

# Changes in specific surface area and morphology of kaolin group minerals with pressure treatment

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Pressure can tailor the properties of a material by changing its atomistic arrangements and/or crystal morphology. We have investigated the changes in the adsorption properties of kaolin group minerals as a function of pressure treatment in gigapascal range. External pressure has been applied using a Large Volume Press (LVP) to kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ) and halloysite ( $\text{H}_4\text{Al}_2\text{O}_9\text{Si}_2 \cdot 2\text{H}_2\text{O}$ ), which represent natural 2D layered and 1D nanowire structures, respectively. Powdered samples have been compressed up to 3 GPa in 1 GPa steps at room temperature and recovered by up to  $\sim 0.35\text{g}$  from each pressure step. The BET (Brunauer, Emmett, Teller) measurement was conducted using  $\text{N}_2$  gas to measure the specific surface area, pore size distribution, and pore volume of the pressure-treated samples. Before pressure-treatment, the specific surface area of kaolinite has been measured to be  $10.702(\text{m}^2\text{g}^{-1})$ , which increases to  $12.090(\text{m}^2\text{g}^{-1})$  after 1 GPa,  $17.101(\text{m}^2\text{g}^{-1})$  after 2 GPa, and  $17.864(\text{m}^2\text{g}^{-1})$  after 3 GPa treatments. These lead to a gradual increase in the total volume of adsorbed pores from  $0.01216(\text{cm}^3\text{g}^{-1})$  at ambient, to  $0.01368(\text{cm}^3\text{g}^{-1})$  after 1 GPa,  $0.074337(\text{cm}^3\text{g}^{-1})$  after 2 GPa, and  $0.09546(\text{cm}^3\text{g}^{-1})$  after 3 GPa treatments. Unlike Kaolinite, the specific surface area of halloysite decreases from  $47.476(\text{m}^2\text{g}^{-1})$  from ambient to  $44.348(\text{m}^2\text{g}^{-1})$  after 1 GPa,  $41.579(\text{m}^2\text{g}^{-1})$  after 2 GPa, and  $42.796(\text{m}^2\text{g}^{-1})$  after 3 GPa treatment, which results in overall decrease in the total volume of the pore from  $0.1914(\text{cm}^3\text{g}^{-1})$  at ambient to  $0.1863(\text{cm}^3\text{g}^{-1})$  after 1 GPa,  $0.1572(\text{cm}^3\text{g}^{-1})$  after 2 GPa, and  $0.1187(\text{cm}^3\text{g}^{-1})$  after 3 GPa treatments.

We discuss the contrasting effects of pressure-treatment on the two morphologically-distinct kaolin group minerals based on SEM (Scanning Electron Microscope) images and analyses of selected diffraction peaks measured on each recovered material. We observe that the layers in kaolinite separate into smaller units upon increasing pressure treatment, whereas the tubes in halloysite become flattened, which led to the contrasting changes in surface area and pore volume. Further study is in progress to compare these results to when water is used as a pore-penetrating pressure-transmitting medium.

Keywords: kaolinite, halloysite, high pressure treatment, specific surface area, morphology

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### ABSTRACT

Pressure can tailor the properties of a material by changing its atomistic arrangements and/or crystal morphology. We have investigated the changes in the adsorption properties of kaolin group minerals as a function of pressure treatment in gigapascal range. External pressure has been applied using a Large Volume Press (LVP) to kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ) and halloysite ( $\text{H}_4\text{Al}_2\text{O}_9\text{Si}_2 \cdot 2\text{H}_2\text{O}$ ), which represent natural 2D layered and 1D nanowire structures, respectively. Powdered samples have been compressed up to 3 GPa in 1 GPa steps at room temperature and recovered by up to  $\sim 0.35\text{g}$  from each pressure step. The BET (Brunauer, Emmett, Teller) measurement was conducted using  $\text{N}_2$  gas to measure the specific surface area, pore size distribution, and pore volume of the pressure-treated samples. Before pressure-treatment, the specific surface area of kaolinite has been measured to be  $10.702(\text{m}^2\text{g}^{-1})$ , which increases to  $12.090(\text{m}^2\text{g}^{-1})$  after 1 GPa,  $17.101(\text{m}^2\text{g}^{-1})$  after 2 GPa, and  $17.864(\text{m}^2\text{g}^{-1})$  after 3 GPa treatments. These lead to a gradual increase in the total volume of adsorbed pores from  $0.01216(\text{cm}^3\text{g}^{-1})$  at ambient, to  $0.01368(\text{cm}^3\text{g}^{-1})$  after 1 GPa,  $0.074337(\text{cm}^3\text{g}^{-1})$  after 2 GPa, and  $0.09546(\text{cm}^3\text{g}^{-1})$  after 3 GPa treatments. Unlike Kaolinite, the specific surface area of halloysite decreases from  $47.476(\text{m}^2\text{g}^{-1})$  from ambient to  $44.348(\text{m}^2\text{g}^{-1})$  after 1 GPa,  $41.579(\text{m}^2\text{g}^{-1})$  after 2 GPa, and  $42.796(\text{m}^2\text{g}^{-1})$  after 3 GPa treatment, which results in overall decrease in the total volume of the pore from  $0.1914(\text{cm}^3\text{g}^{-1})$  at ambient to  $0.1863(\text{cm}^3\text{g}^{-1})$  after 1 GPa,  $0.1572(\text{cm}^3\text{g}^{-1})$  after 2 GPa, and  $0.1187(\text{cm}^3\text{g}^{-1})$  after 3 GPa treatments.

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