

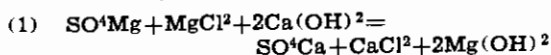
ALIEN PROPERTY CUSTODIAN

PROCESS FOR EXTRACTING MAGNESIA FROM WATER CONTAINING CONVERTIBLE MAGNESIUM SALTS, SUCH AS SEA-WATER, ETC.

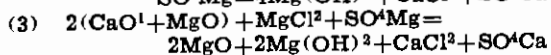
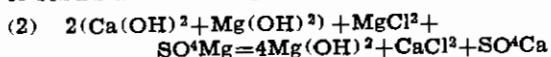
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No Drawing. Application filed December 20, 1939

Many processes have been proposed for the manufacture of magnesia hydroxide from water containing convertible magnesium salts in solution such as, for example, sea water, natural or artificial brines, etc. Generally milk of lime is used according to the well known reaction.



Calcined dolomites have also been used in milk or solid state according to reaction 2 or 3:



Reaction 3 is obtained when the dolomite has been overburnt so as to avoid hydration of MgO contained in the dolomite.

Unhappily, it is always difficult and often impossible or too expensive to secure lime stones or dolomites pure enough to work under these conditions, because the impurities from these raw materials remain in the recovered magnesia.

It is therefore necessary to locate the works only in favourable sites which limits very much the economical possibilities.

Further, the reactions in the solid state are slow and necessitate expensive working.

The process according to the present invention consists with getting reaction (1) from lime-water instead of milk of lime id est from a liquid reagent.

Under this new condition, all noxious impurities from the calcined lime-stone remain as insoluble residues, and it is possible to use even very impure raw materials and to choose therefore freely suitable manufacturing sites.

It seems however that the process according to the invention is subject to such drawbacks as to render its practical use impossible because lime has a very small solubility in water and huge volumes must therefore be used and also because it is well known by experts that precipitation of magnesium salts solutions by alkaline or alkaline-earth solutions give out precipitates which are very voluminous, difficult to filter and which retain such a quantity of water that they are practically of no possible use.

The inventor has found, by a careful study of these two difficulties, that the first one is not a real difficulty and that the second one can be met with success.

The first point is not a real difficulty because in fact the reaction is very rapid and the cost

of pumping and circulating the lyes is a very small item in the total cost price.

Further, the possibility exists to utilize mainly the residual waters from the process for making the necessary lime-water so that pumping be nearly limited to the magnesium salt waters (if local conditions render it useful or necessary).

The second point is solved by precipitating magnesia under special conditions discovered by the inventor which give out a compact and granular material, easy to manipulate and retaining only a normal amount of water.

In fact, if magnesia is precipitated by reaction (1) in a first batch, and if the precipitate, after being separated from the mother-lye, is used as a seed for a new batch and so on, always using the accumulated precipitate from the previous operations as a seed for the next operations, it has been found that (on condition that proper agitation is secured each time during the reaction) the precipitate gets more and more heavy and granular, can be decanted more and more easily and quickly, and retains decreasing quantities of water so that one is perfectly free to regulate, within proper limits, the physical properties of the precipitate. We will now explain the working out of the invention starting for example from sea water and limestone.

The sea water will first, eventually, be purified in known ways from the bicarbonate it contains. It may also be decanted and/or filtered.

From calcined lime-stone, lime-water will, on the other hand, be prepared in fresh water or in residual sea water coming out of the process. In this lime-water noxious impurities are insoluble and soluble impurities are immaterial as they are not precipitated, later on, during the process.

The mixture of the two liquids, sea water and lime-water, in proper proportions give reaction (1) which is practically immediately complete. An excess of one liquid over the other is of no great importance though it is generally better to be slightly short in lime-water. In order to avoid gelatinous precipitates, the reaction is carried as already explained in the presence of an agitated seeding material until the desired quality is obtained. This seeding material may be commercial $\text{Mg}(\text{OH})^2$ or $\text{Mg}(\text{OH})^2$ produced by the process. For example, suitable seeding precipitate (or a previous precipitate obtained from the process) will be mixed with the sea water or with the lime-stone or with both so that the reaction will take place in the presence of a milk of magnesia-seed well agitated.

It is preferred to mix the seed only to the lime

water and to add progressively sea-water under proper agitation to keep the seed in intimate suspension in the reacting liquid.

It is advantageous to carry the process in a continuous way in a reaction vessel coupled with a decanting tank (or other suitable separating device). In the reaction vessel, lime-water mixed with seeding precipitate, is received, together with sea water, properly dispersed and the liquid is continuously agitated to insure proper and quick mixture.

The proportion between the two entering liquids is easily calculated and controlled by known means.

From the reaction capacity the liquid and the suspended precipitate go together by suitable means (such as pumping or overflow) to the decanting-tank (or any suitable separating device) from which the deposited precipitate is pumped back and reinjected in the fresh lime-water entering the reaction capacity whilst the exhausted water overflows and is taken away.

Working goes on in this way and the precipitate is kept circulating in a closed circuit until its physical qualities are satisfactory. After this point is reached, one may regularly take out for disposal a proper fraction of the circulating precipitate.

It is also possible to work on the batch principle and to dispose of the whole of the precipitate and start again a new series of operations.

In working the process, if the sea water (or the brine containing CO^2) are not chemically treated, the precipitate will contain a small proportion of CO^2Ca which is not detrimental for certain purposes.

However it is generally preferred to treat the sea-water (or brine) in known way by addition of lime or lime-water to precipitate CO^2 as CO^2Ca before using it for magnesia production.

It is also possible to treat the sea water or brine by a strong acid such as for example ClH so as to turn all the carbonates and bicarbonates into soluble salts. Free CO^2 is then separated either by a vacuum or by injecting (or properly mixing with) a gas free from CO^2 (such as decarbonated air).

However a very simple method of manufacture consists with using raw solutions of sea water or brines and to dispose of the CO^2Ca which is precipitated with the magnesia by calcining the mixture and washing out the soluble CaO . In this case, if calcination has been carried at a temperature higher than 1200°C . magnesia does not hydrate to any extent during the washing operation and, after drying, anhydrous magnesia is recovered.

Generally it is preferred but not necessary to carry the different operations so that CO^2 from

the atmosphere does not react on the solutions. This may be conveniently realized by using a closed reaction capacity and carrying the decantation under a film of protecting oil.

In certain cases, it may be interesting to utilize dolomite stone for the manufacture of the lime-water used in the process. In this case, magnesia, mixed with the impurities of the raw material is recovered as a residue and this is a second quality stuff which may be used as such or after mixing with the high quality magnesia obtained from the process.

For the manufacture of lime-water, fresh water is preferred if proper supplies are at hand but the exhausted waters from the process may also be used, or mixtures of both. If the sea water or brines contain organic matter in such quantity as to hinder operations, it will be necessary to sterilize it.

As an example, we will now describe the manufacture from sea-water previously filtered on a sand filter.

The lime-water was prepared from fresh water at the start and as seen as the circuit was established, it was obtained from the exhausted sea-water coming out from the reaction. The lime-water was decanted carefully but not filtered.

The proportion of the mixture in the reaction tank was:

	Liters
Sea-water -----	1
Lime-water -----	2,4

The liquid from the reaction tank received in the decanting tank was there separated from the precipitate which was continuously reintroduced together with the lime-water in the reaction tank.

Under these conditions, after treating twenty times the initial volume and passing again twenty times the precipitate through the circuit, a production of 2900 milligrams of $\text{Mg}(\text{OH})^2$ per liter of sea-water treated was recovered. The precipitate composition (filtered under vacuum) was 53% H^2O and 47% $\text{Mg}(\text{OH})^2 + \text{CO}^2\text{Ca}$.

After calcination at 1250°Cent . the material retained 4,8% of CaO and after washing out with fresh water the finished product contained:

	Per cent
MgO -----	98,8
Impurities -----	1,2

When manufacturing in the same conditions from sea water previously purified and from filtered lime-water made out of fresh water calcined magnesia retained only 0,5% impurities, without requiring any washing out of CaO .

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