Immobilized coupling product (CP) of chitosan with humic acid (HA) was prepared in order to overcome the solubility limitations in using chitosan and HA as an adsorbent for heavy metals. Two CPs were prepared; FCP from Fluka humic acid and JCP from Ajloun humic acid (Jordan). The prepared CPs were found to be insoluble in the pH range from 2 to 12. The CPs were studied by elemental analysis, Ba(OH)<sub>2</sub> titration, potentiometric titration, FTIR, solid <sup>13</sup>C-NMR, SEM and XRD. The results indicated the formation of covalent amide/ester bonds and electrostatic bonds (~~~NH<sub>3</sub><sup>+</sup>...<sup>-</sup>OOC~~~) in the CP. The uptake of Pb(II) and Cd(II) by CP was found to increase with increasing pH and ionic strength, while, the reverse was observed in the case of  $Cr_2O_7^{2-}$  which indicated a unique behavior of CPs compared with free HA and chitosan. The adsorption capacities of Pb(II), Cd(II), and Cr(VI) were 33.0, 24.7 and 56.5 mg/g, respectively, in the case of FCP, and 54.9, 20.1 and 54.1 mg/g, respectively, in the case of JCP. The effect of time and temperature on adsorption were investigated and was found to be fast, endothermic and entropy driven. The SEM indicated tight surface of CPs due to the strong bonding between chitosan and humic acid. To overcome this problem, CPs were spread over silica gel to produce a high capacity adsorbent (486 mg Pb/g CP) which facilitated removal of low concentrations of Pb < 30 ppm with about 100 % efficiency.