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(71) Applicant: **S.A. LHOIST RECHERCHE ET DEVELOPPEMENT** [BE/BE]; Rue Charles Dubois 28, 1342 Ottignies Louvain-la-Neuve (BE).

(72) Inventors: **GÄRTNER, Robert S.**; O. de Kerchove d'Exaerdestraat 186, 1501 Buizingen (BE). **BALLARD, Deborah A.**; 7575 Surfside Drive, Fort Worth, Texas 76135 (US). **LIGNER, Emmanuelle**; Rue de Sirault 41,

7334 Villerot (BE). **LORGUILLOUX, Marion**; Rue des Canadiens 57, 7110 Strépy Bracquegnies (BE).

(74) Agent: **CALYSTA NV**; Lambroekstraat 5A, 1831 Diegem (BE).

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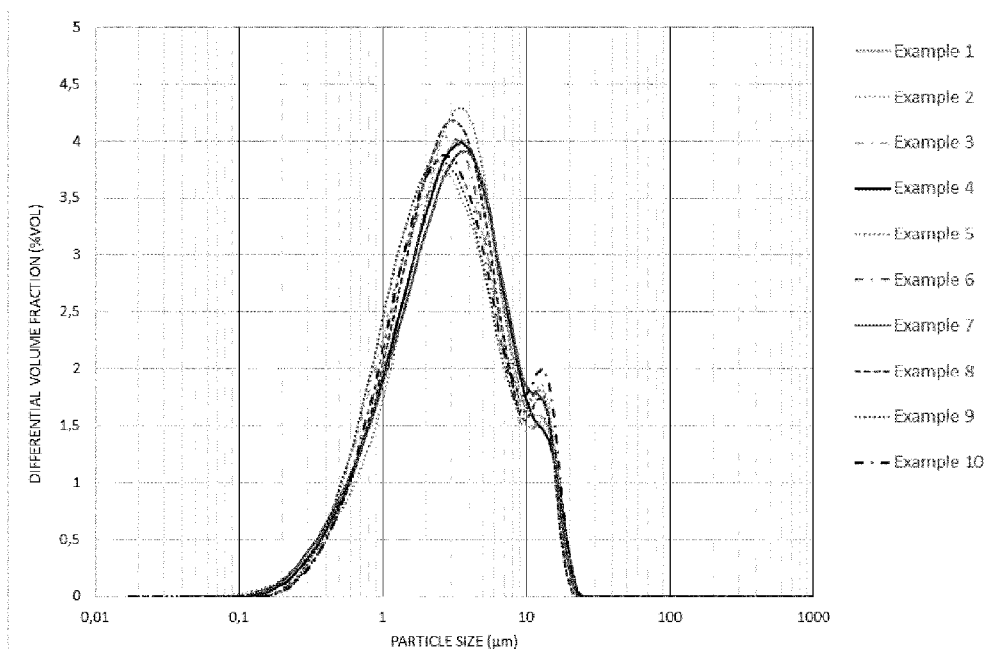


Figure 1

(57) Abstract: Ready-to-use powder formulation and its manufacturing process for making a high solids highly reactive milk of lime with improved stability and settling, comprising particles of high calcium lime, an organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5wt% and a dispersant polymer at an amount of 0.03 to 0.3 wt%, and containing at least 90wt% of high calcium lime, said ready-to-use powder formulation having a d₉₇ < 40µm and a d₅₀ comprised between 2 and 15µm.



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**« READY-TO-USE POWDER FORMULATION FOR MAKING A HIGHLY
REACTIVE, HIGH SOLIDS MILK OF LIME »**

The present invention relates to a ready-to-use powder formulation for making a highly reactive, high solids milk of lime having an improved viscosity and settling stability and to its manufacturing process.

5 Lime is a calcium-magnesium based compound herein after called calcium-based compound.

Calcium based compounds such as CaO and Ca(OH)₂ have many practical uses. For instance, these substances are used in treating drinking water, wastewater and sewage, industrial streams, in flue gas cleaning, as soil neutralizing
10 agents and nutrients, for ground stabilization, for construction, and as components of building materials or as strong base or calcium-source chemicals.

"Quicklime" is an industrial product mainly made of calcium oxide, i.e. CaO and some impurities at a level of a few %, while "hydrated lime" is an industrial product mainly made of calcium hydroxide, i.e. Ca(OH)₂ and some impurities at a
15 level of a few %, both sometimes being informally referred to as "lime". Quicklime is usually in the form of lumps or pebbles but it can also be a powder. Dry hydrated lime is usually a powder. In the further, we will refer to quicklime by its main component, CaO, and to hydrated lime by its main component, Ca(OH)₂.

Quicklime can be hydrated to form hydrated lime (also called
20 sometimes slaked lime).

The slaked lime is comprised of a set of solid particles, mainly of calcium dihydroxide of formula Ca (OH)₂, and is the result of industrial slaking or hydration of a quicklime with water, also referred to as a hydration reaction. This product is also known under the name of hydrated lime. Subsequently, the
25 dihydroxide will be simply called calcium hydroxide.

The hydration reaction is a heavily exothermic reaction and is chemically as follows:

$$\text{CaO} + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2$$
As a function of the extent of the excess water implementation, there are several possibilities to obtain an industrially slaked lime starting with quicklime (see in particular BOYNTON, R.S., Chemistry and Technology
30 of lime and limestone, Second ed., New York: Wiley, 1980, XII, pp.327-328). The

extinction mode also determines the form in which is presented the slaked lime: dry hydrate, paste, suspension/milk of lime.

In a slaking mode "via a dry route" (hydration) the amount of added water is limited to that which is required for the slaking reaction, increased with the amount lost as steam because of the exothermic nature of the reaction. Upon exiting the hydrator, the obtained product is powdery and generally comprises both at least 2% of residual non-hydrated CaO and less than 2% of humidity, with a maximum of 4% of humidity. It may be packaged and sold directly, after optional steps for controlling grain size, but without any preliminary drying and de-agglomeration step.

In a previous slaking mode alternative, the hydration may be achieved with a larger excess of water according to WO 97/14650. In this case, the obtained hydrate contains humidity of the order of 15 to 30% by mass. Because of this humidity, the hydrated lime requires a drying and de-agglomeration step before storage and transport. The drawback of adding a drying/de-agglomeration step in the production line is compensated by the fact that the water excess used during the reaction allows development of the specific surface area and of the porous volume of the hydrate which may then, by means of these characteristics, be used in specific applications, notably for the treatment of acid gases. This slaking mode according to WO 97/14650, will be called a slaking mode via a "quasi-dry route".

In a slaking mode "via a wet route", the amount of added water is in very large excess as compared with the amount strictly required for the slaking reaction. A milk of lime is then obtained, i.e. an aqueous suspension of slaked lime particles.

When the amount of water used for the slaking reaction is a little smaller, the obtained product is pasty and this is referred to as slaking "via a putty route" (lime putty).

In the present document, a slaking mode "via a non-wet route" will designate slaking modes, grouping slaking modes via a dry route, via a quasi-dry route and any intermediate between both of them (a hydrate obtained with a humidity comprised between 2 and 15%) excluding the two slaking modes via a humid route and via a slurry route.

Depending on the use of lime, in order to further process powdery compounds and improve the ease, with which they are handled, dry CaO or dry Ca(OH)₂ are often mixed with water to form an aqueous suspension, i.e., a slurry, called milk of lime, which is a fluid suspension of slaked lime, also called hydrated

lime, which will inevitably include some impurities, in particular silica, alumina, unburnt limestone (CaCO_3), magnesium oxide or magnesium hydroxide to the extent of a few percent.

Such a suspension is obtained either by slaking quicklime (calcium oxide— CaO) with a large excess of water, or by mixing hydrated lime with water (slurrying step). The resulting aqueous suspensions are characterized by the concentration of the mass of the solid matter (% solids), the chemical reactivity of the slurry to neutralize an acid and the distribution of the sizes of the particles in suspension (strongly influencing the suspension viscosity and settling behavior).

These characteristics determine the properties of the slurry, mainly its viscosity and its reactivity.

When a milk of lime is obtained by slurrying hydrated lime, hydrated lime particles are suspended in water. The hydrated lime is produced by common atmospheric hydrators, which may or may not have size classifying systems. In this case, in the hydrator, a slaking step "via a non-wet route" is carried out where water is added to quicklime, possibly in a pre-mixer, at a specific mass ratio and allowed to mix together. The particle size distribution will vary depending upon the nature of the quicklime starting material used, the applied process conditions, as well as from the manufacturing process or manufacturing plant (presence of a size classifying system or not, presence of a screening or milling system or not, presence of a pre-mixer or not, ...). Milk of lime made from hydrated lime will have a particle size distribution similar to that of the hydrate from which it is produced, and the solids content can vary typically from 5 to 50 w %.

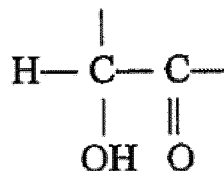
Milk of lime made from quicklime (by slaking quicklime via a putty route or a wet route), in a commercial, continuous process, is notably produced by common tank reactors or by special paste slaker, detention slaker, or ball mill slaker (Boyton, 1980). In all cases, quicklime is added to an excess amount of water and mixed together, to produce slurry with a solid content ranging from 5 to 50 w %. The water reacts with the quicklime particles during the slaking operation in an exothermic reaction to form slaked lime. During the atmospheric slaking of quicklime with an excess of water, the temperature of hydration is below 100° C. (212° F.). The particle size distribution of the milk of lime or of the particles of the milk of lime is a function of both the nature of the quicklime, the slaking process conditions and the coarse fraction removal systems, which include screening, settling and milling.

Lime slurries can be made either in batches or in a continuous process. The process of slaking and its resulting milk of lime qualities are widely used industrially across many industries and as a result reasonably well understood.

Of course, the biggest demand for ready-to-use milk-of-lime on the market involves high solid concentration slurries since shipping milk of lime involves a lot of water transportation. Unfortunately, high solid concentration slurries are viscous and more difficult to handle since viscosity typically increases quite rapidly, especially when the milk of lime is very reactive, meaning that it contains hydrated lime with fine particle size.

WO2006/050567 discloses milk of lime containing dispersant and viscosity modifiers, which can be added to an aqueous slurry or paste of lime to lower the viscosity and improve the flow characteristics of the slurry which in turn makes the high-solids product easier to handle. According to this document, the milk of lime comprises polycarboxylate dispersants and carbohydrate dispersants. Preferably, the dispersant comprises a polycarboxylate dispersant in combination with a carbohydrate dispersant with a non-volatile weight ratio of the polycarboxylate dispersant to carbohydrate dispersant is in the ratio 1 :20 to 10:1, more preferably 1:20 to 1:1.

The recited carbohydrates in WO2006/050567 which have been found to be suitable for lowering the viscosity of calcium hydroxide aqueous slurries comprise aldoses, saccharides, disaccharides, polysaccharides and synthetic derivatives from such precursors, for example, glycerol, sorbitol, mannitol, gluconic, citric, isocitric, lactic, tartaric acids and salts thereof, dextrose, maltose, glucose, lactose, saccharose, maltotriose, maltotetraose, both alpha and beta glucoheptonic acids and salts thereof. Many of these are commercially available in syrup form, e.g., glucose syrup and corn syrup. Carbohydrates are compounds of carbon, hydrogen, and oxygen that contain the saccharose unit or its first reaction product.



and in which the ratio of hydrogen to oxygen is the same as in water.

Polycarboxylate dispersants useful according to WO2006/050567 are molecules or polymers that contain multiple carboxyl (COOH) groups which can

form salts with metals and amine, such as styrene maleic anhydride copolymers in the form of their sodium or other alkali metal salts. Generally, excellent performance is achieved when the polycarboxylate dispersant is in the form of an addition copolymer. Suitable copolymers include salts of polyether polycarboxylates. These
5 may be formed from acrylic acid and hydrophilic polyalkylene oxides such as polyethylene and polypropylene oxides. An example of such a copolymer is Ethacryl(R) G from Lyondell Chemie Nederland B. V (currently produced by Arkema, France). This is a polyether polycarboxylate, sodium salt supplied as a 40% aqueous solution.

10 According to WO2006/050567, both the carbohydrate dispersant and polycarboxylate dispersant have been added to the slaking water before adding the quicklime in the water. The viscosity was stable for about 48 hours (i.e. meaning that viscosity was not increasing above 1000 mPa s (cP).

Unfortunately, such milk of lime can settle over time and document
15 EP1999082 deals with this issue.

Indeed, document EP1999082 discloses milk of lime having a great fineness and a dry matter content between 1 and 60 wt% having rheological properties maintained in time (such as dynamic viscosity, shearing yield or plastic viscosity) at levels compatible with their utilization, implementation, handling in time
20 in stationary, dynamic or mixed conditions. According to this document, this aim is achieved in a milk of lime by the addition of one or several additives chosen among carbohydrates or some of their derivatives, such as the ones obtained by oxidation or by hydrogenation of these carbohydrates.

The milk of lime according to EP 1999082 can be obtained by slaking
25 calcic lime and/or dolomitic lime in water, by slurring in water powdery hydrated lime and/or hydrated dolomitic lime or even by dilution of calcium and/or magnesium hydrate paste or calcic and/or dolomitic lime paste in water. The additives are disclosed to be added anywhere in the process (to the quicklime, to the hydrated lime, before, during or after the slaking process or dilution process or
30 even to the slaking water being the illustrated way in the examples). The resulting milk of lime shows a viscosity below 2000 mPa.s (cP) (as measured with a Haake Rotovisco RT20 viscosimeter at a shearing speed of 5 s⁻¹), preferably below 1500 mPa.s, at best below 1200 mPa.s after an ageing period of 12 hours under agitation at 600 rpm.

The possible additives according to EP 1999082 are chosen from the long list made by erythrose, threose, xylose, ribose, allose, glucose, galactose, fructose, mannose, sucrose, lactose, maltose, trehalose, starch, modified starch, hydrolysed starch, glycogen, inuline, modified inuline, cellulose, modified cellulose, pectins, dextrans and cyclodextrins, those glucides further filed into oligosaccharides or polysaccharides, according to their effective degree of polymerization, chitin, glucan, glycosaminoglucans, agar, pectin, xanthene gum, guar gum, carob, glucomannane, sorbitol or glucitol, erythritol, xylitol, lactitol, maltitol, glucosamine or oxidized forms such as uronic acids (specifically galacturonic acid, glucuronic acid), gluconic acid, as well as the salts of these acids, originating from syrups and/or molasses derived from sugar cane, beets and chicory or in solid form, such as powder, in the form of a paste or as an aqueous or non-aqueous solution. They can be crystalline, semi-crystalline or amorphous.

While those documents offer promising solutions for improving the stability of milk of lime, the stability is still limited over time, thereby creating many difficulties when the milk of lime is manufactured at the lime producer facilities and then shipped to- and stored at the user's facilities.

It is also known from Belgian Patent BE1026329 B1 or international patent application WO2020/094607 that the combination of carbohydrate additive, notably sucrose, and a polyether-polyacrylate dispersant such as Rheopersperse dispersant available at Coatex SAS, can achieve low viscosity and limited viscosity increase over time, i.e. 14 days of storage under intermittent agitation (5 min/h), even for a fine hydrated lime, i.e. down to d_{50} of ca. 2 μm and $d_{98} < 10 \mu\text{m}$ (see example 4, Table 7)

However, even if of high solids content, the lime milk usually contains more than 50 weight % of water to be transported. The present invention aims to solve at least a part of the aforementioned drawbacks by providing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling allowing shipping the lime in a dry state and preparing the milk of lime at the user's facilities by simply adding the dry hydrated lime to water to form a slurry with these unique properties.

To solve this problem, it is provided according to the present invention a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling comprising solid particles of high calcium dry hydrated lime, , an organic stabilizing agent in C₄ to C₁₈ bearing one or

more hydroxyl group(s) at an amount from 0.3 to 5 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation and a dispersant polymer at an amount of 0.03 to 0.3 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation containing
5 at least 90wt% of high calcium lime based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation having a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm .

The wording "particles of high calcium lime" within the meaning of the present invention means particles containing at least 88 wt% of hydrated lime,
10 preferably at least 90 wt%, more preferably at least 92 wt% and even more preferably at least 93 wt% hydrated lime relative to the weight of the solid matter, such as the particles. The content of hydrated lime in the solid matter can be determined by the amount of available lime according to the standard EN 459-2, §5.8.

It has been indeed highlighted according to the present invention that
15 it is possible to provide a dry formulation which can be manufactured off-site and simply added to water on site.

According to the present invention, the organic stabilizing agent and the dispersant polymer are already contained in the ready-to-use formulation without impairing the properties of the final milk of lime. According to the present
20 invention, it was surprisingly possible, against all expectations to have the dispersant polymer easily & swiftly recovering from its collapsed state by addition of water. Further, the dispersant polymer become active again, while the presence of calcium ions would have thought away the man skilled in the art from adding it to a calcium-based compound as it is generally considered that calcium ions are detrimental to
25 the unfolding of the collapsed polymer and to the regaining of the dispersant functionality. Indeed, dispersant polymers, like polycarboxylate polymers change their so-called conformation (the "outer shape" of the polymer chain) both with concentration and calcium content. Both will force the polymer into a more compact form, which will reduce its efficiency as a dispersant. A good dispersant
30 should have stretched chains to create distance between the solid particle by so-called steric repulsion. A contracted or collapsed conformation occurring by complete removal of the solvent (i.e. the water), will act less or even not at all as a dispersant.

Further, organic stabilizing agent in C_4 to C_{18} bearing one or more
35 hydroxyl group(s) like sugars contain typically either aldehyde (e.g. glucose and its

di-, tri- and oligosaccharides) or ketone groups (e.g. fructose and its di-, tri or oligosaccharides). Such groups are well known to be highly susceptible to the so-called aldol reaction or aldol addition under alkaline conditions, which can convert the aldehyde into an alcohol or an acid in the case of polyalcohol – or lead to shortening of the carbon-chain in the polyol. This is especially likely in keto-sugars like fructose. This is explained in some detail in "Sugar Activation by Alkali" by P.A. Shaffer & T.E. Friedemann in Journal of Biological Chemistry (<https://www.jbc.org/content/86/1/345.full.pdf>) or "Concentrated Lactic Acid Production from Invert Sugar in Alkaline Solution" by Rinlada Sirisangsawang & Prakob Kitchaiya in Chem. Eng. Trans, No. 75, 2019 (<https://www.aidic.it/cet/19/75/103.pdf>).

Due to this decomposition, it is even more surprising according to the present invention that the organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) which would possibly decompose to different products, keep their initial similar effect in stabilizing lime slurry viscosity over time.

In addition, the dispersant polymer in combination with an organic stabilizing agent made it possible to provide a ready-to-use powder formulation stable on storage under a dry state and which can form after being slurried a highly reactive, high solids milk of lime having an improved viscosity and settling stability.

When the milk of lime is prepared on site with a standard slurring equipment, e.g. a stirred tank, it shows a stable and improved viscosity in such a way that it does not exceed 1350 mPa s, preferably 1200 mPa s, more preferably 1000 mPa s, preferably it does not exceed 600 mPa s, more preferably 500 mPa s, more preferably 400 mPa s after 14 days of dispersion in water, either in resting state or in sheared state.

By the term viscosity, it is meant in the present application, dynamic or absolute viscosity measured in the centipoise (cP) unit or in the millipascal-second (mPa s) unit. One centipoise is equal to one millipascal-second (mPa s) in the International system of units. The viscosity according to the present invention is measured according to standard industry practice, as by the use of a "Brookfield DV III Rheometer" viscometer, with the suitable spindle according to the observed torque or viscosity, typically LV spindle N° 3, at 100 rpm. The measurement was taken on the 30th second, once the viscometer motor was turned on.

According to the present invention, the viscosity measurements have been made under different states, i.e. under a "resting state" or "at rest" and under a "sheared state", or "under agitation".

5 Within the meaning of the present invention, it is meant by the terms "resting state" or "at rest", a state which is considered typically achieved after 2 to 4 hrs at rest, i.e. without agitation, movement or flow. In this state, it is assumed, that the lime particles have arranged themselves in a network, which increases the viscosity and the yield strength, i.e. the force required to make the product flow.

10 By the term "sheared state" or "under agitation", it is meant the opposite state where the aforementioned network-state has been destroyed, so that the resistance to flow is reduced. While viscosity is still decreasing with increasing movement, the sheared state has lower viscosity and lower yield strength. The agitation can be continuous or applied for a part of the day, each day, like during one hour per day.

15 According to the present invention, the stable and improved viscosity was observed even for the very high solid contents (such as between 40 wt% to 60 wt%) while keeping a very fine particle size distribution, thereby allowing the preparation of a milk of lime having also a high reactivity which can be advantageous for applications such as pH adjustment in general, remineralization, neutralization, precipitation, coating, lubrication or flocculation.

20 In a preferred embodiment, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is present at an amount from 0.4 to 2.0 wt%, most preferably 0.5 to 1.2 wt% based on the total weight of the ready-to-use powder formulation. The given wt% are defined by the wt% of non-volatile matter, i.e. the content of organic stabilizing agent and not the amount of solution or syrup when the organic stabilizing agent is not commercialized under solid form. The amount of organic stabilizing agent can be determined by conventional analytic method, such as by extraction of the organic stabilizing agent with a solvent from the particles of high calcium lime and characterizing the amount of organic stabilizing agent by HPLC & GC-MS.

25 In another preferred embodiment, said dispersant polymer is present at an amount of 0.04 to 0.25 wt%, most preferably 0.05 to 0.20 wt%, based on the total weight of the ready-to-use powder formulation. The given wt% are defined by the wt% of non-volatile matter, i.e. the content of dispersant polymer and not the amount of solution of dispersant polymer under which form it is commercialized. The

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amount of dispersant polymer can be determined by conventional analytic method, such as by extraction of the dispersant polymer with a solvent from the particles of high calcium lime and characterizing the amount of dispersant polymer by HPLC & GC-MS & gel-permeation chromatography & FTIR.

5 Preferably, the dispersant polymer is a hydrophilic polymer dispersant ideally soluble in water, i.e. a dispersant polymer characterized by a high content in hydrophilic groups, such as ether, ester, hydroxyl and carboxylate groups.

Advantageously, said ready-to-use powder formulation has a d_{97} lower than or equal to 30 μm , preferably lower than or equal to 20 μm , most
10 preferably lower than or equal to 15 μm .

In addition, said ready-to-use powder formulation has a d_{97} greater than or equal to 10 μm , preferably greater than or equal to 12 μm .

In a further advantageous embodiment of the ready-to-use formulation, the particles have a d_{50} lower than 12 μm , more preferably lower than
15 10 μm , particularly lower than or equal to 8 μm , more particularly lower than or equal to 6 μm .

The notation d_x represents a characteristic diameter expressed in μm , measured by laser granulometry in methanol after sonication, compared to which the size of $x\%$ by volume of the particles measured are less than or equal to this
20 diameter.

In a preferred embodiment, the ready-to-use powder formulation is packaged in an industrial container, such as in 25 to 50 kg bags or about 1 m^3 big bags, in 10 m^3 , 20 m^3 or larger silo, transportable containers and the like.

In a preferred embodiment, the ready-to-use powder formulation for
25 making a high solids highly reactive milk of lime having an improved stability and settling has a silica content expressed in SiO_2 equivalent lower than 1.5 wt%, preferably lower than or equal to 1.1 wt%, particularly lower than or equal to 0.85 wt%, more preferably lower than or equal to 0.65 wt%, even more particularly lower than or equal to 0.5 wt% based on the total weight of the ready-to-use powder
30 formulation.

In another preferred embodiment, the ready-to-use powder formulation according to the present invention has an aluminum content expressed in Al_2O_3 equivalent lower than 0.40 wt%, preferably lower than or equal to 0.35 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than
35 or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

In yet another preferred embodiment, the ready-to-use powder formulation according to the present invention has a sulfur content expressed in SO_3 equivalent lower than 0.5 wt%, preferably lower than or equal to 0.4 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

According to the present invention, the particles of the ready-to-use powder formulation have a specific surface area measured by manometric nitrogen adsorption and calculated using the Brunauer, Emmett and Teller model (BET method) after degassing 2 h at 190°C comprised between 5 and $45 \text{ m}^2/\text{g}$, more preferably between 7 and $30 \text{ m}^2/\text{g}$, more particularly between 8 and $25 \text{ m}^2/\text{g}$.

According to the present invention, the particles of the ready-to-use powder formulation have a total BJH pore volume, constituted of pores, the diameter of which are higher than or equal to 17 \AA and less than or equal to 1000 \AA greater than or equal to $0.02 \text{ cm}^3/\text{g}$, preferably greater than or equal to $0.022 \text{ cm}^3/\text{g}$ and generally lower or equal to $0.15 \text{ cm}^3/\text{g}$, preferably lower than $0.10 \text{ cm}^3/\text{g}$, even between 0.025 and $0.08 \text{ cm}^3/\text{g}$ obtained by the desorption of nitrogen.

According to the present invention, the particles of the ready-to-use powder formulation have a total BJH pore volume made up of pores having a diameter between 100 and 300 \AA , obtained by nitrogen desorption, greater than or equal to $0.01 \text{ cm}^3/\text{g}$, preferably greater than or equal to 0.02, preferably greater than or equal to $0.03 \text{ cm}^3/\text{g}$.

By the expression "BJH pore volume", it is meant according to the present invention, the pore volume measured by manometry with nitrogen adsorption at 77 K after degassing under vacuum at a temperature lying between 150 and 250°C , notably at 190°C . for at least 2 hours and calculated according to the BJH method using the desorption graph, with the hypothesis of a cylindrical pore geometry.

By the term "total pore volume", according to the present invention, it is meant the BJH pore volume made up of pores having a diameter less than or equal to 1000 \AA (17 to 1000 Angströms).

In another preferred embodiment according to the present invention, the particles of the ready-to-use powder formulation presents a monomodal size distribution.

By the terms monomodal, it is meant that the particle size distribution curve does not present more than one peak.

In a particular embodiment of the present invention, the ready-to-use powder formulation present having a flowability comprised in the range of 1.2 to 2.0, preferably between 1.3 and 1.8 as measured according to ASTM D6128 standard.

The advantageous flowability of the ready-to-use powder formulation according to the present invention is kept at a very good level despite the addition of organic stabilizing agent and/or dispersant polymer through an aqueous solution of them. Indeed, typically, even small amounts of water (1 to 2wt%) can impair the flow behavior of powdery hydrated lime, especially for ultrafine hydrated lime used according to the present invention. The flowability of the ready-to-use powder formulation is an important aspect to contribute to the "ready-to-use" aspect of the formulation according to the present invention.

The behaviour of powder in a storage silo may be simulated with another method using a rheometer for powder, such as the Brookfield powder flow tester (PFT) according to the ASTM D6128 norm. In this method, a powder sample is introduced into the equipment and is subjected to compaction increasing over time. At each compression stage (principal consolidation stress), a specific torque is applied to the powder until failure (non-confined failure restriction). The response of the powder to the restriction applied is recorded on a computer, which evaluates the static cohesion of the tested sample. The results are shown by a graph which is compared to the ASTM references.

In another particular embodiment according to the present invention, the ready-to-use powder formulation presents a moisture content comprised between 0.2 and 3 wt%, preferably between 0.3 and 2 wt%, most preferably between 0.5 and 1.5 wt%, based on the total weight of the powder formulation as measured by infra-red thermobalance at 150°C. Alternatively, similar result can be obtained by thermogravimetric analysis at the same temperature.

According to the present invention, the moisture content is measured by a loss of ignition test at 150° C.

Preferably, the organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups is chosen amongst an uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a monosaccharide, a disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

More preferably, said dispersant polymer is a water-soluble polymer preferably having an active polycarboxylate ether concentration of at least 10 wt%, more preferably of at least 15 wt% and even more preferably of at least 20 wt%.

5 Other embodiments of the ready-to-use powder formulation for making a milk of lime having an improved stability according to the present invention are mentioned in the appended claims.

The present invention also relates to a process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

10 The process according to the present invention comprises the steps of

- A hydration step of quicklime in a hydrator during which solid particles mainly comprised of quicklime are contacted with water and hydrated to form high calcium dry hydrated lime solid particles, said hydrator having different zones, said solid particles being transported from a feeding zone to an exit zone of hydrated lime solid particles for example by a screw, said hydration step comprising a series of aqueous phase additions at a series of locations between the feeding zone and the exit zone A first addition of an organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% relative to the total weight of the ready-to-use powder formulation, and

20 • A second addition of a dispersant polymer at an amount of 0.03 to 0.3 wt% active polymer, i.e. based on non-volatile content relative to the total weight of the ready-to-use powder formulation,

- One amongst said first addition and said second addition being performed in the hydrator to said solid particles upstream of the collecting zone, optionally through at least one addition of said series of aqueous addition, and the other amongst said first addition and said second addition being performed before, during or after said hydration step,

- A size control step of said high calcium dry hydrated lime solid particles to provide a predetermined amount of high calcium dry hydrated lime solid particles having a particle size distribution with a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm and

30 • A collecting step of a ready -to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling comprising solid particles of high calcium dry hydrated lime, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to

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5 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation and said dispersant polymer at an amount of 0.03 to 0.3 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation containing at least 90wt% of high calcium lime based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation having a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm .

By the terms "series of aqueous phase additions", it is meant, within the meaning of the present invention that the process comprises 1, 2, 3, ...20, 40, 100, or even more additions of aqueous phase along the hydrator, and when the number of aqueous phase addition is at least 2, the aqueous phases can be the same or different to each other.

By the terms "series of locations", it is meant, within the meaning of the present invention that the hydrator comprises 1, 2, 3, ...20, 40, 100, or even more locations where an aqueous phase can be added, and when the number of locations is at least 2, the aqueous phase added at one location can be the same or different to the addition at another location.

By the terms "solid particles mainly comprised of quicklime", it is meant within the meaning of the present invention that the solid particles comprise at least 50% of quicklime. Indeed, the solid particles in the feeding zone can be the entering quicklime or the entering partially hydrated lime (i.e. quicklime to which a small portion of water was added upstream the feeding zone to increase the control of the hydration process. Such water addition provides particles having a core of quicklime and typically a shell of hydrated lime. The thickness of the hydrated lime layer depends on the amount of water added to the solid particles. "solid particles mainly comprised of quicklime" mean also the particles of quicklime or partially hydrated lime which starts encountering the hydration process, especially when water or moisture is added in the feeding zone..

It has been surprisingly realized according to the present invention that the dispersant polymer retains its dispersing function after having been added and stored on a dry hydrated lime powder, as the dispersant function requires, that the dispersant polymer is in its unfolded/ stretched conformation. This conformation is only normally attained in solution, due to the effect of the water molecules, which bond to the polymer's functional groups and prevent the polymer from contracting.

During drying, i.e. either intentionally in a drying step or by evaporation due to contact with ambient or process air, e.g. in a classification or pneumatic conveying equipment, on the lime, the dispersant polymer should contract to into a bunched shape, as in a dried state the functional groups attract each other. It was
5 in fact surprising to note that from such dried state in a hydrate formulation according to our invention, the dispersant polymer easily recovers its functionality when slurried with water to form the milk of lime. This would indicate that the dispersant polymer actually readily unfolds again. This is indeed unexpected as the bond between functional groups is quite strong and the water should be hindered
10 from entering the hardened polymer (like dissolving dried-in bread-dough).

During the hydration step according to the present invention, the quicklime is fed to a hydrator through a feeding zone. Possibly, quicklime is beforehand pre-hydrated in a prehydrator located upstream the feeding zone. Solid particles of mainly quicklime (i.e. quicklime or partially pre-hydrated quicklime) are
15 conveyed along the hydrator and undergoes the hydration before reaching the collecting zone, at the end of the hydrator under the form of high calcium dry hydrated lime solid particles, which can then further been classified or dried or both for forming the ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

20 Between the feeding zone and the collecting zone, a "non-wet slaking step" is performed in a reaction zone by addition at one or more location of one or more aqueous phase, which can be the same or different to each other. In this intermediate zone, the population of solid particles is a mixture of hydrated particles, partially prehydrated particles and quicklime depending on the progression of the
25 hydration process, the location along the conveying way of the solid particles, but also the mixing conditions and the distance between the addition point in the hydrator and where the particles are located. If the addition is performed centrally the particles present at the periphery of the hydrator can be less hydrated than the particles present in the near proximity of the addition point. If the addition is
30 performed peripherally in the hydrator, the particles near the central axis of the hydrator are less hydrated, with the difference of the level of hydration becoming smaller and smaller when the particles are at the end of their convey in the hydrator.

It was indeed observed according to the present invention that performing one amongst the first and second addition in the hydrator allows to reach
35 a ready-to-use powder formulation for making a high solids highly reactive milk of

lime having an improved stability and settling which is homogenous and very well controlled beside being easy to integrate to industrial production of slaked lime.

Preferably, in the process according to the present invention, said size control step is chosen amongst a dry or wet milling step, a mil classification step, an
5 air classification step or a combination of two or more thereof before collecting said ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

Preferably, the process according to the present invention comprises a packaging step of the ready-to-use powder formulation for making a milk of lime
10 having an improved stability in an industrial container, such as in 25 to 50 kg bags or about 1 m³ big bags, in 10 m³, 20 m³ or larger silo, transportable containers and the like.

In a preferred embodiment according to the present invention, said one amongst said first addition and said second addition is performed at one or more
15 locations in the hydrator to said dry hydrated lime solid particles, optionally through at least one addition of said series of aqueous additions.

In another preferred embodiment, said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to partially hydrated lime solid particles, optionally through at least one addition of said
20 series of aqueous phase additions.

In yet another preferred embodiment, said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to said solid particles mainly comprised of quicklime, optionally through at least one addition of said series of aqueous phase additions.

Of course, it is contemplated according to the present invention that
25 two or three of the three previous embodiments described above can be combined.

In one embodiment according to the present invention, the one amongst said first addition and said second addition is a solid-state addition or alternatively a liquid state addition.

In one embodiment, the other amongst said first addition and said
30 second addition is a solid-state addition or alternatively a liquid state addition.

In one further embodiment, the other amongst said first addition and said second addition is performed to at least one aqueous phase addition during said one amongst said first addition and said second addition.

In one further embodiment, the other amongst said first addition and said second addition is performed to at least one aqueous phase addition before the hydration step.

5 In one further embodiment, the other amongst said first addition and said second addition is performed to said solid particles mainly comprised of quicklime before the hydration step.

In one further embodiment according to the present invention, the other amongst said first addition and said second addition is performed to partially hydrated lime solid particles during hydration step, preferably in the reaction zone.

10 In one embodiment according to the present invention, the other amongst said first addition and said second addition is performed to said dry hydrated lime solid particles upstream of the collecting zone.

Indeed, in one variant of the preferred embodiment, the first addition is performed before said slaking step, i.e. to the slaking water or to the quicklime. For example, it is contemplated that the organic stabilizing agent is in solid state and is mixed to the quicklime before the slaking step. It is also for example foreseen that the organic stabilizing agent is in solid or liquid state and added to the water for slaking the quicklime.

15 In another variant according to the present invention, because the other amongst said first addition and said second addition can also be the second addition, it is contemplated that the second addition is performed before said slaking step, i.e. to the slaking water or to the quicklime. For example, it is contemplated that the polymer dispersant is in solid state and is mixed to the quicklime before the slaking step. It is also for example foreseen that the polymer dispersant is in solid or liquid state and added to the water for slaking the quicklime.

20 As explained before, quicklime is typically slaked in a hydrator having different zones, said solid particles being transported from the feeding zone (quicklime entry) to the collecting zone (slaked lime exit) for example by a screw. The water being possibly added at one location or at several locations between the feeding zone and the collecting zone. It is foreseen according to the present invention that the first addition is performed during said slaking step, i.e. to the slaking water or to the quicklime or quicklime at least partially hydrated depending on the location between the quicklime entry and hydrated lime exit, at once or progressively along the traveling of the solid lime between the quicklime entry and the hydrated lime exit.

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According to the present invention, the second addition can be performed after said slaking step, i.e. to the slaked lime, optionally before or after a further drying step.

In yet another variant, because the other amongst said first addition and said second addition can also be the second addition, it is foreseen according to the present invention that the second addition is performed during said slaking step, i.e. to the slaking water or to the quicklime at least partially hydrated depending on the location between the quicklime entry and hydrated lime exit, at once or progressively along the traveling of the solid lime between the quicklime entry and the hydrated lime exit.

In still another variant, the second addition is performed after said slaking step, i.e. to the slaked lime, optionally before or after a further drying step.

The first addition and the second addition can be performed together or separately. One amongst the first and second addition can be performed before the slaking step and the other amongst the first and second additions can be performed during or after the slaking step.

According to the present invention, the dispersant polymer is added under a solid form or under a form of a suspension or solution.

Preferably, when the dispersant polymer is added as a dry powder, it has a maximum particle size lower than or equal to 500 μm , preferably lower than or equal to 200 μm . When the dispersant polymer is added as a solution, the solution presents a dispersant polymer content lower than or equal to 10wt%, preferably lower than or equal to 20wt%.

In addition, the organic stabilizing agent can be added under a solid form or under a form of a suspension or solution.

Preferably, when the organic stabilizing agent is added as a dry powder, it has a maximum particle size lower than or equal to 500 μm , preferably lower than or equal to 200 μm . When the organic stabilizing agent is added as a solution, the solution presents an organic stabilizing agent content greater than or equal to 15wt%, preferably greater than or equal to 30wt%.

It is foreseen according to the present invention that the dispersant polymer can be added under a suspension or a solution and the organic stabilizing agent can be added under a solid form or a suspension or solution form. Also, It is foreseen according to the present invention that the dispersant polymer can be

added under a solid form and the organic stabilizing agent can be added under a solid form or a suspension or solution form.

In a particular embodiment, the hydration step is carried out in presence of calcium nitrate or calcium chloride at an amount of 0.3 to 2 wt%, preferably between 0.5 and 1.5 wt% with respect to the weight of solid particles mainly comprised of quicklime.

In one embodiment, said second addition is an addition of a water-soluble dispersant polymer preferably having an active polycarboxylate ether concentration of at least 10 wt%, more preferably of at least 15 wt% and even more preferably of at least 20 wt%.

In another preferred embodiment, said dispersant polymer is added at an amount of 0.04 to 0.25 wt%, most preferably 0.05 to 0.20 wt%, based on the total weight of the ready-to-use powder formulation.

Preferably, the dispersant polymer is a hydrophilic polymer dispersant ideally soluble in water, i.e. a dispersant polymer characterized by a high content in hydrophilic groups, such as ether, ester, hydroxyl and carboxylate groups.

In one embodiment, said first addition is an addition of at least one organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups which is chosen amongst a uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a monosaccharide, a disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

In a preferred embodiment, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is added at an amount from 0.4 to 2.0 wt%, most preferably 0.5 to 1.2 wt% based on the total weight of the ready-to-use powder formulation.

In a variant of the process according to the present invention, said process further comprises a step of spraying an additional amount of organic stabilising agent in C₄ to C₁₈ bearing one or more hydroxyl groups and/or of a polymer dispersant on the high calcium dry hydrated lime solid particles.

Advantageously, said ready-to-use powder formulation has a d₉₇ lower than or equal to 30 µm, preferably lower than or equal to 20 µm, most preferably lower than or equal to 15µm.

In addition, said ready-to-use powder formulation has a d₉₇ greater than or equal to 10 µm, preferably greater than or equal to 12 µm.

In a further advantageous embodiment of the ready-to-use formulation, the particles have a d_{50} lower than 12 μm , more preferably lower than 10 μm , particularly lower than or equal to 8 μm , more particularly lower than or equal to 6 μm .

5 Other embodiments of the process according to the present invention are mentioned in the appended claims.

Other characteristics and advantages of the present invention will be derived from the non-limitative following description, and by making reference to the drawings and the examples.

10 Figure 1 shows the full particle size distribution of the samples of hydrated lime according to examples 1 to 10.

Figure 2 shows the results of both viscosity measurement at rest and viscosity measurement under 1 hour/day gentle agitation for milk of lime obtained with hydrated lime samples from examples 1 and 7.

15 Figure 3 shows the viscosity over time for the milk of lime obtained with hydrated lime samples from examples 7 and 8.

Figure 4 shows the viscosity over time at rest of milks of lime prepared from the samples from examples 2 and 3

20 Figure 5 shows the viscosity over time of milks of lime prepared from the samples from examples 2 and 3 under a gentle agitation during one hour each day.

Figure 6 shows the viscosity over time at rest of milks of lime prepared from the samples from examples 9 and 10.

Figure 7 shows the viscosity over time of milks of lime prepared from the samples from examples 9 and 10 under a gentle agitation during one hour each day.

25 The present invention relates to a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling comprising particles of high calcium lime, i.e. particles containing at least 88 wt% of hydrated lime, preferably at least 90 wt%, more preferably at least 92 wt% and even more preferably at least 93 wt% hydrated lime relative to the weight of the solid matter, such as the particles. The content of hydrated lime in the solid matter can be determined by the amount of available lime according to the standard EN 459-2, §5.8, which can be manufactured off-site and simply added to water on site.

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The manufacturing off-site can accordingly be realized at the lime manufacturer site and then shipped to the final customer without requiring shipping water and improving the storage time of the product.

5 The present invention relates accordingly also to a process for manufacturing the ready-to-use formulation for further making a high solids highly reactive milk of lime having an improved stability and settling and the ready-to-use powder formulation directly obtained by the process according to the present invention.

The ready-to use powder formulation comprises:

- 10 (i) an organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt%, preferably below or equal to 3 wt% and more preferably below or equal to 2 wt%
- 15 (ii) a dispersant polymer at an amount of 0.03 to 0.3 wt% based on the total weight of the ready-to-use powder formulation, preferably between 0.04 to 0.25 wt%, more preferably between 0.05 and 0.22 wt%

The ultimate ready-to-use powder formulation contains at least 90 wt% of high calcium lime based on the total weight of the ready-to-use powder formulation and has a particle size distribution such that the characteristic diameter $d_{97} \leq 40 \mu\text{m}$ and the characteristic diameter d_{50} is comprised between 2 and 15 μm .

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According to the present invention, the organic stabilizing agent and the dispersant polymer are already contained in the ready-to-use formulation without impairing the properties of the final milk of lime since the dispersant polymer easily & swiftly recovers from its collapsed state by addition of water and becomes active again and very importantly made it possible to provide a ready-to-use powder formulation stable on storage under a dry state and which can form after being slurried a highly reactive, high solids milk of lime having an improved viscosity and settling stability.

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30 When the milk of lime is prepared on site with a standard slurring equipment, e.g. a stirred tank, it shows a stable and improved viscosity in such a way that it does not exceed 1350 mPa s, preferably 1200 mPa s, more preferably 1000 mPa s, preferably it does not exceed 600 mPa.s, more preferably 500 mPa s, more preferably 400 mPa s after 14 days of dispersion in water, either in resting state or in

35 sheared state.

According to the present invention, the viscosity measurements have been made under different states, i.e. under a "resting state" or "at rest" and under a "sheared state", or "under agitation".

In a preferred embodiment, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is present at a preferred amount from 0.4 to 2.0 wt%, most preferably 0.5 to 1.2 wt% based on the total weight of the ready-to-use powder formulation. The given wt% are defined by the wt% of non-volatile matter, i.e. the content of organic stabilizing agent.

In another preferred embodiment, said dispersant polymer is present at an amount of 0.03 to 0.25 wt%, most preferably 0.04 to 0.20 wt%, non-volatile matter based on the total weight of the ready-to-use powder formulation. The given wt% are defined by the wt% of non-volatile matter, i.e. the content of dispersant polymer.

The weight ratio in non-volatile content between the dispersant polymer and the organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is comprised between 0.04 and 0.4 and preferably between 0.06 and 0.35.

Preferably, the dispersant polymer is a hydrophilic polymer dispersant ideally soluble in water, i.e. a dispersant polymer characterized by a high content in hydrophilic groups, such as ether, ester, hydroxyl and carboxylate groups.

Advantageously, said ready-to-use powder formulation has a d_{97} lower than or equal to 30 μm , preferably lower than or equal to 20 μm , most preferably lower than or equal to 15 μm .

In addition, said ready-to-use powder formulation has a d_{97} greater than or equal to 10 μm , preferably greater than or equal to 12 μm .

In a further advantageous embodiment of the ready-to-use formulation, the particles have a d_{50} lower than 12 μm , more preferably lower than 10 μm , particularly lower than or equal to 8 μm , more particularly lower than or equal to 6 μm .

The notation d_x represents a characteristic diameter expressed in μm , measured by laser diffraction granulometry in methanol as carrier solvent after sonication (applied after dispersion in carrier solvent is performed), compared to which the size of x% by volume of the particles measured are less than or equal to this diameter.

In a preferred embodiment, the ready-to-use powder formulation is packaged in an industrial container, such as in 25 to 50 kg bags or about 1 m³ big bags, in 10 m³, 20 m³ or larger silo, transportable containers and the like.

5 The ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling has preferably a silica content expressed in SiO₂ equivalent lower than 1.5 wt%, preferably lower than or equal to 1.1 wt%, particularly lower than or equal to 0.85 wt%, more preferably lower than or equal to 0.65 wt%, even more particularly lower than or equal to 0.5 wt% based on the total weight of the ready-to-use powder formulation.

10 The ready-to-use powder formulation according to the present invention has preferably an aluminum content expressed in Al₂O₃ equivalent lower than 0.40 wt%, preferably lower than or equal to 0.35 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

15 The ready-to-use powder formulation according to the present invention has preferably a sulfur content expressed in SO₃ equivalent lower than 0.5 wt%, preferably lower than or equal to 0.4 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

20 The particles of the ready-to-use powder formulation have typically a specific surface area measured by manometric nitrogen adsorption and calculated using the Brunauer, Emmett and Teller model (BET method) after degassing 2 h at 190°C comprised between 5 and 45 m²/g, more preferably between 7 and 30 m²/g, more particularly between 8 and 25 m²/g.

25 The particles of the ready-to-use powder formulation have further typically a total BJH pore volume, constituted of pores, the diameter of which are higher than or equal to 17 Å and less than or equal to 1000 Å greater than or equal to 0.02 cm³/g, preferably greater than or equal to 0.022 cm³/g and generally lower than or equal to 0.15 cm³/g, preferably lower than 0.10 cm³/g, even, between 0.025 and
30 0.08 cm³/g, obtained by the desorption of nitrogen.

The particles of the ready-to-use powder formulation have generally a total BJH pore volume made up of pores having a diameter between 100 and 300 Å, obtained by nitrogen desorption, greater than or equal to 0.01 cm³/g, preferably greater than or equal to 0.02, preferably greater than or equal to 0.03 cm³/g.

The particles of the ready-to-use powder formulation presents a monomodal size distribution.

The ready-to-use powder formulation presents particularly a flowability comprised in the range of 1.2 to 2.0, preferably between 1.3 and 1.8 as measured
5 according to ASTM D6128 standard.

Further, the ready-to-use powder formulation presents generally a moisture content comprised between 0.2 and 3 wt%, preferably between 0.3 and 2 wt%, most preferably between 0.5 and 1.5 wt%, based on the total weight of the powder formulation as measured by infra-red thermobalance at 150°C.
10 Alternatively, similar result can be obtained by thermogravimetric analysis at the same temperature.

Preferably, the organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups is chosen amongst a uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a
15 monosaccharide, a disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

A preferred group of organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups comprise a mixture of monosaccharides as organic stabilizing agent, like a mixture of two or more amongst fructose, glucose, dextrose
20 or an inverted sugar syrup, sorbitol or xylitol, gluconic acid or sodium gluconate or disaccharides, like sucrose, maltose, lactose or saccharose.

More preferably, said dispersant polymer is a water-soluble polymer preferably having an active polycarboxylate ether concentration of at least 10 wt%, more preferably of at least 15 wt% and even more preferably of at least 20 wt%.

25 The present invention also relates to a process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

The process according to the present invention comprises the steps of

- A hydration step of quicklime in a hydrator during which solid particles
30 mainly comprised of quicklime are contacted with water and hydrated to form high calcium dry hydrated lime solid particles, said hydrator having different zones, said solid particles being transported from a feeding zone to an exit zone of hydrated lime solid particles for example by a screw or a paddle shaft, said hydration step comprising a series of aqueous phase additions at a series of locations between the
35 feeding zone and the exit zone A first addition of an organic stabilizing agent in C₄

to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% relative to the total weight of the ready-to-use powder formulation, and

- A second addition of a dispersant polymer at an amount of 0.03 to 0.3 wt% active polymer, i.e. based on non-volatile content relative to the total weight of the ready-to-use powder formulation,

- One amongst said first addition and said second addition being performed in the hydrator to said solid particles upstream of the collecting zone, optionally through at least one addition of said series of aqueous addition, and the other amongst said first addition and said second addition being performed before, during or after said hydration step,

- A size control step of said high calcium dry hydrated lime solid particles to provide a predetermined amount of high calcium dry hydrated lime solid particles having a particle size distribution with a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm and

- A collecting step of a ready -to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling comprising solid particles of high calcium dry hydrated lime, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation and said dispersant polymer at an amount of 0.03 to 0.3 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation containing at least 90wt% of high calcium lime based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation having a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm

It has been surprisingly realized according to the present invention that the dispersant polymer retains its dispersing function after having been added and stored on a dry hydrated lime powder, as the dispersant function requires, that the dispersant polymer is in its unfolded/ stretched conformation. This conformation is only normally attained in solution, due to the effect of the water molecules, which bond to the polymer's functional groups and prevent the polymer from contracting.

During drying, i.e. either intentionally in a drying step or by evaporation due to contact with ambient or process air, e.g. in a classification or pneumatic conveying equipment, on the lime, the dispersant polymer should contract to into a

bunched shape, as in a dried state the functional groups attract each other. It was in fact surprising to note that from such dried state in a hydrate formulation according to our invention, the dispersant polymer easily recovers its functionality when slurried with water to form the milk of lime. This would indicate that the
5 dispersant polymer actually readily unfolds again. This is indeed unexpected as the bond between functional groups is quite strong and the water should be hindered from entering the hardened polymer (like dissolving dried-in bread-dough).

During the hydration step according to the present invention, the quicklime is fed to an hydrator through a feeding zone. Possibly, quicklime is
10 beforehand pre-hydrated in a prehydrator located upstream the feeding zone. Solid particles of mainly quicklime (i.e. quicklime or partially pre-hydrated quicklime) are conveyed along the hydrator and undergoes the hydration before reaching the collecting zone, at the end of the hydrator under the form of high calcium dry hydrated lime solid particles, which can then further be classified or dried or both
15 for forming the ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

Between the feeding zone and the collecting zone, a "non-wet slaking step" is performed by addition at one or more location of one or more aqueous phase, which can be the same or different to each other. In this intermediate zone,
20 the population of solid particles is a mixture of hydrated particles, partially prehydrated particles and quicklime depending on the progression of the hydration process, the location along the conveying way of the solid particles, but also the mixing conditions and the distance between the addition point in the hydrator and where the particles are located. If the addition is performed centrally the particles
25 present at the periphery of the hydrator can be less hydrated than the particles present in the near proximity of the addition point. If the addition is performed peripherally in the hydrator, the particles near the central axis of the hydrator are less hydrated, with the difference of the level of hydration becoming smaller and smaller when the particles are at the end of their convey in the hydrator.

30 It was indeed observed according to the present invention that performing one amongst the first and second addition in the hydrator allows to reach a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling which is homogenous and very well controlled beside being easy to integrate to industrial production of slaked lime.

Preferably, in the process according to the present invention, said size control step is chosen amongst a dry or wet milling step, a mil classification step, an air classification step or a combination of two or more thereof before collecting said ready-to-use powder formulation for making a high solids highly reactive milk of lime
5 having an improved stability and settling.

Preferably, the process according to the present invention comprises a packaging step of the ready-to-use powder formulation for making a milk of lime having an improved stability in an industrial container, such as in 25 to 50 kg bags or about 1 m³ big bags, in 10 m³, 20 m³ or larger silo, transportable containers and the
10 like.

In a preferred embodiment according to the present invention, said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to said dry hydrated lime solid particles, optionally through at least one addition of said series of aqueous additions.

In another preferred embodiment, said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to partially hydrated lime solid particles, optionally through at least one addition of said series of aqueous phase additions.
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In yet another preferred embodiment, said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to said solid particles mainly comprised of quicklime, optionally through at least one addition of said series of aqueous phase additions.
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Of course, it is contemplated according to the present invention that two or three of the three previous embodiments described above can be combined.

In one embodiment according to the present invention, the one amongst said first addition and said second addition is a solid-state addition or alternatively a liquid state addition.
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In one embodiment, the other amongst said first addition and said second addition is a solid-state addition or alternatively a liquid state addition.

In one further embodiment, the other amongst said first addition and said second addition is performed to at least one aqueous phase addition during said one amongst said first addition and said second addition.
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In one further embodiment, the other amongst said first addition and said second addition is performed to at least one aqueous phase addition before the hydration step.
35

In one further embodiment, the other amongst said first addition and said second addition is performed to said solid particles mainly comprised of quicklime before the hydration step, preferably in the reaction zone.

5 In one further embodiment according to the present invention, the other amongst said first addition and said second addition is performed to partially hydrated lime solid particles during hydration step.

In one embodiment according to the present invention, the other amongst said first addition and said second addition is performed to said dry hydrated lime solid particles upstream of the collecting zone.

10 Indeed, in one variant of the preferred embodiment, the first addition is performed before said hydration step, i.e. to the slaking (hydration) water or to the quicklime. For example, it is contemplated that the organic stabilizing agent is in solid state and is mixed to the quicklime before the hydration step. It is also for example foreseen that the organic stabilizing agent is in solid or liquid state and added to the
15 water for hydrating the quicklime.

In another variant according to the present invention, because the other amongst said first addition and said second addition can also be the second addition, it is contemplated that the second addition is performed before said hydration step, i.e. to the slaking (hydration) water or to the quicklime. For example,
20 it is contemplated that the polymer dispersant is in solid state and is mixed to the quicklime before the hydration step. It is also for example foreseen that the polymer dispersant is in solid or liquid state and added to the water for hydrating the quicklime.

As explained before, quicklime is typically slaked in a hydrator having
25 different zones, said solid particles being transported from the feeding zone (quicklime entry) to the collecting zone (slaked lime exit) for example by a screw. The water being possibly added at one location or at several locations between the feeding zone and the collecting zone. It is foreseen according to the present invention that the first addition is performed during said hydration step, i.e. to the
30 slaking (hydration) water or to the quicklime or quicklime at least partially hydrated depending on the location between the quicklime entry and hydrated lime exit, at once or progressively along the traveling of the solid lime between the quicklime entry and the hydrated lime exit.

According to the present invention, the second addition can be performed after said hydration step, i.e. to the slaked lime, optionally before or after a further drying step.

In yet another variant, because the other amongst said first addition
5 and said second addition can also be the second addition, it is foreseen according to the present invention that the second addition is performed during said hydration step, i.e. to the slaking (hydration) water or to the quicklime at least partially hydrated depending on the location between the quicklime entry and hydrated lime exit, at once or progressively along the traveling of the solid lime between the quicklime
10 entry and the hydrated lime exit.

In still another variant, the second addition is performed after said hydration step, i.e. to the slaked lime, optionally before or after a further drying step.

The first addition and the second addition can be performed together or separately. One amongst the first and second addition can be performed before
15 the hydration step and the other amongst the first and second additions can be performed during or after the hydration step.

According to the present invention, the dispersant polymer is added under a solid form or under a form of a suspension or solution.

Preferably, when the dispersant polymer is added as a dry powder, it
20 has a maximum particle size lower than or equal to 500 μm , preferably lower than or equal to 200 μm . When the dispersant polymer is added as a solution, the solution presents a dispersant polymer content lower than or equal to 10wt%, preferably lower than or equal to 20wt%.

In addition, the organic stabilizing agent can be added under a solid
25 form or under a form of a suspension or solution.

Preferably, when the organic stabilizing agent is added as a dry powder, it has a maximum particle size lower than or equal to 500 μm , preferably lower than or equal to 200 μm . When the organic stabilizing agent is added as a solution, the solution presents an organic stabilizing agent content greater than or
30 equal to 15wt%, preferably greater than or equal to 30wt%.

It is foreseen according to the present invention that the dispersant polymer can be added under a suspension or a solution and the organic stabilizing agent can be added under a solid form or a suspension or solution form. Also, It is foreseen according to the present invention that the dispersant polymer can be

added under a solid form and the organic stabilizing agent can be added under a solid form or a suspension or solution form.

In one embodiment, said second addition is an addition of a water-soluble dispersant polymer preferably having an active polycarboxylate ether
5 concentration of at least 10 wt%, more preferably of at least 15 wt% and even more preferably of at least 20 wt%.

In a preferred embodiment, the process comprises a step of spraying an additional amount of organic stabilising agent in C₄ to C₁₈ bearing one or more hydroxyl groups and/or of a polymer dispersant on the high calcium dry hydrated
10 lime solid particles.

In yet a preferred embodiment, the hydration step is carried out in presence of calcium nitrate or calcium chloride at an amount of 0.3 to 2 wt%, preferably between 0.5 and 1.5 wt% with respect to the weight of solid particles mainly comprised of quicklime.

In another preferred embodiment, said dispersant polymer is added at an amount of 0.04 to 0.5wt%, most preferably 0.05 to 0.2wt%, based on the total weight of the ready-to-use powder formulation.

Preferably, the dispersant polymer is a hydrophilic polymer dispersant ideally soluble in water, i.e. a dispersant polymer characterized by a high content in
20 hydrophilic groups, such as ether, ester, hydroxyl and carboxylate groups.

In one embodiment, said first addition is an addition of at least one organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups which is chosen amongst a uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a monosaccharide, a
25 disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

In a preferred embodiment, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is added at an amount from 0.4 to 2.0 wt%, most preferably 0.5 to 1.2 wt% based on the total weight of the ready-to-use powder formulation.

30

Examples

Examples 1 to 3.- Hydration of quicklime with sorbitol as organic stabilizing agent and Neomere® Tech 646 from Chryso SAS as dispersant polymer

Quicklime used for the hydration trials is a < 90 µm lime from Germany. The quicklime present the following composition: CaO(tot) 93.5%wt, MgO 1.0 %wt,

SiO₂ 1.0 %wt, Al₂O₃ 0.3 %wt, SO₃ 0.13% wt and presents a reactivity characterized by a t₆₀ of 2.0 min as measured according to the European standard EN459-2 .

Quicklime was fed to a pilot lab hydrator at a lime feeding rate of 200 g/min. The hydrator is working in a continuous mode and the flowrate of aqueous phase added to the hydrator is controlled to carry out a non-wet slaking, meaning that a powdery hydrated lime is obtained at the end of the hydration step. More precisely, the aqueous phase flow rate was adapted to the moisture measured at the outlet of the hydrator. The target moisture was around 2%

Sorbitol and Neomere® Tech 646 were added to aqueous phase added during the hydration reaction or hydration reaction according to the amounts mentioned in table 1.

At the outlet of the hydrator, the product was collected and stored in a bucket for some days before being classified or milled with the Hosokawa air/mill classifier. The parameter setting of the Hosokawa classifier were chosen according to the particle size distribution target. PSD target was a d₉₇ at 20µm and d₅₀ at 3µm. The mentioned amount in the table is the wt% non-volatile matter added to the lime (i.e. not taking the solvent into account)

Table 1.-

Example	Calcium nitrate (Wt% based on the quicklime weight)	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Classification <20 µm
E1	1	1.1	0.17	Air classification
E2	1	1.5	0.22	Milling classification
E3	1	1.8	0.22	Milling classification

20

Examples 4 to 6.- Hydration of quicklime with inverted sugar mixture as organic stabilizing agent and Neomere® Tech 646 from Chryso SAS as dispersant polymer

Example 1 was reproduced except that sorbitol was replaced by inverted sugar mixture containing 70 wt% carbohydrate content and 30% water has been used. The carbohydrate content is made by 33 wt% glucose, 33 wt% fructose and 33 wt% sucrose.

5 The amount of organic stabilizing agent (inverted sugar mixture) and dispersant polymer (Neomere® Tech 646) used are mentioned in table 2. The mentioned amount in the table is the wt% non-volatile matter added to the lime (i.e. not taking the solvent into account)

Table 2.-

Example	Calcium nitrate (Wt% based on the quicklime weight)	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Classification <20 µm
E4	1	1.3	0.17	Air classification
E5	1	1.5	0.22	Milling classification
E6	1	1.8	0.22	Milling classification

10

Examples 7 to 10.- Hydration of quicklime with carbohydrate mixture as organic stabilizing agent and Neomere® Tech 646 from Chryso SAS as dispersant polymer

15 Example 1 was reproduced except that sorbitol was replaced by a inverted sugar mixture under the form of a syrup containing 70wt% carbohydrate content and 30% water has been used. The carbohydrate content is made by about 40 wt% fructose, about 53% glucose, about 3 wt% maltose and between 3 and 4 wt% higher sugar. This mixture is commonly called & commercially available under the trade name high fructose corn syrup.

20 The amount of organic stabilizing agent (carbohydrate mixture) and dispersant polymer (Neomere® Tech 646) used are mentioned in table 3. The mentioned amount in the table is the wt% non-volatile matter added to the lime (i.e. not taking the solvent into account)

Table 3.-

Example	Calcium nitrate (Wt% based on the quicklime weight)	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Wt% of the organic stabilizing agent with respect to the weight of ready-to-use hydrated lime particles	Classification <20 μm
E7	1	1.1	0.17	Air classification
E8	1	1.1	0.17	Milling classification
E9		1.5	0.22	Milling classification
E10	1	1.8	0.22	Milling classification

Porosity measures

5 All the hydrated lime samples produced from examples 1 to 10 have been submitted to a manometric nitrogen adsorption test and the BET specific surface area was calculated using the Brunauer, Emmett and Teller model (BET method) after degassing 2 h at 190°C The results are mentioned in table 4.

10 All the hydrated lime samples produced from examples 1 to 10 have been submitted to a total BJH pore volume measurement by manometry with nitrogen adsorption at 77 K after degassing under vacuum at a temperature at 190° C. for at least 2 hours and calculated according to the BJH method using the desorption graph, with the hypothesis of a cylindrical pore geometry and taking the pores, for which the diameters are 17 to 1000 Å, into account. The results are mentioned in table 4.

15

Table 4.-

Example	BET specific surface area m^2/g	BJH total pore volume cm^3/g
E1	9.9	0.028
E2	8.1	0.029
E3	7.1	0.022

E4	7.7	0.025
E5	7.8	0.031
E6	7.4	0.030
E7	7.7	0.025
E8	7.6	0.027
E9	6.8	0.023
E10	6.8	0.023

As it can be seen, all the hydrated lime samples produced from examples 1 to 10 water exhibited a BET specific surface area in the range 7 to 10 m²/g and a BJH total pore volume comprised between 0.022 and 0.032.

5

Particle size distribution measures

The particle size distribution of the hydrated lime samples produced from examples 1 to 10 have been measured by laser diffraction after sonication in methanol as solvent carrier (applied after dispersion in the carrier solvent). **Error! Reference source not found.** gives the characteristic diameter d₉₇ and d₅₀ for all the

10 hydrated lime samples from examples 1 to 10.

Table 5.-

Example	d₅₀ (μm)	d₉₀ (μm)
E1	3.1	15.3
E2	2.7	15.3
E3	2.8	15.2
E4	3.2	15.6
E5	3.2	15.7
E6	2.9	15.7
E7	3.2	15.3
E8	3.0	14.8
E9	2.7	14.8
E10	3.0	16.0

Figure 1 shows the full particle size distribution. As it can be seen, All the hydrated lime samples produced from examples 1 to 10 are equivalent in term of

particle size distribution The top cut is sharp, and no tails can be seen on the PSD curves.

Slurrying and viscosity stability measurement

5 The hydrated lime obtained from examples 1, 2 and 3, 7 to 10 have been slurried to form milk of lime with a solid lime content of 45 wt%. The milks of lime were prepared by slurrying the ready-to-use formulation made by hydrated lime particles to which a first organic stabilizing agent and a second polymer dispersant have been added during the hydration process.

10 Dispersion was done using a 80 mm propeller stirrer at ca. 300 rpm producing ca. 2.5 dm³ of 45%wt suspension. Stability of suspension viscosity was monitored for ca. 2 weeks by a Brookfield DV III Rheometer with LV spindle N° 3, at 100 rpm, following 2 methods: at rest and under 1hr/day gentle agitation. The measurement was taken on the 30th second, after the rheometer/viscometer motor was turned on.

15 Additionally, settling of the sample was measured on a graduated 1ltr bottle of suspension. Degree of settling is defined as percentage of clear supernatant over the fluid height in the bottle.

The results of the viscosity measurement at rest and viscosity measurement under 1 hour/day gentle agitation of the milk of lime obtained with the hydrated lime sample from example 1 and example 7 are presented in figure 2.

20 The results of the viscosity measurement at rest and viscosity measurement under 1 hour/day gentle agitation of the milk of lime obtained with the hydrated lime sample from example 7 and example 8 are presented in figure 3. It can be seen that the viscosity development over time is relatively stable. It can be seen that the viscosity of the milk of lime prepared from the sample of example 7 at day 0 is almost by a factor of 2 lower for the milk of lime obtained with the mill classified sample from example 8.

25 Overall, the viscosity stabilities, both at rest and under 1hr/day agitation, are lower for the mill classified sample, but are following the same trend as for the air classified sample. Indeed, the value of the viscosity stability is good the day of the slurrying, but is increasing sharply during the first 5 days. In the end, the viscosity increase tends to stabilize on the following days. Mill classification of the

hydrated lime samples seems to give better results on the viscosity stability of the milk of lime over time. Indeed, with this technology, all the hydrate is milled down to a particle size below 20 μ m. There is no loss of additive in the final product compared to air classification (where a disproportionally high fraction of the additives ends up
5 in the reject fraction, possibly due to agglomeration by the additives.

Milk of lime obtained by slurring the hydrated lime samples from example 2 and 3 have been compared to milk of lime obtained from hydrated lime samples from examples 2 and 3, but on which an additional step was performed. On the hydrated lime samples from examples 2 and 3, an addition of further organic
10 stabilizing agent (sorbitol) was applied by spraying a solution containing 50 wt% non-volatile matter of sorbitol with respect to the weight of the solution in order to add to the hydrated lime samples of examples 2 and 3 and additional content of sorbitol of 0.3 wt%. Sample from example 2 contained already 1.5 wt% and totalized after the spraying of sorbitol an organic stabilizing agent (sorbitol) content of 1.8 wt% non-
15 volatile matter with respect to the weight of the sample from example 2. Sample from example 3 contained already 1.8 wt% sorbitol and totalized after the spraying of sorbitol an organic stabilizing agent (sorbitol) content of 2.1 wt% non-volatile matter with respect to the weight of the sample from example 3.

Figure 4 and 5 shows respectively the viscosity over time at rest and
20 under a gentle agitation during one hour each day. The initial viscosities measured are very low and in the range 30 to 75 cps. This proves that a sufficient amount of the first (sorbitol) and second dispersant (Neomere® Tech 646) survived the hydration reaction and is still active enough to affect the viscosities.

As it can be seen of figure 4 showing the viscosity evolution over time
25 at rest, the viscosity at rest started to increase only after 10 days while still staying below the limit of 1200 mPa.s. Further, as it can be seen from figure 5 showing the viscosity evolution over time with 1 hour a day gentle agitation, the viscosity increased drastically after the first day, reaching a peak the second day. Then the viscosity started to decrease slowly day after day, arriving even below 500 mPa.s
30 after 3 to 5 days, depending on the dose. However, viscosities measured both on resting (figure 4) as well as 1hr/day agitated samples (figure 5) were improved and below the limit by adding an extra dose of stabilizer by spraying.

Milk of lime obtained by slurring the hydrated lime samples from example 9 and 10 have been compared to milk of lime obtained from hydrated lime samples from examples 9 and 10, but on which an additional step was performed. On the hydrated lime samples from examples 9 and 10, an addition of further organic
5 stabilizing agent (the same carbohydrate mixture as in the examples 7 to 10) was applied by spraying a solution containing 50 wt% non-volatile matter of the carbohydrate mixture of example 7 to 10 with respect to the weight of the solution in order to add to the hydrated lime samples of examples 9 and 10 an additional
10 content of carbohydrate mixture of 0.3 wt%. Sample from example 9 contained already 1.5 wt% and totalized after the spraying of the carbohydrate mixture used in examples 7-10 an organic stabilizing agent (carbohydrate mixture) content of 1.8 wt% non-volatile matter with respect to the weight of the sample from example 9. Sample from example 10 contained already 1.8 wt% non-volatile matter of
15 carbohydrates and totalized after the spraying of the carbohydrate mixture of examples 7-10 an organic stabilizing agent (carbohydrate) content of 2.1 wt% non-volatile matter with respect to the weight of the sample from example 10.

Figure 6 and 7 shows respectively the viscosity over time at rest and under a gentle agitation during one hour each day. The initial viscosities measured are very low and in the range 30 to 75 cps. This proves that a sufficient amount of the
20 first (carbohydrate mixture of examples 7-10) and second dispersant (Neomere® Tech 646) survived the hydration reaction and is still active enough to affect the viscosities.

As it can be seen from figure 6 showing the viscosity evolution over time at rest, the the milk of lime exhibited very good viscosity stabilities over time at rest.
25 All the samples are far below 500 mPa.s and adding an extra dose of organic stabilizing agent improved the behavior of the milk of lime even further at rest.

As it can be seen from figure 7 showing the viscosity evolution over time and under gentle 1hr/day agitation, the the milk of lime exhibited very good viscosity stabilities over time under gentle 1hr/day agitation. All the samples are far
30 below 500 mPa.s and adding an extra dose of organic stabilizing agent improved the behavior of the milk of lime even further under gentle 1hr/day agitation.

« CLAIMS »

1. Ready-to-use powder formulation, for making a high solids highly reactive milk of lime having an improved stability and settling, comprising solid particles of high calcium dry hydrated lime, , an organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation and a dispersant polymer at an amount of 0.03 to 0.3 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation containing at least 90wt% of high calcium lime based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation having a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm .

2. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 1, having a silica content expressed in SiO₂ equivalent lower than 1.5 wt%, preferably lower than or equal to 1.1 wt%, particularly lower than or equal to 0.85 wt%, more preferably lower than or equal to 0.65 wt%, even more particularly lower than or equal to 0.54 wt% based on the total weight of the ready-to-use powder formulation.

3. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 1 or claim 2, having an aluminum content expressed in Al₂O₃ equivalent lower than 0.4 wt%, preferably lower than or equal to 0.35 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

4. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 1 to 3, , having an sulfur content expressed in SO₃ equivalent lower than 0.5 wt%, preferably lower than or equal to 0.4 wt%, more preferably lower than or equal to 0.30 wt%, even more particularly lower than or equal to 0.25 wt% based on the total weight of the ready-to-use powder.

5. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 1 to 4, having a specific surface area measured by manometric

nitrogen adsorption and calculated using the Brunauer, Emmett and Teller model (BET method) after degassing 2 h at 190°C comprised between 5 and 45 m²/g, more preferably between 7 and 30 m²/g, more particularly between 8 and 25 m²/g.

5 6. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 1 to 5, having a flowability comprised in the range of 1.2 to 2.0.

7. Ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 1 to 6, having a moisture content comprised between 0.2 and 3
10 wt%, preferably between 0.3 and 2 wt% based on the total weight of the powder formulation as measured by infra-red thermobalance at 150°C or thermogravimetric analysis at the same temperature.

8. Ready-to-use powder formulation for making a milk of lime having an improved stability according to any of the claims 1 to 7, wherein said
15 organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups is chosen amongst a uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a monosaccharide, a disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

9. Ready-to-use powder formulation for making a milk of lime
20 having an improved stability according to any of the claims 1 to 8, wherein the weight ratio in non-volatile content between the dispersant polymer and the organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) is comprised between 0.04 and 0.4 and preferably between 0.06 and 0.35.

10. Process for manufacturing a ready-to-use powder formulation
25 for making a high solids highly reactive milk of lime having an improved stability and settling comprising the steps of:

- A hydration step of quicklime in a hydrator during which solid particles mainly comprised of quicklime are contacted with water and hydrated to form high calcium dry hydrated lime solid particles, said hydrator having different zones, said
30 solid particles being transported from a feeding zone to an exit zone of hydrated lime solid particles, said hydration step comprising a series of aqueous phase additions at a series of locations between the feeding zone and the exit zone,

- A first addition of an organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% relative to the total weight
35 of the ready-to-use powder formulation, and

- A second addition of a dispersant polymer at an amount of 0.03 to 0.3 wt% active polymer, i.e. based on non-volatile content relative to the total weight of the ready-to-use powder formulation,

- One amongst said first addition and said second addition being performed in the hydrator to said solid particles upstream of the collecting zone, optionally through at least one addition of said series of aqueous addition, and the other amongst said first addition and said second addition being performed before, during or after said hydration step,

- A size control step of said high calcium dry hydrated lime solid particles to provide a predetermined amount of high calcium dry hydrated lime solid particles having a particle size distribution with a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm and

- A collecting step of a ready -to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling comprising solid particles of high calcium dry hydrated lime, said organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl group(s) at an amount from 0.3 to 5 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation and said dispersant polymer at an amount of 0.03 to 0.3 wt% non-volatile matter based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation containing at least 90wt% of high calcium lime based on the total weight of the ready-to-use powder formulation, said ready-to-use powder formulation having a $d_{97} \leq 40 \mu\text{m}$ and a d_{50} comprised between 2 and 15 μm .

11. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 10, wherein said size control step is chosen amongst a dry or wet milling step, a mil classification step, an air classification step or a combination of two or more thereof before collecting said ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling.

12. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 10 or claim 11, wherein said one amongst said first addition and said second addition is performed at one or more locations in the

hydrator to said dry hydrated lime solid particles, optionally through at least one addition of said series of aqueous additions.

13. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 12, wherein said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to partially hydrated lime solid particles, optionally through at least one addition of said series of aqueous phase additions.

14. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 13, wherein said one amongst said first addition and said second addition is performed at one or more locations in the hydrator to said solid particles mainly comprised of quicklime, optionally through at least one addition of said series of aqueous phase additions.

15. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 14, wherein one amongst said first addition and said second addition is a solid-state addition.

16. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 15, wherein one amongst said first addition and said second addition is a liquid state addition.

17. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 16, wherein the other amongst said first addition and said second addition is a solid-state addition.

18. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 17, wherein the other amongst said first addition and said second addition is a liquid state addition.

19. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 18, wherein the other amongst said first addition and said second addition is performed to at least one aqueous phase addition during said one amongst said first addition and said second addition.

20. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 19, wherein the other amongst said first addition and said second addition is performed to at least one aqueous phase addition before the hydration step.

21. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 20, wherein the other amongst said first addition and said second addition is performed to said solid particles mainly comprised of quicklime before the hydration step.

22. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 21, wherein the other amongst said first addition and said second addition is performed to partially hydrated lime solid particles during the hydration step.

23. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 22, wherein the hydration step is carried out in presence of calcium nitrate or calcium chloride at an amount of 0.3 to 2 wt%, preferably between 0.5 and 1.5 wt% with respect to the weight of solid particles mainly comprised of quicklime.

24. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 23, wherein the other amongst said first addition and said second addition is performed to said dry hydrated lime solid particles upstream of the collecting zone.

25. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to any of the claims 10 to 24, wherein said second addition is an addition of a water-soluble dispersant polymer preferably having an active polycarboxylate ether concentration of at least 10 wt%, more preferably of at least 15 wt% and even more preferably of at least 20 wt%.

26. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 10 to 25, wherein said first addition is an addition of at

least one organic stabilizing agent in C₄ to C₁₈ bearing one or more hydroxyl groups is chosen amongst an uronic or aldonic acid of a carbohydrate, e.g., sodium gluconate, or a polyol, or a carbohydrate chosen amongst a monosaccharide, a disaccharide, an oligosaccharide or a polysaccharide or combination thereof.

5 27. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 10 to 26, further comprising a step of spraying an additional amount of organic stabilising agent in C₄ to C₁₈ bearing one or more hydroxyl groups and/or of a polymer dispersant on the high calcium dry hydrated
10 lime solid particles.

 28. Process for manufacturing a ready-to-use powder formulation for making a high solids highly reactive milk of lime having an improved stability and settling according to claim 10 to 27, where the weight ratio in non-volatile content between the dispersant polymer and the organic stabilizing agent in C₄ to C₁₈
15 bearing one or more hydroxyl group(s) is comprised between 0.04 and 0.4 and preferably between 0.06 and 0.35.

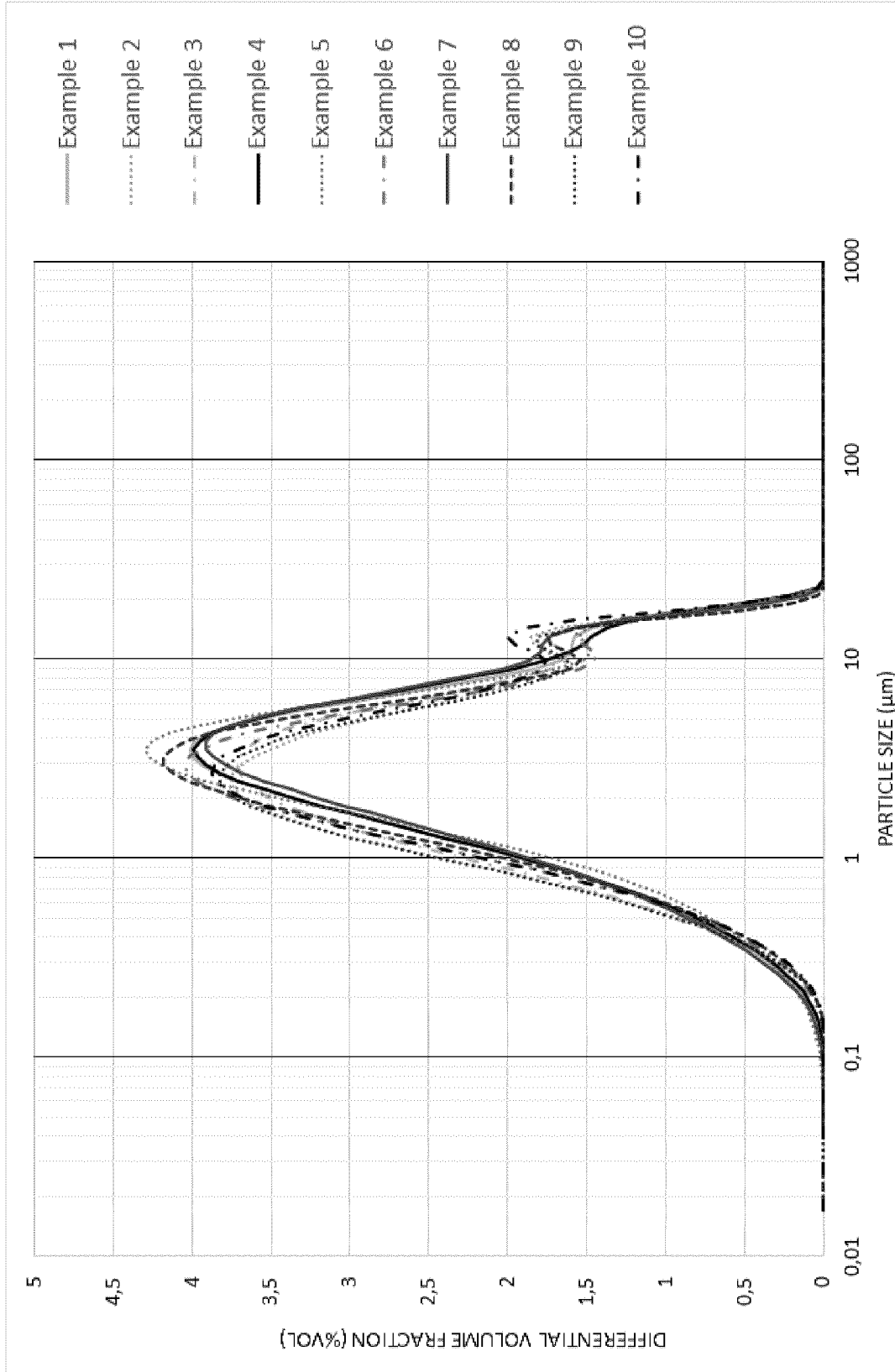


Figure 1

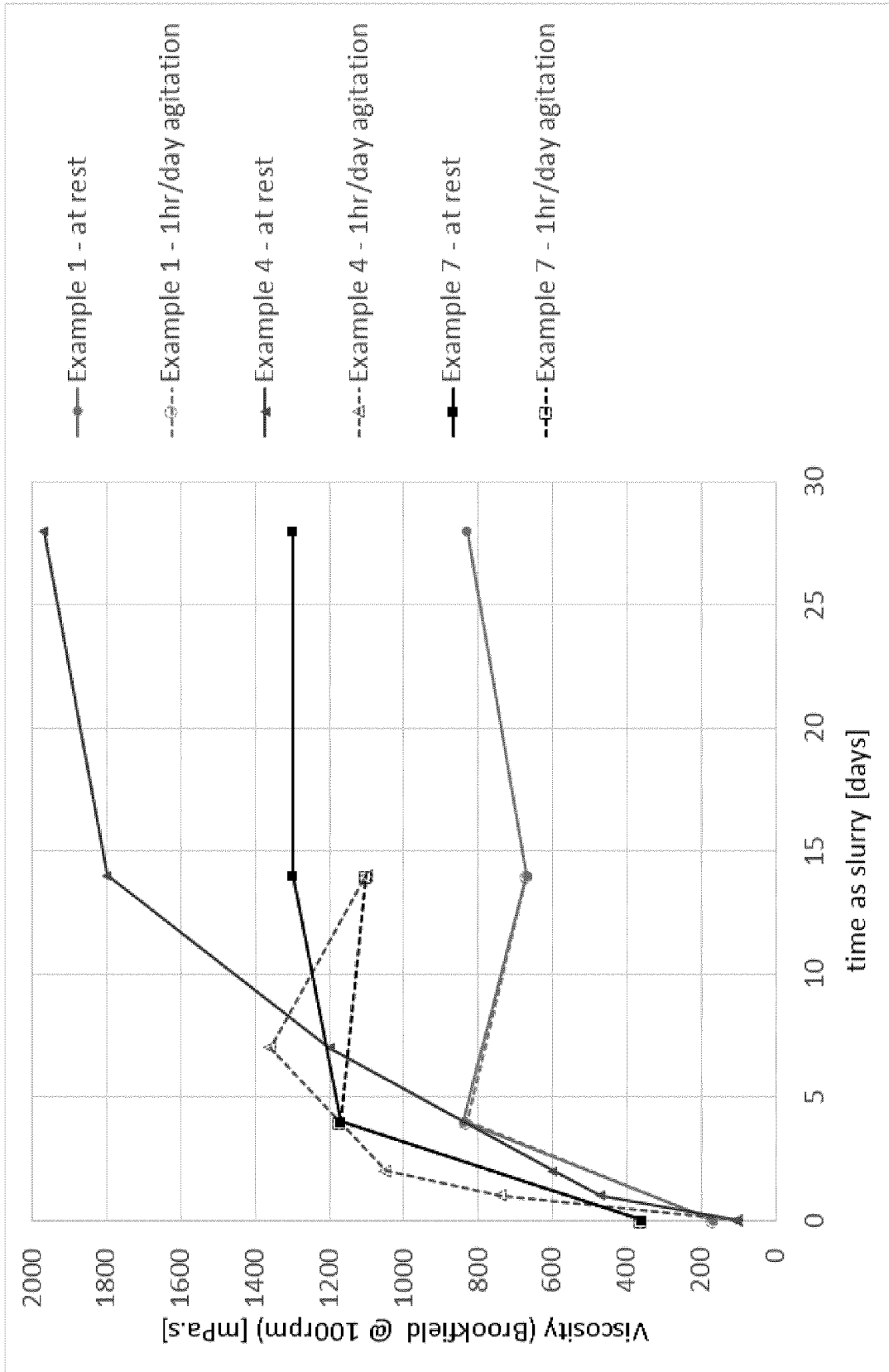


Figure 2

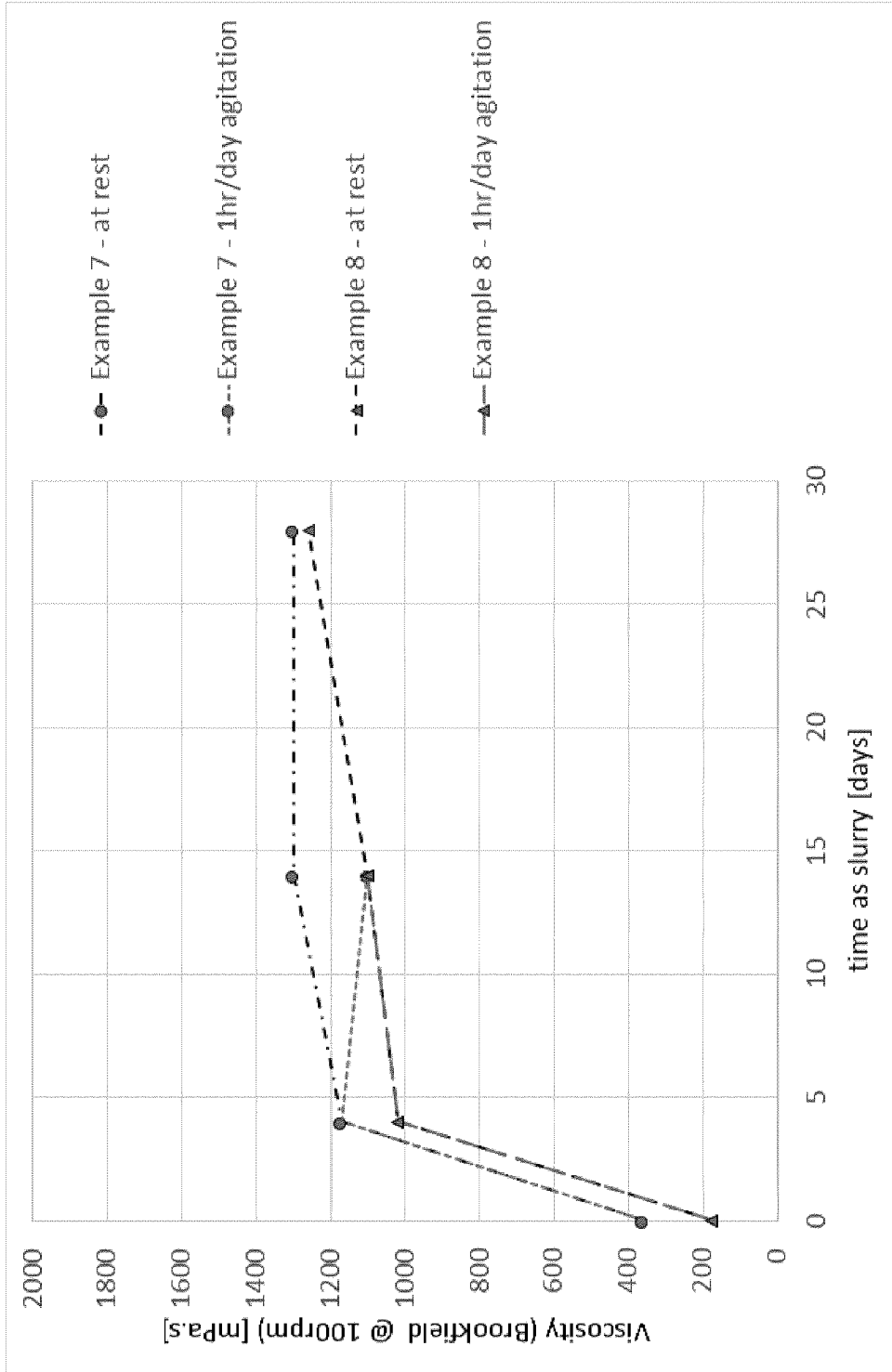


Figure 3

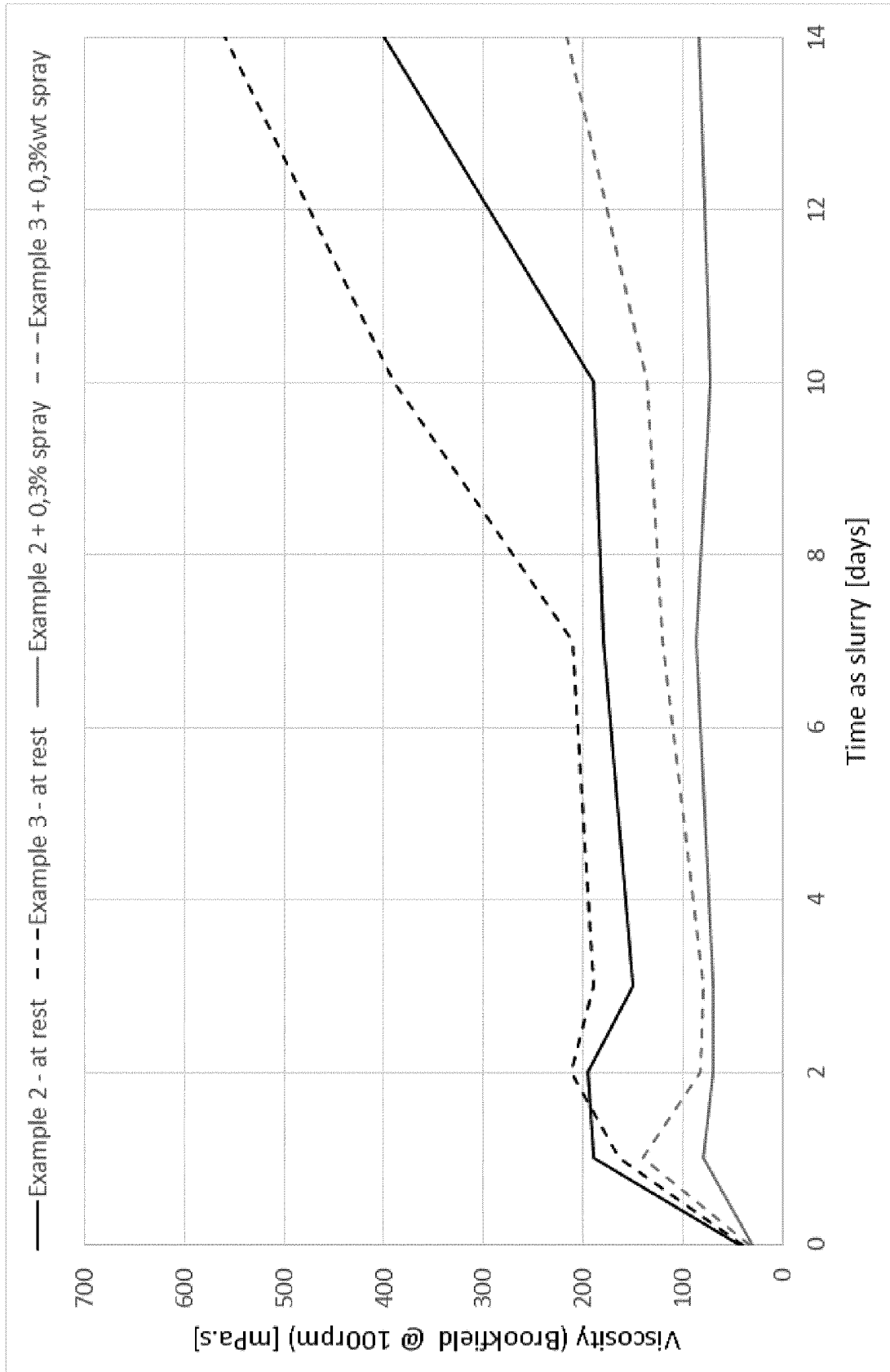


Figure 4

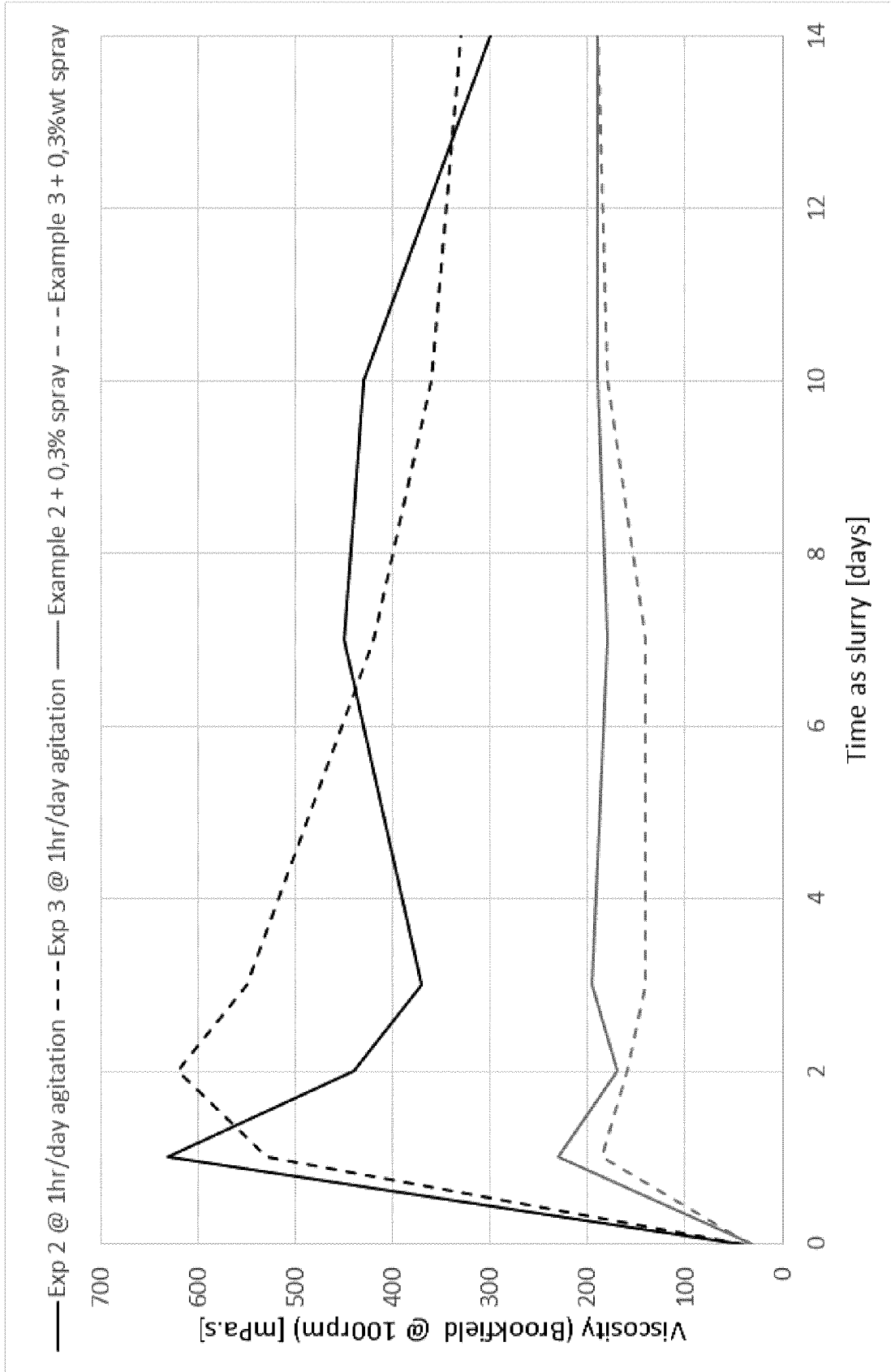


Figure 5

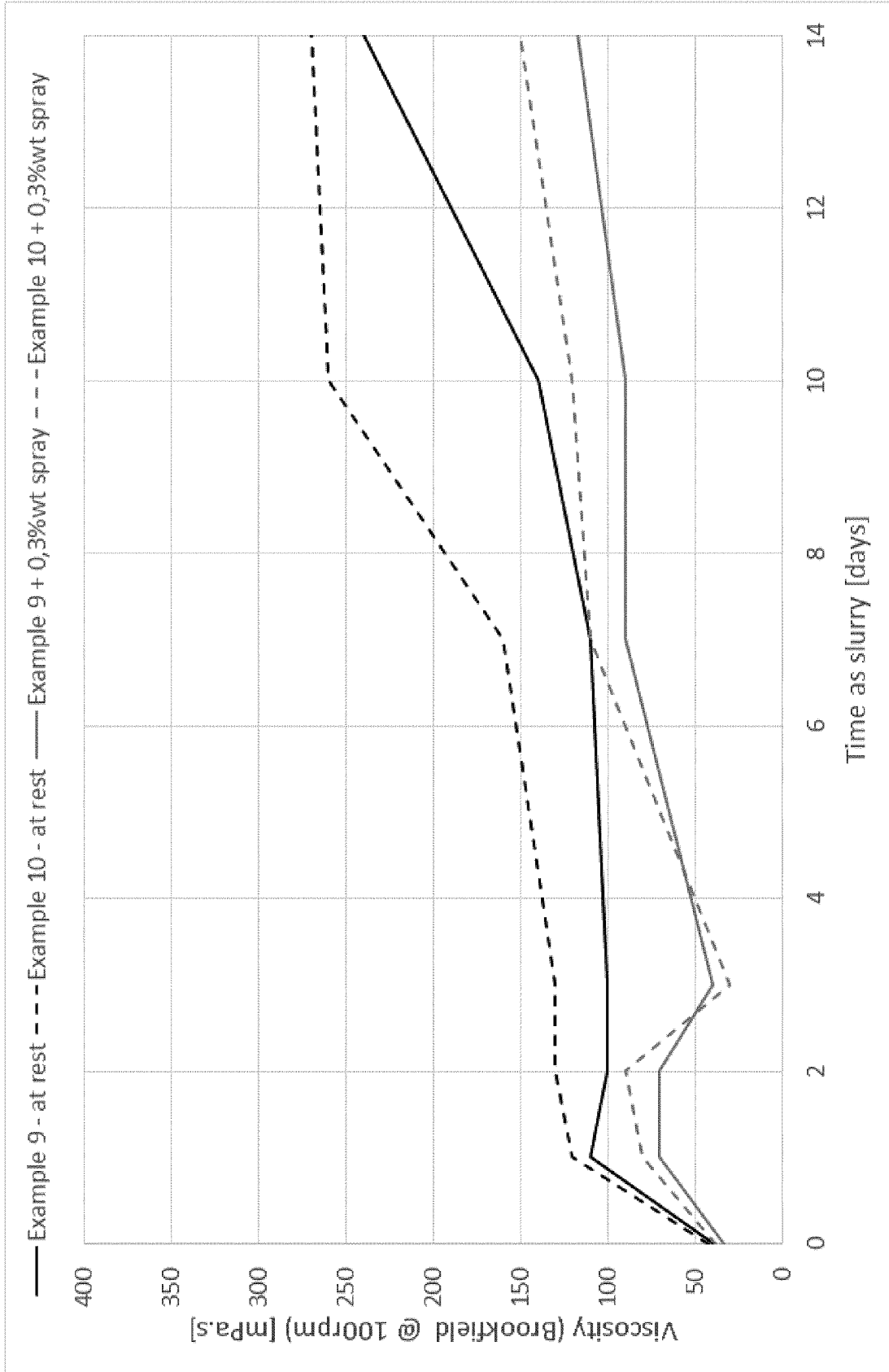


Figure 6

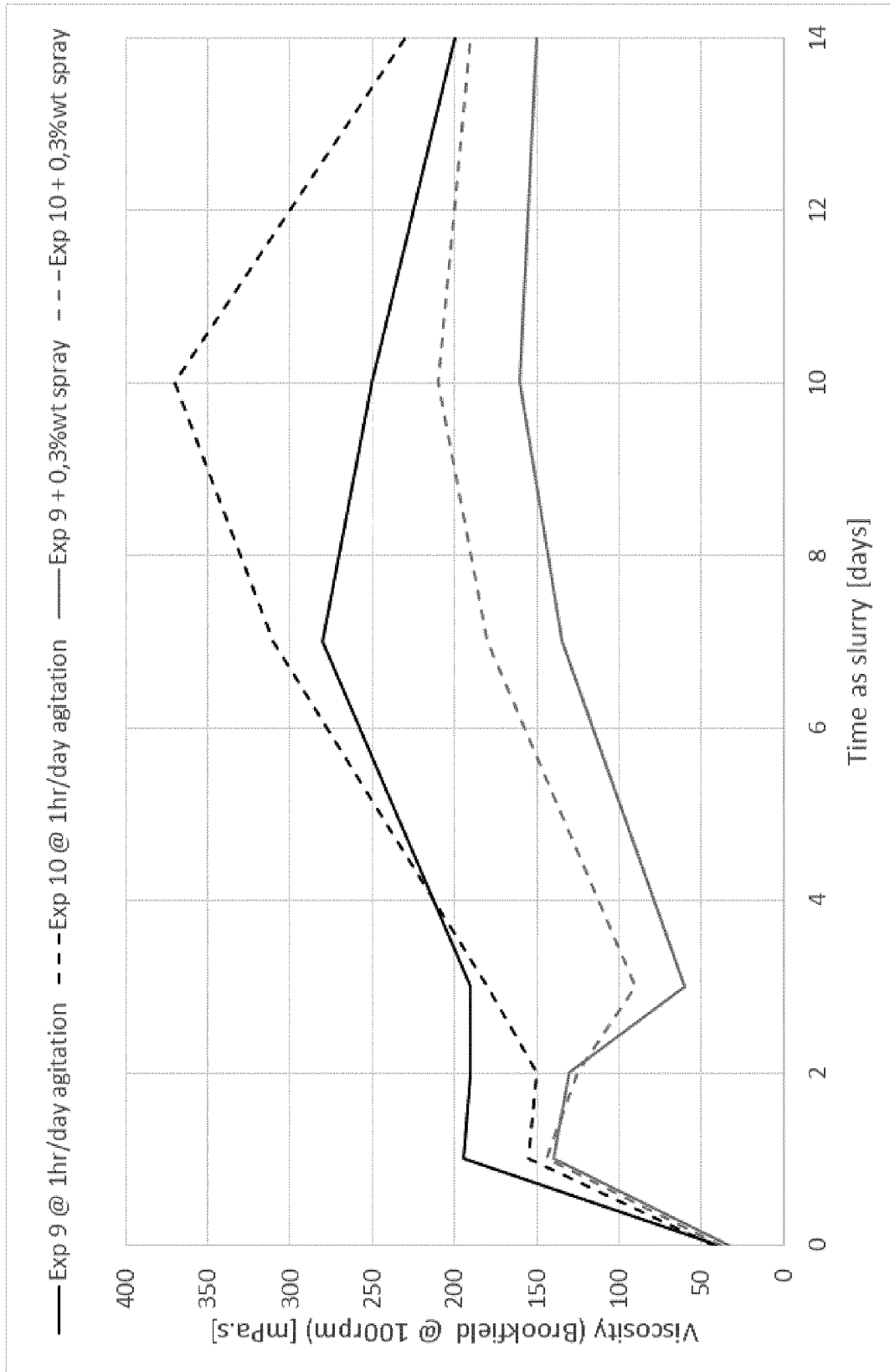


Figure 7

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2021/062030

A. CLASSIFICATION OF SUBJECT MATTER
INV. C04B2/04 C04B2/06 C04B40/00
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	BE 1 026 329 B1 (CARMEUSE RESEARCH AND TECH [BE]) 6 January 2020 (2020-01-06) page 13, lines 21-22, paragraphs 1,24,32,35,49,50; examples 1-7; tables 1-7 -----	1-28
A	FR 3 060 020 A1 (LHOIST RECH ET DEVELOPPEMENT SA [BE]) 15 June 2018 (2018-06-15) the whole document -----	1-28

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents :

<p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"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</p> <p>"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</p> <p>"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</p> <p>"&" document member of the same patent family</p>
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Date of the actual completion of the international search 13 July 2021	Date of mailing of the international search report 19/07/2021
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Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Bonneau, Sébastien
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No PCT/EP2021/062030

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