Emergent environmental issues, ever-shrinking global petroleum reserves, and unstable fossil fuel costs continue to spur interest in the development of sustainable biofuels from renewable feed-stocks. The development and viability of all biofuel fermentations, however, remains limited by numerous factors, including both feedback inhibition by toxic products as well as inefficient and energy intensive product separation processes. To circumvent both feedback inhibition and product recovery issues, researchers have turned their attention to incorporating energy efficient separation techniques into fermentations via in situ product recovery (ISPR) approaches. This thesis focuses on the characterization of two novel adsorbents for the recovery of alcohol biofuels from model aqueous solutions as the first step towards developing an effective product recovery strategy for sustainable biofuel synthesis. First, a hydrophobic silica aerogel was evaluated as a biofuel adsorbent through characterization of its equilibrium behavior with both conventional second generation biofuels. The adsorbed alcohol capacity at biologically relevant conditions proved to be marginal as a result of limited diffusion and adsorption within the pores. However, pore intrusion and adsorption is greatly enhanced at exceedingly high concentrations of alcohol in the aqueous phase, resulting in the observance of Type IV adsorption isotherms for all biofuels studied. Additionally, pore intrusion significantly affects the dynamic binding efficiency within a packed adsorption column. However, as a result of the low adsorptive capacity, the silica aerogel would not be effective in future biofuel ISPR applications. The second class of materials evaluated as biofuel adsorbents through characterization of equilibrium and kinetic behavior was mesoporous carbons. Variations in synthetic conditions enabled facile tuning of specific surface area and pore morphology of adsorbents. The adsorbed alcohol capacity increased with elevated specific surface area of the adsorbents. Although adsorption capacity is comparable to commercially-available, hydrophobic polymer adsorbents of similar surface area, pore morphology and structure of mesoporous carbons greatly influenced adsorption rates. Multiple cycles of adsorbent regeneration rendered no impact on adsorption equilibrium or kinetics. The high chemical and thermal stability of mesoporous carbons provide potential significant advantages over other commonly examined biofuel adsorbents. Correspondingly, mesoporous carbons should be further studied for biofuel ISPR applications.