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THERMOANALYTICAL AND ADSORPTION INVESTIGATIONS ON DEALUMINATED Y-ZEOLITES OF DIFFERENT PREPARATION

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ABSTRACT

Y-zeolites dealuminated by the thermochemical treatment and by the extraction with ethylenediaminetetraacetic acid are characterized by DTA/DTG and adsorption measurements.

INTRODUCTION

Dealuminated Y-zeolites are important in view of their use as promoter in cracking catalysts. By modification of the Y structure samples may be obtained with an aluminium content per unit cell between that of the parent zeolite (55 aluminium atoms) and about 2 [1]. The content of aluminium in the structure determined not only the catalytic activity and selectivity [2] but also the thermal stability.

In this paper the thermal stability is studied on two series of samples of different preparation. For further characterization the adsorption capacity was determined from adsorption isotherms:

EXPERIMENTAL

Samples preparation

Series A: Samples prepared by thermochemical treatment, i.e. by thermal decomposition of NH_4Y zeolites in the presence of water vapour.

Series B: Samples prepared by extraction of NaY with ethylenediaminetetraacetic acid (EDTA) in aqueous solution.

NaY and NaX commercial and from own preparation are used for comparison of the course of the changes in their properties.

For the study of thermal properties a Netzsch Thermoanalyser and a Derivatograph (MOM) was used. The conditions were:

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	Netzsch Thermoanalyser	Derivatograph
sample mass, series A	37 - 50 mg	100 mg
series B	45 - 70 mg	100 mg
inert material	Al_2O_3	Al_2O_3
DTA sensitivity	0.05 mV	1 : 3
DTG sensitivity		1 : 10
heating rate	5° / min.	5° / min.
temperature range	25 - 1400°C	25 - 1000°C
gas atmosphere	N_2 (80 ccm/min.)	air

Adsorption isotherms were measured gravimetrically at 25°C using a McBain balance.

RESULTS AND DISCUSSION

The samples are listed in Table 1. Here the extrapolated onset temperature for the lattice destruction as well as for the peak maximum are given in columns 3 and 4. In the last two columns the adsorption capacity for water from the TG curve and for hexane from the adsorption isotherms is shown.

Figure 1 contains the DTA curves in the temperature range below 500°C for series A, series B and NaY. In this range water desorption takes place. Corresponding to the decreasing amounts of water with diminishing Al content the DTA curves become smoothed and the peak maximum as well as the end of the desorption are shifted to lower temperatures.

The hexane adsorption capacity (column 6) for the investigated samples agrees with the estimated water content not only in the dependence on Al content but also in the numerical values.

In Figure 2 the destruction temperature is plotted against the number of Al atoms. In the range from 81 to 55 Al atoms per unit cell the destruction temperature is nearly constant. Between 55 and 50 Al a remarkable increase of stability is observed. Below 55 Al atoms the stability again depends weakly on the Al content. A strong rise in stability is observed at very low Al content. The discontinuous dependence of the thermal stability on the aluminium content seems to be related to the silicon, aluminium ordering in the tetrahedral zeolite lattice [3, 4] and will be considered later [5]

Table 1

Sample	N_{Al} (number of Al atoms per unit cell)	Temperature of structure destruction (° C)		Adsorption capacity (ccm/g)	
		T_{on}(extr.)	T_p	H_2O (from the TG curve)	n-hexane (from adsorption isotherms)
NaX	81	830	855	0.28	-
NaX	74	840	880	0.26	-
NaY	62	875	895	0.26	-
NaY*)	55	875†)	-	0.24	0.29
Series A					
1	52	933	946	0.25	0.27
2	41	959	972	0.24	0.27
3	30	980	992	0.21	0.24
4	15	~960	993	0.19	0.20
5	8	994	1003	0.10	0.18
6	2	>1300†)	-	0.11	0.22
Series B					
1	49	944	972	0.25	0.29
2	41	945	972	0.24	0.27
3	32	965	995	0.23	0.26
4	26	1027	1044	0.23	0.24

*) Parent zeolite for series A and B. †) Sintering

No significant differences can be detected in the thermal stability for the different series of samples, i.e. the thermal stability and also the adsorption capacity are only determined by the aluminium content and not by the conditions of sample preparation.

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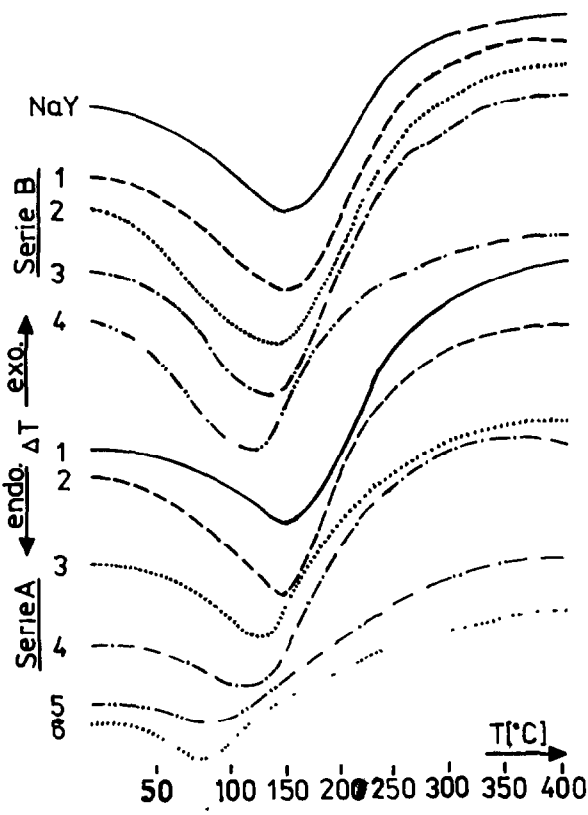

← Figure 1
Temperature dependence of the water desorption (samples of Table 1).

Figure 2
↓ Destruction temperature in dependence on the number of Al atoms per unit cell

• $T_{on(extr.)}$:
x Netzsch, series B, NaX, NaY
▣ " , series A
△ Derivatograph

T_p:
○ Netzsch, Derivatograph