

Characterization of Activated Carbon (Norit R1 Extra) by Adsorption Equilibria, Impedance Spectroscopy and ESCA-Measurements

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Introduction

To calculate and optimize industrial adsorption processes, basic information on the adsorbents, i.e. its inner surface, pore size distribution, number of active sites, etc. is needed. To get this quantities for activated carbon (AC) Norit R1 Extra, we have performed various measurements of adsorption isotherms (AIs), dielectric measurements, ESCA-measurements and mercury porosimetry.

Ar Adsorption Isotherm

The Ar AI has been measured at $T = 87$ K with an ASAP 2010 from micromeritics. The resulting pore size distribution is sketched in Figure 1 and the calculated value for the total pore volume is $0.626 \text{ cm}^3/\text{g}$.

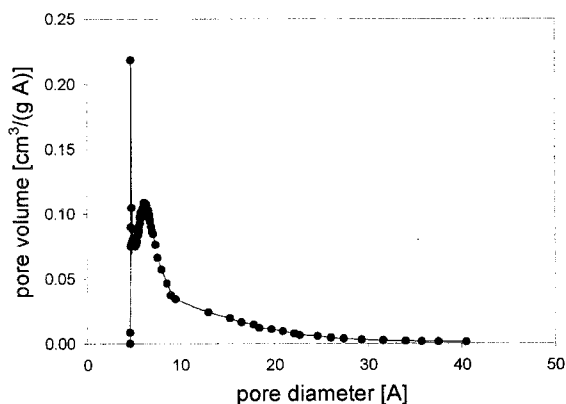


Figure 1. Pore size distribution of AC Norit R1 Extra.

N₂ Adsorption Isotherm

The N₂ AI measurements were carried out at 77 K using an ASAP 2400 (micromeritics). The AI is shown in Figure 2. The resulting values for the surface area and the pore volume are listed in table 1.

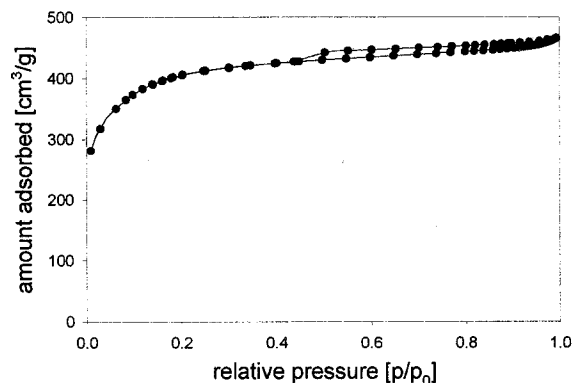


Figure 2. Adsorption and desorption isotherms of N₂ on AC Norit R1 Extra, $T = 77$ K.

Table 1. Analysis of the N₂ 77K AI and the CO₂ AIs (298 K, 323 K) on AC Norit.

micropore volume	total pore volume	micropore area	external surface
$0.616 \text{ cm}^3/\text{g}$	$0.715 \text{ cm}^3/\text{g}$	$1332.6 \text{ m}^2/\text{g}$	$74.7 \text{ m}^2/\text{g}$
method	N ₂ (77 K)	CO ₂ (298K)	CO ₂ (323K)
surface area m^2/g	1407.3	783.9	691.3

CO₂ Adsorption Isotherms

The AIs of sub- ($T = 298$ K) and supercritical ($T = 323$ K) CO₂ on AC Norit are shown in Figure 3. The experimental data represent AIs of Type II (IUPAC classification) reflecting mainly adsorption and capillary condensation in the micro- and mesopores of the activated carbon. The pore size distribution was calculated by the BJH-method. Agreement with the pore size distribution calculated from the Ar 87 K AI measurements and these from CO₂ AIs was satisfying. The resulting surface area was determined by a linear

BET-plot and the numerical value is about ½ of the surface area calculated from N₂ 77 K AI (cp. Table 2).

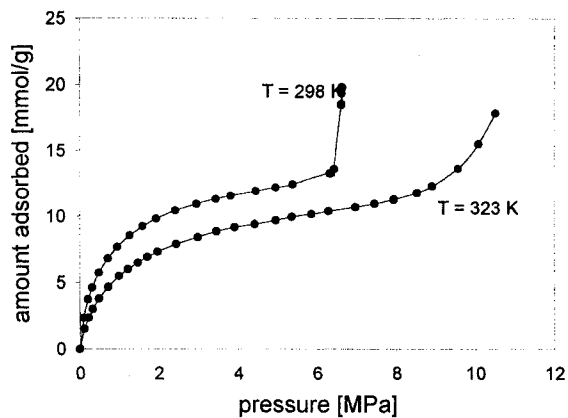


Figure 3 Adsorption isotherms of CO₂ on AC Norit R1 Extra, T = 298 K and T = 323K.

ESCA-Investigations

The ESCA measurements (Electron Spectroscopy for Chemical Analysis) deliver information about the concentration of different atoms and active sites in the activated carbon the results are listed in table 2.

Table 2. Concentrations of different atoms and active sites in AC Norit R1 Extra

atom	C	O	N	Ca	S	Si
portion %	93.7	4.8	0.4	0.2	0.2	0.7
active sites	-CH _x -O-C-		-C=O	O=C-O	-CO ₃	-C??
portio %	70	13	6	5	4	2

He-Adsorption isotherm

The bulk volume of the AC Norit has been determined by a He-pycnometer to 0.442 cm³/g. Adsorption equilibria of Helium on AC Norit R1 Extra have been measured at ambient temperature (298 K, 323 K) in the pressure range 0,1 MPa - 1.2 MPa. The isotherms increase steeply at low pressures and then reach a plateau with a saturation load. The amount of Helium being adsorbed tends nearly to be constant or at higher pressures to increase monotonously with increasing pressure. This slow increase of the AC sample mass can be explained by assuming Helium to

be *absorbed* in the chaotic quasi graphite structure of the AC.

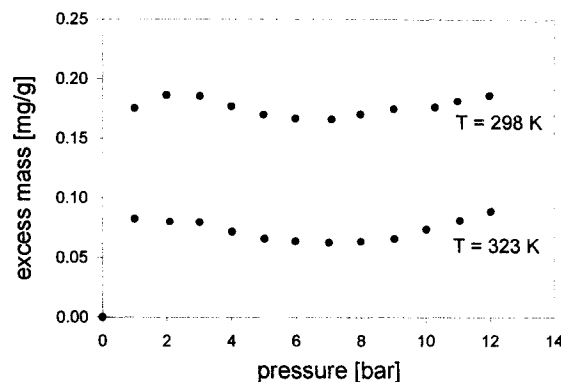


Figure 4. Adsorption isotherms of He on AC Norit R1 Extra, T = 298 K and T = 323K.

Mercury Porosimetry

The meso- and macropore analysis using a mercury porosimeter (Autopore II, micromeritics) shows a maximum in the pore size range of 1 µm. In the range between 0.5 and 0.01 µm and between 5 and 70 µm were no pores detected. The resulting total volume in the meso- and macropore range is 0.684 ml/g.

Impedance Spectroscopy

Physisorption equilibria of gases on porous solids can be characterized by measuring the (frequency dependent) capacitance of a capacitor filled with a sample adsorbens. This quantity strongly depends not only on the physico-chemical structure of the empty adsorbens in vacuum, but also on the permanent or induced dipole moments of the molecules adsorbed. The resulting impedance spectra measured during a regeneration process of the AC Norit R1 Extra are shown in Figure 5.

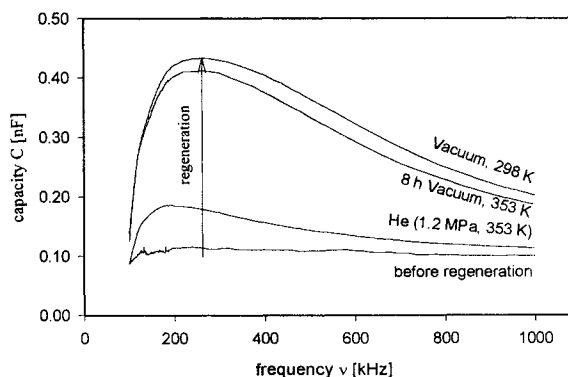


Figure 5. Impedance spectra of the regeneration process of the AC Norit R1 Extra, 100 kHz < ν < 1 MHz.