
Natural and Synthetic Copper Phyllosilicates Studied by XPS

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Abstract: X-ray photoelectron spectroscopy (XPS) has been used to characterize the bonding state of Cu^{2+} , Si^{4+} , Al^{3+} , and O^{2-} ions in structural (octahedral and interlamellar) or adsorbed position in phyllosilicates. Five smectites, 5 kaolinites, and 1 chrysocolla with Cu(II) in known positions (octahedral, interlamellar, or surface adsorbed) have been investigated. Their spectra were compared with those of pure Cu metal and of pure Cu(I) and Cu(II) oxides.

The line for Cu $2p_{3/2}$ (binding energy of 935.4 eV) and well-defined shake-up lines (binding energy of about 943 eV) observed after 1 hr of X-ray irradiation are characteristic of Cu(II) in phyllosilicate octahedral sites. But due to the photoreduction effect, they show Cu(I) oxidation states (Cu $2P_{3/2}$, binding energy of 933.2 eV and near absence of shake-up lines) for the phyllosilicates with adsorbed Cu or in interlamellar positions. The kinetics of photoreduction distinguishes octahedral from interlamellar positions, and the latter from a surface adsorbed position. The enlargement of the FWHM (full width at half maximum) of XPS lines has been used to describe crystallochemical parameters linked to local ordering around the probe cations. Crystallization produces decreasing O 1 s and Cu 2p (octahedral cation) line widths but has no effect on the Si 2p (tetrahedral cation) line width. The enlargement of FWHM for all ion lines of the lattice is linked to the nature ($\text{Cu} > \text{Mg} > \text{Al}$) and the number and amount of structural cations in the phyllosilicates.

Key Words: Chrysocolla • Cu • ESCA • Kaolinite • Smectite • XPS

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