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PROCESS FOR LIGHTENING PLASTERBOARDS

The present invention relates to a process for the manufacture of lightened plasterboards.

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A plasterboard is a prefabricated parallelepipedal component made of plaster (calcium sulphate dihydrate) covered with cardboard or paper or inorganic fibres on each of its faces. The composite thus formed exhibits
10 good mechanical properties, the sheets on the faces acting both as framework and as facing.

The plaster core is obtained from a plaster slurry formed principally by mixing hydratable calcium sulphate and
15 water, if appropriate with the addition of conventional adjuvants. The term "hydratable calcium sulphate" should be understood as meaning, in the present account, anhydrous calcium sulphate (anhydrite II or III) or a calcium sulphate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) in its α or β
20 crystalline form. Such compounds are well known to a person skilled in the art and are generally obtained by the calcination of a gypsum.

The slurry rapidly sets by hydration of the plaster. The
25 boards are subsequently heated in dryers in order to remove the excess water.

The lightening of plasterboards, in addition to their mechanical strength, is what is really at stake here. In
30 order to lighten the plasterboard, it is commonplace to introduce air into the slurry by adding a foam to the plaster slurry.

The reduction of the density of the plasterboards is
35 desired for its two-fold economic advantage: first, it makes it possible to lighten the product and thus to facilitate its transportation and, secondly, it makes it possible to overcome the limitation on the production



throughput by the reduction in the demand for plaster. This is because the calcination of the gypsum takes time and it is possible for this stage to be a limiting component on the speed of the production line. The
5 decrease in the demand for plaster makes it possible in addition to reduce the production costs related to the calcination. Thus, the reduction of the density of the plasterboards makes it possible to decrease the transportation costs, to increase the line speed and, at
10 the same time, to decrease the production costs.

Various surfactant compositions for lightening plasterboards are known. Often, these compositions comprise alkyl ether sulphates.

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Thus, a surfactant composition based on alkyl sulphates and alkyl ether sulphates is disclosed in WO 9516515. In this composition, the ratio of alkyl sulphates to alkyl di- or triether sulphates is at least 12 : 1, preferably
20 between 30 : 1 and 60 : 1. Alkyl sulphates alone are undesirable. This is because a plasterboard obtained with an alkyl sulphate alone exhibits very mediocre lightening.

25 The document US 5 643 510 discloses the use of a mixture of alkyl sulphates and of alkyl ether sulphates in order to control the size of the air bubbles in plasterboards. The document indicates that alkyl sulphates, which form unstable foams, are not used alone as the production of a
30 board with a given density would require a consumption approximately 3 times greater than that of a surfactant comprising an alkyl ether sulphate. The preferred surfactant is thus a mixture comprising approximately 10% of alkyl ether sulphate.

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However, the use of alkyl sulphates instead of an alkyl ether sulphate as surfactant has certain advantages. This is because, whereas the alkyl sulphate can be obtained by

direct sulphation of the corresponding fatty alcohol, it is necessary to pass through an ethoxylation stage to obtain the alkyl ether sulphate. This stage is not only expensive but can additionally result in the formation of
5 undesirable by-products.

The problem which the invention intends to solve thus consists in providing a process for the manufacture of inexpensive lightened plasterboards using a surfactant
10 which is inexpensive and readily available.

As a general rule, the quality of a surfactant is evaluated with respect to the volume of foam which it makes it possible to obtain. This is because, for the
15 production of a given volume of foam, it appears logical that more will be consumed of a surfactant with a low foaming power. This has economic effects on the cost price of the plasterboard.

20 However, the stability of the foam before it is introduced into the plasterboard and then in the latter also constitutes a not insignificant factor. Thus, at an equal volume, a foam of low stability will result in a less lightened plasterboard than a stable foam. According
25 to US 5 643 510, alkyl sulphates form foams of low stability in plasterboard.

Furthermore, it is clear that the plasterboard must, even lightened, furthermore exhibit characteristics comparable
30 to unlightened boards, such as the level of mechanical strength. The quality of the bonding between the plaster slurry and the faces of the boards, in dry as in wet surroundings, also constitutes a quality requirement which has to be satisfied.

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The invention is based on the observation that, while certain alkyl sulphate compositions generally exhibit a slight excess consumption with respect to mixtures with

alkyl ether sulphates, they make it possible to obtain plasterboards exhibiting a considerable reduction in weight. The possible additional economic cost is largely compensated for by such a reduction in weight. This is
5 because the saving generated by the reduction in weight can represent up to 7 times the additional cost related to the additional consumption of surfactant. The balance therefore remains very positive overall.

10 The plasterboards thus lightened additionally exhibit entirely acceptable characteristics of mechanical strength and of bonding with the external faces.

This surprising effect appears to be related to an
15 interaction between the surfactant, the foam and the plaster slurry.

This is because, while the foaming power of these alkyl sulphates is often lower compared with the usual
20 surfactants comprising alkyl ether sulphates, it turns out that some of them make it possible to obtain foams compatible with the plaster slurry. Furthermore, contrary to the teaching of the prior art, it has been found that the excess consumption is very moderate and may even, in
25 some cases, be nonexistent.

It appears that this effect is at least partially due to a very good compatibility between the plaster slurry and the foam, a function of their respective viscosities.
30 More particularly, it appears that the fluidity of the two phases can be adjusted so that the mixing takes place without excessive loss of foam volume. Thus, the effectiveness of the foam generated by the surfactant is improved.

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It thus appears that the consumption of surfactant and its ability to lighten are not solely a function of the foaming power of the latter but are also dependent on the

complex interplay of the other parameters of the process. There is reason to believe that, in particular, the physicochemical characteristics of the plaster slurry, and in particular its fluidity, have an influence on the
5 lightening effect obtained.

A subject-matter of the invention is thus a process for the manufacture of plasterboards exhibiting a core density of less than 0.77 comprising the stages of:

- 10 - formation of a foam from water and a surfactant composed of at least one alkyl sulphate of formula $H(CH_2)_nOSO_3^-M^+$, in which n is a number between 6 and 16, M is a monovalent cation and the mean number of carbon atoms n_m is from 9 to 10, and
- 15 - introduction of the foam obtained into a plaster slurry,

characterized in that the amount of surfactant introduced does not exceed 0.32 g/l of plaster slurry.

20 The term "density" is understood to mean the ratio of the mass of a given volume of plaster slurry to the mass of this volume of water. The term "core density" is understood to mean the density of the plasterboard with the exclusion of the material covering the faces.

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Preferably, the consumption of surfactant is less than 0.24 g/l and in particular less than 0.16 g/l of plaster slurry. Generally, the consumption is greater than 0.05 g/l of plaster slurry for boards with a thickness of
30 12.5 mm and 0.02 g/l for boards with a thickness of 6 mm.

The plasterboard produced preferably exhibits a compressive strength of greater than 2 MPa and in particular of greater than 3 MPa.

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According to a preferred embodiment, the surfactant comprises 5 to 60% by weight of dodecyl sulphate.

According to a preferred embodiment, the surfactant comprises 40 to 95% by weight of decyl sulphate. According to another preferred embodiment, the surfactant comprises 5 to 60% by weight of octyl sulphate. The particularly preferred surfactant comprises 5 to 15% by weight of sodium octyl sulphate and 85 to 95% by weight of sodium decyl sulphate.

The particularly preferred surfactant exhibits a mean number of carbons of between 9.5 and 10, preferably from 9.7 to 9.9.

Preferably, M is chosen from sodium, potassium, magnesium and ammonium.

According to one embodiment, the surfactant additionally comprises a sequestering agent and/or a hydrotropic agent.

The invention also relates to the use of a surfactant for lightening plasterboards composed of at least one alkyl sulphate of formula $H(CH_2)_nOSO_3^-M^+$, in which n is a number between 6 and 16, M is a monovalent cation and the mean number of carbon atoms n_m is from 9 to 10, the amount used being less than 0.32 g/l of plaster slurry.

Preferably, the said surfactant is used to produce plasterboards exhibiting a core density of less than 0.77, in particular of less than 0.74 and very particularly of less than 0.72. However, the density of the core is generally greater than 0.48.

Other characteristics and advantages of the invention will now be described in detail in the account which follows.

The plaster composition comprises plaster, water and a surfactant composition. It can furthermore comprise other additives as conventionally used.

5 Furthermore, the foam formed by the targeted surfactant composition makes it possible to obtain comparable reductions in weight for plasterboards prepared with different types of gypsum. Thus, the surfactant composition is advantageous in that it is relatively
10 insensitive to the quality of gypsum employed.

Furthermore, the characteristics of the foam formed by the surfactant composition are relatively insensitive to temperature. Thus, when the temperature of the water
15 varies, the foam volume generated remains substantially constant.

In addition, it turned out that the quality of bonding between the plaster and the external sheet of the
20 plasterboards is outstanding.

The mechanical characteristics of plasterboards are usually evaluated by the flexural strength of the core, the hardness of the core, the ball hardness and the
25 compressive strength of the plasterboard. The resistance which the plasterboard makes to a ¼-inch nail head being driven in, which is conventionally called the "nail pull test" or punching strength, is also of great practical importance. The conditions of this test are described in
30 ASTM Standard C473-Method B.

Furthermore, it turns out that the plasterboards obtained show good bonding between the plaster and the external sheet.

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An appropriate surfactant composition for the implementation of the process is, for example, a mixture of C₈ and C₁₀ alkyl sulphates, such as Emal A10 DE, sold

by Kao Corporation SA. However, it is also possible to carry out mixing starting from other alkyl sulphates or from mixtures of alkyl sulphates to produce a composition having a mean carbon number of 9 to 10.

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The alkyl sulphates corresponding to the formula $H(CH_2)_n-OSO_3M$ are generally obtained by sulphation of the corresponding alcohols. In principle, n is generally an even number because of the better availability of these alcohols. However, alkyl sulphates with n uneven can also
10 be used in the context of the invention.

The chain of the alkyl sulphates participating in the composition preferably comprises from 8 to 12 carbon
15 atoms.

The surfactant composition can additionally include a hydrotropic agent. Such hydrotropic agents are, for example, methanol, ethanol, isopropanol, ethylene glycol, propylene glycol, polyethylene glycol and polypropylene
20 glycol, as well as ethylene glycol monoalkyl ethers, alkyl polyglycosides and their mixtures.

The composition can advantageously comprise a sequestering or chelating agent which makes it possible
25 to keep in solution magnesium or calcium ions, in particular in hard water. Such sequestering or chelating agents are, for example, hydroxycarboxylic acids and their salts, aldoses and ketoses, inorganic complexing agents, more particularly phosphates, borates and
30 polyphosphates, organic complexing agents chosen more particularly from the group consisting of EDTA, NTA, and the like, and phosphoric acid derivatives with a polymer structure which comprise hydroxyl and/or amine and/or
35 carboxylate groups.

The foam obtained makes it possible to introduce, into the plasterboard, from 0.01 to 0.04% by weight of

surfactant (expressed on a dry basis) with respect to the weight of the board. It represents a volume of between 20 and 40% of that of the plasterboard.

- 5 The foam-based plasterboard can moreover advantageously comprise adjuvants which are conventionally used, such as plasticizers, accelerators, starch, and the like.

The invention will be better understood from the
10 viewpoint of the examples which follow, which are given by way of illustration and without implied limitation.

EXAMPLES

Example 1

- 15 Miniboards with an area of 0.1 m² and with a thickness of 12.5 mm are prepared from St Loubès plaster, which is a plaster obtained by flash calcination of a natural gypsum having the following characteristics:

- gypsum content : 68.8%
- 20 - anhydrite : 0.90%
- magnesia : 3.70%
- dolomite : 8.80%
- talc : 0.80%
- phlogopite : 1.10%
- 25 - microline : 3.80%
- quartz : 9.50%
- celestine : 0.60%
- clinochlore : 2.00%.

- 30 These boards are prepared in the following way:

A foam is prepared by stirring, for 1 minute in a foam generator of the Hamilton Beach type set to a voltage of 55 volts, a mixture of 5.25 ml of a 50 g/l solution of a composition composed of sodium alkyl sulphates comprising
35 47% by weight of C₈ alkyl and 53% by weight of C₁₀ alkyl, the mean carbon number n_m , taking into account the molar masses of the constituents, being 9, with 170 ml of water at 22°C. The foam is subsequently introduced into a

mixture of 700 g of water at a temperature of 50°C and 1130 g of plaster at a temperature of 22°C. The plaster slurry is deposited between two sheets of cardboard. The excess after filling is removed. The miniboard is
5 subsequently dried in an oven at a steadily increasing temperature of from 100°C to 200°C over 15 min, and then a steadily decreasing temperature from 200°C to 90°C over 25 min.

10 Example 2

Plasterboards are manufactured according to Example 1 but replacing the surfactant composition with the same amount of a surfactant comprising 25% by weight of C₈ alkyl sulphate and 75% by weight of C₁₀ alkyl sulphate. The mean
15 number of carbon atoms n_m in the composition is 9.5.

Example 3

Plasterboards are manufactured according to Example 1 but replacing the surfactant composition with the same amount
20 of a surfactant comprising 11% by weight of C₈ alkyl sulphate and 89% by weight of C₁₀ alkyl sulphate. The mean number of carbon atoms n_m in the composition is 9.8.

Example 4

25 Plasterboards are manufactured according to Example 1 but replacing the surfactant composition with the same amount of a surfactant comprising 33.3% by weight of C₈ alkyl sulphate, 58.4% by weight of C₁₀ alkyl sulphate and 8.3% by weight of C₁₂ alkyl sulphate. The mean number of
30 carbon atoms n_m in the composition is 9.5.

Example 5

Plasterboards are manufactured according to Example 1 but replacing the surfactant composition by the same amount
35 of a surfactant comprising 75% by weight of C₈ alkyl sulphate and 25% by weight of C₁₀ alkyl sulphate. The mean number of carbon atoms n_m in the composition is 8.5.

Example 6

Plasterboards are manufactured according to Example 1 but replacing the surfactant composition with the same amount of a surfactant comprising an alkyl sulphate (AS) and an alkyl ether sulphate (AES). This surfactant is sold by Stepan under the name Alphafoamer.

The boards prepared according to the examples, once dried to constant weight in agreement with French Standard NF P 72-302, are weighed and their core densities are determined. The results are recorded in Table 1 below.

Table 1

Example	n _m	Foam volume [ml]	Density of the board
1	9.0	688	0.728
2	9.5	688	0.712
3	9.8	695	0.692
4	9.5	700	0.698
5*	8.5	680	0.771
6*	AS + AES	750	0.700

* Comparative examples

The compositions according to the invention make it possible, without excess consumption in comparison with mixtures of alkyl sulphates and of alkyl ether sulphates, to obtain comparable reductions in weight. Example 3 illustrates a preferably used surfactant composition.

To evaluate the mechanical properties of the boards, the compressive strength is measured from a 5 × 5 cm² sample withdrawn from the board. The ball hardness and the load at failure of the core (three-point bending test) are measured according to NF Standard P 72-302.

The hardness of the core and the punching strength (nail pull test) are measured according to ASTM Standard C473 Method B.

5 The results obtained demonstrate that, with a surfactant composition according to the prior art (Example 6), when the core densities are equivalent, the mechanical performance obtained is inferior to that which is achieved by virtue of the process according to the
10 invention.

The performance obtained with the composition according to Example 5 corresponds to boards having a higher core density.

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Thus, at a satisfactory mechanical performance, the preferred implementation corresponds to that which results in the maximum reduction in weight. The results of these measurements are recorded in Table 2 below.

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Table 2

Example	Amount surfactant used (g/l)	Compressive stress [MPa]	Ball hardness [mm]	Core hardness [DaN]	Nail pull [DaN]
1	0.139	3.72	18.70	9,60	37
2	0.139	3.63	19.90	11.40	38
3	0.136	3.46	18.65	9.55	34.8
4	0.138	3.52	18.78	10.25	35.9
5*	0.152	4.52	17.30	14.00	42
6*	0.134	2.59	19.8	7.6	32.6

* Comparative examples

25 Furthermore, the plasterboards prepared were characterized by the measurement of the bonding between the cardboard and the core. The measurement consists in tearing off the cardboard and in evaluating the percentage of detachment of the cardboard from the core.
30 The dry bonding test is carried out on a dry board. The

wet bonding test after 2 hours is carried out after rehumidification for 2 hours at 30°C in a controlled atmosphere at 90% humidity. The results are recorded in Table 3 below.

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Table 3

Example	Dry bonding (% detachment)	Wet bonding after 2 hours (% detachment)
1	0	6
2	0	4
3	0	4
4	0	5
5*	0	7
6*	3	30

* Comparative examples

- 10 The results demonstrate the superiority of the compositions according to the invention with regard to the bonding, whether dry bonding or wet bonding after 2 hours, in particular with respect to surfactants comprising alkyl ether sulphates.

CLAIMS

1. Process for the manufacture of plasterboards exhibiting a core density of less than 0.77 comprising the stages of:
 - formation of a foam from water and a surfactant composed of at least one alkyl sulphate of formula $\text{H}(\text{CH}_2)_n\text{OSO}_3^-\text{M}^+$, in which n is a number between 6 and 16, M is a monovalent cation and the mean number of carbon atoms n_m is from 9 to 10, and
 - introduction of the foam obtained into a plaster slurry, characterized in that the amount of surfactant introduced does not exceed 0.32 g/litre of plaster slurry.
2. Process according to Claim 1, in which the consumption of surfactant is less than 0.24 g/l of plaster slurry.
3. Process according to either of the preceding claims, in which the plasterboard produced exhibits a compressive strength of greater than 2 MPa.
4. Process according to one of the preceding claims, in which the surfactant comprises 5 to 60% by weight of dodecyl sulphate.
5. Process according to one of the preceding claims, in which the surfactant comprises 40 to 95% by weight of decyl sulphate.
6. Process according to one of the preceding claims, in which the surfactant comprises 5 to 60% by weight of octyl sulphate.

7. Process according to one of the preceding claims, in which M is chosen from sodium, potassium, magnesium and ammonium.
- 5 8. Process according to one of the preceding claims, in which the surfactant comprises 5 to 25% by weight of sodium octyl sulphate and 75 to 95% by weight of sodium decyl sulphate.
- 10 9. Process according to one of the preceding claims, in which the surfactant comprises 5 to 15% by weight of sodium octyl sulphate and 85 to 95% by weight of sodium decyl sulphate.
- 15 10. Process according to one of the preceding claims, in which the surfactant additionally comprises a sequestering agent.
- 20 11. Process according to one of the preceding claims, in which the surfactant additionally comprises a hydrotropic agent.
- 25 12. Use of a surfactant for lightening plasterboards composed of at least one alkyl sulphate of formula $\text{H}(\text{CH}_2)_n\text{OSO}_3^-\text{M}^+$, in which n is a number between 6 and 16, M is a monovalent cation and the mean number of carbon atoms n_m is from 9 to 10, the amount used being less than 0.32 g/l of plaster slurry.
- 30 13. Use according to the preceding claim, in which the plasterboard exhibits a core density of less than 0.77.