

Adhesive Performance Improvements With Next Generation Surface Insensitive Cyanoacrylates

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Commonly known as instant adhesives, cyanoacrylates have been widely used in assembly processes for the past 50 years. They are single-component adhesive formulations that cure at room temperature, bond to a wide variety of substrates, and have exceptionally fast cure speeds.

Cyanoacrylate adhesives are ideally suited for bonding applications in high-throughput and fast cycle time manufacturing operations. These adhesives are used for bonding various components on loud speakers such as surrounds, gaskets, and voice coils, and for bonding plastics, metals, and elastomers on disposable medical devices such as catheters and tubesets.

The first cyanoacrylates were predominantly ethyl cyanoacrylate monomer based materials. They helped many customers realize significant gains in productivity versus slower curing solvent-based, water-based, or multi-component products.

In 1986, the first surface insensitive cyanoacrylate (SICA) adhesive was developed. SICA formulations used patented technology to improve cure speed on dry or acidic surfaces such as pine, polyolefin plastics, and plated metals. The resulting products helped manufacturers increase productivity on an even wider variety of substrates.

Recently introduced next generation SICA adhesives offer improved shelf-life stability and even faster initial cure speeds than current surface insensitive technology. Next generation products maintain performance properties such as cure speed and viscosity better as they age. Therefore, end users enjoy a fast-curing cyanoacrylate product from the first to the last drop in the bottle.

Test Methodology

A comparison test between four next generation SICA products from Henkel versus current cyanoacrylate technology from 13 adhesive manufacturers was performed in order to benchmark performance improvements. As the data presented shows, next generation SICA products exhibit vastly improved shelf-life stability compared to current surface insensitive cyanoacrylates.

The tested adhesives were selected based upon their overall market share and breadth of their parent manufacturer's product line. Products that were described by product and technical data as "surface insensitive" were selected first. In some cases products described as "very fast curing", "high performance", or "for difficult to bond substrates" were tested. A single adhesive was tested from 13 competitive suppliers.

All products were tested in their original containers from the manufacturer. Containers were either 20 gram or 1 ounce bottles or aluminum tubes. All product labels were removed from the packaging to ensure unbiased testing. In addition, every effort was made to obtain the most recent lot of product available along with any support documentation such as certificates of conformance and dates of manufacture.

To facilitate the comparison, all products were grouped into one of four categories based on viscosity: wicking, low viscosity, high viscosity, and gel. Comparisons were made only among products of similar viscosity. Wicking grade products were generally less than 20 cP, low viscosity products approximately 100 cP, high viscosity products 300-700 cP, and thixotropic gels packaged in aluminum tubes.

Testing was conducted at both room temperature (70°F) and at 50°C. All cyanoacrylate adhesive manufactures recommend that product be stored in refrigerated (40°F) conditions. Although many adhesive distributors do comply, end-users rarely do. Therefore, testing at room temperature rather than refrigerated conditions was conducted.

The testing at 50°C was an accelerated aging test with products tested after 0 (control), 2, 4, 6, and 8 weeks of aging. Historically, two weeks of aging at 50°C is roughly equivalent to six months of aging at room temperature. The room temperature test is a real time test with test points at 0 (control) 3, 6, 9, and 12 months. Testing at room temperature will more accurately simulate the product variation customers will normally observe in production. The results presented in this article summarize only accelerated aging testing. The real-time testing is currently ongoing and is scheduled for completion in the Fall of 2004.

Table 1: test matrix.

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	Weeks at 50°C				Months at Room Temperature				
	0	2	4	6	8	3	6	9	12
Fixture Time- steel									
Fixture Time- polycarbonate									
Fixture Time- Pine									
Viscosity									

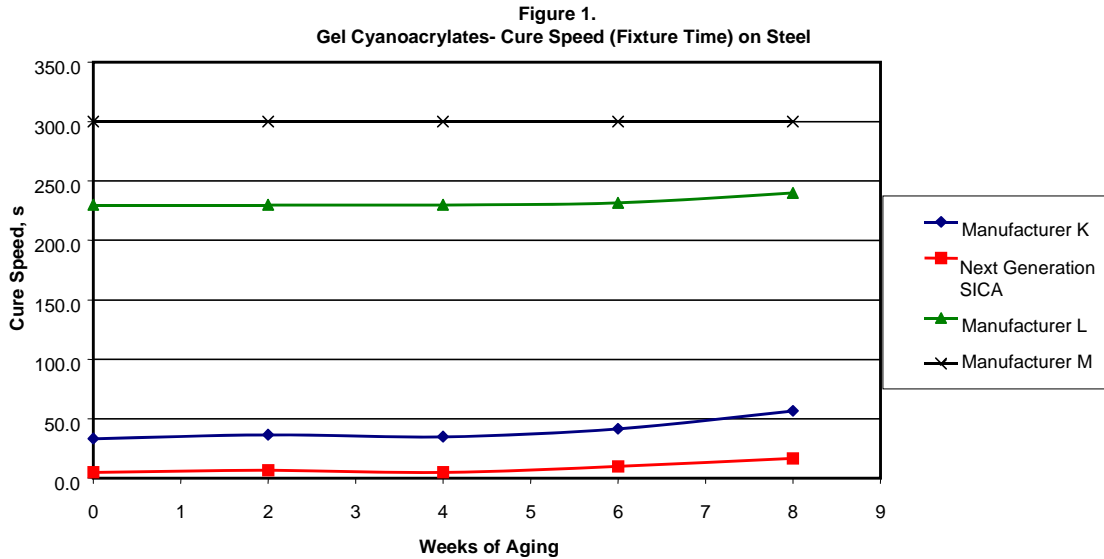
Fixture time and viscosity tests compared the performance of the various products. The fixture time test was performed on three different substrates: steel, polycarbonate, and pine. Steel and polycarbonate are common substrates in many cyanoacrylate adhesive applications, and are broadly representative of performance on metals and plastics. Historically, many cyanoacrylate adhesives have achieved very slow fixture speeds on pine, making it an excellent substrate to determine if a product is truly surface insensitive.

Fixture time is commonly defined as the cure time required for a lap shear assembly to support a 3 Kg weight. For cyanoacrylate adhesives, fixture time is normally measured in seconds. In this specific test, a bond area of 0.5 square inches was used, which results in a bond strength of 13.2 psi. Testing was stopped after 5 minutes, even if no fixture occurred.

Viscosity measurements were obtained with a Haake RT20 Rotovisco Viscometer with a 60 mm cone and plate. The cone and plate type viscometer requires only 1-2 milliliters of product for testing as compared to 300 milliliters required for a Brookfield type viscometer. The cone and plate were washed with a phosphoric acid solution to prevent curing during testing.

Results

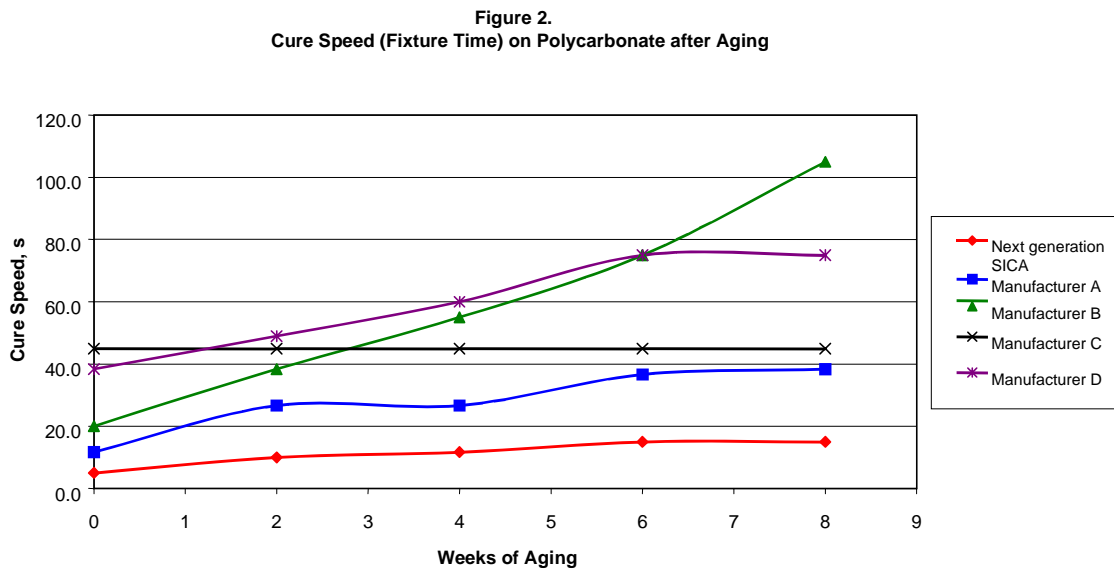
Figure 1: cure speed results of various gel cyanoacrylate products on steel.



As the data in Figure 1 shows, the next generation SICA has the fastest initial fixture time on steel. Fixture speed increases slightly throughout the eight weeks of aging. The closest performing product (Manufacturer K) had an initial fixture speed of 33 seconds versus 5 seconds for the next generation SICA. At the end of the eight weeks of aging, the fixture time of the Manufacturer K product increased by 24 seconds to 57 seconds, while the next generation SICA increased only 12 seconds to 17 seconds.

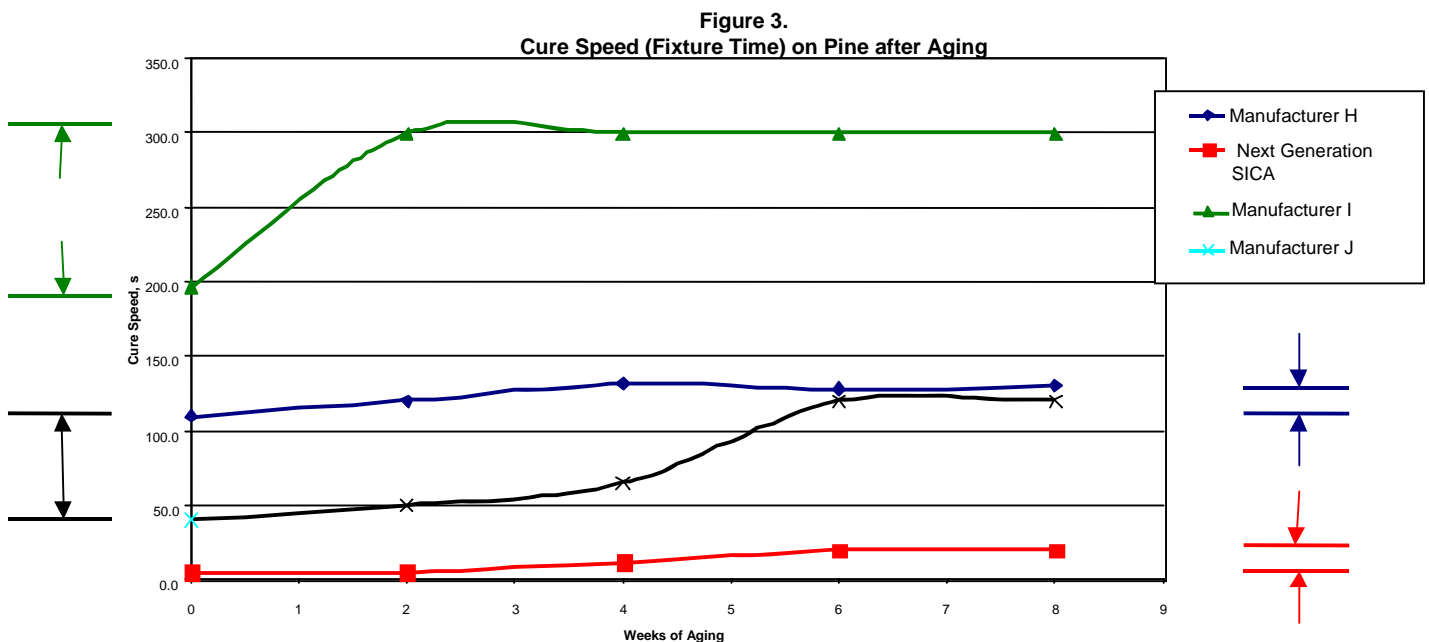
All other products, had fixture times in excess of 200 seconds throughout the heat aging test. As testing was stopped after 300 seconds, it is likely that the fixture times for the Manufacturer M product would have been much longer. All of the products shown in Figure 1 are marketed as "surface insensitive" products. As the testing shows, there is a wide disparity in performance for seemingly similar products. Similar results were obtained on pine and polycarbonate.

Figure 2: fixture time results of various wicking grade products on polycarbonate.



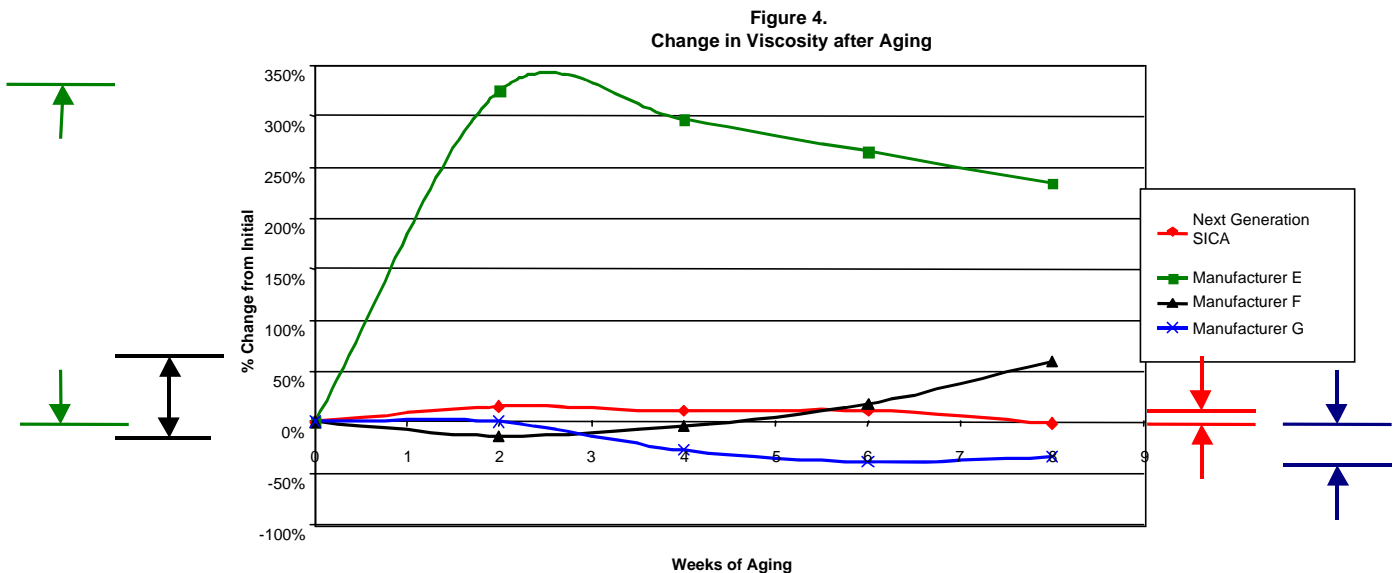
The Manufacturer A product and the next generation SICA are “surface insensitive cyanoacrylates”. The products from Manufacturers B and C are standard ethyl grade adhesives. Because cyanoacrylate products typically have fast cure speeds on polycarbonate, the least amount of variation among the products was seen. The next generation SICA performed the best, with an initial fixture time of less than 10 seconds. Fixture time increased to only 15 seconds throughout the heat aging test. The competitive surface insensitive product from Manufacturer A had an initial fixture time of 15 seconds that increased to 38 seconds by the end of the aging test. The ethyl-based products from Manufacturers B and C had initial fixture speeds of 20 and 40 seconds respectively. Their fixture times continued to slow throughout the test to 105 seconds and 75 seconds respectively.

Figure 3: fixture time testing of various high viscosity cyanoacrylates on pine.



The Manufacturer H product is labeled “very fast curing.” The Manufacturer I product is a standard ethyl based adhesive, while Manufacturer J has a surface insensitive product. As the data indicates, the next generation SICA out-performs current surface insensitive technology and standard ethyl-based technology. The next generation SICA had an initial fixture speed of 5 seconds compared to 40 and 110 seconds for the Manufacturer J surface insensitive product and the Manufacturer H “very fast curing” product . The ethyl-based Manufacturer I product had an initial fixture speed of 200 seconds. After two weeks of aging, the Manufacturer I product reached the maximum fixture time of 300 seconds. After eight weeks of aging, the fixture times for the Manufacturer J and H products increased to 120 and 130 seconds respectively.

Figure 4: change in viscosity over time with various low viscosity cyanoacrylate products.



The Manufacturer E product is a surface insensitive grade. The Manufacturer F material is highly stabilized for viscosity consistency. Manufacturer G is an ethyl based cyanoacrylate. Figure 3 shows that the next-generation SICA adhesive had the least viscosity variation. Throughout the heat-aging test, its viscosity changed by no more than 15 percent. While typical viscosity tends to increase over time due to thickening of the adhesive, variation either positive or negative is undesirable. For applications requiring precisely metered amounts of adhesive, viscosity variation can lead to endless tweaking of dispensing parameters especially in pressure-time systems.

The data collected as part of this test indicates that next generation SICAs offer significant improvement in fixture speed retention and viscosity stability compared to other available surface insensitive cyanoacrylate products. Fixture speed is the most significant difference in performance, especially on pine and steel.

Conclusion

Next generation SICA formulations have minimized the variability in fixture speed and viscosity throughout the entire shelf-life of the product. These materials also offer the fastest fixture speeds attainable in the industry on dry or acidic surfaces.

To the end user, these performance improvements mean reduced scrap, fewer inspection steps, simplified process control, more reliable and precise dispensing, fewer fixtures needed for process, a smaller area footprint for process, and more consistent throughput.

Many production processes rely upon custom fixtures to move assemblies through the process. If an adhesive's cure speed were to slow, additional fixtures are needed to extend the cure time without disrupting the cycle time of the process. Additional fixtures, cost money and require additional space. The example below illustrates these issues in greater detail.

In a hypothetical assembly process with a 10 second cycle time under optimal conditions, a cyanoacrylate adhesive fixtures in 5 seconds. The process works well as the cycle time exceeds the fixture time. However, based on the data above, a customer using the Manufacturer C instant adhesive will experience a significant increase in fixture time if the adhesive batch is older than 12 months. (Fixture time could increase as much as six times.) Since the adhesive that once fixtured in 5 seconds now takes 30 seconds, the cycle time is too short. At this point the engineer can either increase the work-in-process by adding more fixtures, reduce the throughput of the process, use of an accelerator to speed fixture, or change to more recent lot of product. All of these options either reduce the efficiency of the process or add cost.

Room temperature testing of these same adhesives is currently being conducted. These results will be analyzed in a similar fashion to the 50°C accelerating aging data. Verification testing of the accelerated aging data by an independent third party is also planned.

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