J. Indian Chem. Soc.,
Vol. 93, April 2016, pp. 437-442

Bentonite minerals and its TGA, DSC and PXRD studies

Ashok Kr. Jha

Department of Chemistry, G. B. College, (T.M. Bhagalpur University), Naugachia-853 204, Bihar, India

E-mail : ashokjha39@gmail.com

Manuscript received 04 August 2014, revised 06 January 2016, accepted 06 January 2016

Abstract : Bentonite is hydrated alumina-silicate clay composed of the smectite class mineral Montmorillonite. The deposits are widely distributed in Rajmahal Hills of Jharkhand. A large number of samples collected from different places available in different colours revealed the fact that they are good ion exchangers. Some of them gave positive stain test with benzidine. Percentage of alumina and silica known for different samples accounted for different adsorption behaviour. These clay minerals have a layered structure based on sheets with 6-membered rings of silica tetrahedral formed by the condensation of sheets of silica tetrahedral with sheets of Al or Mg octahedra. Investigations on thermal behaviour carried out using TGA, DSC experiments present almost a loss of mass about 18.9% showing the loss of molecular water and dehydroxylation. X-Ray diffraction patterns of the samples consist peaks of Montmorillonite minerals. The mineral has been considered to constitute an interstratified mineral with a structure composed of Kaolin and Montmorillonite layers. The proportion of Montmorillonite layers is abundant in the inner part, but at the margin the layers of Kaolin are present.

Keywords : Bentonite, TGA, DSC, PXRD.

Introduction

With a view to assess the properties, bentonite samples from different places from Rajmahal Hills have been collected. Rajmahal Hills rise abruptly from places forming a wall of 1000′ to 2000′ height which just cut into the Gangetic valley. The samples of some places of Rajmahal Hills have not been thoroughly studied till now. So the samples have been collected from important places e.g. Madro fossil park, foot of the Hill near Jawahar Navodaya Vidyalya, Sahibganj, Kendua Bishanpur situated 7 km from Barharwa Junction. Bentonites available in the range of Rajmahal Hills are nontoxic and low cost adsorbent[2]. Bentonite is an absorbent aluminium Phyllosilicate consisting mostly of Montmorillonite[3,4]. Other minerals present in the Hills are Kaolinite, Illite and Vermiculite. Most of the samples responding to positive stain test to benzidine solution are Montmorillonite. The Montmorillonite samples are good adsorbent[5,6]. This is the reason that the bentonites of this area have great industrial utility though not utilized properly. Physico-chemical studies have shown pH and swelling power. A single layer of Montmorillonite consists of two tetrahedral silica sheets and one octahedral sheet. The adsorption capacity of the mineral is dependent on the layer charge[7–9]. TGA and DSC studies showing loss of mass of 18.9% between 296 and 1173 K help understand the structure of bentonite[10–13].

The PXRD analysis important for the clay minerals make understand the diffraction characteristic of bentonites[11–13].

These studies open the vast scope of industrial utility of bentonite minerals. Due to higher proportion of exchangeable cations of sodium and magnesium, bentonites are exploited for softening of hard water[14,15]. Bentonites are ecofriendly, cheap and low cost adsorbent for the removal of fluoride from water. Wide range of bentonites of different colour and grade can be exploited for industrial utility e.g. wine treatment decolorisation and adsorption of radioactive wastes.

Experimental

The clay mineral used in the present work has been collected from different places of Jharkhand and tested for Montmorillonite by benzidine solution. The finely powdered bentonite to 300 μm is dried over 1 h. The thermal behaviour of the bentonite has been carried out using TGA and DSC experiments showing weight loss at different temperature. DSC developed by Perkin-Elmer USA has been used.

The PXRD studies of the dried samples have been done at 0_2 Theta position using Cu as anode material. The diffractometer type used was D8. Specimen length was fixed at 10 nm and receiving slit size was 0.1000 mm. RHB1 and RHB3 stands for bentonite sample of Madro fossil park and Kendua Bishanpur respectively.

Results and discussion

The bentonites presented a total loss of mass of about 18.9% between 373 K and 1073 K (Fig. 3). The first weight loss is explained due to sample hygroscopicity i.e. the loss of molecular water from the exchange layer[10]. The second loss of weight occurs due to the removal of water composition from the bentonite. The third weight loss is due to the dehydroxylation of the silicate lattice. Some samples display a total weight loss of 5.539% which is different from bentonite clear from Fig. 4. These samples may be Kaolinite or Illite. Fig. 1 and Fig. 2 show the DSC curves of RHB1 and RHB3 at Fig. 1 and Fig. 2 show the DSC curves of RHB1 and RHB3 at different temperatures. The endothermic reaction indicates the presence of Montmorillonite layer. When an endothermic transition occurs; the energy absorbed by the sample is compensated by an increased energy input to the sample in order to maintain a zero temperature difference. The abscissa indicates the transition temperature and the peak area measures the total energy transfer to the sample. Decomposition of the sample showing DSC endotherm indicates the presence of impurities along with Montmorillonite.

The PXRD analysis of some samples of bentonites have been carried out shown in Figs. 5 and 6, which is liable to be confused with a basal reflexion from chlorite or vermiculite. Imperfections of the crystal affect the diffraction characteristic and important for the clay mineral. This may be due to highly symmetrical arrangement of the atoms in the various layers and weak binding force between them. The layers may be displaced with respect to one another.

Another imperfection is due to the fact that clay mineral layers are very similar having silica tetrahedral sheets and closely packed layers of hydrogen and hydroxyls.

The d values of X-ray powder data of some bentonite minerals of Rajmahal Hills have been reported which is in agreement with previous reported values in the literature. It can also be concluded that Montmorillonite clay mineral are present in all the samples along with Kaolinite in small proportion.

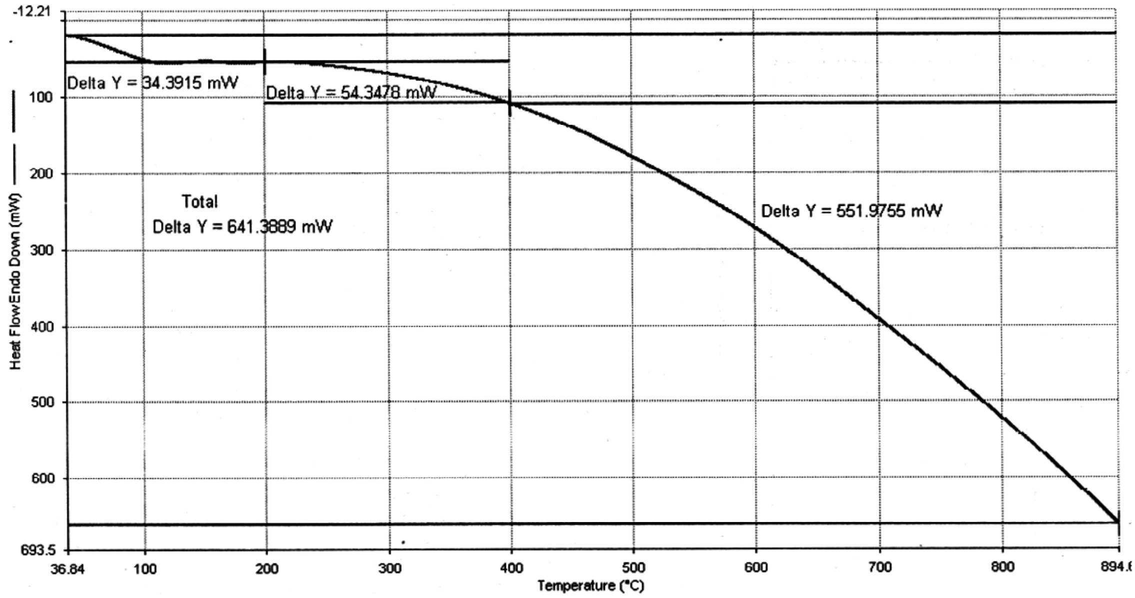

Fig. 1. Only DSC curves of RHB1.

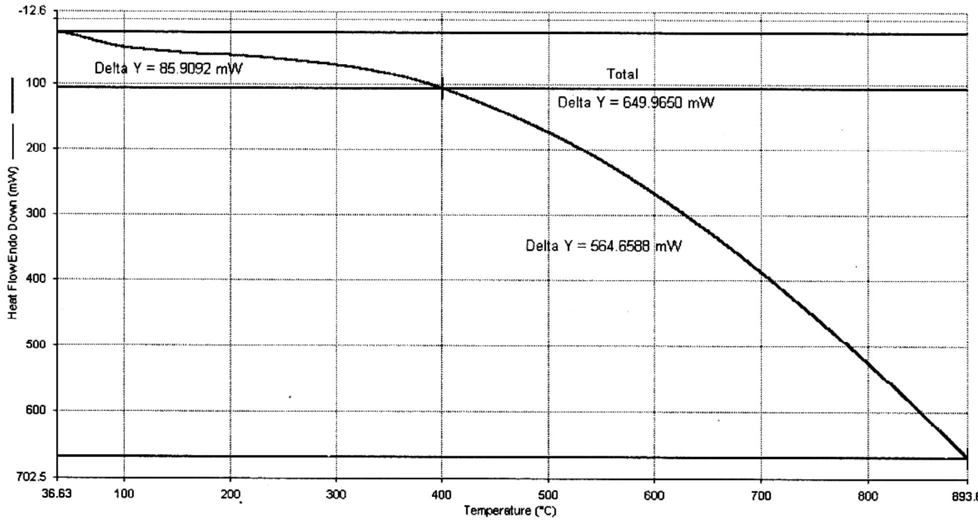

Fig. 2. Only DSC curves of RHB3.

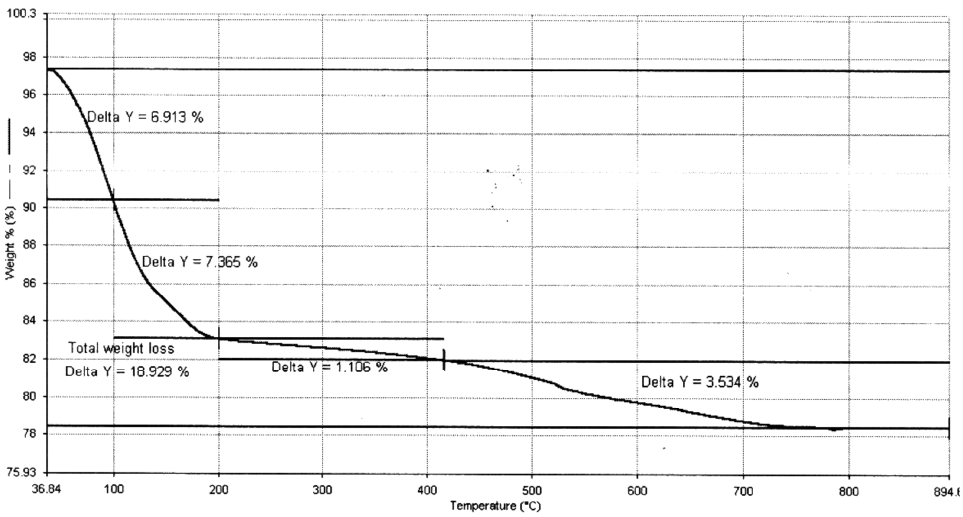

Fig. 3. Thermo gravimetric analysis (TGA) curves of RHB1.

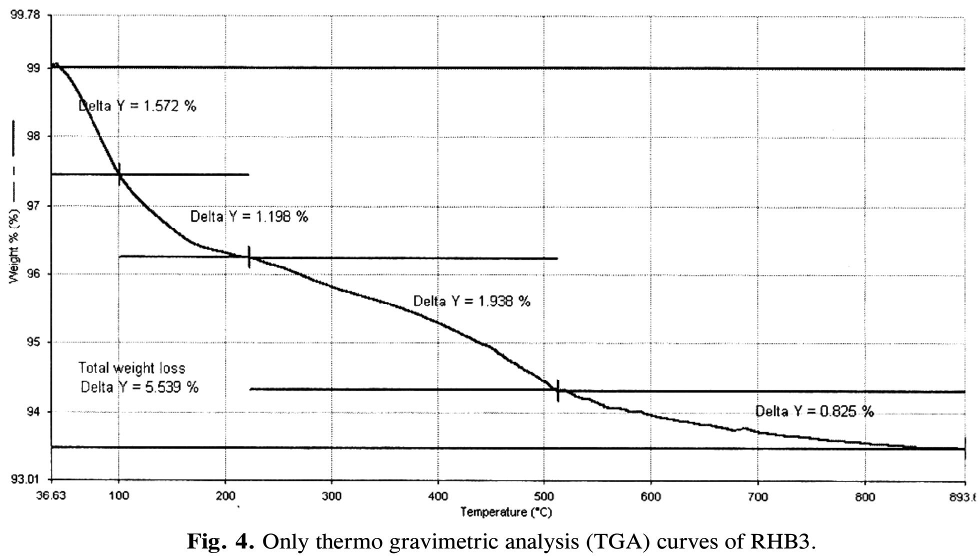

Fig. 4. Only thermo gravimetric analysis (TGA) curves of RHB3.

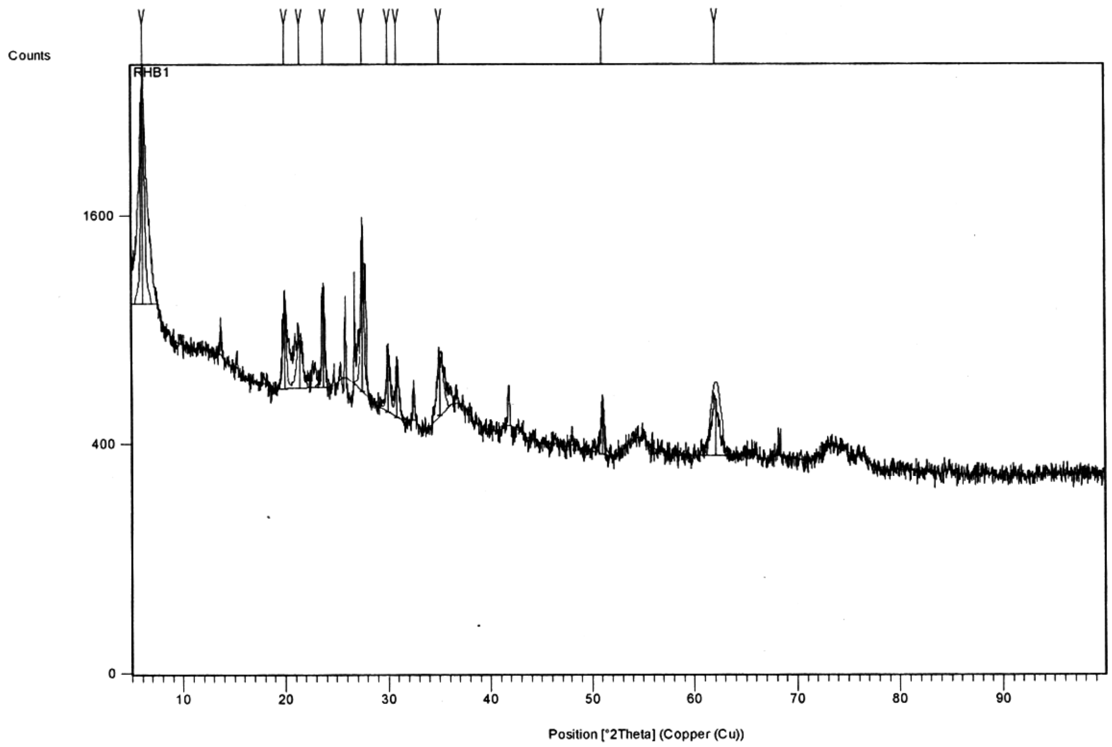

Fig. 5. Powdered X-ray diffraction (PXRD) of RHB1.

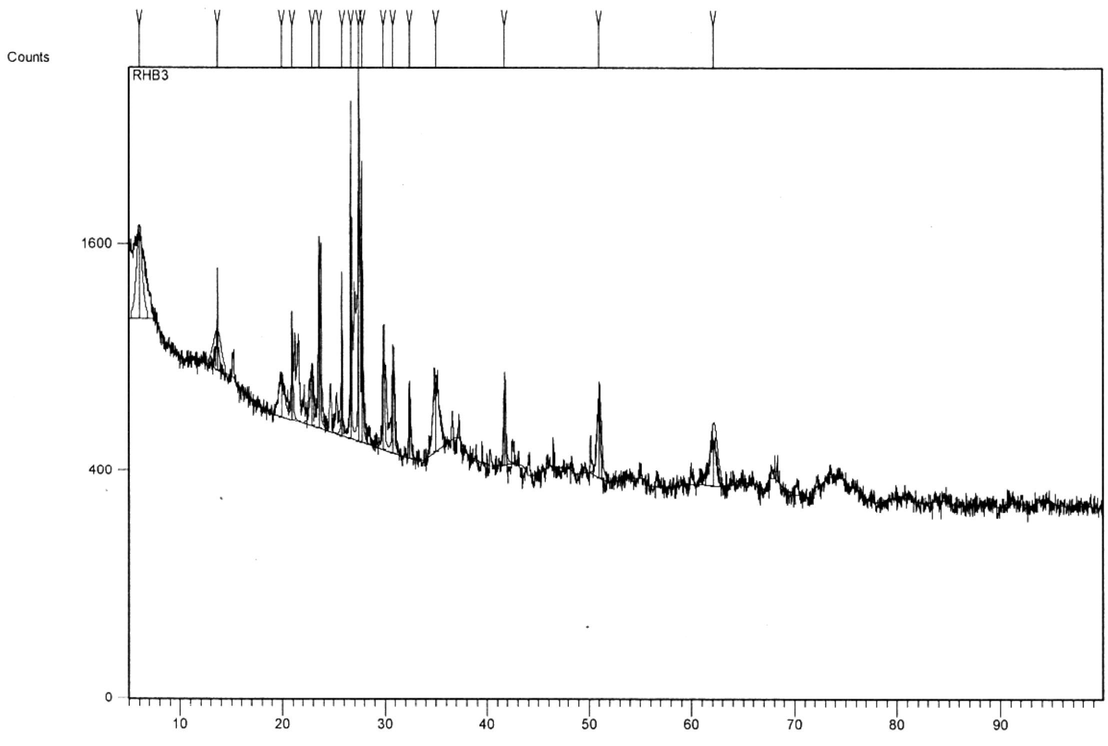

Fig. 6. Powdered X-ray diffraction (PXRD) of RHB3.

Table 1 describes the literature values of d (Å) for bentonites of different places and Table 2 shows the calculated powdered X-ray data. Table 3 and Table 4 show d spacings and height (cts) for bentonite samples RHB1 and RHB3 respectively.

Table 1. Literature[a]

Bihar d (Å)	Rajula d (Å)	Akli d (Å)	Jaipur d (Å)
7.23 (K)	10.10 (I)	7.17 (K)	4.49 (M)
4.48 (M)	4.48 (M)	4.48 (M)	3.34 (I)
2.57 (M)	2.58 (M)	2.57 (M)	2.59 (M)
15.77 (M)	15.40 (M)	15.50 (M)	2.57 (I)

[a]S. K. Guha and Sudhir Sen, *Trans. Ind. Ceramic Soc.*, 1973, **32**, 97.

Table 2. Calculated X-ray powder data of Montmorillonite and Kaolinite (literature)[a]

Montmorillonite d (Å)	Kaolinite d (Å)
4.500	7.150
2.600	4.400
2.250	4.330
1.760	4.172
1.503	3.736
1.301	3.573
1.252	3.148
–	3.098
–	2.748
–	2.566
–	2.483
–	2.335

[a]S. Ambi, "The study of bentonite minerals from Rajmahal, Bihar", IIT, Delhi, 1977.

Table 3. Powdered X-ray diffraction (PXRD) of RHB1

Peak List : (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. int. [%]
6.1334	1604.14	0.3365	14.41051	100.00
19.9676	461.69	0.2991	4.44678	28.78
21.4269	228.37	0.5983	4.14712	14.24
23.7572	501.70	0.2991	3.74535	31.28
27.5275	828.85	0.2991	3.24033	51.67
30.0483	265.22	0.2991	2.97399	16.53
30.9289	239.89	0.2991	2.89130	14.95
35.1143	260.40	0.5983	2.55567	16.23
51.0660	192.82	0.2991	1.78859	12.02
62.0838	193.33	1.0944	1.49380	12.05

Table 4. Powdered X-ray diffraction (PXRD) of RHB3

Peak List : (Bookmark 3)

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. int. [%]
6.0027	558.94	0.5983	14.72396	21.46
13.6461	196.33	0.8974	6.48919	7.54
19.8985	180.94	0.5983	4.46206	6.95
20.9231	548.25	0.1496	4.24583	21.05
22.8987	271.86	0.3739	3.88377	10.44
23.5689	1007.35	0.1496	3.77484	38.67
25.8082	806.27	0.0935	3.45216	30.95
26.6688	2088.12	0.0748	3.34268	80.16
27.4816	2604.78	0.0935	3.24565	100.00
27.7773	1735.15	0.0748	3.21176	66.61
29.8874	593.06	0.1496	2.98964	22.77
30.8195	451.68	0.1870	2.90131	17.34
32.4280	310.52	0.1496	2.76098	11.92
34.9999	302.42	0.5983	2.56377	11.61
41.7045	406.28	0.1870	2.16580	15.60
50.9712	348.97	0.3739	1.79169	13.40
62.1093	165.46	0.7296	1.49325	6.35

Acknowledgement

Thanks to University Grants Commission for the grant of research project in Chemistry.

References

1. D. Jha, B. K. Mishra, S. S. Mishra and A. K. Jha, *J. Haematol. and Ecotoxicol.,* 2010, **5**, 1.
2. A. K. Jha and B. Mishra, *J Indian Chem. Soc.*, 2012, **89**, 519.
3. G. Lagaly, "Surface and Interlayer Reactions : Bentonites as Adsorbents", 1995, pp. 137-144.
4. D. Guyonnet, E. Gaucher, H. Gaboriau, H. Ponsc, C. Clinard, V. Norotte and G. Didier, *Journal of Geotechnical Engineering*, 2005, **131**, 740.
5. M. V. Kok, *Energy Sources*, 2002, **24**, 899.
6. F. Ayari, E. Srasra and M. Trabelsi-Ayadi, *Desalination*, 2005, **185**, 391.
7. Rapport annual de Federation de I' industrie minerale, 2004.
8. E. Srasra, F. Bergaya and J. J. Fripiat, *Clays Clay Miner.*, 1994, **42**, 237.
9. S. Chevalier, R. Franck, J. F. Lambert, D. Barthomeuf and H. Suquet, *Appl. Catal. A : Gen.*, 1994, **110**, 153.
10. Hicham Zaitan, Daniel Bianchi, Ouafae Achak and Tarik Chafik, *J. Hazard. Mater.*, 2008, **153**, 852.
11. Z. Yermiyahu, I Lapides and S. Yariv, *J. Them. Anal. Calorim.*, 2002, **69**, 317.

12. Z. Yermiyahu, I. Lapides and S. Yariv, *Appl. Clay Sci.*, 2005, 33.

13. A. C. Newman and G. Brown, *Chem. Clays. Clay Miner.*, 1987, **6**, 1.

14. K. Kuroda, *Hyomen*, 1989, **27**, 77.

15. R. E. Grim, "Clay Mineralogy", Int. Series in Earth and Planetary Sci., 1968

16. A. R. Mermut (Ed.), "Layer Charge Characteristics of 2 : 1 Silicate Clay Minerals", CMS Workshop Lecture Series, Vol. 6, Clay Minerals Society, 1994

17. G. W Brindley and G. Brown, "Crystal Structure of Clay Minerals and their X-Ray Identification", Mineralogical Society, Monographs, 1980, No. 5.

18. Ashok Kr. Jha, Arun Kr. Jha, Arun Kr. Mishra, Vineeta Kumari and B. Mishra, *Asian Journal of Water, Environment and Pollution*, 2011, **8**, 93.