

## Making of gypsum plaster in Bhutan – An experience

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*Received 13 February 2006; accepted 02 June 2006*

Paper deals with characterization of gypsum, its calcination to  $\beta$ -hemihydrate plaster, chemistry and constitution, setting and expansion. Configuration of Bhutanese gypsum plant and production of gypsum plaster by the process suggested by CBRI vis-à-vis to process adopted by the plant have been explained. Gypsum at 180°C with 1 h retention time gave plaster of better setting and strength properties acceptable to market complying relevant standards.

**Keywords:** Bhutan, Calcination, Gypsum plaster

**IPC Code:** E04F13/02

### Introduction

Gypsum occurs in several forms, most common among which is dihydrate- hydrated calcium sulphate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) found as selenite, alabaster and satin spar and anhydrite ( $\text{CaSO}_4$ ). In Bhutan, gypsum mines are located in north in Pemagachal area. Most of gypsum mined in this area is transported to Penden Cement Plant in Bhutan and other adjacent Indian States (West Bengal, Assam, Arunachal Pradesh, etc.). Rajasthan accounts for 95% production of total gypsum in India<sup>1</sup>. Considerable researches have been accomplished at Central Building Research Institute (CBRI), Roorkee, India, to identify the quantity and nature of impurities in waste phosphogypsum and other variety of by-product gypsum and to beneficiate them for the development and production of value-added building materials<sup>2-6</sup>. Several processes developed by CBRI have been accepted by the Indian industry.

In this paper, characterization of gypsum, its calcination to  $\beta$ -hemihydrate plaster, setting and expansion and chemistry and constitution have been discussed. Configuration of Bhutanese Gypsum plant and the production of gypsum plaster by the process suggested by CBRI vis-à-vis to process adopted by the plant to improve the properties of the plaster have been detailed.

### Properties of Gypsum

Pure dihydrate is white or colourless and has following chemical composition, expressed as oxides:

CaO, 32.5;  $\text{SO}_3$ , 46.6; and  $\text{H}_2\text{O}$ , 20.9 %. Its bulk weight is about 2300  $\text{kg/m}^3$  and hardness 1.5-2.0 on Moh's scale. It usually contains varying amount of clay, slate, anhydrite, chalk, dolomite, silica and iron as well as water. Pure anhydrite is white or sometimes gray, blue, red, or brown in pale shades due to presence of certain impurities. Its chemical composition, expressed by weight as oxides, is: CaO, 41.2; and  $\text{SO}_3$ , 58.8 %. Its bulk weight is about 3000  $\text{kg/m}^3$  and its hardness 3.0-3.5 on Moh's scale. The requirements of mineral gypsum needed for different gypsum industries are listed in Table 1.

### Plaster of Paris/Calcined Gypsum

Plaster is produced from calcination of gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) that partially dehydrates to produce a hemihydrate ( $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ ) [ $\text{CaSO}_4 \cdot 2\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 1/2\text{H}_2\text{O} + 3/2 \text{H}_2\text{O}$ ].

### Production of Plaster at Bhutan – CBRI Study

#### Type of Bhutan Plant

M/s Durk Satair Corporation, Ltd, Samdrup Jongkhar, Bhutan produces plaster of Paris in the Gypsum plant located at Khothakpa, Pemagatchal. The objective of CBRI was to regulate setting time of the plaster, which was fast setting (5-7 min). The required setting time of plaster for commercial application was 10-12 min without addition of any chemical retarder or additive.

Gypsum plant comprised of 6 units (capacity per unit, 1 ton/ 4 h) installed in a battery. Natural gypsum of high purity, collected from Bhutanese mines, is snow white and used for the production of plaster of

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Table 1— Specifications of mineral gypsum in different industries<sup>10</sup>

Constituents	Surgical plaster, %	Ammonium sulphate fertilizer, %	Pottery, %	Cement, %	Reclamation of soil, %
CaSO <sub>4</sub> .2H <sub>2</sub> O*	96.0	85-90	85.0	70-75	70
Free water	1.0	---	1.0	---	---
CO <sub>2</sub> **	1.0	---	3.0	---	---
SiO <sub>2</sub> and other insoluble matter**	0.7	---	6.0	---	---
Fe <sub>2</sub> O <sub>3</sub> +Al <sub>2</sub> O <sub>3</sub> **	0.1	1.5	1.0	---	---
MgO**	0.5	1.0	1.5	3.0---	---
NaCl**	0.01	0.003	0.1	0.5---	---
Na <sub>2</sub> O*---	---	---	---	0.7 Na	---
Fineness**	---	---	---	---	Residue on 2 mm sieve, nil & on 0.2 mm sieve, 50

Table 2—Chemical composition of Bhutan gypsum

Constituents	%
SiO <sub>2</sub>	8.0-9.62
Al <sub>2</sub> O <sub>3</sub>	0.51-1.0
Fe <sub>2</sub> O <sub>3</sub>	0.03-0.5
CaO	28.0-30.0
MgO	0.16-0.20
K <sub>2</sub> O	0.04-0.6
Na <sub>2</sub> O	0.16-0.18
SO <sub>3</sub>	41.15-43.90
CaSO <sub>4</sub> .2H <sub>2</sub> O	88.0-92.0
Moisture	6.0-8.0
Loss on ignition	12.7-14.0

Paris. Each unit of gypsum calcinator consists of two major assemblies (1) Calcinator and (2) Furnace. Calcinator comprises of a cylindrical shell (height 5' 6", diam 4.6") fitted with two pairs of churning blades on the stirrer shaft with chain at the bottom, bevel and pinion duly electrified through 20 hp motor and reduction gear box producing 18 RPM.

The production cycle comprised of crushing of gypsum boulders, pulverization and calcination. Ground gypsum (size, 1 mm sieve) is taken to storage bin (capacity, 20 ton) and fed to each calcinator through screw conveyor. Optimum temperature of calcination was 120-165°C. Duration of calcination was 5.0 h per charge. Calcined material is finally ground in the pulverizer (capacity 2 ton/h) before bagging.

#### Setting Time Studies

Setting time of the plaster was 5-7 min. Whole operation was pondered upon and found that gypsum pebbles/pieces (size, 1.5-2.5 cm) retained in the gypsum feed were main cause of rapid setting of the plaster. It was decided to remove gypsum pebbles from gypsum feed so that they may not enter into calcinator as these pebbles remain under calcined in the plaster and act as an activator for plaster, resulting into rapid crystallization of the plaster and thus fast

setting of gypsum plaster takes place. Plaster samples were brought to CBRI for DTA and SEM studies.

Bhutanese gypsum (Table 2) was taken for calcination trials. Composition of gypsum falls in the category of pottery to fertilizer grades (Table 1). To get plaster of required setting time, calcination trials of gypsum powder (without gypsum pebbles) were undertaken in calcinator. Calcination temperature was raised to 180°C gradually. When temperature approached 180°C, burners were minimized to retain temperature for 1 h so that soluble anhydrite ( $\gamma$ -CaSO<sub>4</sub>) may be formed. The appearance of soluble anhydrite tends to enhance the setting time by coating the plaster of Paris. Mineralogical composition of the plaster samples was done as per reputed method<sup>7-8</sup>. Gypsum plasters produced by several calcination trials was ground to pass 90  $\mu$  Indian sieve and then tested as per IS 2542 (Part1)-1978<sup>9</sup> for various physical properties.

#### Results and Discussion

Almost all the plaster samples contain quite high amount of under calcined gypsum, which is responsible for the fast setting of the plaster (Table 3). Instead of discharging plaster at 165-170°C (usual temperature), calcination temperatures was increased to 180°C and retained for 1 h at that temperature. The plaster on evaluation showed an improvement in the hemihydrate as well as the insoluble anhydrite contents. It was noted that the gypsum content was minimized to improve the hemihydrate content. Increased quantity of soluble anhydrite forms a coating over hydrating hemihydrate particles and thus improves its setting time (Table 4) as the soluble anhydrite is a slow setting compound, which temporarily suppresses hydration of plaster to help in lengthening the setting of the plaster. It can be further seen that consistency of the plaster is reduced leading to increase in bulk density and compressive strength of the plaster.

Table 3—Mineral compositions of plaster samples produced in gypsum plant at 120-165°C, (Practice being followed at the plant)

Plaster design	$\beta$ - Hemihydrate, %	Soluble anhydrite, %	Insoluble anhydrite, %	Gypsum, %
Sample I	75.6(85.6)*	4.0 (9.0)*	2.00 (3.00)*	18.40 (2.40)*
Sample II	75.0 (85.0)	6.0 (11.0)	2.00 (2.10)	17.00 (1.45)
Sample III	77.0 (87.0)	7.0(11.5)	1.00 (1.00)	15.00 (0.50)
Sample IV	76.0 (86.0)	6.5 (10.5)	1.00 (2.00)	16.50 (1.50)
Sample V	76.0 (86.0)	7.0 (10.0)	2.50 (2.50)	14.50 (1.50)

\*Figures in parenthesis are at 180°C (Modified process)

Table 4 — Properties of plasters produced at different temperatures

Plaster design	Consistency, %	Setting time, Min	Bulk density, g/cc	Compressive strength, MPa
Sample I	59.0 (55.0)*	4.0 (9.00)*	1.27 (1.31)*	5.34 (9.400)*
Sample II	60.0 (49.0)	8.0 (12.0)	1.224 (1.45)	5.35 (10.60)
Sample III	54.0 (50.0)	7.0 (14.0)	1.239 (1.37)	5.13 (10.08)
Sample IV	58.0 (50.0)	9.0 (12.5)	1.239 (1.38)	4.95 (11.76)
Sample V	60.0 (53.0)	8.0 (12.0)	1.21 (1.55)	5.80 (10.85)

\*Figures in parenthesis are at 180°C

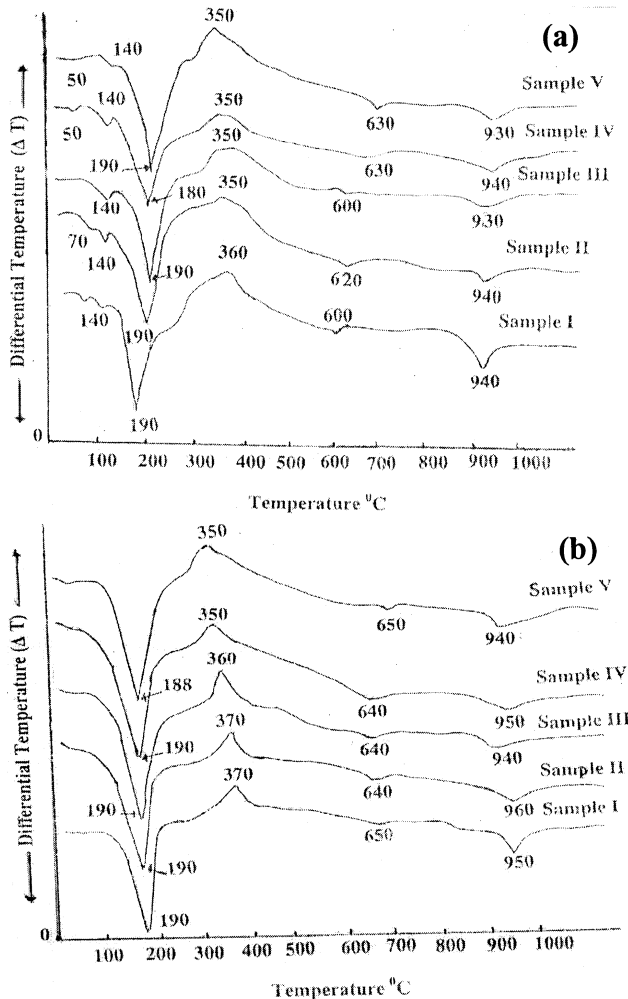


Fig. 1— DTA of calcined gypsum/plaster samples: a) Bhutan methods; b) CBRI Method

Calcination results were confirmed by differential thermograms (DTA) of the plaster samples produced in the plant (Fig.1a). Endotherms at 50-70°C show presence of gypsum in the plaster samples. Endotherms at 140 and 180-190°C indicate double dehydration peaks for inversion of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  into  $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$  and  $\text{CaSO}_4$  ( $\gamma$ ) phases respectively. Gypsum plaster sample V showed largest endotherm at 190°C due to enhanced formation of hemihydrate plaster. Exotherms at 350-360°C denotes inversion of  $\text{CaSO}_4$  ( $\gamma$ ) into  $\text{CaSO}_4$  ( $\beta$ ). Endotherms formed at 600-630°C and at 930-940°C are due to dehydroxylation of kaolinite mineral and decomposition of  $\text{CaCO}_3$  present in the plaster samples.

Absence of endotherms at 50-70°C in DTA of plaster samples produced by CBRI method shows disappearance of uncalcined gypsum (Fig. 1b). Intensity of hemihydrate endotherms (188-190°C) as well as exotherms (350-370°C) increases over the intensity of hemihydrate peaks obtained in case of plasters produced by the process adopted by the plant (Fig. 1a), confirming thereby, increased conversion of gypsum into plaster. There was slight shifting of endotherm temperatures due to clay mineral kaolinite (640-650°C) and decomposition of  $\text{CaCO}_3$  (940-960°C). This may be ascribed to better calcination of gypsum and fine grinding of the raw feed.

SEM of the plasters produced by methods as adopted by the plant (Fig. 2a) shows formation of anedral to subhedral gypsum plaster crystals with twinning and variable sizes. Stacking and distribution of crystals is not uniform showing under calcination

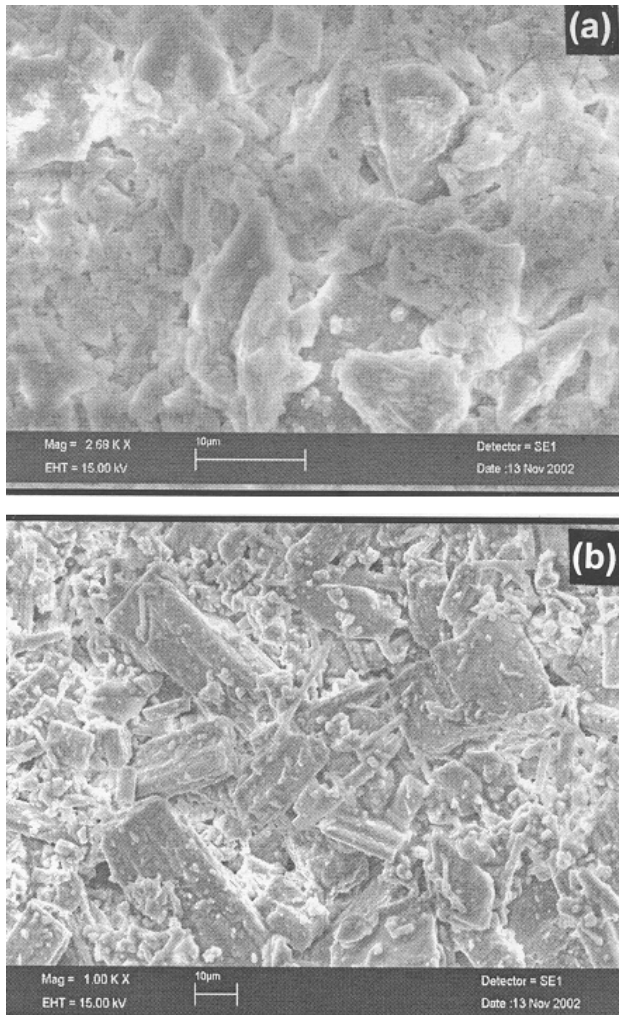


Fig. 2— SEM of typical calcined gypsum/plaster: a) Bhutan method; b) CBRI Method

of gypsum. Whereas, formation of subhedral to euhedral prismatic crystals with lath and tabular habit showing proper stacking and distribution indicate formation of properly calcined plaster (Fig. 2b).

### Conclusions

Quality of mineral gypsum available in Bhutan is of high purity but  $\beta$ -hemihydrate plaster being produced at Druck Satair Pemagatchal Plaster Plant is not

consistence due to improper calcination of gypsum. Coarse fraction (gypsum pebbles, 1.5-2.5 cm) present in gypsum feed was responsible for the poor calcination of gypsum. It was found to be under calcined thus spoiling quality of the plaster. Removal of coarse fraction from the feed and calcination of gypsum at 180°C with 1 h extra retention time gave plaster of better setting and strength properties acceptable to market complying relevant standards. Coarse fraction was recommended to be mixed with raw gypsum before grinding.

### Acknowledgements

Author thanks Director, CBRI, Roorkee for allowing publication of the paper. Thanks are also due to M/s Durk Satair Corporation Ltd, Samdrup (Bhutan) for sponsoring the visit of author to Bhutan to carry out the study.

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