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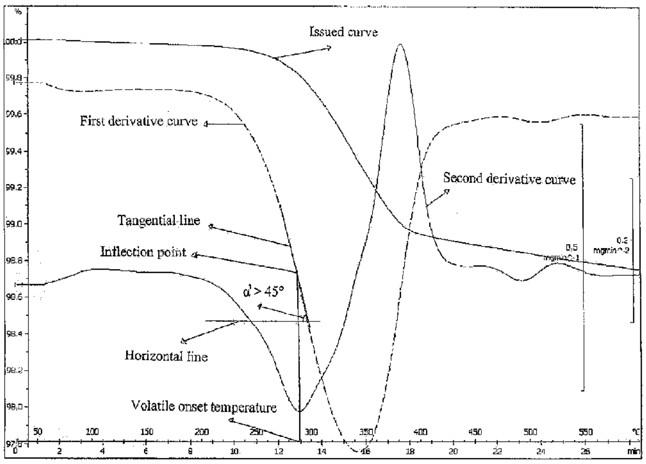
(54) Title: TREATED MINERAL FILLER PRODUCTS, PROCESS FOR THE PREPARATION THEREOF AND USES OF SAME

Figure 2



(57) Abstract: The present invention relates to treated mineral filler products comprising a) at least one mineral filler, b) a treatment layer located on the surface of said mineral filler(s), said treatment layer comprising at least one saturated C_8 to C_{24} aliphatic carboxylic acid; and at least one di and/or trivalent cation salt of one or more saturated C_8 to C_{24} aliphatic carboxylic acid, wherein the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 51 :49 to 75:25; and said treatment layer is present in an amount of at least 2.5 mg/m^2 of said mineral filler. Furthermore the present invention relates to processes to prepare such treated mineral filler products, and to their uses, notably in plastic applications, and especially in polypropylene (PP)- or polyethylene (PE)-based breathable or extrusion coating film applications.

Treated mineral filler products, process for the preparation thereof and uses of same

The present invention relates to the domain of treated mineral filler products, to processes to prepare such treated mineral filler products, and to their uses, notably in plastic applications, and especially in polypropylene (PP)- or polyethylene (PE)-based breathable or extrusion coating film applications.

Mineral fillers are often used as particulate fillers in polymer products. The presence of volatiles associated with mineral fillers that evolve at temperatures reached during the application of such mineral fillers and/or in the processing of such mineral filler-comprising products may lead to the degradation of the quality of the final mineral-comprising polymer product. This is particularly a problem encountered in the preparation of mineral filler-comprising and more particularly calcium carbonate-comprising, PP- or PE-based breathable or extrusion coating films.

Moreover, volatiles may lead to a reduction in the tensile and tear strength of such a film, and may degrade its visible aspects, in particular of its visible uniformity.

Volatiles can generate excessive foaming of the mineral filled polymer melt during a step of compounding, causing unwanted product build-up at the vacuum extraction and hence, forcing a reduced output rate.

Such volatiles may, for example, be:

- inherently associated with the mineral filler ("inherent volatiles"), and is especially associated water, and/or

- introduced during the treatment of the mineral filler ("added volatiles"), for example to render the mineral filler more dispersible within a plastic medium, and/or

- generated by the reaction of inherent organic materials and/or added organic materials, with the mineral filler; such reactions may especially be induced or enhanced by the temperatures reached during the introduction and/or processing of the polymeric material comprising the treated mineral filler, such as during the extrusion or compounding process; and/or

- generated by the degradation of inherent organic materials and/or added organic materials, forming CO_2, water and possibly low molecular mass fractions of these organic materials; such a degradation may especially be induced or enhanced by the temperatures reached during the introduction and/or processing of the polymeric material comprising the treated mineral filler, such as during the extrusion or compounding process.

As such, the skilled man is in need of a mineral filler:

a) that is suitable for application, notably in the plastic industry;
b) featuring as high a “volatile onset temperature” as possible, as defined hereafter;
c) leading to a limited quantity of volatiles evolved over the range of temperatures typically reached during the implementation of said mineral filler (hereafter “total volatiles”).

One obvious means to increase the volatile onset temperature and to limit the quantity of volatiles associated with a mineral filler is to avoid or limit the use of certain common filler treatment additives. However, often, as in the case when a mineral filler is applied in a plastic application, such additives are needed to ensure other functions.

In the case of breathable film applications, additives are introduced to provide the mineral filler with a hydrophobic coating and to improve the dispersability of the mineral filler in the film precursor material as well as possibly to improve the processability of this film precursor material and/or properties of the final application products. An elimination of such additives would unacceptably compromise the resulting film quality.

Thus, an additive for treating mineral fillers should provide the resulting mineral filler product with:

- a workable viscosity, notably of less than 10 000 mPa·s, at 180°C,
- an increased "volatile onset temperature",
- simultaneously limited "total volatiles",
- without compromising the mineral hydrophobicity, and hence film quality.

When the prior art makes reference to treated mineral fillers, and especially calcium carbonate, treatment comprising aliphatic carboxylic acids, and aliphatic carboxylic acid salts, is mentioned. However, it fails to provide any specific teaching with respect to providing the mineral fillers with the above mentioned features.

In this respect, **WO 00/20336** relates to an ultrafine natural calcium carbonate, which may optionally be treated with one or several fatty acids or one or several salts or mixtures thereof, and which is used as a rheology regulator for polymer compositions. Whereas fatty acid and fatty acid salt mixtures are generally mentioned in this document, nowhere are such mixtures exemplified nor are any preferential dosing amounts of the fatty acid relative to fatty acid salt referred to. Indeed, only stearic acid treatments of calcium carbonate are presented in the examples.

Likewise, **US 4,407,986** recites a precipitated calcium carbonate that is surface-treated with a dispersant that may include higher aliphatic acids and their metal salts in order to limit the addition of lubricant additives when kneading this calcium carbonate with crystalline polypropylene and to avoid the formation of calcium carbonate aggregates that limit the impact strength of the polypropylene.

In **EP 0 325 114**, relating to non-sagging underseal compositions for motor vehicles based on polyvinyl chloride which has improved rheological and adhesion properties, Example 7 discloses a mixture of an ammonium salt of 12-hydroxystearic acid in combination with a fatty acid (in a weight ratio of 1:1) used to treat a mineral filler.

WO 03/082966 relates to a cross-linkable and/or cross-linked nanofiller composition which, in optional embodiments, may additionally include fillers that may or may not be coated with stearic acid, stearate, silane, siloxane and/or titanate. Such nanofiller compositions are used to increase barrier properties, strength and heat distortion temperatures, making them useful in medical, automotive, electrical, construction and food application.

US 2002/0102404 describes dispersible calcium carbonate particles coated on their surface with a combination of saturated and unsaturated aliphatic carboxylic acids and salts thereof along with an organic compound such as a phthalic ester, which are used in adhesive compositions to improve viscosity stability and adhesion properties. Whereas mixtures of fatty acids and fatty acid salts are generally mentioned, the examples include only treatments of calcium carbonate with mixtures of fatty acid salts.

Moreover, **US 2002/0102404** requires the implementation of a mixture of saturated and unsaturated aliphatic carboxylic acids/salts. The presence of unsaturated aliphatic carboxylic acids/salts increases the risk of unwanted in situ side reactions with the double bond during processing of any unsaturated aliphatic carboxylic acid/salt-comprising material. Additionally, the presence of unsaturated aliphatic carboxylic acids/salts may result in discoloration of, or unwanted odour development, and notably rancid odours, in the material in which they are implemented.

Claim 11 of **WO 92/02587** indicates that a saponified sodium salt solution of at least one high molecular weight unsaturated fatty acid or combination of at least one high molecular weight unsaturated fatty acid and at least one high molecular weight unsaturated fatty acid, may be added to a pre-heated slurry of precipitated calcium carbonate, to ultimately produce a desired level of fatty acid coating on the calcium carbonate before proceeding with further process steps.

The abstract of **JP54162746** discloses a composition comprising given relative amounts of rigid vinyl chloride resin, fatty acid treated-colloidal calcium carbonate, and barium stearate used in order to improve the heat stability of the vinyl chloride composition.

US 4,520,073 describes mineral filler materials with improved hydrophobic coatings prepared by pressure coating of porous minerals using steam as a carrier for the coating material. Said coating material may be selected, among other options, from long chain aliphatic fatty acids and their salts.

WO 01/32787 describes a particulate alkaline earth metal carbonate material product which has on its particles a coating of hydrophobic material comprising a composition formed of (a) a first component which comprises the reaction product of the alkaline earth metal carbonate and at least one given aliphatic carboxylic acid and (b) a second component having a carbonate release temperature substantially higher than the first component comprises a compound of formula $CH_3(CH_2)_mCOOR$, wherein, among other options, R is a Group II metal radical; further limitations are provided regarding the amounts of each component to be implemented. Notably, it is indicated that the second component forms at least 10 % by weight of the coating composition. Whereas a wide range of possible weight ratios are very generally indicated on page 13, line 20: "weight ratio of the first component to the second component may be from 10:80 to 90:10" (preferentially from 20:80 to 80:20), all of the further discussion and all of the given examples focus the skilled man solely on weight ratios between 50:50 and 90:10, which might be due to the fact that the only quite generally listed ratios where the fatty acid salt weight fraction exceeds that of the fatty acid (i.e. 80:20 and 90:10) provide treatment agents of unworkably high viscosities, namely above 10 000 mPa·s.

Additional prior art, namely **WO 99/61521** and **WO 2005/075353**, which suggest a reduction of only the inherent water and picked up humidity of the starting mineral filler, entirely missed the point of reducing the other volatiles besides water which contribute to the total volatiles.

Thus, it can be taken from the prior art that carboxylic acids and/or carboxylic acid salts are commonly used treating agents for mineral fillers for different purposes.

However, no leading line or direction is available to the skilled man in the prior art, even in an indistinct manner, which would solve the following multifaceted technical problem with the help of these treatment agents:

- to treat a mineral filler such that it is sufficiently hydrophobic for applications in plastics requiring dispersability of said mineral filler in the polymer medium, and notably such that said filler is more hydrophobic than if treated with only an aliphatic carboxylic acid salt;
- to treat a mineral filler with a treatment agent featuring a workable viscosity, that is to say a viscosity of less than 10 000 mPa·s at 180°C;
- to increase the volatile onset temperature such that this onset temperature is significantly greater than that of a mineral filler treated with only an aliphatic carboxylic acid;
- to limit the total quantity of volatiles issued between 25 and 280°C;
- to identify a treatment agent that achieves the above regardless of whether or not the mineral filler(s) undergo a salt exchange on contact with carboxylic acids to created carboxylic acid salts at the filler surface.

Consequently, it is the object of the present invention to provide a treated mineral filler product having the above characteristics.

A further object of the present invention also resides in a process for the preparation of such a treated mineral filler product, as well as to obtain a corresponding product by this process.

Finally, the use of such mineral filler products in plastic applications are also an object of the present invention.

For the purpose of the present application, the "**volatile onset temperature**" is defined as the temperature at which volatiles - including volatiles introduced as a result of common mineral filler preparation steps including grinding, with or without grinding aid agents, beneficiation, with or without flotation aid or other agents, and other pre-treatment agents not expressly listed above, detected according to the thermogravimetric analysis described hereafter – begin to evolve, as observed on a thermogravimetric (TGA) curve, plotting the mass of remaining sample (y-axis) as a function of temperature (x-axis), the preparation and interpretation of such a curve being defined hereafter.

For the purpose of the present application, the "**total volatiles**" associated with mineral fillers and evolved over a temperature range of 25 to 280°C is characterised according to % mass loss of the mineral filler sample over a temperature range as read on a thermogravimetric (TGA) curve.

TGA analytical methods provide information regarding losses of mass and volatile onset temperatures with great accuracy, and is common knowledge; it is, for example, described in "Principles of Instrumental analysis", fifth edition, Skoog, Holler, Nieman, 1998 (first edition 1992) in Chapter 31 pages 798 to 800, and in many other commonly known reference works. In the present invention, thermogravimetric analysis (TGA) is performed using a Mettler Toledo TGA 851 based on a sample of 500 +/- 50 mg and scanning temperatures from 25 to 280°C at a rate of 20°C/minute under an air flow of 70 ml/min.

The skilled man will be able to determine the "**volatile onset temperature**" by analysis of the TGA curve as follows: the first derivative of the TGA curve is obtained and the inflection points thereon between 150 and 280°C are identified. Of the inflection points having a tangential slope value of greater than 45° relative to a horizontal line, the one having the lowest associated temperature above 200°C is identified. The temperature value associated with this lowest temperature inflection point of the first derivative curve is the "**volatile onset temperature**". An illustration of such an evaluation is given in Figures 1 and 2, described in the examples section hereafter.

The "**total volatiles**" evolved on the TGA curve is determined using Star^{e} SW 9.01 software. Using this software, the curve is first normalised relative to the original sample weight in order to obtain mass losses in % values relative to the original sample. Thereafter, the temperature range of 25 to 280°C is selected and the step horizontal (in

German: "Stufe horizontal") option selected in order to obtain the % mass loss over the selected temperature range.

Throughout the present document, a molten state is defined as the state in which a material is entirely liquid, in other words is entirely melted. Whereas the phenomenon of melting occurs at constant temperature on application of energy, a substance is qualified as being molten as of the moment following melting when the temperature begins to rise, as observed on a curve plotting temperature versus energy input obtained by thermogravimetric analysis (TGA). The details of such an analysis are provided hereafter.

Throughout the present document, the specific surface area (in m^2/g) of the mineral filler is determined using the BET method, which is well known to the skilled man (ISO 9277:1995). The total surface area (in m^2) of the mineral filler is then obtained by multiplication of the specific surface area and the mass (in g) of the mineral filler prior to treatment.

Throughout the present document, a dry mineral filler is understood to be a mineral filler having less than 0.2 % by weight of water relative to the mineral filler weight. The % water is determined according to the Coulometric Karl Fischer measurement method, wherein the mineral filler is heated to 220°C, and the water content released as vapour and isolated using a stream of nitrogen gas (at 100 ml/min) is determined in a Coulometric Karl Fischer unit.

Throughout the present document, the hydrophobicity of a mineral filler product is evaluated by determining the minimum alcohol to water ratio in an alcohol-water mixture needed for the settling of a majority of said mineral filler product, where said mineral filler product is deposited on the surface of said alcohol-water mixture by passage through a specific sieve.

Throughout the present document, the value d_x represents the diameter relative to which X % by weight of the particles have a diameter less than d_x, and is determined based on measurements made using Malvern Mastersizer^{TM} X instrumentation (with software version 2.18 and using the OHD presentation and analysis model).

Throughout the present document, the term saturated means having an iodine number of less than 5, preferably less than 1 g I_2/100g sample. This iodine number determination is well-known to the skilled man, and namely implements a determination of the iodine addition to a 100 g sample by back-titration of the surplus iodine with sodium thiosulfate.

Throughout the present document, all viscosity values are measured in a PHYSICA MCR 300 equipped with a CP50 – 1 instrumentation at a shear rate of 5 s^{-1} and scanning temperatures from 200 to 130 °C.

To respond to the above needs of the skilled man, the Applicant has identified a treated mineral filler product comprising:

a) at least one mineral filler;

b) a treatment layer located on the surface of said mineral filler(s), said treatment layer comprising:

- at least one saturated C_8 to C_{24} aliphatic carboxylic acid; and
- at least one di and/or trivalent cation salt of one or more saturated C_8 to C_{24} aliphatic carboxylic acid; wherein

- the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 51:49 to 75:25; and
- said treatment layer is present in an amount of at least 2.5 mg/m^2 of said mineral filler.

According to one preferred embodiment of the invention, the inventive treated mineral filler product features a total volatiles between 25 and 280°C of less than 0.25 % by mass, and preferably of less than 0.23 % by mass, e.g. of from 0.04 to 0.21 % by mass, preferably from 0.08 to 0.15 % by mass, more preferably from 0.1 to 0.12 % by mass.

In another preferred embodiment, the treated mineral filler product features a volatile onset temperature of greater than or equal to 270°C, and preferably of greater than or equal to 290°C, most preferably of greater than or equal to 300°C.

Preferably, the treated mineral filler product according to the invention features a higher volatile onset temperature than the same mineral filler having a treatment layer but wherein the aliphatic carboxylic acid salt is replaced with the corresponding aliphatic carboxylic acid such that the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 0:100 to 50:50.

In another preferred embodiment, the treated mineral filler product is more hydrophobic than the same mineral filler having a treatment layer but wherein the aliphatic carboxylic acid(s) are entirely replaced with a corresponding aliphatic carboxylic acid alkali or earth alkali metal salt(s).

Additionally, it is preferred that said treatment layer has a weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) of from 55:45 to 75:25, more preferably is from about 60:40 to 70:30, e.g. from 64:36 to 67:33.

Preferred mineral filler(s) are calcium carbonate-comprising mineral fillers and/or plate-like mineral-comprising mineral fillers and/or quartz-comprising mineral fillers and/or clay-comprising mineral fillers. Most preferably, said mineral filler(s) are calcium carbonate-comprising mineral fillers. In such a case, these calcium carbonate-comprising mineral fillers may be precipitated calcium carbonate (PCC), namely featuring one or more of aragonitic, vateritic and calcitic mineralogical crystal forms, and/or natural ground calcium carbonate (NGCC), namely one or more of marble, limestone, or chalk, and/or dolomite.

Most preferably, said calcium carbonate-comprising mineral fillers are marble and/or dolomite.

Alternatively or additionally, these fillers include plate-like mineral fillers, such as talc.

As regards the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s), these are preferably selected from among calcium, magnesium, strontium and aluminium salts, and mixtures thereof, and more preferably are selected from among calcium salts, magnesium salts and mixtures thereof.

In another embodiment, the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are di and/or trivalent cation salt of one or more C_{10} to C_{18}, and preferably are di and/or trivalent cation salt of one or more C_{12} to C_{18} aliphatic carboxylic acid(s).

In another embodiment, the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of di and/or trivalent cation salt of one or more aliphatic monocarboxylic acid(s).

These di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid may also or alternatively be salt(s) of one or more linear aliphatic carboxylic acid(s), and/or may be salt(s) of one or more hydroxylated (i.e. OH group-comprising) aliphatic carboxylic acid(s).

In particular, the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) may be salt(s) of one or more fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acid(s), and most preferably are salts of stearic and/or palmitic acid(s).

According to another embodiment of the invention, the di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid feature an equivalent isolated viscosity of more than 100 000 mPa·s, and preferably of more than 1 000 000 mPa·s, at 180 °C when measured in a PHYSICA MCR 300 equipped with a CP50 – 1 instrumentation at a shear rate of $5 s^{-1}$ and scanning temperatures from 200 to 130 °C.

The C_8 to C_{24} aliphatic carboxylic acid of the di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid and the C_8 to C_{24} aliphatic carboxylic acid(s) may or may not be the same.

As regards the C_8 to C_{24} aliphatic carboxylic acid(s), these may be C_{10} to C_{18}, and are preferably C_{12} to C_{18} aliphatic carboxylic acid(s).

These C_8 to C_{24} aliphatic carboxylic acid(s) may also or alternatively be aliphatic monocarboxylic acids, and/or linear aliphatic carboxylic acids and/or hydroxylated (i.e. OH group-comprising) aliphatic carboxylic acids.

In a preferred embodiment, these C_8 to C_{24} aliphatic carboxylic acid(s) are fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acids or mixtures thereof, and most preferably are stearic and/or palmitic acid.

The total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) in the treatment agent preferably accounts for greater than 2.7, more preferably at least 3, especially at least 3.2, e.g. 3.5 mg of total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s)/m^2 of mineral filler(s).

It is also preferred that the equivalent isolated mixture of the aliphatic carboxylic acid salts(s) and the aliphatic carboxylic acid(s) features a viscosity of less than 10 000, preferably of less than 1 000, and more preferably of less than 500 mPa·s at 180 °C. Indeed, above a value of 10 000 mPa·s, a treatment agent is largely unworkable.

In a more preferred embodiment, the aliphatic carboxylic acid(s) is a 1:1 stearic acid:palmitic acid mixture, and the aliphatic carboxylic acid salt(s) is a magnesium or calcium stearate.

The treatment agent may also further contain additional agents that do not correspond to a C_8 to C_{24} aliphatic carboxylic acid, nor to a di and/or trivalent cation salt of a C_8 to C_{24} aliphatic carboxylic acid. In such a case, this additional treatment agent is preferably a siloxane, and in particular a polydimethylsiloxane (PDMS).

A further aspect of the present invention is a process for the preparation of such a treated mineral filler product, characterised in that the process comprises the following steps:

(a) providing at least one dry mineral filler;
(b) providing at least one saturated C_8 to C_{24} aliphatic carboxylic acid(s);

(c) providing at least one di and/or trivalent cation salt of one or more saturated C_8 to C_{24} aliphatic carboxylic acid;

(d) contacting said mineral filler(s) of step (a), in one or more steps under heating, with:

   a. the aliphatic carboxylic acid(s) of step (b);
   b. the aliphatic carboxylic acid salt(s) of step (c), whereupon

(e) a treatment layer comprising said aliphatic carboxylic acid(s) and said aliphatic carboxylic acid salt(s) is formed on the surface of said mineral filler(s) resulting in a treated mineral filler product; wherein

- the weight ratio of all of said aliphatic carboxylic acid salt(s) provided in step (c) : all of said aliphatic carboxylic acid(s) provided in step (b) is from 51:49 to 75:25;
- the total weight of all of said aliphatic carboxylic acid salt(s) and all of said aliphatic carboxylic acid(s) located on the surface of the mineral filler is at least 2.5 mg/m^2 of the mineral filler provided in step (a);
- during step (d), the temperature is adjusted such that all of the aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) are molten.

In a preferred embodiment, the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 55:45 to 75:25, more preferably is from about 60:40 to 70:30, e.g. from 64:36 to 67:33.

The mineral filler(s) in (a) may, in a preferred embodiment, be a calcium carbonate-comprising mineral filler and/or plate-like mineral-comprising mineral filler and/or quartz-comprising mineral filler and/or clay-comprising mineral filler; more preferably it is a calcium carbonate-comprising mineral filler.

In the latter case, this calcium carbonate-comprising mineral fillers may be a precipitated calcium carbonate (PCC), namely one or more of the aragonitic, vateritic and calcitic mineralogical crystal forms, and/or a natural ground calcium carbonate (NGCC), namely one or more of marble, limestone, or chalk, and/or dolomite.

The calcium carbonate-comprising mineral fillers are preferably marble and/or dolomite.

Alternatively or additionally, these fillers may include plate-like mineral fillers such as talc.

As regards the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) , these are preferably selected from among calcium, magnesium, strontium and aluminium salts, and mixtures thereof, and more preferably are selected from among calcium salts, magnesium salts and mixtures thereof.

Alternatively or additionally, these may be di and/or trivalent cation salts of one or more C_{10} to C_{18}, and preferably are di and/or trivalent cation salt of one or more C_{12} to C_{18} aliphatic carboxylic acid(s).

In another embodiment, said di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of di and/or trivalent cation salt of one or more aliphatic monocarboxylic acid(s).

In yet another embodiment, said di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid are salt(s) of one or more linear aliphatic carboxylic acid(s). In another embodiment, they are C_8 to C_{24} aliphatic carboxylic acid salt(s) of one or more hydroxylated (i.e. OH group-comprising) aliphatic carboxylic acid(s).

In a preferred embodiment, said C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of one or more fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acid(s), and most preferably are salts of stearic and/or palmitic acid(s).

Said di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid may feature an equivalent isolated viscosity of more than 100 000 mPa·s, and preferably of more than 1 000 000 mPa·s, at 180 °C when measured in a PHYSICA MCR 300 equipped with a CP50 – 1 instrumentation at a shear rate of 5 s^{-1} and scanning temperatures from 200 to 130 °C.

Said C_8 to C_{24} aliphatic carboxylic acid of the di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid and the C_8 to C_{24} aliphatic carboxylic acid(s) may or may not be equivalent.

As regards said C_8 to C_{24} aliphatic carboxylic acid(s), these may be C_{10} to C_{18}, and preferably are C_{12} to C_{18} aliphatic carboxylic acid(s).

In a preferred embodiment, said C_8 to C_{24} aliphatic carboxylic acid(s) are aliphatic monocarboxylic acids. Alternatively or additionally, they may be linear aliphatic carboxylic acids and/or hydroxylated (i.e. OH group-comprising) aliphatic carboxylic acids.

In another preferred embodiment, said C_8 to C_{24} aliphatic carboxylic acid(s) are fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acids or mixtures thereof, and most preferably are stearic and/or palmitic acid.

In one embodiment of the invention, the total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) accounts for greater than 2.7, more preferably at least 3, especially at least 3.2, e.g. 3.5 mg of total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s)/m^2 of mineral filler(s).

It is preferred that the equivalent isolated mixture of the aliphatic carboxylic acid salts(s) and the aliphatic carboxylic acid(s) features a viscosity of less than 10 000, preferably of less than 1 000, and more preferably of less than 500 mPa·s at 180 °C. Indeed, above a value of 10 000 mPa·s, a treatment agent is largely unworkable.

In another preferred embodiment, the aliphatic carboxylic acid is a 1:1 stearic acid:palmitic acid mixture, and the aliphatic carboxylic acid salt(s) is a magnesium or calcium stearate.

It is also possible that additional treatment agents that do not correspond to a C_8 to C_{24} aliphatic carboxylic acid, nor to a di and/or trivalent cation salt of a C_8 to C_{24} aliphatic carboxylic acid are implemented in the process of the present invention. In such a case, it is preferred that this additional treatment agent is a siloxane, and more preferably a polydimethylsiloxane (PDMS).

As regards the mineral filler provided to step (a), it may previously have been dry or wet ground, and preferably dry ground, optionally with a grinding agent. It is also common that such a mineral filler undergoes a beneficiation step (such as a flotation, bleaching or magnetic separation step) to remove impurities.

In order to optimise the particle size distribution characteristics, it is also standard to subject the mineral filler(s) to a classification step. Indeed, it may be preferred to implement mineral filler(s) in step (a) featuring a d_{50} of 0.5 to 10 microns, and more preferably featuring a d_{50} of 1.5 to 1.8 microns. A mineral filler d_{98} of less than 25 microns may also be advantageous.

The process of the present invention may be a continuous or batch process.

When implementing the aliphatic carboxylic acid(s) provided to step (b), it is preferably that these be in a molten state. The same is true of the aliphatic carboxylic acid salt(s) provided to step (c).

Step (d) of contacting the mineral filler with the aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) preferably takes place under mixing conditions. The skilled man will adapt these mixing conditions (such as the configuration of mixing pallets and mixing speed) according to his process equipment.

It is preferred that in step (d), all or part of said aliphatic carboxylic acid salt(s) of step (c) and all or part of said aliphatic carboxylic acid(s) of step (b), and preferably all of said aliphatic carboxylic acid salt(s) of step (c) and all of said aliphatic carboxylic acid(s) of step (b), are first contacted with one another and mixed to form a molten mixture prior to contacting any of said mineral filler(s).

In the case where an additional additive is implemented, such as siloxane, this would then generally be introduced in the process following step (d).

Another object of the invention resides in the treated mineral filler product obtained by the process of the invention. Such treated mineral fillers are characterised by a higher volatile onset temperature than a comparable mineral filler obtained according to the same process but wherein the aliphatic carboxylic acid salt is replaced with the corresponding aliphatic carboxylic acid such that the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 0:100 to 50:50.

Such treated mineral filler products may also generally provide a total volatiles between 25 and 280°C of less than 0.25 %, and preferably of less than 0.23 % by mass, e.g. of from 0.04 to 0.21 % by mass, preferably from 0.08 to 0.15 % by mass, more preferably from 0.1 to 0.12 % by mass.

The volatile onset temperature of such treated mineral filler products may also, generally, be greater than or equal to 270°C, preferably greater than or equal to 290°C, most preferably of greater than or equal to 300°C.

Moreover, such treated mineral filler products are generally more hydrophobic than the same mineral filler having the same treatment layer but wherein the aliphatic carboxylic acid(s) are entirely replaced with a corresponding aliphatic carboxylic acid alkali or earth alkali metal salt(s).

Such treated mineral filler products as described above may advantageously be implemented in a process of mixing and/or extruding and/or compounding and/or blow moulding with plastic materials, and preferably with polyolefins or thermoplastics such as polyethylenes (PE), polypropylenes (PP) and/or polyurethanes (PU), particularly to obtain films, namely stretched / oriented films, and preferably breathable films, or extrusion coating films.

The following figures, examples and tests will additionally illustrate the invention without in any way limiting its scope.

Description of the figures:

Figure 1 presents the TGA curve obtained for the treated mineral filler product of comparative Example 1.

Figure 2 presents the TGA curve obtained for the treated mineral filler product of Example 5 according to the invention.

EXAMPLES

All measurement methods implemented in the examples are described hereabove.

In all cases, the hydrophobicity of the resulting material was compared to that of a material treated according to the same process but wherein the aliphatic carboxylic acid is entirely replaced by the corresponding calcium or magnesium salt. The indication "yes" implies that the material is more hydrophobic than the purely salt treated comparison.

Stearic acid and dry palmitic acid powder mixtures used in the tests hereafter feature a weight ratio of stearic acid:palmitic acid of 56:44 based on pure forms of the acids obtained from Fluka.

Calcium stearate used in the tests hereafter, commercialised under the trade name Ceasit I, was obtained from Baerlocher.

Magnesium stearate used in the tests hereafter was obtained from Siegfried Handel.

Magnesium laurate used in the tests hereafter was synthesized by reaction of lauric acid, purum grade obtained from Fluka, and sodium hydroxide, purum grade from Fluka, followed by precipitation with magnesium hydroxide, purum grade from Fluka.

Example 1 – Comparative Example

500 g of a cyclone-classified, marble from Carrara, Italy, dry ground using a glycol-based dry grinding aid and featuring a d_{50} of approximately 2.2 microns and a specific surface area of 3.6 was added to an MTI Mixer and the mixing was activated at 500 rpm. Thereafter a 1:1 mixture of dry stearic acid powder and dry palmitic acid powder at room temperature was introduced to the mixer in a quantity so as to obtain the mg of treatment agent per m^2 of marble indicated in Table 1, and the mixer contents were heated to 130°C. The contents of the mixer were mixed at 130°C under a stirring speed of 500 rpm for a period of 10 minutes.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Figure 1 presents the TGA curve obtained for the treated mineral filler product of Example 1. On Figure 1, the broad continuous line represents the % remaining sample weight relative to the original sample weight as a function of both temperature and time as issued by the TGA instrumentation software. The dashed line represents the first derivative of this issued curve, and the narrow continuous line represents the second derivative of this issued curve. A tangential line is drawn at the inflection point of the second derivative curve having an angle (α, also figured) of at least 45° at the lowest associated temperature above 200°C. The temperature associated with this inflection point is the volatile onset temperature.

Example 2 – Comparative Example

500 g of a cyclone-classified, marble from Carrara, Italy, dry ground using a glycol-based dry grinding aid and featuring a d_{50} of approximately 2.2 microns and a specific surface area of 3.6 was added to an MTI Mixer and the mixing was activated at 500 rpm. Thereafter calcium stearate powder at room temperature was introduced to the mixer in a quantity so as to obtain the mg of treatment agent per m^2 of marble indicated in Table 1, and the mixer contents were heated to 180°C. The contents of the mixer were mixed at 180°C under a stirring speed of 500 rpm for a period of 10 minutes.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 3 – Comparative Example

500 g of a cyclone-classified, marble from Carrara, Italy, dry ground using a glycol-based dry grinding aid and featuring a d_{50} of approximately 2.2 microns and a specific surface area of 3.6 was added to an MTI Mixer and the mixing was activated at 500 rpm. Separately, a 1:1 mixture of dry stearic acid powder and dry palmitic acid powder was mixed by hand with calcium stearate, also in powder form, in the relative amounts listed in Table 1 at a temperature of 180°C in a beaker. Once a visually homogeneous molten mixture of the acid and salt were obtained, this molten mixture was allowed to cool to form a powder. The so obtained powder was thereafter added to the marble in the MTI Mixer in a quantity so as to obtain the mg of treatment agent per m^2 of marble indicated in Table 1. The contents of the mixer heated to 130°C and were mixed at 180°C under a stirring speed of 500 rpm for a period of 10 minutes.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 4 – Comparative Example

Example 3 above was repeated but implementing the relative amounts of aliphatic carboxylic acid to aliphatic carboxylic acid salt listed in Table 1.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 5 – Example of the Invention

Example 3 above was repeated but implementing the relative amounts of aliphatic carboxylic acid to aliphatic carboxylic acid salt listed in Table 1.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Figure 2 presents the TGA curve obtained for the treated mineral filler product of Example 5. On Figure 2, the broad continuous line represents the % remaining sample weight relative to the original sample weight as a function of both temperature and time as issued by the TGA instrumentation software. The dashed line represents the first derivative of this issued curve, and the narrow continuous line represents the second derivative of this issued curve. A tangential line is drawn at the inflection point of the second derivative curve having an angle (α', also figured) of at least 45° at the lowest associated temperature above 200°C. The temperature associated with this inflection point is the volatile onset temperature.

Example 6 – Example of the Invention

Example 3 above was repeated but replacing the marble with a marble featuring a d_{50} of approximately 2.2 microns and a specific surface area of 3.6, and implementing the relative amounts of aliphatic carboxylic acid to aliphatic carboxylic acid salt listed in Table 1 and so as to obtain the mg of treatment agent per m^2 of marble indicated in Table 1.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 7 – Example of the Invention

Example 3 above was repeated but replacing the marble with a marble featuring a d_{50} of approximately 6.5 microns and a specific surface area of 1.8 m^2/g, implementing the relative amounts of aliphatic carboxylic acid to aliphatic carboxylic acid salt listed in Table 1 so as to obtain the mg of treatment agent per m^2 of marble indicated in Table 1.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 8 – Example of the Invention

Example 3 above was repeated but replacing calcium stearate by magnesium laurate and implementing the relative amounts of aliphatic carboxylic acid to aliphatic carboxylic acid salt listed in Table 1.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 9 – Comparative Example

500 g of a cyclone-classified dolomite, dry ground using a glycol-based dry grinding aid and featuring a d_{50} of approximately 3.4 microns and a specific surface area of 2.9 m^2/g, was added to an MTI Mixer and the mixing was activated at 500 rpm. Separately, dry lauric acid powder was mixed by hand with aluminium stearate, also in powder form, in the relative amounts listed in Table 1 at a temperature of 180°C in a beaker. Once a visually homogeneous molten mixture of the acid and salt were obtained, this molten mixture was allowed to cool to form a powder. The so obtained powder was thereafter added to the dolomite in the MTI Mixer in a quantity so as to obtain the mg of treatment agent per m^2 of dolomite indicated in Table 1. The contents of the mixer were heated and mixed at 180°C under a stirring speed of 500 rpm for a period of 10 minutes.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Example 10 –Example of the Invention

500 g of a cyclone-classified dolomite, dry ground using a glycol-based dry grinding aid and featuring a d_{50} of approximately 3.4 microns and a specific surface area of 2.9 m^2/g, was added to an MTI Mixer and the mixing was activated at 500 rpm. Separately, dry lauric acid powder was mixed by hand with aluminium stearate, also in powder form, in the relative amounts listed in Table 1 at a temperature of 180°C in a beaker. Once a visually homogeneous molten mixture of the acid and salt were obtained, this molten mixture was allowed to cool to form a powder. The so obtained powder was thereafter added to the dolomite in the MTI Mixer in a quantity so as to obtain the mg of treatment

agent per m^2 of dolomite indicated in Table 1. The contents of the mixer were heated and mixed at 180°C under a stirring speed of 500 rpm for a period of 10 minutes.

The product so obtained was thereafter analysed; the results are presented in Table 1.

Table 1

Test	1	2	3	4	5	6	7	8	9	10
	Comparison	**Comparison**	**Comparison**	**Comparison**	**Invention**	**Invention**	**Invention**	**Invention**	**Comparison**	**Invention**
Mineral	$CaCO_3$	$CaCO_3$	$CaCO_3$	$CaCO_3$	$CaCO_3$	$CaCO_3$	$CaCO_3$	$CaCO_3$	Dolomite	Dolomite
Carboxylic acid	Stearic / palmitic acid (56:44)		Stearic / palmitic acid (56:44)	Stearic / palmitic acid (56:44)	Stearic / palmitic acid (56:44)	Stearic / palmitic acid (56:44)	Stearic / palmitic acid (56:44)	Stearic / palmitic acid (56:44)	Lauric acid	Lauric acid
Carboxylic acid salt		Ca stearate	Ca stearate	Ca stearate	Ca stearate	Ca stearate	Ca stearate	Mg Laurate	Aluminium stearate	Aluminium stearate
Total treatment agent [mg/m^2 of mineral]	3.1	2.9	2.9	3.5	3.1	3.8	3.2	3.2	3.0	3.0
Weight ratio carboxylic acid salt(s) : carboxylic acid(s)	0 : 100	100 : 0	33 : 67	50 : 50	67 : 33	75 : 25	75 : 25	70 : 30	40 : 60	60 : 40
Isolated carboxylic acid salt viscosity [mPa·s]		1 600 000	1 600 000	1 600 000	1 600 000	1 600 000	1 600 000	1 600 000	567 000	567 000
Treatment agent viscosity	--	--	< 100 mPa·s	< 100 mPa·s	< 100 mPa·s	2600 mPa·s	2600 mPa·s	9000 mPa·s	653 mPa.s	6 740 mPa.s
Total volatiles [% by mass] (± 0.01%)	0.36%	0.08%	0.31%	0.34%	0.15%	0.21%	0.08%	0.04%	0.23%	0.21%
Volatile onset temperature	240°C	303°C	240°C	240°C	290°C	300°C	300°C	290°C	237°C	255°C
More hydrophobic than when treated with equivalent carboxylic acid salt	yes	Not applicable	yes	yes	yes	yes	yes	yes	yes	yes

The results of Table 1 clearly demonstrate that only the inventive process simultaneously implements treatment agents having a workable viscosity and results in treated products featuring the desired low "volatile onset temperature", low "total volatiles" and required hydrophobicity.

CLAIMS

1. A treated mineral filler product comprising:

a) at least one mineral filler;

b) a treatment layer located on the surface of said mineral filler(s), said treatment layer comprising:

  - at least one saturated C_8 to C_{24} aliphatic carboxylic acid; and
  - at least one di and/or trivalent cation salt of one or more saturated C_8 to C_{24} aliphatic carboxylic acid;

characterised in that:

- the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 51:49 to 75:25; and
- said treatment layer is present in an amount of at least 2.5 mg/m^2 of said mineral filler.

2. The treated mineral filler product according to claim 1, **characterised in that** it features a total volatiles between 25 and 280°C of less than 0.25 %, and preferably of less than 0.23 % by mass, e.g. of from 0.04 to 0.21 % by mass, preferably from 0.08 to 0.15 % by mass, more preferably from 0.1 to 0.12 % by mass.

3. The treated mineral filler product according to claim 1 or 2, **characterised in that** it features a volatile onset temperature of greater than or equal to 270°C, and preferably of greater than or equal to 290°C, most preferably of greater than or equal to 300°C.

4. The treated mineral filler product according to any of claims 1 to 3, **characterised in that** it has a higher volatile onset temperature than the same mineral filler having a treatment layer but wherein the aliphatic carboxylic acid salt is replaced with the corresponding aliphatic carboxylic acid such that the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 0:100 to 50:50.

5. The treated mineral filler product according to any of claims 1 to 4, **characterised in that** it is more hydrophobic than the same mineral filler having a treatment layer but wherein the aliphatic carboxylic acid(s) are entirely replaced with a corresponding aliphatic carboxylic acid alkali or earth alkali metal salt(s).

6. The treated mineral filler product according to any of claims 1 to 5, **characterised in that** said weight ratio of all of said aliphatic carboxylic acid salt(s):all of said aliphatic carboxylic acid(s) is from 55:45 to 75:25, more preferably is from about 60:40 to 70:30, e.g. from 64:36 to 67:33.

7. The treated mineral filler product according to any of claims 1 to 6, **characterised in that** the mineral filler(s) in (a) are calcium carbonate-comprising mineral fillers and/or plate-like mineral-comprising mineral fillers and/or quartz-comprising mineral fillers and/or clay-comprising mineral fillers, and are preferably calcium carbonate-comprising mineral fillers.

8. The treated mineral filler product according to claim 7, **characterised in that** the calcium carbonate-comprising mineral fillers are precipitated calcium carbonate (PCC), namely one or more of the aragonitic, vateritic and calcitic mineralogical crystal forms, and/or natural ground calcium carbonate (NGCC), namely one or more of marble, limestone, or chalk, and/or dolomite.

9. The treated mineral filler product according to claim 8, **characterised in that** the calcium carbonate-comprising mineral fillers are marble and/or dolomite.

10. The treated mineral filler product according to claim 7, **characterised in that** the plate-like mineral fillers are talc.

11. The treated mineral filler product according to any of claims 1 to 10, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are selected from among calcium, magnesium, strontium and aluminium salts, and mixtures thereof, and preferably are selected from among calcium salts, magnesium salts and mixtures thereof.

12. The treated mineral filler product according to any of claims 1 to 11, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are di and/or trivalent cation salt of one or more C_{10} to C_{18}, and preferably are di and/or trivalent cation salt of one or more C_{12} to C_{18} aliphatic carboxylic acid(s).

13. The treated mineral filler product according to any of claims 1 to 12, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of di and/or trivalent cation salt of one or more aliphatic monocarboxylic acid(s).

14. The treated mineral filler product according to any of claims 1 to 13, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of one or more fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acid(s), and most preferably are salts of stearic and/or palmitic acid(s).

15. The treated mineral filler product according to any of claims 1 to 14, **characterised in that** the di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid feature an equivalent isolated viscosity of more than 100 000 mPa·s, and preferably of more than 1 000 000 mPa·s, at 180 °C when measured in a PHYSICA MCR 300 equipped with a CP50–1 instrumentation at a shear rate of 5 s^{-1} and scanning temperatures from 200 to 130 °C.

16. The treated mineral filler product according to any of claims 1 to 15, **characterised in that** the C_8 to C_{24} aliphatic carboxylic acid(s) are C_{10} to C_{18}, and preferably a C_{12} to C_{18} aliphatic carboxylic acid(s).

17. The treated mineral filler product according to any of claims 1 to 16, **characterised in that** the C_8 to C_{24} aliphatic carboxylic acid(s) are fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acids or mixtures thereof, and most preferably are stearic and/or palmitic acid.

18. The treated mineral filler product according to any of claims 1 to 17, **characterised in that** the total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) accounts for greater than 2.7, more preferably at least 3, especially at least 3.2, e.g. 3.5 mg of total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s)/m^2 of mineral filler(s).

19. The treated mineral filler product according to any of claims 1 to 18, **characterised in that** the equivalent isolated mixture of the aliphatic carboxylic acid salts(s) and the aliphatic carboxylic acid(s) features a viscosity of less than 10 000, preferably of less than 1 000, and more preferably of less than 500 mPa·s at 180 °C.

20. The treated mineral filler product according to any of claims 1 to 19, **characterised in that** the aliphatic carboxylic acid is a 1:1 stearic acid:palmitic acid mixture, and the aliphatic carboxylic acid salt(s) is a magnesium or calcium stearate.

21. Process for the preparation of a treated mineral filler product, **characterised in that** said process comprises the following steps:

(a) providing at least one dry mineral filler;
(b) providing at least one saturated C_8 to C_{24} aliphatic carboxylic acid(s);
(c) providing at least one di and/or trivalent cation salt of one or more saturated C_8 to C_{24} aliphatic carboxylic acid;
(d) contacting said mineral filler(s) of step (a), in one or more steps under heating, with:
    a. the "aliphatic carboxylic acid(s)" of step (b);
    b. the "aliphatic carboxylic acid salt(s)" of step (c); whereupon
(e) a treatment layer comprising said aliphatic carboxylic acid(s) and said aliphatic carboxylic acid salt(s) is formed on the surface of said mineral filler(s) resulting in a treated mineral filler product;

characterised in that:

- the weight ratio of all of said aliphatic carboxylic acid salt(s) provided in step (c) : all of said aliphatic carboxylic acid(s) provided in step (b) is from 51:49 to 75:25;
- the total weight of all of said aliphatic carboxylic acid salt(s) and all of said aliphatic carboxylic acid(s) located on the surface of the mineral filler is at least 2.5 mg/m^2 of the mineral filler provided in step (a);
- during step (d), the temperature is adjusted such that all of the aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) are molten.

22. Process according to claim 21,
characterised in that said weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 55:45 to 75:25, more preferably is from about 60:40 to 70:30, e.g. from 64:36 to 67:33.

23. Process according to claim 21 or 22,
characterised in that the mineral filler(s) in (a) are calcium carbonate-comprising mineral fillers and/or plate-like mineral-comprising mineral fillers and/or quartz-comprising mineral fillers and/or clay-comprising mineral fillers, and are preferably calcium carbonate-comprising mineral fillers.

24. Process according to claim 23,
characterised in that the calcium carbonate-comprising mineral fillers are precipitated calcium carbonate (PCC), namely one or more of the aragonitic, vateritic and calcitic mineralogical crystal forms, and/or natural ground calcium carbonate (NGCC), namely one or more of marble, limestone, or chalk, and/or dolomite.

25. Process according to claim 24,
characterised in that the calcium carbonate-comprising mineral fillers are marble and/or dolomite.

26. Process according to claim 23,
characterised in that the plate-like mineral filler is talc.

27. Process according to any of claims 21 to 26,
characterised in that the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are selected from among calcium, magnesium, strontium and aluminium salts, and mixtures thereof, and preferably are selected from among calcium salts, magnesium salts and mixtures thereof.

28. Process according to any of claims 21 to 27, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are di and/or trivalent cation salt of one or more C_{10} to C_{18}, and preferably are di and/or trivalent cation salt of one or more C_{12} to C_{18} aliphatic carboxylic acid(s).

29. Process according to any of claims 21 to 28, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of di and/or trivalent cation salt of one or more aliphatic monocarboxylic acid(s).

30. Process according to any of claims 21 to 29, **characterised in that** the di and/or trivalent cation salt(s) of one or more C_8 to C_{24} aliphatic carboxylic acid(s) are salt(s) of one or more fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acid(s), and most preferably are salts of stearic and/or palmitic acid(s).

31. Process according to any of claims 21 to 30, **characterised in that** the di and/or trivalent cation salt(s) of a C_8 to C_{24} aliphatic carboxylic acid feature an equivalent isolated viscosity of more than 100 000 mPa·s, and preferably of more than 1 000 000 mPa·s, at 180 °C when measured in a PHYSICA MCR 300 equipped with a CP50–1 instrumentation at a shear rate of 5 s^{-1} and scanning temperatures from 200 to 130 °C.

32. Process according to any of claims 21 to 31, **characterised in that** the C_8 to C_{24} aliphatic carboxylic acid(s) are C_{10} to C_{18}, and preferably a C_{12} to C_{18} aliphatic carboxylic acid(s).

33. Process according to any of claims 21 to 32, **characterised in that** the C_8 to C_{24} aliphatic carboxylic acid(s) are fatty acid(s), especially stearic and/or palmitic and/or myristic and/or lauric acids or mixtures thereof, and most preferably are stearic and/or palmitic acid.

34. Process according to any of claims 21 to 33, **characterised in that** the total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s) accounts for greater greater than 2.7, more preferably at least 3, especially at least 3.2, e.g. 3.5 mg of total aliphatic carboxylic acid(s) and aliphatic carboxylic acid salt(s)/m^2 of mineral filler(s).

35. Process according to any of claims 21 to 34, **characterised in that** the equivalent isolated mixture of the aliphatic carboxylic acid salts(s) and the aliphatic carboxylic acid(s) features a viscosity of less than 10 000, preferably of less than 1 000, and more preferably of less than 500 mPa·s at 180 °C.

36. Process according to any of claims 21 to 35, **characterised in that** the aliphatic carboxylic acid is a 1:1 stearic acid:palmitic acid mixture, and the aliphatic carboxylic acid salt(s) is a magnesium or calcium stearate.

37. Process according to any of claims 21 to 36, **characterised in that** the aliphatic carboxylic acid(s) provided to step (b) are in a molten state.

38. Process according to any of claims 21 to 37, **characterised in that** the aliphatic carboxylic acid salt(s) provided to step (c) are in a molten state.

39. Process according to any of claims 21 to 38, **characterised in that** in step (d), all or part of said aliphatic carboxylic acid salt(s) of step (c) and all or part of said aliphatic carboxylic acid(s) of step (b), and preferably all of said aliphatic carboxylic acid salt(s) of step (c) and all of said aliphatic carboxylic acid(s) of step (b), are first contacted with one another and mixed to form a molten mixture prior to contacting any of said mineral filler(s).

40. Treated mineral filler product obtained by the process of any of claims 21 to 39, **characterised in that** it features a higher volatile onset temperature than the same mineral filler obtained according to the same process but wherein the aliphatic carboxylic acid salt is replaced with the corresponding aliphatic carboxylic acid such that the weight ratio of all of said aliphatic carboxylic acid salt(s) : all of said aliphatic carboxylic acid(s) is from 0:100 to 50:50.

41. Treated mineral filler product according to claim 40, **characterised in that** it features a total volatiles between 25 and 280°C of less than 0.25 %, and preferably of less than 0.23 % by mass, e.g. of from 0.04 to 0.21 % by mass, preferably from 0.08 to 0.15 % by mass, more preferably from 0.1 to 0.12 % by mass.

42. Treated mineral filler product according to claim 40 or 41, **characterised in that** it features a volatile onset temperature of greater than or equal to 270°C, preferably greater than or equal to 290°C, most preferably of greater than or equal to 300°C.

43. Treated mineral filler product according to any of claims 40 to 43, **characterised in that** it is more hydrophobic than the same mineral filler having the same treatment layer but wherein the aliphatic carboxylic acid(s) are entirely

replaced with a corresponding aliphatic carboxylic acid alkali or earth alkali metal salt(s).

44. Use of a product according to any of claims 1 to 20 or 40 to 43 in a process of mixing and/or extruding and/or compounding and/or blow moulding with plastic materials.

45. The use according to claim 44,
characterised in that the plastic materials comprise polyolefins or thermoplastics such as polyethylenes (PE), polypropylenes (PP) and/or polyurethanes (PU).

46. Films comprising the products of any of claims 1 to 20 or 40 to 43.

47. The films according to claim 46,
characterised in that they are selected from the group comprising stretched and/or oriented films, and preferably breathable films, or extrusion coating films.

Figure 1

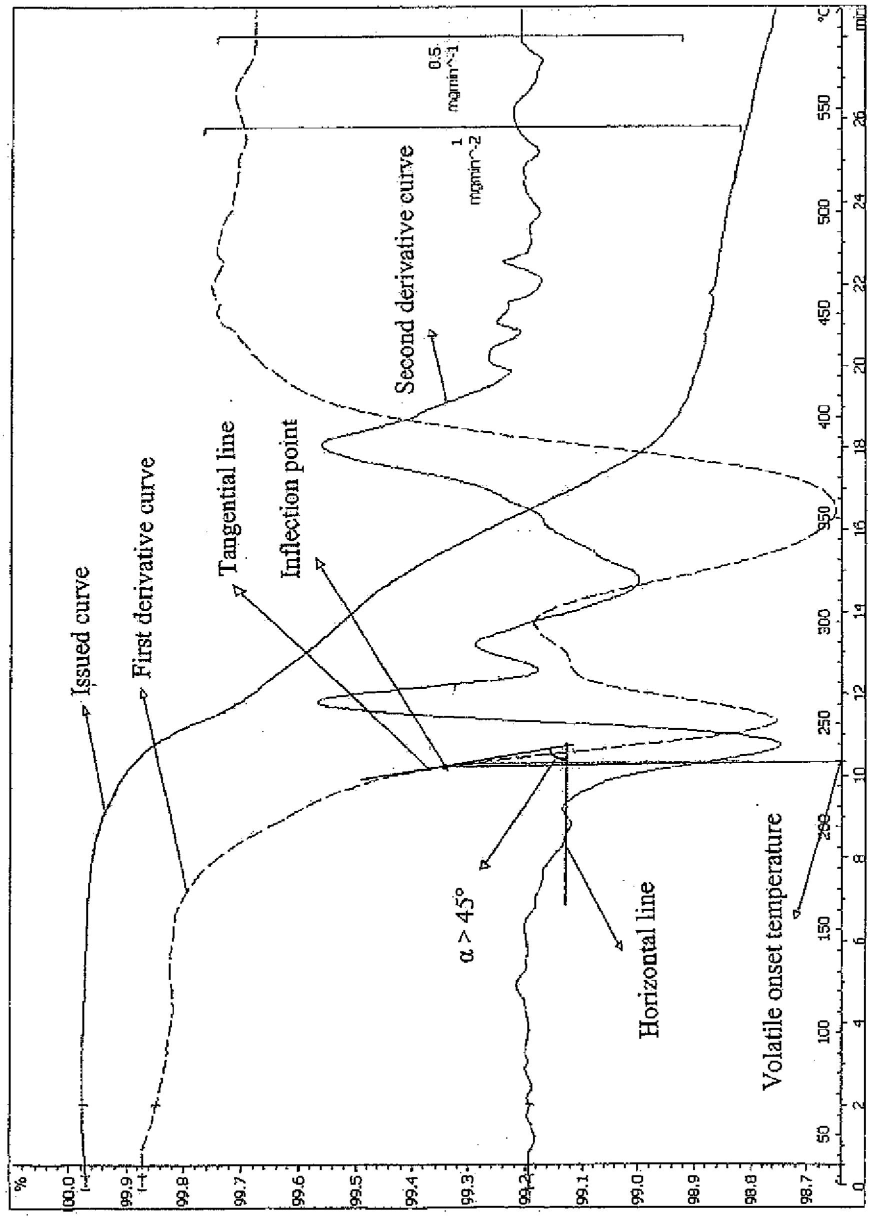
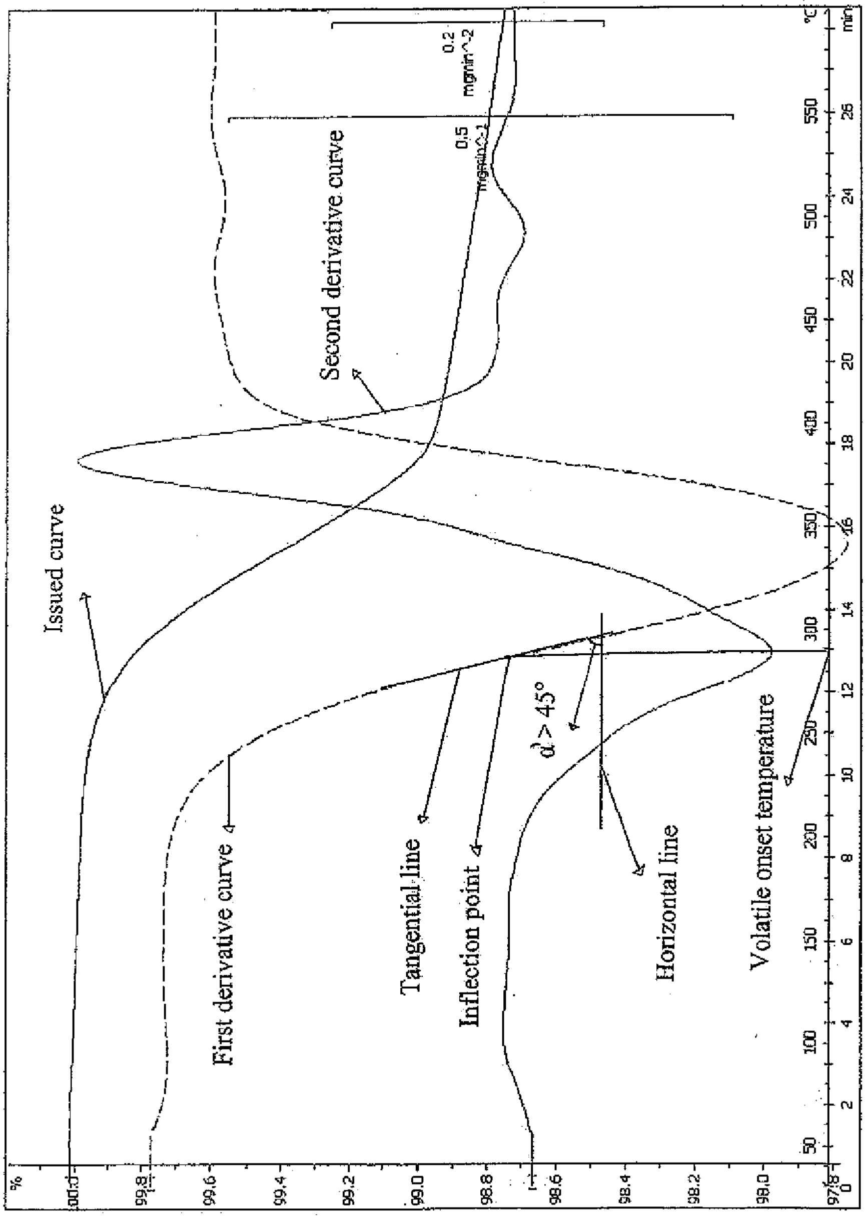

Issued curve
First derivative curve
Tangential line
Inflection point
Second derivative curve
α > 45°
Horizontal line
Volatile onset temperature
0.5 mgmin^-1
1 mgmin^-2
%
100.0
99.9
99.8
99.7
99.6
99.5
99.4
99.3
99.2
99.1
99.0
98.9
98.8
98.7
50
100
150
200
250
300
350
400
450
500
550
°C
0
2
4
6
8
10
12
14
16
18
20
22
24
26
min

Figure 2


Issued curve
Second derivative curve
First derivative curve
Tangential line
Inflection point
Horizontal line
Volatile onset temperature
α > 45°
0.2 mgmin^-2
0.5 mgmin^-1
%
100.0
99.8
99.6
99.4
99.2
99.0
98.8
98.6
98.4
98.2
98.0
97.8
°C
50
100
150
200
250
300
350
400
450
500
550
min
0
2
4
6
8
10
12
14
16
18
20
22
24
26

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2009/060741

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08K9/00

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)
C08K

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 practical, search terms used)

EPO-Internal

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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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- "E" earlier document but published on or after the international filing date
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- "O" document referring to an oral disclosure, use, exhibition or other means
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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
23 September 2009	01/10/2009

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Glomm, Bernhard

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2009/060741

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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