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

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This invention relates to an improved process for extracting Magnesia from aqueous solutions containing Magnesium Salts convertible by lime such as sea-water, natural or artifical brines, etc.

Generally milk lime is used according to the well-known reaction.

$$(1)\quad SO^4Mg+Mg\ Cl^2+2Ca(OH)^2= SO^4Ca+CaCl^2+2Mg(OH)^2$$

Milks of calcined dolomites containing caustic lime have been used to the same purpose.

Unhappily, the reaction (1) with milk necessitates expensive working as magnesia is recovered in the form of precipitates which are very voluminous, difficult to filter and to wash and which retain very large quantities of water.

It has been found that by using lime water which is a liquid and very diluted reagent very good results are obtained when the reaction is carried under the following conditions:

Mixing lime-water with the magnesium brine, separating the precipitate which is formed and continuously reinjecting the precipitate from one operation in the reaction liquids used for the next operation so as to obtain finally, after a number of successive operations a heavy precipitate easily recovered, filtered and washed.

This lime-water process has only one drawback, it requires the manipulation of large volumes of liquids and therefore large equipments.

I have now found that, if operations are properly carried out, it is possible to obtain precipitates of a quality equivalent to the precipitates obtained from lime-water, by using instead of a liquid reagent such as lime-water reagents such as milk of lime or milk of calcined dolomite more or less diluted so that the reaction takes place between the magnesium salt solution and solid particles kept in liquid suspension. The result is that the volumes of liquids to handle and the volumes of equipment are considerably reduced.

For instance, the extraction of Magnesia from sea-water will require for one ton of MgO the manipulation of about five hundred cubic meters of sea-water and about fourteen hundred cubic meters of lime-water. In my improved process, the volume of sea-water is of course not altered but for the suspension of lime in water I can use a very small volume of water.

This quantity varies according to the ways and means employed to work out my invention which will be explained later on but it can be reduced eventually just to the quantity necessary for making the milk of lime or the milk of dolomite i. e. well under fifteen cubic meters for one ton of magnesia.

The fact that these results can be obtained by substitution of a solid suspension for a liquid reagent is rather surprising because the conditions of reaction and pH during the reaction are not the same in the two cases.

In carrying out the invention the following steps are used:

(I) Making a milk from the reagent (calcined lime, calcined dolomite, etc.). At this step heavy impurities such as unburnt or overburnt stones are easily disposed of.

(II) Making from the milk a suspension of solid particles in diluting liquid. At this step, smaller impurities may be eliminated.

(III) Mixing the magnesium salts solution with the suspension from step II together with previously formed precipitate of magnesia so that precipitation from the new reaction will always be realised in the presence of agitated seeding products regularly sent back to be fed by new reactions until proper physical qualities of the final precipitate are obtained.

(IV) Separating the precipitates from the mother liquids and sending back the separated precipitate in III until proper qualities are obtained.

In this step IV, I may work on the discontinuous principle and recover the precipitates in batch operations but it is better to work in a regular continuous circuit. In this case, when the precipitate has reached the required physical qualities (density, filtrability, washability, etc.), only a part of the precipitate is sent back to reaction and the rest is drawn out for utilisation.

Step I.—This does not require special explanation as it is a usual and well-known industrial operation it requires a very small amount of water which may be any of the three qualities mentioned for use as diluting liquid in Step II hereunder. At this step heavy impurities are better sieved out or otherwise disposed of.

Step II.—At this step we have to consider: (*a*) The separation of smaller impurities; (*b*) The diluting liquid; (*c*) The diluting operation.

(*a*) is usually simply carried by a rough decantation whereby heavier and coarser particles are eliminated. Any equivalent means can be employed for instance a Sieve, etc.

(*b*) for diluting water, I may employ either fresh water, or the residual exhaust water from the process but I find more advantageous to use as diluting liquid a portion of the liquid resulting from reaction in Step III and still containing the precipitate which has been formed during said reaction. By using the reaction liquid sent back from Step III to Step II, the real consumption of diluting liquid is reduced to losses and the cost is reduced to merely small pumping expenses as this reaction liquid is utilised without any sort of preparation or treatment. Further it acts as a seed and facilitates the precipitation of dense material.

(c) The diluting operation is very simple; it may be carried in any mixing tank provided with proper mixing devices. It wil be avantageous to provide means to separate at this step small but heavy impurities which will have escaped the treatment in step I.

Step III.—This step is better carried out in a reaction tank where the magnesium brine is mixed with the diluted reagent from Step II and with precipitate sent back from Step IV. This precipitate may be admitted in the reaction tank either separately or mixed with the magnesium brine. I generally prefer to mix it with the diluted reagent before admission in the tank.

The reaction tank may be a simple tank provided with proper mixing devices (specially in case of batch operation). For continuous work I generally prefer to use a reaction tank in the shape of a longitudinal channel provided with mixing devices where I admit first the diluted reagent together with the seeding precipitate at one end and where I inject progressively the magnesium brine in due proportion, the speed of travel of the products from one end to the other being so regulated that reaction is practically finished during the time taken by the liquid to reach the exhaust.

Step IV.—This step may be carried by any device able to separate the precipitate from the mother-lye. I generally prefer to use a decanting tank.

From this separating device, the precipitate is sent back to Step III until by working again and again new reactions in the presence of the precipitates from previous reactions the desired physical qualities are obtained; when the precipitate is recovered and operations started again.

In continuous work the precipitate is sent back as a whole until proper qualities are obtained and thereafter a proper portion of the precipitate may be regularly drawn out and disposed of whilst the rest goes back as a seed in Step III.

The relative volume proportions of the different tanks for operating the process will be easily determined by small scale tests.

As an example the following proportions have given very good results, starting from sea-water and lime milk. The figures refer to a production of one ton MgO per hour recovered as $Mg(OH)^2$:

(1°) Proportion of reagents:
Sea-water per hour____cubic meters__ 500
CaO (plus impurities and insolubles) ______________________kilogs__ 1400

(2°) Volume of diluting tank_cubic meters__ 500

The mixture of milk and diluting liquid (which was taken back from the reaction tank without separating the precipitate) was agitated and passed through the tank in 10 to 15 minutes. The volume of diluting liquid was 2000 to 3000 cubic meters per hour.

(3°) Volume of reaction tank__cubic meters__ 150

(4°) Surface of decanting tank____ _______________cubic meters__ 1000 to 1500

Another advantageous way of carrying out the invention consists with using a series of tanks through which the products flow progressively.

In the first tank the milk of reagent and the seeding precipitate from the decanting apparatus are admitted and mixed together with a proportion of the liquid from the last tank of the series, in the following tanks the magnesium brine is admitted in fractions. Agitation is carried out in each tank. The overflow from the last tank goes to decantation wherefrom, when the "regime" is reached, a proportion of precipitate is sent back to the first tank and the surplus disposed of for recovering the precipitate to be disposed of. In this form of working the proportion of diluting liquid may be reduced on condition that effective agitation is provided and sufficient time is allowed for the reaction to be practically finished in the last tank.

Of course, these successive tanks may be replaced by any equivalent apparatus such as a channel-tank provided with proper means to insure the desired successive flow, mixtures and agitation all along the channel.

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