Microscopic characterization of Activated Carbon for Fluoride Removal.

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Water supply in Mexico relies on groundwater subtraction, affecting 75% of the total population (1). In the north part of the country fluoride has a natural origin in groundwater and almost all water sources exceed the actual permissible levels of exposure (2). The MCL (maximum contaminant level) established by WHO is of 1.5 mg/L. Water that contains fluoride above the MCL must to be treated. Among the different techniques for fluoride depletion are precipitation, ion exchange and the use of different adsorbents such as activated carbon (AC).

AC has to be modified in order to develop affinity for certain contaminants, such as fluoride (3-5). AC surface can be acidic, basic and neutral depending on the presence of surface functional groups. As such, modification of chemical characteristics is taken to adjust the inherent surface functionality. It has been widely recognized that chemical species removal by AC adsorption is due predominantly to the surface complex formation between the species and the surface functional groups. This is especially significant in the case of removing inorganics and metals from aqueous solutions (3).

In the present work, aluminum and iron were loaded into commercial AC (Aquactiv, Clarimex). Impregnation was made using 15 g of AC and 250 mL of Al $(NO_3)_3 \cdot 9\ H_2O$ with an Aluminum concentration of 15 ppm. Adsorption was made in a mechanical shaker (SI-600 JEIO TECH) at 150 rpm, 30 °C and a pH=3.5. After that period, AC was washed and impregnated with a 0.05 M iron nitrate solution. Afterwards it was washed until neutral pH and it was dried at 120 °C for 24 h. Finally AC was calcined in a furnace at 500 °C during one hour. Fluoride removal was attained with well water provided from Chihuahua City, and fluoride concentration was measured by a potentiometric method (4). Samples were studied in high resolution transmission electron microscopy (HRTEM) by Z-contrast and bright field micrographs in STEM mode using a JEOL JEM-2200FS microscope operated at 200kV and spherical aberration correction for scanning mode (STEM). The chemical analysis was made by characteristic X-ray energy dispersion spectroscopy (EDS) with an INCA-X-Sight (Oxford Instruments) energy dispersive spectrometer coupled to the microscope.

Fluoride removal increases almost 30% when AC is aluminum and iron loaded. The capacity measured according to Langmuir model was of 14.61 mg/g. At pH 5.5, the fixed pH value used during isotherm processing, aluminum and iron could form complexes with fluoride. Fluoride sorption in this impregnated material is chemical in nature. The high electronegativity of fluoride must direct a specific interaction with Al and Fe. In order to strength explanation regarding its strong affinity for these metals and the high fluoride capacity, several HRTEM micrographies and EDS analysis was performed.

In figure 1, HRTEM micrography for Aluminum and Iron activated carbon loaded evidences both elements among other major ones, finely distributed forming nanostructures along the surface. These

fine elemental dispersion provides the main nucleus which attain fluoride adsorption.

For instance, chemical modification by Fe and Al impregnation is detrimental for fluoride removal, forming homogeneously distributed complexes into the activated carbon surface.

References:
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[6] This research was performed by the aid of CONACYT and the Nanotechnology Laboratory at CIMAV

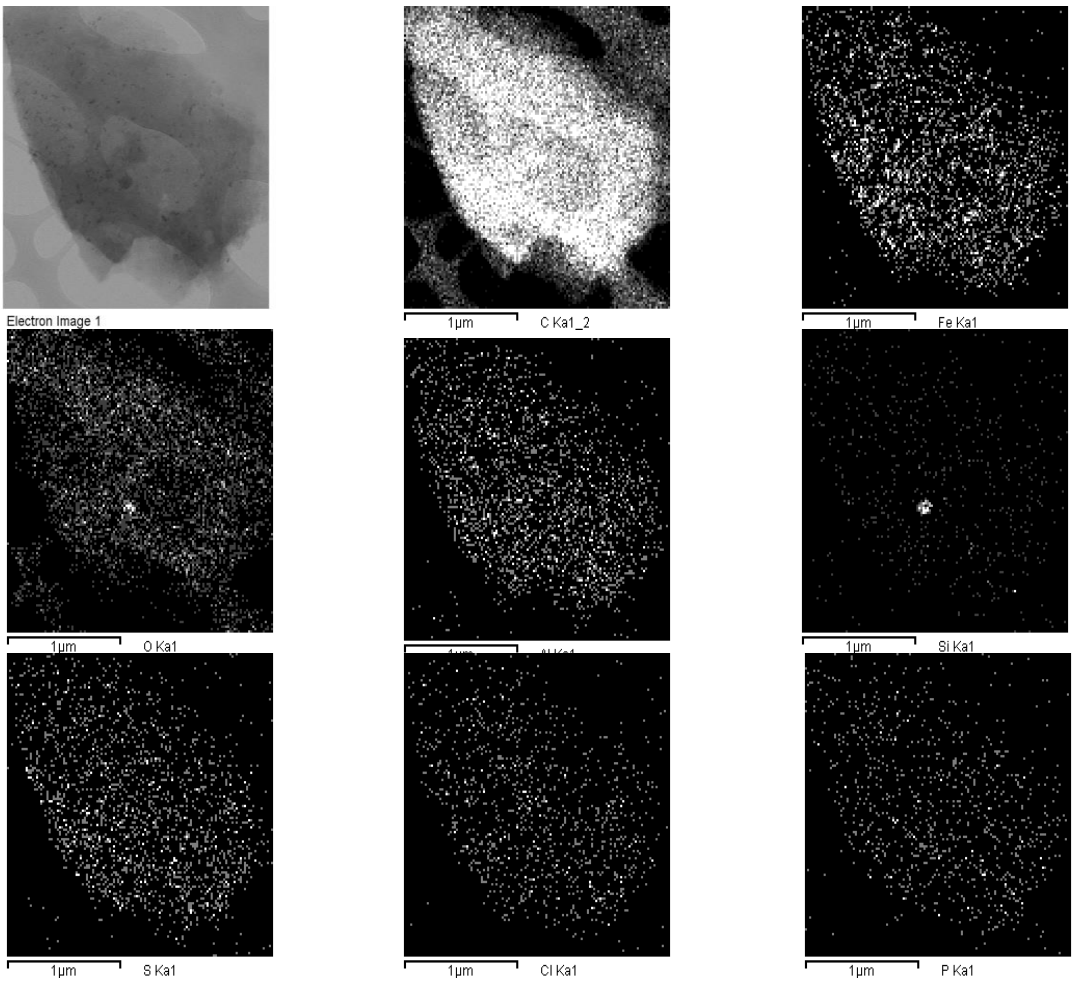

Figure 1. HRTEM-EDS for activated carbon loaded with Aluminum and Iron