

## **Aluminum ion occupancy in the structure of synthetic saponites: Effect on crystallinity**

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### **ABSTRACT**

Two series of saponites with fixed (Si+Al)/Mg and Si/Mg ratios, respectively, were synthesized by using hydrothermal methods. The obtained products were characterized by XRD, XRF, <sup>27</sup>Al, and <sup>29</sup>Si MAS NMR, SEM, and TEM. XRD patterns showed that well-ordered saponites were obtained in the initial Si/Al ratio range of 5.43–7.89. Beyond this Si/Al ratio range, poorly crystallized saponites were obtained with small crystallized particles, which can be seen from TEM images. When intercalating saponite with surfactant, the intercalated products displayed strong and well-ordered (00 $l$ ) reflections, indicating that layered saponite has been successfully synthesized in the present study. <sup>27</sup>Al MAS NMR spectra demonstrated that well-crystallized synthetic saponites had a higher Al(IV)/Al(VI) ratio than the poorly crystallized samples, which is an important factor affecting the crystallinity of synthetic saponite. A one-to-one substitution (i.e., 1 Al<sup>3+</sup> → 1 Mg<sup>2+</sup>) actually occurred in the octahedral sheet and this substitution had a negative effect on the crystallinity of the synthetic saponites. After grafting the synthetic saponites with silane, the decreased intensity of the <sup>29</sup>Si NMR signal at –86 ppm and the increased intensity of Q<sup>3</sup> Si(0Al) and Q<sup>3</sup> Si(1Al) signals strongly suggested that the signal at ca. –86 ppm corresponded to Q<sup>2</sup> Si at the layer edges of saponite.

**Keywords:** Synthetic saponite, occupancy of aluminum ion, crystallinity, <sup>27</sup>Al and <sup>29</sup>Si MAS NMR, isomorphous substitution