Journal of Thermal Analysis, Vol. 44 (1995) 733-738

THERMAL, IR, CATION EXCHANGE AND ADSORPTION STUDIES OF NEW SYNTHETIC ZEOLITE (DEOLITE)

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(Received January 18, 1994; in revised form May 20, 1994)

Abstract

Cadmium(II) and Pb(II) ions replace the sodium(I) ion in commercial molecular sieve deolite. These exchanged derivatives have been interacted with adsorbates like CO_2 and NH_3 and the new exchanged and adsorbed derivatives characterized by their TG and IR spectral data. It is concluded that deolite can be effectively employed for removal of hazardous Cd(II) and Pb(II) ions as well as the gaseous adsorbates.

Keywords: Deolite, IR, TG, zeolite

Introduction

Commercial deolite, a desicant having similar properties to 4A type zeolite has already been thermally and structurally studied by authors [1–3]. In the present case of investigation deolite has been interacted with Cd(II) and Pb(II) ions. These exchanged derivatives have been used for adsorbing CO_2 and NH_3. The aim of the present study is to test their stabilities as cation exchangers. Cadmium and lead are known air and water pollutants having toxic effect on the pulmonary and renal systems [4]. Similarly, carbon dioxide and ammonia have hazardous effects which necessitates their removal from both water and the atmosphere for health safety [5]. The new exchanged and adsorbed derivatives have been characterized by their TG and IR analysis and results have been reported here.

Experimental

The deolite sample was obtained from Ras Enterprises, Bombay as a white odourless powder and procedure used for the preparation of exchanged and ad-

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0368–4466/95/ $ 4.00

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sorbed derivatives has been described earlier [6]. An aqueous solution of Analar cadmium(II) nitrate-tetrahydrate and lead(II) chloride were used for exchange. Composition study has been carried out by Simadzu simultaneous X-ray fluorescence spectrophotometer. TG analysis was performed by Perkin Elmer thermobalance at the heating rate of 15 deg/min in air. IR spectra were obtained by Perkin Elmer 397 spectrophotometer using KBr phase in the range of 4000–400 cm^{-1}. Alkali metals were estimated by Elico digital flame photometer.

Results and discussion

The analytical data of deolite and its derivatives are given in Table 1. On the basis of obtained analytical data it has been suggested that deolite is a fairly good ion exchanger for these ions. The mechanism of cation exchange and ad-

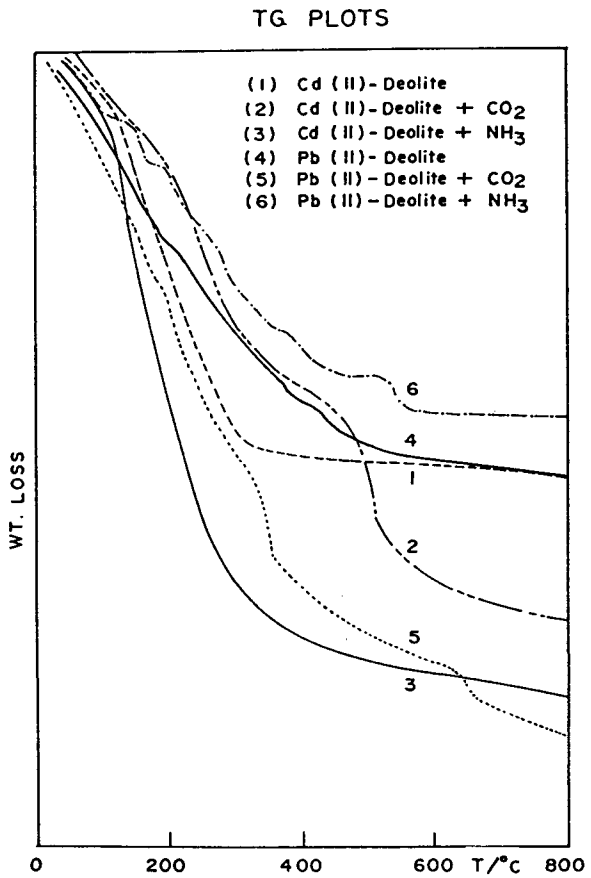

Fig. 1

sorption [7–9] can be explained on the basis of earlier work done on similar cation and adsorbate species and zeolite molecular sieves.

Table 1 Analytical data of zeolites

Sample	SiO_2 / %	Al_2O_3 / %	Na_2O / %	Degree of exchange / %	Si/Al –
Deolite	41.82	22.59	14.00		1.64
Cd(II)/exchanged Deolite	40.92	22.22	0.65	95.35	1.84
Pb(II)/exchanged Deolite	40.25	21.05	0.74	94.71	1.69

TG plots are shown in Fig. 1 and TG data are given in Table 2. These data are discussed over the lower and higher temperature range, i. e. below and above 750°C approximately. In the case of all six samples major mass loss is attributed mainly to dehydration. Above this lower temperature limit, thermal processes like desorption and decomposition have been observed in Cd(II)/Deolite + adsorbed CO_2 samples. The maximum loss occurs upto 753 K which indicates lack of physically adsorbed CO_2, while ammonia interaction is expected to form complex species like $[Cd(NH_3)_4](OH)_2$, and shows an abrupt and very fast mass loss step due to deammoniation. From the TG plots of Pb(II)–deolite and its adsorbed derivatives, it is clear that the major step of weight loss occurs between 353 and 633 K. Further weight loss beyond 633 K takes place at much slower rate upto 1073 K. Carbon dioxide sample exhibits maximum weight loss upto 393 K.

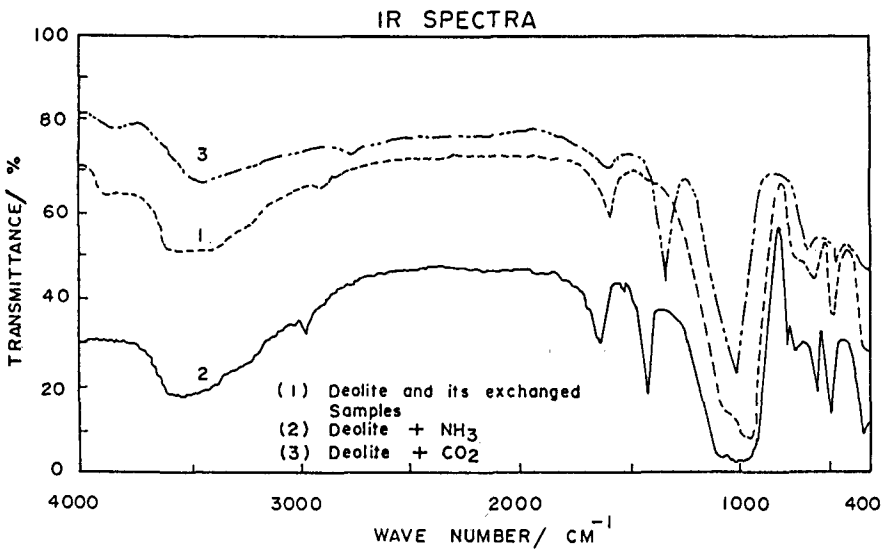

Fig. 2

Table 2 Summary of thermal data of zeolites

No.	Zeolite	Weight losses				Rate of reaction / ($\times 10^{-2}$ min^{-1})		Activation energy / kJ·mol^{-1}	
		Total loss / %	loss by steps / %		T/K	$n=1$	$n=2$	$n=1$	$n=2$
1.	Cd(II)/	19.25	2.25	upto	393	46.00	12.80	21.48	14.95
	Deolite		15.30	upto	539	9.68	5.50	30.64	22.50
			1.70	after	593	0.26	0.72	58.34	36.12
2.	Cd(II)/	22.37	14.49	upto	753	10.00	3.44	20.00	15.59
	Deolite		3.83	upto	793	6.05	5.50	116.58	63.08
	$+CO_2$		4.05	after	793	1.46	1.46	153.72	79.06
3.	Cd(II)/	24.85	20.30	upto		32.50	8.80	31.49	19.91
	Deolite		2.25	upto		2.48	0.33	67.23	44.19
	$+NH_3$		1.30	after		0.15	0.51	53.43	31.83
4.	Pb(II)/	12.20	5.19	upto	493	18.60	6.50	19.50	12.97
	Deolite		4.79	upto	693	3.31	2.42	31.09	20.34
			2.22	after	693	0.55	1.11	79.29	46.80
5.	Pb(II)/	23.80	15.02	upto		13.00	4.89	20.01	13.70
	Deolite		2.46	upto		7.90	5.75	138.98	97.92
	$+CO_2$		2.01	upto		0.73	0.83	73.44	48.54
			2.31	after		1.31	2.61	215.83	101.20
6.	Pb(II)/	23.00	16.82	upto	653	14.00	5.09	23.14	15.67
	Deolite		3.37	upto	813	1.13	1.24	37.44	26.85
	$+NH_3$		2.24	upto	833	5.25	10.50	286.88	12.39
			0.57	after	833				

Table 3 Principle infrared frequencies cm^{-1}

S. No.	Deolite and its exchanged derivatives	Deolite $+CO_2$	Deolite $+NH_3$	Assignments
1.	3620–3340 (sb)	3600 (sb)	3540 (sb)	OH stretching
2.	1630 (s)	1645 (s)	1640 (s)	Water bending
3.		1440 (s)	1460 (s)	CO_3(II), NH_4(I) ions respectively
4.	1000 (sb)	1040 (sb)	1020 (s)	Asymmetric T–O stretching
5.	710 (m)	715 (ms)	725 (ms)	Symmetric T–O stretching
6.	560 (ms)	530 (ms)	540 (ms)	T–O bending
7.	440 (ws)	485 (ws)	445 (ws)	Double ring
8.	330 (sb)	340 (sb)	320 (sb)	Pore opening

Abbreviations: s = strong; sb = sharp broad; sm = sharp medium; m = medium; ms = medium sharp; wb = weak broad; ws = weak shoulder

Kinetic parameters of the various thermal changes were evaluated by using $g(\alpha)$ *vs.* time and $\ln[g(\alpha)/T^2]$ *vs.* $1000/T$ plots for $n=1$ and 2. The values corresponding to $n=1$ are given in Table 1. The activation energy obtained for the various thermal events indicates the nature of the reactions.

IR spectra are shown in Fig. 2. Exchanged derivatives give approximately the same IR spectrum as it is obtained in the case of deolite [10]. Adsorption of CO_2 produces a sharp band in the region of 1395 cm^{-1} attributed to ν_1 vibration of physically adsorbed CO_2 held linearly to the surface. Ammonia adsorbed on exchanged sample produces $NH_4(I)$ ion as indicated by a band at 1450 cm^{-1} [11].

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Zusammenfassung — Kadmium(II)- und Pb(II)-Ionen ersetzen das Natrium(I)-Ion im handelsüblichen Molekularsieb Deolite. Diese ionengetauschten Derivative traten mit Adsorbaten wie CO_2 und NH_3 in Wechselwirkung und die neuen ausgetauschten und adsorbierten Derivative wurden durch ihre TG- und IR-Daten charakterisiert. Man zog den Schluß, daß Deolite effektiv zur Entfernung von gefährlichen Cd(II)- und Pb(II)-Ionen als auch der gasförmigen Adsorbate eingesetzt werden kann.