



TECHNOLOGY OF OBTAINING MGO FROM EPSOMITE LOCAL RAW MATERIAL

Orazberdiyev Kyyasberdi

student of the Oguzhan Engineering Technologies University of Turkmenistan
Ashgabat, Turkmenistan

Ovezsahedov Saparmyrat

Lecturer of the Oguzhan Engineering Technologies University of Turkmenistan
Ashgabat, Turkmenistan

Ryzayev Kerim

Lecturer of the Oguzhan Engineering Technologies University of Turkmenistan
Ashgabat, Turkmenistan

Introduction

A simple, efficient and inexpensive method of purifying magnesium sulfate from mixtures of epsomite and halite. The method allows for the preparation of substantially pure magnesium sulfate products. The method generally involves heating a mixture of epsomite and halite to form clusters of lower hydrated magnesium sulfate crystals and subsequently applying slight pressure to the clusters so that they collapse to yield fine, less hydrated magnesium sulfate crystals which can then be easily separated from the comparatively coarse halite crystals. The less hydrated magnesium sulfate crystals can be recrystallized to provide highly purified epsomite which can then be dehydrated to provide kieserite or anhydrous magnesium sulfate.

Technical field

The invention relates generally to the preparation of substantially pure magnesium sulfate products and more particularly to a method for recovering such products from mixtures of epsomite and halite.

Background art

Magnesium sulfate monohydrate ($\text{MgSO}_4 \cdot \text{H}_2\text{O}$) and the higher hydrates of magnesium sulfate are naturally occurring materials having many uses. For example, they are known as fireproofing materials, as catalyst carriers, for use in warp-sizing and loading cotton goods, weighting silk, dyeing and calico printing,

as an ingredient in mineral waters, and are used in ceramics, fertilizers, paper, cosmetic lotions, dietary supplements, laxatives, and as a therapeutic bath salt.

A number of methods are known in the prior art for the production and purification of magnesium sulfate (MgSO_4) and its hydrates. For example, these materials are sometimes produced by reacting sulfuric acid (H_2SO_4) with either magnesia (MgO) or magnesite (MgCO_3). However, this method is very expensive and the magnesium sulfate produced may still require subsequent purification due to impurities present in the magnesia or magnesite feed stock.

Alternatively, hydrated magnesium sulfate may be produced by dissolving kieserite (magnesium sulfate monohydrate) and then subsequently refrigerating this resulting solution.

Kieserite is mined in large quantities, for example in Germany, and after washing, supplies most of the world's magnesium sulfate market. It contains various impurities, however, including halite (i.e., sodium chloride) and, in addition, it comprises only 89-93% magnesium sulfate monohydrate. Moreover, since the mined kieserite is only partly soluble in ambient water it is not acceptable for use in certain applications, for example, as a therapeutic bath salt or as a laxative.

Epsomite is a heptahydrate of magnesium sulfate having the chemical formula $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. It is often found mixed with halite (i.e., NaCl) in nature. Such mixtures are readily isolated from seawater by solar evaporation. In the past, however, these mixtures have not been relied upon as a source of magnesium sulfate or its hydrates since epsomite and halite react upon contact in certain aqueous solutions to form double salts, i.e., a condition wherein two salts crystallize from a melt or from a solution in simple stoichiometric proportions, forming a distinct crystal lattice. The double salts formed in this case are mainly of magnesium and sodium sulfate. An example of such a double salt is astrakanite ($\text{MgSO}_4 \cdot \text{Na}_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$)

Consequently, the separation of epsomite and halite must take place in a medium in which the two substances cannot react, i.e., so as not to form double salts. A simple, cost-effective method has thus long been desired for separating these two materials in a manner to provide substantially pure magnesium sulfate products without the formation of double salts as described above.

The present invention provides a simple, cost-effective method of separating substantially all the magnesium sulfate present in an epsomite-halite mixture to obtain substantially purified magnesium sulfate products without the formation of double salts as described above.

In the method of the present invention, magnesium sulfate is separated from epsomite-halite mixtures by converting the epsomite portion of the mixture in the manner described below into fine magnesium sulfate crystals (i.e., substantially more fine than the epsomite crystals) having a degree of hydration lower than epsomite (i.e., $<7\text{H}_2\text{O}$). Due to their smaller size, the resultant less-hydrated crystals may then be readily separated from the comparatively larger and coarser halite crystals.

More specifically, the epsomite-halite mixture is first heated at a temperature above about 47°C ., i.e., the temperature at which epsomite crystals become unstable, but below about 83°C ., the melting temperature of epsomite. Heating at this temperature causes the epsomite crystals to break down into clusters formed of a plurality of fine crystals of magnesium sulfate hexahydrate or of lower hydrates of magnesium sulfate, which remain clumped together such that the clusters typically maintain the shape of the original epsomite crystals. The amount of hexahydrate or lower hydrated crystals that are formed depends on the length and temperature of the heating, i.e., the lower the temperature and the shorter the heating period, the greater the likelihood that the crystals will be composed mostly of magnesium sulfate hexahydrate. Conversely, higher temperatures and/or longer periods of heating tend to produce magnesium sulfate products with a correspondingly lower degree of hydration.

Applying a minimal amount of pressure to the crystal clusters thus formed causes them to collapse and separate into a plurality of individual magnesium sulfate hexahydrate ($\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$) or lower hydrated crystals. These newly formed magnesium sulfate crystals are not affected by the heat or pressure applied as described above. These fine crystals are then separated from the comparatively coarse halite crystals by methods well known in the art, such as screening.

Thereafter, in one embodiment of the invention, the hexahydrate or lower hydrate crystals thus formed may be subsequently recrystallized by hydrating them in, for example, an atmospheric crystallizer to form high grade epsomite crystals. "High grade", as used herein means a product of about 99.97% purity. These high grade crystals may then, if desired, be further recrystallized in, for example, a vacuum crystallizer to obtain a final product comprising pure grade epsomite crystals. "Pure" grade as used herein means a product of between about 99.98% to about 100% pure. A suitable vacuum crystallization apparatus is one such as described in U.S. Pat. No. 3,873,275 or U.S. Pat. No. 3,961,904, the disclosures of which are incorporated herein by reference.

In an alternate embodiment of the invention, the hexahydrate or lower hydrate crystals which have been separated from the comparatively coarse halite crystals can be recrystallized with the use of a counter-current belt filter extractor to provide high grade epsomite.

The purified epsomite crystals produced by any of the above-described embodiments may, in turn, be subsequently melted at a temperature of above about 80° C. or dissolved in a solvent at a temperature of about 70° C. to about 80° C. The resultant magnesium sulfate solution, or slurry thereof, can then be dehydrated in a fluidized bed drier. The dehydration is performed at different temperatures to form magnesium sulfate crystals having various degrees of hydration, such as anhydrous magnesium sulfate and magnesium sulfate monohydrate, depending upon the dehydration temperature used. These two products are particularly preferred because they can serve as excellent desiccants. This is because the monohydrate or anhydrous crystals can absorb water to re-form epsomite without altering the crystals' original shape, size or appearance.

Alternatively, the solution or slurry can instead be dehydrated in a spray drier. It is preferred, however, that the magnesium sulfate crystals be dehydrated in a fluidized bed drier because the particles obtained from the spray drying process tend to be too fine for use in certain applications.

Conclusions

It is noted that the above description is merely illustrative of the invention, and other parameters and embodiments may be used without departing from the inventive concept herein. Accordingly, the present invention is only limited by the claims appended hereto.

References

1. 2022 European Biomass Industry Association. Available online: <http://www.eubia.org/cms/wiki-biomass/biofuels/biodiesel/> (accessed on 11 February 2022).
2. Toldrá-Reig, F.; Mora, L.; Toldrá, F. Trends in Biodiesel Production from Animal Fat Waste. *Appl. Sci.* 2020, 10, 3644 <https://doi.org/10.3390/app10103644>.
3. Directive (EU) 2018/2001 of the European Parliament and of the council of 11 December 2018, on the promotion of the use of energy from renewable sources. *Off. J. Eur. Union* 2018, L328/82.
4. Fattah, I.M.R.; Ong, H.C.; Mahlia, T.M.I.; Mofijur, M.; Silitonga, A.S.; Rahman, S.M.A.; Ahmad, A. State of the Art of Catalysts for Biodiesel Production. *Front. Energy Res.* 2020, 8, 101 <https://doi.org/10.3389/fenrg.2020.00101>.
5. Tran-Nguyen, P.L.; Ong, L.K.; Go, A.W.; Ju, Y.; Angkawijaya, A.E. Non-catalytic and heterogeneous acid/base-catalyzed bio-diesel production: Recent and future developments. *Asia-Pac. J. Chem. Eng.* 2020, 15, e2490. <https://doi.org/10.1002/apj.2490>.

6. Dawood, S.; Ahmad, M.; Ullah, K.; Zafar, M.; Khan, K. Synthesis and characterization of methyl esters from non-edible plant species yellow oleander oil, using magnesium oxide (MgO) nano-catalyst. *Mater. Res. Bull.* 2018, 101, 371–379.
<https://doi.org/10.1016/j.materresbull.2018.01.047>.
7. Rabie, A.M.; Shaban, M.; Abukhadra, M.R.; Hosny, R.; Ahmed, S.A.; Negm, N.A. Diatomite supported by CaO/MgO nanocomposite as heterogeneous catalyst for biodiesel production from waste cooking oil. *J. Mol. Liq.* 2019, 279, 224–231.
<https://doi.org/10.1016/j.molliq.2019.01.096>.
8. Dimian, A.C.; Bildea, C.S.; Kiss, A.A. *Technology & Engineering Applications in Design and Simulation of Sustainable Chemical Processes*; Elsevier: Amsterdam, The Netherlands, 2019; pp. 719–734.
<https://doi.org/10.1016/c2015-0-06856-3>.