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# Effects of Freezing on Colloidal Halloysite: Implications for Temperate Soils

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**Abstract:** The literature reports that freezing of aqueous aluminosilicate clay suspensions can produce clay aggregates that disperse with agitation. Our experiments indicate that colloidal suspensions extracted from north Idaho loess soils (Boralfs), when frozen, form silt- and sand-sized agglomerates that resist dispersion with agitation. XRD and TEM analyses showed that colloids are dominantly halloysite between 0.10 to 0.30  $\mu\text{m}$  in diameter. The colloidal halloysite has anomalously high mole ratios of Si/Al and a high Fe content. Freeze-produced agglomerates are light yellow to yellowish-brown in color, occur in the form of plates, wedges, cusps, or laths, and often exhibit uniform optical properties, suggesting a crystalline product. Selected area electron diffraction, however, indicates that the agglomerates are poly-crystalline. With proper orientation, agglomerates produce acute bisectrix interference figures that are optically negative with variable  $2V$ , generally  $<30^\circ$ . XRD analyses and IR spectra imply that the agglomerates are halloysite. Measured optical properties, however, are different than those reported for halloysite and may be affected by the high Fe content and polycrystalline nature of the agglomerates. Grains, exhibiting similar optical properties as laboratory-produced agglomerates, are a minor proportion of the very-fine sand fraction in some horizons of the soils studied. Freeze-aided agglomeration of colloidal material may be an important process in temperate climates. It may be overlooked because of particle destruction by soil pretreatments or morphological order and/or optical similarity to some micaceous minerals.

**Key Words:** Fragipan • Idaho • Loess • Tephra • Transmission electron microscopy • X-ray diffraction

*Clays and Clay Minerals*; December 1991 v. 39; no. 6; p. 642-650; DOI: [10.1346/CCMN.1991.0390610](https://doi.org/10.1346/CCMN.1991.0390610)  
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