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Size dependent pore size distribution of shales by gas physisorption

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Gas physisorption, in particular nitrogen adsorption-desorption, is a traditional technique for characterization of geomaterials including the organic rich shales. The low pressure nitrogen is used together with adsorption-desorption physical models to study the pore size distribution (PSD) and porosity of the porous samples. The samples are usually crushed to a certain fragment size to measure these properties however there is not yet a consistent standard size proposed for sample crushing. Crushing significantly increases the surface area of the fragments e.g. the created surface area is differentiated from that of pores using BET technique.
In this study, we show that the smaller fragment sizes lead to higher cumulative pore volume and smaller pore diameters. It is also shown that some of the micro-pores are left unaccounted because of the correction of the external surface area. In order to illustrate this, the nitrogen physisorption is first conducted on the identical organic rich shale samples with different sizes: 20-25, 45-50 and 63-71 μm. We then show that such effects are not only a function of pore structure changes induced by crushing, but is linked to the inability of the physical models in differentiating between the external surface area (BET) and micro-pores for different crushing sizes at relatively low nitrogen pressure. We also discuss models currently used in nano-technology such as t-method to address this issue and their advantages and shortcoming for shale rock characterization.