Thermochimica Acta, 186 (1991) 311–314
Elsevier Science Publishers B.V., Amsterdam

Note

Thermogravimetric studies of a Cr(III)- and Zr(IV)-exchanged new synthetic zeolite (Deolite)

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(Received 2 January 1991)

Commercial Deolite, a desiccant with properties similar to those of 4A-type zeolite, has been interacted with Cr(III) and Zr(IV) cations to prepare cation-exchanged derivatives of Deolite. This work is devoted to the discussion of the TG, DTA and DTG behaviour of the exchanged derivatives of Deolite. Similar studies have also been carried out on other synthetic zeolites by earlier workers [1–4].

EXPERIMENTAL

Details of the sample preparation using synthetic Deolite (obtained from Ras Enterprises, Bombay) have been reported previously [5]. The TG data were recorded on a Perkin–Elmer thermobalance at a heating rate of 10 °C min^{-1}. The TG plots are shown in Figs. 1 and 2. The IR spectra were recorded in KBr pellets on a Perkin–Elmer 397 spectrophotometer and are shown in Fig. 3.

RESULTS AND DISCUSSION

The Cr(III)-interacted Deolite exhibits a somewhat different thermal behaviour compared with that reported for other cation-interacted derivatives [1–4]. This is due to the tendency of the Cr(III) ions to form hydrated species as well as being oxidised to higher valency states. Thus it loses about a third of its total weight loss in a fast step up to 373 K, followed by a second dehydration step up to 493 K, resulting in a further weight loss of 6.9%. Both the DTG and DTA plots confirm these thermal events. Beyond 493 K, decomposition of the hydrated chromium begins. The decomposition is indicated by an exothermic DTA peak at around 713 K. The final weight-loss step starts at around 973 K leading to the final decomposition at around 1200 K, confirmed by a shallow exothermic peak. The four-step weight loss sequence gave an average dehydration activation energy of 57.3 kJ mol^{-1} for $n = 1$ and 39.0 kJ mol^{-1} for $n = 2$.

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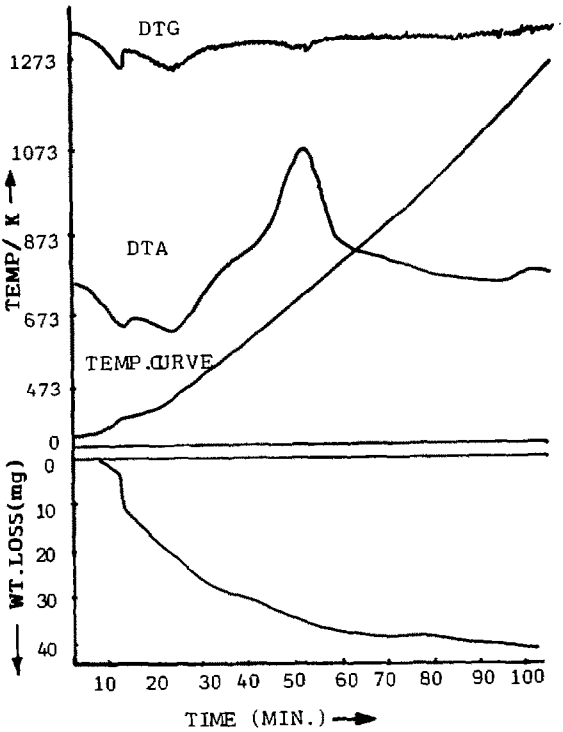

Fig. 1. TG plot of the Cr(III)-exchanged Deolite.

The weight-loss sequence for Zr(IV)-interacted Deolite begins around 333 K and proceeds up to 513 K at which temperature most of the water has been lost. Further water loss due to dehydration proceeds at a much slower

TABLE 1

TG data, kinetic parameters of thermochemical processes and IR spectra data

Zeolite	Total weight loss (%)	Weight loss (%) at different temperatures	Rate of reaction (min^{-1})		Activation energy ($kJ\ mol^{-1}$)		IR band (cm^{-1})
			$n=1$	$n=2$	$n=1$	$n=2$	
Cr(III) Deolite	21.4	6.0 up to 373 K	3.6×10^{-1}	1.3×10^{-1}	59.9	36.7	3400,1645
		6.9 up to 493 K	5.7×10^{-2}	3.1×10^{-2}	40.3	28.2	1070,790
		4.6 up to 713 K	1.4×10^{-2}	1.2×10^{-2}	51.1	36.9	620,455
		3.9 after 713 K	5.6×10^{-3}	7.3×10^{-3}	77.7	54.1	
Zr(IV) Deolite	23.8	4.1 up to 393 K	4.5×10^{-1}	1.4×10^{-1}	63.9	39.0	3400.1645
		8.6 up to 453 K	1.7×10^{-1}	8.4×10^{-2}	73.4	44.7	1075,790
		8.2 up to 513 K	7.9×10^{-2}	7.5×10^{-2}	152.8	85.3	620,450
		2.9 after 513 K	1.84×10^{-2}	4.2×10^{-2}	244.8	132.6	

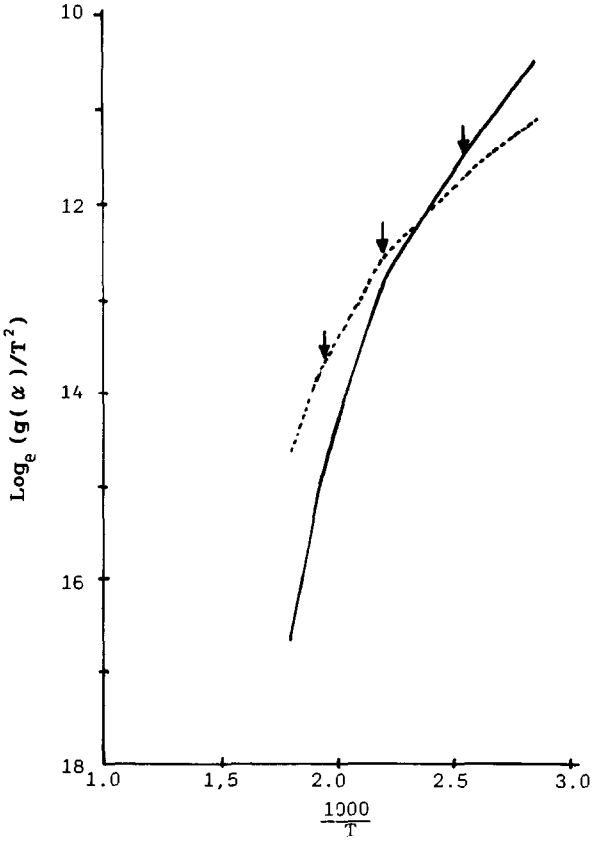

Fig. 2. TG plot of the Zr(IV)-exchanged Deolite: ———, $n = 1$; ----, $n = 2$.

rate beyond 513 K and a total weight loss 23.8% is obtained. The DTA data for this Deolite have been reported earlier [6].

The IR spectra of the two zeolite derivatives represent typical hydrated zeolite structures. The absorption band at 1000 cm^{-1} is related to the Si/Al

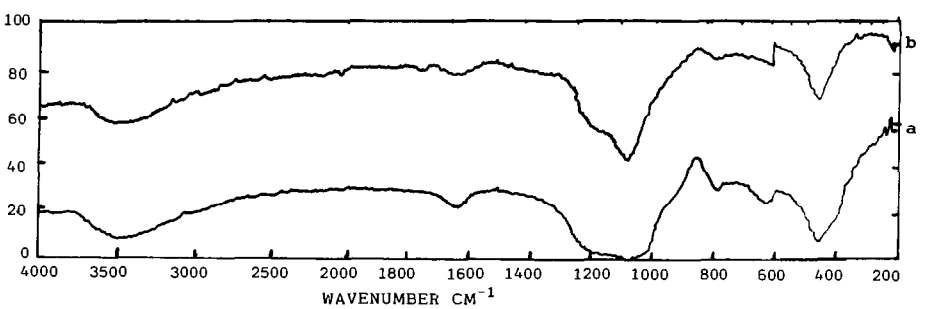

Fig. 3. IR spectra of Cr(III)-exchanged Deolite (curve a) and Zr(IV)-exchanged Deolite (curve b).

ratio in the zeolite framework [7], and a decrease in its frequency is mainly due to the increase in aluminium in the aluminosilicate tetrahedra.

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