80% (1.7/8.67), and an increase in surface area of 5.7 times (3286/573); (iv) milled rice flour, a decrease in particle size of D10 of 89.1% (1.42/13.0), and an increase in surface area of 10.3 times (3278/318). Those skilled in the art, having the benefit of the present disclosure, will recognize that the tuneable process technology of producing ultra-fine products in accordance with this disclosure may provide increased surface area up to 4,000 m^2/kg by decreasing particle size of D10 down to 1 µm compared to the base material from which it was derived. In an embodiment, ultra-fine products in accordance with this disclosure may have a surface area of 100-4,000 m^2/kg and a particle size D10 of 1-200 µm.

Table 4. Particle size and surface area of powders

Sample	D10 (µm)	D50 (µm)	D90 (µm)	Surface Area (m^2/kg)
Milled corn starch 1	1.82	3.84	7.15	3073
Milled corn starch 2	3.71	6.21	9.61	1892
Milled modified tapioca starch	1.70	3.65	6.71	3286
Milled rice flour	1.42	3.29	6.78	3278
Native corn starch	8.80	17.7	55.7	401
Modified tapioca starch	8.67	15.0	25.9	573
Native rice flour	13.0	32.6	153	318

[79] Particle size and surface area of dispersion were analyzed using Malvern Mastersizer 3000 wet module. Effect of different lipid formulation on particle size and surface area of milled material is shown in Table 5. As shown in Table 5, spray drying of milled material with lipid formulation decreased the particle size and increased the surface area compared to when lipid formulation was not present.

Table 5. Particle size and surface area in 10 % soluble solids (S.S) aqueous dispersion

Sample in 10 % S.S aqueous dispersion	D90 (µm)	Surface Area (m^2/kg)

Milled corn starch 1	8.04	1780
Milled corn starch-1 with PAF lipid formulation	10.9	1642
Milled corn starch-1 with ME lipid formulation	10.1	1670
Milled corn starch-1 with EM lipid formulation	11.6	1624
Spray dried milled corn starch 1	22.9	905.8
Spray dried milled corn starch-1 incorporated with PAF lipid formulation	15.9	1099
Milled corn starch 2	11.9	1283
Milled corn starch-2 with PAF lipid formulation	12.5	1252
Milled corn starch-2 with ME lipid formulation	13.9	1277
Milled corn starch-2 with EM lipid formulation	13.1	1248
Spray dried milled corn starch-2	23.4	792.2
Spray dried milled corn starch-2 incorporated with PAF lipid formulation	16.1	1083
Milled modified tapioca starch	6.69	1817
Milled modified tapioca starch with PAF lipid formulation	6.76	1826
Milled modified tapioca starch with ME lipid formulation	7.49	1775
Milled modified tapioca starch with EM lipid formulation	7.06	1833
Spray dried milled modified tapioca starch	20.3	1053
Spray dried milled modified tapioca starch incorporated with PAF lipid formulation	18.6	1190
Milled rice flour	10.4	1635
Milled rice flour with PAF lipid formulation	10.6	1546
Milled rice flour with ME lipid formulation	13.4	1411
Milled rice flour with EM lipid formulation	13.3	1355
Spray dried milled rice flour	24.0	845.7
Spray dried milled rice flour incorporated with PAF lipid formulation	34.5	813.9

[80] Example 7

[81] Color and whiteness of dry powders. Color characterization was analyzed using a colorimeter, i.e., HunterLab ColorFlex EZ. Changes in the whiteness (L*) of dry powders were monitored. Color characteristics are shown in Table 6. As shown in Table 6, a suitable process technology combined with addition of lipid formulation, e.g., milling, spray drying, and PAF formulation, provides products with retained whiteness characteristics compared to the base material from which it is derived.

Table 6. Whiteness of dry powders

Sample	Dry Powder Whiteness (L*)			
	Native	Milled	Spray dried slurry mixed (with no lipid formulation)	Spray dried slurry mixed incorporated with PAF formulation
Modified tapioca starch	95.41	94.20	92.59	92.55
Rice flour	92.71	95.31	92.76	92.29

Corn starch	96.96	96.13	95.88	96.44

[82] Example 8

[83] Color characterization was analyzed using HunterLab ColorFlex EZ. Changes in the whiteness (L*) of 10 % S.S aqueous dispersion due to heating at 60° Celsius in a water bath for 30 min were monitored. Color characteristics are shown in Table 7. As shown in Table 7, a suitable lipid formulation, e.g., PAF formulation, provides protection against loss in whiteness in ultra-fine starch or flour particle products, specifically spray dried milled modified tapioca starch, milled rice flour, spray dried milled rice flour, milled corn starch, spray dried milled corn starch, with the exception of milled modified tapioca starch (without spray drying). As shown in Table 7, a suitable lipid formulation, e.g., PAF formulation, provides protection against loss in whiteness in ultra-fine starch or flour particle products made using spray drying. Ultra-fine starch or flour particle products made with a PAF formulation and spray drying provides increased heat stability as evidenced by reduced whiteness loss over products made with spray drying and without a PAF formulation.

Table 7. Whiteness of 10 % S.S aqueous dispersion

Sample in 10 % S.S aqueous dispersion	**Whiteness before heating (L*)**	**Whiteness after heating (L*)**	**Differences***
Milled modified tapioca starch	83.3	82.5	0.8
Milled modified tapioca starch with PAF lipid formulation	83.7	82.8	0.9
Spray dried milled modified tapioca starch	81.1	79.9	1.2
Spray dried milled modified tapioca starch with PAF lipid formulation	82.0	81.8	0.2
Milled rice flour	84.3	82.3	2.1
Milled rice flour with PAF lipid formulation	85.2	83.4	1.8
Spray dried milled rice flour	83.5	78.3	5.2
Spray dried milled rice flour with PAF lipid formulation	83.7	81.6	2.1
Milled corn starch	86.7	82.2	4.5
Milled corn starch with PAF lipid formulation	88.2	85.0	3.3
Spray dried milled corn starch	81.2	79.4	1.8
Spray dried milled corn starch with PAF lipid formulation	84.3	83.3	1.1

*Differences in whiteness before and after heating

[84] Example 13

[85] Differential Scanning Calorimetery (DSC). The thermal characteristics of dry products were monitored using TA instrument DSC2500. 10 milligrams DS of sample and 30 milligrams of DI water were added in a DSC pan and equilibrated overnight (about 16-20 hours) at room temperature. The DSC parameters were set to 5 °Celsius/min rate from 30 °Celsius to 170 °Celsius. The temperature associated with the gelatinization process, peak temperature, was analyzed on the DSC thermograms using Trios software. Peak temperature characteristics analyzed by DSC are shown in Table 8. As shown in Table 8, milling and addition of lipid formulation, in e.g., PAF formulation, provides improved heat stability as characterized by higher DSC peak temperature compared to the base material where it is derived.

Table 8. Peak temperature characteristics analyzed by DSC

Sample	Peak Temperature (°Celsius)				
	Native	Milled	Dry mixed incorporated with PAF formulation	Freeze dried slurry mixed incorporated with PAF formulation	Spray dried slurry mixed incorporated with PAF formulation
Modified tapioca starch	68.6	72.7	73.8	76.6	77.4
Rice flour	69.3	72.0	ND*	ND*	70.0
Corn starch	67.9	69.5	71.0	71.6	72.1

*ND: not detected

[86] Example 14

[87] X-ray diffraction (XRD) patterns of corn starch samples are shown in FIG. 12. The crystallinity and amylose-lipid complexation characteristics of dry products were monitored using Bruker D8 Advance equipped with a Cu Kα radiation source $\lambda = 1.5406$ Å, operating at 40 kv and 40 mA. The dry products were corn starch or derived from corn starch, with relative crystallinity % (RC) and relative intensity % (RI) at $2\theta=19.8^{o}$ (%) identified as follows: a) native, RC 38%, RI 0%; b) milled, RC 36.9%, RI 4.1%; c) dry mixed incorporated with PAF formulation, RC 35%, RI 6.8%; d) freeze dried slurry mixed incorporated with PAF formulation, RC 38.0%, RI 16.7%; e) spray dried slurry mixed incorporated with PAF formulation, RC 36.8%, RI 17.1%; and f) paste, RC 0%, RI NA. The relative intensities were recorded in a scattering angle range (2θ) of 4.0–34.0° with a scintillation counter at a scanning speed of 0.02° min^{-1} in coupled two theta scanning type. The relative crystallinity (RC) was expressed as a percentage and was calculated from the crystalline (I_c) and amorphous areas (I_a) obtained in each diffraction pattern using the following equation: $RC\ (\%) = (I_c-I_a)/I_c \times 100$.

[88] Paste was made by cooking starting ingredient in deionized water (DI) water at 95 ºCelsius for 30 min and immediately freeze dried. X-ray diffraction of the paste was used as amorphous area (I_a) with RC of 0% (as shown in FIG. 12).

[89] Referring to FIG. 12, peak intensity reflections at 2θ of 15, 17, 18, and 23º are associated to A-type pattern of native crystalline structure. The diffraction peaks at 2θ=19.8º were due to the formation of starch-lipid complex, whereas the peaks at 2θ=21.3º are attributed to the free and not complexed lipids (See Chao, C., Yu, J., Wang, S., Copeland, L., Wang, S., (2017). Mechanisms underlying the formation of complexes between maize starch and lipids. Journal of Agricultural and Food Chemistry 66(1), 272-278).

[90] The relative crystallinity (RC) characteristics analyzed by XRD are shown in Table 9. As shown in Table 9, milling and incorporation with lipid formulation, in e.g., PAF formulation, kept the granular integrity; as native crystallinity of the product was retained above 88% compared to the base material where it is derived.

Table 9. Relative crystallinity characteristics analyzed by XRD

	Relative Crystallinity (RC, %)				
Sample	Native	Milled	Dry mixed incorporated with PAF formulation	Freeze dried slurry mixed incorporated with PAF formulation	Spray dried slurry mixed incorporated with PAF formulation
Milled corn starch 1	38.0	36.9	35.0	38.0	36.8
Milled corn starch 2	38.0	37.0	34.6	37.5	36.6
Modified tapioca starch	36.8	32.6	32.4	32.7	32.8
Rice flour	34.3	30.9	30.5	32.1	36.5

[91] Example 15

[92] X-ray diffraction (XRD). The amount of amylose-lipid complex formation was analyzed from intensity of peak at 2θ=19.8º. Table 10 presents relative intensity at 2θ=19.8º compared to the base material where it is derived. As shown in Table 10, incorporation with lipid formulation, in e.g., PAF formulation, increased the complex formation up to 25 % compared to the base material where it is derived.

Table 10. Relative intensity at 2θ=19.8° characteristics analyzed by XRD

Sample	Relative intensity at 2θ=19.8° (RI, %)			
	Milled	Dry mixed incorporated with PAF formulation	Freeze dried slurry mixed incorporated with PAF formulation	Spray dried slurry mixed incorporated with PAF formulation
Milled corn starch 1	4.1	6.8	16.7	**17.1**
Milled corn starch 2	2.5	2.5	12.6	**14.3**
Modified tapioca starch	8.4	9.2	13.2	**21.3**
Rice flour	1.9	15.7	16.5	**22.6**

[93] Example 16

[94] X-ray diffraction (XRD). The effect of particle size on amount of amylose-lipid complex formation was analyzed from intensity of peak at 2θ=19.8°. Table 11 presents relative intensity at 2θ=19.8° compared to the base material where it is derived. As shown in Table 9, the claimed process technology of producing ultra-fine products enhanced the amylose-lipid complexation even at ambient conditions; whereas smaller particle size product, in e.g., corn starch, presented higher amylose-lipid complex formation in compared to larger particle sizes. As shown in Table 11, increase in moisture content, in e.g., dry mixed vs slurry mixed, resulted in enhanced interaction between lipid formulation and base micronized material thus increasing in amylose-lipid complexation up to 16.7%. As shown in Table 11, increase in drying temperature, in e.g., using spray drying versus freeze drying, resulted in enhanced interaction between lipid formulation and base micronized material thus increasing in amylose-lipid complexation up to 17.1 %.

Table 11. Relative intensity at 2θ=19.8° of corn starches at different particle sizes analyzed by XRD.

Corn starch sample	D50 (μm)	Relative intensity at 2θ=19.8° (RI, %)		
		Dry mixed incorporated with PAF formulation	Freeze dried slurry mixed incorporated with PAF formulation	Spray dried slurry mixed incorporated with PAF formulation
Milled-1	3.84	6.8	16.7	17.1
Milled-2	6.21	2.5	12.6	14.3
Native	17.7	0.0	6.0	8.2

[95] Example 17

[96] Color absorbance. The absorption capacity of products was monitored by absorbed color using spectrophotometric analysis. A 1% w/w solution of dye, in e.g., Brilliant Green, was made

in DI water. 0.1 grams DS of starch/flour and 9.9 grams of DI water were added in a centrifuged tube and well mixed. The tube then centrifuged for 5 minutes at 1000 xg and supernatant was analyzed at 625nm using an Agilent Cary 60 UV-Vis. Relative color absorbance was calculated using equation below: Relative color absorbance (%) = 100 x (absorbance of Brilliant Green-absorbance of sample)/absorbance of Brilliant Green Higher relative color absorbance indicates higher absorption capacity of samples. Relative color absorbance characteristics are shown in Table 12. As shown in Table 12, enhanced adsorption capacity of up to 90% was observed.

Table 12. Relative color absorbance

Sample	Relative color absorbance (%)		
	Native	Milled	Spray dried slurry mixed incorporated with PAF formulation
Modified tapioca starch	87.4	88.9	80.2
Rice flour	80.5	89.5	83.4
Corn starch	4.5	19.8	23.6

[97] Benefits of the present disclosure include:

a. A method of developing heat stable lipid complexes by retaining granular integrity and native crystallinity due to improved interactions resulted from increase in surface areas of lipids and ultrafine starch/flour;
b. A tune-able process technology is disclosed to increase surface area and incorporate lipid formulation while whiteness of product is highly retained;
c. Improved dispersion/opacity was observed due to incorporation of claimed lipid formulation with ultra-fine starch/flour;
d. Retention of crystallinity and granular integrity was observed in the milled and incorporated with lipid formulation product;
e. The higher amylose-lipid complex making ability was observed for the starch/flour with lower particle size (as determined using XRD); and
f. The disclosed technology enables formation of amylose-lipid inclusion complex with easily oxidizable lipids and heat sensitive ingredients such as flavors, colors, and botanical extracts.

[98] The present invention provides selection of simple, clean and cost-effective processes and conditions to produce a variety of ultra-fine starch/flour particles exhibiting water solubility of greater than 75% and stability to at least about a 48-hour time span. These stabilities and solubilities of the ultra-fine starch/flour particles of the present invention exceed those typical of

conventional products in the market. The ultra-fine starch/flour particles of the present invention provide an improved utility in food and industrial applications not attainable by conventional products. Those of skill in the art having the benefit of the present disclosure will recognize that the unique ultra-fine (also called submicron) starch/flour particles and products, compositions, and powder formulations disclosed herein provide the following benefits:

a. Use of ultra-fine starch particles produced using a simple, cost effective, non-chemically modified process as a bulking agent for certain food applications such as dry mixes, sweeteners, etc.
b. Improved sensory and organoleptic functionality in food systems, such as bakery filings and icing, cereal bars, extruded snacks, margarines, low fat spreads, shortenings, confectionary, certain high moisture foods-like sour cream, yogurt, cheese, processed cheese, and beverages.
c. Use as a carrier for flavors, micro/macro nutrients, enzymes and dietary supplements.
d. Improved texture delivery in foods where a range of solubility and stability can be dialed in for improved adhesion and building desirable textures such as crisp, crunch, etc. that is important for end user's eating experience.
e. Improved carbohydrate and protein solubility for favorable nutritional functionality in foods and feeds.
f. Improved particulate composition for coating application in industrial, cosmetics, paper and improved adhesion.

[99] Color absorbance. Those skilled in the art will having the benefit of the present disclosure will recognize that the method of present invention provides novel ultra-fine starch particles useful for food applications, as carriers, and in coatings applications.

[100] This disclosure has been described with reference to certain exemplary embodiments, compositions, and uses thereof. However, it will be recognized by those of ordinary skill in the art that various substitutions, modifications, or combinations of any of the exemplary embodiments may be made without departing from the spirit and scope of the disclosure. Thus, the disclosure is not limited by the description of the exemplary embodiments, but rather by the appended claims as originally filed.

WHAT IS CLAIMED IS:

1. A method of forming an ultra-fine starch or flour product comprising:

at least one of steps (a) or (b),

(a) heating a mixture of water and native or modified starch or flour to a temperature in the range of 25° Celsius to less than 200° Celsius, extruding the mixture with a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw to produce an extrudate; or

(b) forming a mixture of water, a lipid, and native or modified starch or flour, and drying the mixture of water, lipid, and native or modified starch or flour to produce a dried lipid starch intermediate or dried lipid flour intermediate; and

at least one of steps (c) or (d),

(c) prior to either steps (a) or (b), milling the native or modified starch or flour to reduce particle size of the native or modified starch or flour; or

(d) breaking apart the extrudate produced in (a), or breaking apart the dried lipid starch intermediate or flour intermediate produced in (b),

thus producing an ultra-fine starch or flour particle product with high water solubility as compared to a starch or flour particle product that is produced wherein extruding of the mixture in (a) is with a screw configuration consisting of a low-shear forward conveying screw and devoid of a high-shear mixing screw, or a starch or flour intermediate produced in (b) without a lipid;

wherein the method is devoid of chemical or enzyme reaction.

2. The method of claim 1 wherein the particle size of the ultra-fine starch or flour particle product is 1-200 μm and a surface area of 100-4,000 m^2/kg.

3. The method of claim 1 wherein the whiteness of the ultra-fine starch or flour particle product is 97 or less in L* colormetric scale.

4. The method of claim 1 wherein the gelatinization temperature of the ultra-fine starch or flour particle product is in the range of 65 to 80 °Celsius.

5. The method of claim 1 wherein, using step (a), the ultra-fine starch or flour particle product retains crystallinity in the range of 85 to 98% compared to the native or modified starch or flour from which it was derived.

6. The method of claim 1 wherein, using step (b), the ultra-fine starch or flour particle product with lipid retains crystallinity in the range of 85 to 100% compared to the native or modified starch or flour from which it was derived.

7. The method of claim 1 wherein, using step (b), the ultra-fine starch or flour particle product with lipid has an amylose-lipid complex formation that is at least 25% greater as compared to the base material from which it was derived.

8. The method of claim 1 wherein the ultra-fine starch or flour particle product has enhanced absorption capacity of up to 90%.

9. The method of claim 1 wherein the *in vitro* digestibility of the ultra-fine starch or flour particle product is in the range of 10 to 80 %.

10. The method of claim 1 wherein the starting native or modified starch or flour contains a starch content of at least 30% by weight and is selected from the group consisting of maize, wheat, barely, rice, potato, tapioca, waxy tapioca, peas, fava beans, and lentil.

11. The method of claim 1 wherein, using step (b), the lipid comprises a fatty acid or corresponding mono-, di-, or triglycerides with a chain length of 6 to 22.

12. The method of claim 11 wherein the chain length is 10 to 18.

13. The method of claim 11 wherein the chain length is 12 to 16.

14. A method of making a food product comprising incorporating the ultra-fine starch or flour particle product made in accordance with claim 1, wherein the food product is selected form the group consisting of a soup product, a dairy product, a processed meat product, a yogurt product, a dressing product, a frozen food product, a juice product, a confectionary product, and a bakery product.

15. A method of forming ultra-fine starch or flour particle product, comprising:

(a) mixing a starch or de-germed flour, or a combination of any thereof with water, thus producing a mixture;

(b) heating the mixture to a temperature in the range of 25° Celsius to less than 200° Celsius;

(c) extruding the mixture with a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw, thus producing an extrudate; and

(d) breaking apart the extrudate, thus producing an ultra-fine starch or flour particle product with high water solubility as compared to a starch or flour particle product that is produced wherein the extruding of the mixture is with a screw configuration consisting of a low-shear forward conveying screw and devoid of a high-shear mixing screw;

wherein the method is devoid of chemical or enzyme reaction.

16. The method of claim 15 wherein the starch or de-germed flour is selected from the group consisting of corn, wheat, barley, rice, potato, tapioca, waxy tapioca, peas, fava beans, and lentil.

17. The method of claim 15wherein the at least one low-shear conveying screw is a forward cut flight screw, and the at least one high-shear mixing screw is a forward Shearlock screw.

18. The method of claim 15 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw, at least one high-shear mixing screw, and a second low-shear forward conveying screw.

19. The method of claim 15 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw, a first high-shear mixing screw, a second low-shear forward conveying screw, and a second high-shear mixing screw, the second high-shear mixing screw selected from the group consisting of a forward Shearlock screw, a reverse Shearlock screw, and combinations thereof.

20. The method of claim 15 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw comprising a forward cut flight screw, a first high-shear mixing screw comprising a forward Shearlock screw, a second low-shear forward conveying screw comprising a forward cut flight screw, a second high-shear mixing screw comprising a forward Shearlock screw, a third high-shear mixing screw comprising a reverse Shearlock screw, a fourth high-shear mixing screw comprising a reverse Shearlock screw, a fifth high-shear mixing screw comprising a forward Shearlock screw, and a sixth high-shear mixing screw comprising a forward Shearlock screw,

wherein the first, second, third, fourth, fifth and sixth high-shear mixing screws are each selected from the group consisting of a 4 x 45° forward Shearlock screw, a 3 x 45° forward Shearlock screw, a 3 x 45° reverse Shearlock screw, a 2 x 45° reverse Shearlock screw, a 3 x 30° forward Shearlock screw, and combinations thereof.

21. The method of claim 15 wherein

the first high shear mixing screw is a 4 x 45° forward Shearlock screw;

the second-high shear mixing screw is a 3 x 45° forward Shearlock screw;

the third high shear mixing screw is a 3 x 45° reverse Shearlock screw;

the fourth high shear mixing screw is a 2 x 45° reverse Shearlock screw;

the fifth high shear mixing screw is a 3 x 30° forward Shearlock screw; and

the sixth high shear mixing screw is a 3 x 45° forward Shearlock screw.

22. The method of claim 20 further comprising a shallow flight, cut flight cone screw following the sixth high shear mixing screw in series.

23. The method of claim 21 further comprising a shallow flight, cut flight cone screw following the sixth high shear mixing screw in series.

24. The method of claim 15 wherein the screw configuration comprising a first screw configuration and a second screw configuration, wherein the first screw configuration is parallel to the second screw configuration.

25. The method of claim 15 wherein the breaking apart the extrudate comprises applying pressure to the extrudate.

26. The method of claim 25 wherein the applying pressure to the extrudate is selected from the group consisting of roll pressing, grinding, or milling, and combinations thereof.

27. An apparatus comprising:

a heater configured to heat a mixture of a starch or de-germed flour, or a combination thereof, with water to a temperature in the range of 25° Celsius to less than 200° Celsius;

a screw configuration comprising in series at least one low-shear forward conveying screw and at least one high-shear mixing screw, wherein the screw configuration is configured to extrude the mixture to produce an extrudate; and

at least one of roll press, a grinder, a mill, or a spray dryer configured to break apart the extrudate to produce an ultra-fine starch or flour particle product without chemical or enzyme reaction, wherein the ultra-fine starch or flour particle product has a high water solubility as compared to a starch or flour particle product that is devoid of chemical or enzyme reactants that is produced wherein the screw configuration consists of a low-shear forward conveying screw and is devoid of a high-shear mixing screw.

28. The apparatus of claim 27 wherein the at least one low-shear conveying screw is a forward cut flight screw, and the at least one high-shear mixing screw is a forward Shearlock screw.

29. The apparatus of claim 27 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw, at least one high-shear mixing screw, and a second low-shear forward conveying screw.

30. The apparatus of claim 27 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw, a first high-shear mixing screw, a second low-shear forward conveying screw, and a second high-shear mixing screw, the second high-shear mixing screw selected from the group consisting of a forward Shearlock screw, a reverse Shearlock screw, and combinations thereof.

31. The apparatus of claim 27 wherein the screw configuration comprises, in series, a first low-shear forward conveying screw comprising a forward cut flight screw, a first high-shear mixing screw comprising a forward Shearlock screw, a second low-shear forward conveying

screw comprising a forward cut flight screw, a second high-shear mixing screw comprising a forward Shearlock screw, a third high-shear mixing screw comprising a reverse Shearlock screw, a fourth high-shear mixing screw comprising a reverse Shearlock screw, a fifth high-shear mixing screw comprising a forward Shearlock screw, and a sixth high-shear mixing screw comprising a forward Shearlock screw,

wherein the first, second, third, fourth, fifth and six high-shear mixing screws are each selected from the group consisting of a 4 x 45° forward Shearlock screw, a 3 x 45° forward Shearlock screw, a 3 x 45° reverse Shearlock screw, a 2 x 45° reverse Shearlock screw, a 3 x 30° forward Shearlock screw, and combinations thereof.

32. The apparatus of claim 31 wherein

the first high shear mixing screw is a 4 x 45° forward Shearlock screw;

the second-high shear mixing screw is a 3 x 45° forward Shearlock screw;

the third high shear mixing screw is a 3 x 45° reverse Shearlock screw;

the fourth high shear mixing screw is a 2 x 45° reverse Shearlock screw;

the fifth high shear mixing screw is a 3 x 30° forward Shearlock screw; and

the sixth high shear mixing screw is a 3 x 45° forward Shearlock screw.

33. The apparatus of claim 30 further comprising a shallow flight, cut flight cone screw following the sixth high shear mixing screw in series.

34. The apparatus of claim 32 further comprising a shallow flight, cut flight cone screw following the sixth high shear mixing screw in series.

35. The apparatus of claim 27 wherein the screw configuration comprises a first screw configuration and a second screw configuration, wherein the first screw configuration is parallel to the second screw configuration.

36. An ultra-fine starch or grain-based particle extruded product comprising ultra-fine starch or grain-based particles characterized by a peak size of around 0.12 μm at a volume density of about 4%, wherein the extruded product is devoid of chemical or enzyme reactants.

37. An ultra-fine starch or grain-based particle extruded product comprising ultra-fine starch or grain-based particles characterized by a percent solubility in water in the range of about 75-95% for up to at least 48 hours, wherein the extruded product is devoid of chemical or enzyme reactants.

38. The ultra-fine starch or grain-based particle extruded product of claim 27, wherein the percent solubility in water is in the range of about 85-95% for up to at least 48 hours.

39. A method of forming an ultra-fine starch or flour product comprising:

(a) forming a mixture of water, a lipid, and native or modified starch or flour, and drying the mixture of water, lipid, and native or modified starch or flour to produce a dried lipid starch intermediate or dried lipid flour intermediate; and

at least one of steps (b) or (c),

(b) prior to step (a), milling the native or modified starch or flour to reduce particle size of the native or modified starch or flour; or

(c) breaking apart the dried lipid starch intermediate or flour intermediate produced in (a),

thus, producing an ultra-fine starch or flour particle product with high water solubility as compared to a starch or flour intermediate produced in (b) without a lipid;

wherein the method is devoid of chemical or enzyme reaction.

40. The method of claim 39, wherein in (c) breaking apart the dried lipid starch intermediate or flour intermediate is selected from the group consisting of roll pressing, grinding, or milling, and combinations thereof.


100
106
106
106
104
102
106
106
106

FIG. 1

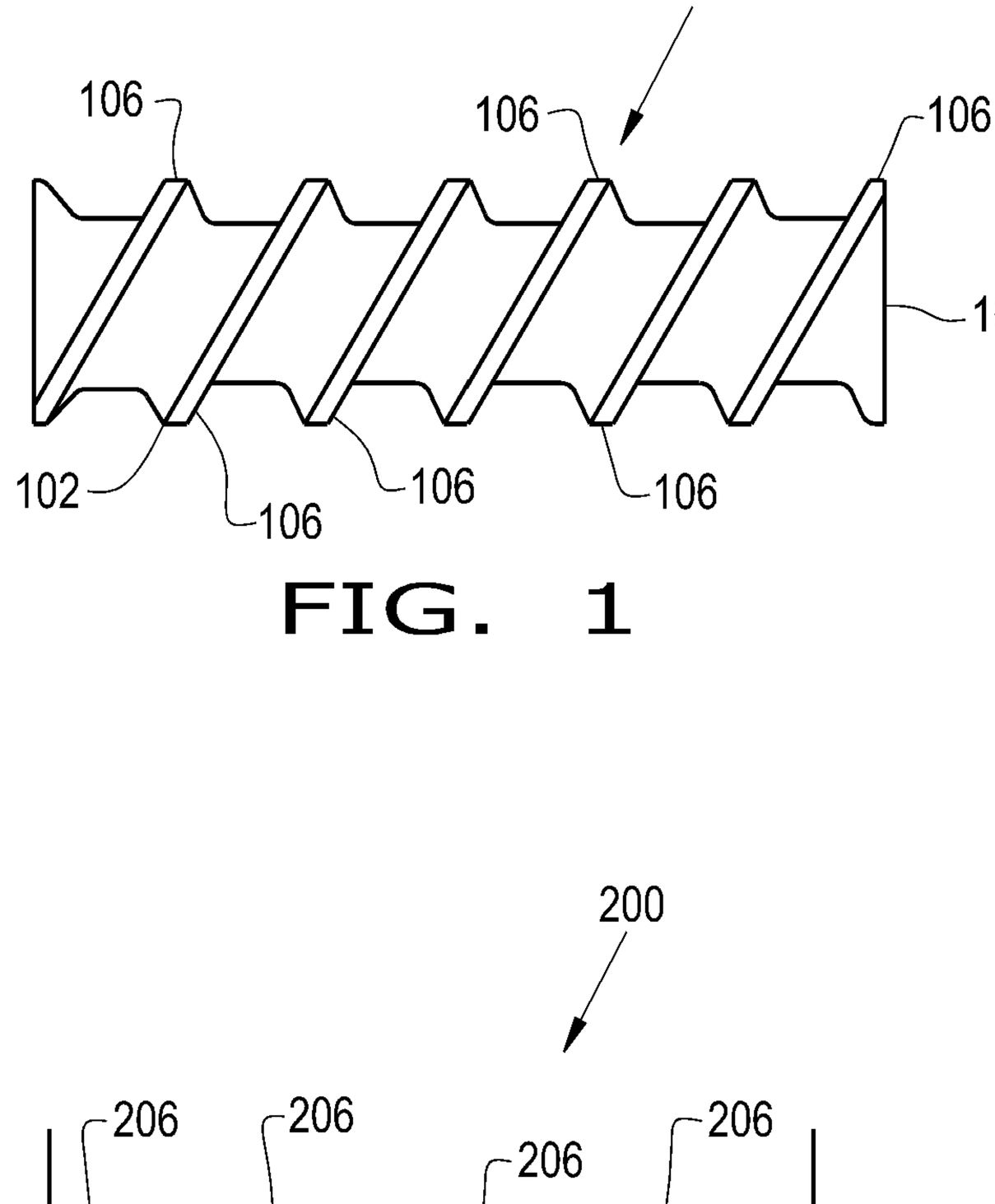
200
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FIG. 2

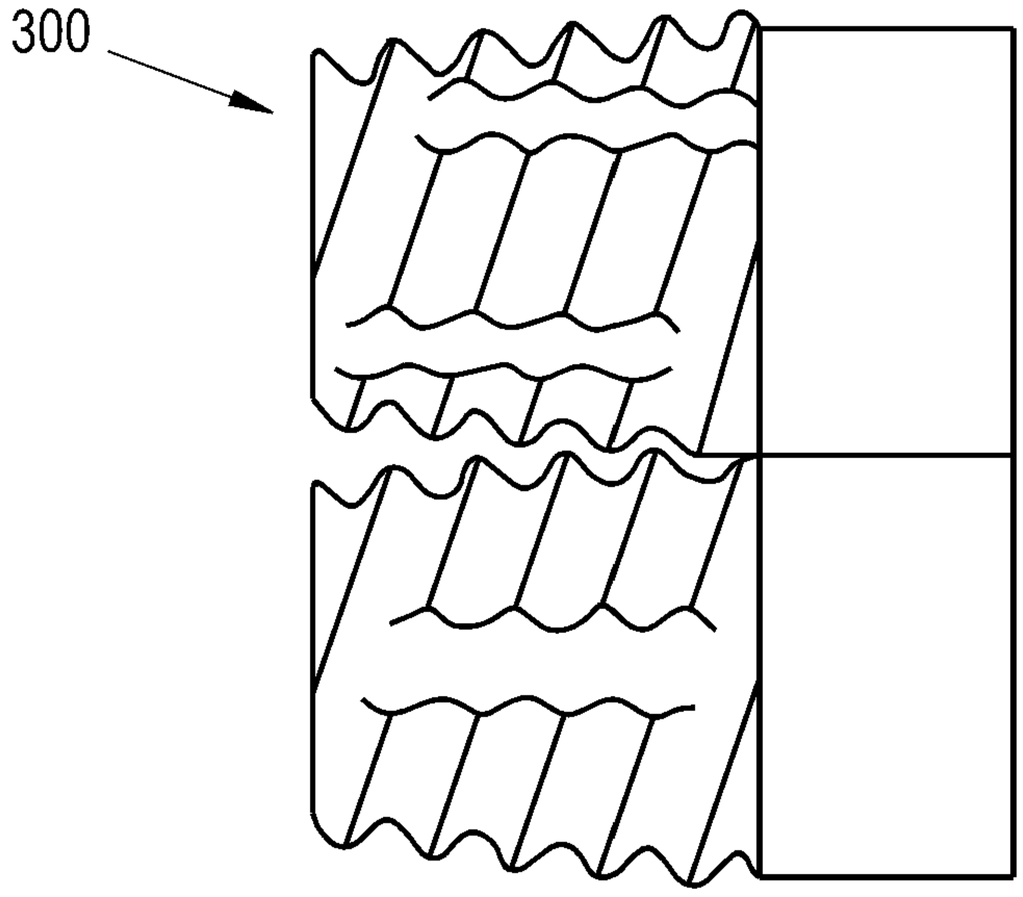

FIG. 3

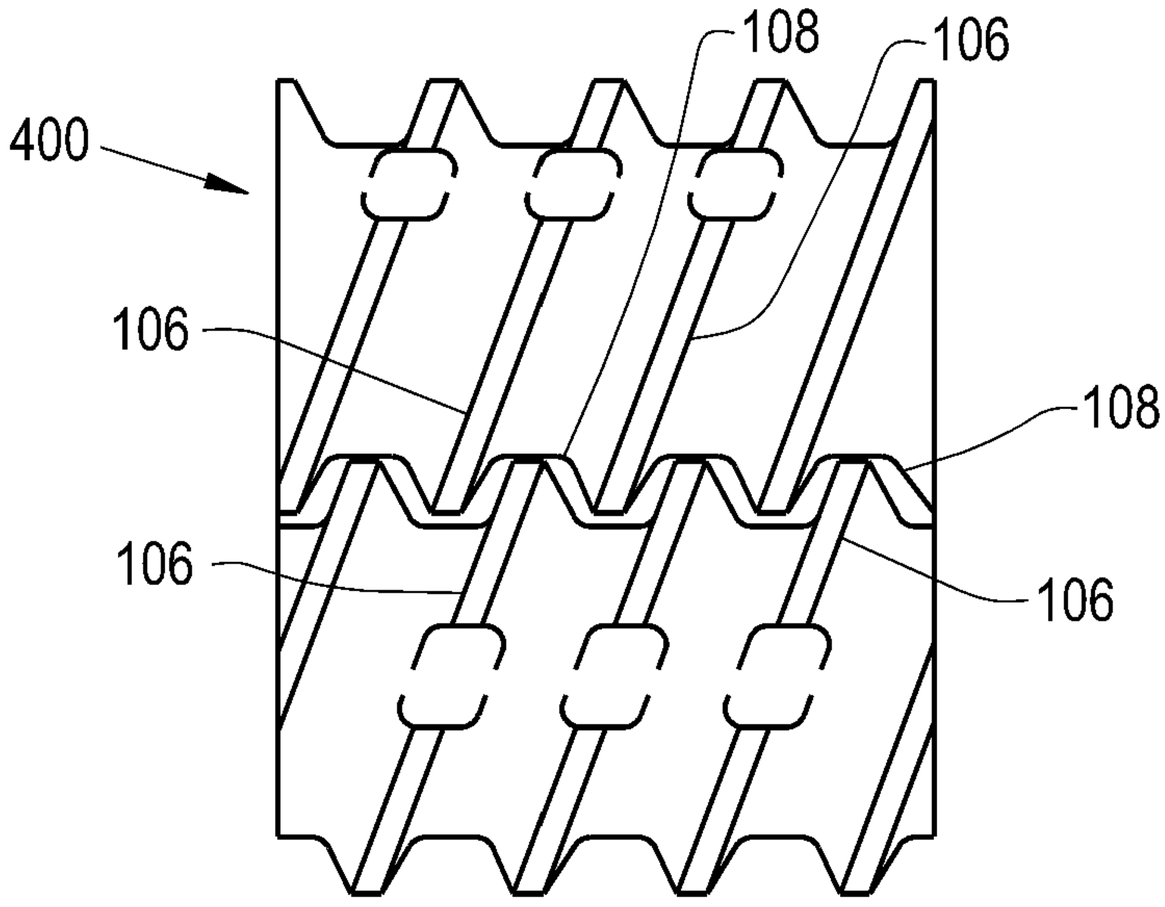

FIG. 4

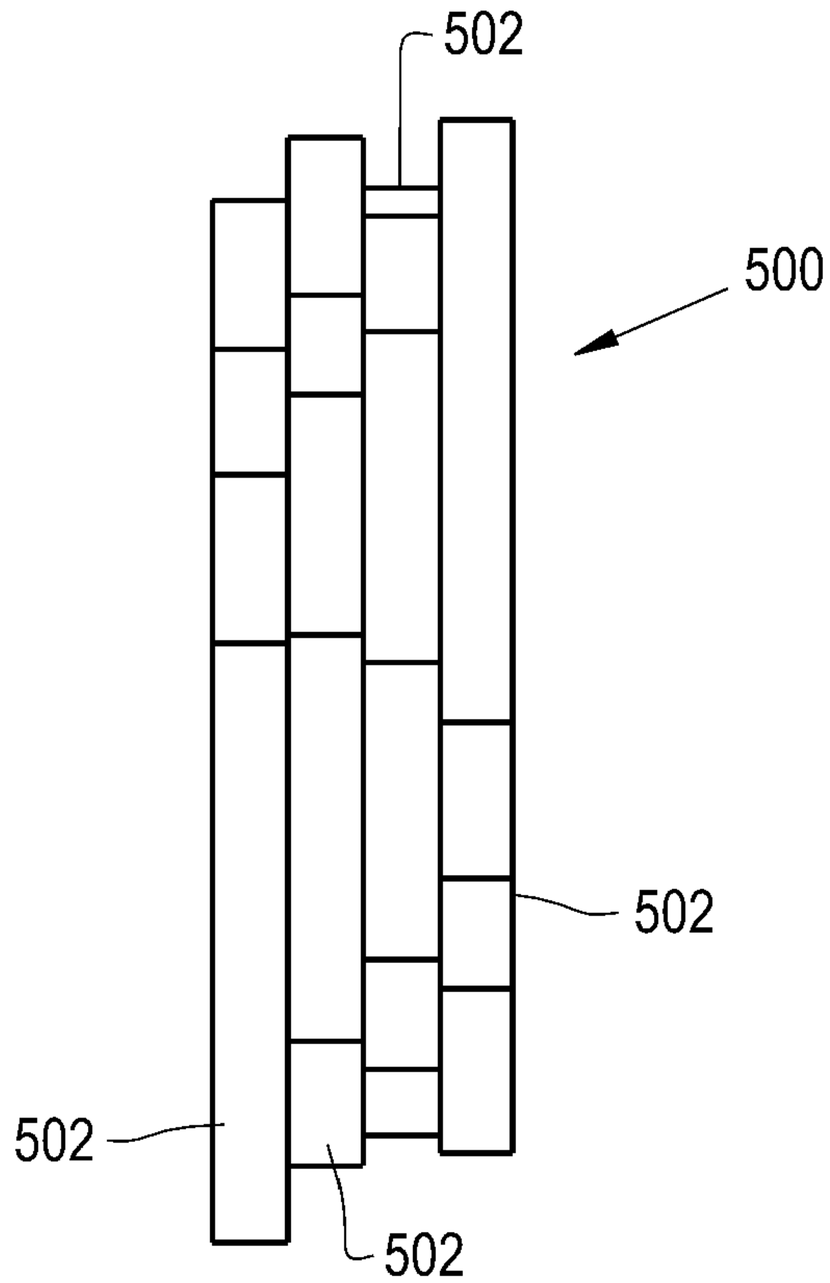

FIG. 5

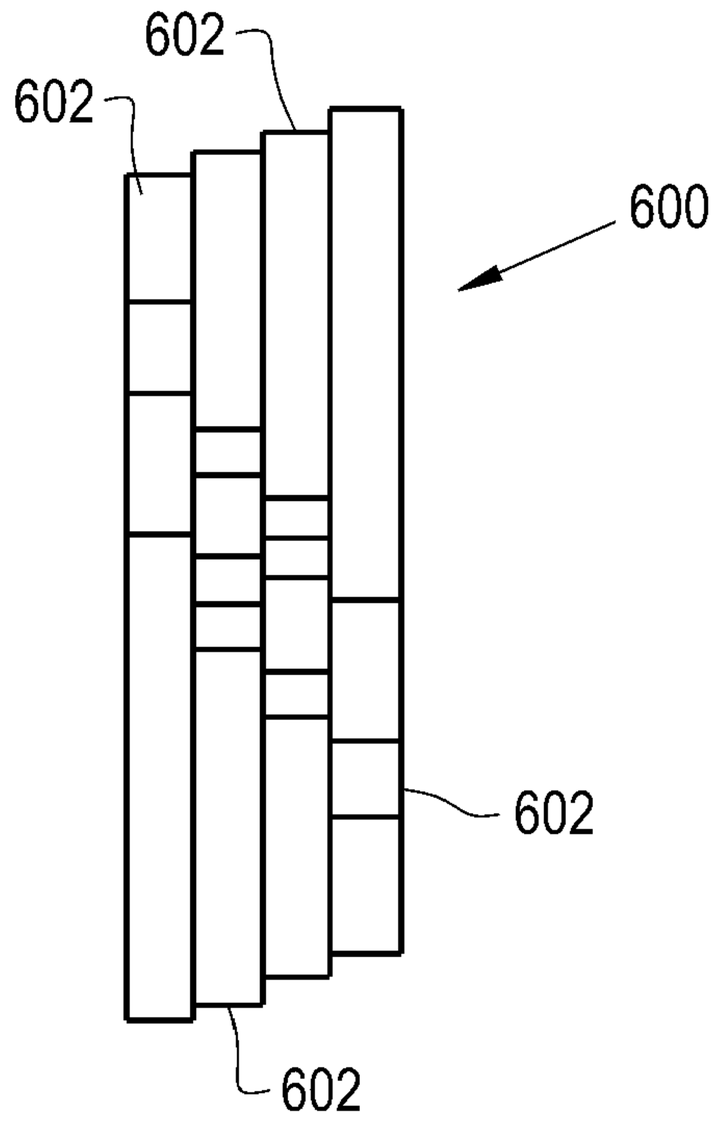

FIG. 6

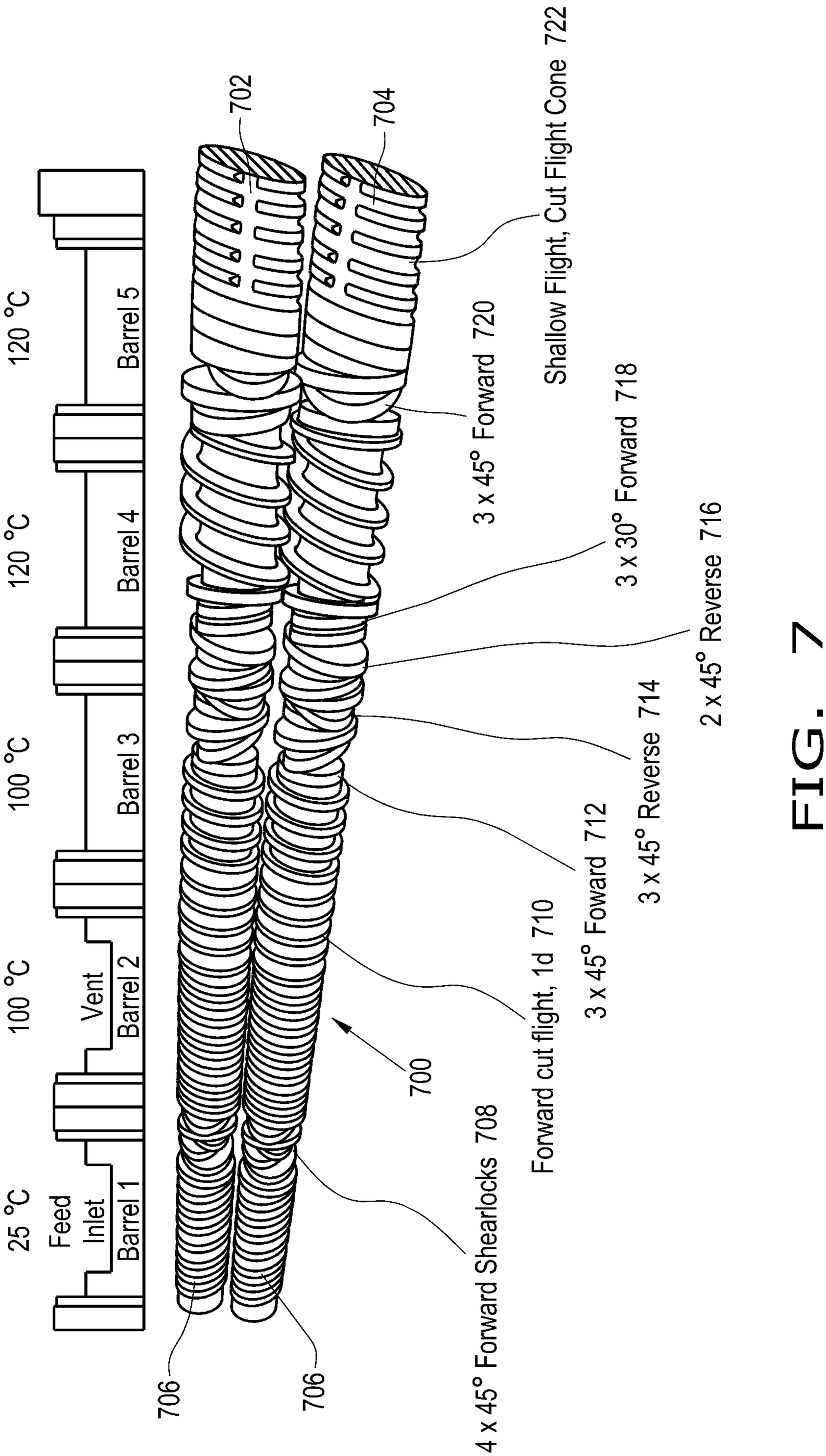
Feed Inlet
Barrel 1
Vent
Barrel 2
Barrel 3
Barrel 4
Barrel 5
25 °C
100 °C
100 °C
100 °C
120 °C
120 °C
702
704
722
Shallow Flight, Cut Flight Cone
720
3 x 45° Forward
718
3 x 30° Forward
716
Reverse
714
2 x 45° Reverse
712
3 x 45° Reverse
710
Foward
3 x 45°
Forward cut flight, 1d
700
708
4 x 45° Forward Shearlocks
706
706

FIG. 7

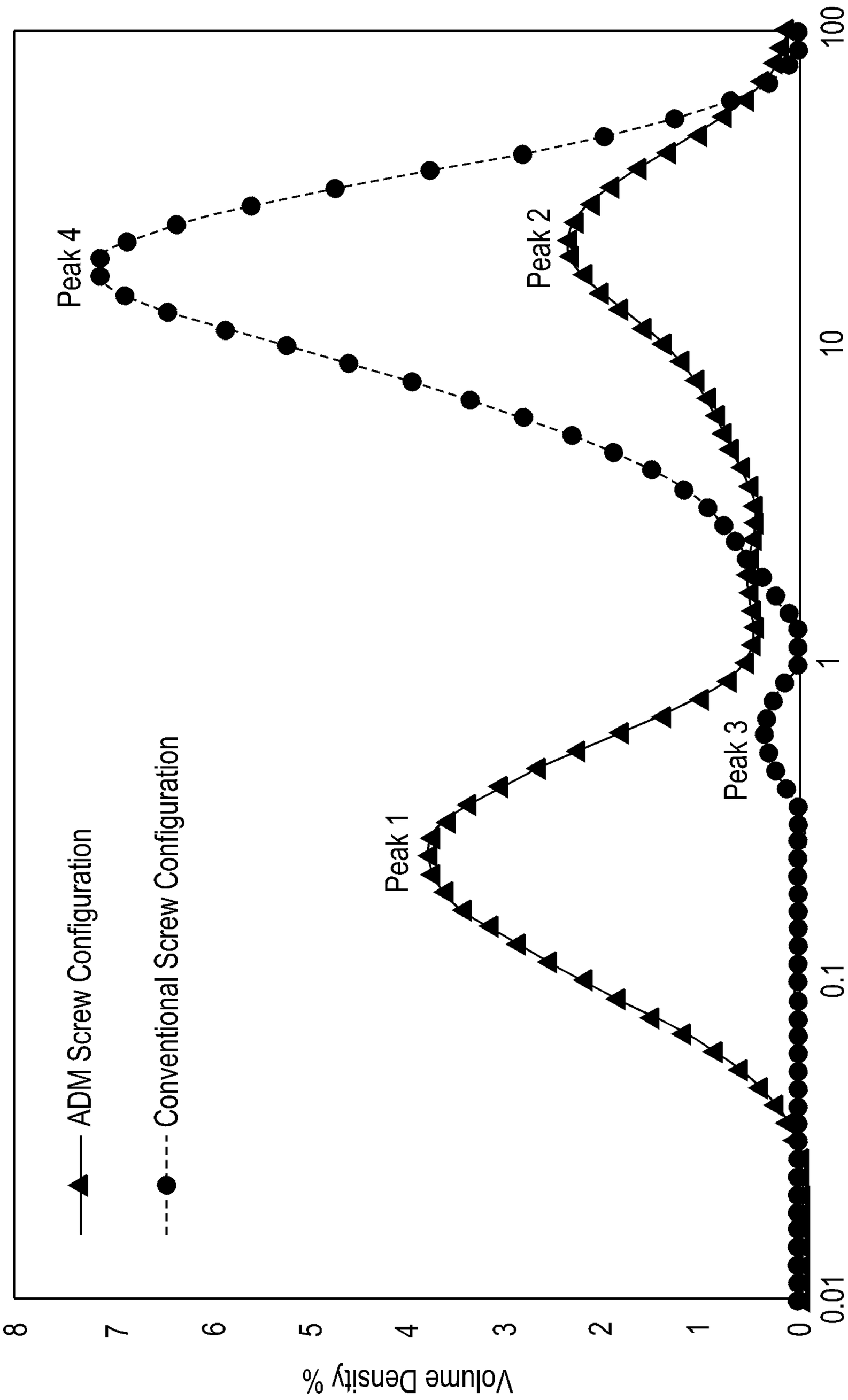
ADM Screw Configuration
Conventional Screw Configuration
Peak 1
Peak 2
Peak 3
Peak 4
100
10
1
0.1
0.01
0
1
2
3
4
5
6
7
8
Size Classes (µm)
Volume Density %

FIG. 8

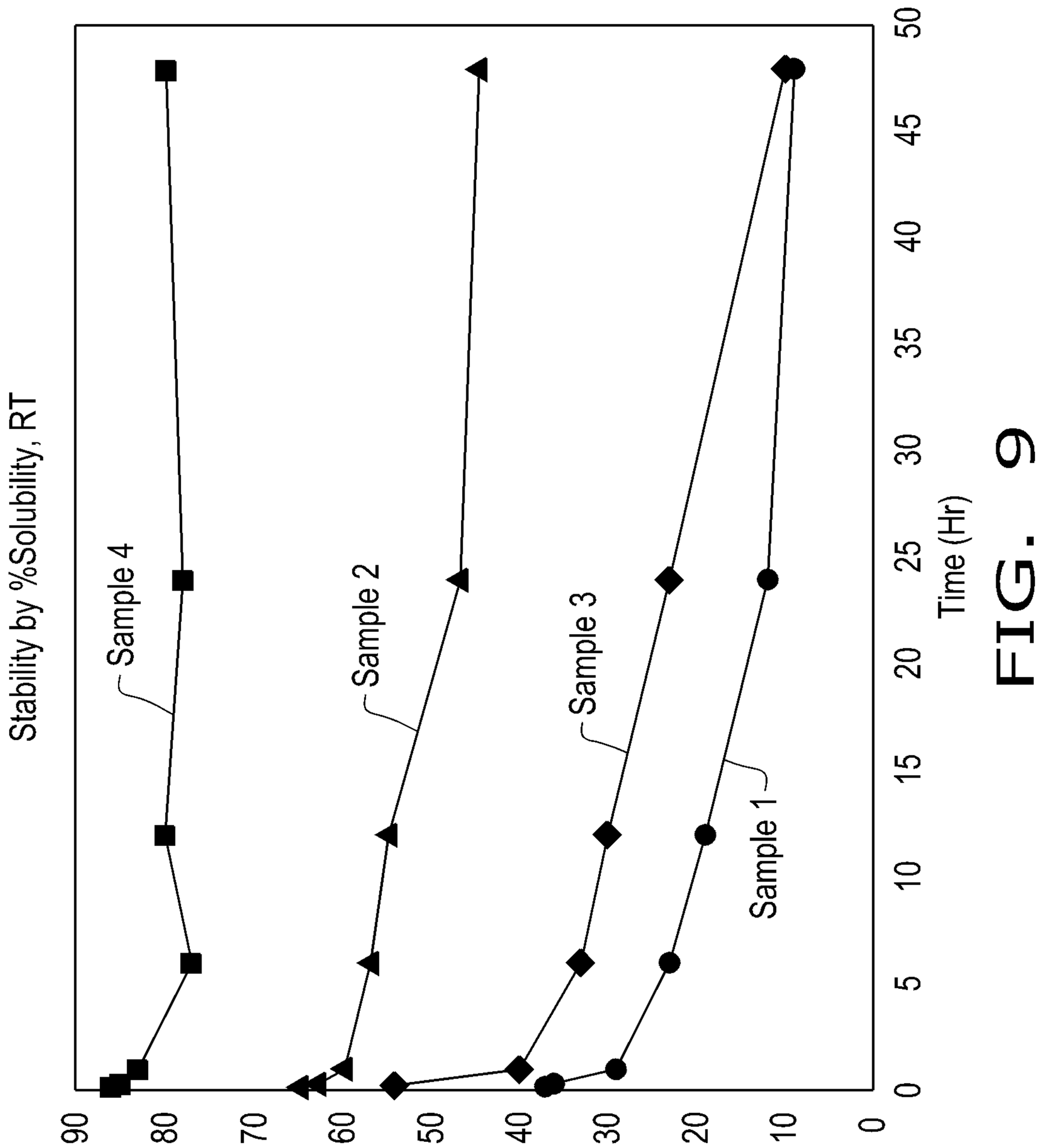
Stability by %Solubility, RT
Sample 4
Sample 2
Sample 3
Sample 1
0
10
20
30
40
50
60
70
80
90
0
5
10
15
20
25
30
35
40
45
50
Time (Hr)
% Solubility

FIG. 9

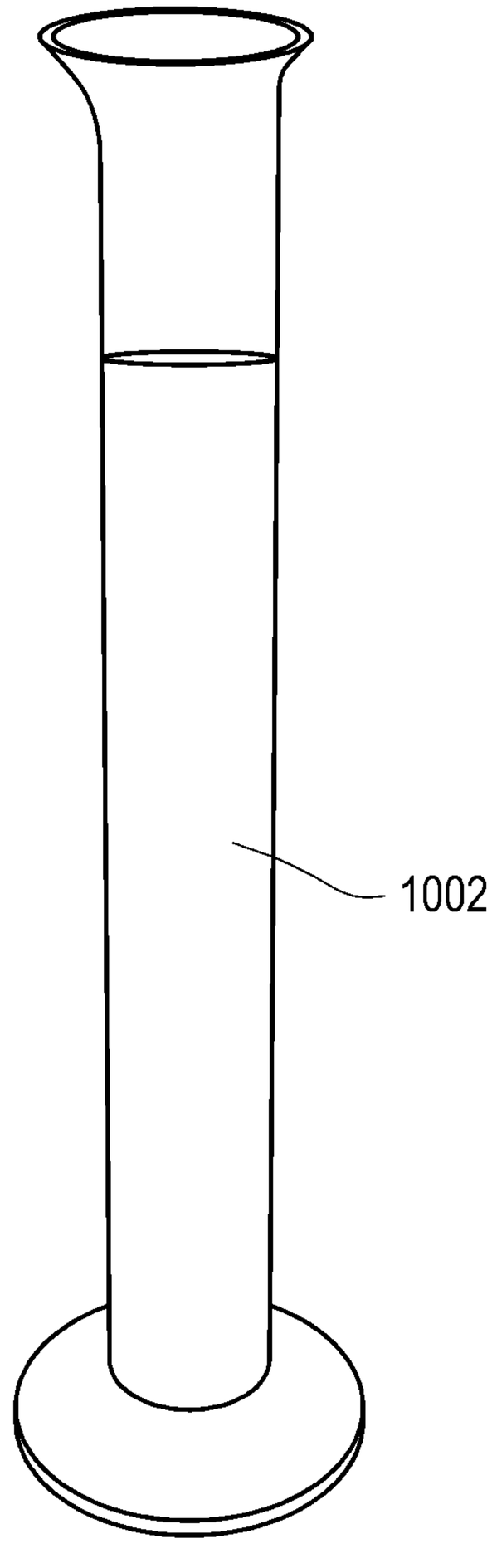

FIG. 10

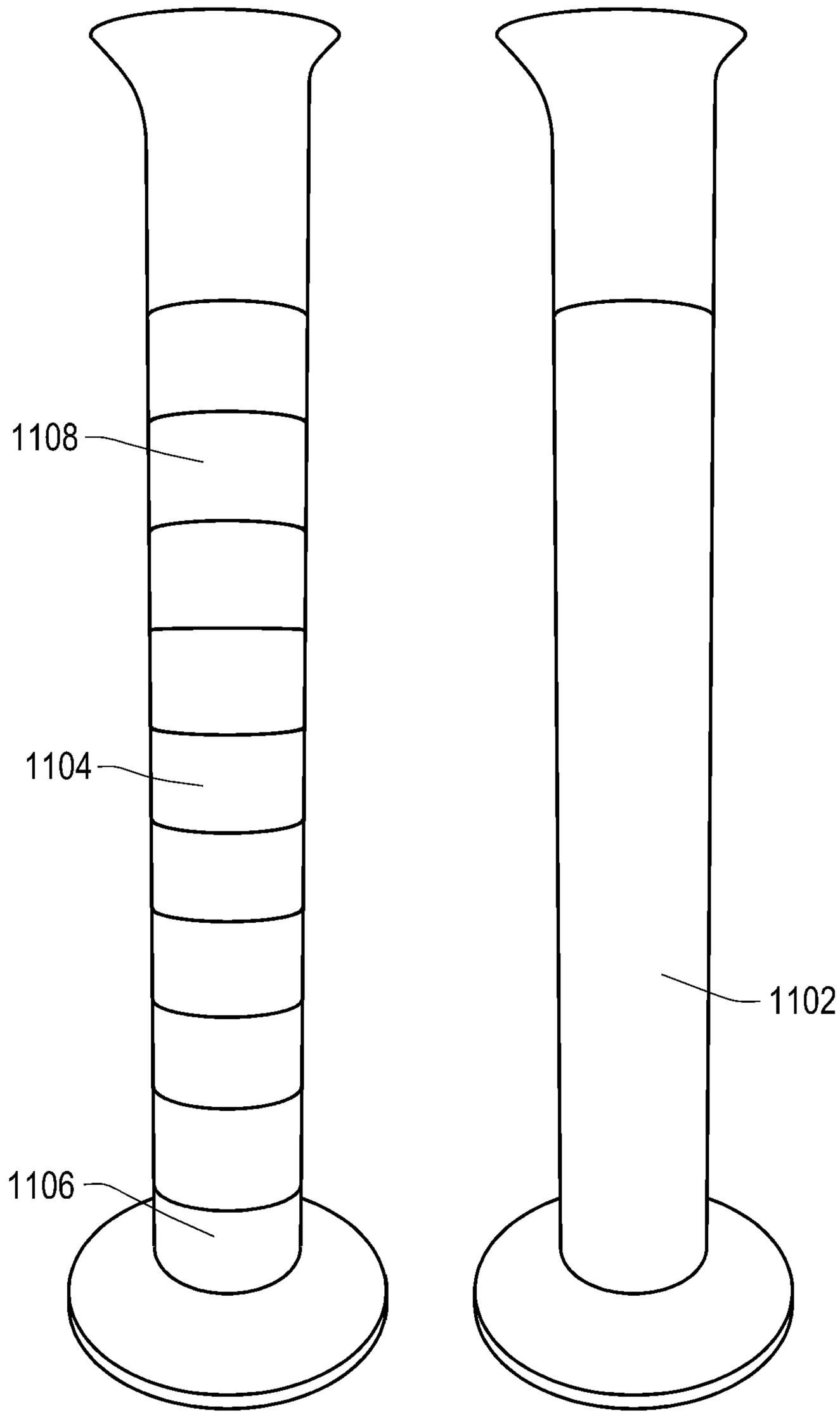

FIG. 11

Corn Starch	RC* (%)	RI** (%)
(a) Native	38.0	0
(b) Milled	36.9	4.1
(c) Dry mixed incorporated with PAF formulation	35.0	6.8
(d) Freeze dried slurry mixed incorporated with PAF formulation	38.0	16.7
(e) Spray dried slurry mixed incorporated with PAF formulation	36.8	17.1
(f) Paste	0	NA

*RC: Relative Crystallinity (%)

**RI: Relative intensity at 20=19.8° (%)

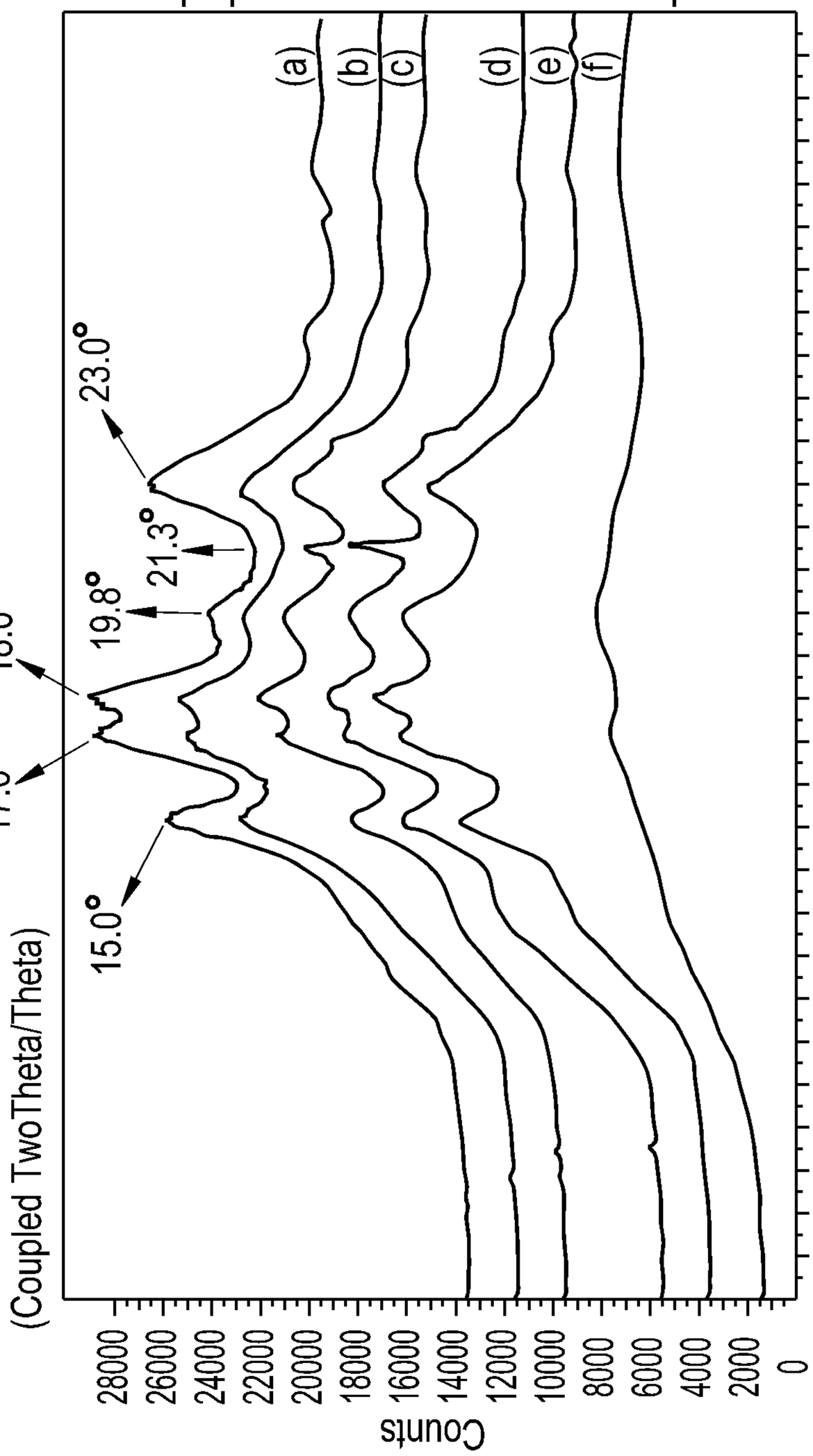

FIG. 12

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2020/064673

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C08B 30/14; A21C 11/16; A23L 29/10; B29B 7/42; C08J 3/12; C08L 3/02 (2021.01)
CPC - C08B 30/14; A21C 11/16; A23L 29/10; B29B 7/42; C08J 3/12; C08L 3/02 (2021.02)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
see Search History document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
see Search History document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
see Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2015/0024113 A1 (GRAIN PROCESSING CORPORATION) 22 January 2015 (22.01.2015) entire document	1-6, 8-19, 24-30, 35, 39, 40
Y	US 4,985,269 A (IRVIN et al) 15 January 1991 (15.01.1991) entire document	1-6, 8-19, 24-30, 35
Y	US 2015/0147442 A1 (GRUPO ALTEX SA DE CV) 28 May 2015 (28.05.2015) entire document	3
Y	GB 1 299 205 A (HAYASHIBARA COMPANY) 13 December 1972 (13.12.1972) entire document	5, 6, 8
Y	MARTENS et al., Amylopectin structure and crystallinity explains variation in digestion kinetics of starches across botanic sources in an in vitro pig model, Journal of Animal Science and Biotechnology, Vol. 9, No. 91, 29 December 2018 [retrieved on 02 February 2021]. Retrieved from the Internet: <URL: https://jasbsci.biomedcentral.com/articles/10.1186/s40104-018-0303-8>. entire document	9
Y	SIMONS, Characterization of edible bean flours: Properties and functionality, North Dakota State University, Ph.D., Thesis, August 2013 [retrieved on 03 February 2021]. Retrieved from the Internet: <URL: https://library.ndsu.edu/ir/bitstream/handle/10365/26923/Characterization%20of%20Edible%20Bean%20Flours%20Properties%20and%20Functionality.pdf?sequence=1>. Pgs. 55-79	17, 19, 28, 30
Y	US 3,443,990 A (DECNOP) 13 May 1969 (13.05.1969) entire document	39, 40
A	US 6,413,567 B1 (DUDACEK et al) 02 July 2002 (02.07.2002) entire document	1-40

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
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"D" document cited by the applicant in the international application
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
04 February 2021	MAR 12 2021
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, VA 22313-1450 Facsimile No. 571-273-8300	Authorized officer Blaine R. Copenheaver Telephone No. PCT Helpdesk: 571-272-4300

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2020/064673

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2011/0170369 A1 (EK et al) 14 July 2011 (14.07.2011) entire document	1-40