

STUDIES IN CHLORINATION: PART I - MAGNESITE(*)

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Preparation of anhydrous magnesium chloride by dehydration from hydrated magnesium chloride is not straight forward after dihydrate stage due to hydrolysis. Magnesium oxide and oxy-chloride contaminate the final product in uncertain amounts.

The major uses of anhydrous magnesium chloride are for extraction of magnesium metal and preparing fluxing material used in melting, refining and casting of magnesium and magnesium-base alloys for which it should be free from magnesium oxide and oxychloride etc.

The present paper describes a process to prepare anhydrous magnesium chloride synthetically, by which the disadvantages of incomplete reaction and hydrolysis are eliminated.

The preliminary processing of raw materials, study of their characteristics, time and temperature of chlorination reaction, rate of gas flow etc. have been examined. On the basis of data, optimum conditions have been established and proposals have been put forward to design a production unit with a capacity of 25 Kg/day. Calcined magnesite crushed to -60 mesh mixed with calcined petroleum coke of -60 mesh in the ratio of 10:3 together with magnesium chloride solution as binder have been briquetted into cylindrical shape of about 12 mm dia under various pressures.

The briquettes are heat treated under standardised condition to reduce free moisture and volatiles and their physical properties are measured. Concurrently, pellets of about 0.7 Cm dia from the same composite mix are prepared in a drum pelletiser and heat-treated under identical conditions.

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Both the briquettes and pellets are chlorinated. The chlorination is carried out in a 25 mm quartz tube heated externally by a vertical tube furnace. The lower part of the tube is packed with graphite particles which supports the charge. Chlorine is injected by a side tube at the bottom of the graphite bed which flows up through the graphite bed and the charge.

The reaction product being liquid at reaction temperature passes through graphite bed and collects in a silica receptacle fitted at the lower end of the quartz tube. The top is open to atmosphere under an exhaust duct.

Among the various variables studied are the temperature, variation of gas flow rate, time of reaction, quantity of carbon addition.

Optimum conditions for briquette are as follows:

Bulk density of the briquettes	1.39 g/cc
Porosity	44.04%
Crushing strength	28.69 Kg per Sq.cm.
Amount of chlorine	Theoretical
Temperature	900°C
Time of reaction	2 hours.

Sufficient coke is used (on the basis of MgO:C ratio 10:3) so that its fixed carbon content is approximately equal to the stoichiometric amount of carbon necessary to reduce the magnesium oxide of the calcined magnesite for its subsequent chlorination. Under these conditions 68.32% conversion has been obtained.

Optimum conditions for MgO + C pellets are

Bulk density	1.230 g/cc
Porosity	46.37%
Crushing strength	29.86 34.74 Kg per Sq.cm.
Amount of chlorine	Theoretical
Temperature	900°C
Reaction time	2 hours

under these condition 75.06% conversion has been obtained.

The magnesium chloride obtained analyses as MgO - 0.07%, Moisture - 0.12, CaCl₂ - 0.48%, MgCl₂ - 99.23%.

It can be concluded that on the basis of conversion efficiency pellets are better as feed charge for chlorination than briquettes.
