Solvent Stability of Functionalized Silica Gels

Regular or chromatographic silica gels manufactured from water glass may be susceptible to dissolution by some solvents (methanol over 20% v/v). When this phenomenon occurs, the solution appears slightly cloudy as observed after running a flash column. The incomplete polymerization of the silica monomers (SiO_4^{4-}) is responsible for this effect as shown in figure 1.

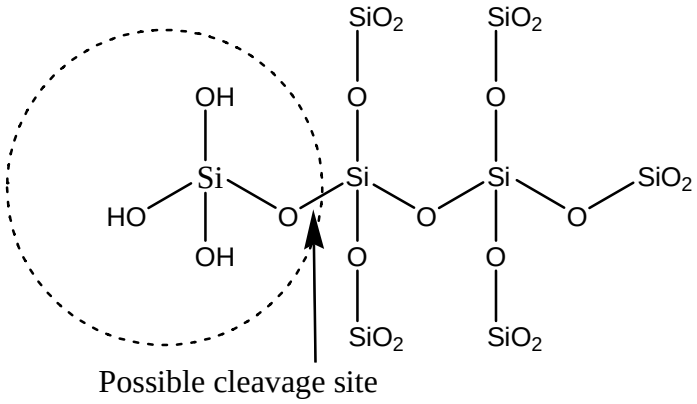

Figure 1.*Regular silica gel with monomer susceptible to dissolution.*

This type of silicate tetrahedron attached to only one other tetrahedron is named Q1 (^{29}Si NMR nomenclature) where a silicate tetrahedron linked to 2 other tetrahedrons is called Q2, to 3 others, Q3 and finally, a silicate tetrahedron linked to 4 other tetrahedrons is called Q4. In a standard silica gel, most of the silicates are Q4 and Q3 with a little quantity of Q2 and Q1. These last two types are the least stable and are responsible for the dissolution of silica in methanol.

SiliCycle® functional gels, however, do not suffer from this shortcoming because, like reversed-phase silica gels, they are end-capped. End capping is a method by which residual silanol groups (-OH) are reacted to render the surface unreactive and hydrophobic, and thus protects it from the solvent dissolution effect. This step typically follows the initial grafting reaction. Figure 2 demonstrates the steps of producing a functional silica gel from bare silica gel. We have tested different functional silica gels under reflux conditions in methanol and toluene and we observed no dissolution of silica or functional group cleavage.

As it is common to use 100% methanol with reversed-phase chromatographic silica gels, we have ensured, by utilizing the above process, excellent stability when you use SiliCycle® functionalized silica gels.

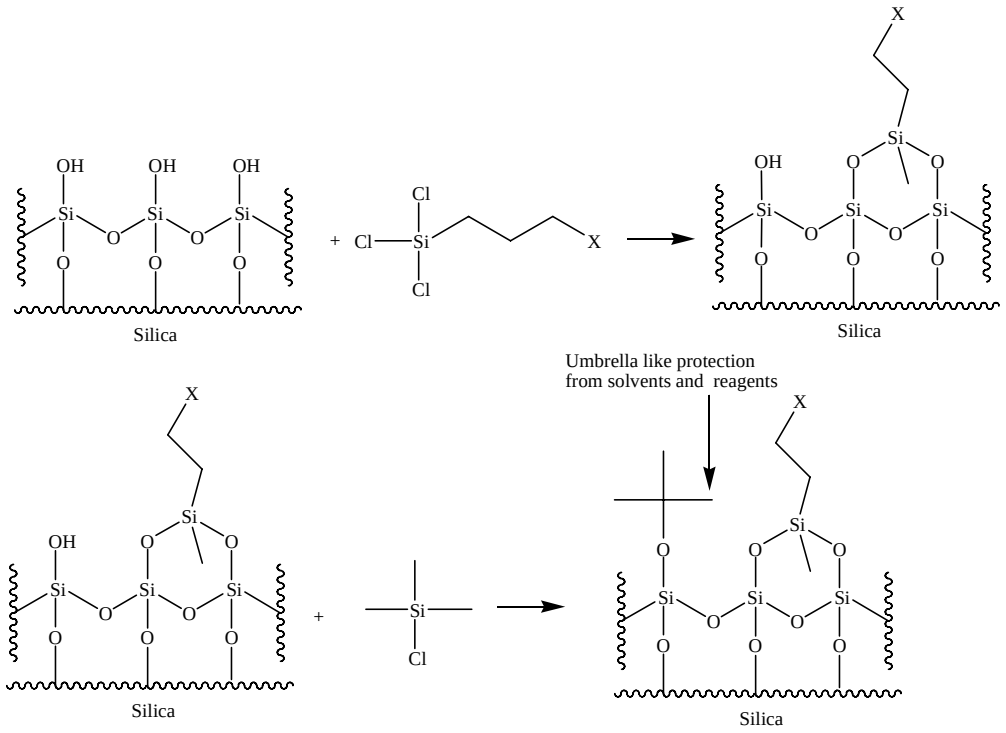

Figure 2. *End capping: Reaction steps to render a functionalized silica surface unreactive.*