in US 5,475,123 (1995) naming Bos as inventor, which is hereinafter called “unmodified ZMG”. A second form of ZMG is also known from the Patent literature – US 7,074,949 also naming Bos as inventor (2006). In the method of the 2006 patent, zinc monoglycerolate is synthesized, either directly or indirectly, from hydrozincite ($Zn_5(CO_3)_2(OH)_6$). Hence, this material is referred to hereinafter as “hydrozincite-ZMG”.

However, over time there are increased pressures on costs in polymer production, and it would be advantageous to develop new or modified nucleating agents that provide an even more favorable balance between the cost of the agent at the required loading levels and cost savings obtained during manufacturing of polymer products containing the agent at that loading level.

It is an object of the embodiments disclosed herein to provide a new or modified nucleating agent with an improved nucleating effect at low loadings, polymer compositions containing the nucleating agent and an associated method for the production of the nucleating agent.

SUMMARY

We have now found that we are able to produce a modified form of zinc monoglycerolate that provides an improved nucleating effect, particularly when compared to unmodified zinc monoglycerolate. The new form of zinc monoglycerolate is hereafter named “GMS-ZMG” (named after the glycerol monostearate (GMS) modifier predominately used), and provides a better nucleating effect than that achieved with more than twice the amount of unmodified zinc monoglycerolate. We produce this product (GMS-ZMG) using a new process of preparation.

According to one embodiment, there is provided a zinc monoglycerolate in the form of agglomerates of crystallites, wherein the crystallite size based on the average coherence domain length is not more than 30nm in the <100> direction (i.e. along the <100> zone axis; hereafter often referred to as the <100> direction), and not more than 60nm in the <011> direction, as determined by the Scherrer equation via powder X-ray diffraction. The product also suitably has an aspect ratio (measured or computed as <100>/<011> coherent domain lengths) of less than 0.65, for example less than 0.60 or less than 0.56. The aspect ratio in the preferred embodiments is less than 0.44.

In an aspect, there is provided a zinc monoglycerolate in the form of agglomerates of crystallites, wherein the crystallite size based on the average coherence domain length is less than 30nm in the <100> direction (size along the <100> zone axis), and less than 60nm in the <011> direction (size along the <100> zone axis), as determined by the Scherrer equation via powder X-ray diffraction; and the aspect ratio computed by <100>/<011> coherent domain lengths is less than 0.65, preferably less than 0.56.

Through the process described herein, the applicant has produced a form of zinc monoglycerolate that contains an incorporated modifier (e.g. GMS-ZMG), which results in the formation of agglomerates of crystallites, where the crystallites are extremely small. The agglomerates contain multiple crystallites, and are consequently significantly larger than the individual crystallites

The applicant has conducted considerable research into ways to modify the zinc monoglycerolate in order to improve the efficiency of crystallization, through increasing the crystallization temperature and/or speeding up the time period to complete crystallization of the polymer.

The applicant achieved this result following many years of effort, during which a range of attempts were made to reduce the crystallite size of zinc monoglycerolate

The applicant has now successfully achieved small crystal size of the zinc monoglycerolate through the use of a modifier and through the control of production conditions of the zinc monoglycerolate. The key innovation, as compared to prior art, is the ability to use the readily commercially available and relatively inexpensive “indirect or French process” zinc oxide (e.g. as obtainable from Umicore Zinc Chemicals) as the starting point, as opposed to hydrozincite (as described in US 7,074,949). For a description of the “indirect or French process” for zinc oxide production, see Moezzi, A., McDonagh, A. M., & Cortie, M. B. (2012) Zinc oxide particles: Synthesis, properties and applications. *Chemical Engineering Journal*, *185*, 1-22. The modifier does not alter the fundamental chemical structure of zinc monoglycerolate.

According to another aspect, there is provided a process for the preparation of zinc monoglycerolate, comprising reacting a zinc compound, which is different from hydrozincite (i.e. it is a zinc compound other than hydrozincite, and is not derived from hydrozincite), with glycerol and a modifier, in the presence of an acid catalyst, wherein:
- the zinc compound is selected from the group consisting of zinc oxide, zinc carbonate, zinc hydroxide, zinc carboxylate and combinations thereof, or/and
- the reaction is conducted in the presence of less than 10% by weight water, based on the total weight of the reaction composition.

In preferred embodiments, both conditions are used in the process.

The present application also describes preferred temperature conditions for the production of zinc monoglycerolate from zinc oxide and glycerol, in the presence of an acid catalyst and a modifier, to control the crystallite and agglomerate size of the product.

In another aspect, there is provided a zinc monoglycerolate obtainable by the process as defined above.

By producing zinc monoglycerolate in the specified modified form described above, the applicant has been able to produce polymer compositions containing the zinc monoglycerolate (nu

cleating agent) (GMS-ZMG), in which the polymer compositions have a relatively high crystallization temperature at a low loading of the zinc monoglycerolate.

Thus, according to a further aspect, there is provided a composition comprising an organic polymer and the zinc monoglycerolate described above. The zinc glycerolate may be present in an amount of from 0.01% to 20% by weight of the polymeric composition.

According to another embodiment, there is provided a composition comprising an organic polymer and a nucleating agent comprising zinc monoglycerolate, wherein the zinc monoglycerolate has a crystallite size based on the average coherence domain length of not more than 30nm in the <100> direction, and not more than 60nm in the <011> direction, as determined by the Scherrer equation via powder X-ray diffraction; and the ratio of the above coherent domain lengths (which may be referred to as the aspect ratio) is less than 0.65, for example less than 0.60 or less than 0.56, preferably less than 0.44. Having an aspect ratio within this range indicates that the stack height of the zinc glycerol platelets is smaller than their sheet length.

In another aspect, there is provided a use of the zinc monoglycerolate as defined above to increase the crystallization temperature or the mechanical properties, preferably the E-modulus or yield strength, of an organic polymer.

In another aspect, there is provided a use of the zinc monoglycerolate as defined above as nucleating agent in an organic polymer.

The present application provides for the use of the zinc monoglycerolate of the type described above (GMS-ZMG) as a nucleating agent. Also provided is the use of the zinc monoglycerolate of the type described above (GMS-ZMG) to improve (increase) the crystallisation temperature of an organic polymer. This may be by way of increasing the crystallisation temperature of an organic polymer at low loading, e.g. 400ppm or less.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows the XRD pattern of modified zinc monoglycerolate according to the embodiment of the present invention (GMS-ZMG). Also shown is the XRD pattern of unmodified ZMG. The X-axis shows 2θ (in degrees), and the Y-axis shows the counts (peak intensity). The peak at 2θ = 10.9° is the (100) peak of zinc monoglycerolate. Both XRD patterns have been normalized such that the (100) peak has the same intensity. The (100) peak of GMS-ZMG is broader than in unmodified ZMG, i.e. GMS-ZMG crystallites are smaller.
Figures 2a and 2b show the SEM images of unmodified ZMG (Figure 2a) and modified ZMG (Figure 2b) of an embodiment of the invention. The scale bar in each image is 1μm.

Figures 3a, 3b and 4 are additional SEM images for the modified zinc monoglycerolate. The scale bar in each image is 100nm.

Figure 5 shows the XRD pattern of hydrozincite-ZMG (comparative example 1), compared with XRD plot of GMS-ZMG (Example 1). The X-axis is the 2θ axis (in degrees), and the Y-axis is