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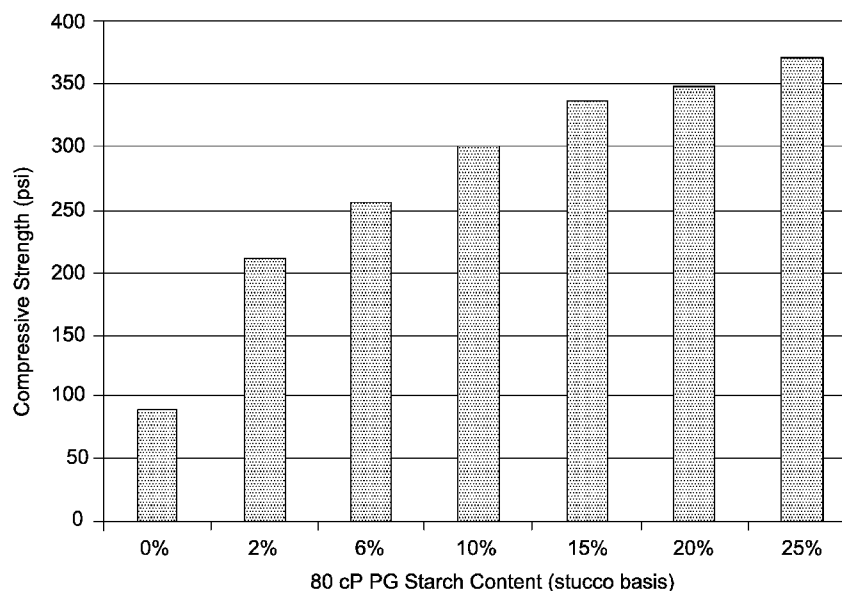


FIG. 4

(57) Abstract: Disclosed is a gypsum board comprising a set gypsum core disposed between two cover sheets. The set gypsum core is formed from a slurry comprising stucco, water, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method described herein. The starch is present in an amount of greater than 10% by weight of the stucco. In preferred embodiments, the board has good strength properties even at lower densities, such as about 31 pcf or less. Also disclosed are a related method of making a gypsum wallboard and a slurry.

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- *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
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GYPSUM WALLBOARD AND RELATED METHODS AND SLURRIES

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This patent application claims the benefit of U.S. Patent Application No. 15/186,147, filed June 17, 2016, which is incorporated by reference.

BACKGROUND

[0002] Starches generally contain two types of polysaccharides (amylose and amylopectin) and are classified as carbohydrates. Some starches are pregelatinized, typically through thermal means. Generally, pregelatinized starches can form dispersions, pastes, or gels with cold water.

[0003] One use for pregelatinized starches is in the preparation of gypsum wallboard. During manufacture of the board, stucco (i.e., calcined gypsum in the form of calcium sulfate hemihydrate and/or calcium sulfate anhydrite), water, starch, and other ingredients as appropriate are mixed, typically in a pin mixer as the term is used in the art. A slurry is formed and discharged from the mixer onto a moving conveyor carrying a cover sheet with one of the skim coats (if present) already applied (often upstream of the mixer). The slurry is spread over the paper (with skim coat optionally included on the paper). Another cover sheet, with or without skim coat, is applied onto the slurry to form the sandwich structure of desired thickness with the aid of, e.g., a forming plate or the like.

[0004] The mixture is cast and allowed to harden to form set (i.e., rehydrated) gypsum by reaction of the calcined gypsum with water to form a matrix of crystalline hydrated gypsum (i.e., calcium sulfate dihydrate). It is the desired hydration of the calcined gypsum that enables the formation of the interlocking matrix of set gypsum crystals, thereby imparting strength to the gypsum structure in the product. Heat is required (e.g., in a kiln) to drive off the remaining free (i.e., unreacted) water to yield a dry product.

[0005] Often, pregelatinized starches add water demand to the process. To compensate for the water demand and allow for sufficient fluidity during manufacture, water content must be added into the stucco slurry. This excess water creates inefficiencies in the manufacture, including increased drying time, slower manufacturing line speeds, and higher energy costs. Reducing the amount of water in the system has proven to be very difficult without

compromising other critical aspects of commercial product, including board weight and strength.

[0006] Another challenge is reducing the weight of gypsum board while maintaining strength. One measure of the strength of board is "nail pull resistance," sometimes simply referred to as "nail pull." To reduce the weight of the board, foaming agent can be introduced into the slurry to form air voids in the final product. Replacing mass with air in the gypsum board envelope reduces weight, but that loss of mass also results in less strength. Compensating for that loss in strength is a significant obstacle in weight reduction efforts in the art.

[0007] It will be appreciated that this background description has been created by the inventors to aid the reader, and is not to be taken as a reference to prior art nor as an indication that any of the indicated problems were themselves appreciated in the art. While the described principles can, in some regards and embodiments, alleviate the problems inherent in other systems, it will be appreciated that the scope of the protected innovation is defined by the attached claims, and not by the ability of the claimed invention to solve any specific problem noted herein.

BRIEF SUMMARY

[0008] In one aspect, the disclosure provides a board (e.g., gypsum wallboard) comprising a set gypsum core disposed between two cover sheets. The set gypsum core is formed from a slurry comprising stucco, water, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method described herein. The starch is present in an amount of greater than 10% by weight of the stucco. In preferred embodiments, the board has good strength properties even at lower densities, such as about 31 pcf or less. For example, at a thickness of one-half inch (about 1.3 cm), the board preferably meets at least one of the following: a compressive strength of at least about 170 psi, a nail pull resistance of at least about 65 lb (about 29 kg), an average core hardness of at least about 11 lb (about 5 kg), an average flexural strength of at least about 36 lb (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, or other strength property described herein. The nail pull resistance, core hardness, and flexural strength are determined in accordance with ASTM standard C473-10, method B.

[0009] In another aspect, the disclosure provides a method of making a gypsum wallboard. The method comprises mixing at least water, stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method. The starch is present in an amount of greater than 10% by weight of the stucco. The method also comprises disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly; cutting the wet assembly into a board; and drying the board. In preferred embodiments, the board has good strength properties even at lower densities. For example, at a thickness of one-half inch (about 1.3 cm), the board preferably meets at least one of the following: a compressive strength of at least about 170 psi, a nail pull resistance of at least about 65 lb (about 29 kg), an average core hardness of at least about 11 lb (about 5 kg), an average flexural strength of at least about 36 lb (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, or other strength property described herein. The nail pull resistance, core hardness, and flexural strength are determined in accordance with ASTM standard C473-10, method B.

[0010] In another aspect, the disclosure provides a slurry comprising water, stucco, and pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method. The starch is present in an amount of greater than 10% by weight of the stucco. When the slurry is cast and dried as board comprising a set gypsum composition, the set gypsum composition comprises a continuous crystalline matrix substantially of calcium sulfate dihydrate. The board can be made at low densities, e.g., a density of about 31 pcf or less, while maintaining good strength properties. In preferred embodiments, and at a thickness of one-half inch (about 1.3 cm), the board meets at least one of the following: a compressive strength of at least about 170 psi, a nail pull resistance of at least about 65 lb (about 29 kg), an average core hardness of at least about 11 lb (about 5 kg), an average flexural strength of at least about 36 lb (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, or other strength property described herein. The nail pull resistance, core hardness, and flexural strength are determined in accordance with ASTM standard C473-10, method B.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0011] FIG. 1 is an amylogram plotting viscosity (left y-axis) and temperature in Celsius (right y-axis) versus time (x-axis) that shows pasting profiles of starches extruded at a

moisture content of 16 wt.% with the solid content of testing slurry being 10 wt.% as set forth in Example 2.

[0012] FIG. 2 is an amylogram plotting viscosity (left y-axis) and temperature in Celsius (right y-axis) versus time (x-axis) that shows pasting profiles of starches extruded at a moisture content of 13 wt.% with the solid content of testing slurry being 10 wt.% as set forth in Example 2.

[0013] FIG. 3 is a graph plotting temperature versus time showing the temperature (in Fahrenheit) rise set (TRS) hydration rate of two slurries containing pregelatinized, partially hydrolyzed starches treated with alum in an amount of 3 wt.% and retarder in amounts of 0.05 wt.% and 0.0625 wt.%, respectively, and a third slurry containing a conventional pregelatinized corn starch having a viscosity of 773 centipoise and retarder in an amount of 0.05 wt.% as set forth in Example 3.

[0014] FIG. 4 is a bar graph of board compressive strength shown at various weight percentages of starch, as set forth in Example 5.

DETAILED DESCRIPTION

[0015] Embodiments of the disclosure provide gypsum board (e.g., gypsum wallboard), methods of preparing gypsum board, and slurries. The gypsum board comprises a set gypsum core disposed between two cover sheets. The core is formed from a slurry comprising stucco, water, and at least one pregelatinized starch having a particular type of mid-range viscosity starch, having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method. In preferred embodiments, the starch is present in an amount of greater than 10% by weight of the stucco (e.g., from about 10 wt.% to about 25 wt.%). Advantageously, the board can be an ultra light weight board, while having sufficient strength properties.

[0016] To achieve the desired mid-range viscosity, particularly a viscosity range of from about 20 cP to about 300 cP, the starch is typically in the form of pregelatinized, partially hydrolyzed starches. Surprisingly and unexpectedly, with such viscosity range, typically with partial hydrolysis, the starch can be added to a slurry at elevated levels for forming the board to achieve increasing board strength (e.g., in an amount of at least 10% by weight of the stucco). The inventors have also found that the pregelatinized and partially hydrolyzed starch demands less water. In some embodiments, the starch is extruded.

[0017] In some embodiments, the starch preferably is prepared by pregelatinizing and acid-modifying starch in a single step in an extruder. See, e.g., US 2015/0010767, which method is incorporated by reference. Pregelatinizing and acid-modifying starch in a single step in an extruder has considerable advantages in comparison to pregelatinizing and acid-modifying starch in separate steps. For example, the method of making pregelatinized, partially hydrolyzed starch allows for a higher board output, faster production, and lower energy costs without sacrificing desired properties (e.g., viscosity, fluidity, cold water solubility, etc.) as described herein.

[0018] In addition, it has been found that the extrusion conditions (e.g., high temperature and high pressure) can significantly increase the acid hydrolysis rate of starch. Surprisingly and unexpectedly, this single step process makes possible using a weak acid, such as alum, and/or smaller amounts of strong acid, for starch acid-modification. Either acid form provides a mechanism where protons from acids catalyze the hydrolysis of starch. Conventional acid-modification processes include purification and neutralization steps. The use of a weak acid (e.g., alum) and/or a small amount of a strong acid avoids the need for any neutralization step and the subsequent purification step typically required in conventional systems to purify the starch of salts resulting from the neutralization step, in accordance with some embodiments of the disclosure.

[0019] The extrusion process, in accordance with embodiments of the disclosure can pregelatinize the starch, and can partially hydrolyze starch molecules in a single step. The hydrolyzation may occur due to hydrolyzation from the extruder and/or may occur due to addition of acid (i.e., acid-modification), as described below. Thus, the extrusion process in one step provides both physical modification (pregelatinization) and chemical modification (acid-modification, partially acid hydrolysis). The pregelatinization provides the ability for the starch to impart strength (e.g., on a final product such as gypsum board). Acid-modification beneficially partially hydrolyzes the starch to low water demand, e.g., in gypsum board manufacturing processes. Thus, the product of methods of preparing starch in accordance with embodiments of the disclosure can be pregelatinized and partially hydrolyzed starch.

[0020] The extrusion desirably can provide a highly efficient acid-modification reaction. The pregelatinization and acid-modification in the extruder occurs at elevated temperatures and/or pressures as described herein and can result in an acid hydrolysis rate that can be, e.g., approximately 30,000 times or greater faster than conventional acid hydrolysis rates at lower

temperatures (e.g., 50°C) and/or pressures. The rate of acid hydrolysis is further increased through the use of low moisture (about 8 wt.% to about 25 wt.%) levels in the starch precursor and hence through an increased concentration of reactants. Because of this high efficiency of acid-modification, the inventors have found that, surprisingly and unexpectedly, a weak acid or a very low level of strong acid can be used in the starch precursor to achieve optimal acid-modification and avoid the need for neutralization and purification which are costly, time consuming, and inefficient requirements of conventional systems.

[0021] In accordance with some embodiments, the hydrolysis is designed to convert the starch into smaller molecules within an optimum size range, which is defined herein by the desired viscosity of the pregelatinized, partially hydrolyzed starch. If the starch is over hydrolyzed, it can be converted into unduly small molecules (e.g., oligosaccharides or sugars), which can result in less board strength than that provided by the pregelatinized, partially hydrolyzed starch of desired viscosity.

[0022] The pregelatinized, partially hydrolyzed starch can be prepared by (i) mixing at least water, non-pregelatinized starch, and an acid to form a wet starch precursor having a moisture content of from about 8 wt.% to about 25 wt.%. The acid can be: (1) a weak acid that substantially avoids chelating calcium ions, (2) a strong acid in an amount of about 0.05 wt.% or less by weight of the starch, or (3) any combination thereof. The wet starch precursor is pregelatinized and acid-modified in one step in an extruder at an elevated die temperature and/or pressure as described herein. The starch is hydrolyzed to a degree that results in a desired viscosity, e.g., as described herein.

[0023] Thus, in some embodiments, a pregelatinized, partially hydrolyzed starch can be made by mixing at least water, non-pregelatinized starch, and a weak acid that substantially avoids chelating calcium ions to make a wet starch precursor having a moisture content of from about 8 wt.% to about 25 wt.%. The wet starch is then fed into an extruder. While in the extruder at a die temperature of about 150°C (about 300°F) to about 210°C (about 410°F), the wet starch is pregelatinized and acid-modified, such that it is at least partially hydrolyzed.

[0024] In further embodiments, a pregelatinized, partially hydrolyzed starch can be made by mixing at least water, non-pregelatinized starch, and a strong acid to make a wet starch precursor having a moisture content of from about 8 wt.% to about 25 wt.%, wherein the strong acid is in an amount of about 0.05 wt.% or less by weight of the starch. The wet starch is then fed into an extruder. While in the extruder at a die temperature of about 150°C (about

300°F) to about 210°C (about 410°F), the wet starch is pregelatinized and acid-modified, such that it is at least partially hydrolyzed.

[0025] Desirably, the resulting pregelatinized, partially hydrolyzed starch has low water demand when included in a stucco slurry and can be useful in the manufacture of board (e.g., gypsum board) with good strength, in preferred embodiments. Thus, in another aspect, the disclosure provides a method of making gypsum board using starch prepared with the method of pregelatinizing and acid-modifying in a single step in an extruder. In some embodiments, the pregelatinized, partially hydrolyzed starches have low water demand relative to other pregelatinized starches known in the art.

[0026] As a result, pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure can be included in a stucco slurry (e.g., by a feed line into a pin mixer) with good fluidity. In some embodiments, higher amounts of the pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure can be included since excess water is not needed to be added to the system, such that even higher strengths and lower board densities can be achieved. The resulting board exhibits good strength properties (e.g., having good core hardness, nail pull resistance, compressive strength, etc., or any relationship therebetween, based on any combination of values for each provided herein). Advantageously, inclusion of starch prepared according to the method of the disclosure during the manufacture of gypsum board enables the production of ultra low density product because of the strength enhancements. The gypsum board can be in the form of, e.g., gypsum wallboard (often referred to as drywall), which can encompass such board used not only for walls but also for ceilings and other locations as understood in the art.

Pregelatinization and Acid-Modification

[0027] Starches are classified as carbohydrates and contain two types of polysaccharides, namely linear amylose, and branched amylopectin. Starch granules are semi-crystalline, e.g., as seen under polarized light, and are insoluble in water at room temperatures. Gelatinization is the process by which the starch is placed in water and heated ("cooked"), such that the crystalline structure of the starch granules is melted, and the starch molecules are dissolved in water, resulting in good dispersion. It has been found that, when transforming a starch granule to gelatinized form, initially, the starch granule provides little viscosity in water because starch granules are water insoluble. As the temperature increases, the starch granule

swells and the crystalline structure melts at the gelatinization temperature. Peak viscosity is achieved when the starch granule has maximum swelling. Further heating will break the starch granules and dissolve the starch molecules in water, with a precipitous drop-off in viscosity. After cooling, the starch molecule will re-associate to form a 3-D gel structure, with the viscosity increasing due to the gel structure. Some commercial starches are sold in a pregelatinized form, while others are sold in the granular form. In accordance with some embodiments, the granular form undergoes at least some degree of gelatinization. To illustrate, the starch is pregelatinized prior to its addition to gypsum slurry, also referred to herein as stucco slurry (typically in a mixer, e.g., a pin mixer).

[0028] Thus, as used herein, "pregelatinized" means that the starch has any degree of gelatinization, e.g., before it is included in the gypsum slurry. In some embodiments, the pregelatinized starch can be partially gelatinized when included in the slurry, but becomes fully gelatinized when exposed to elevated temperature, e.g., in the kiln during the drying step to remove excess water. In some embodiments, the pregelatinized starch is not fully gelatinized, even upon exiting the kiln so long as the starch meets the mid-range viscosity characteristic of some embodiments when under the conditions according to the Viscosity Modifying Admixture (VMA) method.

[0029] When viscosity is referred to herein, it is in accordance with the VMA method, unless otherwise indicated. According to this method, viscosity is measured using a Discovery HR-2 Hybrid Rheometer (TA Instruments Ltd) with a concentric cylinder, a standard cup (diameter of 30 mm) with vane geometry (diameter of 28 mm and length of 42.05 mm).

[0030] When the starch is obtained, differential scanning calorimetry (DSC) techniques can be used to determine whether the starch is fully gelatinized. The DSC step can be utilized to observe whether starch is fully gelatinized, e.g., to confirm that no retrogradation has occurred. One of two procedures can be adopted, depending on the temperature required to fully gelatinize the starch, which can also be determined by DSC as one of ordinary skill in the art will appreciate.

[0031] Procedure 1 is utilized where the DSC reveals that the starch is fully gelatinized or has a gelatinization temperature at or below 90°C. Procedure 2 is utilized where the gelatinization temperature is above 90°C. Since the viscosity is measured while the starch is in water, procedure 2 uses pressure cooking in a sealed vessel to allow for superheating to temperatures above 100°C without causing the water to appreciably evaporate. Procedure 1

is reserved for starches already fully gelatinized or for starches having gelatinization temperature up to 90°C, because, as discussed below, the gelatinization takes place in the rheometer which is an open system and cannot create pressurized conditions for gelatinization. Thus procedure 2 is followed for starches having higher gelatinization temperatures. Either way, starch (7.5 g, dry basis) is added into water for a total weight of 50 g when the viscosity is measured.

[0032] In procedure 1, the starch is dispersed in the water (15% starch of the total weight of starch and water) and the sample is immediately transferred to a cylinder cell. The cell is covered with aluminum foil. The sample is heated from 25°C to 90°C at 5°C/min and a shear rate of 200 s⁻¹. The sample is held at 90°C for 10 min at a shear rate of 200 s⁻¹. The sample is cooled from 90°C to 80°C at 5°C/min and a shear rate of 200 s⁻¹. The sample is held at 80°C for 10 min at a shear rate of 0 s⁻¹. The viscosity of the sample is measured at 80°C and a shear rate of 100 s⁻¹ for 2 min. The viscosity is the average of the measurements taken between 30 seconds to 60 seconds.

[0033] Procedure 2 is used for starches having gelatinization temperature greater than 90°C. The starch is gelatinized according to the methods well-known in the starch industry (e.g., by pressure cooking). The gelatinized starch water solution (15% of total weight) is immediately transferred into the rheometer measuring cup and equilibrated at 80°C for 10 minutes. The viscosity of the sample is measured at 80°C and a shear rate of 100 s⁻¹ for 2 minutes. The viscosity is the average of the measurement from 30 seconds to 60 seconds.

[0034] Viscograph and DSC are two different methods to describe starch gelatinization. Degree of starch gelatinization can be determined by, for example, thermogram from DSC, e.g., using peak area (melting of crystal) for calculation. A viscogram (from viscograph) is less desirable to determine degree of partial gelatinization but is a good tool to obtain data such as the viscosity change of starch, gelatinization maximum, gelatinization temperature, retrogradation, viscosity during holding, viscosity at the end of cooling, etc. For degree of gelatinization, the DSC measurements are done in the presence of excess water, particularly at or above 67% by weight. If water content of starch/water mixture is less than 67%, gelatinization temperature will increase as water content decreases. It is difficult to melt starch crystals when available water is limited. When water content of starch/water mixture reaches 67%, gelatinization temperature is kept constant no matter how much more water is added into the starch/water mixture. Gelatinization onset temperature indicates the starting

temperature of gelatinization. Gelatinization end temperature indicates the end temperature of gelatinization. Enthalpy of gelatinization represents the amount of crystalline structure melted during gelatinization. By using the enthalpy from a starch DSC thermogram, the degree of gelatinization can be determined.

[0035] Different starches have different gelatinization onset temperatures, end temperatures, and gelatinization enthalpies. Therefore, different starches may become fully gelatinized at different temperatures. It will be understood that a starch is fully gelatinized when the starch is heated beyond the end temperature of gelatinization in excess water. In addition, for any particular starch, if the starch is heated below the end temperature of gelatinization, the starch will be partially gelatinized. Thus, partial and not full gelatinization will occur when starch in the presence of excess water is heated below gelatinization end temperature, e.g., as determined by DSC. Full gelatinization will occur when starch in the presence of excess water is heated above gelatinization end temperature, e.g., as determined by DSC. The degree of gelatinization can be adjusted in different ways, such as, for example, by heating the starch below the gelatinization end temperature to form partial gelatinization. For example, if the enthalpy for fully gelatinizing a starch is 4 J/g, when the DSC shows the gelatinization enthalpy of the starch as being only 2 J/g, this means 50% of the starch has been gelatinized. Fully gelatinized starch would not have the DSC thermogram gelatinization peak (enthalpy = 0 J/g) when it is measured by DSC.

[0036] As noted, the degree of gelatinization can be any suitable amount, such as about 70% or more, etc. However, smaller degrees of gelatinization will more closely approximate granular starch and may not take full advantage of the strength enhancement, better (more complete) dispersion, and/or water demand reduction of some embodiments of the disclosure. Thus, in some embodiments, it is preferred that there is a higher degree of gelatinization, e.g., at least about 75%, at least about 80%, at least about 85%, at least about 90%, at least about 95%, at least about 97%, at least about 99%, or full (100%) gelatinization. Starch with lower degree of gelatinization can be added to slurry with additional gelatinization (e.g., to 100%) taking place in the kiln in the case of gypsum board. For purposes of addition to slurry, by "fully gelatinized," it will be understood that the starch is sufficiently cooked at or above its gelatinization temperature or to otherwise achieve full gelatinization as can be seen from DSC techniques. Although some small degree of retrogradation upon cooling may be expected, the starch will still be understood as "fully gelatinized" for addition to gypsum slurry, in some embodiments as one of ordinary skill in the art will recognize. In contrast, for

purposes of the VMA method discussed herein, such retrogradation is not accepted in making the viscosity measurement.

[0037] The starch molecule can be acid-modified, e.g., to hydrolyze glycosidic bonds between glucose units to achieve desired molecular weight. One benefit of acid-modifying starch such that a reduction in molecular weight is achieved is that the water demand will decrease. Conventional pregelatinized starches that were not also acid-modified had a very high water demand, which is associated with higher energy costs. It has been conventionally believed that it is generally preferred that the modification take place before gelatinization because it tends to be more efficient and less cost intensive. Surprisingly and unexpectedly, however, the inventors have found that pregelatinization and acid-modification can be incorporated into a single step, such that they can occur simultaneously rather than in series.

Method of Preparing Starch

[0038] In accordance with some embodiments, prior to entry into the extruder, a wet starch precursor is prepared. The wet starch precursor can be prepared by any suitable method. For example, in some embodiments, the wet starch precursor is prepared by adding to a starch raw material water and an acid that is (a) a weak acid that substantially avoids chelating calcium ions, and/or (b) a strong acid in a small amount.

[0039] Any suitable starch raw material can be selected to prepare the wet starch precursor so long as it can be used to make pregelatinized, partially hydrolyzed starch, such as one meeting the mid-range viscosity characteristic of some embodiments of the disclosure. As used herein, "starch" refers to a composition that includes a starch component. As such, the starch can be 100% pure starch or may have other components such as those commonly found in flours such as protein and fiber, so long as the starch component makes up at least about 75% by weight of the starch composition. The starch can be in the form of a flour (e.g., corn flour) containing starch, such as flour having at least about 75% starch by weight of the flour, e.g., at least about 80%, at least about 85%, at least about 90%, at least about 95%, etc.). Any suitable unmodified starch or flour can be used to prepare the precursor of the pregelatinized, partially hydrolyzed starches of the disclosure. For example, the starch can be CCM260 yellow corn meal, CCF600 yellow corn flour (Bunge North America), Clinton 106 (ADM), and/or Midsol 50 (MGP Ingredients).

[0040] The wet starch precursor can be prepared to have any suitable moisture content, such that desired levels of pregelatinization and acid-modification are achieved in an

extruder. In some embodiments, for example, it is desirable that the wet starch precursor have a moisture content of from about 8 wt.% to about 25 wt.% by weight of the total starch precursor, such as from about 8 wt.% to about 23 wt.%, e.g., from about 8 wt.% to about 21 wt.%, from about 8 wt.% to about 20 wt.%, from about 8 wt.% to about 19 wt.%, from about 8 wt.% to about 18 wt.%, from about 8 wt.% to about 17 wt.%, from about 8 wt.% to about 16 wt.%, from about 8 wt.% to about 15 wt.%, from about 9 wt.% to about 25 wt.%, from about 9 wt.% to about 23 wt.%, from about 9 wt.% to about 21 wt.%, from about 9 wt.% to about 20 wt.%, from about 9 wt.% to about 19 wt.%, from about 9 wt.% to about 18 wt.%, from about 9 wt.% to about 17 wt.%, from about 9 wt.% to about 16 wt.%, from about 9 wt.% to about 15 wt.%, from about 10 wt.% to about 25 wt.%, from about 10 wt.% to about 23 wt.%, from about 10 wt.% to about 21 wt.%, from about 10 wt.% to about 20 wt.%, from about 10 wt.% to about 19 wt.%, from about 10 wt.% to about 18 wt.%, from about 10 wt.% to about 17 wt.%, from about 10 wt.% to about 16 wt.%, from about 10 wt.% to about 15 wt.%, from about 11 wt.% to about 25 wt.%, from about 11 wt.% to about 23 wt.%, from about 11 wt.% to about 21 wt.%, from about 11 wt.% to about 20 wt.%, from about 11 wt.% to about 19 wt.%, from about 11 wt.% to about 18 wt.%, from about 11 wt.% to about 17 wt.%, from about 11 wt.% to about 16 wt.%, from about 11 wt.% to about 15 wt.%, from about 12 wt.% to about 25 wt.%, from about 12 wt.% to about 23 wt.%, from about 12 wt.% to about 21 wt.%, from about 12 wt.% to about 20 wt.%, from about 12 wt.% to about 19 wt.%, from about 12 wt.% to about 18 wt.%, from about 12 wt.% to about 17 wt.%, from about 12 wt.% to about 16 wt.%, from about 12 wt.% to about 15 wt.%, from about 13 wt.% to about 25 wt.%, from about 13 wt.% to about 23 wt.%, from about 13 wt.% to about 21 wt.%, from about 13 wt.% to about 20 wt.%, from about 13 wt.% to about 19 wt.%, from about 13 wt.% to about 18 wt.%, from about 13 wt.% to about 17 wt.%, from about 13 wt.% to about 16 wt.%, from about 13 wt.% to about 15 wt.%, from about 14 wt.% to about 25 wt.%, from about 14 wt.% to about 23 wt.%, from about 14 wt.% to about 21 wt.%, from about 14 wt.% to about 20 wt.%, from about 14 wt.% to about 19 wt.%, from about 14 wt.% to about 18 wt.%, from about 14 wt.% to about 17 wt.%, from about 14 wt.% to about 16 wt.%, or from about 14 wt.% to about 15 wt.% all based on the total weight of the wet starch precursor. It will be understood that when preparing the wet starch, the moisture contents described herein include ambient moisture as well as water added.

[0041] While not wishing to be bound by any particular theory, it is believed that a lower moisture content leads to greater friction in the extruder. In some embodiments, the wet

starch can be prepared to have a moisture content that allows for sufficient mechanical energy input when the wet starch is fed through the extruder, such that friction prevents the wet starch from moving through the extruder too easily. The increased friction can increase the disruption of hydrogen bonding in the starch.

[0042] Any suitable weak acid that substantially avoids chelating calcium ions may be mixed into the wet starch. Without wishing to be bound by any particular theory, chelation includes the weak acid, for example, forming a coordination complex with calcium or otherwise interfering with the formation of gypsum crystals within the gypsum slurry. Such interference may be the reduction in number of gypsum crystals formed, retardation (decreased rate) of formation of the crystals, decreasing interactions among the gypsum crystals, etc. The term "substantially" with respect to not chelating calcium ions generally means that at least 90% (e.g., at least 92%, at least 95%, at least 96%, at least 97%, at least 98%, or at least 99%) of the available calcium ions are not chelated to the acid.

[0043] Weak acids in accordance with embodiments of the disclosure can be defined as those having a pKa value from about 1 to about 6, e.g., from about 1 to about 5, from about 1 to 4, from about 1 to 3, from about 1 to 2, from about 1.2 to about 6, from about 1.2 to about 5, from about 1.2 to about 4, from about 1.2 to about 3, from about 1.2 to about 2, from about 2 to about 6, from about 2 to about 5, from about 2 to about 4, from about 2 to about 3, from about 3 to about 6, from about 3 to about 5, from about 3 to about 4, from about 4 to about 6, or from about 4 to about 5. As is understood in the art, the pKa value is a measure of the strength of an acid; the lower the pKa value, the stronger the acid.

[0044] Weak acids that substantially avoid chelating calcium ions are characterized, for example, by a lack of multi-binding sites, such as multiple carboxyl functional groups (COO⁻), which tend to bind calcium ions. In some embodiments, the weak acid has a minimal amount of multi-binding sites, such as multi-COO⁻ groups, or is substantially free of multi-binding sites, such as multi-COO⁻ groups, such that, for example, chelation is minimal (i.e., substantially avoided) or gypsum crystal formation is not substantially impacted relative to the crystal formation in the absence of the weak acid. In some embodiments, for example, aluminum sulfate (alum) is an appropriate weak acid to use in preparing the wet starch since it substantially avoids chelating calcium ions. Alum does not have multi-binding sites.

[0045] In some embodiments, alum is added into the wet starch precursor in any suitable form, such as in liquid containing alum of desired solids content. For example, the liquid

alum can be included in an aqueous solution where the alum is present in any suitable amount. Other weak acids can be added similarly.

[0046] The wet starch can be mixed to include any suitable amount of a weak acid that substantially avoids chelating calcium ions, such that the pregelatinized, partially hydrolyzed starch is prepared with desired viscosity and low water demand and is not over hydrolyzed into sugar. For example, in some embodiments, such weak acid is included in an amount of from about 0.5 wt.% to about 5 wt.% based on the weight of the starch, such as from about 0.5 wt.% to about 4.5 wt.%, e.g., from about 0.5 wt.% to about 4 wt.%, from about 0.5 wt.% to about 3.5 wt.%, from about 0.5 wt.% to about 3 wt.%, from about 1 wt.% to about 5 wt.%, from about 1 wt.% to about 4.5 wt.%, from about 1 wt.% to about 4 wt.%, from about 1 wt.% to about 3.5 wt.%, from about 1 wt.% to about 3 wt.%, from about 1.5 wt.% to about 5 wt.%, from about 1.5 wt.% to about 4.5 wt.%, from about 1.5 wt.% to about 4 wt.%, from about 1.5 wt.% to about 3.5 wt.%, from about 1.5 wt.% to about 3 wt.%, from about 2 wt.% to about 5 wt.%, from about 2 wt.% to about 4.5 wt.%, from about 2 wt.% to about 4 wt.%, from about 2 wt.% to about 3.5 wt.%, from about 2 wt.% to about 3 wt.%, from about 2.5 wt.% to about 5 wt.%, from about 2.5 wt.% to about 4.5 wt.%, from about 2.5 wt.% to about 4 wt.%, from about 2.5 wt.% to about 3.5 wt.%, or from about 2.5 wt.% to about 3 wt.%. It will be understood that these amounts encompass the weak acid component, and, when the weak acid is in a solution, excludes the water or other components of the solution.

[0047] The wet starch precursor can be prepared to optionally further comprise secondary acids that can chelate calcium ion, such as tartaric acid. Thus, in some embodiments, a secondary acid, such as tartaric acid, can be combined with any suitable weak acid that does not chelate calcium ions. Tartaric acid is known to retard gypsum crystallization. However, in combination with the non-chelating weak acid, tartaric acid can avoid substantial retarding of gypsum crystallization, such that the hydrolysis reaction via acid-modification is optimized. Besides tartaric acid, other secondary acids, such as succinic acid or malic acid, may be beneficial so long as they do not surpass the accelerating effect of alum. In some embodiments, the wet starch precursor includes both alum and tartaric acid.

[0048] If included, secondary acids (e.g., tartaric acid) can be present in any suitable amount. For example, tartaric acid can be present in an amount of from about 0.1 wt.% to about 0.6 wt.% based on the weight of the starch, e.g., from about 0.1 wt.% to about 0.4 wt.%, from about 0.2 wt.% to about 0.3 wt.%.

[0049] In some embodiments, oil can optionally be added to the wet starch to improve the conveyability of starch inside the extruder. Possible oils include canola oil, vegetable oil, corn oil, soybean oil, or any combination thereof, in some embodiments. For example, in some embodiments, canola oil or one of the aforementioned substitutes can optionally be added in an amount of from about 0 wt.% to about 0.25 wt.% by weight of the starch, e.g., from about 0.1 wt.% to about 0.2 wt.%, from about 0.1 wt.% to about 0.15 wt.%, from about 0.15 wt.% to about 0.25 wt.%, from about 0.15 wt.% to about 0.2 wt.%, or from about 0.2 wt.% to about 0.25 wt.%.

[0050] In accordance with some embodiments, the wet starch precursor is prepared by mixing water, non-pregelatinized starch, and a small amount of a strong acid. In some embodiments, the strong acid has a pKa of about -1.7 or less. Any such strong acid can be used and, in some embodiments, the strong acid comprises sulfuric acid, nitric acid, hydrochloric acid, or any combination thereof. Sulfuric acid, alone or in combination with other acids, is preferred in some embodiments because sulfate ion can accelerate gypsum crystallization in gypsum board.

[0051] The amount of strong acid is relatively small, such as about 0.05 wt.% or less by weight of the starch, e.g., about 0.045 wt.% or less, about 0.04 wt.% or less, about 0.035 wt.% or less, about 0.03 wt.% or less, about 0.025 wt.% or less, about 0.02 wt.% or less, about 0.015 wt.% or less, about 0.01 wt.% or less, about 0.005 wt.% or less, about 0.001 wt.% or less, about 0.0005 wt.% or less, such as from about 0.0001 wt.% to about 0.05 wt.%, from about 0.0001 wt.% to about 0.045 wt.%, from about 0.0001 wt.% to about 0.04 wt.%, from about 0.0001 wt.% to about 0.035 wt.%, from about 0.0001 wt.% to about 0.03 wt.%, from about 0.0001 wt.% to about 0.025 wt.%, from about from about 0.0001 wt.% to about 0.02 wt.%, from about 0.0001 wt.% to about 0.015 wt.%, from about 0.0001 wt.% to about 0.01 wt.%, from about 0.0001 wt.% to about 0.005 wt.%, from about 0.0001 wt.% to about 0.001 wt.%, from about 0.0001 wt.% to about 0.0005 wt.% by weight of the starch. It will be understood that these amounts encompass the strong acid component, and, when the strong acid is in a solution, excludes the water or other components of the solution. For example, conventional strong acid-modification uses 2% sulfuric acid solution, with starch solid of ~35% (2 g sulfuric acid for 35 g starch). The percent is based on pure sulfuric acid components. It is calculated as the weight of sulfuric acid component divided by the weight of the wet starch. For example, if the sulfuric acid is 50% wt.% based on the water (which means that half the weight of the solution is pure sulfuric acid), then the weight of the sulfuric

acid solution is doubled when calculating wt.% of acid to starch. To illustrate, for 100 g starch, 0.1 g pure sulfuric acid is added to achieve 0.1 wt.%. If the concentration of sulfuric solution is 50%, 0.2 g of the 50% sulfuric acid solution is added to achieve 0.1 wt.%.

[0052] It will be understood that there are different grades of acids (>95%, 98%, 99.99%). These differences are encompassed by the term "about" in relation to the amount of strong acid in the starch precursor. One of ordinary skill in the art will readily be able to determine the wt.% described herein to include the different grades. The amounts of strong acid used in accordance with some embodiments of the disclosure are considerably smaller than what were included in conventional systems which used, e.g., at least about 2 g of sulfuric acid for 35 g of starch. In some embodiments, the strong acid in small amounts as described above can be used in combination with a weak acid that does not chelate calcium ions, such as alum, as described herein.

[0053] Some embodiments provide for feeding the wet starch precursor through an extruder, such that the wet starch precursor is pregelatinized and acid-modified in a single step in the extruder. It will be appreciated that an extruder is a machine generally used to melt and process polymers into a desired shape by melting the polymer and pumping it through a die. The extruder can also mix the polymer with other ingredients, such as color dyes, reinforcing fibers, mineral fillers, etc. The purpose of the extruder is to disperse and distribute all of the ingredients fed into it and to melt the ingredients with a constant temperature and pressure.

[0054] Configurations and arrangements for extruders are known in the art. In general, an extruder comprises a feed hopper to deliver the feed material, a preconditioner comprising heat jackets for conditioning polymer with plasticizer (e.g., water), an extruder modular head comprising heating zones, and a die assembly. Extruders generally include a feed auger, a knife, and screw(s). The feed auger is present to help convey the wet starch precursor into the extruder. The knife is present to cut the string-like pregelatinized, partially hydrolyzed starch into small pellets, such that they can be ground. The screw(s) help mix the wet starch precursor, convey the wet starch precursor through the extruder, and provide mechanical shearing. An extruder can be of the single-screw or twin-screw varieties as will be understood by one of ordinary skill in the art. See, e.g., Leszek Moscicki, *Extrusion-Cooking Techniques*, WILEY-VCH Verlag & Co. KGaA, 2011.

[0055] In single-screw extruders, the screw generally comprises a feed section with deep channels for transporting the solids from the throat of the feeder and compressing them, a

compression section at which point the screw's channels become progressively less deep and the polymer is melted, and a metering section with shallow channels that conveys the melted polymer to the die. Some screws are designed to include mixing devices (e.g., pins extending from the screw).

[0056] Twin-screw extruders generally have two screws that rotate either in the same direction (i.e., co-rotating) or in opposite directions (i.e., counter-rotating). The two screws may rotate with non-intermeshing or fully intermeshing flights. Whereas in the case of single-screw extruders, the material being fed fills the entire screw channel, in the case of twin-screw extruders, only part of the screw channel is filled, such that downstream feed ports or vents can be utilized for the addition of certain ingredients.

[0057] The die assembly generally comprises a plate, spacer, and die head. When extruding materials, the process can be either continuous, such that the material is extruded in an indefinite length, or semi-continuous, such that the material is extruded in pieces. Materials being extruded may be hot or cold.

[0058] The disclosure provides a method of preparing pregelatinized, partially hydrolyzed starch in an extruder. Any suitable extruder can be used, such as a single-screw extruder (e.g., the Advantage 50 available from American Extrusion International, located in South Beloit, IL) or a twin-screw extruder (e.g., the Wenger TX52 available from Wenger located in Sabetha, KS).

[0059] As described herein, non-pregelatinized starch, an acid in the form of a weak acid that substantially avoids chelating calcium ions and/or a strong acid in a small amount, and water are mixed and fed into the extruder. In some embodiments, additional water may be added to the extruder. While in the extruder, a combination of heating elements and mechanical shearing melts and pregelatinizes the starch, and the weak acid partially hydrolyzes the starch to a desired molecular weight indicated by viscosity as desirable as described herein. The conditions in the extruder, because of the mechanical energy, will also cause the starch molecules to degrade, which partially produces the same effect of acid-modification. It is believed that because the conditions in an extruder (e.g., high reaction temperature and high pressure) in accordance with some embodiments facilitate this chemical reaction, a weak acid and/or low amounts of a strong acid can be used. The method of preferred embodiments, thus, improves the efficiency of starch acid-modification.

[0060] The main screw(s) can be operated at any suitable speed, such that desired mixing and mechanical shearing are achieved. For example, in some embodiments the main screw

can be operated at a speed of about 350 RPM ($\pm$ about 100 RPM). The feed auger can be operated at any suitable speed to achieve desired feeding rate. For example, in some embodiments the feed auger can be operated at a speed of about 14 RPM ($\pm$ about 5 RPM).

[0061] The knife can be operated at any suitable speed. For example, in various embodiments the knife can be operated at a speed of from about 400 RPM to about 1,000 RPM, e.g., from about 400 RPM to about 900 RPM, from about 400 RPM to about 800 RPM, from about 400 RPM to about 700 RPM, from about 400 RPM to about 600 RPM, from about 400 RPM to about 500 RPM, from about 500 RPM to about 1,000 RPM, from about 500 RPM to about 900 RPM, from about 500 RPM to about 800 RPM, from about 500 RPM to about 700 RPM, from about 500 RPM to about 600 RPM, from about 600 RPM to about 1,000 RPM, from about 600 RPM to about 900 RPM, from about 600 RPM to about 800 RPM, from about 600 RPM to about 700 RPM, from about 700 RPM to about 1,000 RPM, from about 700 RPM to about 900 RPM, from about 700 RPM to about 800 RPM, from about 800 RPM to about 1,000 RPM, from about 800 RPM to about 900 RPM, or from about 900 RPM to about 1,000 RPM.

[0062] The wet starch can be pregelatinized and acid-modified in an extruder having a die at any suitable temperature, such that the wet starch becomes sufficiently pregelatinized without burning the materials. For example, the wet starch can be pregelatinized and acid-modified in an extruder having a die at a temperature of from about 150°C (about 300°F) to about 210°C (about 410°F), e.g., in various embodiments, from about 150°C to about 205°C (about 400°F), from about 150°C to about 199°C (about 390°F), from about 150°C to about 193°C (about 380°F), from about 150°C to about 188°C (about 370°F), from about 150°C to about 182°C (about 360°F), from about 154°C (about 310°F) to about 210°C, from about 154°C to about 205°C (about 400°F), from about 154°C to about 199°C, from about 154°C to about 193°C, from about 154°C to about 188°C, from about 154°C to about 182°C, from about 160°C (about 320°F) to about 210°C, from about 160°C to about 205°C (about 400°F), from about 160°C to about 199°C, from about 160°C to about 193°C, from about 160°C to about 188°C, from about 160°C to about 182°C, from about 166°C (about 330°F) to about 210°C, from about 166°C to about 205°C, from about 166°C to about 199°C, from about 166°C to about 193°C, from about 166°C to about 188°C, from about 166°C to about 182°C, from about 171°C (about 340°F) to about 210°C, from about 171°C to about 205°C, from about 171°C to about 199°C, from about 171°C to about 193°C, from about 171°C to about 188°C, from about 171°C to about 182°C, from about 177°C (about 350°F) to about 210°C, from about 177°C to

about 205°C, from about 177°C to about 199°C, from about 177°C to about 193°C, from about 177°C to about 188°C, or from about 177°C to about 182°C. While the die of the extruder can be any sufficient temperature as described herein, the die temperature generally exceeds the melting temperature of the starch crystals.

[0063] The degree of gelatinization can be any suitable amount, such as at least about 70% or more, e.g., at least about 75%, at least about 80%, at least about 85%, at least about 90%, at least about 95%, at least about 97%, at least about 99%, or full (100%) gelatinization. In the case of making wallboard as described below, starch with such lower degrees of gelatinization can be added to stucco slurry, e.g., with additional gelatinization (for example, to 100%) taking place in the kiln.

[0064] The pressure in the extruder can be at any suitable level, such that appropriate conditions for pregelatinization and acid-modification are achieved. Pressure inside the extruder is determined by the raw material being extruded, moisture content, die temperature, and screw speed, which will be recognized by one of ordinary skill in the art. For example, the pressure in the extruder can be at least about 2,000 psi (about 13,800 kPa), such as at least about 2,250 psi (about 15,500 kPa), at least about 2,500 psi (about 17,200 kPa), at least about 2,750 psi (about 19,000 kPa), at least about 3,000 psi (about 20,650 kPa), at least about 3,500 psi (about 24,100 kPa), at least about 4,000 psi (about 27,600 kPa), or at least about 4,500 psi (about 31,000 kPa). In some embodiments, the pressure can be from about 2,000 psi to about 5,000 psi (34,500 kPa), e.g., from about 2,000 psi to about 4,500 psi, from about 2,000 psi to about 4,000 psi, from about 2,000 psi to about 3,500 psi, from about 2,000 psi to about 3,000 psi, from about 2,000 psi to about 2,500 psi, from about 2,500 psi to about 5,000 psi, from about 2,500 psi to about 4,500 psi, from about 2,500 psi to about 4,000 psi, from about 2,500 psi to about 3,500 psi, from about 2,500 psi to about 3,000 psi, from about 3,000 psi to about 5,000 psi, from about 3,000 psi to about 4,500 psi, from about 3,000 psi to about 4,000 psi, from about 3,000 psi to about 3,500 psi, from about 3,500 psi to about 5,000 psi, from about 4,000 psi to about 5,000 psi, from about 4,000 psi to about 4,500 psi, or from about 4,500 psi to about 5,000 psi.

[0065] It has been found that preparing pregelatinized, partially hydrolyzed starch in a single step in an extruder is considerably faster than pregelatinizing and acid-modifying starch in two steps in series. Significantly greater amounts of pregelatinized, partially hydrolyzed starch can be prepared with the method according to preferred embodiments than starch prepared with any other method. The higher production amount and faster output rate

are because of high reaction rate at high temperature and/or high pressure. In some embodiments, pregelatinization and acid-modification occur in less than about 5 minutes, such as less than about 4 minutes, e.g., less than about 3 minutes, less than about 2 minutes, less than about 90 seconds, less than about 75 seconds, less than about 1 minute, less than about 45 seconds, less than about 30 seconds, less than about 25 seconds, less than about 20 seconds, less than about 15 seconds, or less than about 10 seconds. In addition, in some embodiments, the pregelatinization and acid-modification occur at a rate in the extruder bound by any two of the foregoing points. For example, the pregelatinization and acid-modification rate can be between about 10 seconds and 5 minutes, e.g., between about 10 seconds and about 4 minutes, between about 10 seconds and about 3 minutes, between about 10 seconds and about 2 minutes, between about 10 seconds and about 90 seconds, between about 10 seconds and about 75 seconds, between about 10 seconds and about 1 minute, between about 10 seconds and about 45 seconds, between about 10 seconds and about 30 seconds, between about 10 seconds and about 25 seconds, between about 10 seconds and about 20 seconds, or between about 10 seconds and about 15 seconds.

[0066] The method of preparing pregelatinized, partially hydrolyzed starch can be a continuous process occurring at any sufficient rate. In some embodiments, starch is pregelatinized and acid-modified at a production output rate in an extruder of at least about 100 kg/hr, such as at least about 150 kg/hr, at least about 200 kg/hr, at least about 250 kg/hr, at least about 300 kg/hr, at least about 350 kg/hr, at least about 400 kg/hr, at least about 450 kg/hr, 500 kg/hr, at least about 550 kg/hr, e.g., at least about 600 kg/hr, at least about 650 kg/hr, at least about 700 kg/hr, at least about 750 kg/hr, at least about 800 kg/hr, at least about 850 kg/hr, at least about 900 kg/hr, at least about 950 kg/hr, at least about 1,000 kg/hr, at least about 1,050 kg/hr, at least about 1,100 kg/hr, at least about 1,150 kg/hr, at least about 1,200 kg/hr, at least about 1,250 kg/hr, at least about 1,300 kg/hr, at least about 1,350 kg/hr, at least about 1,400 kg/hr, at least about 1,450 kg/hr, or at least about 1,500 kg/hr. In addition, in some embodiments, the production output rate in an extruder can be bound by any two of the foregoing points. For example, the production output rate can be between about 100 kg/hr and about 1,500 kg/hr (e.g., between about 100 kg/hr and about 1,500 kg/hr, between about 100 kg/hr and 1,000 kg/hr, between about 250 kg/hr and about 1,500 kg/hr, between about 250 kg/hr and about 1,000 kg/hr, between about 600 kg/hr and about 1,250 kg/hr, between about 650 kg/hr and about 1,200 kg/hr, between about 700 kg/hr and about 1,100 kg/hr, between about 750 kg/hr and about 1,000 kg/hr, etc.).

[0067] It has been found by the inventors that in some embodiments the conditions in an extruder (e.g., high temperature and high pressure) are particularly conducive to efficiently and sufficiently pregelatinize and acid-modify starch in a single step. When the extruder mixes the wet starch, it creates very high friction, thereby generating heat. The shear force is created by the screw in the extruder because the space between the screw and chamber in the extruder is very small. Specific mechanical energy (SME) describes mechanical energy of an object per unit of mass. SME will depend on the moisture content. Higher moisture content (e.g., for purposes of fluidity) will result in low viscosity and low friction and, thus, a smaller SME. If more moisture is present, a smaller SME will result because of low viscosity and low friction. The moisture contents in the wet starch precursor of the disclosure as described herein provide effective SME.

[0068] In the extruder, because of the conditions provided by some embodiments as described herein, the starch is pregelatinized highly efficiently. While not wishing to be bound by any particular theory, it is believed that the good mixing in the extruder in accordance with some embodiments requires less water for reaction in an extruder. Very low moisture content facilitates a high concentration of reactant, which can accelerate the chemical reaction rate. The high temperature of the extruder also significantly accelerates the reaction rate. When the starch leaves the extruder, the reaction has occurred, such that it is pregelatinized and partially hydrolyzed.

[0069] In conventional acid-modification, starch is added into a strong acid solution. This conventional method uses significantly more water and acid than the surprising and unexpected method of simultaneously pregelatinizing and acid-modifying starch in one step in an extruder as described herein rather than in series. Conventional acid-modification takes several hours. After the reaction has taken place, the acid needs to be neutralized, purified, and washed away. The neutralization and purification steps are time consuming and costly.

[0070] It was conventionally thought to be undesirable to use a weak acid that substantially avoids chelating calcium ions or a strong acid in a small amount in conventional acid-modification. This is because, in the conventional method, the weaker the acid is or the smaller the amount of a strong acid is, the longer acid-modification takes. Thus, a strong acid (e.g., having a pKa of below about -1.7) in high amounts was desired in conventional acid-modification. Surprisingly and unexpectedly, when pregelatinized, partially hydrolyzed starch is prepared in an extruder according to embodiments of the disclosure using a weak acid or a strong acid in a small amount as described herein, there is no need for neutralization

and purification steps, due to the mild acidic condition and less interference with gypsum crystallization, respectively. In some embodiments, there can still be acid present in the pregelatinized, partially hydrolyzed starch.

Properties of Starch and Advantages of Using the Starch in Gypsum Board

[0071] The starch prepared in an extruder, in accordance with some embodiments, can be any pregelatinized, partially hydrolyzed starch. In some embodiments, the starch can be prepared to have various properties as desired (e.g., mid-range viscosity, cold water solubility, cold water viscosity, etc.) as described herein.

[0072] Pregelatinized, partially hydrolyzed starches prepared in an extruder in accordance with some embodiments can be suitable for use in gypsum board. For application in gypsum board, for instance, pregelatinization and acid-modification are beneficial, e.g., for strength purposes by achieving a desired viscosity (and, hence, molecular weight range) in accordance with embodiments of the disclosure as described herein. In the method of making wallboard discussed herein, the starch that is introduced into the stucco slurry can be at least about 70% gelatinized, e.g., at least about 75% gelatinized, at least about 80% gelatinized, at least about 85% gelatinized, at least about 90% gelatinized, at least about 95% gelatinized, at least about 97% gelatinized, or 100% gelatinized (i.e., fully gelatinized).

[0073] Furthermore, feeding a wet starch comprising a weak acid that substantially avoids chelating calcium ions as described herein into an extruder, in accordance with embodiments of the disclosure, hydrolyzes the starch, such that a desired viscosity is achieved, thus indicating a desired molecular weight range is achieved. Viscosity thereby indicates the molecular weight of the pregelatinized, partially hydrolyzed starch, as will be appreciated by one of ordinary skill in the art.

[0074] In some embodiments, pregelatinized, partially hydrolyzed starch prepared in accordance with some embodiments can be prepared to have a "mid-range" viscosity (i.e., having a viscosity from about 20 centipoise to about 300 centipoise) when the pregelatinized, partially hydrolyzed starch is subjected to conditions according to the VMA method with the pregelatinized, partially hydrolyzed starch in water in an amount of 15% by weight of the total weight of the pregelatinized, partially hydrolyzed starch and water. Thus, the VMA method is used to determine whether the pregelatinized, partially hydrolyzed starch exhibits the mid-range viscosity characteristic when subjected to the conditions of the VMA method. This does not mean that the pregelatinized, partially hydrolyzed starch must be added to the

gypsum slurry under these conditions. Rather, when adding the pregelatinized, partially hydrolyzed starch to slurry, it can be in wet (in various concentrations of starch in the water) or dry forms, and it need not be fully gelatinized as described herein or otherwise under the conditions set forth in the VMA method.

[0075] In some embodiments, the mid-range viscosity of the pregelatinized starch can be from about 20 centipoise to about 300 centipoise, such as from about 20 centipoise to about 250 centipoise, from about 30 centipoise to about 200 centipoise, or from about 50 centipoise to about 300 centipoise. In embodiments of the disclosure, the viscosity of the pregelatinized starch when tested under the VMA method can be, e.g., as listed in Tables 1A and 1B. In the tables, an "X" represents the range "from about [corresponding value in top row] to about [corresponding value in left-most column]." The indicated values represent viscosity of the pregelatinized starch in centipoise (cP). For ease of presentation, it will be understood that each value represents "about" that value. For example, the first "X" in Table 1A is the range "about 20 centipoise to about 25 centipoise."

Table 1A

[illegible]

Table 1B

End Point for Viscosity Range (centipoise)		100	125	150	175	200	225	250	275	300
	125	X								
	150	X	X							
	175	X	X	X						
	200	X	X	X	X					
	225	X	X	X	X	X				
	250	X	X	X	X	X	X			
	275	X	X	X	X	X	X	X		
	300	X	X	X	X	X	X	X	X	

[0076] Thus, the viscosity of the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can have a range between and including any of the aforementioned endpoints set forth in Tables 1A or 1B. Alternatively, in some embodiments, the pregelatinized, partially hydrolyzed starch has a viscosity (10% solids, 93°C) of from about 5 Brabender Units (BU) to about 33 BU, measured according to the Brabender method described herein, e.g., from about 10 BU to about 30 BU, from about 12 BU to about 25 BU, or from about 15 BU to about 20 BU.

[0077] In some embodiments, pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure can provide significant benefits to the strength of the product (e.g., wallboard) to which they are applied. Since starch contains glucose monomers containing three hydroxyl groups, starch provides many sites for hydrogen bonding to gypsum crystals. While not wishing to be bound by any particular theory, it is believed that the molecular size of pregelatinized, partially hydrolyzed starch prepared in accordance with some embodiments allows for optimal mobility of starch molecules to align starch molecules with the gypsum crystals to facilitate good binding of starch to gypsum crystals to strengthen the resulting crystalline gypsum matrix, e.g., via hydrogen bonding.

[0078] Conventional pregelatinized starches prepared according to another method than that which is described herein, e.g., having higher viscosities, which would have either longer chain lengths and higher molecular weight (viscosity that is too high) and shorter chain lengths and lower molecular weights (viscosity that is too low), respectively, do not provide the same combination of benefits. The inventors have found dissolved starch molecules with,

for example, mid-range viscosity (representing mid range molecular weight of starch) allows for optimal mobility of starch molecules to align starch molecules with gypsum crystals to facilitate good starch and gypsum hydrogen-bonding and core strength in some embodiments.

[0079] Pregelatinized, partially hydrolyzed starch prepared in accordance with some embodiments of the disclosure also provides advantages with respect to water demand, in some embodiments. Adding conventional pregelatinized starch to gypsum slurry requires that additional water be added to the gypsum slurry in order to maintain a desired degree of slurry fluidity. This is because conventional pregelatinized starch increases the viscosity and reduces the fluidity of the gypsum slurry. Thus, the use of pregelatinized starch in conventional systems has resulted in an increase in water demand such that even more excess water would be required in the gypsum slurry.

[0080] Pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure, particularly with the desired mid-range viscosity, demands less water so that the effect on water demand in the gypsum slurry is reduced, especially in comparison to conventional starches. Furthermore, because of the efficiency of the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure, such that less starch can be used, the positive impact on water demand can be even more significant in accordance with some embodiments of the disclosure. This lower water demand provides considerable efficiencies during manufacture. For example, excess water requires energy input for drying. The speed of the line must be slowed to accommodate the drying. Thus, by reducing the water load in the gypsum slurry, less energy resources and cost can be seen, as well as faster production rates. In some embodiments, the increase in water demand in a gypsum slurry is less than the increase in water demand required by other starches such as pregelatinized starches having viscosity above 300 centipoise (e.g., a corn starch having a viscosity of about 773 centipoise), e.g., prepared by a different method.

[0081] Any suitable non-pregelatinized starch can be selected in preparing a pregelatinized, partially hydrolyzed starch so long as it is sufficient to be pregelatinized and acid-modified in an extruder. As used herein, "starch" refers to a composition that includes a starch component. As such, the starch can be 100% pure starch or may have other components such as those commonly found in flours such as protein and fiber, so long as the starch component makes up at least about 75% by weight of the starch composition. The starch can be in the form of a flour (e.g., corn flour) containing starch, such as flour having at

least about 75% starch by weight of the flour, e.g., at least about 80%, at least about 85%, at least about 90%, at least about 95%, etc.). By way of example, and not in any limitation, the starch can be in the form of a corn flour containing starch.

[0082] In some embodiments, the pregelatinized, partially hydrolyzed starch prepared in accordance with some embodiments can be prepared to have desired cold water solubility. Conventional pregelatinization techniques involve making starch cold water soluble and generally require cooking starch in an excess amount of water. However, these conventional techniques are not efficient. Extrusion, in accordance with embodiments of the disclosure, which allows for a combination of heating and mechanical shearing, is surprisingly and unexpectedly an energy efficient method that can be used to produce pregelatinized, partially hydrolyzed starch in a one step process having a low moisture content with cold water solubility. Cold water solubility is defined as having any amount of solubility in water at room temperature (about 25°C). It was discovered that starches exhibiting solubility in cold water can provide significant benefits to the strength of gypsum products (e.g., wallboard). Cold water soluble starches of preferred embodiments have a cold water solubility greater than about 70% and, when added to a set gypsum core, can increase the strength of the gypsum core. Generally, it has been found that a higher cold water solubility indicates a higher degree of gelatinization, and higher strength. The solubility of the pregelatinized starch in water is defined as the amount of starch that dissolves in room temperature water divided by the total amount of starch.

[0083] In some embodiments, the cold water solubility of the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure is at least about 70%, at least about 75%, at least about 80%, at least about 85%, at least about 90%, or at least about 95%, e.g., from about 70% to about 100%, from about 75% to about 100%, from about 80% to about 100%, from about 85% to about 100%, from about 90% to about 100%, from about 95% to about 100%, from about 70% to about 99%, etc., from about 75% to about 99%, from about 80% to about 99%, from about 85% to about 99%, from about 90% to about 99%, from about 95% to about 99%. In preferred embodiments, the cold water solubility of the extruded pregelatinized, partially hydrolyzed starch is from about 90% to about 100%.

[0084] While not wishing to be bound by any particular theory, it is believed that a combination of mechanical and thermal energy during extrusion is responsible for the cold water solubility of the pregelatinized, partially hydrolyzed starch prepared in accordance with

embodiments of the disclosure. It is believed that when the starch undergoes extrusion, the hydrogen bonds between the starch molecules are broken. When the extruded starch is dissolved in water, the starch forms hydrogen bonds with the water molecules. After the pregelatinization process, the extruded pregelatinized, partially hydrolyzed starch molecules are free to hydrogen-bond with the gypsum crystals, thus imparting higher strength to the gypsum product. Accordingly, because starches exhibiting solubility in cold water improves the strength of gypsum wallboard, less starch is required compared with conventional starches.

[0085] In some embodiments, the pregelatinized, partially hydrolyzed starch has a cold water viscosity (10% solids, 25°C) of from about 10 BU to about 100 BU, measured according to the Brabender method described herein, e.g., from about 20 BU to about 100 BU, from about 30 BU to about 100 BU, from about 40 BU to about 90 BU, from about 50 BU to about 80 BU, or from about 60 BU to about 70 BU. In some embodiments, the starch has a cold water viscosity of a 10% slurry of the starch in water when measured at 25 °C of from about 60 cP to about 160 cP, as measured with a Brookfield viscometer with #2 spindle and at a rotation speed of 30 rpm. For example, the cold water viscosity of a 10% slurry of the starch in water when measured at 25 °C can be from about 60 cP to about 150 cP, from about 60 cP to about 120 cP, from about 60 cP to about 100 cP, from about 70 cP to about 150 cP, from about 70 cP to about 120 cP, from about 70 cP to about 100 cP, from about 80 cP to about 150 cP from about 80 cP to about 120 cP, from about 80 cP to about 100 cP, from about 90 cP to about 150 cP, from about 90 cP to about 120 cP, from about 100 cP to about 150 cP, or from about 100 cP to about 120 cP.

Use of Starch in Making Board

[0086] In some embodiments, a board (e.g., gypsum wallboard) can be made by forming a pregelatinized, partially hydrolyzed starch by) mixing at least water, non-pregelatinized starch, and an acid to form a wet starch precursor having a moisture content of from about 8 wt.% to about 25 wt.%, the acid selected from: a weak acid that substantially avoids chelating calcium ions, a strong acid in an amount of about 0.01 wt.% or less by weight of the starch, or any combination thereof.

[0087] The wet starch precursor is then fed into an extruder in which the temperature of the die is about 150°C (about 300°F) to about 210°C (about 410°F) where the wet starch is pregelatinized and acid-modified, such that it is at least partially hydrolyzed. The

pregelatinized, partially hydrolyzed starch can then be mixed with at least water and stucco to form a slurry, which can then be disposed between a first cover sheet and a second cover sheet to form a wet assembly. The wet assembly can then be cut into a board, which is then dried. Preferably, the set gypsum core of the board has a compressive strength greater than a set gypsum core made with a starch prepared under a different method.

[0088] Surprisingly and unexpectedly, in accordance with preferred embodiments, it has been found that elevated amounts of the pregelatinized starch can be used to increase strength in the resulting board core (e.g., by enhancing the strength of the gypsum crystal matrix). Thus, in preferred embodiments, the pregelatinized starch is added to the slurry for forming the board core in an amount of greater than 10% by weight of the stucco.

[0089] For example, in some embodiments, the pregelatinized starch can be provided in an amount of about 10.1% by weight of the stucco to about 25% by weight of the stucco, e.g., about 10.1% by weight of the stucco to about 22% by weight of the stucco, about 10.1% by weight of the stucco to about 20% by weight of the stucco, about 10.1% by weight of the stucco to about 15% by weight of the stucco, about 11% by weight of the stucco to about 25% by weight of the stucco, about 11% by weight of the stucco to about 22% by weight of the stucco, about 11% by weight of the stucco to about 20% by weight of the stucco, about 11% by weight of the stucco to about 15% by weight of the stucco, about 12% by weight of the stucco to about 25% by weight of the stucco, about 12% by weight of the stucco to about 22% by weight of the stucco, about 12% by weight of the stucco to about 20% by weight of the stucco, about 12% by weight of the stucco to about 15% by weight of the stucco, about 13% by weight of the stucco to about 25% by weight of the stucco, about 13% by weight of the stucco to about 22% by weight of the stucco, about 13% by weight of the stucco to about 20% by weight of the stucco, about 13% by weight of the stucco to about 15% by weight of the stucco, about 14% by weight of the stucco to about 25% by weight of the stucco, about 14% by weight of the stucco to about 22% by weight of the stucco, about 14% by weight of the stucco to about 20% by weight of the stucco, about 15% by weight of the stucco to about 25% by weight of the stucco, about 15% by weight of the stucco to about 22% by weight of the stucco, about 15% by weight of the stucco to about 20% by weight of the stucco, about 15% by weight of the stucco to about 18% by weight of the stucco, about 18% by weight of the stucco to about 25% by weight of the stucco, about 18% by weight of the stucco to about 22% by weight of the stucco, or about 20% by weight of the stucco to about 25% by weight of the stucco.

[0090] Advantageously, because of the increased benefit to board core resulting from the use of the elevated amounts of pregelatinized starch (e.g., above 10 wt.% on a starch basis), gypsum wallboard can be prepared at particularly ultra light densities (and hence weights, since density is a function of board weight at particular thickness). In preferred embodiments, the resulting board can be prepared to have a dry density of about 31 pcf or less, e.g., about 30 pcf or less, about 29 pcf or less, about 28 pcf or less, about 27 pcf or less, about 26 pcf or less, about 25 pcf or less, about 24 pcf or less, about 23 pcf or less, about 22 pcf or less, about 21 pcf, or about 20 pcf or less. In some embodiments, the board has a dry density of from about 15 pcf to about 31 pcf, e.g., from about 15 pcf to about 30 pcf, from about 15 pcf to about 29 pcf, from about 15 pcf to about 28 pcf, from about 15 pcf to about 27 pcf, from about 15 pcf to about 26 pcf, from about 15 pcf to about 25 pcf, from about 15 pcf to about 24 pcf, from about 15 pcf to about 23 pcf, from about 15 pcf to about 22 pcf, from about 15 pcf to about 21 pcf, from about 15 pcf to about 20 pcf, from about 15 pcf to about 19 pcf, from about 15 pcf to about 18 pcf, from about 19 pcf to about 31 pcf, from about 19 pcf to about 30 pcf, from about 19 pcf to about 29 pcf, from about 19 pcf to about 28 pcf, from about 19 pcf to about 27 pcf, from about 19 pcf to about 26 pcf, from about 19 pcf to about 25 pcf, from about 19 pcf to about 24 pcf, from about 19 pcf to about 23 pcf, from about 19 pcf to about 22 pcf, or from about 19 pcf to about 21 pcf.

[0091] Pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can be added to the slurry in combination with other starches, in some embodiments for various applications. For example, in the case of gypsum wallboard as described below, pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can be combined with other starches to enhance both core strength and paper-core bond, particularly if some increase in water demand is accepted.

[0092] Thus, in some embodiments of the disclosure, gypsum slurry may include one or more pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure, as well as one or more other types of starches. Other starches can include, for example, pregelatinized starches having viscosity below 20 centipoise and/or above 300 centipoise. One example is pregelatinized corn starch (e.g., having a viscosity over 500 centipoise, such as about 773 centipoise). The other starches may also be in the form of, e.g., non-pregelatinized starches, such as acid-modified starches, as well as alkylated starches, e.g., ethylated starches, which are not gelatinized, etc. The combination of starches may be

pre-mixed (e.g., in a dry mix, optionally with other components such as stucco, etc., or in a wet mix with other wet ingredients) before addition to the gypsum slurry, or they can be included in the gypsum slurry one at a time, or any variation thereof. Any suitable proportion of pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure and other starch may be included.

[0093] For example, the starch content of pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure as a percentage of total starch content to be added to gypsum slurry can be, e.g., at least about 10% by weight, such as at least about 20%, at least about 30%, at least about 40%, at least about 50%, at least about 60%, at least about 70%, at least about 80%, at least about 90%, at least about 95%, at least about 99%, at least about 100%, or any range in between). In some embodiments, the ratio of pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure to other starch can be about 25:75, about 30:70, about 35:65, about 50:50, about 65:35, about 70:30, about 75:25, etc.

[0094] In addition to the starch component, the slurry is formulated to include water, stucco, foaming agent (sometimes referred to simply as "foam"), and other additives as desired, in some embodiments. Surprisingly and unexpectedly, in accordance with some embodiments, particularly those exhibiting a mid-range viscosity, it has been found that the amount of water needed to be added to maintain the slurry fluidity at the same level it would be without the pregelatinized, partially hydrolyzed starch prepared in an extruder in accordance with embodiments of the disclosure, is less than the increase in the amount of water needed when using a starch prepared according to a different method. The stucco can be in the form of calcium sulfate alpha hemihydrate, calcium sulfate beta hemihydrate, and/or calcium sulfate anhydrite. The stucco can be fibrous or non-fibrous. Foaming agent can be included to form an air void distribution within the continuous crystalline matrix of set gypsum. In some embodiments, the foaming agent comprises a major weight portion of unstable component, and a minor weight portion of stable component (e.g., where unstable and blend of stable/unstable are combined). The weight ratio of unstable component to stable component is effective to form an air void distribution within the set gypsum core. See, e.g., U.S. Patents 5,643,510; 6,342,284; and 6,632,550.

[0095] It has been found that suitable void distribution and wall thickness (independently) can be effective to enhance strength, especially in lower density board (e.g., below about 35 pcf). See, e.g., US 2007/0048490 and US 2008/0090068. Evaporative water

voids, generally having voids of about 5 μm or less in diameter, also contribute to the total void distribution along with the aforementioned air (foam) voids. In some embodiments, the volume ratio of voids with a pore size greater than about 5 microns to the voids with a pore size of about 5 microns or less, is from about 0.5:1 to about 9:1, such as, for example, from about 0.7:1 to about 9:1, from about 0.8:1 to about 9:1, from about 1.4:1 to about 9:1, from about 1.8:1 to about 9:1, from about 2.3:1 to about 9:1, from about 0.7:1 to about 6:1, from about 1.4:1 to about 6:1, from about 1.8:1 to about 6:1, from about 0.7:1 to about 4:1, from about 1.4:1 to about 4:1, from about 1.8:1 to about 4:1, from about 0.5:1 to about 2.3:1, from about 0.7:1 to about 2.3:1, from about 0.8:1 to about 2.3:1, from about 1.4:1 to about 2.3:1, from about 1.8:1 to about 2.3:1, etc. In some embodiments, the foaming agent is present in the slurry, e.g., in an amount of less than about 0.5% by weight of the stucco such as about 0.01% to about 0.5%, about 0.01% to about 0.4%, about 0.01% to about 0.3%, about 0.01% to about 0.2%, about 0.01% to about 0.1%, about 0.02% to about 0.4%, about 0.02% to about 0.3%, about 0.02% to about 0.2%, etc., all by weight of the stucco.

[0096] Additives such as accelerator (e.g., wet gypsum accelerator, heat resistant accelerator, and climate stabilized accelerator) and retarder are well known and can be included, in some embodiments. See, e.g., U.S. Patents 3,573,947 and 6,409,825. In some embodiments where accelerator and/or retarder are included, the accelerator and/or retarder each can be in the gypsum slurry in an amount on a solid basis of, e.g., from about 0% to about 10% by weight of the stucco (e.g., about 0.1% to about 10%), such as, for example, from about 0% to about 5% by weight of the stucco (e.g., about 0.1% to about 5%). Other additives as desired may be included, e.g., to impart strength to enable lower weight product with sufficient strength, to avoid permanent deformation, to promote green strength, for example, as the product is setting on the conveyor traveling down a manufacturing line, to promote fire resistance, to promote water resistance, etc.

[0097] For example, the slurry can optionally include at least one dispersant to enhance fluidity in some embodiments. Like the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure and other ingredients, the dispersants may be included in a dry form with other dry ingredients and/or in a liquid form with other liquid ingredients in the core slurry. Examples of dispersants include naphthalenesulfonates, such as polynaphthalenesulfonic acid and its salts (polynaphthalenesulfonates) and derivatives, which are condensation products of naphthalenesulfonic acids and formaldehyde; as well as polycarboxylate dispersants, such as polycarboxylic ethers, for example, PCE211, PCE111,

1641, 1641F, or PCE 2641-Type Dispersants, e.g., MELFLUX 2641F, MELFLUX 2651F, MELFLUX 1641F, MELFLUX 2500L dispersants (BASF), and COATEX Ethacryl M, available from Coatex, Inc.; and/or lignosulfonates or sulfonated lignin. Lignosulfonates are water-soluble anionic polyelectrolyte polymers, byproducts from the production of wood pulp using sulfite pulping. One example of a lignin useful in the practice of principles of embodiments of the present disclosure is Marasperse C-21 available from Reed Lignin Inc.

[0098] Lower molecular weight dispersants are generally preferred. Lower molecular weight naphthalenesulfonate dispersants are favored because they trend to a lower water demand than the higher viscosity, higher molecular weight dispersants. Thus, molecular weights from about 3,000 to about 10,000 (e.g., about 8,000 to about 10,000) are preferred. As another illustration, for PCE211 type dispersants, in some embodiments, the molecular weight can be from about 20,000 to about 60,000, which exhibit less retardation than dispersants having molecular weight above 60,000.

[0099] One example of a naphthalenesulfonate is DILOFLO, available from GEO Specialty Chemicals. DILOFLO is a 45% naphthalenesulfonate solution in water, although other aqueous solutions, for example, in the range of about 35% to about 55% by weight solids content, are also readily available. Naphthalenesulfonates can be used in dry solid or powder form, such as LOMAR D, available from GEO Specialty Chemicals, for example. Another exemplary naphthalenesulfonate is DAXAD, available from Hampshire Chemical Corp.

[00100] If included, the dispersant can be included in any suitable (solids/solids) amount, such as, for example, from about 0.1% to about 5% by weight based on the weight of the stucco, e.g., from about 0.1% to about 4%, from about 0.1% to about 3%, from about 0.2% to about 3%, from about 0.5% to about 3%, from about 0.5% to about 2.5%, from about 0.5% to about 2%, from about 0.5% to about 1.5%, etc.

[0100] In some embodiments, one or more phosphate-containing compounds can also be optionally included in the slurry, if desired. For example, phosphate-containing components useful in some embodiments include water-soluble components and can be in the form of an ion, a salt, or an acid, namely, condensed phosphoric acids, each of which comprises two or more phosphoric acid units; salts or ions of condensed phosphates, each of which comprises two or more phosphate units; and monobasic salts or monovalent ions of orthophosphates as well as water-soluble acyclic polyphosphate salt. See, e.g., U.S. Patents 6,342,284; 6,632,550; 6,815,049; and 6,822,033.

[0101] Phosphate compositions if added in some embodiments can enhance green strength, resistance to permanent deformation (e.g., sag), dimensional stability, etc. Trimetaphosphate compounds can be used, including, for example, sodium trimetaphosphate, potassium trimetaphosphate, lithium trimetaphosphate, and ammonium trimetaphosphate. Sodium trimetaphosphate (STMP) is preferred, although other phosphates may be suitable, including for example sodium tetrametaphosphate, sodium hexametaphosphate having from about 6 to about 27 repeating phosphate units and having the molecular formula $\text{Na}_{n+2}\text{P}_n\text{O}_{3n+1}$ wherein $n=6-27$, tetrapotassium pyrophosphate having the molecular formula $\text{K}_4\text{P}_2\text{O}_7$, trisodium dipotassium tripolyphosphate having the molecular formula $\text{Na}_3\text{K}_2\text{P}_3\text{O}_{10}$, sodium tripolyphosphate having the molecular formula $\text{Na}_5\text{P}_3\text{O}_{10}$, tetrasodium pyrophosphate having the molecular formula $\text{Na}_4\text{P}_2\text{O}_7$, aluminum trimetaphosphate having the molecular formula $\text{Al}(\text{PO}_3)_3$, sodium acid pyrophosphate having the molecular formula $\text{Na}_2\text{H}_2\text{P}_2\text{O}_7$, ammonium polyphosphate having 1,000-3,000 repeating phosphate units and having the molecular formula $(\text{NH}_4)_{n+2}\text{P}_n\text{O}_{3n+1}$ wherein $n=1,000-3,000$, or polyphosphoric acid having two or more repeating phosphoric acid units and having the molecular formula $\text{H}_{n+2}\text{P}_n\text{O}_{3n+1}$ wherein n is two or more.

[0102] The phosphate can be included in some embodiments in a dry form or in a form in water (e.g., a phosphate solution from about 5% to about 20%, such as about a 10% solution). If included, the phosphate can be in any suitable amount (solids/solids basis), such as from about 0.01% to about 0.5% by weight based on the weight of stucco, e.g., from about 0.03% to about 0.4%, from about 0.1% to about 0.3%, or from about 0.12% to about 0.4% by weight based on the weight of stucco.

[0103] Suitable additives for fire-resistant (e.g., fire-rated) and/or water resistant product can also optionally be included, including, e.g., siloxanes for water resistance. Examples of suitable fire resistant additives include high expansion particulates, high efficiency heat sink additives, fibers, or the like, or any combination thereof, as described in U.S. Patent 8,323,785, which description of such additives is hereby incorporated by reference. Vermiculite, aluminum trihydrate, glass fibers, and a combination thereof can be used in some embodiments.

[0104] For example, the high expansion particulates useful in accordance with some embodiments can exhibit a volume expansion after heating for one hour at about 1560 °F (about 850 °C) of about 300% or more of their original volume. In some embodiments, high expansion vermiculites can be used that have a volume expansion of about 300% to about

380% of their original volume after being placed for one hour in a chamber having a temperature of about 1560 °F (about 850 °C). If included, high expansion particulate, such as vermiculite, can be present in any suitable amount. In some embodiments, it is present in an amount from about 1% to about 10%, e.g., about 3% to about 6%, by weight of stucco.

[0105] Aluminum trihydrate (ATH), also known as alumina trihydrate and hydrated alumina, can increase fire resistance due to its crystallized or compound water content. ATH is a suitable example of a heat sink additive. Such high efficiency heat sink (HEHS) additives have a heat sink capacity that exceeds the heat sink capacity of comparable amounts of gypsum dihydrate in the temperature range causing the dehydration and release of water vapor from the gypsum dihydrate component of the panel core. Such additives typically are selected from compositions, such as aluminum trihydrate or other metal hydroxides that decompose, releasing water vapor in the same or similar temperature ranges as does gypsum dihydrate. While other HEHS additives (or combinations of HEHS additives) with increased heat sink efficiency relative to comparable amounts of gypsum dihydrate can be used, preferred HEHS additives provide a sufficiently-increased heat sink efficiency relative to gypsum dihydrate to offset any increase in weight or other undesired properties of the HEHS additives when used in a gypsum panel intended for fire rated or other high temperature applications. If included, heat sink additive, such as ATH, is present in any suitable amount. In some embodiments, it is included in an amount from about 1% to about 8%, e.g., from about 2% to about 4% by weight of stucco.

[0106] The fibers may include mineral fibers, carbon and/or glass fibers and mixtures of such fibers, as well as other comparable fibers providing comparable benefits to the panel. In some embodiments, glass fibers are incorporated in the gypsum core slurry and resulting crystalline core structure. The glass fibers in some of such embodiments can have an average length of about 0.5 to about 0.75 inches and a diameter of about 11 to about 17 microns. In other embodiments, such glass fibers may have an average length of about 0.5 to about 0.675 inches and a diameter of about 13 to about 16 microns. If included, fibers, such as glass fibers, is present in any suitable amount, such as, from about 0.1% to about 3%, e.g., from about 0.5% to about 1% by weight of stucco.

[0107] In some embodiments, the gypsum board product exhibits fire resistance beyond what is found in conventional wallboard. To achieve fire resistance, the board can optionally be formed from certain additives that enhance fire resistance in the final board product, as

described herein. Some fire resistant board is considered "fire rated" when the board passes certain tests while in an assembly.

[0108] In accordance with some embodiments, gypsum board is configured to meet or exceed a fire rating pursuant to the fire containment and structural integrity requirements of UL U305, U419, U423, and/or equivalent fire test procedures and standards, e.g., where the board contains fire resistant additives discussed herein. The present disclosure thus provides gypsum board (e.g., reduced weight and density at thickness of 1/2 inch or 5/8 inch), and methods for making the same, that are capable of satisfying fire ratings (e.g., 17 min., 20 min., 30 min., 3/4 hour, one-hour, two-hour, etc.) pursuant to the fire containment and structural integrity procedures and standards of various UL standards such as those discussed herein, in some embodiments.

[0109] The gypsum board can be tested, e.g., in an assembly according to Underwriters Laboratories UL U305, U419, and U423 specifications and any other fire test procedure that is equivalent to any one of those fire test procedures. It should be understood that reference made herein to a particular fire test procedure of Underwriters Laboratories, such as, UL U305, U419, and U423, for example, also includes a fire test procedure, such as one promulgated by any other entity, that is equivalent to the particular UL standard in question.

[0110] For example, the gypsum board in some embodiments is effective to inhibit the transmission of heat through an assembly constructed in accordance with any one of UL Design Numbers U305, U419 or U423, the assembly having a first side with a single layer of gypsum panels and a second side with a single layer of gypsum panels. Surfaces of gypsum boards on the first side of the assembly are heated in accordance with the time-temperature curve of ASTM E119-09a, while surfaces of gypsum panels on the second side of the assembly are provided with temperature sensors pursuant to ASTM E119-09a. In some embodiments of fire resistant board, when heated, the maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 50 minutes, and/or or the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 50 minutes. In some embodiments, the board has a density of about 40 pounds per cubic foot or less. Desirably, the board has good strength as described herein, such as a core hardness of at least about 11 pounds (5 kg), e.g., at least about 13 pounds (5.9 kg), or at least about 15 pounds (6.8 kg).

[0111] In some embodiments, when the surfaces on the first side of the assembly of fire resistant gypsum board with fire resistant additive in the concentrated layer are heated, the

maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 55 minutes, and/or the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 55 minutes. In other embodiments, when the surfaces of gypsum board on the first side of the assembly are heated the maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 60 minutes and/or the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 60 minutes. In other embodiments, when the surfaces of gypsum panels on the first side of the assembly are heated, the maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 50 minutes, and/or the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 50 minutes. In other embodiments, when the surfaces of gypsum panels on the first side of the assembly are heated, the maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 55 minutes, and/or the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 55 minutes. In other embodiments, when the surfaces of gypsum panels on the first side of the assembly are heated, the maximum single value of the temperature sensors is less than about 325 °F plus ambient temperature after about 60 minutes, and the average value of the temperature sensors is less than about 250 °F plus ambient temperature after about 60 minutes.

[0112] In some embodiments, fire resistant gypsum board with fire resistant additive in the concentrated layer is effective to inhibit the transmission of heat through the assembly when constructed in accordance with UL Design Number U305 so as to achieve a one hour fire rating under ASTM E119-09a. In some embodiments, the board is effective to inhibit the transmission of heat through the assembly when constructed in accordance with UL Design Number U419 so as to achieve a one hour fire rating under ASTM E119-09a. In some embodiments, the gypsum board is effective to inhibit the transmission of heat through the assembly when constructed in accordance with UL Design Number U423 so as to achieve a one hour fire rating under ASTM E119-09a. In some embodiments, the board has a Thermal Insulation Index (TI) of about 20 minutes or greater and/or a High Temperature Shrinkage (S) of about 10%. In some embodiments, the board has a ratio of High Temperature Thickness Expansion (TE) to S (TE/S) of about 0.2 or more.

[0113] Furthermore, in some embodiments, the gypsum board can be in the form of reduced weight and density, fire resistant gypsum board with High Temperature Shrinkage of

less than about 10% in the x-y directions (width-length) and High Temperature Thickness Expansion in the z-direction (thickness) of greater than about 20% when heated to about 1560 °F (850 °C). In yet other embodiments, when used in wall or other assemblies, such assemblies have fire testing performance comparable to assemblies made with heavier, denser commercial fire rated panels. In some embodiments, the High Temperature Shrinkage of the panels typically is less than about 10% in the x-y directions (width-length). In some embodiments, the ratio of z-direction High Temperature Thickness Expansion to x-y High Temperature Shrinkage is at least about 2 to over about 17 at 1570 °F (855 °C).

[0114] In some embodiments, a fire resistant gypsum board formed according to principles of the present disclosure, and the methods for making same, can provide a panel that exhibits an average shrink resistance of about 85% or greater when heated at about 1800 °F (980 °C) for one hour. In other embodiments, the gypsum board exhibits an average shrink resistance of about 75% or greater when heated at about 1800 °F (980 °C) for one hour.

[0115] The board of some fire-related product according to the disclosure can have a Thermal Insulation Index (TI) of about 17 minutes or greater, e.g., about 20 minutes or greater, about 30 minutes or greater, about 45 minutes or greater, about 60 minutes or greater, etc.; and/or a High Temperature Shrinkage (at temperatures of about 1560°F (850 °C)) of less than about 10% in the x-y directions and expansion in the z-direction of greater than about 20%. Fire-resistant embodiments can have a relatively higher density, e.g., the gypsum board can have a density (D) of about 40 pounds per cubic foot or less (e.g., 37 pcf or less, 35 pcf or less, 33 pcf or less, or the other densities listed herein). The gypsum layers between the cover sheets can be effective to provide the gypsum board with a ratio of TI/D of about 0.6 minutes/pounds per cubic foot (0.038 minutes/(kg/m³)) or more.

[0116] The fire or water resistance additives can be included in any suitable amount as desired and effective. For example, if included, the fire or water resistance additives can be in an amount from about 0.5% to about 10% by weight of the stucco, such as from about 1% to about 10%, about 1% to about 8%, about 2% to about 10%, about 2% to about 8% by weight of the stucco, etc.

[0117] If included, in some embodiments, the siloxane preferably is added in the form of an emulsion. The slurry is then shaped and dried under conditions which promote the polymerization of the siloxane to form a highly cross-linked silicone resin. A catalyst which promotes the polymerization of the siloxane to form a highly cross-linked silicone resin can

be added to the gypsum slurry. In some embodiments, solventless methyl hydrogen siloxane fluid sold under the name SILRES BS 94 by Wacker-Chemie GmbH (Munich, Germany) can be used as the siloxane. This product is a siloxane fluid containing no water or solvents. It is contemplated that about 0.3% to about 1.0% of the BS 94 siloxane may be used in some embodiments, based on the weight of the dry ingredients. For example, in some embodiments, it is preferred to use from about 0.4% to about 0.8% of the siloxane based on the dry stucco weight.

[0118] The slurry formulation can be made with any suitable water/stucco ratio, e.g., from about 0.4 to about 1.3. However, because the pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure reduce the amount of water required to be added to the slurry to accommodate them, as compared with other starches (e.g., conventional pregelatinized starch prepared according to a different method), the slurry can be formulated with a water/stucco ratio input that is lower in some embodiments than what is conventional for other starch-containing gypsum slurries, especially at low weight/density. For example, in some embodiments, the water/stucco ratio can be from about 0.4 to about 1.1, from about 0.4 to about 0.9, from about 0.4 to about 0.85, from about 0.45 to about 0.85, from about 0.55 to about 0.85, from about 0.55 to about 0.8, from about 0.6 to about 0.9, from about 0.6 to about 0.85, from about 0.6 to about 0.8, etc.

[0119] The cover sheets can be formed of any suitable material and basis weight. Advantageously, board core formed from slurry comprising pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure provides sufficient strength in board even with lower basis weight cover sheets such as, for example, less than 45 lbs/MSF (e.g., about 33 lbs/MSF to 45 lbs/MSF) even for lower weight board (e.g., having a density of about 35 pcf or below) in some embodiments. However, if desired, in some embodiments, heavier basis weights can be used, e.g., to further enhance nail pull resistance or to enhance handling, e.g., to facilitate desirable "feel" characteristics for end-users.

[0120] In some embodiments, to enhance strength (e.g., nail pull strength), especially for lower density board, one or both of the cover sheets can be formed from paper and have a basis weight of, for example, at least about 45 lbs/MSF (e.g., from about 45 lbs/MSF to about 65 lbs/MSF, from about 45 lbs/MSF to about 60 lbs/MSF, from about 45 lbs/MSF to about 55 lbs/MSF, from about 50 lbs/MSF to about 65 lbs/MSF, from about 50 lbs/MSF to about 60 lbs/MSF, etc.). If desired, in some embodiments, one cover sheet (e.g., the "face" paper

side when installed) can have aforementioned higher basis weight, e.g., to enhance nail pull resistance and handling, while the other cover sheet (e.g., the “back” sheet when the board is installed) can have somewhat lower weight basis if desired (e.g., weight basis of less than about 45 lbs/MSF, e.g., from about 33 lbs/MSF to about 45 lbs/MSF or from about 33 lbs/MSF to about 40 lbs/MSF).

[0121] Board weight is a function of thickness. Since boards are commonly made at varying thicknesses, board density is used herein as a measure of board weight. As noted herein, preferred embodiments of the disclosure have particular utility at lower densities where the enhanced strength provided by the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure advantageously enables the use of lower weight board with good strength and lower water demand than board made from other starches. For example, the elevated amounts of pregelatinized starch in accordance with preferred embodiments can be used to prepare ultra light weight board (e.g., at a density of 31 pcf or less) with good strength properties. However, advantages of the pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can be seen across various board densities, e.g., about 40 pcf or less, such as from about 20 pcf to about 40 pcf, from about 24 pcf to about 37 pcf, etc.

[0122] In other embodiments, board density can be from about 20 pcf to about 35 pcf, e.g., from about 20 pcf to about 34 pcf, from about 20 pcf to about 33 pcf, from about 20 pcf to about 32 pcf, from about 20 pcf to about 31 pcf, from about 20 pcf to about 30 pcf, from about 20 pcf to about 29 pcf, from about 20 pcf to about 28 pcf, from about 20 pcf to about 27 pcf, from about 20 pcf to about 26 pcf, from about 20 pcf to about 25 pcf, from about 20 pcf to about 24 pcf, from about 20 pcf to about 23 pcf, from about 20 pcf to about 22 pcf, from about 21 pcf to about 35 pcf, from about 21 pcf to about 34 pcf, from about 21 pcf to about 33 pcf, from about 21 pcf to about 32 pcf, from about 21 pcf to about 31 pcf, from about 21 pcf to about 30 pcf, from about 21 pcf to about 29 pcf, from about 21 pcf to about 28 pcf, from about 21 pcf to about 27 pcf, from about 21 pcf to about 26 pcf, from about 21 pcf to about 25 pcf, from about 21 pcf to about 24 pcf, from about 21 pcf to about 23 pcf, from about 24 pcf to about 35 pcf, from about 24 pcf to about 34 pcf, from about 24 pcf to about 33 pcf, from about 24 pcf to about 32 pcf, from about 24 pcf to about 31 pcf, from about 24 pcf to about 30 pcf, from about 24 pcf to about 29 pcf, from about 24 pcf to about 28 pcf, from about 24 pcf to about 27 pcf, or from about 24 pcf to about 26 pcf.

[0123] The pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure can be added to slurry to provide strength enhancement to product according to the disclosure, which can be especially beneficial at lower weight/density. For example, in some embodiments, the board made according to embodiments of the disclosure has a compressive strength of at least about 400 psi (2,750 kPa) at a density of 29 pcf as tested according to the method set forth in Example 4. Advantageously, in various embodiments at various board densities as described herein, the board produced by the method according to some embodiments can be prepared to have a compressive strength of at least about 170 psi (1,170 kPa), e.g., from about 170 psi to about 1,000 psi (6,900 kPa), from about 170 psi to about 900 psi (6,200 kPa), from about 170 psi to about 800 psi (5,500 kPa), from about 170 psi to about 700 psi (4,800 kPa), from about 170 psi to about 600 psi (4,100 kPa), from about 170 psi to about 500 psi (3,450 kPa), from about 170 psi to about 450 psi (3,100 kPa), from about 170 psi to about 400 psi (2,760 kPa), from about 170 psi to about 350 psi (2,410 kPa), from about 170 psi to about 300 psi (2,070 kPa), or from about 170 psi to about 250 psi (1,720 kPa). In some embodiments, the board has a compressive strength of at least about 450 psi (3,100 kPa), at least about 500 psi (3,450 kPa), at least about 550 psi (3,800 kPa), at least about 600 psi (4,100 kPa), at least about 650 psi (4,500 kPa), at least about 700 psi (4,800 kPa), at least about 750 psi (5,200 kPa), at least about 800 psi (5,500 kPa), at least about 850 psi (5,850 kPa), at least about 900 psi (6,200 kPa), at least about 950 psi (6,550 kPa), or at least about 1,000 psi (6,900 kPa). In addition, in some embodiments, the compressive strength can be bound by any two of the foregoing points. For example, the compressive strength can be between about 450 psi and about 1,000 psi (e.g., between about 500 psi and about 900 psi, between about 600 psi and about 800 psi, etc.).

[0124] In some embodiments, board made according to the disclosure meets test protocols according to ASTM Standard C473-10. For example, in some embodiments, when the board is cast at a thickness of $\frac{1}{2}$ inch, the board has a nail pull resistance of at least about 65 lb_f (pounds force) as determined according to ASTM C473-10, Method B, e.g., at least about 68 lb_f, at least about 70 lb_f, at least about 72 lb_f, at least about 75 lb_f, at least about 77 lb_f, etc. In various embodiments, the nail pull resistance can be from about 68 lb_f to about 100 lb_f, e.g., from about 68 lb_f to about 95 lb_f, from about 68 lb_f to about 90 lb_f, from about 68 lb_f to about 85 lb_f, from about 68 lb_f to about 80 lb_f, from about 68 lb_f to about 77 lb_f, from about 68 lb_f to about 75 lb_f, from about 68 lb_f to about 72 lb_f, from about 68 lb_f to about

70 lb_f, from about 70 lb_f to about 100 lb_f, from about 70 lb_f to about 95 lb_f, from about 70 lb_f to about 90 lb_f, from about 70 lb_f to about 85 lb_f, from about 70 lb_f to about 80 lb_f, from about 70 lb_f to about 77 lb_f, from about 70 lb_f to about 75 lb_f, from about 70 lb_f to about 72 lb_f, from about 72 lb_f to about 100 lb_f, from about 72 lb_f to about 95 lb_f, from about 72 lb_f to about 90 lb_f, from about 72 lb_f to about 85 lb_f, from about 72 lb_f to about 80 lb_f, from about 72 lb_f to about 77 lb_f, from about 72 lb_f to about 75 lb_f, from about 75 lb_f to about 100 lb_f, from about 75 lb_f to about 95 lb_f, from about 75 lb_f to about 90 lb_f, from about 75 lb_f to about 85 lb_f, from about 75 lb_f to about 80 lb_f, from about 75 lb_f to about 77 lb_f, from about 77 lb_f to about 100 lb_f, from about 77 lb_f to about 95 lb_f, from about 77 lb_f to about 90 lb_f, from about 77 lb_f to about 85 lb_f, or from about 77 lb_f to about 80 lb_f.

[0125] With respect to flexural strength, in some embodiments, when cast in a board of ½ inch thickness, the board has a flexural strength of at least about 36 lb_f in a machine direction (e.g., at least about 38 lb_f, at least about 40 lb_f, etc) and/or at least about 107 lb_f (e.g., at least about 110 lb_f, at least about 112 lb_f, etc.) in a cross-machine direction as determined according to the ASTM standard C473, method B. In various embodiments, the board can have a flexural strength in a machine direction of from about 36 lb_f to about 60 lb_f, e.g., from about 36 lb_f to about 55 lb_f, from about 36 lb_f to about 50 lb_f, from about 36 lb_f to about 45 lb_f, from about 36 lb_f to about 40 lb_f, from about 36 lb_f to about 38 lb_f, from about 38 lb_f to about 60 lb_f, from about 38 lb_f to about 55 lb_f, from about 38 lb_f to about 50 lb_f, from about 38 lb_f to about 45 lb_f, from about 38 lb_f to about 40 lb_f, from about 40 lb_f to about 60 lb_f, from about 40 lb_f to about 55 lb_f, from about 40 lb_f to about 50 lb_f, or from about 40 lb_f to about 45 lb_f. In various embodiments, the board can have a flexural strength in a cross-machine direction of from about 107 lb_f to about 130 lb_f, e.g., from about 107 lb_f to about 125 lb_f, from about 107 lb_f to about 120 lb_f, from about 107 lb_f to about 115 lb_f, from about 107 lb_f to about 112 lb_f, from about 107 lb_f to about 110 lb_f, from about 110 lb_f to about 130 lb_f, from about 110 lb_f to about 125 lb_f, from about 110 lb_f to about 120 lb_f, from about 110 lb_f to about 115 lb_f, from about 110 lb_f to about 112 lb_f, from about 112 lb_f to about 130 lb_f, from about 112 lb_f to about 125 lb_f, from about 112 lb_f to about 120 lb_f, or from about 112 lb_f to about 115 lb_f.

[0126] In addition, in some embodiments, board can have an average core hardness of at least about 11 lb_f, e.g., at least about 12 lb_f, at least about 13 lb_f, at least about 14 lb_f, at least about 15 lb_f, at least about 16 lb_f, at least about 17 lb_f, at least about 18 lb_f, at least about 19 lb_f, at least about 20 lb_f, at least about 21 lb_f, or at least about 22 lb_f, as determined according

to ASTM C473-10, method B. In some embodiments, board can have a core hardness of from about 11 lb_f to about 25 lb_f, e.g., from about 11 lb_f to about 22 lb_f, from about 11 lb_f to about 21 lb_f, from about 11 lb_f to about 20 lb_f, from about 11 lb_f to about 19 lb_f, from about 11 lb_f to about 18 lb_f, from about 11 lb_f to about 17 lb_f, from about 11 lb_f to about 16 lb_f, from about 11 lb_f to about 15 lb_f, from about 11 lb_f to about 14 lb_f, from about 11 lb_f to about 13 lb_f, from about 11 lb_f to about 12 lb_f, from about 12 lb_f to about 25 lb_f, from about 12 lb_f to about 22 lb_f, from about 12 lb_f to about 21 lb_f, from about 12 lb_f to about 20 lb_f, from about 12 lb_f to about 19 lb_f, from about 12 lb_f to about 18 lb_f, from about 12 lb_f to about 17 lb_f, from about 12 lb_f to about 16 lb_f, from about 12 lb_f to about 15 lb_f, from about 12 lb_f to about 14 lb_f, from about 12 lb_f to about 13 lb_f, from about 13 lb_f to about 25 lb_f, from about 13 lb_f to about 22 lb_f, from about 13 lb_f to about 21 lb_f, from about 13 lb_f to about 20 lb_f, from about 13 lb_f to about 19 lb_f, from about 13 lb_f to about 18 lb_f, from about 13 lb_f to about 17 lb_f, from about 13 lb_f to about 16 lb_f, from about 13 lb_f to about 15 lb_f, from about 13 lb_f to about 14 lb_f, from about 14 lb_f to about 25 lb_f, from about 14 lb_f to about 22 lb_f, from about 14 lb_f to about 21 lb_f, from about 14 lb_f to about 20 lb_f, from about 14 lb_f to about 19 lb_f, from about 14 lb_f to about 18 lb_f, from about 14 lb_f to about 17 lb_f, from about 14 lb_f to about 16 lb_f, from about 14 lb_f to about 15 lb_f, from about 15 lb_f to about 25 lb_f, from about 15 lb_f to about 22 lb_f, from about 15 lb_f to about 21 lb_f, from about 15 lb_f to about 20 lb_f, from about 15 lb_f to about 19 lb_f, from about 15 lb_f to about 18 lb_f, from about 15 lb_f to about 17 lb_f, from about 15 lb_f to about 16 lb_f, from about 16 lb_f to about 25 lb_f, from about 16 lb_f to about 22 lb_f, from about 16 lb_f to about 21 lb_f, from about 16 lb_f to about 20 lb_f, from about 16 lb_f to about 19 lb_f, from about 16 lb_f to about 18 lb_f, from about 16 lb_f to about 17 lb_f, from about 17 lb_f to about 25 lb_f, from about 17 lb_f to about 22 lb_f, from about 17 lb_f to about 21 lb_f, from about 17 lb_f to about 20 lb_f, from about 17 lb_f to about 19 lb_f, from about 17 lb_f to about 18 lb_f, from about 18 lb_f to about 25 lb_f, from about 18 lb_f to about 22 lb_f, from about 18 lb_f to about 21 lb_f, from about 18 lb_f to about 20 lb_f, from about 18 lb_f to about 19 lb_f, from about 19 lb_f to about 25 lb_f, from about 19 lb_f to about 22 lb_f, from about 19 lb_f to about 21 lb_f, from about 19 lb_f to about 20 lb_f, from about 21 lb_f to about 25 lb_f, from about 21 lb_f to about 22 lb_f, or from about 22 lb_f to about 25 lb_f.

[0127] Due at least in part to the mid-range viscosity characteristic that results in some embodiments of the disclosure, these standards (e.g., nail pull resistance, flexural strength, and core hardness) can be met even with respect to ultra light density board (e.g., about 31 pcf or less) as described herein.

[0128] It has also been found by the inventors that pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure demonstrate temperature rise set (TRS) hydration rates that are comparable to or surpass those of conventional pregelatinized starches prepared according to a different method. The desired setting time may depend on the formulation, and the desired setting time can be determined by one of ordinary skill in the art depending on plant conditions and available raw materials.

[0129] Product according to embodiments of the disclosure can be made on typical manufacturing lines. For example, board manufacturing techniques are described in, for example, U.S. Patent 7,364,676 and U.S. Patent Application Publication 2010/0247937. Briefly, in the case of gypsum board, the process typically involves discharging a cover sheet onto a moving conveyor. Since gypsum board is normally formed “face down,” this cover sheet is the “face” cover sheet in such embodiments.

[0130] Dry and/or wet components of the gypsum slurry are fed to a mixer (e.g., pin mixer), where they are agitated to form the gypsum slurry. The mixer comprises a main body and a discharge conduit (e.g., a gate-canister-boot arrangement as known in the art, or an arrangement as described in U.S. Patents 6,494,609 and 6,874,930). In some embodiments, the discharge conduit can include a slurry distributor with either a single feed inlet or multiple feed inlets, such as those described in U.S. Patent Application Publication 2012/0168527 A1 (Application No. 13/341,016) and U.S. Patent Application Publication 2012/0170403 A1 (Application No. 13/341,209), for example. In those embodiments, using a slurry distributor with multiple feed inlets, the discharge conduit can include a suitable flow splitter, such as those described in U.S. Patent Application Publication 2012/0170403 A1. Foaming agent can be added in the discharge conduit of the mixer (e.g., in the gate as described, for example, in U.S. Patents 5,683,635 and 6,494,609) or in the main body if desired. Slurry discharged from the discharge conduit after all ingredients have been added, including foaming agent, is the primary gypsum slurry and will form the board core. This board core slurry is discharged onto the moving face cover sheet.

[0131] The face cover sheet may bear a thin skim coat in the form of a relatively dense layer of slurry. Also, hard edges, as known in the art, can be formed, e.g., from the same slurry stream forming the face skim coat. In embodiments where foam is inserted into the discharge conduit, a stream of secondary gypsum slurry can be removed from the mixer body to form the dense skim coat slurry, which can then be used to form the face skim coat and hard edges as known in the art. If included, normally the face skim coat and hard edges are

deposited onto the moving face cover sheet before the core slurry is deposited, usually upstream of the mixer. After being discharged from the discharge conduit, the core slurry is spread, as necessary, over the face cover sheet (optionally bearing skim coat) and covered with a second cover sheet (typically the "back" cover sheet) to form a wet assembly in the form of a sandwich structure that is a board precursor to the final product. The second cover sheet may optionally bear a second skim coat, which can be formed from the same or different secondary (dense) gypsum slurry as for the face skim coat, if present. The cover sheets may be formed from paper, fibrous mat or other type of material (e.g., foil, plastic, glass mat, non-woven material such as blend of cellulosic and inorganic filler, etc.).

[0132] The wet assembly thereby provided is conveyed to a forming station where the product is sized to a desired thickness (e.g., via forming plate), and to one or more knife sections where it is cut to a desired length. The wet assembly is allowed to harden to form the interlocking crystalline matrix of set gypsum, and excess water is removed using a drying process (e.g., by transporting the assembly through a kiln). Surprisingly and unexpectedly, it has been found that board prepared according to the disclosure with pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure requires significantly less time in a drying process because of the low water demand characteristic of the starch. This is advantageous because it reduce energy costs.

[0133] It also is common in the manufacture of gypsum board to use vibration in order to eliminate large voids or air pockets from the deposited slurry. Each of the above steps, as well as processes and equipment for performing such steps, are known in the art.

[0134] The pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can be used in formulating various products, such as, for example, gypsum wallboard, acoustical (e.g., ceiling) tile, joint compound, gypsum-cellulosic fiber products, such as gypsum-wood fiber wallboard, and the like. In some embodiments, such product can be formed from slurry according to embodiments of the disclosure.

[0135] As such, pregelatinized, partially hydrolyzed starch prepared in an extruder in accordance with embodiments of the disclosure can have beneficial effect, as described herein, in product besides paper-faced gypsum board in embodiments of the disclosure. For example, pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure can be used in mat-faced products (e.g., woven) where board cover sheets are in the form of fibrous mats. The mats can optionally bear a finish to reduce water permeability. Other ingredients that can be included in making such mat-faced

product, as well as materials for the fibrous mats and methods of manufacture, are discussed in, e.g., U.S. Patent 8,070,895, as well as U.S. Patent Application Publication 2009/0247937.

[0136] In addition, gypsum-cellulosic product can be in the form of cellulosic host particles (e.g., wood fibers), gypsum, pregelatinized, partially hydrolyzed starch prepared in accordance with embodiments of the disclosure, and other ingredients (e.g., water resistant additives such as siloxanes) as desired. Other ingredients and methods of manufacture are discussed in, e.g., U.S. Patents 4,328,178; 4,239,716; 4,392,896; 4,645,548; 5,320,677; 5,817,262; and 7,413,603.

Illustrative Examples of Embodiments

[0137] In one embodiment, gypsum board comprises a set gypsum core disposed between two cover sheets, the core formed from a slurry comprising stucco, water, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco. The board has a density of about 31 pcf or less and at a thickness of one-half inch (about 1.3 cm), the board meets at least one of the following: a compressive strength of at least about 170 psi, a nail pull resistance of at least about 65 lb_f (about 29 kg), an average core hardness of at least about 11 lb_f (about 5 kg), an average flexural strength of at least about 36 lb_f (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, or other strength property described herein. The nail pull resistance, core hardness, and flexural strength are determined in accordance with ASTM standard C473-10, method B.

[0138] In another embodiment, the pregelatinized starch has a viscosity of from about 20 cP to about 200 cP.

[0139] In another embodiment, the pregelatinized starch has a viscosity from about 30 cP to about 150 cP.

[0140] In another embodiment, the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.

[0141] In another embodiment, the pregelatinized starch is present in an amount of from about 12% to about 25% by weight of the stucco.

[0142] In another embodiment, the board has a density of about 15 pcf to about 31 pcf.

[0143] In another embodiment, the board has a density of about 15 pcf to about 27 pcf.

[0144] In another embodiment, the board has a density of about 19 pcf to about 24 pcf.

[0145] In another embodiment, the board has a compressive strength of from about 170 psi to about 250 psi.

[0146] In another embodiment, the starch has a cold water solubility of at least about 70% (e.g., at least about 75%, at least about 80%, at least about 85%, at least about 90%, at least about 95%, etc.) when measured at 25 °C.

[0147] In another embodiment, the starch has a cold water viscosity of a 10% slurry of the starch in water when measured at 25 °C of from about 60 cP to about 160 cP.

[0148] In another embodiment, a method of making a gypsum wallboard comprises mixing at least water, stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco. The slurry is disposed between a first cover sheet and a second cover sheet to form a wet assembly. The wet assembly is cut into a board and the board is dried. The board has a density of about 31 pcf or less and at a thickness of one-half inch (about 1.3 cm), the board meets at least one of the following: a compressive strength of at least about 170 psi, a nail pull resistance of at least about 65 lb_f (about 29 kg), an average core hardness of at least about 11 lb_f (about 5 kg), an average flexural strength of at least about 36 lb_f (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, or other strength property described herein. The nail pull resistance, core hardness, and flexural strength are determined in accordance with ASTM standard C473-10, method B.

[0149] In another embodiment, the pregelatinized starch has a viscosity from about 30 cP to about 200 cP.

[0150] In another embodiment, the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.

[0151] In another embodiment, the pregelatinized starch is present in an amount of from about 12% to about 25% by weight of the stucco.

[0152] In another embodiment, the board has a density of from about 15 pcf to about 27 pcf.

[0153] In another embodiment, a slurry comprises water, stucco, and pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco; wherein when the slurry is cast and dried as board comprising a set gypsum composition, the set gypsum composition comprises a continuous crystalline matrix

substantially of calcium sulfate dihydrate, wherein said board has a density of about 31 pcf or less, and at a thickness of one-half inch (about 1.3 cm), the board meets at least one of the following: a nail pull resistance of at least about 65 lb_f (about 29 kg), an average core hardness of at least about 11 lb_f (about 5 kg), and an average flexural strength of at least about 36 lb_f (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, the nail pull resistance, the core hardness and the flexural strength determined in accordance with ASTM standard C473-10, method B.

[0154] In another embodiment, the pregelatinized starch has a viscosity from about 30 cP to about 200 cP.

[0155] In another embodiment, the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.

[0156] In another embodiment, the board has a density of from about 15 pcf to about 27 pcf.

[0157] It shall be noted that the preceding are merely examples of embodiments. Other exemplary embodiments are apparent from the entirety of the description herein. It will also be understood by one of ordinary skill in the art that each of these embodiments may be used in various combinations with the other embodiments provided herein.

[0158] The following examples further illustrate the invention but, of course, should not be construed as in any way limiting its scope.

EXAMPLE 1

[0159] This Example illustrates the preparation of pregelatinized, partially hydrolyzed starches.

[0160] Nine pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure were prepared for various tests of specific properties (e.g., viscosity, fluidity, strength). These nine starches (in Compositions 1D-1L) were tested alongside three commercially available starches (in Compositions 1A-1C).

[0161] Wet starch precursors were prepared by mixing a degerminated corn flour commercially available as CCM 260 Yellow Corn Meal from Bunge North America (St. Louis, MO) in an amount of 100 kg, varying amounts of aluminum sulfate (alum), a weak acid that substantially avoids chelating with calcium ions, and/or tartaric acid (less than 20 wt.% of the total of weak acids), and varying amounts of water. The wet starch precursors were fed into a single screw extruder commercially available as Advantage 50 from

American Extrusion International (South Beloit, IL). In the extruder, the wet starch precursors were pregelatinized and acid-modified in a single step, such that they occurred simultaneously.

[0162] Table 2 describes the parameters of the extrusion of the corn flour in the presence of acid. The residence time of extrusion (i.e., the time for pregelatinization and acid-modification) was less than 30 seconds. All percents are based on the total weight of the starch, except moisture, which is based on the total wet weight, expressed as the sum of water, starch, and other additives.

[0163] The resulting pregelatinized, partially hydrolyzed starches were evaluated against a conventional pregelatinized corn starch having a viscosity of 773 centipoise designated Composition 1A (comparative), as well as two low water-demand starches prepared by extrusion of acid-modified corn starches, commercially available as Clinton 277 (ADM, Chicago, IL) and Caliber 159 (Cargill, Wayzata, MN), designated Composition 1B (comparative) and Composition 1C (comparative), respectively.

Table 2

Substrate	Corn Flour
Canola oil	0.25 wt. %
Liquid alum	1 wt. % - 4 wt. %
Tartaric acid	0 wt. % - 0.3 wt. %
Moisture of starch during extrusion	10 wt. % - 20 wt. %
Main screw (RPM)	350
Feed auger speed (RPM)	14
Die temperature (°F)	350-370
Knife speed (RPM)	400-1,000

[0164] Table 3 details the various moisture contents for extrusion and acid contents during extrusion for Compositions 1D-1L. Compositions 1D-1H and 1L were prepared with a moisture content of 16 wt. %, while Compositions 1I-1K were prepared with a moisture content of 13 wt. %. Compositions 1D-1G and Compositions 1I-1L were prepared with liquid alum in an amount of ranging from 1 wt. % to 4 wt. %, while Composition 1H included liquid alum and tartaric acid.

Table 3

Composition	Moisture	Acid
1A	16 wt. %	NA
1B	19 wt. %	NA
1C	19 wt. %	NA
1D	16 wt. %	1 wt. % alum
1E	16 wt. %	2 wt. % alum
1F	16 wt. %	3 wt. % alum
1G	16 wt. %	4 wt. % alum
1H	16 wt. %	2 wt. % alum; 0.3 wt. % tartaric acid
1I	13 wt. %	1 wt. % alum
1J	13 wt. %	2 wt. % alum
1K	13 wt. %	3 wt. % alum
1L	16 wt. %	3 wt. % alum

[0165] Examples 2-4 below test the Compositions described in Table 3 for various properties. In Example 2, Compositions 1B-1L were evaluated with regards to viscosity in amylograph tests. Example 3 tested slurries prepared with one of Compositions 1A, 1D-1I, and 1K-1L for fluidity, which was evaluated by means of a slump test. Compositions 1F and 1L were prepared using the same moisture content and amount of acid, but in Example 3 had different amounts of retarder because fluidity was evaluated at the same setting rate as control 1A.

[0166] This data was then further corroborated by measuring the time to 50% hydration for the slurries. This illustrated how much time it took for the slurries to set. Example 4 tested slurries prepared with Compositions 1A, 1D-1I, and 1K for strength, which was evaluated by means of a compressive strength test described herein.

EXAMPLE 2

[0167] This example illustrates the viscosity of pregelatinized, partially hydrolyzed starches prepared in an extruder in accordance with embodiments of the disclosure. Compositions 1D-1K were tested in comparison to extruded commercially available acid-modified starches (Compositions 1B-1C), specifically with regards to how viscosity changes based on the amount of acid (e.g., alum) and moisture content, defined by the level of moisture of the wet starch that is fed through the extruder.

[0168] In preparation for testing, the Compositions were mixed with water into a starch slurry, such that the starch slurries contained the Compositions in an amount of 10 wt.%. It will be noted that the term "solution" is used when the starch is fully gelatinized and completely dissolved and the term "slurry" is used when the starch is not completely dissolved. Each Composition was then tested for viscosity at different temperatures by the Amylograph technique described herein. The results of the tests were plotted in FIGS. 1 and 2, which are amylograms evaluating the viscosity of pregelatinized, partially hydrolyzed starches at different temperatures by plotting viscosity (left y-axis) and temperature (right y-axis) versus time (x-axis). The temperature curve is overlaid against each sample. The same temperature profile was used for each sample. The other curves show the viscosity of the starches.

[0169] The initial viscosity at 25°C was an indicator of the fluidity of a slurry system containing any one of Compositions 1B-1K. 25°C is the temperature at which the starch will be mixed with stucco and other ingredients to make board. At this temperature, furthermore, the viscosity of the starch is negatively correlated with the fluidity of the stucco slurry.

[0170] The viscosity at trough (93°C) was an indicator of the molecular weight of any one of Compositions 1B-1K. At a temperature of 93°C, the starch molecules are completely dissolved in water. The viscosity of the starch solutions at 93°C is positively correlated with the molecular weight of the starch, which results from partial hydrolysis.

[0171] FIG. 1 is an amylogram plotting viscosity (left y-axis) and temperature (right y-axis) over a fifty minute period (x-axis). Comparative Compositions 1B and 1C and Compositions 1D-1H, as described herein, were mixed into starch solutions in an amount of 10% by weight based on the weight of the solution. To avoid forming lumps, starch was added into the water in a mixing cup of a Waring blender while mixed at low speed for 20 seconds. The starch solutions were then evaluated using a Viscograph-E (C.W. Brabender® Instruments, Inc., South Hackensack, NJ). According to the Brabender viscosity measurement procedure as referred to herein, viscosity is measured using a C.W. Brabender Viscograph, e.g., a Viscograph-E that uses reaction torque for dynamic measurement. It is to be noted that, as defined herein, the Brabender units are measured using a sample cup size of 16 fl. oz (about 500 cc), with a 700 cmg cartridge at an RPM of 75. One of ordinary skill in the art also will readily recognize that the Brabender units can be converted to other viscosity measurements, such as centipoises (e.g., $cP = BU \times 2.1$, when the measuring cartridge is 700 cmg) or Krebs units, as described therein. The pasting profiles of Compositions 1D-1H

extruded at 16 wt.% moisture content are shown in FIG. 1 along with comparative Compositions 1B and 1C.

[0172] Considering Compositions 1D-1H, as alum increased from 1 wt.% to 4 wt.%, the initial viscosity decreased from 70 Brabender Unit (BU) to 10 BU, while the molecular weight decreased as well. The initial viscosities and viscosities at 93°C of Compositions 1D-1H were reduced as low as those of Compositions 1B and 1C. Compositions 1B and 1C represent conventional viscosity limits of low water demand starches.

[0173] The results of Compositions 1D-1H shown in FIG. 1 demonstrate that optimal acid-modification can be achieved during extrusion. These results further suggest that the method of preparing pregelatinized, partially hydrolyzed starch successfully reduced the viscosity (molecular weight) of the starch. No viscosity peak was observed between 70°C to 90°C, indicating that Compositions 1D-1H were fully gelatinized. Had Compositions 1D-1H not been fully gelatinized, there would have been an increase in viscosity. The full gelatinization of the starch Compositions were confirmed by differential scanning calorimetry (DSC).

[0174] FIG. 2 is a second amylogram plotting viscosity (left y-axis) and temperature (right y-axis) over a fifty minute period (x-axis). Comparative Compositions 1B and 1C and Compositions 1I-1K, all as described herein, were mixed into starch solutions in an amount of 10% by weight based on the weight of the solution. To avoid forming lumps, starch was added into the water in a mixing cup of a Waring blender while mixed at low speed for 20 seconds. The starch solutions were then evaluated using a Viscograph-E. The pasting profiles of Compositions 1I-1K extruded at 13 wt.% moisture content are shown in FIG. 2 along with comparative Compositions 1B and 1C.

[0175] Similar trends observed with Compositions 1D-1H were observed with Compositions 1I-1K. In particular, the method of preparing pregelatinized, partially hydrolyzed starch in an extruder as described herein successfully reduced the viscosity of Compositions 1I-1K.

[0176] As alum increased from 1 wt.% to 3 wt.%, the initial viscosity decreased from 75 BU to 14 BU, while the molecular weight also decreased. The initial viscosities and the viscosity at 93°C of Compositions 1I - 1K were reduced as low as those of Compositions 1B and 1C.

[0177] In addition, the results of Compositions 1I-1K shown in FIG. 2 demonstrate that optimal acid-modification can be achieved during extrusion. No viscosity peak was observed between 70°C to 90°C, indicating that Compositions 1I-1K were fully gelatinized.

[0178] Furthermore, these results show that at a lower moisture content, more starch hydrolysis can be achieved at a given acid level than at a higher moisture content because at a low moisture content there is more mechanical energy and, thus, more starch degradation, such that the starch will become smaller using the same acid level.

EXAMPLE 3

[0179] This Example illustrates the fluidity of gypsum slurries containing Compositions 1A (comparative), 1D-1I, and 1K-1L. The Compositions were evaluated with regards to fluidity using a slump test that will be appreciated by one of ordinary skill in the art.

[0180] In preparation for testing, slurries were prepared with each of Compositions 1A (comparative), 1D-1I, and 1K-1L in an amount of 2 wt.% and the formulation outlined in Table 4, using a water stucco ratio (WSR) of 1.0.

Table 4

Ingredient	Weight (g)
Stucco	400
Heat resistance accelerator	4
Starch Composition	8
Sodium trimetaphosphate 10% solution	8
Dispersant	2
Retarder 1% solution	20
Gauging water	357
PFM-33 foam (0.5% solution)	25

[0181] The starch was weighed into a dry mix comprising stucco having over 95% purity and heat resistance accelerator. Water, sodium trimetaphosphate (10 wt.% solution), dispersant, and retarder were weighed into the mixing bowl of a Hobart Mixer. The dry mix was poured into the mixing bowl of a mixer available as N50 5-Quart Mixer from Hobart

(Troy, OH), soaked for 10 seconds, and mixed at speed II for 30 seconds. For foam preparation, a 0.5% solution of Hyonic® PFM-33 soap (available from GEO® Specialty Chemicals, Ambler, PA) was formed, and then mixed with air to make the air foam. The air foam was added to the slurry using a foam generator.

[0182] Each slurry was then put into a cylinder, having a diameter of 4.92 cm (1.95 in.) and a height of 10 cm (3.94 in.). The cylinder was then lifted, allowing the slurry to freely flow. The diameters of the slumps that formed were then measured to illustrate fluidity of the slurries and are recorded in Table 5. Table 5 also includes the results of a time to 50% hydration test explained in further detail below. The retarder of Composition 1L was increased from 0.05 wt% to 0.0625 wt.% so that Composition 1L had the same setting rate as reference Composition 1A.

Table 5

Composition	Retarder	Slump (cm)	Time to 50% Hydration (minutes)
1A	0.05 wt.%	13.7 cm (5 3/8 in)	4
1D	0.05 wt.%	16.5 cm (6 1/2 in)	3.8
1E	0.05 wt.%	15.2 cm (6 in)	3.6
1F	0.05 wt.%	16.2 cm (6 3/8 in)	3.7
1G	0.05 wt.%	16.2 cm (6 3/8 in)	3.3
1H	0.05 wt.%	17.8 cm (7 in)	3.7
1I	0.05 wt.%	15.9 cm (6 1/4 in)	3.6
1K	0.05 wt.%	18.4 cm (7 1/4 in)	3.4
1L	0.0625 wt.%	18.4 cm (7 1/4 in)	4

[0183] As can be observed from Table 5, slurries prepared with Compositions 1D-1I and 1K showed larger slump sizes than the slurry prepared with Composition 1A (comparative). They also set faster than Composition 1A (comparative), indicating slurries containing Compositions 1D-1I and 1K had better fluidity than the slurry containing Composition 1A.

[0184] In addition, the time to 50% hydration was measured for the slurries for purposes of comparing the slump size when the slurries set at the same rate. The temperature profiles of the slurries were measured using software as one of ordinary skill in the art will appreciate.

[0185] This additional test was conducted to confirm that the slump tests were correct, specifically to illustrate that the large slumps observed with slurries including pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure resulted from improved fluidity in comparison to Composition 1A (comparative), not slow hydration.

[0186] Composition 1H, prepared with 2 wt.% alum and 0.3 wt.% tartaric acid, effectively hydrolyzed starch to a low viscosity and had less impact on the hydration rate, because tartaric acid and alum had opposite effect on hydration rate.

[0187] FIG. 3 is a graph plotting temperature versus time, showing the temperature rise set (TRS) hydration rate. Compositions 1F and 1L with 0.05% and 0.0625% of retarder, respectively, hydrate faster or at the same rate as Composition 1A (comparative).

[0188] As seen in FIG. 3, Composition 1L, with 0.0625 wt.% of retarder, had the same hydration rate as Composition 1A (comparative). The slump size of Composition 1L with 0.065 wt.% retarder was 18.415 cm (7 1/4"), was significantly larger than Composition 1A.

[0189] This result suggests that the larger slump sizes observed with slurries including pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure were due to high fluidity and not to slower setting. Furthermore, pregelatinized, partially hydrolyzed starches prepared in accordance with embodiments of the disclosure will allow for wallboards using less water without sacrificing fluidity.

EXAMPLE 4

[0190] This Example illustrates the strength of gypsum disks prepared with slurries containing Compositions 1A (comparative), 1D-II, and 1K. Strength was evaluated using a compressive strength test described herein.

[0191] To prepare for testing, slurries were prepared with each of Compositions 1A (comparative), 1D-II, and 1K-1L in an amount of 2 wt.% and the parameters outlined in Table 4 above.

[0192] A water stucco ratio (WSR) of 1.0 and air foam were used to make gypsum disks with a final density of 29 pcf. The starch was weighed into a dry mix comprising stucco and heat resistance accelerator. Water, sodium trimetaphosphate 10% solution, dispersant, and

retarder were weighed into the mixing bowl of a Hobart Mixer. The dry mix was poured into the mixing bowl of a mixer available as N50 5-Quart Mixer from Hobart (Troy, OH), soaked for 10 seconds, and mixed at speed II for 30 seconds. For foam preparation, a 0.5% solution of Hyonic® PFM-33 soap (available from GEO® Specialty Chemicals, Ambler, PA) was formed, and then mixed with air to make the air foam. The air foam was added to the slurry using a foam generator. The foam generator was run at a rate sufficient to obtain the desired board density of 29 pcf. After foam addition, the slurry was immediately poured to a point slightly above the tops of the molds. The excess was scraped as soon as the plaster set. The molds had been sprayed with mold release (WD-40™). The disks had a diameter of 10.16 cm (4 in.) and a thickness of 1.27 cm (0.5 in.).

[0193] After the disks had hardened, the disks were removed from the mold, and then dried at 110°F (43°C) for 48 hours. After removing from the oven, the disks were allowed to cool at room temperature for 1 hour. The compressive strength was measured using a materials testing system commercially available as SATEC™ E/M Systems from MTS Systems Corporation (Eden Prairie, Minnesota). The load was applied continuously and without a shock at speed of 0.04 inch/min (with a constant rate between 15 to 40 psi/s). The results are shown in Table 6.

Table 6

Composition	Compressive Strength (PSI) at 29 pcf
1A	396
1D	439
1E	388
1F	476
1G	419
1H	417
1I	455
1K	426

[0194] As seen in Table 6, the foam disks containing Compositions 1D-1I and 1K had compressive strengths comparable to, or better than that which contained Composition 1A (comparative), indicating pregelatinized, partially hydrolyzed starches could reduce water demand without sacrificing their strength enhancing property.

EXAMPLE 5

[0195] This example demonstrates the effect on strength by changing the amount of starch on gypsum disks prepared from slurries of compositions 5A-5G. Particularly, composition 5A was a comparative composition inasmuch as it included no starch. Compositions 5B-5G included pregelatinized, partially hydrolyzed starch in the form of a pregelatinized starch having a viscosity of 88 cP, as measured according to the VMA Method, as set forth herein (see also US 2014/0113124 A1). The starch was a corn starch. None of the samples included retarder solution. The compositions are set forth in Table 7. The weight percentages are provided on a stucco basis.

Table 7

Composition	Stucco (g)	HRA (g)	Starch (g)	STMP 10% Solution (g)	Dispersant (g)	Water (g)
5A (control)	250 (100 wt%)	2.5 (1 wt.%)	0 (0 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	744 (297.6 wt.%)
5B	250 (100 wt. %)	2.5 (1 wt.%)	5 (2 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	744 (297.6 wt.%)
5C	250 (100 wt.%)	2.5 (1 wt.%)	15 (6 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	754 (301.6 wt.%)
5D	250 (100 wt.%)	2.5 (1 wt.%)	25 (10 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	769 (307.6 wt.%)
5E	250 (100 wt.%)	2.5 (1 wt.%)	37.5 (15 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	782 (312.8 wt.%)
5F	250 (100 wt.%)	2.5 (1 wt.%)	50 (20 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	794 (317.6 wt.%)
5G	250 (100 wt.%)	2.5 (1 wt.%)	62.5 (25 wt.%)	5 (0.2 wt.%)	1.25 (0.5 wt.%)	806 (322.4 wt.%)

[0196] The disks were prepared from the separate slurry compositions 5A-5G. Each composition was prepared from dry and wet mixes that were combined. Each wet mix was prepared by weighing the water, dispersant, retarder 1% solution, dispersant, and sodium trimetaphosphate 10% solution in a mixing bowl of a Waring blender (model CB15),

commercially available from Conair Corp. (East Windsor, New Jersey). The sodium trimetaphosphate 10% solution was prepared by dissolving 10 parts (weight) of sodium trimetaphosphate in 90 parts (weight) of water. The remaining ingredients, particularly, the stucco, heat resistant accelerator, and starch (if present), were weighed and prepared in a dry mix. The heat resistant accelerator was composed of ground up land plaster and dextrose. The dry mix was poured into the blender with the wet ingredients, and soaked for 10 seconds and then mixed at high speed for 10 seconds.

[0197] The slurry was immediately poured into a ring (4" diameter and 0.5" thick) type of mold which was suited for forming a disk sample. The surface of the molds had previously been sprayed with mold release in the form of WD40™, commercially available from WD-40 Company, San Diego, CA. The slurry was poured to a point slightly above the top of the ring of the molds. The excess slurry was scraped as soon as the plaster was set. After the disks hardened in the mold, the disks were removed from the mold, then dried at 110°F (43°C) for 48 hours. Following removal from the oven, the disks were allowed to cool at room temperature for one hour. Upon completion of drying, the final disks had dimensions of a diameter of 4 inches (10.16 cm), and a thickness of 0.5 inches (1.27 cm).

[0198] Strength was tested by measuring board compressive strength (BCS). The compressive strength was measured using the materials testing system commercially available as ATS Systems from Applied Test Systems, Inc. The load was applied continuously and without a shock at speed of 1.0 inch/min (with a constant rate between 15 to 40 psi/s). The results are shown in FIG. 4.

[0199] FIG. 4 shows the results from tests conducted with compositions 5D-5F, i.e., with pregelatinized starch at 10 wt.%, 15 wt.%, 20 wt.%, and 25 wt.%. FIG. 4 shows the results for the compositions of Table 7. As seen in FIG. 4, the results show that board compressive strength increased as the pregelatinized and acid-modified starch content increased from 0% to 25% by weight of stucco. In contrast to pregelatinized and acid-modified starch, other pregelatinized starches, generally having a viscosity above 300 cP according to the VMA method, appear to have diminished influences on compressive strength at higher starch concentrations.

EXAMPLE 6

[0200] This example demonstrates the flowability of a stucco slurry containing an elevated amount (i.e., 20 wt.%, stucco basis) of pregelatinized starch having a viscosity of 88

cP, as measured according to the VMA Method. The starch was a corn starch, i.e., the same starch at that viscosity set forth in Example 5. The composition of the slurry is set forth in Table 8. The composition was prepared and using ingredients as described in Example 5.

Table 8

Ingredient	Amount (g)	Amount (wt. %)	Amount (wt.%, stucco basis)
Stucco	400	47.4%	---
HRA	4	0.47%	1
Starch	80	9.48%	20
Dispersant	2	0.24%	0.5
STMP 10% Solution	8	0.95%	0.2
Water	350	41.5%	87.5
Total:	844	100%	211

[0201] The flowability was evaluated by a slump test. The slurry, having a water to stucco ratio (WSR) of 0.90, was poured into a 2 inch (5.08 cm) diameter cylinder that was 4 inch (10.16 cm) tall. The cylinder was open on each end and placed on one end on a flat smooth surface, with excessive slurry scraped off. Then the cylinder was immediately lifted to allow the slurry to rush out and form a slump. The diameter of the resulting patty was measured. A larger diameter indicated a higher flowability.

[0202] The stucco slurry, which contained 20 wt.% (stucco basis) of the 88 cP pregelatinized starch had a slump size of 6.75 inch (17 cm), indicating the slurry containing 20 wt.% (stucco basis) of the starch was processable. Such a slump size indicated flowability because slurry with an approximate 6 inch slump typically is able to completely spread to the full width of a production table. This demonstrated that the slurry was suitable for useful in wallboard manufacturing processing despite the elevated amount of starch. This processability is useful because wallboard manufacture typically requires a certain amount of flowability in forming the wallboard sandwich precursor structure.

EXAMPLE 7

[0203] This example demonstrates the effect on various properties of very light boards (having a board weight of 800 lb/MSF) for one-half inch thick board having a board core prepared from the slurry of composition 7 set forth in Table 9.

[0204] The boards had either paper cover sheets or uncoated glass mats in the form of Duraglass 8924G/Duraglass8924, commercially available from Johns Manville, Denver, CO. The Duraglass 8924G was used on the face side of the board, while the Duraglass 8924 was used on the back side of the board.

Table 9 – Composition 7

Ingredient	Amount (g)	Amount (wt. %)	Amount (wt.%, stucco basis)
Stucco	300	37.8	---
HRA	9	1.13	3
Starch	48	6.05	16
Dispersant	3	0.38	1
STMP 10% Solution	6	0.76	0.2
Water	427	53.85	142
Total:	793	100	---

[0205] The composition was prepared from dry and wet mixes that were combined. The wet mix was prepared by weighing the water, dispersant, and sodium trimetaphosphate 10% solution in a mixing bowl of a Hobart Mixer (model N50), commercially available from Hobart (Troy, Ohio). The sodium trimetaphosphate 10% solution was prepared by dissolving 10 parts (weight) of sodium trimetaphosphate in 90 parts (weight) of water. The starch had a viscosity of 88 cP, as measured according to the VMA Method. The starch was a corn starch and the same starch at that viscosity as in Examples 5-6. It was added into the solution and mixed in a Waring Blender (model CB 15) for 30 seconds. The remaining ingredients, particularly the stucco and heat resistant accelerator, were weighed and prepared in a dry mix. The heat resistant accelerator was composed of ground up land plaster and dextrose. The dry mix was poured into the blender with the wet ingredients, and soaked for 10 seconds and then mixed at speed 2 for 25 seconds.

[0206] Foam was added in order to reduce disk density (and hence weight). For foam preparation, a 0.5% solution of Hyonic™ PFM-33 soap (available from GEO Specialty Chemicals, Ambler, PA) was formed and then mixed with air to make the air foam. The air foam was added to the slurry using a foam generator. The foam generator was operated at a rate sufficient to obtain a core density of 18.4 pcf.

[0207] After foam addition, the slurry was immediately poured into a 12" x 12" x 1/2" envelop made from face paper on the face side of the board and back paper on the back side

of the board, or uncoated glass mat Duraglass 8924G/ Duraglass 8924. After the gypsum had hardened, the envelop sample was removed from the mold, and then dried at 110°F (43°C) for 48 hours.

[0208] Three boards were prepared, identified as Boards 1-3. Boards 1 and 3 were prepared with cover sheets in the form of 48 lb/MSF face (Manila) paper. Board 2 was prepared with Duraglass 8924G/ Duraglass 8924 cover sheets which had a basis weight of 24 lbs/MSF. Board 3 was a commercial lightweight control board, and was prepared from a slurry which did not include the mid-range viscosity pregelatinized starch but instead included a higher viscosity starch at 773 cP. Table 10A provides the board weight and density of each board, with two boards prepared for each of Boards 1-3. The caliper was determined by a digital thickness measuring device and refers to the thickness of the dry board.

Table 10A

Board ID	Face Material	Caliper (in)	Board Weight (#/MSF)	Density (pcf)
Board 1	Paper	0.467	820	21.1
		0.466	815	21.0
Board 2	Glass Mat	0.459	828	21.6
		0.464	803	20.8
Board 3 (control)	Paper	0.496	1351	32.7
		0.496	1319	31.9

[0209] As seen in Table 10B, a 3 inch (7.6 cm) by 10 inch (25.4 cm) sample was cut from 12 inch (30.5 cm) by 12 inch (30.5 cm) laboratory board (Boards 1 and 2) and plant production board (Board 3, control), and tested for flexural strength properties by using an Instron Universal Testing Machine model no. 3345 (Instron Corp, Norwood, MA). The flexural strength is related to the maximum load the sample supported before failure.

Flexural properties of the tested boards were evaluated by supporting the specimen near the ends and applying a transverse load midway between the supports of the device. The long side of the board was in the machine direction or the cross machine direction of the face material. The supports were either parallel (i.e., in the machine direction of the face material) or perpendicular (i.e., in the cross machine direction of the face material), to the long side of the test sample. A mechanical property of interest is the Modulus of Rupture (MOR), calculated based on the following equation (1):

$$M = \frac{3 \times P \times L}{2 \times b \times d^2}$$

(Equation 1)

wherein:

M = Modulus of Rupture;

P = Breaking Load;

L = Distance between Knife Edges on which the Sample is Supported;

b = Average Specimen Breadth; and

d = Average Specimen Depth.

[0210] From a stress-strain curve of the flexural test, the Instron Universal Testing machine determines the Modulus of Elasticity (MOE). The linear portion of the stress-strain curve indicates the MOE or Young's modulus and a non-linear portion indicates non-linear deformation of material. Each board was tested twice in the perpendicular direction and twice in the parallel direction. Each test was conducted using the "face down" side of the face material to test its effects on mechanical properties contributed by the face material.

Table 10B

Board ID	Board Type and Face Material	Test		3" x 10" Sample		
				Flexural Load (lb _f)	MOR (psi)	MOE (psi)
Board 1	Lab board with 48 lbs/MSF Face Paper	Perpendicular	Face Down	48.6	892	165905
		Parallel	Face Down	25.4	468	104813
Board 2	Lab board with 24	Perpendicular	Face Down	44.4	843	75242

	lbs/MSF Glass Mat	Parallel	Face Down	38.1	709	58848
Board 3 (control)	Ultralight board with 48 lbs/MSF Face Paper	Perpendicular	Face Down	70.7	1151	253038
		Parallel	Face Down	31.4	512	196675

[0211] The flexural load information in Table 10B was adjusted using Equation 1 in order to account for the change of laboratory size to the sample size required by ASTM 473-10. In particular, the flexural load data was extrapolated to a board sample size of 12 inch (30.5 cm) by 16 inch (40.6 cm) at a thickness of one-half inch. The equivalent flexural load is set forth in Table 10B.

[0212] In addition, for each sample, a factor of safety for a three foot overhang was evaluated. A factor of safety of three feet or greater is typically necessary to maintain integrity when the product is subjected to dynamic loads in transit or handling.

Table 10C

Board ID	Board Type and Face Material	Test		12" x 16" x ½" Sample	Factor of Safety for 3' Overhang
				ASTM C473 – Equivalent Flexural Load (lb _f)	
Board 1	Lab board with 48 #/MSF Manila Paper	Perpendicular	Face Down	127	9.4
		Parallel	Face Down	67	N/A
Board 2	Lab board with 24 #/MSF Glass Mat	Perpendicular	Face Down	120	8.6
		Parallel	Face Down	101	N/A
Board 3 (control)	Ultralight with 48 #/MSF Manila Paper	Perpendicular	Face Down	164	7.8
		Parallel	Face Down	73	N/A

[0213] As seen in Tables 10A-10C, the equivalent flexural load was provided for each laboratory board in both perpendicular and parallel tests. The factor of safety is determined by the momentum capacity for the board divided by the momentum for a three foot overhang. Thus, this data showed that the reduced weight board gains a greater factor of safety subjected to dynamics load in transit and handling. For three foot overhang, the board

experienced a bending moment by self imposed load from its own weight with unsupported end of three feet.

[0214] As seen in Tables 10A-10C, the equivalent flexural load was provided for each laboratory board in both perpendicular and parallel tests. Moment capacity is determined by the flexural test and relates to the breaking load from the bending force on specimen multiplied by the length between two supports for the full width of the specimen. The factor of safety, defined by the ratio of the moment capacity to the moment from 3' overhang, is greater than 3 so as to ensure the board having sufficient resistance to the load without breaking. As seen in Table 11, the values for Boards 1 and 2 at 9.4 and 8.6 were higher than that of the control Board 3 at 7.8. Thus, this data showed that the reduced weight board gains a greater factor of safety subjected to dynamics load in transit and handling. Although the flexural load for the lower weight 800 lb/MSF board of Board 1 had a lower flexural load than that of the commercial control board, it was surprised to find out that the lower weight Board 1 had much lower moment from 3' overhang as compared with control Board 3, resulting higher safety of factor.

[0215] Furthermore, three additional boards were tested and identified as boards 4-6. The laboratory boards were prepared as discussed above. The density information is shown below in Table 11. The boards were tested for thermal resistance using the Fox 314 Test System (commercially available from TA Instruments, Wood Dale, IL), and in accordance with ASTM C518-10 standard test method for steady-state thermal transmission properties by means of heat flow meter apparatus.

Table 11

Lab Board ID	Face Material	Board Density (pcf)	R-Value (h.ft. ² °F/Btu)
Board 4	Ultralight board with 48 #/MSF Face Paper	30.0	0.5
Board 5	Lab board with 24 #/MSF Glass Mat	19.5	1.1
Board 6	Lab board with 24 #/MSF Glass Mat	19.8	1.0

[0216] The R-Value parameter is expressed as the thickness of the material normalized to the thermal conductivity. R-value is a measure of thermal resistance of the building materials. The higher the value of R, the better the thermal insulation. As seen in Table 11, Boards 5 and 6 exhibited an R-value of twice the normal production board, which was surprising and unexpected because of the density change. These boards were formed from slurry containing pregelatinized starch with a viscosity of 88 cP and have a much higher insulation property (e.g., two times the R-value) of board formed from slurries containing pregelatinized starches with much higher viscosities (e.g., above about 300 cP), where all viscosities are measured according to the VMA method. In some embodiments, a board according to the present disclosure has an R-Value of at least about 1.0 h.ft².°F/Btu (e.g., from about 0.5 h.ft².°F/Btu to about 5 h.ft².°F/Btu), as measured according to the ASTM C518-10 standard test method for steady-state thermal transmission properties by means of heat flow meter apparatus.

EXAMPLE 8

[0217] This example illustrates the strength generated by board prepared from pregelatinized starches of mid-range viscosity.

[0218] In particular, three gypsum disks were prepared with slurries containing Compositions 8A-8C. Slurries, disks, and strength were prepared and evaluated as described in Example 4. Each of Compositions 8A-8C contained the formulation set forth in Table 12.

Table 12

Ingredient	Amount (g)	Amount (wt. %)	Amount (wt.%, stucco basis)
Stucco	300	31.45	---
HRA	9	0.94	3
PG Starch	60	6.29	20
Dispersant	3	0.31	1
STMP 10% Solution	6	0.63	0.2
Water	576	60.38	192
Total:	954	100	---

[0219] The weight of the pregelatinized (PG) starch in each case on a stucco basis was 20 wt.%. The difference between Compositions 8A-8C was in the viscosity of the pregelatinized starch that was included. The starches of Compositions 8A, 8B, and 8C were corn starches. In Composition 8A, the pregelatinized starch had a viscosity of 59 cP according to the VMA method. Composition 8B included the pregelatinized starch with a

viscosity of 88 cP according to the VMA method, while Composition 8C included the pregelatinized starch with a viscosity of 195 cP according to the VMA method. The results are reported in Table 13.

Table 13

Composition for Preparing Disk	Viscosity (cP)	Compressive Strength (PSI) at 20 pcf
8A	59	285
8B	95	293
8C	195	305

[0220] This example illustrates the benefit to strength with using elevated amounts of pregelatinized starch (20 wt.%, stucco basis) at a range of representative mid-range viscosities. As seen in Table 13, all three disks exhibited good compressive strength. These values are below 400 PSI as in Example 4, but represent good compressive strength at the low board density of 20 pcf.

[0221] The use of the terms “a” and “an” and “the” and “at least one” and similar referents in the context of describing the invention (especially in the context of the following claims) (e.g., in relation to acids, raw material starches, or other components or items) are to be construed to cover both the singular and the plural, unless otherwise indicated herein or clearly contradicted by context. The use of the term “at least one” followed by a list of one or more items (for example, “at least one of A and B”) is to be construed to mean one item selected from the listed items (A or B) or any combination of two or more of the listed items (A and B), unless otherwise indicated herein or clearly contradicted by context. The terms “comprising,” “having,” “including,” and “containing” are to be construed as open-ended terms (i.e., meaning “including, but not limited to,”) unless otherwise noted. Recitation of ranges of values herein are merely intended to serve as a shorthand method of referring individually to each separate value falling within the range, unless otherwise indicated herein, and each separate value is incorporated into the specification as if it were individually recited herein. All methods described herein can be performed in any suitable order unless otherwise indicated herein or otherwise clearly contradicted by context. The use of any and all examples, or exemplary language (e.g., “such as”) provided herein, is intended merely to better illuminate the invention and does not pose a limitation on the scope of the invention

unless otherwise claimed. No language in the specification should be construed as indicating any non-claimed element as essential to the practice of the invention.

[0222] Preferred embodiments of this invention are described herein, including the best mode known to the inventors for carrying out the invention. Variations of those preferred embodiments may become apparent to those of ordinary skill in the art upon reading the foregoing description. The inventors expect skilled artisans to employ such variations as appropriate, and the inventors intend for the invention to be practiced otherwise than as specifically described herein. Accordingly, this invention includes all modifications and equivalents of the subject matter recited in the claims appended hereto as permitted by applicable law. Moreover, any combination of the above-described elements in all possible variations thereof is encompassed by the invention unless otherwise indicated herein or otherwise clearly contradicted by context.

CLAIMS:

1. A gypsum board comprising:
a set gypsum core disposed between two cover sheets, the core formed from a slurry comprising stucco, water, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco;
the board having a density of about 31 pcf or less and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10, method B.
2. The gypsum board of claim 1, wherein the pregelatinized starch has a viscosity of from about 20 cP to about 200 cP.
3. The gypsum board of claim 2, wherein the pregelatinized starch has a viscosity from about 30 cP to about 150 cP.
4. The gypsum board of any one of claims 1-3, wherein the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.
5. The gypsum board of any one of claims 1-4, wherein the pregelatinized starch is present in an amount of from about 12% to about 25% by weight of the stucco.
6. The gypsum board of any one of claims 1-5, wherein the board has a density of about 15 pcf to about 31 pcf.
7. The gypsum board of any one of claims 1-6, wherein the board has a density of about 15 pcf to about 27 pcf.
8. The gypsum board of any one of claims 1-7, wherein the board has a density of about 19 pcf to about 24 pcf.
9. The gypsum board of any one of claims 1-8, wherein the board has a compressive strength of from about 170 psi to about 250 psi.
10. The gypsum board of any one of claims 1-9, wherein the starch has a cold water solubility of at least about 70% when measured at 25 °C.

11. The gypsum board of any one of claims 1-10, wherein the pregelatinized starch, in a 10% slurry in water when measured at 25 °C has a cold water viscosity of from about 60 cP to about 160 cP.

12. A method of making a gypsum board comprising:

a) mixing at least water, stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco;

b) disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly;

c) cutting the wet assembly into a board; and

d) drying the board;

the dried board having a density of about 31 pcf or less and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10, method B.

13. The method of claim 12, wherein the pregelatinized starch has a viscosity from about 30 cP to about 200 cP.

14. The method of claim 12 or 13, wherein the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.

15. The method of claim 14, wherein the pregelatinized starch is present in an amount of from about 12% to about 25% by weight of the stucco.

16. The method of any one of claims 12-15, wherein the board has a density of from about 15 pcf to about 27 pcf.

17. A slurry comprising:

water, stucco, and pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco; wherein

when the slurry is cast and dried as board comprising a set gypsum composition, the set gypsum composition comprises a continuous crystalline matrix substantially of calcium sulfate dihydrate, wherein said board has a density of about 31 pcf or less, and at a thickness

of one-half inch (about 1.3 cm), the board meets at least one of the following: (i) a nail pull resistance of at least about 65 lb (about 29 kg), (ii) an average core hardness of at least about 11 lb (about 5 kg), and (iii) an average flexural strength of at least about 36 lb (about 16 kg) in a machine direction and/or about 107 lb (about 48.5 kg) in a cross-machine direction, the nail pull resistance, the core hardness and the flexural strength determined in accordance with ASTM standard C473-10, method B.

18. The slurry of claim 17, wherein the pregelatinized starch has a viscosity from about 30 cP to about 200 cP.

19. The slurry of claim 17 or 18, wherein the pregelatinized starch is present in an amount of from about 10.1% to about 25% by weight of the stucco.

20. The slurry of any one of claims 17-19, wherein the board has a density of from about 15 pcf to about 27 pcf.

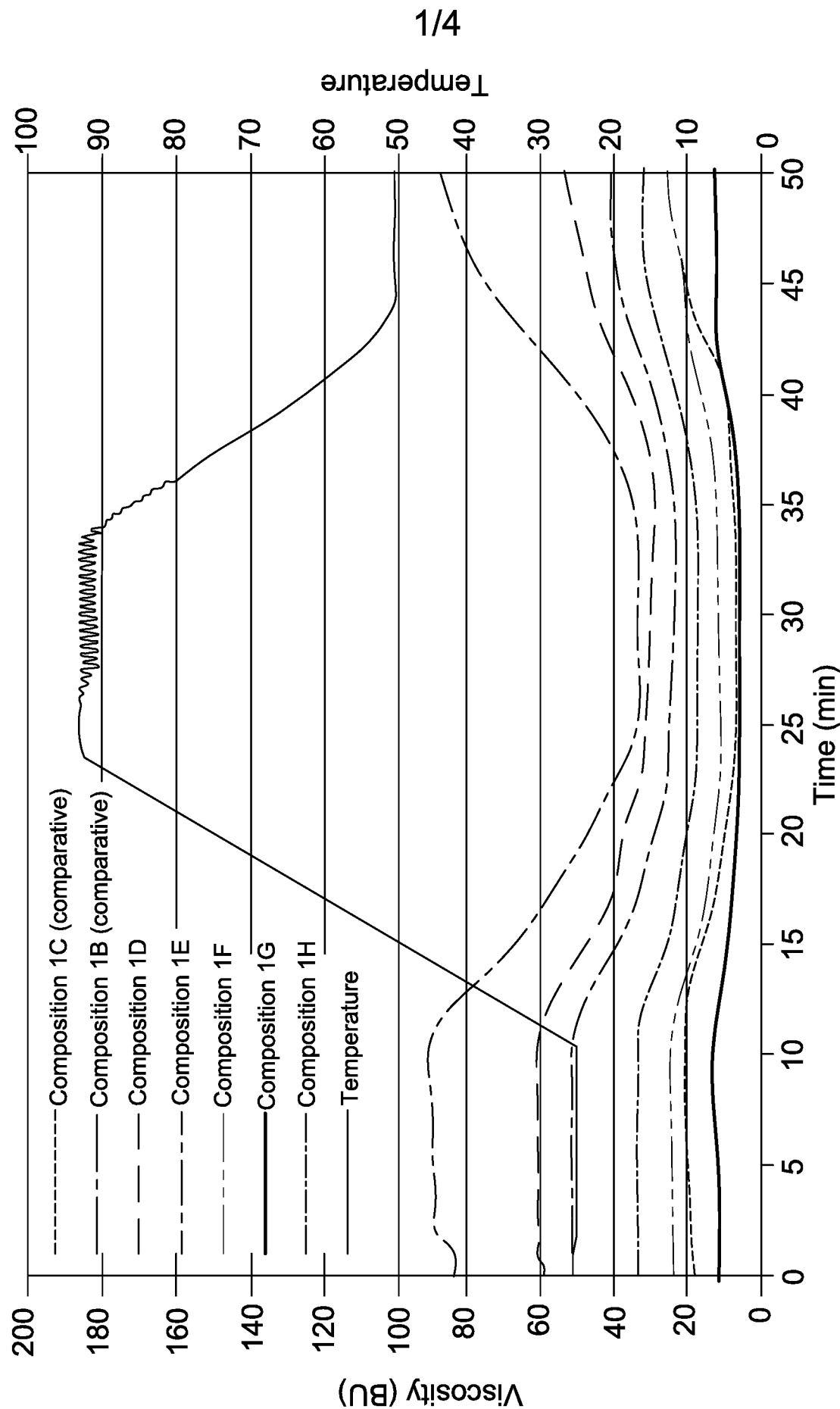


FIG. 1

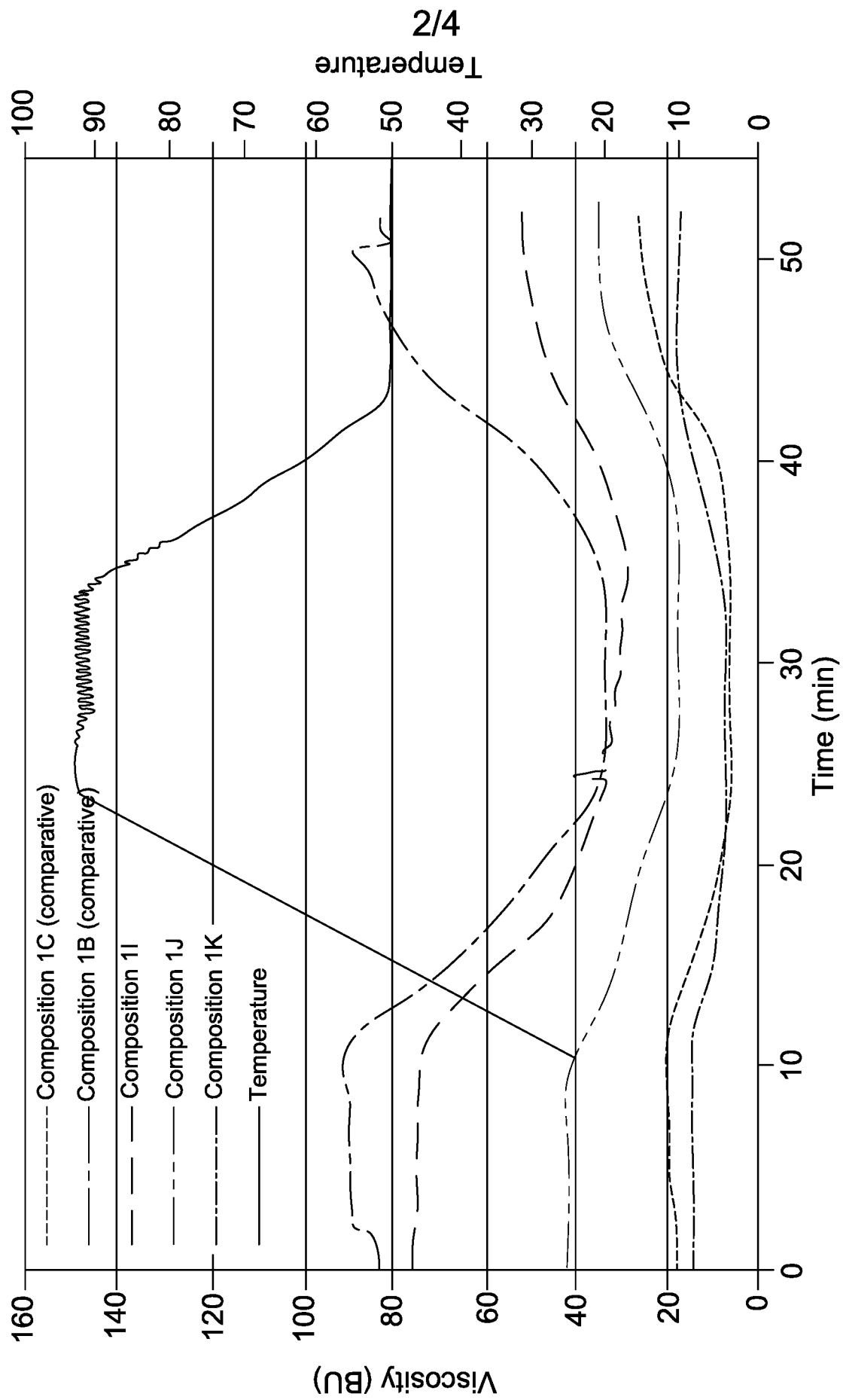


FIG. 2

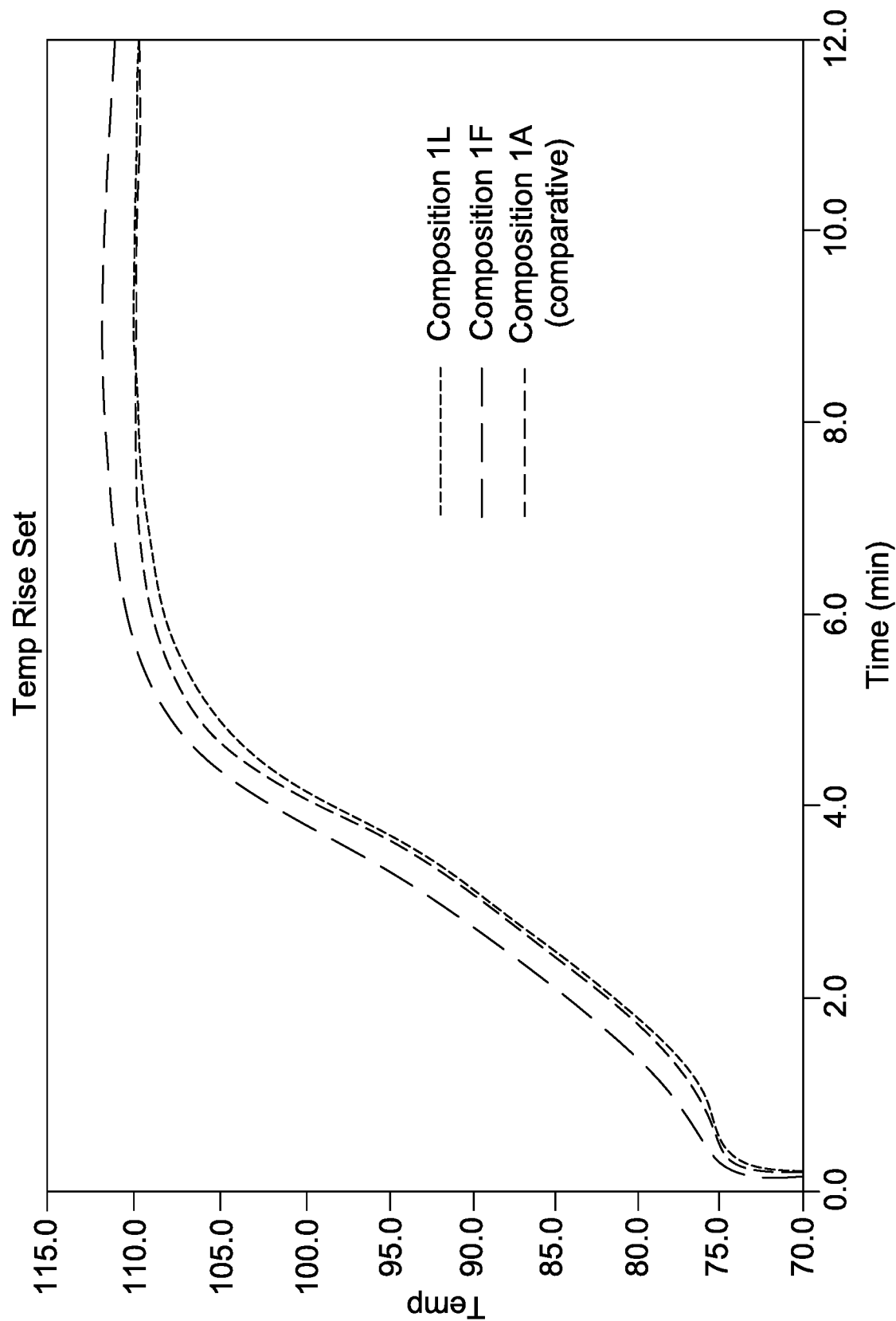


FIG. 3

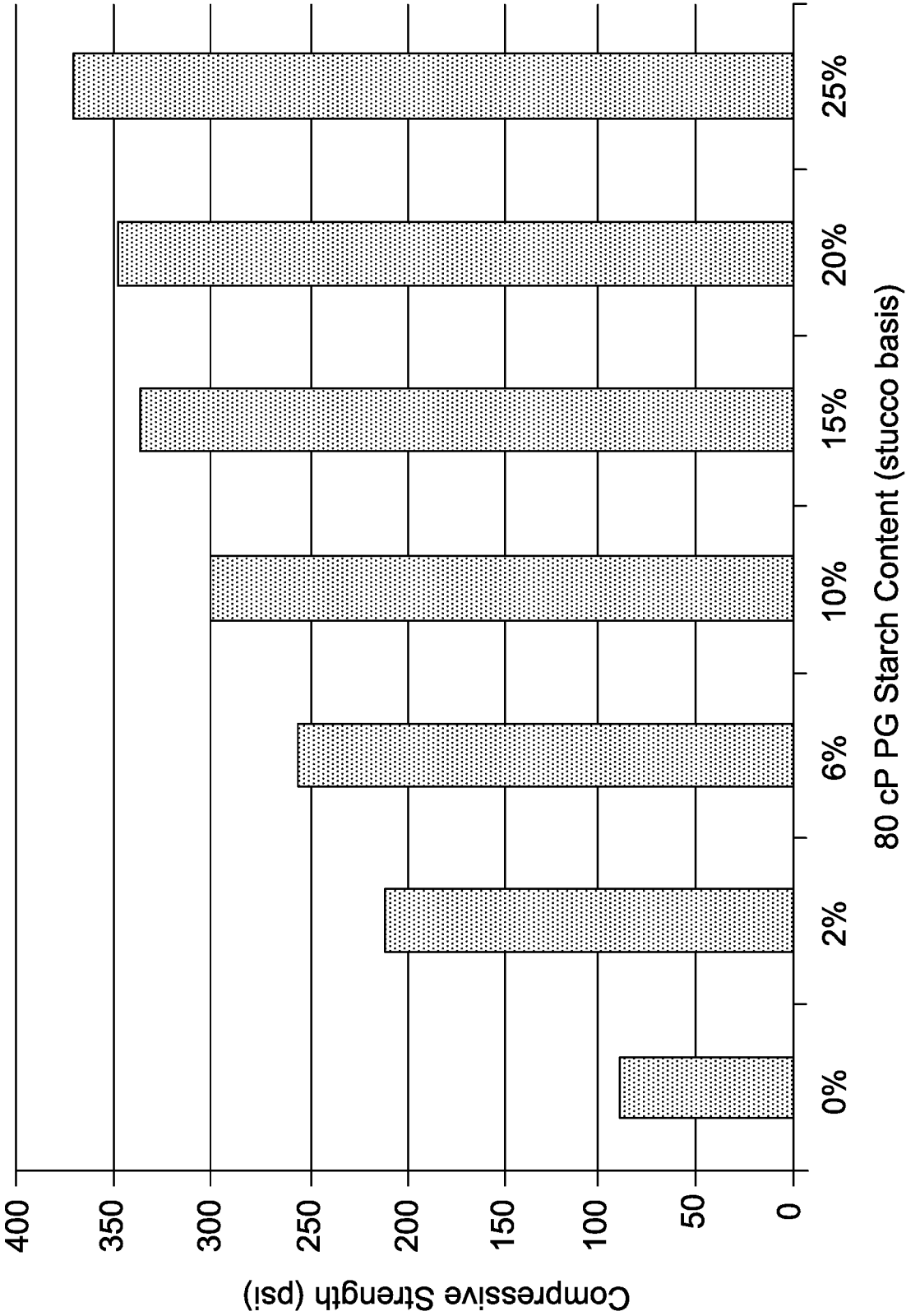


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No

PCT/US2016/038714

A. CLASSIFICATION OF SUBJECT MATTER
 INV. C04B28/14
 ADD.

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)
 C04B

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

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2015/050804 A1 (UNITED STATES GYPSUM CO [US]) 9 April 2015 (2015-04-09) paragraph [0005] - paragraph [0160]; claims 1-10; figures 1-3; examples 1-4 -----	1-20
X	WO 2012/122102 A2 (GEORGIA PACIFIC GYPSUM LLC [US]; FRANCIS HUBERT C [US]) 13 September 2012 (2012-09-13) paragraphs [0001], [0009] - paragraph [0052]; claims 1-21; figures 1,2; examples 1,2 -----	12,13, 16-18,20
A		14,15,19
X	US 2008/070026 A1 (YU QIANG [US] ET AL) 20 March 2008 (2008-03-20) formulation B; samples 6-13 in table 7; paragraph [0001] - paragraph [0049]; claims 1-9; examples 1,2,6,7; tables 1,2,6,7 -----	1-3,6-8, 10,11



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier 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

8 February 2017

Date of mailing of the international search report

21/02/2017

Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2016/038714

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2015050804 A1	09-04-2015	AU 2014329817 A1 CA 2925619 A1 CN 105722861 A EP 3052527 A1 JP 2016535116 A KR 20160065869 A TW 201514315 A WO 2015050804 A1	05-05-2016 09-04-2015 29-06-2016 10-08-2016 10-11-2016 09-06-2016 16-04-2015 09-04-2015
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US 2008070026 A1	20-03-2008	AR 069160 A1 BR PI0819230 A2 CA 2703358 A1 CN 101855067 A CN 103496204 A CO 6280413 A2 EP 2212105 A1 ES 2585704 T3 JP 2011502094 A RU 2010118087 A TW 200922775 A US 2008070026 A1 WO 2009058558 A1	30-12-2009 05-05-2015 07-05-2009 06-10-2010 08-01-2014 20-05-2011 04-08-2010 07-10-2016 20-01-2011 10-12-2011 01-06-2009 20-03-2008 07-05-2009

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Date of mailing (day/month/year) 22 October 2018 (22.10.2018)	
Applicant's or agent's file reference 4621 WO	IMPORTANT NOTIFICATION
International application No. PCT/US2016/038714	International filing date (day/month/year) 22 June 2016 (22.06.2016)

1. The following indications appeared on record concerning:			
☐ the applicant		☒ the inventor	
☐ the agent		☐ the common representative	
Name and Address SONG, Weixin, D. 125 S. Old Creek Road Vernon Hills, Illinois 60061 United States of America		State of Nationality	State of Residence
		Telephone No.	
		Facsimile No.	
		E-mail address	
2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:			
☐ the person		☐ the name	
☒ the address		☐ the nationality	
		☐ the residence	
Name and Address SONG, Weixin, D. 1068 Huaihai Road Middle Building 2, Unit 7C Shanghai 200031 China		State of Nationality	State of Residence
		Telephone No.	
		Facsimile No.	
		E-mail address ☐ Notifications by e-mail authorized	
3. Further observations, if necessary:			
4. A copy of this notification has been sent to:			
☒ the receiving Office		☐ the International Preliminary Examining Authority	
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☐ the Authority(ies) specified for supplementary search		☐ the elected Offices concerned	
		☐ other:	

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PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

To:
see form PCT/ISA/220

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No. PCT/US2016/038714	International filing date (day/month/year) 22.06.2016	Priority date (day/month/year) 17.06.2016
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International Patent Classification (IPC) or both national classification and IPC
INV. C04B28/14

Applicant
UNITED STATES GYPSUM COMPANY

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. **FURTHER ACTION**

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA:  European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016	Date of completion of this opinion see form PCT/ISA/210	Authorized Officer Büscher, Olaf Telephone No. +31 70 340-0	
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Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:
 - ☒ the international application in the language in which it was filed.
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).
2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43bis.1(a))
3. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:
 - a. ☐ forming part of the international application as filed:
 - ☐ in the form of an Annex C/ST.25 text file.
 - ☐ on paper or in the form of an image file.
 - b. ☐ furnished together with the international application under PCT Rule 13ter.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.
 - c. ☐ furnished subsequent to the international filing date for the purposes of international search only:
 - ☐ in the form of an Annex C/ST.25 text file (Rule 13ter.1(a)).
 - ☐ on paper or in the form of an image file (Rule 13ter.1(b) and Administrative Instructions, Section 713).
4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
5. Additional comments:

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>5, 9, 15</u>
	No: Claims	<u>1-4, 6-8, 10-14, 16-20</u>
Inventive step (IS)	Yes: Claims	
	No: Claims	<u>1-20</u>
Industrial applicability (IA)	Yes: Claims	<u>1-20</u>
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item VIII

Certain observations on the international application

1 Article 6 PCT

- 1.1 The "viscosity" of a product is a parameter for which the provisions of the PCT Guidelines, Part II, Chapter 5.36 apply (further reference is made to the EPC-Guidelines, Part F, Chapter IV, 4.11, 4.18, Chapter III, 2 and 11; Part G, Chapter VI, 6).

Parameters are characteristic values, which may be values of directly measurable properties (e.g. the melting point of a substance, the flexural strength of a steel, the resistance of an electrical conductor). This fundamental condition is provided by linking the "viscosity" parameter of the present application to the so-called "VMA-method". However, the "VMA method" is not a well-known and commonly applied method in the technical field of building materials to determine the viscosity of starch, but is a method developed and used exclusively by the applicant in the present application.

Consequently, the present application contains the deficiency that a well-known parameter ("viscosity") is used to define the subject-matter for which protection is sought, whereby this parameter itself is defined by an unusual method (VMA-method) of determining this parameter.

The feature "viscosity" determined by the "VMA method" renders the scope of claims 1-20 unclear (Article 6 PCT).

A fair comparison with the prior art is limited by the use of this parameter. However, since the applicant has chosen to define the invention by aforementioned parameter own hos volition, the burden of proof rests with the applicant, that prior art pregelatinized starches are not falling under the parametric definition.

- 1.2 Claim 17 defines the amount of pregelatinized starch by an open range (greater than 10%). This allows the scope of claim 17 protection for slurries containing 98% pregelatinized starch and 1% stucco and 1% water. This is technically not plausible and therefore unclear (Article 6 PCT). Claim 17 further defines a further utilization of the slurry, by casting and drying as board. This cannot define the slurry further.

- 1.3 Claim 1 defines the parameters "density" and "core hardness" by open ranges. Neither has it been shown that a board with an infinite hardness has been manufactured or how it can be manufactured, nor is this technically plausible. The same applies for the "density". The parameters "density" and "core hardness" defined by the open ranges render the scope of claim 1 unclear (Article 6 PCT). It is a basic requirement that it must be possible for the skilled person to perform an invention over the whole range claimed, otherwise the requirements of Article 5 PCT are not sufficiently satisfied. It therefore appears that the open parameters "density" and "core hardness" cannot be achieved over the whole range and claims 1-20 do not fulfill the requirements of Article 5 PCT.
- 1.4 The defined board density is a function of the amount of foaming agent used (see also experimental section). Without the use of foaming agents the claimed board density cannot be obtained. Claims 1-20 are lacking an essential feature (Article 6 PCT taken in combination with Rule 6.3(b) PCT).
- 1.5 The term "about" used in claims 1-20 renders the features associated therewith unclear (Article 6 PCT).
- 1.6 The expression "incorporated herein by reference" used in the description is unclear (Article 5 PCT and PCT Guidelines, Part II, 4.26) and should be deleted (see [0001], [0017], [0103]).
- 1.7 The units used throughout the application are not systematically SI units and are not additionally expressed in terms of the units stipulated by Rule 10.1(a) and (b) PCT (e.g. [0125]).
- 1.8 The explanations and interpretations stipulated in [0221] are not recognized by the PCT and the PCT Guidelines.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents:

- D1 WO 2015/050804 A1 (UNITED STATES GYPSUM CO [US]) 9 April 2015
- D2 WO 2012/122102 A2 (GEORGIA PACIFIC GYPSUM LLC [US]; FRANCIS HUBERT C [US]) 13 September 2012
- D3 US 2008/070026 A1 (YU QIANG [US] ET AL) 20 March 2008

1 Preliminary remarks

- 1.1 Claim 1 defines an end product (a gypsum board) by components and properties of the slurry used to manufacture the end product.

Neither can the finished end product contain "stucco" (calcium sulfate hemihydrate is hydrated to gypsum) or water, nor can it retroactively be determined which viscosity the "pregelatinized starch" had, before it was used to manufacture the end product. As explained in [0028] of the application upon drying the green board at elevated temperatures the pregelatinized starch becomes fully gelatinized. It is impossible to determine the degree of pregelatinization and viscosity after that process step.

The product of claim 1 can only be compared with the prior art in terms of the technical features of the end product (gypsum board). Thus, the scope of claim 1 can only embrace a gypsum board comprising: a set gypsum core between 2 cover sheets, the board having a density of about 31 pcf or less and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10, method B.

- 1.2 The working examples disclosed in the experimental section of the present application cannot be used to substantiate a technical effect and an inventive step over the prior art. Example 5 shows in table 7 a suit of comparative and "inventive" examples. The ISA observes that samples 5B-5D although being considered as "inventive" are not falling under scope of claim 1, because they are not containing the pregelatinized starch in an amount *greater than* 10 wt.-

% (this definition explicitly excludes the value 10 and only includes every value greater than 10). The ISA noticed to its surprise that the "not inventive" examples 5B-5D are not shown anything special over the "inventive samples" (5E-5G). Likewise, example 3 contains only 2 wt.-% pregelatinized starch by weight of stucco (table 6). The same applies for example 4.

The features of claims 1, 12 and 17 can only be considered as juxtaposition of arbitrarily selected features for which no inventive step can be acknowledged.

2 **Article 33(1) PCT, Article 33(2) PCT & Article 33(3) PCT**

2.1 The present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of independent claims 1, 12 and 17 is not new (Article 33(2) PCT).

2.1.1 **Document D1** discloses ([0005]-[0160], examples 1-4; fig. 1-3; claims 1-10):

(1) a gypsum board having a core hardness measured according to ASTM C473-10 of >11 lbs ([0113]; [0155]) and a board density of 29 pcf. ([0154]);

(2) a method of making a gypsum board comprising:

a) mixing at least water, stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method ([0072]-[0073]; table 1A-1C), wherein the starch is present in an amount of greater than *about* 10% by weight of the stucco ([0086]-[0087]; [0091]-[0092]);

b) disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly;

c) cutting the wet assembly into a board; and

d) drying the board;

the dried board having a density of 29 pcf and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10 (claim 8; ([0113]; [0155]; [0154]);

(3) a **slurry** comprising: water stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco (claim 8; ([0072]-[0073]; table 1A-1C; ([0086]-[0087])).

Remarks:

(1) D1 discloses that the pregelatinized starch can be present in an amount of about 10% by weight of the stucco. It can therefore be present in an amount greater 10% by weight;

(2) D2 further explicitly discloses that increasing amounts of pregelatinized starch beyond the range of 0.1-10 % by weight of stucco does not improve strength, but higher amounts can be tolerated ([0087]; [0092]). This teaching will become highly relevant, once Article 33(3) PCT will be examined in a further prosecution of the present application.

The subject-matter of independent claims 1, 12 and 17 is not new over document D1 (Article 33(1) PCT).

2.1.2 **Document D2** discloses ([01]; [09]-[52], examples 1-2; claims 1-21; fig. 1-2):

(1) a **method of making a gypsum board** comprising:

a) mixing at least water, stucco, and at least one pregelatinized starch, wherein the starch is present in an amount of greater than 4% ([11]; [31]) by weight of calcined gypsum;

b) disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly ([12]);

c) cutting the wet assembly into a board; and

d) drying the board ([41]-[48]; fig. 1);

(2) a **slurry** comprising: water stucco, and at least one pregelatinized starch, wherein the starch is present in an amount of greater than 4% by weight of calcined gypsum ([11]; [31]). The hardened slurry obtains a density of 28-31 pcf. ([51]).

Remarks:

- (1) the range of "more than 4%" includes "greater 10%";
- (2) D2 is silent on the viscosity of the pregelatinized starch. Burden of proof is resting with the applicant to show that the starch of D2 does not have a viscosity of 20-300 cP if measured according to the applicants "VMA method";
- (3) D2 is silent on "core hardness". The "core hardness" must be a function of the ingredients of the slurry used to make the board. Otherwise, the claims would lack essential features of how this parameter can be obtained. Since the slurry of D2 contains identical features as the slurry and the board of the present application, the slurry and the board of D2 must have identical properties.

The subject-matter of independent claims 12 and 17 is not new over document D2 (Article 33(2) PCT).

2.1.3 **Document D3** discloses ([0001]-[0049], examples 1, 2, 6, 7; claims 1-9):

(1) a **gypsum board** having a core hardness measured according to ASTM C473-10 of >11 lbs (boards 3 and 4 in table 6 of example 6 prepared by using formulation B of example 2; [0053]) and a board density of 10-34 pcf. (samples 6-13 in table 7 of example 7, prepared by using formulation B of example 2).

The subject-matter of independent claim 1 is not new over document D3 (Article 33(2) PCT).

2.2 **Dependent claims**

Since it is impossible to retroactively determine the viscosity of pregelatinized starch before it was used to manufacture a gypsum board in the gypsum board *per se*, claims 2 and 3 are not clear and thus not new over the prior art (Article 33(1) PCT).

Identical objections apply for claims 10 and 11. These claims are not clear and thus not new over the prior art (Article 33(2) PCT).

The board of D1 can have a density of 29 pcf. Claim 6 is therefore not new over D1 (Article 33(2) PCT).

29 pcf. is *about* 27 pcf. Claims 7, 16 and 20 are therefore not new over D1 (Article 33(2) PCT).

The starch can have a viscosity of 30-200 cP ([0073]). Claims 11, 13 and 18 are therefore not new over D1 (Article 33(2) PCT).

10% by weight of stucco is *about* 10.1 wt.-% by weight of stucco. Claims 4, 14 and 19 are not new over D1 (Article 33(2) PCT).

The board of D2 can have a density of 21 or 24 pcf. Claims 8 and 16 are therefore not new over D2 (Article 33(2) PCT).

The slurry of claim 17 cannot be further defined by the density of another manufactured from this slurry as in claim 20. Claim 20 cannot be new over the prior art (Article 33(2) PCT).

Dependent claims 5, 9 and 15 do not appear to contain any additional features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT with respect to Article 33(3) PCT, because neither are surprising technical effects derivable from their characterising features, nor do these claims solve a technical problem in a non-obvious manner..

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44bis)

Applicant's or agent's file reference 4621 WO	FOR FURTHER ACTION	See item 4 below
International application No. PCT/US2016/038714	International filing date (<i>day/month/year</i>) 22 June 2016 (22.06.2016)	Priority date (<i>day/month/year</i>) 17 June 2016 (17.06.2016)
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237		
Applicant UNITED STATES GYPSUM COMPANY		

1.	This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 bis.1(a).																								
2.	This REPORT consists of a total of 11 sheets, including this cover sheet.																								
	In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.																								
3.	This report contains indications relating to the following items: <table style="width: 100%;"> <tr> <td style="width: 5%; text-align: center;">☒</td> <td style="width: 25%;">Box No. I</td> <td style="width: 70%;">Basis of the report</td> </tr> <tr> <td style="text-align: center;">☐</td> <td>Box No. II</td> <td>Priority</td> </tr> <tr> <td style="text-align: center;">☐</td> <td>Box No. III</td> <td>Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</td> </tr> <tr> <td style="text-align: center;">☐</td> <td>Box No. IV</td> <td>Lack of unity of invention</td> </tr> <tr> <td style="text-align: center;">☒</td> <td>Box No. V</td> <td>Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</td> </tr> <tr> <td style="text-align: center;">☐</td> <td>Box No. VI</td> <td>Certain documents cited</td> </tr> <tr> <td style="text-align: center;">☐</td> <td>Box No. VII</td> <td>Certain defects in the international application</td> </tr> <tr> <td style="text-align: center;">☒</td> <td>Box No. VIII</td> <td>Certain observations on the international application</td> </tr> </table>	☒	Box No. I	Basis of the report	☐	Box No. II	Priority	☐	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability	☐	Box No. IV	Lack of unity of invention	☒	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement	☐	Box No. VI	Certain documents cited	☐	Box No. VII	Certain defects in the international application	☒	Box No. VIII	Certain observations on the international application
☒	Box No. I	Basis of the report																							
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☒	Box No. VIII	Certain observations on the international application																							
4.	The International Bureau will communicate this report to designated Offices in accordance with Rules 44bis.3(c) and 93bis.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44bis .2).																								

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland Facsimile No. +41 22 338 82 70	Date of issuance of this report 18 December 2018 (18.12.2018) Authorized officer Agnès Wittmann-Regis e-mail: pct.team6@wipo.int
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PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

To:
see form PCT/ISA/220

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing (day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference see form PCT/ISA/220

FOR FURTHER ACTION See paragraph 2 below
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International application No. PCT/US2016/038714	International filing date (day/month/year) 22.06.2016	Priority date (day/month/year) 17.06.2016
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International Patent Classification (IPC) or both national classification and IPC INV. C04B28/14

Applicant UNITED STATES GYPSUM COMPANY

1. This opinion contains indications relating to the following items:
- ☒ Box No. I Basis of the opinion
 - ☐ Box No. II Priority
 - ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
 - ☐ Box No. IV Lack of unity of invention
 - ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
 - ☐ Box No. VI Certain documents cited
 - ☐ Box No. VII Certain defects in the international application
 - ☒ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA:  European Patent Office P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Fax: +31 70 340 - 3016	Date of completion of this opinion see form PCT/ISA/210	Authorized Officer Büscher, Olaf Telephone No. +31 70 340-0	
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Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:
 - ☒ the international application in the language in which it was filed.
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).
2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43bis.1(a))
3. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:
 - a. ☐ forming part of the international application as filed:
 - ☐ in the form of an Annex C/ST.25 text file.
 - ☐ on paper or in the form of an image file.
 - b. ☐ furnished together with the international application under PCT Rule 13ter.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.
 - c. ☐ furnished subsequent to the international filing date for the purposes of international search only:
 - ☐ in the form of an Annex C/ST.25 text file (Rule 13ter.1(a)).
 - ☐ on paper or in the form of an image file (Rule 13ter.1(b) and Administrative Instructions, Section 713).
4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
5. Additional comments:

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>5, 9, 15</u>
	No: Claims	<u>1-4, 6-8, 10-14, 16-20</u>
Inventive step (IS)	Yes: Claims	
	No: Claims	<u>1-20</u>
Industrial applicability (IA)	Yes: Claims	<u>1-20</u>
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Re Item VIII

Certain observations on the international application

1 Article 6 PCT

- 1.1 The "viscosity" of a product is a parameter for which the provisions of the PCT Guidelines, Part II, Chapter 5.36 apply (further reference is made to the EPC-Guidelines, Part F, Chapter IV, 4.11, 4.18, Chapter III, 2 and 11; Part G, Chapter VI, 6).

Parameters are characteristic values, which may be values of directly measurable properties (e.g. the melting point of a substance, the flexural strength of a steel, the resistance of an electrical conductor). This fundamental condition is provided by linking the "viscosity" parameter of the present application to the so-called "VMA-method". However, the "VMA method" is not a well-known and commonly applied method in the technical field of building materials to determine the viscosity of starch, but is a method developed and used exclusively by the applicant in the present application.

Consequently, the present application contains the deficiency that a well-known parameter ("viscosity") is used to define the subject-matter for which protection is sought, whereby this parameter itself is defined by an unusual method (VMA-method) of determining this parameter.

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- 1.2 Claim 17 defines the amount of pregelatinized starch by an open range (greater than 10%). This allows the scope of claim 17 protection for slurries containing 98% pregelatinized starch and 1% stucco and 1% water. This is technically not plausible and therefore unclear (Article 6 PCT). Claim 17 further defines a further utilization of the slurry, by casting and drying as board. This cannot define the slurry further.

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The product of claim 1 can only be compared with the prior art in terms of the technical features of the end product (gypsum board). Thus, the scope of claim 1 can only embrace a gypsum board comprising: a set gypsum core between 2 cover sheets, the board having a density of about 31 pcf or less and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10, method B.

- 1.2 The working examples disclosed in the experimental section of the present application cannot be used to substantiate a technical effect and an inventive step over the prior art. Example 5 shows in table 7 a suit of comparative and "inventive" examples. The ISA observes that samples 5B-5D although being considered as "inventive" are not falling under scope of claim 1, because they are not containing the pregelatinized starch in an amount *greater than* 10 wt.-

% (this definition explicitly excludes the value 10 and only includes every value greater than 10). The ISA noticed to its surprise that the "not inventive" examples 5B-5D are not shown anything special over the "inventive samples" (5E-5G). Likewise, example 3 contains only 2 wt.-% pregelatinized starch by weight of stucco (table 6). The same applies for example 4.

The features of claims 1, 12 and 17 can only be considered as juxtaposition of arbitrarily selected features for which no inventive step can be acknowledged.

2 **Article 33(1) PCT, Article 33(2) PCT & Article 33(3) PCT**

2.1 The present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of independent claims 1, 12 and 17 is not new (Article 33(2) PCT).

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(1) a **gypsum board** having a core hardness measured according to ASTM C473-10 of >11 lbs ([0113]; [0155]) and a board density of 29 pcf. ([0154]);

(2) a **method of making a gypsum board** comprising:

a) mixing at least water, stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method ([0072]-[0073]; table 1A-1C), wherein the starch is present in an amount of greater than *about* 10% by weight of the stucco ([0086]-[0087]; [0091]-[0092]);

b) disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly;

c) cutting the wet assembly into a board; and

d) drying the board;

the dried board having a density of 29 pcf and a core hardness of at least about 11 lbs, as determined according to ASTM C473-10 (claim 8; ([0113]; [0155]; [0154]);

(3) a **slurry** comprising: water stucco, and at least one pregelatinized starch having a viscosity of from about 20 cP to about 300 cP as measured according to the VMA method, wherein the starch is present in an amount of greater than 10% by weight of the stucco (claim 8; ([0072]-[0073]; table 1A-1C; ([0086]-[0087])).

Remarks:

(1) D1 discloses that the pregelatinized starch can be present in an amount of about 10% by weight of the stucco. It can therefore be present in an amount greater 10% by weight;

(2) D2 further explicitly discloses that increasing amounts of pregelatinized starch beyond the range of 0.1-10 % by weight of stucco does not improve strength, but higher amounts can be tolerated ([0087]; [0092]). This teaching will become highly relevant, once Article 33(3) PCT will be examined in a further prosecution of the present application.

The subject-matter of independent claims 1, 12 and 17 is not new over document D1 (Article 33(1) PCT).

2.1.2 **Document D2** discloses ([01]; [09]-[52], examples 1-2; claims 1-21; fig. 1-2):

(1) a **method of making a gypsum board** comprising:

a) mixing at least water, stucco, and at least one pregelatinized starch, wherein the starch is present in an amount of greater than 4% ([11]; [31]) by weight of calcined gypsum;

b) disposing the slurry between a first cover sheet and a second cover sheet to form a wet assembly ([12]);

c) cutting the wet assembly into a board; and

d) drying the board ([41]-[48]; fig. 1);

(2) a **slurry** comprising: water stucco, and at least one pregelatinized starch, wherein the starch is present in an amount of greater than 4% by weight of calcined gypsum ([11]; [31]). The hardened slurry obtains a density of 28-31 pcf. ([51]).

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- (1) the range of "more than 4%" includes "greater 10%";
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- (3) D2 is silent on "core hardness". The "core hardness" must be a function of the ingredients of the slurry used to make the board. Otherwise, the claims would lack essential features of how this parameter can be obtained. Since the slurry of D2 contains identical features as the slurry and the board of the present application, the slurry and the board of D2 must have identical properties.

The subject-matter of independent claims 12 and 17 is not new over document D2 (Article 33(2) PCT).

2.1.3 **Document D3** discloses ([0001]-[0049], examples 1, 2, 6, 7; claims 1-9):

(1) a **gypsum board** having a core hardness measured according to ASTM C473-10 of >11 lbs (boards 3 and 4 in table 6 of example 6 prepared by using formulation B of example 2; [0053]) and a board density of 10-34 pcf. (samples 6-13 in table 7 of example 7, prepared by using formulation B of example 2).

The subject-matter of independent claim 1 is not new over document D3 (Article 33(2) PCT).

2.2 **Dependent claims**

Since it is impossible to retroactively determine the viscosity of pregelatinized starch before it was used to manufacture a gypsum board in the gypsum board *per se*, claims 2 and 3 are not clear and thus not new over the prior art (Article 33(1) PCT).

Identical objections apply for claims 10 and 11. These claims are not clear and thus not new over the prior art (Article 33(2) PCT).

The board of D1 can have a density of 29 pcf. Claim 6 is therefore not new over D1 (Article 33(2) PCT).

29 pcf. is *about* 27 pcf. Claims 7, 16 and 20 are therefore not new over D1 (Article 33(2) PCT).

The starch can have a viscosity of 30-200 cP ([0073]). Claims 11, 13 and 18 are therefore not new over D1 (Article 33(2) PCT).

10% by weight of stucco is *about* 10.1 wt.-% by weight of stucco. Claims 4, 14 and 19 are not new over D1 (Article 33(2) PCT).

The board of D2 can have a density of 21 or 24 pcf. Claims 8 and 16 are therefore not new over D2 (Article 33(2) PCT).

The slurry of claim 17 cannot be further defined by the density of another manufactured from this slurry as in claim 20. Claim 20 cannot be new over the prior art (Article 33(2) PCT).

Dependent claims 5, 9 and 15 do not appear to contain any additional features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT with respect to Article 33(3) PCT, because neither are surprising technical effects derivable from their characterising features, nor do these claims solve a technical problem in a non-obvious manner..