The present invention relates to a flame retardant quality magnesium hydroxide having a BET surface area of less than approximately 10 m² per gram (uncoated), an average particle size of approximately 0.5 to 10.0 micron, and less than approximately 0.5 % of particles over 10 micron (average particle size measured by laserlight scattering method), and a substantial number of crystals which are generally ovoid in cross section. A process for producing a flame retardant quality magnesium hydroxide is also described.
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MAGNESIUM PROCESS

The present invention relates to a flame retardant magnesium hydroxide and to a process for preparing same.

Magnesium hydroxide is a staple industrial chemical which is used in a wide variety of areas. Magnesium hydroxide finds particular application as a component in thermoplastic synthetic resins to impart flame retardancy. However, the incorporation into thermoplastic synthetic resins can adversely effect the physical properties of the resins, in particular impact strength and elongation at break.

It is known in the prior art that the elongation at break and other properties of such thermoplastic resins may be improved by utilising magnesium hydroxide which is characterised by specific strain, crystallite size and surface area characteristics ("the desired type"). Production of this type of magnesium hydroxide is, for example, described in United Kingdom Patent 1,514,081 to Kyowa Chemical Industry Co. Ltd. The process of preparing such a magnesium hydroxide product requires that a magnesium salt solution is treated with base in substoichiometric quantities at low temperatures (eg. less than 60°C) to precipitate a basic salt. The basic salt is then recrystallised in its mother liquor under fairly severe conditions (eg. 145°C to 200°C). Whilst such a process is effective, the process is very inefficient and conversion rates of magnesium are low, eg. of the order of 50 to 70%. Low magnesium recovery may lead to poor process economics and may also affect the subsequent disposal of the resulting ammoniacal salt solutions. Moreover, the magnesium hydroxide formed by the process contains crystals that appear to be flat and hexagonal in shape.

An alternative process for preparing magnesium hydroxide has been disclosed in Czech Patent 275,256, 1990, to RIIC. The RIIC patent describes a process for preparing magnesium hydroxide wherein a solution containing magnesium and calcium nitrates is treated with an excess of ammonia (eg. of 1.2 to 2.5 the stoichiometric amount) to generate a precipitate. It appears that no basic salt is generated, and autoclave recrystallisation is conducted under more mild conditions (eg. 130°C to 160°C for 10 to 90 minutes). It is suggested that the
ability to recrystallise under these conditions is due to the presence of calcium and ammonium nitrates and free ammonia. However, the magnesium hydroxide product formed is not of the desired type, and thermoplastic resins into which it is incorporated do not demonstrate a high elongation at break. The process also suffers from difficulties in purification, due to the large concentration of calcium present (eg. 5 to 100 g/l). Moreover, the by-product ammonium nitrate formed cannot be used (eg. as a fertiliser or explosive) without further purification to remove calcium.

It is accordingly an object of the present invention to overcome, or at least alleviate, one or more of the difficulties relating to the prior art.

Accordingly, in a first aspect of the present invention there is provided a flame retardant magnesium hydroxide having

- a BET surface area of less than approximately 10 m² per gram (uncoated), preferably less than approximately 6 m² per gram;
- an average particle size of approximately 0.5 to 10.0 micron, preferably approximately 0.5 to 5.0 micron, and less than approximately 0.5% of particles over 10 micron (average particle size measured by laserlight scattering method); and
- a substantial number of crystals which are generally ovoid in cross section.

Surprisingly the magnesium hydroxide according to the present invention contains a substantial number of crystals having a novel shape that is generally ovoid in cross section.

In a preferred embodiment there is provided a flame retardant magnesium hydroxide containing a substantial number of crystals being generally ovoid in cross section with a low BET specific surface area less than approximately 10 m²/g, a median particle size of approximately 0.5 to 10.0 micron and an oil absorption less than approximately 70 mL/100g, preferably less than approximately 60 mL/100g, most preferably less than approximately 50 mL/100g (oil absorption measured under International Standard ISO 787/5-1980(E)).

In a further preferred aspect of the present invention, the magnesium hydroxide may be coated with a surface active agent, preferably an anionic surfactant. A fatty acid surface active agent is preferred.
The anionic surface active agent may be selected from the group consisting of stearic acid, oleic acid, lauric acid, palmitic acid, sodium stearate, potassium behenate, sodium montanate, potassium stearate, sodium oleate, potassium oleate, sodium palmitate, potassium palmitate, sodium laurate, potassium laurate, sodium dilauryl benzenesulfonate, potassium octadecylsulfate, sodium laurylsulfonate, disodium 2-sulfoethyl α-sulfostearate and ammonium salts of fatty acids, for example ammonium stearate. These anionic surfactants may be used either alone or as mixtures of two or more.

Ammonium stearate and sodium stearate are particularly preferred coating agents.

The magnesium hydroxide product may be incorporated into a polymeric composition in order to minimise the loss of useful properties including tensile elongation and tensile strength. Accordingly in a further preferred aspect there is provided a polymeric composition including a thermoplastic polymer, thermoset or elastomer; and a magnesium hydroxide component containing a substantial number of crystals being generally ovoid in cross section with a low BET specific surface area less than 10 m²/g, a median particle size of approximately 0.5 to 10.0 micron and preferably an oil absorption less than approximately 70 mL/100 g (oil absorption measured under International Standard ISO 787/5-1980(E)).

The thermoplastic polymer may be a thermoplastic synthetic resin. The thermoplastic synthetic resin may be selected from the group consisting of homo- or co-polymers of styrene, homo- or co-polymers of propylene, homo- or co-polymers of olefins including ethylene, vinyl resins, polyester resins, polycarbonate resins, nylon resins, acetate resins, acetal resins and blends thereof. A co-polymer of ethylene, such as ethylene vinyl acetate (EVA) is preferred.

The thermoset may be selected from, for example, unsaturated polyester resins, epoxy resins, acrylic resins and blends thereof.

The elastomer may be selected from, for example, styrene-butadiene rubber (SBR), ethylene-propylene (EP), ethylene-propylene diene monomer (EPDM) and blends thereof.
Various conventional additives may further be incorporated in the polymeric compositions in accordance with this invention.

Examples of these additives are coloring agents (organic and inorganic pigments) such as isoindolinone, cobalt aluminate, carbon black, or cadmium sulfide; other fillers such as calcium carbonate, alumina, zinc oxide or talc; antioxidants such as 2,6-di-t-butyl-4-methylphenol, 2,2'-methylenebis (4-methyl-6-t-butylphenol), dilauryl thiodipropionate or tridecyl phosphite; ultraviolet absorbers such as 2-hydroxy-4-methoxy benzophenone, 2(2'-hydroxy-5-methylphenyl)benzotriazole, 2-ethylhexyl-2-cyano-3,3-diphenyl acrylate, phenyl salicylate or nickelbisoctyl phenyl sulfide; plasticizers such as di-2-ethyl hexyl phthalate, di-n-butyl phthalate, butyl stearate, or epoxidized soybean oil; and lubricants such as zinc stearate, calcium, aluminium and other metal soaps, or polyethylene wax.

The polymeric composition may be provided in the form of polymeric articles, for example melt-shaped articles.

The magnesium hydroxide component may be a coated magnesium hydroxide. In this embodiment, a thermoplastic EVA product may exhibit a high elongation at break, eg. of greater than approximately 200%, preferably greater than approximately 500% and a high limiting oxygen index, eg. of greater than approximately 35% v/v, preferably greater than approximately 40% v/v.

The polymeric articles may take the form of cable sheathing or insulation materials.

In a preferred aspect of the present invention there is provided a process for preparing a flame retardant quality magnesium hydroxide which includes

providing

a magnesium chloride solution; and

a source of ammonia;

adding a stoichiometric excess of ammonia to the solution to form a magnesium hydroxide precipitate; and

subjecting the magnesium hydroxide to a hydrothermal recrystallisation.

It has been found that by utilising inter alia the process described above a magnesium hydroxide product with a high elongation at break may be produced
in high yield, eg. approximately 80% or greater (based on magnesium). The magnesium hydroxide product may in turn be of flame retardant quality.

Surprisingly, applicant has found that the magnesium hydroxide produced according to inter alia the process of the present invention contains a substantial number of crystals having a novel shape that is generally ovoid in cross section.

The magnesium chloride solution preferably contains from approximately 20-130 g/l Mg, more preferably from approximately 30-120 g/l Mg.

In a further aspect of the present invention the process may include the preliminary step of

providing

a magnesium-containing material; and

a source of inorganic acid; and

treating the magnesium-containing material with the acid to form magnesium chloride.

The inorganic acid is preferably hydrochloric acid. The hydrochloric acid may be provided in the form of an aqueous solution of approximately 20 to 35% by weight hydrochloric acid.

The magnesium-containing material may be of any suitable type. The magnesium-containing material may be a magnesium-containing ore. A magnesite or dolomite ore may be used. A magnesite-feed material such as macrorystalline magnesite ore or cryptocrystalline magnesite ore may be used. By-product magnesium-containing materials may be used. By-product materials, containing Mg(OH)_2 and/or MgO for example, may be used. A magnesite ore or magnesium oxide derived from magnesite ore is preferred.

Where the starting material contains MgO, it may be leached with ammonium chloride as an alternative to the acid leach referred to above.

Accordingly, in a alternative preferred aspect of the present invention the process may include the preliminary step of

providing

a magnesium oxide-containing material; and

ammonium chloride; and

leaching the magnesium oxide-containing material with the ammonium
chloride to form magnesium chloride.

The leaching step may be conducted at elevated temperature, e.g. from approximately 60°C to 120°C, preferably from approximately 80°C to 100°C. The leaching step may be assisted by the injection of steam.

Ammonia generated during the leaching step may be utilised to form the ammonium hydroxide utilised in the later precipitation. For example the ammonia may be mixed with water in a suitable absorption device to form ammonium hydroxide.

Thus, the ammonium chloride by-product formed according to this aspect of the present invention may be recycled.

Accordingly, in a preferred aspect of the present invention, the process may further include the subsequent step of recycling at least a portion of the ammonium chloride so formed to the leach step.

Alternatively or in addition, the ammonium chloride formed as a by-product may be utilised industrially, eg. as a fertilizer ingredient. This is particularly so where a magnesite raw material is used, as the calcium content of the by-product is very low.

The leachate from the leaching process may be subjected to a purification step prior to further processing.

Accordingly in a preferred aspect the process according to the present invention further includes subjecting the leachate to a filtration step to remove residue.

In the process of the present invention, a starting solution low in detrimental impurities is particularly preferred. Whilst applicant does not wish to be restricted by theory, it is postulated that magnesite or MgO derived from magnesite is particularly useful in making such a solution as it is low in detrimental impurities and the resulting solution is readily purified. Magnesium oxide is preferred to magnesite as the calcination step involved in its production may remove organic matter which may inhibit the reaction and also adversely affect product colour.

Accordingly, in a preferred aspect of the present invention, the magnesium chloride solution has a low organic content, a low transition element content
and/or is low in sulphur and/or boron compounds.

Where the magnesium-containing material is a magnesite, the magnesite-feed material may be calcined in any known manner. The magnesite-feed material may be heated at temperatures above approximately 650°C, preferably in the range of approximately 800 to 1000°C under calcining conditions. The heating may continue for approximately 1 to 8 hours, preferably approximately 3 to 5 hours.

The magnesium-containing material may be subjected to suitable crushing and/or grinding steps prior to salt solution formation.

The magnesium chloride solution so formed may be buffered to an approximately neutral pH value before further processing including purification as discussed below.

In a preferred aspect of the present invention a relatively pure magnesium chloride solution is used. By the term "relatively pure" as used herein we mean that trace impurities in the solution are at a level such that they do not substantially interfere with the process. Preferably, the relatively pure magnesium chloride solution contains less than approximately 100 ppm, more preferably less than approximately 10 ppm, and most preferably less than approximately 1 ppm transitional element impurities. The transitional element impurities may be metal impurities. In a particularly preferred form, the magnesium chloride solution contains less than approximately 50 ppm, more preferably less than approximately 10 ppm, most preferably less than approximately 1 ppm Fe and/or Mn. Alternatively or in addition the relatively pure magnesium chloride solution preferably contains less than approximately 1000 ppm, more preferably less than approximately 500 ppm, most preferably less then 100 ppm sulphur impurities. Alternatively or in addition the relatively pure magnesium chloride solution preferably contains less than approximately 200 ppm, more preferably less than approximately 100 ppm, most preferably less than approximately 50 ppm boron impurities. Applicant has found that the level of Ca impurities does not substantially affect the efficiency of the process.

Depending on the starting material used, it is preferred in certain circumstances to subject the magnesium chloride solution to a purification step to
remove trace impurities. The trace impurities may be transition element impurities. The transition element impurities may be metal impurities, for example Fe, Ni, Cu and/or Mn.

Accordingly, in a preferred aspect, the process of the present invention includes a preliminary purification step which includes:

- providing a magnesium chloride solution;
- a source of base; and
- a source of oxidant;

adding the source of base to the magnesium chloride solution to raise the pH thereof;

- treating the magnesium chloride solution with the source of oxidant to oxidise trace impurities therein; and
- removing trace impurities as their insoluble hydroxides.

The source of base may be of any suitable type which will raise the pH of the magnesium salt solution. For example, ammonium hydroxide (NH₄OH) and magnesium oxide (MgO) have been found to be suitable. Caustic magnesium oxide is preferred.

Preferably, the pH of the magnesium salt solution is raised to approximately pH 4-7, more preferably approximately pH 5-6.

The source of oxidant may be of any suitable type. For example peroxide (H₂O₂) or chlorine (Cl₂) may be used. Chlorine gas (Cl₂) is preferred.

The trace impurities may be removed as their insoluble hydroxides by filtration.

In the precipitation step according to the present invention, the purified magnesium chloride solution is then treated with a stoichiometric excess of ammonia. The ammonia may be provided in the form of ammonium hydroxide.

The amount of ammonia used may be such that the ammonia is present in the range of approximately 100 to 250% of the stoichiometric amount, preferably 150 to 250%, more preferably approximately 175 to 200% of the stoichiometric amount.
It is preferred to conduct the precipitation step at a temperature between approximately 30°C and approximately 80°C.

The ammonia addition time is preferably from approximately 2 seconds to approximately 2 hours, more preferably from approximately 10 seconds to approximately 1 hour.

The hydrothermal recrystallisation step may be conducted at temperatures of approximately 130°C to 300°C or higher. The hydrothermal recrystallisation step is typically conducted in an autoclave which may be operated batchwise or continuously. In this case the hydrothermal recrystallization is preferably conducted at temperatures of approximately 150°C to approximately 220°C, more preferably approximately 185°C to approximately 210°C. A tube reactor may also be used as a form of continuous autoclave. In this case the hydrothermal recrystallisation is preferably conducted at approximately 150°C to approximately 300°C. The hydrothermal recrystallisation may continue from approximately 1 minute to approximately 6 hours, preferably approximately 5 minutes to approximately 2 hours.

The magnesium hydroxide product so formed may be subjected to a purification step. The magnesium hydroxide product may be filtered and the cake washed with water to produce a purified magnesium hydroxide product.

In a preferred aspect of the present invention the process for preparing a purified hydroxide magnesium product may further include

subjecting the purified Mg(OH)₂ product to a drying step.

The drying step may be conducted utilising conventional techniques.

The drying may be conducted utilising a band dryer, pneumatic dryer, fluid bed dryer or the like or a combination thereof.

The Mg(OH)₂ product may be subjected to a calcining step to produce an MgO product. Calcining conditions may be similar to those used for magnesite treatment as discussed above.

In a further aspect of the present invention there is provided a flame retardant quality magnesium hydroxide whenever prepared according to the process as described above.
The present invention will now be more fully described with reference to the accompanying examples. It should be understood, however, that the description following is illustrative only and should not be taken in any way as a restriction on the generality of the invention described above.

Fig. 1 is a flow diagram illustrating a preferred form of one aspect of the process of the present invention.

Fig. 2 is a flow diagram illustrating a preferred form of a further aspect of the process of the present invention.

Figs. 3(a) to 3(d) are scanning electron micrographs showing magnesium hydroxide containing crystals produced according to Example 16.

Figs. 4(a) to 4(d) are scanning electron micrographs showing magnesium hydroxide containing crystals produced according to Example 15 (0.25h).

Figs. 5(a) to 5(d) are scanning electron micrographs showing magnesium hydroxide containing crystals produced according to Example 15 (2h).

**EXAMPLE 1**

Magnesium hydroxide was precipitated from a purified chloride solution obtained by HCl leaching of Kunwarara magnesite. Transition metal impurities Fe, Mn, Ni and Cu were removed from the leach solution by oxidation/neutralisation using chlorine gas as oxidant and caustic MgO as neutralising reagent. The purified liquor contained 107 g/L Mg, 2.8 g/L Ca and less than 1 mg/L each of Fe, Mn, Ni, Cu. The magnesium hydroxide precipitation was carried out batchwise in an un baffled 5 litre glass beaker. Agitation was by 6-blade turbine driven by an overhead stirrer motor. The temperature was controlled with a hotplate. The operating conditions were as follows:

- concentration of NH$_4$OH: 25% w/w
- NH$_4$OH addition: 1.8 x stoichiometric
- time of NH$_4$OH addition: 19 minutes
- precipitation temperature: 30°C

The slurry from the precipitation was hydrothermally treated in an autoclave at 185°C for 2 hours. Autoclaved magnesium hydroxide was filtered, washed and then dried at 110°C for 12 hours. The dry powder had the following characteristics:
BET specific surface area : 3.5 m²/g
oil absorption (linseed oil) : 50 mL/100 g

**EXAMPLES 2-7**

Magnesium hydroxide was obtained in the same manner as in Example 1 but for the conditions given in Table 1.

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**EXAMPLE 8**

Magnesium hydroxide was precipitated from a purified magnesium chloride solution obtained by leaching of Kunwarara magnesite with dilute HCl. The leach liquor was purified as described in Example 1. The purified solution contained 50.2 g/L Mg, 1.4 g/L Ca and less than 1 mg/L each of Fe, Mn, Ni, Cu. The magnesium hydroxide precipitation was carried out in an unbaffled 5 litre glass vessel. Agitation was by 6-blade turbine driven by an overhead stirrer motor. The temperature was controlled with a hotplate. The operating conditions were as follows:

- concentration of NH₄OH : 30% w/w
- NH₄OH addition : 1.8 x stoichiometric
- time of NH₄OH addition : 19 minutes
- precipitation temperature : 30°C

The slurry from the precipitation was hydrothermally treated in an autoclave at 185°C for 2 hours. The autoclaved product was filtered,
washed and then dried at 110°C for 12 hours. The dry powder had the following characteristics:

BET specific surface area : 1.9 m²/g
oil absorption (linseed oil) : 43.6 mL/100 g

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EXAMPLE 9

Magnesium hydroxide was precipitated from a purified chloride liquor obtained by HCl leaching of Chinese caustic magnesia containing 94.16% MgO, 1.4% CaO, 1.88% SiO₂, 0.77% Fe₂O₃, 0.22% Al₂O₃ and 0.12% SO₃. The leach liquor was purified as described in Example 1. The purified solution contained 121.7 g/L Mg, 7.4 g/L Ca and less than 1 mg/L each of Fe, Mn, Ni, Cu. The precipitation of magnesium hydroxide and the subsequent hydrothermal treatment of the precipitate slurry was performed as described in Example 8. The autoclave product was filtered, washed and then dried at 110°C for 12 hours. The dry powder had the following physical properties:

BET specific surface area : 3 m²/g
oil absorption (linseed oil) : 48.3 mL/100 g

EXAMPLE 10

Magnesium hydroxide was precipitated from a liquor obtained by leaching caustic MgO with an ammonium chloride (AC) recycle solution containing 20.7% w/w ammonium chloride, 3% w/w MgCl₂ and 1.5% w/w CaCl₂. The AC leach liquor contained 54.1 g/L Mg, 10.1 g/L Ca, <1mg/L Fe and Mn, 3.1 mg/L Ni, 3.5mg/L Cu. This solution was processed without further purification as described in Example 1. The dry autoclave product had the following physical properties.

BET specific surface area : 1.6 m²/g
oil absorption (linseed oil) : 39.7 mL/100 g

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EXAMPLE 11

Magnesium hydroxide was precipitated from a liquor obtained by leaching caustic MgO with an ammonium chloride (AC) recycle solution containing 18.3% w/w AC, 3% w/w MgCl₂ and 1.5% w/w CaCl₂. The AC leach liquor contained 57.8 g/L Mg, 7.9 g/L Ca, <1 mg/L Fe and Mn, 4.8 mg/L Ni, 4.1 mg/L Cu. This solution was processed without further purification as described in Example 1. The product from the autoclave was filtered and washed. The wet filter cake was then reslurried in deionised water to a strength of 10% solids. The slurry was heated to 60°C 2.5% w/w (magnesium hydroxide) of stearic acid and the stoichiometric amount of NH₄OH (30 w/w) were added. The slurry was left agitated at 60°C for 1/2 hour. The stearate coated material was then filtered and dried at 110°C for 12 hours. The dry coated powder had the following physical characteristics:

- BET specific surface area : 0.9 m²/g
- median particle size : 3.94 μm (15 minutes ultrasonics)
- oil absorption (linseed oil) : 42.5 mL/100 g

The stearate coated magnesium hydroxide was compounded in a two-roll mill compounder with ethylene vinyl acetate (EVA) 1020VN5 grade at a loading of 60% w/w. The physical properties determined on samples cut from compression moulded sheets of the compound were as follows:

- tensile strength : 8.6 MPa
- elongation at break : 681%
- oxygen index : >40% w/v

EXAMPLE 12

Magnesium hydroxide was precipitated from a liquor obtained by leaching caustic MgO with the composition given in Example 9 with an ammonium chloride (AC) recycle solution containing 21.9% w/w AC, 1.5% w/w MgCl₂ and 2.1% w/w CaCl₂. The AC leach liquor contained 37.5 g/L Mg, 5 g/L Ca, <1 mg/L Fe, Mn and Ni, 1.1 mg/L Cu. This solution was processed without further purification as described in Example 1. Wet, washed product from the autoclave was coated with 2.5% w/w ammonium stearate in the same manner as in Example 11. The stearate coated material was then filtered and dried at 110°C for 12 hours. The
dry coated powder had the following physical characteristics:

- BET specific surface area: 1.4 m$^2$/g
- median particle size: 2.75 μm (15 minutes ultrasonics)
- oil absorption (linseed oil): 33.9 mL/100 g

The stearate coated magnesium hydroxide was compounded with EVA as described in Example 11. The physical properties determined on samples cut from compression moulded sheets of the compound were as follows:

- tensile strength: 7.8 MPa
- elongation at break: 675%
- oxygen index: >40% v/v

**EXAMPLE 13**

Magnesium hydroxide was precipitated from a purified chloride solution obtained by leaching of Kunwarara magnesite with dilute HCl. The purified liquor contained 76.8 g/L Mg, 1.7 g/L Ca, less than 1 mg/L each of Fe, Mn, Ni, Cu. This solution was processed as described in Example 1. Wet, washed autoclave product was coated with 2.5% w/w ammonium stearate in the same manner as in Example 11. The coated material was filtered and dried at 110°C for 12 hours. The dry coated product had the following physical characteristics:

- BET specific surface area: 2.6 m$^2$/g
- median particle size: 1.47 μm (15 minutes ultrasonics)
- oil absorption (linseed oil): 42.8 mL/100 g

The stearate coated product was compounded with EVA as described in Example 11. The physical properties determined on samples cut from compression moulded sheets of the compound were as follows:

- tensile strength: 8.5 MPa
- elongation at break: 711%
- oxygen index: >40% v/v
EXAMPLE 14

Magnesium hydroxide was precipitated from a liquor obtained by leaching caustic MgO with an ammonium chloride (AC) recycle solution containing 11.8% w/w AC, 9.5% w/w MgCl₂ and 1.9% w/w CaCl₂. The AC leach liquor contained 50.8 g/L Mg, 10.5 g/L Ca, <1 mg/L Fe and Mn, 2 mg/L Ni, 2.5 mg/L Cu. This solution was processed without further purification as described in Example 1 except that the precipitation temperature was 60°C. Wet, washed product from the autoclave was coated with 2.5% w/w ammonium stearate in the same manner as in Example 11. The stearate coated product was filtered and dried at 110°C for 12 hours. The dry coated powder had the following physical characteristics:

- BET specific surface area: 2.7 m²/g
- Median particle size: 1.99 μm (15 minutes ultrasonics)
- Oil absorption (linseed oil): 41.9 mL/100 g

The stearate coated magnesium hydroxide was compounded with EVA as described in Example 11. The physical properties determined on samples cut from compression moulded sheets of the compound were as follows:

- Tensile strength: 7.3 MPa
- Elongation at break: 553%
- Oxygen index: >40% v/v

EXAMPLE 15

Magnesium hydroxide was precipitated from a magnesium chloride solution containing 115.5 g/L Mg according to the conditions presented in Example 1. The slurry from the precipitation was hydrothermally treated in an autoclave at 150°C for 0.25 and 2 hours. The products from the autoclave were filtered, washed and dried at 110°C for 12 hours. The dry powders had the following characteristics:
The ovoid magnesium hydroxide crystals produced are shown in Figs. 4(a) to 4(d) (0.25h) and Figs. 5(a) to 5(d) (2h).

**EXAMPLE 16**

Magnesium hydroxide was precipitated from a chloride solution containing 81.5 g/L Mg, 0.05 g/L Ca, 1.7 mg/L Ni and < 1mg/L Fe, Mn. The precipitation was carried out batchwise in a 5 litre baffled SS vessel. Agitation was by 6-blade turbine driven by an overhead stirrer motor. The temperature was controlled by hotplate. The operating conditions were as follows:

<table>
<thead>
<tr>
<th>Concentration of NH₄OH</th>
<th>30% w/w</th>
</tr>
</thead>
<tbody>
<tr>
<td>NH₄OH addition</td>
<td>1.8 x stoichiometric</td>
</tr>
<tr>
<td>Time of NH₄OH addition</td>
<td>19 minutes</td>
</tr>
<tr>
<td>Precipitation temperature</td>
<td>25°C</td>
</tr>
</tbody>
</table>

After completion of the NH₄OH addition the primary slurry was transferred into an autoclave, heated to the target temperature of 185°C and hydrothermally treated at this temperature for 2 hours. The autoclaved magnesium hydroxide was then filtered and washed.

The wet washed filter cake was reslurried in de-ionised water to a strength of 10% solids. The slurry was heated to 60°C. 2.5% w/w (magnesium hydroxide) of stearic acid and the stoichiometric amount of NH₄OH (30% w/w) were added. The slurry was left agitated at 60°C for ½ hour. The stearate coated material was then filtered and dried at 110°C for 72 hours. The dry coated powder had the following physical characteristics:

<table>
<thead>
<tr>
<th>BET specific surface area</th>
<th>1.9 m²/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Median particle size</td>
<td>2.7 µm (15 minutes ultrasonics)</td>
</tr>
<tr>
<td>Oil absorption (linseed oil)</td>
<td>41 mL/100g</td>
</tr>
</tbody>
</table>
The stearate coated magnesium hydroxide was compounded in a two-roll mill compounder with ethylene vinyl acetate (EVA) 1020VN5 grade at a loading of 60% w/w. The physical properties determined on samples cut from compression moulded sheets of the compound were as follows:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>8.2 MPa</td>
</tr>
<tr>
<td>Elongation at break</td>
<td>721%</td>
</tr>
<tr>
<td>Oxygen index</td>
<td>&gt;40% v/v</td>
</tr>
</tbody>
</table>

The ovoid magnesium hydroxide crystals produced are shown in Figs. 3(a) to 3(c).

Finally, it is to be understood that various other modifications and/or alterations may be made without departing from the spirit of the present invention as outlined herein.
CLAIMS:

1. A flame retardant quality magnesium hydroxide having a BET surface area of less than approximately 10 m² per gram (uncoated), an average particle size of approximately 0.5 to 10.0 micron, and less than approximately 0.5% of particles over 10 micron (average particle size measured by laserlight scattering method), and a substantial number of crystals which are generally ovoid in cross section.

2. A magnesium hydroxide according to claim 1 having oil absorption less than approximately 70 mL/100 g (oil absorption measured under International Standard ISO 787/5-1980(E)).

3. A magnesium hydroxide according to claim 1 wherein the BET surface area is less than approximately 6 m²/g and the average particle size is approximately 0.5 to 5.0 micron.

4. A coated magnesium hydroxide product including a flame retardant quality magnesium hydroxide containing crystals being generally ovoid in cross-section, with a low BET specific surface area less than approximately 10 m²/g, a median particle size of approximately 0.5 to 10.0 micron and an oil absorption less than approximately 70 mL/100 g (oil absorption measured under International Standard ISO 787/5-1980(E)) an anionic surface active agent selected from the group consisting of stearic acid, oleic acid, lauric acid, palmitic acid, sodium stearate, potassium behenate, sodium montanate, potassium stearate, sodium oleate, potassium oleate, sodium palmitate, potassium palmitate, sodium laurate, potassium laurate, sodium dilauryl benzenesulfonate, potassium octadecylsulfate, sodium laurylsulfonate, disodium 2-sulfoethyl α-sulfostearate and ammonium salts of fatty acids, and mixtures thereof coated on the magnesium hydroxide.
5. A polymeric composition including
   a thermoplastic polymer, thermoset or elastomer; and
   a magnesium hydroxide component containing a substantial number of
   crystals being generally ovoid in cross section with a low BET specific surface
   area less than 10 m²/g, a median particle size of approximately 0.5 to 10.0 micron
   and an oil absorption less than approximately 70 mL/100 g (oil absorption
   measured under International Standard ISO 787/5-1980(E)).

6. A polymeric composition according to claim 5 wherein the thermoplastic
   polymer is selected from the group consisting of homo- or co-polymers of styrene,
   homo- or co-polymers of propylene, homo- or co-polymers of olefins including
   ethylene, vinyl resins, polyester resins, polycarbonate resins, nylon resins,
   acetate resins, acetal resins and blends thereof; the thermoset is selected from
   the group consisting of unsaturated polyester resins, epoxy resins, acrylic resins
   and blends thereof; and the elastomer is selected from the group consisting of
   styrene-butadiene rubber (SBR), ethylene-propylene (EP), ethylene-propylene
diene monomer (EPDM) and blends thereof.

7. A polymeric composition according to claim 6 wherein the thermoplastic
   polymer is an ethylene vinyl acetate copolymer, and the polymeric composition
   exhibits a high elongation at break and a high limiting oxygen index.

8. A process for preparing a flame retardant quality magnesium hydroxide
   which includes
   providing
   a magnesium chloride solution; and
   a source of ammonia;
   adding a stoichiometric excess of ammonia to the solution to form a
   magnesium hydroxide precipitate; and
   subjecting the magnesium hydroxide to a hydrothermal recrystallisation.
9. A process according to claim 8 wherein when the magnesium chloride solution contains from approximately 20-130 g/l magnesium.

10. A process according to claim 8 wherein the process further includes a preliminary purification step which includes providing

   a magnesium chloride solution;

   a source of base; and

   a source of oxidant;

   adding the source of base to the magnesium chloride solution to raise the pH thereof;

   treating the magnesium chloride solution with the source of oxidant to oxidise trace impurities therein; and

   removing trace impurities as their insoluble hydroxides.

11. A process according to claim 10 wherein the source of base is caustic magnesium oxide and the pH is raised to approximately pH 4-7.

12. A process according to claim 10 wherein the oxidant is chlorine; and

   the trace impurities are removed as their insoluble hydroxides by filtration.

13. A process according to claim 8 wherein, in the precipitation step, ammonia is present in the range of approximately 100% to 250% of the stoichiometric amount and the precipitation step is conducted at a temperature between approximately 30°C and 80°C.

14. A process according to claim 13 wherein the ammonia addition time is from approximately 2 seconds to approximately 2 hours.

15. A process according to claim 13 wherein the hydrothermal recrystallisation step is conducted at temperatures of approximately 130°C to 300°C or higher and continues for from approximately 1 minute to approximately 6 hours.
16. A process according to claim 15 wherein the hydrothermal recrystallization is conducted in an autoclave at temperatures of approximately 150°C to 220°C.

17. A process according to claim 15 wherein the hydrothermal recrystallisation is conducted in a tube reactor at temperatures of approximately 150°C to 300°C.

18. A process according to claim 15 further including subjecting the magnesium product so formed to a purification step; and subjecting the purified magnesium hydroxide product to a drying step.

19. A process according to claim 18 wherein the purification step includes filtering the product and washing the filtered cake so formed with water to produce a purified magnesium hydroxide product.

20. A process according to claim 8 including the preliminary step of providing
   a magnesium-containing material; and
   a source of inorganic acid; and
   treating the magnesium-containing material with the acid to form magnesium chloride.

21. A process according to claim 20 wherein the inorganic acid is hydrochloric acid.

22. A process according to claim 21 wherein the hydrochloric acid is present in the form of an aqueous solution of approximately 20-35% by weight of hydrochloric acid.

23. A process according to claim 20 wherein magnesium containing material is a magnesite ore or magnesium oxide (MgO) derived from a magnesite ore, or a mixture thereof.
24. A process according to claim 8 including the preliminary step of providing
   a magnesium oxide-containing material, and
   ammonium chloride; and
   leaching the magnesium oxide-containing material with the ammonium chloride to form magnesium chloride.

25. A process according to claim 24 wherein the leaching step is conducted at a temperature of approximately 60°C to 120°C, and the leaching step is assisted by the injection of steam.

26. A process according to claim 24 wherein at least a portion of the ammonium chloride formed as a by-product is recycled to the leaching step.

27. A flame retardant quality magnesium hydroxide whenever prepared by a process according to any one of the preceding claims.

28. A flame retardant quality magnesium hydroxide substantially as hereinbefore described with reference to any one of the examples.
Fig. 1.

MAGNESITE (MgCO₃) → LEACH → PURIFICATION (pH = 5-6) → FILTRATION / WASH → RESIDUE TO DUMP

MgO → PURIFICATION (pH = 5-6) → MgCl₂ LIQUOR

Cl₂ → PURIFICATION (pH = 5-6) → Mg(OH)₂ SLURRY

EXCESS NH₄OH → PRECIPITATION Mg(OH)₂

Mg(OH)₂ SLURRY → HYDROTHERMAL TREATMENT (AUTOCLAVE) 185-210°C → FILTRATION / WASH

WATER → HYDROTHERMAL TREATMENT (AUTOCLAVE) 185-210°C → FILTRATION / WASH

BY-PRODUCT AMMONIUM CHLORIDE (AC) LIQUOR (20-30%) → DRYING

WATER → FILTRATION / WASH → Mg(OH)₂ PRODUCT

SUBSTITUTE SHEET (RULE 26)
Fig. 2.

MAGNESIUM-CONTAINING MATERIAL (EG. MgO)

WASH LIQUOR FROM CAKE WASHING

AC (NH₄Cl) LEACH (80-100°C)

NH₃

NH₃ ABSORPTION

HEAT

FILTRATION / WASH

WATER

PRECIPITATION Mg(OH)₂

Mg(OH)₂ SLURRY

HEAT

HYDROTHERMAL TREATMENT (AUTOCLAVE) 180-210°C

NH₄OH EXCESS

ENRICHED RESIDUE EG. HEAVY METALS ETC. TO DUMP

MAKE-UP NH₃

RECYCLE AC TO LEACH

WASH

FILTRATION AND WASH

EVAPORATION

DRYING

Mg(OH)₂ PRODUCT
Fig. 4(a)

Fig. 4(b)
Fig. 5(c)

Fig. 5(d)
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

Int Cl6: C01F 5/14, 5/20 C09K 21/02

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)

IPC: C01F 5114, 5/20, C09K 21/02

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

AU IPC as above

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>EP. A. 0568488 (DEFDED LTD) 23.4.93 (23 April 1993)</td>
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<td>Derwent Abstract Accession No. 92-147439/18 Class A60 E33 JP.A. 04089308 (TOYKUYAMA SODA K.K.) 23 March 1992 (23.03.92)</td>
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<td>Derwent Abstract Accession No. 8800660 (EISLER. J) 12 October 1990 (12.10.90)</td>
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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"&" document member of the same patent family

Date of the actual completion of the international search 31 January 1996

Date of mailing of the international search report 14 FEB 1996

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<td>X</td>
<td>EP.A.0214494 (ASAHI GLASS CO. LTD) 13 August 1986 (13.08.86)</td>
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END OF ANNEX