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Characterization of Limestone Sample from Northeast India and its Implication on Beneficiation

B. Nayak and **K.K. Bhattacharyya**

Mineral Processing Division, National Metallurgical Laboratory, Jamshedpur-831007

Abstract

Two limestone samples, from the same source (Assam, northeast India) have been petro-mineralogically characterized. In hand specimens, both samples are quite similar and are highly fossiliferous. Under optical microscope the fossils that are identified belong mostly to two different Phyllum, viz., Protozoa and Mollusca. The Protozoans are mainly Foraminifera and the Molluscans are Lamellibranchia. Both categories have been petrified to calcium carbonate only and the fossils are embedded in a lime matrix. Due to the dominance of Nummulite fossils, the samples can be named as 'Nummulitic limestones'. The fossil records indicate that the limestones may be of Eocene age (< 65 million years).

Under the microscope calcite (with fine grained aragonite) was observed to be the dominating mineral phase. However, trace occurrence of fine-grained quartz, clay, authigenic mica and chloritic mineral has been observed. X-ray diffraction studies did not reveal the confirmatory presence of any other mineral. The grain size in a sample is highly irregular varying from less than 10 □m to more than 1.5 mm. The aragonite has primarily a cryptocrystalline look and is extremely fine grained. At places the samples have an earthy look. This may be due to the extraneous impregnations of iron- or alumina-rich solutions that dilute the purity of the samples. The petrographic observations supported by chemical analysis indicate that these are very high-grade limestones with less impurities (<4%). These limestones hardly require any beneficiation. If further improvement is required, then very fine grinding (below 325 mesh; i.e, less than 45 microns) has to be adopted to liberate the gangues. It is expected that in size fractions, the finer fraction shall have the highest impurity and the coarser fractions shall be relatively pure. The limestone may be used as a flux in iron and steel industry and may find use in manufacturing chemical grade lime required for refining of sugar, paper making and bleaching of textiles etc.

Keywords: *Fossiliferous Limestone, Mineralogical characterization, Nummulite, Assam.*

INTRODUCTION

Two limestone samples, named as Sample-1 and Sample-3 weighing about 22 kg each, were received from Regional Research Laboratory-Jorhat for characterization studies. The limestone samples thus received were studied for petrographic characters, chemically analyzed and exposed to X-ray diffraction. The compressive strength of the limestone samples were also determined that are discussed in the following paragraphs. For convenience, the abbreviation LS-1 and LS-3 have been used for limestone sample-1 and limestone sample-3 respectively.

MEGASCOPIC (HAND SPECIMEN) CHARACTERS

In hand specimens, both LS-1 and LS-3 are quite similar; they are highly fossiliferous. These are light-grey in colour. However, LS-3 appears more brownish than LS-1. This may be an effect of the extraneous iron-rich solutions on the sample due to surficial exposure. The average size of the rock-

fragments in LS-1 is small (max: 70 mm) whereas in LS-3 it is bigger and some pieces range between 80 to 110 mm. In hand specimens, fossils can be seen ranging in size from half a mm to around 1.5 cm in size. The fossils are mostly spindle shaped, often spherical (oolitic to pisolitic) or oval, and sometimes niddle like skeletal structures. Broadly the fossils are identified as foraminifers (nummulites). Due to the high abundance of the nummulites, these limestones can be called as "Nummulitic Limestones"

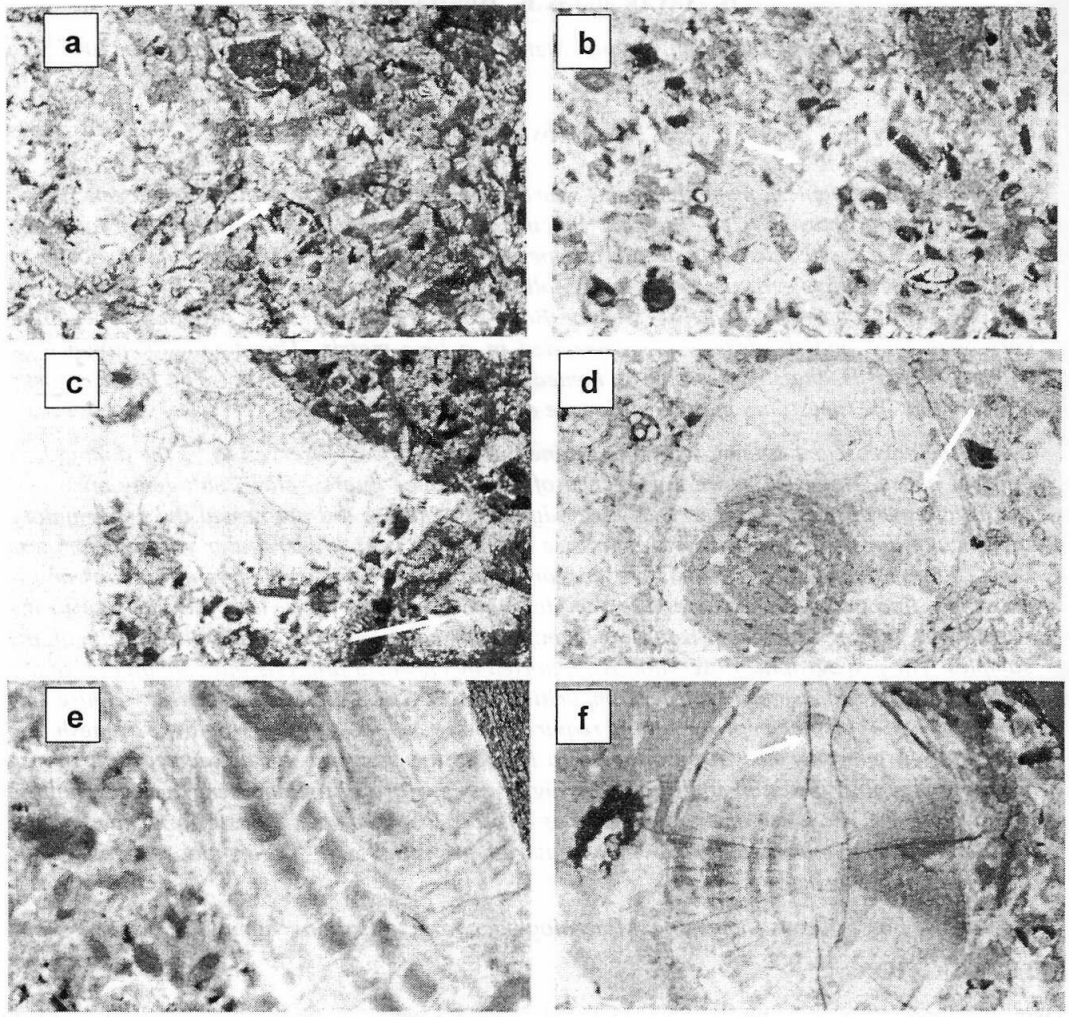


Fig. 1: Optical Photomicrographs of Limestone Sample-1 (LS-1). Photographs are Taken at a Magnification of 5X and Under Partially Crossed Nicols. the Longer Edge of each Photograph Equals to 2.4 Mm. Important Features Described are Marked by Arrows.
(A): A General View of the Limestone Having an Earthy Look; (B): In the Central Portion are Pure Calcite Crystals; (C): Large Calcite Crystals (>1mm) at the Central Portion with some Authigenic Micaceous Mineral at Its Border (Arrow); (D): A Fossil Ooid That Has Almost Been Recrystallised to Calcite with some Inclusions of Quartz (Arrow); (E): A Nummulitid in Which the Internal Structure of the Test is Well Preserved; (F): A Large Nummulite Shell (Nummulites Atacicus ?) That Has Developed some Cracks (Arrow)

MICROSCOPIC CHARACTERS

A number of thin sections (slides) were prepared from LS-1 and LS-3 for optical microscopic studies. Under the plane polarized light and under crossed nicols, calcite with some aragonite were observed to be the dominating mineral phase that covers about 98% of the slide-area. Calcite occurs mainly as authigenic crystals on various shell (fossil) fragments and very often recrystallized. It is mostly fine-grained ranging in size from 10 μm to about 150 μm. However, larger crystals are very often found where the recrystallisation is more prominent and the grain size exceeds 400 μm. Even crystals greater than 1.5 mm are occasionally recorded. The aragonite has primarily a cryptocrystalline look and is extremely fine grained, only few microns. The grain size of aragonite sometimes is too fine to be measured in optical microscope. At some places the sample has an earthy look. This may be due to the extraneous impregnations of iron- or alumina-rich solutions that dilute the purity of the samples. The presence of any gangue mineral is very rare. The presence of very fine-grained authigenic quartz (approximately 40-50 microns) and micaceous or chloritic impurity is recognized only locally.

The samples contain numerous fossils and the fossils, that are identified, belong mostly to two different Phyllum, *viz.*, Protozoa and Mollusca. The Protozoans are mainly Foraminifera and the Molluscans are Lamellibranchia. Both categories have been petrified to calcium carbonate only and the fossils are embedded in a lime matrix. The foraminiferal tests are either unilocular (with one chamber) or multilocular and both benthonic and pelagic/planktonic forms are recognized. The different species of Foraminifera recorded are *Gumbelina globosa, Discocyclina, Lockhartia tipperi, Spiroloculina, Globogerina, Nummulites atacicus, Nummulites lahirii, Nummulites atacicus, Nummulites nuttali, Miscellania miscella, Assilina dandotica.* The few identified Lamellibranchia shells are *Pecten* and *Ostrea.* In addition bacterial remains, organic filaments and other skeletal structures are also preserved in the samples. The occurrence of fossils has been documented in Figs. 1 to 2. While Fig. 1 represents the fossils recorded in limestone sample-1, Fig. 2 represents the fossils of limestone sample-3. All these fossil records indicate that the limestones may be of Eocene age (< 65 million years).

LIBERATION CHARACTERISTICS

The petrographic observations indicate that these are very high-grade limestones with less gangue minerals and hardly require any beneficiation. If further improvement is required, then very fine grinding (below 325 mesh) has to be adopted to liberate all gangues. Since the gangues/impurities are having a finer grain size (less than 45 microns), it is expected that in size fractions, the finer size fraction shall have the highest impurity and the coarser fractions shall be relatively pure.

X-RAY DIFFRACTION STUDIES

Fine powders (below 75 microns) of the limestone samples were exposed to x-ray diffraction using a Seifert make x-ray diffractometer (model: XRD 3003 PTS) with Co-target. The X-ray diffractograms (Figs.3 and 4) are almost identical showing the peaks of only one mineral, i.e, calcite. Minor peaks of very low intensity are observed but could not be identified conclusively. These may be the peaks of some clay minerals whose abundance is very low in the sample.

CHEMICAL COMPOSITION OF THE LIMESTONE SAMPLES

Both the limestone samples are highly reactive to dilute hydrochloric acid and immediately produce effervescence confirming their carbonate-composition. The shells / tests of the fossils are also composed of calcium carbonate and are highly reactive to dil. HCl. A detailed chemical analysis [except loss on ignition (LOI)] by weight-chemical gravimetric method reveals that the limestone samples are of very high quality with lower concentration of impurities. The analytical results are presented in table-1.

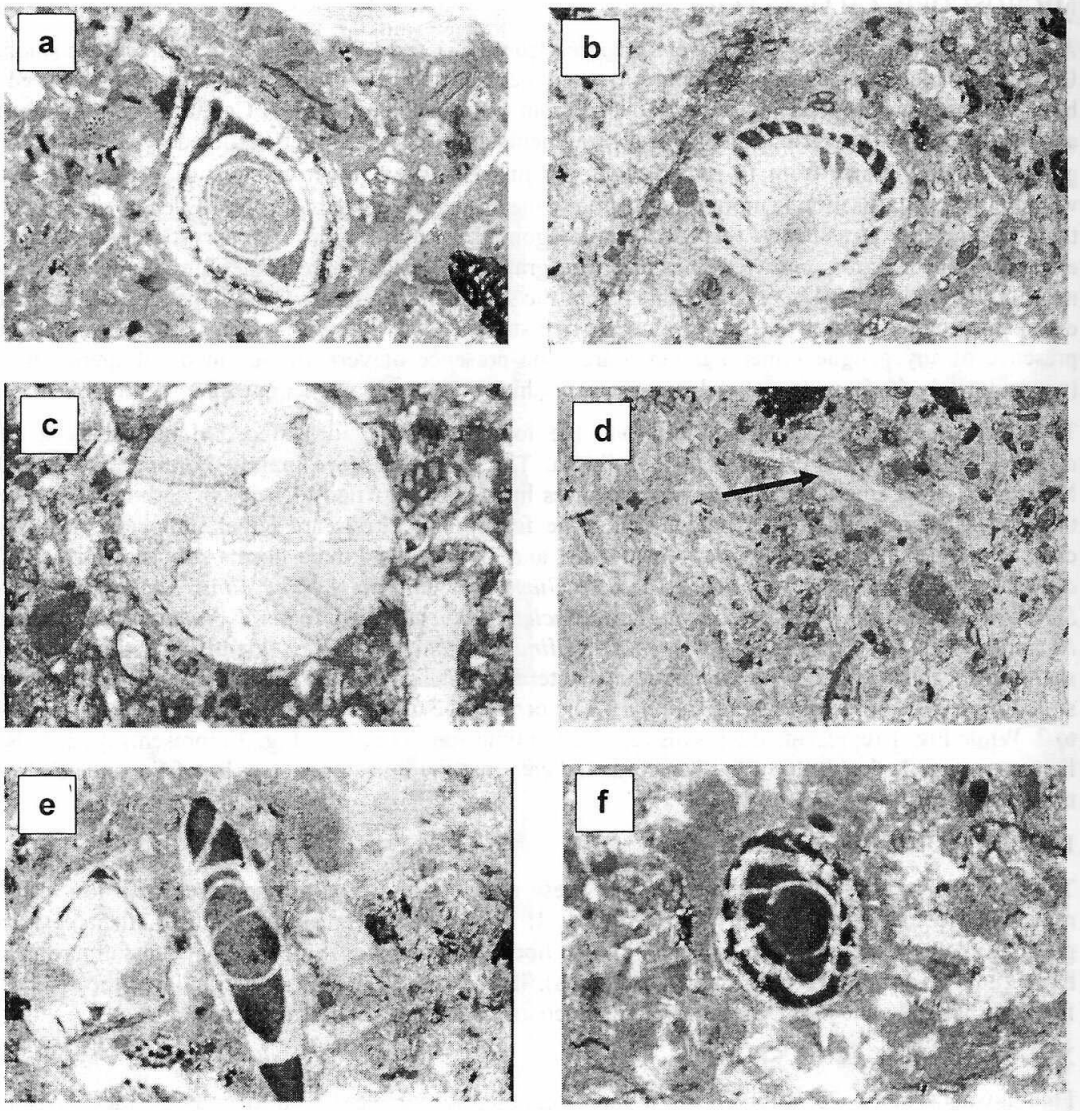
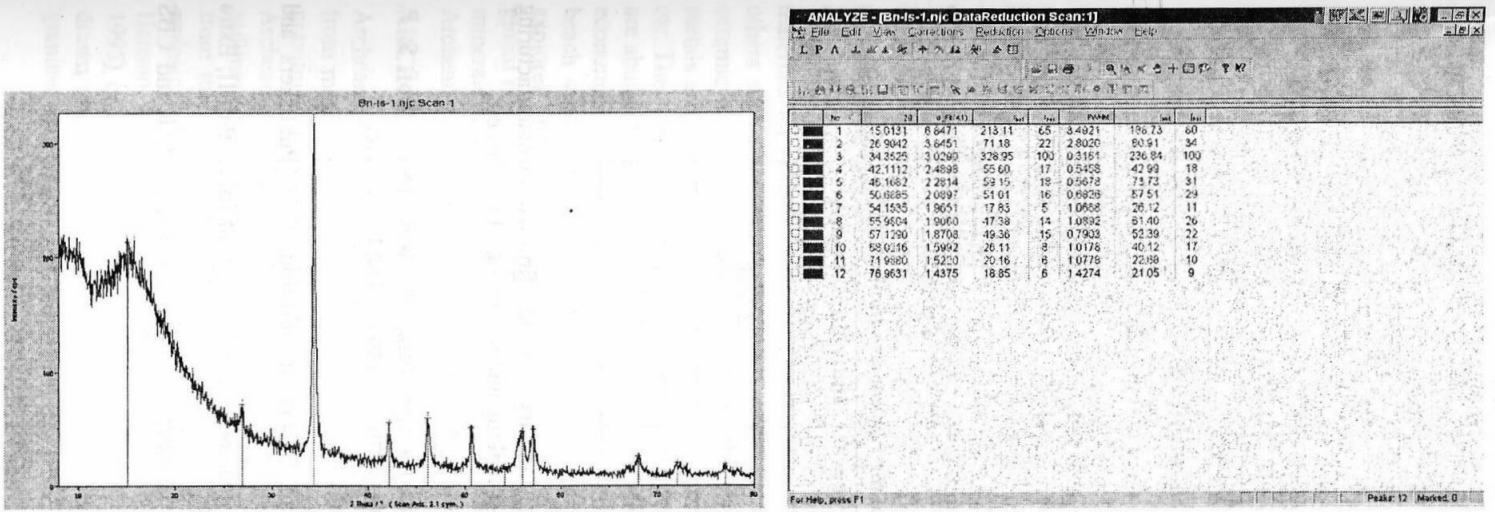


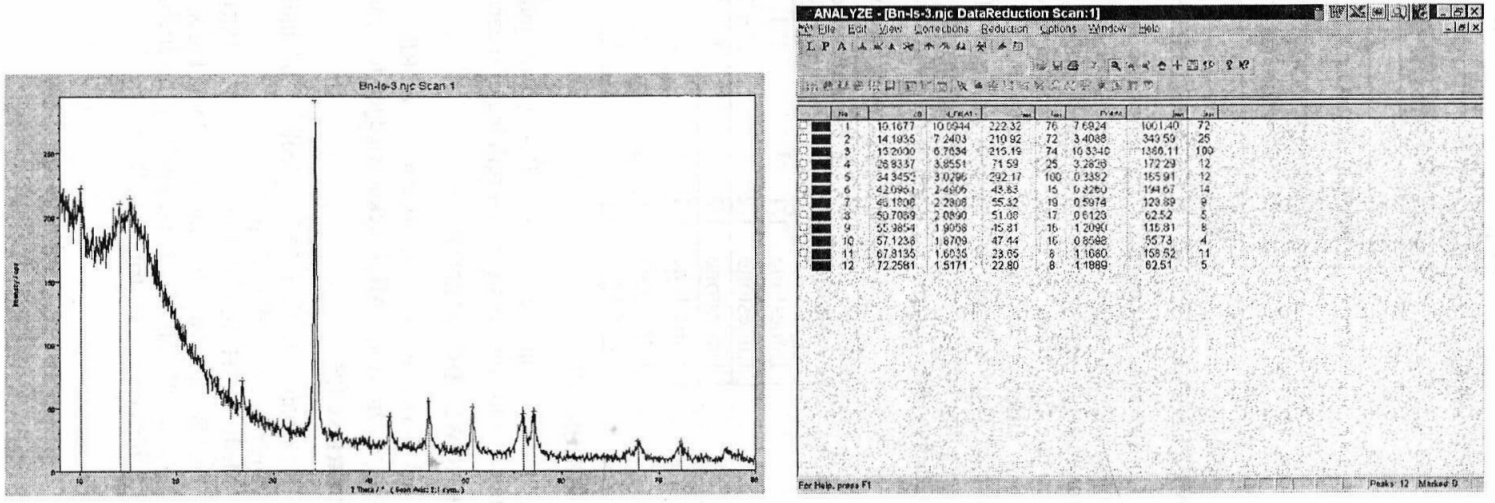
Fig. 2: Optical Photomicrographs of Limestone Sample-3 (LS-3). Photographs are Taken at a Magnification of 5X and Under Partially Crossed Nicols. the Longer Edge of Each Photograph Equals to 2.4 mm. Important Features Described are Marked by Arrows.
(A): A Fossil of Nummulites Lahirii at the Centre of Which are Seen Microcrystalline Calcite in Form of an Ooid; (B): A Shell of Nummulites Nuttali; (C): A Fossil Shell, May be of Discocyclina That Has Completely Been Replaced by Calcite; (D): Numerous Micro-Fossils and an Wedge-Shaped Filament (Arrow) That Has Been Converted to Calcite; (E): A Fossil of Nummulites Atacicus (Left Side) and a Shell of Assilina Dandotica (Darker Shell in the Middle); (F): A Large Test of Globigerina is Seen in the Middle Portion of the Photograph

It can be observed from table-1 that the overall impurity level of LS-3 is higher than that of LS-1. The surficial brown colouration of LS-3 which may be due to extraneous iron-rich solutions is responsible for the higher level of iron and alumina in LS-3.



No	2θ	d (Å)	Net	Int.	Area	Int.
1	15.0131	6.8471	218.11	65	3.4921	186.73	60
2	26.9042	3.6451	71.18	22	2.8020	60.91	34
3	34.3525	3.0293	328.95	100	0.3161	236.84	100
4	42.1112	2.4898	55.60	17	0.5458	42.99	18
5	46.1682	2.2814	59.15	18	0.5678	73.73	31
6	50.6685	2.0897	51.01	16	0.6826	67.51	29
7	54.1535	1.9651	17.85	5	1.0668	26.12	11
8	55.9504	1.9060	47.38	14	1.0892	61.40	26
9	57.1290	1.8708	49.36	15	0.7903	52.39	22
10	58.0216	1.5992	26.11	8	1.0178	40.12	17
11	71.9880	1.5220	20.16	6	1.0778	22.88	10
12	76.9631	1.4375	18.85	6	1.4274	21.05	9

Fig. 3: X-Ray Diffraction Pattern of LS-1 Showing Peaks of Calcite



No	2θ	d (Å)	Net	Int.	Area	Int.
1	10.1677	10.0944	222.32	76	7.6824	1001.40	72
2	14.1935	7.2403	210.92	72	3.4088	349.59	25
3	15.2000	6.7634	215.19	74	10.8340	1366.11	100
4	26.8337	3.8551	71.59	25	3.2828	172.29	12
5	34.3452	3.0296	292.17	100	0.3382	155.91	12
6	42.0961	2.4866	45.83	15	0.8260	194.67	14
7	46.1606	2.2908	55.32	19	0.5974	129.89	9
8	50.7089	2.0890	51.08	17	0.6126	62.52	5
9	55.9854	1.9058	45.81	16	1.2090	115.81	8
10	57.1238	1.8709	47.44	16	0.8598	55.73	4
11	67.8135	1.6035	23.65	8	1.1680	158.52	11
12	72.2581	1.5171	22.80	8	1.1889	62.51	5

Fig. 4: X-Ray Diffraction Pattern of LS-3 Showing Peaks of Calcite

Table 1: Chemical Analysis of the Limestone Samples

Radicals; Wt.%	Sample - 1	Sample - 3
CaO	53.49	52.29
MgO	0.47	0.49
SiO_2	1.96	1.89
Al_2O_3	0.49	0.63
Fe_2O_3	0.68	0.75
P_2O_5	0.025	0.0254
Na_2O	0.033	0.030
K_2O	0.051	0.082

COMPRESSIVE STRENGTH OF THE LIMESTONE SAMPLES

The **Compressive Strength** of the limestone samples were determined using a compression testing machine and following the Indian Standard IS: 3495 (1976). Cubes of the rock (2" X 2" X 2" approximately) were prepared and tested applying the load axially at a uniform rate of 140 kgf/cm^2 per minute till failure occurred. The rock samples were carefully chosen with minimal flaws and tested in different directions. The results thus obtained varied from a minimum 139.7 kgf/cm^2 to a maximum 331.4 kgf/cm^2 with an average figure of **272.4 kgf/cm^2** for LS-1 and **222.7 kgf/cm^2** for LS-3. These figures may be slightly in the lower side because perfect cubes could not be prepared due to limitations in the rock pieces in the as-received sample.

Table 2: Compressive Strength of the Limestone Samples

Sample No.	Compressive Strength (kgf/ cm^2)	Individual Average (kgf/ cm^2)
Limestone – 1/1	331.4	272.4
Limestone – 1/2	276.2	
Limestone – 1/3	209.5	
Limestone – 3/1	259.7	222.7
Limestone – 3/2	268.6	
Limestone – 3/3	139.7	

CONCLUSION

The limestone may be used as a flux in iron and steel industry and may find use in manufacturing chemical grade lime required for refining of sugar, paper making and bleaching of textiles etc.

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