Geophysical Research Abstracts
Vol. 21, EGU2019-3878, 2019
EGU General Assembly 2019

Preliminary study on the phase transformation and thermal behaviors of birnessite with 20 nm and 50 nm particle sizes

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Birnessite is a layer-structured manganese oxide consisting of octahedral MnO_6 layers, with cations and water molecules filling the interlayer space. It is a common clay mineral readily found in nature, such as in soil or deep sea manganese nodules. The excellent cation-exchange ability and chemical activity of birnessite make it important in many applications, such as a pollutant adsorbent in environmental engineering, an electrode substitute in Li-batteries, or a good Eh/pH indicator in geology. According to the relevant literature, particle size is an important factor in controlling the critical properties, i.e., the electrochemical characteristic and adsorbing ability in regard to toxic substances. While the smaller sized birnessite performs better in enhancing the capacity for adsorbing toxicants, the thermal instability may hinder it to some extent, albeit this is still not well understood. Previous studies found that birnessite will dehydrate generally at about 200˚C and transform into tubular manganese oxide above 400˚C. However, the exact mineral phases and temperature ranges remain uncertain, and the effect of particle size for thermal stability is still unclear.

In this study, we used the oxidation method to synthesize birnessite powder (about 20 nm in diameter), and then aged it for 5 days to get the larger sized birnessite (about 50 nm). The two different sized birnessite powders were then heated in both thermogravimetric analysis (TGA) and dilatometer at various temperatures to measure the weight and volume changes. XRD and SEM were used to determine the mineral phases and particle sizes at each specific temperature. The semi-quantitative data showed that the starting 20 nm and 50 nm birnessite powders have similar composition, i.e., their Na/Mn ratios are 0.20-0.22, they all dehydrate before 400˚C, and transform gradually to tunnel mineral $Na_{0.2}MnO_2$. Furthermore, the one at 20 nm transforms at around 500˚C, while the 50 nm one delays the transformational temperature to ∼600˚C. Finally, we used the Master Kinetic Curve model (MKC) developed by our research team to derive a number of curves that can adequately describe and predict the phase transformations of birnessite.