

Novel technique for the preparation of refractory grade magnesia from bittern through double salt route

Niva Nayak & Chitta R Panda*

CSIR-Institute of Minerals & Materials Technology, Bhubaneswar 751 013, India
E-mail: crpanda@immt.res.in

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A novel technique has been developed to prepare refractory grade magnesia from sea bittern, which is a heterogeneous precipitation method to precipitate magnesium as double salt by a suitable carrier reagent. Prepared double salt is converted to magnesium carbonate, which on further calcination gives rise to MgO. The powdered samples of prepared magnesium carbonate and magnesium oxide are characterized by elemental analysis, X-ray powder diffraction, IR, TG/DTA, Particle size analyser and SEM. Commercially available Fluka magnesium carbonate and its calcined product have also been simultaneously characterised for comparison. Prepared boron free magnesia shows high purity of 99.08%, which can be used as basic refractory material.

Keywords: Bittern, Double salt, Magnesium carbonate, Magnesia, Phase assembles

The increased demand for high purity magnesia as basic refractory has been prominent all over the world since last four decades. Particularly, in the magnesia carbon refractory, to be used as the refractory for converter, electrofused magnesia is frequently employed. In the last few decades efforts have been made by several researchers to synthesize high pure magnesia from natural magnesite¹⁻³, seawater⁴⁻¹⁰ and magnesia rich brine¹¹⁻¹⁴. The physical and chemical beneficiation of magnesium ores are having limitations which often restrict the quality of desired product and can create significant corrosion problems due to the use of strong acids. Besides this, the less expensive calcined magnesia, particularly seawater magnesia clinker, is also another alternative. The bitterns rejected from solar salt plants have long been considered as a possible raw material for the recovery of various mineral salts and marine chemicals. With the advent of extremely large solar salt plants where the bittern tonnage is huge, and through the innovation of advanced technology, production of marine chemicals from such plants may be practiced on a reasonably economic basis. Solar evaporation is the cheapest and most economic means for the pre-concentration of sea-bittern. Magnesia is generally prepared by single or double stage sintering process from seawater. Mg(OH)₂ or MgCO₃ are precipitated from sea water by using various precipitating agents like NaOH, Na₂CO₃,

Ca(OH)₂, dolomite etc. All the processes are fundamentally similar but differ only when the purity of the product is concerned. As magnesia is used for refractory purposes, the purity expected should be very high, i.e. >99%. Almost all commercial processes of magnesia production routed through the precipitation of magnesium hydroxide as an intermediate. Although the chemical reactions involved in these processes are very simple, the considerable engineering involved in handling the intermediate produces are extremely complicated. Magnesium hydroxide is an excellent sorbent for many trace metals because of its high specific surface area with an abundance of exposed OH⁻ rich faces¹⁵. Silicon, iron, aluminium, calcium and boron are considered as undesirable impurities in magnesia since they form low melting point compounds and ultimately reduce the hot strength of magnesia refractory^{6,16,17}.

In the present study, attempts have been made to get high purity magnesia through a new process (double salt route) for the recovery of magnesium from bittern, a waste product of the salt industry. As the basic carbonates does not adsorb impurities to the same extent as does the hydroxides, this technique could be a suitable alternative to extract magnesium as double salt from sea bittern with a view to minimize the impurity level in the final product.

Experimental Section

The required raw material 29° Be' bittern was collected from the Huma solar salt plant, situated at Ganjam district of Odisha, India. Commercial grade reagents like ammonium sulphate, ammonium carbonate, ammonium hydroxide and sorbitol are used in the preparation of magnesia. A set of experiments was carried out to optimize the precipitation condition of double salt. 100 mL bittern containing Mg: 4.96 g was treated with varied amount of ammonium sulphate at varied ratio of Mg: (NH₄)₂SO₄, i.e. 1:1, 1:1.25, 1:1.5, 1:1.75 and 1:2 at room temperature. The reaction temperature of the stirred mixture automatically increased to 44°C. Further the mixture was kept on a water bath for 2 h at 60°C. On cooling the double salt precipitated out slowly and the precipitate is filtered at room temperature and oven-dried at 110°C.

In a similar procedure, a set of experiments was carried out to optimize the precipitation condition of magnesium carbonate with ammonium carbonate as precipitating agent. 100 g double salt containing Mg: 8.68 g dissolved in 500 mL water and reacted separately with ammonium carbonate at a varied ratio of Mg: (NH₄)₂CO₃, i.e. 1:1, 1:1.25, 1:1.5, 1:1.75 and 1:2 at room temperature. Ammonium carbonate solution is added slowly to the double salt solution with constant stirring. Precipitation started at a temperature of 40°C and then the system is kept on hot water bath at ~70°C for 2.0 h for complete precipitation of MgCO₃. The solid precipitate (MgCO₃) is separated by filtration and washed with hot distilled water several times to make it free from other dissolved impurities like sulfate, chloride, sodium and potassium. In the final washing 2-3 drops of sorbitol were added to the distilled water and then washed to make the product free from boron. The solid is then oven-dried at 120°C. The dried magnesium carbonate is calcined at 1100°C in a muffle furnace to yield magnesium oxide.

The bittern was analysed by AAS (Shimadzu AA6300), ICP-OES (Perkin-Elmer Optima 2100 DV) and Flame photometer (Elico, Model CL-22D) for the major and trace elements along with the determination of pH and EC by WTW kit (Model Multi 340i). The double salt, filtrates, washings along with the prepared magnesium carbonate and magnesium oxide were also analysed for various elements like Mg²⁺, Ca²⁺, SO₄²⁻, Cl⁻, Na⁺ and K⁺ by the help of above mentioned instruments. In addition to, synthesised

magnesium carbonate and magnesia, commercially available magnesium carbonate (Fluka make) and its calcined product (MgO) were also characterised by XRD (Philips PW 1400), IR (Perkin-Elmer 823 spectrometer), TG/DTA (Shimadzu DT 40), Particle size (Malvern particle size analyser, Model-3600) and SEM (Jeol JSM 840).

Results and Discussion

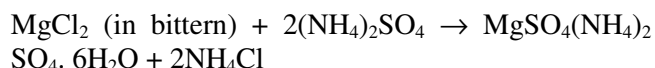
Analysis of bittern

The analysed 29° Be' bittern shows concentration of major elements as Mg - 49.61, Ca - 0.1, SO₄²⁻ - 64.7, Cl⁻ - 191.97, Na - 53.75, K - 10.87 g/L. The major constituents of sea-bitterns are sodium chloride, magnesium sulphate, magnesium chloride and potassium chloride and these are found together with small quantities of sodium sulphate, bromides and some other minor constituents^{18,19}. Measurements of trace metals present in seawater frequently push against the limits of analytical capability in terms of sensitivity due to interference of various radicals. However, the concentrations for many of the metal are found to be B - 48.49, Co - 2.65, Cr - 1.74, Fe - 7.0, Mn - 0.68, Ni - 0.1, Sb - 6.0 mgs/lit, whereas the Al, Si metals could not be detected²⁰. Generally, seawater is alkaline in nature and the pH of sea water ranges from 7.8 to 8.4. The alkalinity is due to the presence of the bicarbonates (HCO₃⁻), carbonates (CO₃²⁻) and the H₂BO₃⁻ in the sea water²¹. As the bicarbonates and carbonates are removed as CaCO₃ during the solar evaporation, the pH of the bittern is no more alkaline. Hence, the pH of the 29° Be' bitterns is found to be 6.71. The electrical conductivity of 29° Be' bittern is 505000 μS/cm. It is dependent upon the ionic concentration and ionic mobility of the mineral content in solution and in general, is an index of the degree to which the water is mineralised.

Precipitation of double salt

The double salt of magnesium and ammonium sulphate is prepared by the addition of stoichiometric proportion of ammonium sulphate to bittern of 29° Be'. As the reaction is exothermic, rise in temperature facilitates the solubility of carrier salt. By adding ammonium sulphate to the bittern solution which is rich in magnesium and by further heating the solution, sulphate ion is generated homogeneously throughout the solution. The double salt is precipitated out on further cooling of the reaction mixture to room

temperature. The fractionation is usually carried out by a heterogeneous precipitation method by shifting a solid alkali sulphate into a solution. In the present investigation different ratios of Mg: (NH₄)₂SO₄ are tried to optimise the best reaction condition for magnesium recovery in the form of double salt i.e. MgSO₄. (NH₄)₂SO₄.6H₂O²². From Table 1 it is observed that the recovery of magnesium in double salt is 96.53% by the addition of stoichiometric amount of ammonium sulphate to bittern. In the second experiment (1:1.25 ratio), with the addition of 25% extra (NH₄)₂SO₄ the recovery of Mg in double salt is 98.56%. Further at higher ratio, the recovery of magnesium decreases and is almost same as first experiment. Assuming 100% recovery of magnesium in sea bittern theoretically, the expected amount of double salt formation should be 73.486 g, where as higher amount of double salt is obtained in 1:1.5, 1:1.75 and 1:2 experiments. Obviously, unreacted (NH₄)₂SO₄ has also been co-precipitated along with double salt. Anticipating the contamination of carrier reagent in the final product, addition of (NH₄)₂SO₄ in 1:1.25 ratio is taken into consideration for the precipitation of double salt. The plausible reaction for the formation of double salt is

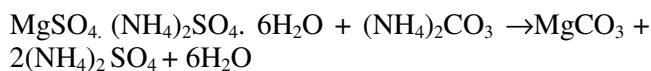


Appreciable amount of magnesium present in seawater as magnesium sulphate along with low percentage of magnesium chloride. When ammonium sulphate is added to the bittern, the magnesium chloride present is converted to magnesium sulphate. The extent of association of Mg²⁺ and SO₄²⁻ increases with decrease in temperature, thereby, forming double salt at room temperature. Simultaneously, the solubility of potassium sulphate, sodium sulphate, calcium carbonate and calcium sulphate increased by the formation of more and more ion pairs between Mg²⁺ and SO₄²⁻ in solution since Mg²⁺ is the effective cation in sea water in terms of effectiveness in complexing²³⁻²⁵.

The solubility of above salts also increases at a faster rate with increase in temperature. Since seawater is a multiphase system, consisting of rather a complex solution of high ionic strength, overall speciation of major elements in sea water and its behaviour are still undefined and have drawn controversy.

Precipitation of magnesium carbonate

Several authors have reported the precipitation of magnesium carbonate from an aqueous magnesium salt solution by different precipitating agents, viz. sodium carbonate, potassium carbonate, sodium and potassium bicarbonate and ammonium carbonate²⁶. The presence of trace amounts of cations, particularly Na⁺ and K⁺ is expected to affect the basicity/base strength distribution of MgO. Hence, Na⁺ and K⁺ contamination in magnesium carbonate will be carried over to the final product reducing the strength of magnesia bricks. With a view to minimise the impurity level and to prepare high pure magnesia, the precipitation of magnesium carbonate has been tried by (NH₄)₂CO₃ as precipitating agent. Varied amount of (NH₄)₂CO₃ of varying ratio with respect to Mg were added to precipitate magnesium carbonate. The reaction was conducted by slow addition of separately dissolved double salt and ammonium carbonate in a minimum volume of water. Experimental condition and magnesium recovery are presented in Table 1.



It is observed that addition of excess (NH₄)₂CO₃ increases the concentration of Mg in magnesium carbonate from 1:1 to 1:2. The percentage of recovery of Mg in magnesium carbonate varies from 54.72 to 76.38% experimentally. At a ratio of 1:1, the recovery was 54.72% and at 1:1.5 it increases to 70.01%. Similarly, at 1:2 ratio the recovery increases to 76.38%, which is further chosen to synthesise more magnesium carbonate for additional investigation.

Table 1 — Extraction percentage of double salt and magnesium carbonate at varied ratio

Varied ratio	Double salt			Magnesium carbonate		
	Wt. of dry Double salt Expt(Theo) in g	Mg in Double salt in g	Recovery of Mg as Double salt (%)	Wt. of dry Magnesium carbonate Expt(Theo) in g	Mg in Magnesium carbonate in g	Recovery of Mg as Magnesium carbonate (%)
1:1	55.1(73.48)	4.78	96.53	18 (30.09)	4.75	54.72
1:1.25	68.0(73.48)	4.89	98.56	20 (30.09)	5.17	59.56
1:1.5	78.5(73.48)	4.66	96.57	23 (30.09)	6.077	70.01
1:1.75	88.5(73.48)	4.80	96.83	23 (30.09)	6.093	70.195
1:2	98.0(73.48)	4.81	96.99	25 (30.09)	6.63	76.38

Maurer²⁷ has studied the precipitation of MgCO_3 by treating MgCl_2 solution obtained by acid leaching of dolomite or magnesite with $(\text{NH}_4)_2\text{CO}_3$ or distillation gas cooler condensate from Solvay process where the basic MgCO_3 formed is calcined to give MgO . Seshadri *et al.*²⁸ reported a useful method for preparation of basic MgCO_3 from bittern using NH_3 and CO_2 at 50°C . Although the removal of Mg was found to be 90.5% in the above referred works when molar ratio of $(\text{NH}_4)_2\text{CO}_3$ to Mg was 2:1, the level of contaminants are also high, which ultimately reduces the hot strength of refractory bricks. Moreover, the cost of the process is markedly high due to consumption of reagent for the precipitation of magnesium hydroxide and ultimate conversion of MgO . Whereas in double salt route the carrier reagent, viz. $(\text{NH}_4)_2\text{SO}_4$, being regenerated during the conversion of MgCO_3 from double salt, which could directly be utilised for double salt precipitation.

Physico-chemical characterisation of magnesium carbonate

The chemical composition of the magnesium carbonate synthesised through double salt route and also the Fluka magnesium carbonate have been analysed. The percentage of Mg in synthesised magnesium carbonate varies from 26.1 to 26.4 and in Fluka magnesium carbonate from 24.12 to 26.23 as shown in Table 2. The major impurities are chloride (0.0398%), calcium (0.0084%) and aluminium (0.0184%) depending upon the bittern composition. The other impurities like Na (0.006%), K (0.0005%), Mn (0.0006%), Fe (0.0019%), Si (0.0017%) are present in less percentage whereas the boron and sulphate contents are almost negligible. Several workers have reported the preparation of basic magnesium carbonate by varying

Table 2 — Chemical composition of Magnesium carbonate (in %)

Element	Prepared MgCO_3	Fluka MgCC
Mg	26.1-26.4	24.12-26.23
SO_4	nd	<0.3
NH_3	nd	nd
Cl	0.0398	<0.01
Na	0.0061	nd
K	0.0005	nd
Ca	0.0084	nd
Mn	0.0006	nd
Fe	0.0019	0.005
Al	0.0184	nd
Si	0.0017	nd
B	nd	nd

*nd-not detected

the magnesium salt, precipitating agent, precipitating condition and ageing period²⁹⁻³¹. Variations in progressing conditions permit the production of a large number of sizes and shapes of magnesium basic carbonate crystals. The basic carbonate does not have the same impurities to the same extents as does the hydroxide, and on dehydration, the open crystalline lattice structure produces an oxide with high surface area and great chemical activity^{32,33}.

The X-ray diffractogram pattern of MgCO_3 in Fig. 1 shows four major peaks having intensity 100, 90, 50 and 30% at dA^0 values 5.809, 2.902, 2.15, 2.5 (2θ - 15.25, 30.8, 41.95, 36) respectively, that matches well with the dA^0 values of $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ as described in the JCPDS file. The reference Fluka magnesium carbonate sample shows similar diffraction pattern providing additional support for the prepared $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ sample. This is also further supported and confirmed by the IR and TG/DTA analysis.

The comparative study of IR spectra for the reference Fluka sample and the synthesized magnesium carbonate seems to be identical except some extra bands obtained for the latter case due probably to different preparation methods. The broad but strong band in the region $3400\text{-}3600\text{ cm}^{-1}$ in both the samples are assigned³⁴ to the $\nu(\text{OH})$ of both the coordinated water and coordinated (terminal) hydroxo groups. In this structure, CO_3^{2-} ions and H_2O molecules alternate statistically and the chains are cross-linked by means of hydrogen bonds between water molecules and CO_3^{2-} ions. Since the carbonate ion participates strongly in

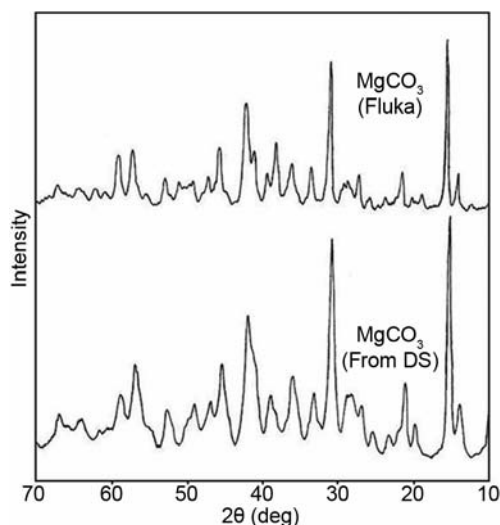


Fig. 1 — Comparative XRD pattern of prepared MgCO_3 and Fluka MgCO_3 .

the main bonding scheme, some reflection of this may be expected in the vibrational spectrum. The internal modes of the CO_3^{2-} ion of the magnesium carbonate compound matches well with the available data³⁴. As expected ν_1 appears as a single sharp band at 1122 cm^{-1} and ν_2 has three components at $892, 865$ and 829 cm^{-1} . The distinct doubling of ν_3 at 1485 and 1424 cm^{-1} and single sharp band for ν_4 at 749 cm^{-1} can also be accounted for CO_3^{2-} ions. The reference sample i.e. basic magnesium carbonate $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$ also shows similar observations, i.e. the presence of vibrational band at the same position and is compared with the prepared sample as shown in Fig. 2.

The thermal decomposition of the prepared magnesium carbonate along with Fluka reference sample was studied in the temperature range 30 to ca. 900°C as shown in Fig. 3. The prepared magnesium carbonate starts decomposing at about 35°C with the loss of coordinate water molecules and the loss of water molecule virtually completes by 350°C . The stoichiometry of the resultant intermediate species is assigned from the gradual weight loss, and they seem to be non-stoichiometric. The delegation process starts in a stepwise manner with the loss of coordinated H_2O molecule, OH molecules and CO_3^{2-} ions. The loss of OH molecule is characterised in multiples of two OH as H_2O molecule leaving one oxygen attached to Mg atom. Similarly, the loss of CO_3^{2-} ion is due to CO_2 release leaving one oxygen attached to Mg atom. At 284°C , two molecules of water have been lost and when 399°C temperature is reached three molecules of water along with another molecule of water from $\text{Mg}(\text{OH})_2$ are lost. The decomposition of CO_3^{2-} starts at after 399°C and between 399 - 692°C all the three CO_2 molecules are escaped out giving rise to the formation of 4MgO .

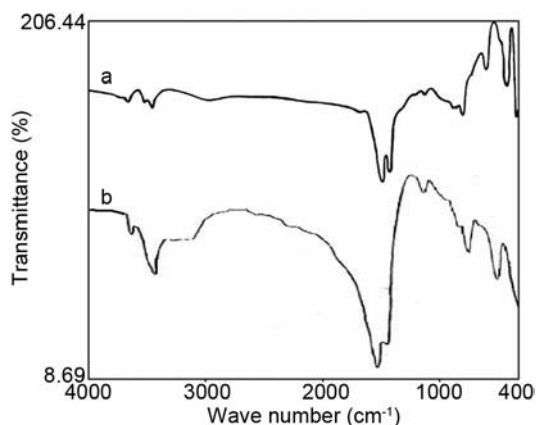


Fig. 2 — Comparative IR Spectra of MgCO_3 prepared from double salt (a) and Fluka MgCO_3 (b).

The DTA curve shows endothermic peaks at 258°C and 443°C for the decomposition of 3 molecule of water and one molecule of water along with decomposition of three molecules of carbonate, respectively. The TG/DTA curve for the reference Fluka MgCO_3 (Fig. 3b) also shows similar curve with three endothermic peaks at $248, 424$ and 489°C . The first peak is due to the loss of $3\text{H}_2\text{O}$ molecule and the other two peaks are due to the decomposition of $\text{Mg}(\text{OH})_2$ and MgCO_3 . Reported data on differential thermal curve for pure $\text{Mg}(\text{OH})_2$ shows an endothermic peak at $\sim 400^\circ\text{C}$, pure MgCO_3 at $\sim 700^\circ\text{C}$ and for $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ multiple peaks are observed due to the presence of H_2O molecule³⁵. The decomposition temperature of the Mg compound also varies due to the presence of water molecule. It is found that the differential thermal curves of prepared sample and the reference samples having multiple peaks do not match with the pure $\text{Mg}(\text{OH})_2$ and MgCO_3 . Thus it is confirmed that the prepared sample contains water molecule and is a hydroxy compound. The analytical and characterizing data initially indicate the formation of basic magnesium carbonate having general formula $x\text{MgCO}_3 \cdot y\text{Mg}(\text{OH})_2 \cdot z\text{H}_2\text{O}$ ³⁶. Subsequently on comparison with reference Fluka magnesium carbonate, the possible structure of the magnesium carbonate is confirmed to be $3\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 3\text{H}_2\text{O}$.

The average particle size D_{50} of prepared MgCO_3 is found to be 16.95μ . About 90% particle of this

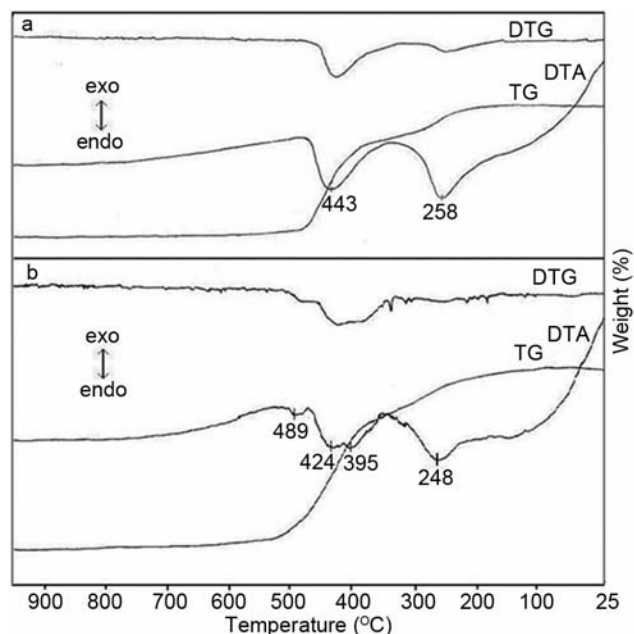


Fig. 3 — TG, DTA and DTG curves of MgCO_3 prepared from double salt (a) compared with Fluka MgCO_3 (b).

sample passes through 41.12μ and 10% particle passes through 3.88μ . But in Fluka magnesium carbonate particle sizes is higher and are 38.283, 83.041, and 9.357 for D_{50} , D_{90} , D_{10} , respectively. The optical surface area which is essentially the external area of the material of assorted size range is found to be $0.659\text{ m}^2/\text{cc}$ for the prepared MgCO_3 and is $0.478\text{ m}^2/\text{cc}$ for the fluka sample. However, the specific surface area of magnesium carbonate sample determined by the single point BET method by measuring the adsorption of nitrogen at liquid nitrogen temperature was found to be $17.57\text{ m}^2/\text{gm}$. Further, the scanning electron micrographs of MgCO_3 particles as shown in the Fig. 4 show a platelet structure (each plate $<10\mu$). Sometimes it shows globular structure looking like a cauliflower (a). Under high magnification the core of globule appears flaky and highly porous (c and d).

Characterisation of MgO

The chemical compositions of MgO obtained by calcination of magnesium carbonate prepared from double salt and Fluka samples are given in Table 3. The purity of sea water MgO is 99.08% which can be compared with the Fluka magnesium oxide of 99.265% pure. There is also absence of sulphate and boron in synthesised magnesia. The impurities are CaO - 0.027%, Al_2O_3 - 0.081% and Cl - 0.061% and the other impurities are in negligible concentration.

The X-ray diffractogram of the prepared magnesium oxide shows three well defined peaks of 10, 50 and 100% at 2θ value of 37, 62.5 and 43, which matches

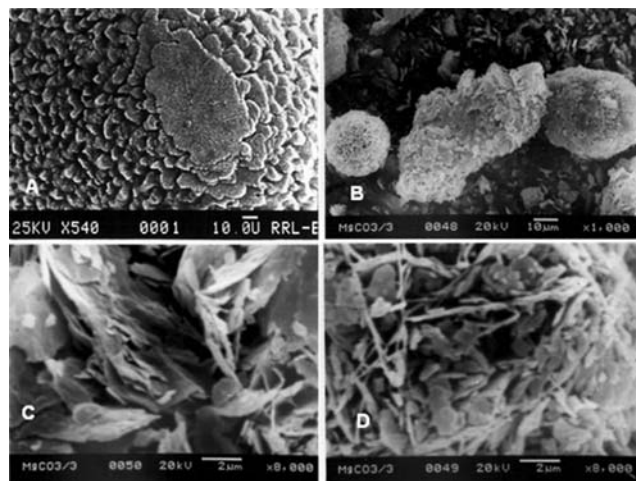


Fig. 4 — SEM micrographs of MgCO_3 prepared from double salt showing (a) platelets, (b) cauliflower shape, (c) flaky and porous nature of MgCO_3 under high magnification and (d) as above in a different sample.

well with the reported values of MgO as described in JCPDS file. These peaks show the presence of MgO and the major is periclase. Similar pattern of peaks are observed for Fluka MgO. Both the diffractogram matches well showing the formation of MgO.

In the IR spectra of prepared MgO and Fluka MgO, the important IR characteristic frequencies of MgO were observed and appropriately assigned. The absorption band at 3404 cm^{-1} shows the presence of adsorbed H_2O molecule. A sharp band observed at lower frequency of 419 cm^{-1} shows the presence of Mg-O vibration^{37,38}. At 1492 cm^{-1} a weak band is seen which corresponds to the presence of a trace of CO_2 . The reference Fluka sample also shows the presence of CO_2 which gives a weak band at 1514 cm^{-1} .

It is observed from the particle size analysis that the average particle size D_{50} of prepared MgO is 4.86μ . About 90% of the particle passes through 16.23μ whereas 10% of the material passes through 1.49μ . The specific surface area/optical surface area which is the outer surface area is found to be $1.8532\text{ m}^2/\text{cc}$. Similarly, MgO from Fluka shows particle size 9.914 , 30.581 and 0.955μ for D_{50} , D_{90} and D_{10} , respectively. The specific surface area is found to be higher i.e. $2.06\text{ m}^2/\text{cc}$. Thus, it can be noted that Fluka MgO of small particle size is having large surface area as compared to prepared sea water magnesia.

Low magnificant SEM photomicrographs of the magnesium carbonate powder at different temperatures are shown in the Fig. 5. With increase in calcination temperature, the platelet size of MgCO_3 gradually reduces. The porosity and looseness of the particles also decrease as the temperature increases. At highest temperature, the grains of MgO appears small and fused and sub-hedral in shape. Thus, it can

Table 3 — Chemical composition of MgO (in %)

Composition	Prepared MgO	Fluka MgO
MgO	99.08	99.265
CaO	0.027	nd
MnO_2	0.002	nd
Fe_2O_3	0.006	<0.169
Al_2O_3	0.081	nd
SiO_2	0.008	nd
Na_2O	0.008	nd
K_2O	0.001	nd
Cl	0.061	<0.0236
SO_4	nd	<0.709
B	nd	nd

*nd-not detected

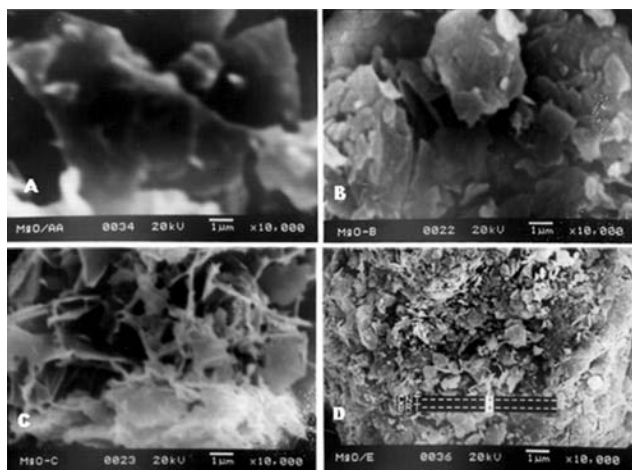


Fig. 5 — SEM micrographs of MgCO_3 calcined at (a) 400°C, (b) 600°C, (c) 800°C and (d) 1100°C.

be concluded that the magnesia has fine grain coagulated nature with a porous structure.

The BET specific surface area of the MgCO_3 particle is found to be 17.57 m^2/g . This MgCO_3 is calcined at different temperature shows that the surface area decreases with increase in temperature. The specific surface area at 400°C is 14.59 m^2/g and at 1000°C is 11.50 m^2/g . It is observed that the specific surface area increases slightly at a temperature of 1100°C. This is probably due to the formation of MgO via adsorption of CO_2 , a decomposition product during calcination of magnesium carbonate. Several investigators³⁹ have reported that the calcinations of MgCO_3 occurs between 600°C to 1000°C. The slight evolution of CO_2 still persists even after 1000°C. Hence, the adsorption of CO_2 by MgO particle is not ruled out. Slight increase in specific surface is also observed at 600°C. Since carbonate begins to decompose at 350°C and not completely decomposed within 600°C this anomaly is obvious.

Conclusion

Unlike known procedures of sea water magnesia entirely a new process, double salt route, has been developed⁴⁰ to overcome the operational difficulties already encountered by $\text{Mg}(\text{OH})_2$ process. Bittern of 29° Be' is subjected to magnesium recovery and no separate boron removal step is required except the addition of 2-3 drops of sorbitol in the last stage of washings. The magnesia recovered is almost free from boron along with other contaminants. The precipitating agent, ammonium sulphate, is recycled in the process to make the process economically

viable. It may be noted that in 100 mL scale the Mg recovery is 70.0%, whereas the Mg recovery is 73.82% in 1000 mL scale and the purity of magnesia is >99%. Hence, the double salt route provides a simpler and easy method of production of high pure magnesia.

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