

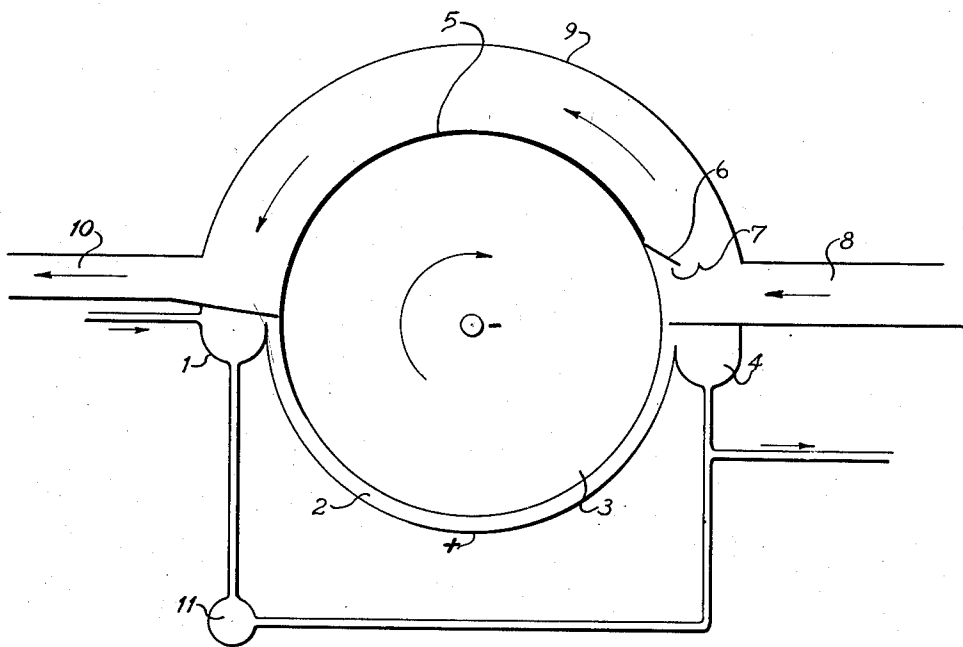
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RECOVERY OF MAGNESIUM FROM SUSPENSIONS IN NON-CONDUCTING LIQUIDS

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RECOVERY OF MAGNESIUM FROM SUSPENSIONS IN NONCONDUCTING LIQUIDS

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This invention relates to a method of recovering finely divided magnesium which is held in suspension in non-conducting fluid, particularly in a hydrocarbon medium.

It is an object of the invention to provide a method for separating suspensions of finely divided magnesium in a non-conducting liquid, to obtain a concentrated solid phase and a cleared liquid phase. One of the primary objects of the invention is to provide means for concentrating magnesium metal dust finely dispersed in a hydrocarbon medium.

Magnesium partly results in the form of dust in the refining, by distillation, of crude electrolytically produced magnesium metal or of magnesium scrap, and particularly in the course of the recovery of metallic magnesium from oxidic magnesium compounds by thermic reduction, carried out at a temperature above the boiling point of metallic magnesium and followed by cooling down the vapor to a temperature below the solidification point of metallic magnesium. Magnesium dust obtained in one or the other of the above ways, particularly when in the state of colloiddally fine subdivision, is highly reactive with oxygen and nitrogen and even spontaneously inflammable. It is old in the art to protect the dust against such unwanted conversions by immersing the same immediately after formation in a hydrocarbon oil medium or, in the case of its resulting from shock chilling the vapor evolved in the course of thermic reduction of magnesium oxide-containing starting materials, by using liquid hydrocarbons as the chilling medium proper. In any case the metallic magnesium is thus obtained in the state of slurry in the hydrocarbon, containing the metal in such a finely divided condition that only a minor proportion may be separated by settling. Conventional methods of separation, such as centrifuging, filtration, etc., will produce a slurry containing a fairly high proportion of solids, but have the disadvantage that a clear hydrocarbon is not recovered. The finely divided solids obtained as centrifuge overflow or filtrate will stay in suspension for periods of weeks without noticeable separation.

I have discovered that finely divided solids of this nature may readily be removed from a non-conducting dispersion medium by electrostatic means. When producing magnesium metal with the aid of an electrically heated reducing agent it is found that the solids are already electrically charged, and therefore, are readily drawn through the suspending medium to an electrode at a high

potential of opposite sign. Normally these particles are positively charged and the repelling electrode will be at a positive potential of about 5,000 to 100,000 volts depending upon gap spacing, the nature of the hydrocarbon liquid, etc., while the collecting electrode will be grounded. When the particles are not sufficiently charged it may be necessary to have the repelling electrode at a higher potential difference above ground. Ionization in very small amount from one electrode is picked up by the particles which become charged and are carried to the collecting electrode of opposite sign where they are collected in the form of a dense slurry. Static or batch apparatus may be utilized to obtain the desired effect, with periodic scraping or flushing off of the solids collected. However, I have found that a particularly suitable apparatus for continuous operation may be utilized which consists, as described in greater detail below and shown schematically in the drawing, of a rotating cylindrical drum partially immersed in a bath of slurry.

The operation of the electrostatic separator may be observed in detail by reference to the drawing. Dirty feed enters the launder 1, extending the length of the drum, and overflows into the annular space 2, between the trough and the rotary drum 3. Drum 3 rotates countercurrent to the flow of the hydrocarbon slurry. Solid particles are drawn to the drum under the influence of the electrostatic field and are removed thereon in the form of a cake. The hydrocarbon medium is progressively clarified as it passes through the annular space and finally overflows as completely clear liquid free of all suspended solids into a discharge launder 4.

The cake 5 containing all of the solids removed from the slurry is carried on the surface of the drum to scraper 6 where it is scraped loose into a suitable conveyor 7 and removed from the system to suitable means for purification (not shown). When produced in the above described manner it is found that the cake contains about 40% solids and 60% hydrocarbon, by weight. In certain cases it is desired to obtain a cake containing 70 to 80% solids for convenience in handling and because of the plasticity desired. This I have found to be accomplished by passing a stream of inert gas, such as methane, natural gas, hydrogen or carbon monoxide, through inlet 8, over the cake 5 under a suitable hood 9, and out through discharge opening 10. The gas stream is preferably passed countercurrent to the drum rotation in order to make fullest use of

available heat. Countercurrent flow of the gas has been found particularly advantageous in those cases where the hydrocarbon slurry introduced into launder 1 is at elevated temperatures. As a means of introducing the required heat to evaporate the hydrocarbon from cake 5, where this heat is not entirely furnished by the hot slurry introduced into launder 1, I have found that several methods may conveniently be used. For example, the gas stream may be heated by external means (not shown) before introduction through opening 8, or hood 9 may be externally heated to heat the gas stream by radiation and convection, or electrical heating elements may be suspended in the gas stream between drum 3 and hood 9. In this manner, any desired proportion of solids and hydrocarbon may be obtained in the cake discharged into conveyor 7 by controlling the flow of gas and the amount of heating employed.

In the practice of my invention I have found that complete separation of suspended solids may be obtained under a wide range of slurry concentration and operating variables, such as drum size, speed of drum rotation, electrostatic potential, etc. Two unexpected phenomena have been discovered in practicing this invention, however, namely that finer particles are easier to remove than larger and more thoroughly agglomerated particles, and also that higher throughput of solids may be accomplished by using more dilute feed. It is preferable although not necessary to introduce into the collecting system some form of conventional settler or thickener to remove any exceptionally large particles. When such large particles are present they have a tendency to deposit as a layer in the bottom of annular space 2 from which they may conveniently be removed hydraulically by streams of hydrocarbon pumped into the annular space through jets, not shown in the drawing.

As an example of the practice of my invention, and also to show the effect of feed dilution the following experiment is illustrative. A slurry of crude magnesium metal dust, containing some magnesium oxide and carbon, suspended in a paraffinic hydrocarbon diluent having a boiling range of 300 to 400° F. was used for the experiment. The apparatus was substantially as described above, with the collecting drum grounded and the lower electrode or trough at a positive electrostatic potential of about 15,000 volts. The original feed contained 10% by weight of solids, and this was successively diluted with solvent to give slurries containing 5% and 2½% by weight of solids. Each of these feeds was then passed into the electrostatic separator while maintaining a constant drum speed to give exposed surface of 19.7 square feet per hour. The rate of feed of each slurry was gradually increased to the maximum which could be maintained to give a clear solvent overflow free of all suspended solids. The following data were obtained:

Concentration of solids in slurry, per cent by weight	Feed rate, gal./hr.	Clear solvent production, gal./hr.	Drum cake production, lbs./hr.
10	0.41	0.28	0.89
5	1.45	1.27	1.88
2½	2.78	2.19	2.24

These data indicate the advantages to be obtained when using a more dilute slurry. It is not

always possible to obtain a slurry having the desired concentration, and it is part of my invention to dilute the slurry obtained with suitable clear solvent. The clear solvent may be obtained by circulation of clear solvent overflow from discharge 4 through pump 11 to the slurry line or directly into launder 1.

The term hydrocarbon as used throughout the specification and in the following claims is to be understood to include any hydrocarbon being normally liquid, or a mixture of hydrocarbons of this kind.

I claim:

1. In the winning of magnesium from its ores by carbothermic reduction wherein by shock chilling there is produced magnesium and accompanying impurities in the form of fine powder, the method of treating said powder which comprises contacting said powder with a liquid hydrocarbon in sufficient amount to wet down all surfaces of the powder, and to form a slurry of said powder and liquid hydrocarbon, diluting said slurry to a thin suspension of from 95% to 97½% liquid hydrocarbon, subjecting said suspension to electrophoretic separation to segregate the solids from the liquid hydrocarbon, and collecting said segregated solids as a cake of powder with the surface of the powder therein protected by liquid hydrocarbon.

2. In the winning of magnesium from its ores by carbothermic reduction wherein by shock chilling with a liquid hydrocarbon there is produced magnesium and accompanying impurities in a finely divided and wetted state, the method of treating such product which comprises adding thereto a liquid hydrocarbon in sufficient amount to form a slurry, diluting said slurry to a thin suspension of from 95% to 97½% liquid hydrocarbon, subjecting said suspension to electrophoretic separation to segregate the solids from the liquid hydrocarbon, and collecting said segregated solids as a cake of powder with the surface of the powder therein protected by liquid hydrocarbon.

3. In the winning of magnesium from its ores by carbothermic reduction wherein by shock chilling there is produced magnesium and accompanying impurities in a finely divided state, the method of treating such product which comprises adding thereto a liquid hydrocarbon in an amount sufficient to form a slurry, diluting said slurry to a thin suspension of from 95% to 97½% liquid hydrocarbon, subjecting said suspension to electrophoretic separation to segregate the solids from the liquid hydrocarbon, and collecting said segregated solids as a cake of powder with the surface of the powder therein protected by liquid hydrocarbon.

4. In the winning of magnesium from its ores by carbothermic reduction wherein by shock chilling there is produced magnesium and accompanying impurities in a finely divided state, the method of treating such product which comprises adding thereto a liquid hydrocarbon in an amount sufficient to form a slurry, then in continuously succeeding steps, diluting said slurry to a thin suspension of from 95% to 97½% liquid hydrocarbon, subjecting said suspension to electrophoretic separation to segregate the solids from the liquid hydrocarbon, and collecting said segregated solids as a cake of powder with the surface of the powder therein protected by liquid hydrocarbon.

5. A method for the recovery of finely divided magnesium from suspensions thereof in a hydro-

carbon liquid which comprises diluting said suspension to a thin suspension of about 2½% to 5% of magnesium, subjecting said suspension to electrophoretic separation to segregate the magnesium from the hydrocarbon liquid, and collecting the magnesium.

6. A method for the recovery of finely divided magnesium from suspensions thereof in a hydro-

carbon liquid which comprises in continuously succeeding steps, diluting said suspension to a thin suspension of about 2½% to 5% of magnesium, subjecting said suspension to electrophoretic separation to segregate the magnesium from the hydrocarbon liquid, and collecting the magnesium.

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