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Quantifying specific surface area, specific edge surface area and specific basal surface area of clay minerals by atomic force microscopy

F. Macht, K. Eusterhues, and K.-U. Totsche

Lehrstuhl für Hydrogeologie, Institut für Geowissenschaften, 07749 Jena, Germany (felix.macht@uni-jena.de, karin.eusterhues@uni-jena.de, kai.totsche@uni-jena.de)

The specific surface area (SSA) of a mineral is an essential parameter for quantifying processes at the solid-liquid interface. Since basal surfaces (00l) and edge surfaces (hk0) of phyllosilicates possess entirely different reactive surface groups, it is furthermore desirable to separately determine the SSA of these crystal planes. Here, we present two methods to measure the SSA of phyllosilicates by atomic force microscopy (AFM):
(1) Assuming an ideal crystal body we determined the height h, the perimeter of the basal plane p, the area of the basal plane a, and the volume v from the AFM-data. With the known mineral density rho, both the edge surface area (ESA) and the basal surface area (BSA) can be calculated.
(2) Our second method is based on a triangulation procedure. Thereby, each height point of the scan is linked with its two nearest neighbors to a triangular polygon. The total SSA is then calculated by summing up the surface area of all polygons, which make up the crystal body, while the ESA is determined by ESA=SSA–BSA.
We applied both methods to ca. 100 AFM images of a montmorillonite and an illite. For the illite we found an average SSA of 126m2/g by method (1) and 128m2/g by method (2). This is in good agreement with 115m2/g as determined by ethylene-glycol-monoethyl-ether adsorption (EGME), but significantly higher than 46m2/g as measured by N2-BET. The ESA was determined as 4m2/g (method 1) and 2m2/g (method 2). For the montmorillonite we found a SSA of 789m2/g by method (1) and 778m2/g by method (2). This is roughly twice as high as 462m2/g (determined by EGME), and ∼10 times higher than the N2-BET value of 71m2/g. The ESA was determined as 12 m^2/g (method 1) and 1 m^2/g (method 2).
In conclusion, AFM-determined SSA values are similar or in the same order of magnitude than EGME values, but 3 -10 times higher than N2-BET values. Both AFM- methods yield very similar results. This shows that the assumption of an ideal crystal body is a good approximation for phyllosilicates. However, by using the triangulation method (2) it should be possible to determine the SSA of more irregular shaped bodies as well.