

## ***Determination of the Kinetics of Curing of Cyanoacrylate-based Adhesives with Fourier Transform Infrared Spectroscopy***

### **Background**

Cyanoacrylate-based adhesives are low-viscosity, rapid curing adhesives that are commonly used for quick fix applications. They cure within a few minutes, and provide a brittle, non-space filling bond between two substrates. The adhesives rely on moisture to initiate the curing reaction.

### **Chemistry**

Cyanoacrylate cements contain a monomer group possessing an unsaturated carbon-carbon bond. The monomer is stabilized with a weak acid to prevent curing in the container. To initiate the reaction, the acid stabilizer must be neutralized with a weak base. Airborne or surface adsorbed water is usually sufficient to neutralize the weak base, although an alkaline surface will also suffice. The amino acid content in skin, in addition to the water content, initiates the polymerization reaction very well. Once the stabilizer is neutralized by the water, the monomer polymerizes via an anionic reaction. Only a small amount of water is necessary to catalyze the initiation reaction. As the monomer polymerizes, the carbon-carbon double bond is consumed, as shown in Figure 1.

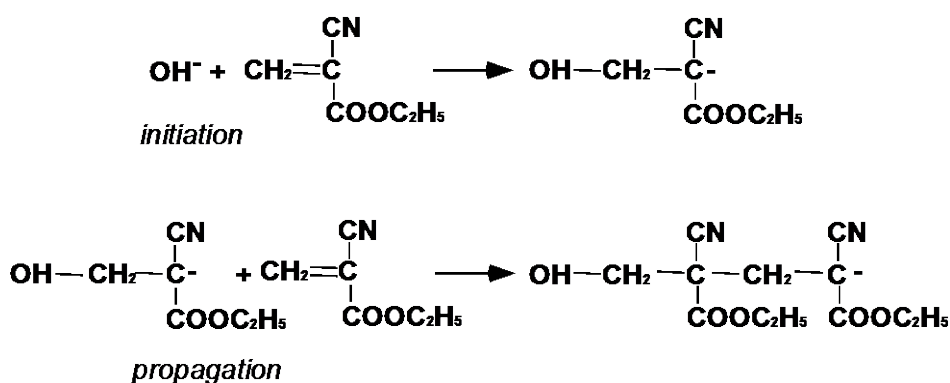


Figure 1: Initiation and propagation reactions for cyanoacrylate-based adhesives when exposed to a weak base.

### **Reaction Kinetics**

In order to monitor the polymerization kinetics of cyanoacrylate adhesives, we often employ Fourier Transform Infrared Spectroscopy (FTIR). FTIR is used to identify and quantify chemical groups, such as C-H bonds or C=C bonds, which absorb infrared light at specific wavelengths.

By identifying a specific chemical group that is consumed in a chemical reaction, FTIR can be used to monitor the kinetics and extent of reaction. In the case of cyanoacrylate adhesives, the carbon-carbon double bond in the monomer is consumed during the polymerization. This bond absorbs infrared light at several wavelengths, and is easily monitored.

The next step was to setup a reproducible reaction environment. In particular, the initiation step had to be reproducible. We found that the surface of borosilicate glass provided a sufficiently basic surface as to initiate the polymerization reaction. Although borosilicate glass absorbs infrared light at wavenumbers below 2000 cm<sup>-1</sup>, a suitable peak related to the C=C bond was found at 3128 cm<sup>-1</sup>, as shown in Figure 2. As the sample polymerizes, this bond decreases in size, as shown in Figure 3.

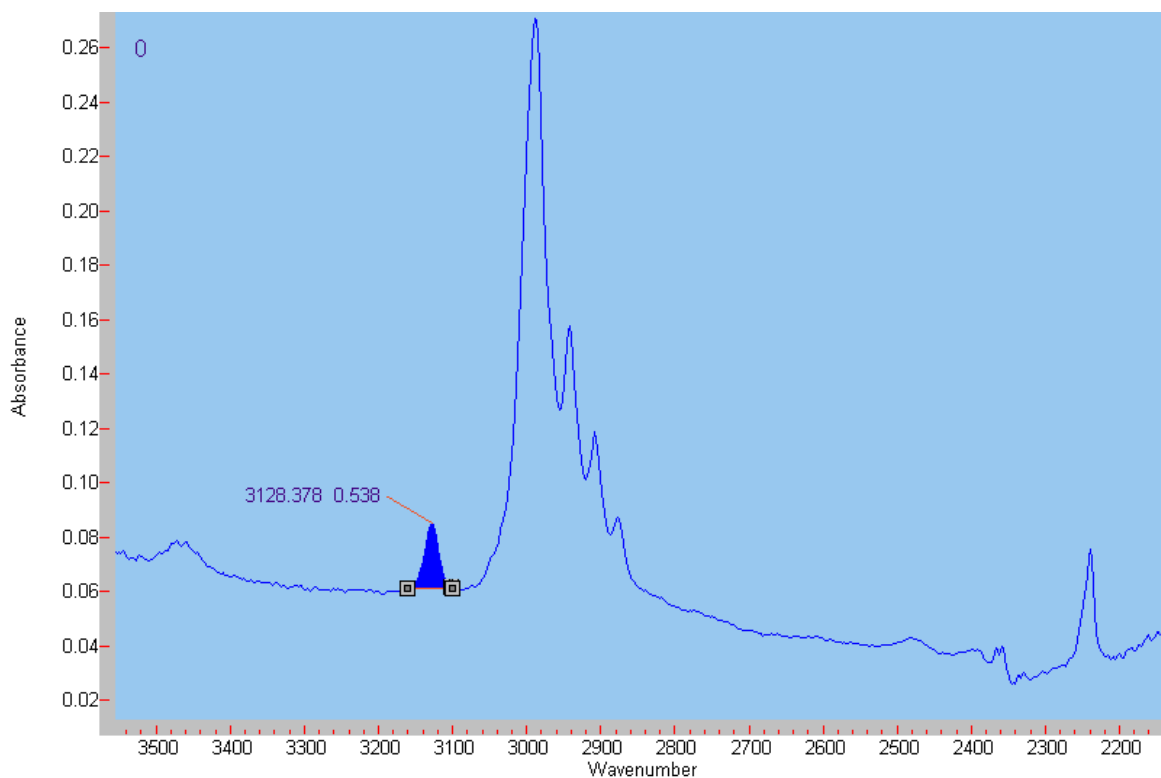


Figure 2: Infrared spectrum showing C=C bond absorption at 3128 cm<sup>-1</sup>.

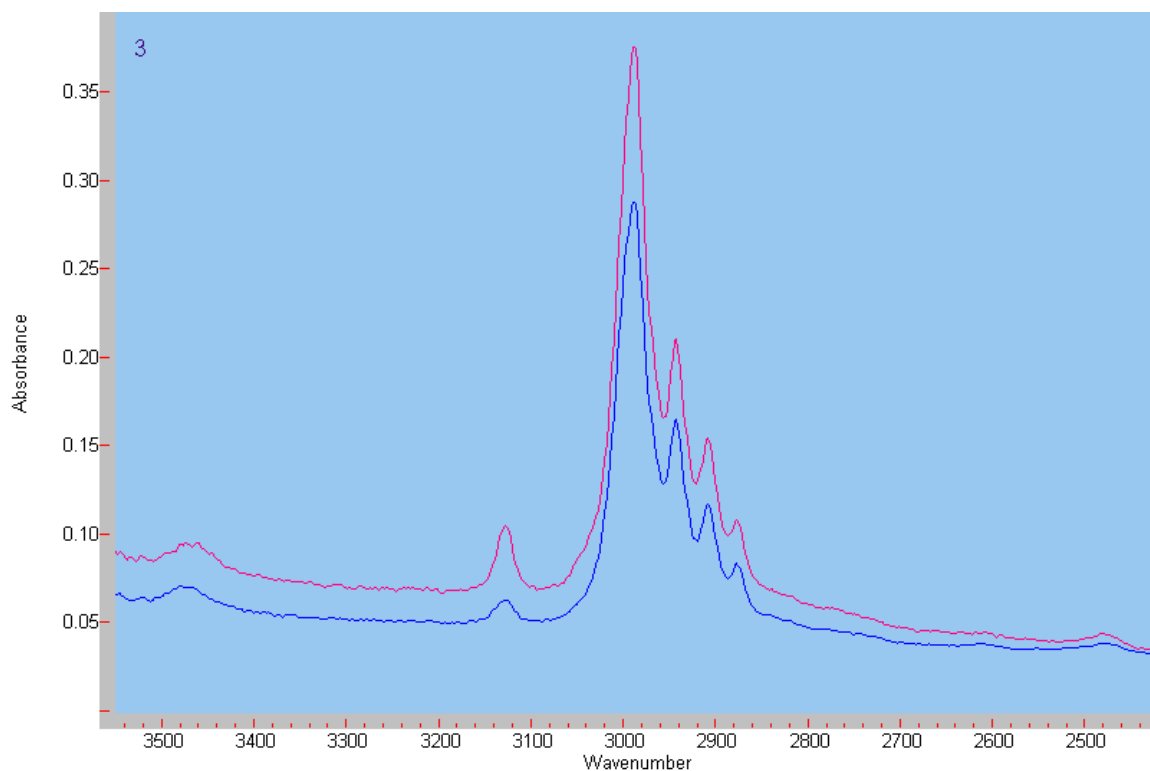


Figure 3: Infrared spectrum showing disappearance of C=C bond absorption at 3128 cm<sup>-1</sup> as sample polymerizes.

To monitor the reaction, a swab was used to apply a commercially-available cyanoacrylate adhesive to the surface of a glass slide. In order to normalize concentration variations, the area under the peak at 3128 cm<sup>-1</sup> was normalized with the methylene stretching peak at 2900 cm<sup>-1</sup>, as shown in Figure 4.

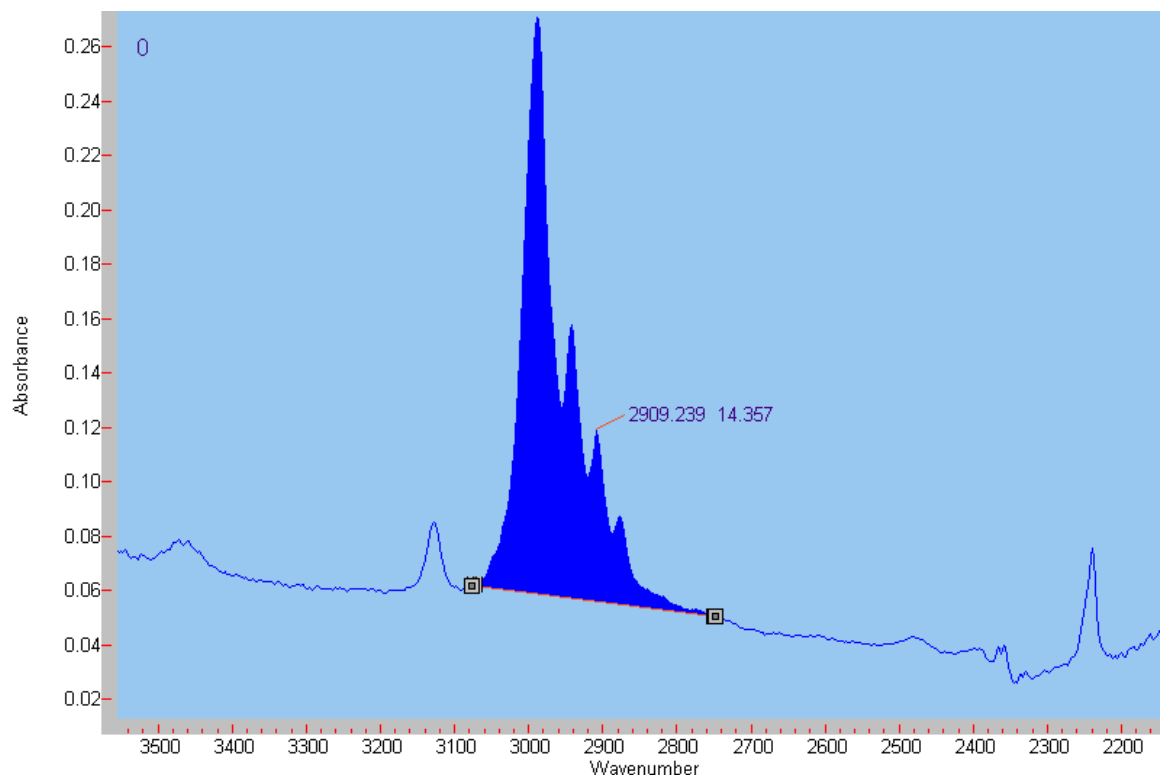


Figure 4: Reference peak at 2900 cm<sup>-1</sup>, showing methyl group stretch. This peak is not affected by the extent of cure, and is thus an ideal candidate to normalize the C=C absorption peak.

The ratio-ing of absorption peaks results in an index, such as the cure index  $I_{cure}$  shown below:

$$I_{cure} = \frac{A_{3128}}{A_{2900}}$$

When  $I_{cure}$  approaches either zero or a steady state value, the material is fully cured.

The curing kinetic results on the commercially-available cyanoacrylate adhesive are shown in Figure 5. The curing curve shows that the material is fully cured between 4-5 minutes. With this technique, the curing behavior in different environments (relative humidity, temperature) and on different substrates can be quantified.

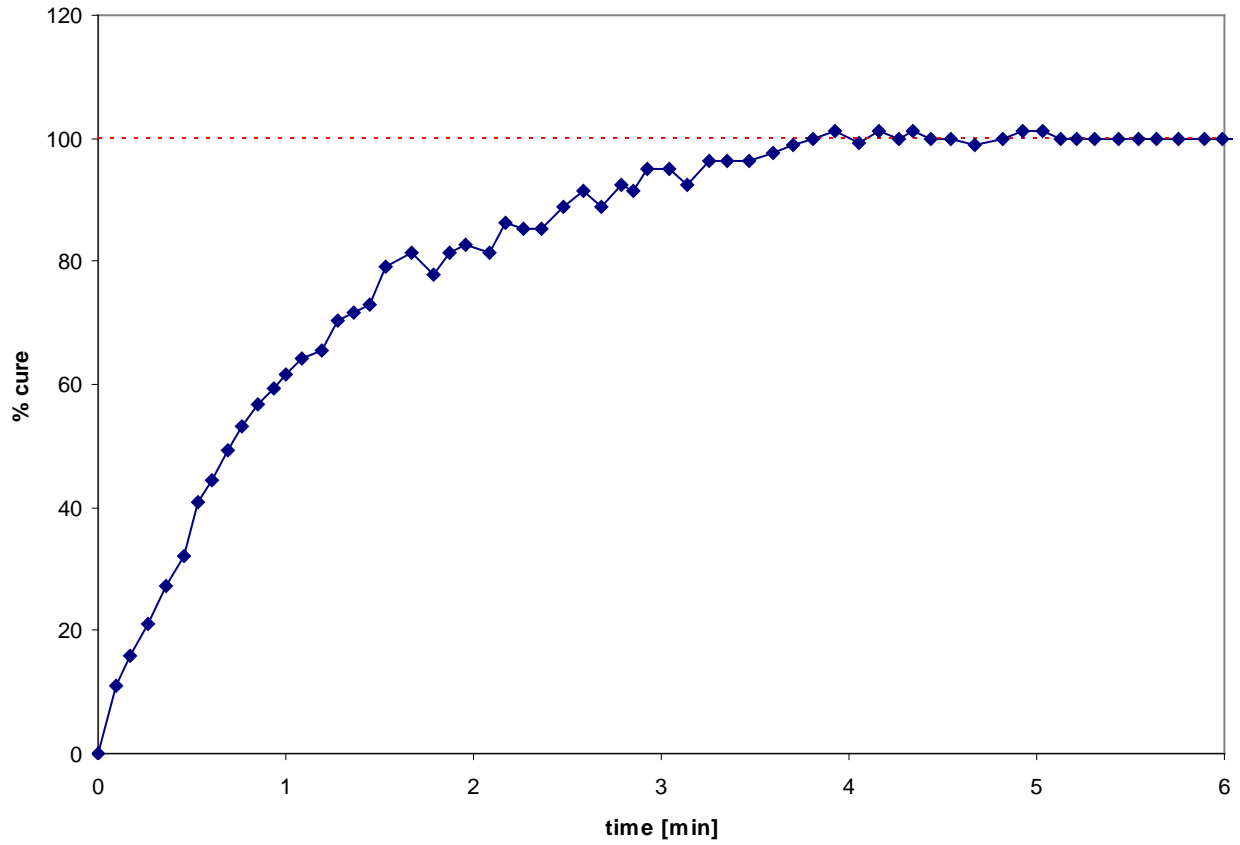


Figure 5: Curing kinetics of commercially-available cyanoacrylate-based adhesive.