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STUDIES ON THE LIME REACTIVITY WITH THERMALLY ACTIVATED CLAY FROM JAMMU REGION (INDIA)

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Clay from Purmandal area of Jammu (J&K State, India) was calcined at various temperatures. The calcined products were subjected to lime reactivity test. The results obtained were correlated with X-ray diffractograms and DTA curves of the raw and calcined samples respectively to explain the pozzolanic activity of the calcined clay.

The great demand to evaluate the clay resources in India has been emphasized by several workers [1-3] which may be of enormous help to the clay processors for ceramic, paper, pharmaceutical, rubber and a number of other industries. Studies on the pozzolanic activity of the burnt clay have been the subject of numerous workers [4-6].

There are large reserves of clays in India which are yet to be characterized properly for their suitable exploitation. In the present case, clays from the Purmandal area of Jammu region in Jammu and Kashmir State of India, for which little information is available in the literature [7-9] has been explained on its pozzolanic activity with respect to temperature of calcination. The main aim of this investigation is to give systematic and detailed report on the good pozzolanic activity of the calcined samples as revealed by X-ray diffraction and DTA studies, and supplementing it with lime reactivity tests.

Experimental

The bulk representative sample of clay for this investigation was collected from the Purmandal area latitude 32° 42' longitude 76° 6' about 33 kms from Jammu. The clay as such is dull white in colour, it occurs as

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fairly hard and compact lumps with large dark grey particles embedded in the clayey matrix. It has a sandy texture. The physical properties and chemical analysis of the sample carried out by conventional method is shown in Tables 1 and 2.

Table 1 Physical properties of Purmandal clay

1.	Colour	Dull white
2.	Plasticity by feel	Poor
3.	Liquid limit	36.2
4.	Plasticity index	4.38
5.	Flow index	5.00
6.	pH of clay suspension (2% S/L)	8.8
7.	Toughness index	0.12
8.	Dry linear shrinkage %	2.5

Table 2 Chemical analysis of Purmandal clay

Constituents	Percent
SiO_2	66.92
Al_2O_3	14.10
Fe_2O_3	1.59
TiO_2	0-.11
CaO	2.28
MgO	1.98
K_2O	3.89
Na_2O	0.19
LOI	9.05

The representative clay sample of minus one cm size was filled in silica dishes and charged inside an electric furnace in the laboratory. The temperature was raised to the desired particular temperature within two hours, soaked for another two hours within a temperature variation of $\pm 5^\circ$ and then the furnace was brought to room temperature. The calcined clay was taken out, powdered to a fineness in accordance with IS 1344.

The differential thermal analysis was carried out on a MOM Derivatograph Model (OD-103) at a heating rate of 10 deg/min. The X-ray analysis of the samples were carried out by powder diffraction technique using Philips X-ray powder diffractometer PW 1350 with Nickel filtered CuK_α radiation (30 KV 15 mA). The samples were scanned with a scanning speed of 1° in 2Θ per minute. The rate meter and chart speed were kept constant for all runs. The lime reactivity test was carried out as per IS 1727 Indian Standard Specification.

Results and discussion

Figure 1 A shows the X-ray powder diffractogram of the raw sample. The interplaner spacings calculated from the diffractogram and peak intensities are given in Table 3. It is seen from the diffractogram that there are two broad reflections near the low angular range from $2\Theta = 5^\circ$ to $2\Theta = 10^\circ$. The '*d*' values of these reflections are 15.504 Å and 9.048 Å. The first reflection is due to montmorillonite. Guha *et al.* [7] have pointed out that the first order basal spacing (ool) of the bentonites can be observed in the region 14 to 17 Å and the same can shift to 17.7 Å on glycerol treatment. The second reflection in this Fig. can be assigned due to the presence of micaceous group. The rest of the values from the Table 3 were also indexed with the standard ASTM data cards. It has been observed that two dimensional (*h k*) bands due to dioctahedral smectites (montmorillonite) are also present. These (*h k*) bands mainly do not provide sharp features [10] as they are diffused and indistinct. Some of the other reflections which have not been

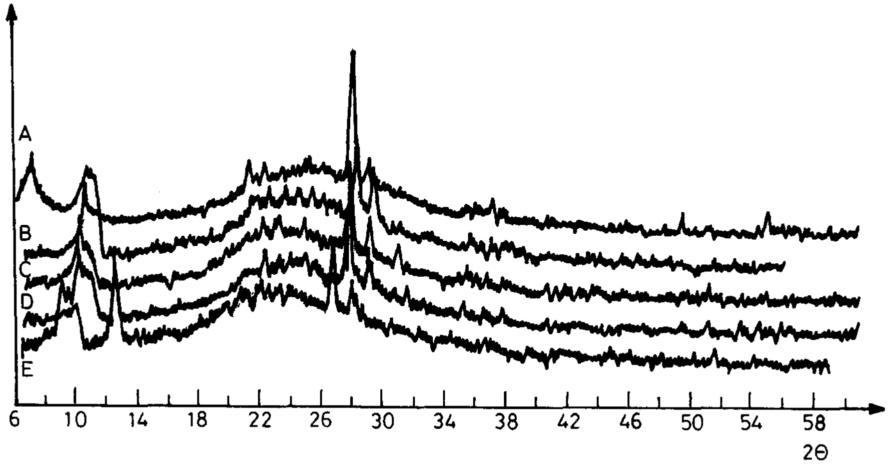

Fig. 1 X-ray powder diffractiogram of the sample from Purmandal area. A - raw sample, B - sample calcined at 750°C, C - sample calcined at 800°C, D - sample calcined at 850°C, E - sample calcined at 900°C

found to be belonging to smectite group of minerals are mainly due to the presence of quartz, felspar and micaceous impurities.

Table 3 Interplaner spacings and peak intensities of the raw clay sample and sample calcined at different temperatures

Raw		750°C		800°C		850°C	
d, Å	*I*	*d*, Å	*I*	*d*, Å	*I*	*d*, Å	*I*
3.3264	15	3.3264	13	3.3264	7	4.7450	7
						3.3264	18
						3.1977	9

d, Å - Interplaner spacing, *I* - peak intensity

Figure 1 BCDE shows the X-ray powder diffractograms of the samples heated to 750, 800, 850 and 900° respectively. At about 750° the diffractogram shows the emergent of some new reflection which are indicative of the transformation of dioctachedral smectite. It is seen from the figure that a new line with '*d* Å' value 9.80 Å have come up which is well comparable with the Bradley and Grim [11] diffraction for montmorillonite ignited to 725°. Some more new lines have also arrived in the diffraction of the sample heated to 850°. But these lines are very weak in intensity and diffuse thereby giving rise to the disordered state of the structure [12].

Figure 2 shows the differential thermal analysis curve of the raw sample. It is observed that there is a main endothermic peak at about 200° and two broad endothermic peaks in the temperature range from 800 to 900°. The initial endothermic peak is due to the water loss which occurs mostly as interlayer water between the silicate sheets. Mackenzie [13] suggested that the amount of interlayer water depends on the dehydration energy of the ad-

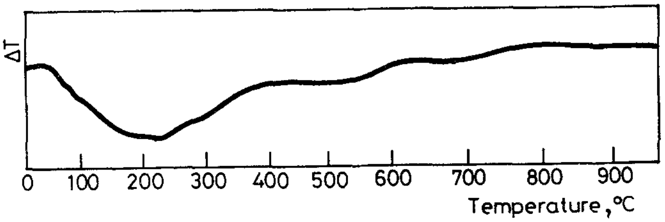

Fig. 2 DTA curve of raw sample from Purmandal area

sorbed cations and on the hydration of the surface. The second and third endotherms may be due to the loss of OH lattice water and there by causing the destruction of the lattice at 900° respectively.

It is reported [14] that clays on heating first, give out their surface and interlayer water. Further heating brings about the continuous loss of the hydroxyl ions in the structure followed by collapse of the structure yielding a very intimate mixture of the oxides or a solid solution. Further heating results in the interaction of these oxides to form spinal structure and finally mullite.

Regarding the pozzolanic activity of the clay minerals, Meilenz *et al.* showed [15] that clay minerals have an optimum temperature of calcination at which it exhibits maximum pozzolanic activity. Srinivasan [16] extended it further and suggested that pozzolanic activity is the highest when the collapse of the structure has just begun, resulting in the nascent and micro crystalline oxides and reminiscent structure with high strain in bonds. The above phenomenon is in agreement with the results got by X-ray and DTA studies carried out on Purmandal clay.

Pozzolanic activity [17] is a measure of the cementing quality of lime pozzolana mixture. The best and the safest method of measuring pozzolanic activity is to measure the strength development in lime, pozzolana, sand mortars. Since the value of pozzolana depends upon it's ability to combine with lime, it is expected that a fair relationship should exist between pozzolanic activity and the amount of lime absorbed or combined. It has been reported that initially the pozzolana absorbs lime rapidly but this does not bear any relation to the value of the pozzolana and may be high with materials of low pozzolanic values. The initial rapid absorption is followed by slower reaction which has been suggested as an index of activity.

Table 4 Lime reactivity value of Purmandal clay

Sl. No.	Temperature of activation, °C	kgs/cm^2
1.	600	50
2.	650	63
3.	700	70
4.	750	86
5.	800	100
6.	850	90
7.	900	81
8.	950	70
9.	1000	65

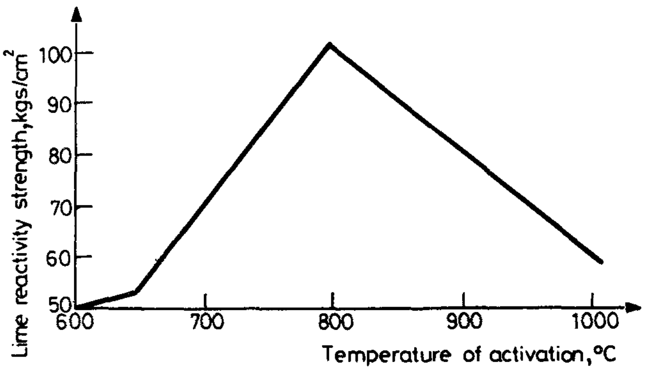

Fig. 3 Lime reactivity strengths of clay samples calcined at various temperatures

Lime reactivity test [17] covers the procedure for determining the reactivity of the pozzolanic materials with hydrated lime as represented by compressive strength of standard mortar test cubes prepared and tested under specific conditions. Lime [18] reactivity tests were carried out as per IS 1727 & IS 4098 on the calcined Purmandal clay samples. The results are reported in Table 4 and Fig. 3. The graph is drawn taking temperature of calcination *vs.* lime reactivity strength which indicates the optimum temperature of calcination for maximum compressive strength is 800°.

Conclusion

Thus from the DTA, X-ray and Lime reactivity studies carried out on Purmandal clay it appears that the clay is montmorillonite (smectite) in nature with good amount of quartz, felspar and micaceous impurities and the structural water is lost in three stages. The structure of the clay becomes distorted at about 800° which is the optimum temperature for maximum activation of the clay. The clay calcined at this temperature is most suitable for making pozzolanic cements.

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References

1 P. K. Rohtagi, K. Rohtagi and Bowender, Technologica Forecasting, Tata McGraw Hill Co., New Delhi 1979.

2 M. Patel, K. Gopakumar and P. K. Rohtagi, Trans. Ind. Ceram. Soc., 39 (1980) 85.

3 D. Thomas, P. Krishna Pillai and M. Lalithambikai, Trans. Ind. Ceram. Soc., 40 (1981) 183.

4 R. Twviziani and H. F. W. Taylor, The Chemistry of Cements, Academic Press, London - New York, 1964 p. 69.

5 E. G. Swenson and P. S. Sereda, J. Appl. Chem., London 17(7) (1967) 198.

6 R. W. Grimahaw, The Chemistry and Physics of Clays Ed. G. 1971.

7 S. K. Guha, S. R. Das and Sen Sudhir, Trans. Ind. Ceram., Soc., 32 (1973) 11.

8 K. N. Goswami and A. K. Gupta, Trans. Ind. Ceram. Soc., 32 (1973).

9 H. R. Khajuria, S. K. Mehta and A. K. Gupta, J. Thermal Anal., 31 (1986) 15.

10 H. Pezerat and J. Mering, C. R. Hebd. Scanc. Acad. Sci., Paris (265), (1967) 529.

11 W. F. Bradley and R. E. Grim, "High Temperature Thermal Effects of Clay and related Minerals", Am. Mineralogist, 36 (1951) 182.

12 K. N. Goswami and A. K. Gupta, "Structural Variations in some Kaolinites", Trans. Ind. Ceram. Soc., 32 (1975) 99.

13 R. C. MacKenzie, Ber. Dait, Keram, Ges., 41 (12) (1964) 696.

14 N. R. Sriniwasan, Road Research Papers, No.1, CRRI, New Delhi, (1956) p. 70, 44, 58.

15 R. C. Meilenz *et al.*, Symposium on the use of Pozzolanic materials and concrete ASTM, Pub. No. 39, 1949.

16 N. R. Srinivasan, Road Research Papers, No.1 CRRI, New Delhi, (1956) p. 70, 49, 50.

17 M. K. Chatterji and D. D. Lahiri, 'Proceedings of the Symp. on Pozzolanas, their Survey, Manufacture and utilization, CRRI, New Delhi 1967, p. 78.

18 IS 1727 (1967), Methods of Test for Pozzolanic Materials, ISI, New Delhi, p. 3.

Zusammenfassung — Bei verschiedenen Temperaturen wurde Tonerde aus dem Gebiet Purmandal in Jammu (J&K State, Indien) kalziniert. Die kalzinierten Produkte wurden einem Kalkreaktivitätstestes unterzogen. Zur Erklärung der Puzzolanaktivität der kalzinierten Tonerdeproben wurde der Zusammenhang zwischen erhaltenen Ergebnissen und Röntgendiffraktogrammen sowie DTA-Kurven von Roh- und kalzinierten Proben ermittelt.