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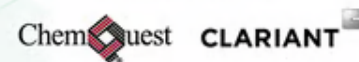
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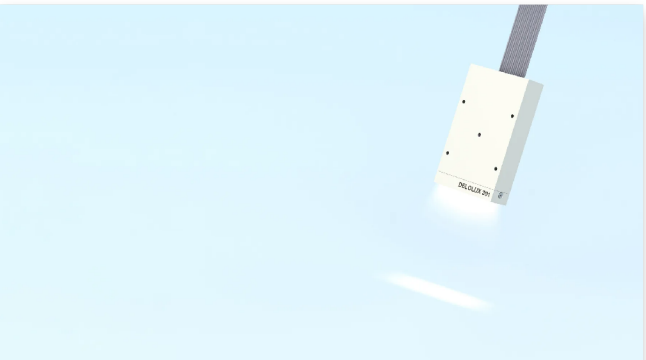
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FROM THE EDITOR

>> Karen Parker, editor-in-chief

ADHESIVE AND SEALANT INDUSTRY MEETS IN NOLA

When ASC President Bill Allmond said to me in a podcast we recorded about the ASC's 2023 Convention and EXPO that he was excited to "bring everybody back together again," I didn't really get the full meaning of that statement. After my first time attending the event last week in New Orleans, I understand it a little better. This industry is a tightknit group. You like each other; you enjoy getting together. Every public space that was available – from the coffee shop to the bar – was full of adhesive and sealant folks chatting it up.

The event reminded me that we do our best work when we interact and collaborate with others. People come together at events like the ASC convention because talking to other people about what you are doing and learning about work others are doing in their respective fields sparks new ideas and new ways to solve problems. Likewise, seeing friends and colleagues in a more relaxed setting offers a pause that allows people to return to work with a more energized and hopeful approach to their jobs. Events give us many opportunities to learn, both formally and informally.

For me, the convention had many educational opportunities, with discussions and presentations focused on some of the most pressing concerns within the industry, such as the supply chain, the regulatory landscape, and sustainability. And technical presentations discussed the latest developments for adhesive formulators. I was especially impressed with Michael Okoroafor's keynote address on Wednesday morning. He is the chief sustainability officer at the spice company McCormick & Co., and he offered insights into how his company partners with suppliers and the broader community to create a more sustainable business model. It was especially interesting because Okoroafor's background is in packaging — which, Okoroafor noted, faces challenges in terms of sustainability and its contribution to GHG emissions. Adhesives will play a key role as the packaging industry solves those problems.

One of the highlights of the event was the presentation of the ASC's Innovation Awards. In its seventh year, the awards honor the top three innovations from the industry. This year, the council received 30 submissions, and independent judges reviewed them against a list of categories and weightings.

The primary award was presented to Albert Giorgini and the team at H.B. Fuller for their development entitled "EV Protect 4006 Lightweight Encapsulant for Lithium-Ion Batteries." This technology represents a two-component foaming, flame-retardant polyurethane encapsulant that prevents or inhibits thermal runaway in lithium-ion battery packs. During the awards ceremony, we watched a video that demonstrates how the technology inhibits runaway fires in those packs. Congratulations to H.B. Fuller for this award and for developing a technology that will make the electric vehicles we drive much safer.

Nvirovate received the first runner up award for a development called "Soybean Oil PSA: Brand ESG & Sustainability Enabler." The company is a start-up that is developing pressure-sensitive adhesives from soy oil, creating a closed-loop, sustainable product that will be better for the environment. Xlynx received the second runner up prize for its innovation, "Development of Universal Covalent Adhesives for Use with Low Surface Energy Polymers." Make sure to read [this article](#) in the October 2022 issue of **ASI** that features the technology from Xlynx.

Speaking of new technology, I am excited for you to read this month's **ASI**, with articles on innovations available to adhesive formulators. Within the eMagazine is an article from King Industries about the performance of non-tin catalysts as tin replacements, providing desirable regulatory and performance attributes. Also featured is the article from Cardolite's presentation at the 2022 Polyurethanes Technical Conference, which discusses the application of renewable, cashew nutshell liquid-based polyols in CASE and polyurethane foams. I hope you enjoy this issue of **ASI**, and I hope to see you soon at another industry event!

SILANE TERMINATED POLYMER REACTIONS WITH NON-TIN CATALYSTS

Research demonstrates the performance of non-tin catalysts as a replacement for tin, satisfying regulatory concerns and performance requirements.

By John Florio, Dymin Morillo, and Dylan LaFerriere of King Industries, Inc., USA

Polymers based on organosilane chemistries are widely used in a variety of applications including adhesives, sealants, and coatings. Organosilanes are capable of forming covalent bonds between organic and inorganic compounds. They are gaining interest as alternatives to more hazardous chemistries, for example, polyurethane applications that use hazardous polyisocyanates. Organosilane polymers can promote adhesion, weatherability, and reinforcement of coatings, adhesives, sealants, and fillers. Hence, they are widely used for DIY projects and commercial construction projects for a variety of indoor and outdoor applications on a variety of substrates.

Organosilanes can be designed as coupling agents that form links between inorganic substrates and organic polymers (Figure 1). In some cases, organosilane coupling agents can act as co-catalysts to provide a synergistic effect with certain other more conventional catalysts.

VIEW FIGURE 1

Organosilane crosslinking chemistries include one-component moisture cured systems that can be based on different functionalized backbone polymers that have alkoxy silane terminal groups. The backbone chemistries of these systems could be polyether, polysiloxane, or polyurethane.

Organosilane polymers can crosslink with other functional groups to form products with structural properties through a variety of mechanisms that involve two basic reactions: hydrolysis of alkoxy silane groups and condensation of formed silanol groups with other silanol groups or with alkoxy silane groups. The product of the condensation reaction is a crosslinked siloxane (-Si-O-Si-). The basic silanol condensation reaction mechanism forming a siloxane and a volatile byproduct is shown in Figure 2. The volatile byproducts are sometimes a limiting factor for some applications. All of these reactions can be accelerated by several different catalyst mechanisms. The curing process can be very complex, with cure rates dependent on several variables.

VIEW FIGURE 2

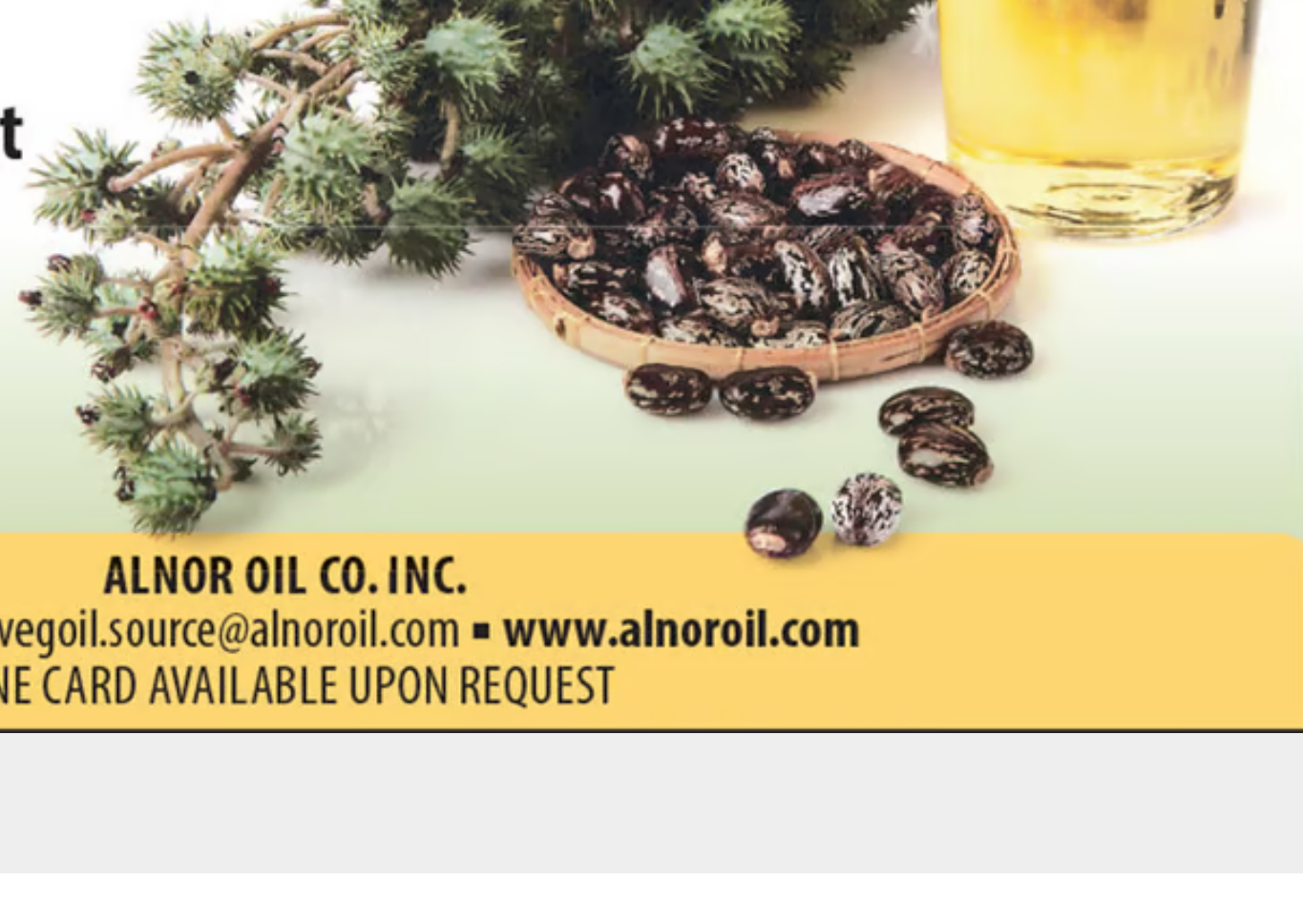
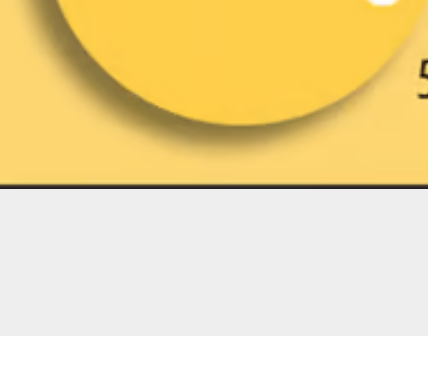
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This article initially reviews the crosslinking process of alkoxy silane terminated systems, followed by an in-depth summary of different catalyst processes for organosilane chemistries. Finally, catalyst evaluations of three organosilane chemistries is presented to address tin catalyst environmental and toxicity issues.

Crosslinking Organosilane

Figure 3 shows a basic structure for a dialkoxy silane polymer. Both hydrolysis and condensation processes are involved in curing one-component moisture-cure alkoxy silane systems, and the reaction rates for both processes can be increased by incorporating acid, base, or organometal catalysts. Figure 4 shows a sequence for the hydrolysis of a dialkoxy silane generating a silanol and two moles of alcohol byproduct. Siloxane generating condensation reaction mechanisms are shown in Figures 5 and 6. Figure 5 shows the silanol-silanol condensation reaction generating the siloxane and a water byproduct, and Figure 6 shows a mechanism for a silanol-alkoxy silane condensation reaction generating a crosslinked siloxane structure and an alcohol byproduct. In practice, hydrolysis and condensation occur concurrently unless special efforts are made to separate the steps.¹

VIEW FIGURE 3

VIEW FIGURE 4

VIEW FIGURE 5

VIEW FIGURE 6

Catalysis of Organosilane Crosslinking²

The hydrolysis and condensation reaction rates of organosilane systems are dependent on pH of the system and on the substituents on the silicon. The rate minimum for the hydrolysis and condensation reactions occurs at approximately pH 7 and pH 4, respectively. Each pH change of one unit in either direction produces a ten-fold rate acceleration assuming an excess of water is available. At pH > 10, hydrolysis of the first intermediate, $RSi(OR)_2OH$, is inhibited due to the ionization of the silanol group.³ The effect of pH on the organosilane hydrolysis and condensation reaction rates is illustrated in Figure 7. Both hydrolysis and condensation reactions are reversible. Alcohols will reverse the silane hydrolysis.

VIEW FIGURE 7

Osterholtz and Pohl described specific mechanisms for each of the acid and base catalyzed reactions. Torry, Campbell, Cunliffe and Tod studied activity of organometallic compounds in organosilane systems and propose tin compounds as the most active. Tin catalysts must initially hydrolyze to form the active species.⁴ Diocetyl tin and dibutyl tin compounds that catalyze many of these crosslinking reactions include dioctyl tin diacetylacetate and dibutyl tin dilaurate. However, concerns about toxicity of tin compounds have driven formulators to explore other catalyst options. Finding a non-tin catalyst that could provide sufficient reaction acceleration for all silane crosslinking chemistries is difficult. Some acids might provide good acceleration of the crosslinking reaction, but the acid might also accelerate rearrangement of the formed polysiloxane backbone, causing product degradation. Some tertiary amines could accelerate the crosslinking reaction, but they might also contribute color and odor.

Base Catalyzed Hydrolysis of Alkoxy silanes

Osterholtz and Pohl proposed a bimolecular nucleophilic displacement reaction depicted in Figure 8 for base catalysis of alkoxy silane hydrolysis, involving a pentacoordinate intermediate, and two different transition states. In the presence of a base and water, the transition state T.S.1 is formed with a partial negative charge on the alkoxy silane silicon. T.S.1 then dissociates to generate the pentacoordinate intermediate, which subsequently breaks down to the desired silanol through a second transition state. Negative charge development on the silicon in the transition state (T.S.1 or T.S.2) is considerable. This is supported by experimental observations where electron-withdrawing substitution on the alkyl groups attached to the Si accelerates the rates of hydrolysis. This is due to the stabilization of the developing negative charge, which lowers the energy of the transition states.

In General Base Catalysis of the hydrolysis of alkoxy silanes, any basic species accelerates the reaction by assisting the removal of a proton from water in the transition state. In Specific Base Catalysis of the hydrolysis of alkoxy silanes, the hydroxide anion accelerates the reaction rate by directly attacking the substrate.

VIEW FIGURE 8

Base Catalyzed Condensation of Silanols

Chojnowski and Chrzczonowicz found that secondary and tertiary amines (piperidine, triethylamine, tri-n-butylamine) catalyzed the condensation of a series of dialkyl- or diarylsilane diols in aqueous dioxane.⁵ They proposed that primary and secondary amines catalyzed the reactions by nucleophilic attack on silicon, which was followed by the rapid attack of dialkylsilane diol on the formed silamine, as shown below in Figure 9.

Acid Catalyzed Hydrolysis of Alkoxy silanes

The mechanism for acid catalyzed hydrolysis (Figure 10) is a rapid equilibrium protonation of the substrate, followed by a bimolecular S_N2 -type displacement of the leaving group by water.

Acid Catalyzed Condensation of Silanols

The acid catalyzed condensation reaction proceeds by an initial protonation of the silanol followed by an S_N2 displacement reaction at the Si, leading to formation of water and regeneration of the acid catalyst (Figure 11).

VIEW FIGURE 9

VIEW FIGURE 10

VIEW FIGURE 11

Tin Catalyst Mechanism

Tin catalyst can be in the form of dioctyl tin and dibutyl tin diacetylacetate and dibutyl tin dilaurate (DBTDL). Catalysis of organosilanes with tin compounds begins with hydrolysis of the tin compound. The tin compound undergoes hydrolysis and forms an organotin hydroxide (Figure 12), which is the true catalytic species.⁶ The organotin hydroxide reacts with an alkoxy silane group to form an organotin silanolate (Figure 13). The tin silanolate will react readily with alcohols and water to form the silanol (Figure 14). The organosilanol will also react with formed silanol groups to produce siloxane linkages, and the organotin hydroxide catalyst is regenerated (Figure 15).

Activity of the tin catalysts can be dependent on the pH of the system. For example, additional acid can affect the hydrolysis mechanism in Figure 12 by forcing the equilibrium to the left and preventing the formation of the active tin hydroxide. Also, strongly acidic species may interact with already formed basic tin hydroxide to prevent further reaction with alkoxy silane groups.

VIEW FIGURE 12

VIEW FIGURE 13

VIEW FIGURE 14

VIEW FIGURE 15

Tin Regulations

There has been increased examination of human health risks and environmental issues associated with tin compounds since restrictions for anti-fouling paints for marine markets began in the early 1980s. In 1999, the International Maritime Organization adopted a global resolution prohibiting the use of organotin compounds acting as biocides in anti-fouling systems by January 1, 2008.⁷ Organotin compounds are currently regulated by The International Maritime Organization's Antifouling System (IMO AFS) convention and the U.S. Organotin Antifouling Paint Control Act (OAPCA). The European Commission Decision 2009/425/EC claims organotin compounds in consumer products pose a risk to human health, increasing the scope of restrictions beyond maritime markets.

2009/425/EC, which includes restrictions on the use of dibutyl tin, dioctyl tin, and tri-substituted organotin compounds, was incorporated into ANNEX XVII of REACH through regulation (EU) 276/2010.⁸

EU regulations restrict tin to $\leq 0.1\%$ metal, with global restrictions likely becoming more restrictive. Many formulators of organosilane systems are proactively striving to develop tin-free systems. The dosage of tin metal that is often required to achieve sufficient cure of moisture cured organosilane polymer systems is typically very close to the REACH $\leq 0.1\%$ limit, with the possible exception of trimethoxy silane polymers that usually require less catalyst.

Catalyst Options

As described in the catalysis section, the organosilane reactions can be catalyzed with acids, bases, and metal compounds. Organotin compounds are very effective catalysts, and they are commonly used in these systems. The motivation for replacing tin catalysts can be for performance issues, but often the motivation is to address regulatory issues. Extensive studies were conducted to identify compounds that could be considered alternatives to tin catalysts in organosilane systems. Amine-type compounds like guanidines can act as catalysts for some organosilane polymers, but even if they sufficiently catalyze the reaction, they are skin sensitizers and can contribute other undesirable characteristics like odor or color.

The control systems in each of the following experiments were catalyzed with a tin catalyst. The non-tin catalyst highlighted in these experiments are CATALYST 670 (zinc/amine compound) and CATALYST 648, a modified version of CATALYST 670. Compared to tin compounds, zinc compounds do not carry the environmental and biological restrictions of tin compounds. Extensive utilization of zinc compounds in the field of medicine exemplifies their non-toxic nature.

Experiment I is based on a dimethoxymethylsilyl (DMS) polyether polymer and Experiment II is based on a trimethoxymethylsilyl (TMS) polymer. Experiment III addresses the issue of methanol generation by using an organosilane based on a diethoxysilyl (DES) polyether polymer.

Experimental

Fully formulated single-component moisture-cure alkoxy silane systems were used in the following experiments. The uncatalyzed one-component formulations were stored in dispense cartridges. Approximately 30 grams of uncatalyzed material was dispensed into a container with a caulk gun before addition of the catalyst. The material was mixed on a speed mixer. An adjustable doctor blade was used to apply approximately 3 mm of the blends onto a paper substrate. The degree of dryness was determined by using a Model 415 Drying Time Tester.¹⁰ The dryness test involved applying a force onto a paper disk that covered a test site on the casting for 60 seconds. The results are based on the amount of tack and on visual impressions that developed from the applied force. The dryness testing was done at 25 °C and 50% relative humidity. Degree 1 of the DIN 53 150 method was substituted with a glove test to determine touch dry. Table 1 defines the rating system.

VIEW TABLE 1

Where tested, hardness of the castings was determined with a Shore A¹¹ hardness tester after the castings were allowed to cure under ambient conditions for 2 weeks. Other mechanical properties were measured on an Instron¹² tester using dogbone shaped samples cut from 3 mm thick castings.

Experiment I. Moisture-cure system based on dimethoxymethylsilane (DMS) polymer¹³

The control system in this experiment was catalyzed with dioctyl tin diacetylacetate (DOTDAA) in a system based on a polyether backbone DMS polymer. The dosage of tin catalyst in the control system was 0.6% DOTDAA. The tin content of DOTDAA is approximately 21%. At 0.6%, the tin content in the formulated control system is approximately 0.12%, which would not comply with EU regulations. The formulation is in Table 2.

Dryness ratings are in Table 3. The DOTDAA and CATALYST 670 catalyzed systems achieved the highest degree of dryness in 6 hours. Mechanical properties were not significantly different (Table 4). The DOTDAA and CATALYST 670 castings developed similar tensile stress (toughness), modulus (stiffness) and strain (elongation).

Tested guanidine compounds were not effective catalysts except for high dosages of 1-phenylguanidine. The high dosage, however, caused severe surface and appearance defects in the cured product.

VIEW TABLE 2

VIEW TABLE 3

VIEW TABLE 4

Experiment II. Moisture-cure system based on diethoxysilane (DES) polymer¹⁶

The basic uncatalyzed DES formulation is in Table 5. DOTDAA was essentially not active in this system. At 0.5% and 1.0% on total formulation weight, DOTDAA catalyzed castings required more than 120 hours to achieve a dryness rating of 7 (Table 6). Dry times with CATALYST 670 were significantly faster than the tin catalyzed systems. The CATALYST 670 system developed dry times that were comparable to the DMS system.

The DOTDAA system required a month of ambient cure before it was suitable for mechanical testing. Even so, the casting had very weak properties (Table 7).

Diethoxysilanes are of particular interest from a toxicity perspective, as the volatile byproduct of the crosslinking reaction is ethanol instead of toxic methanol.

VIEW TABLE 5

VIEW TABLE 6

VIEW TABLE 7

Experiment III. Moisture-cure system based on trimethoxysilane (TMS) polymer

Typical tin catalyst dosages in trimethoxysilane polymer systems are less than in dimethoxysilane polymer systems. The control system in this experiment was catalyzed with 0.06% DBTDL. Although the CATALYST 670 and 648 dosages in this experiment were still higher than the tin catalyst dosage, sufficient cure was achieved when their dosages were reduced by half. CATALYST 670 can work synergistically with additional silane adhesion promoters like 3-aminopropyltriethoxysilane.

The mechanical properties in Table 10 show similar hardness development for each system. However, the DBTDL system had lower % strain (elongation) than each of the non-tin systems.

VIEW TABLE 8

VIEW TABLE 9

VIEW TABLE 10

VIEW TABLE 11

Conclusion

Increasing regulatory restrictions have formulators of organosilane adhesives and sealants searching for alternatives to tin catalysts. CATALYST 670 and CATALYST 648 were effective non-tin catalysts in three one-component organosilane experiments that required acceleration of both the hydrolysis and condensation reactions.

Also addressed in this work was the catalysis of a non-methanol-emitting moisture-cure organosilane system based on a diethoxysilane polymer. In this case, CATALYST 670 catalyzed the crosslinking reaction while tin catalysts were essentially not active even at levels that doubled the maximum tin concentrations allowable in current EU regulations.

Acknowledgements

The authors would like to thank Dr. Ravi Ravichandran, David Switala, and Matthew Gadman for contributions made to this article.

Learn more about King Industries at www.kingindustries.com.

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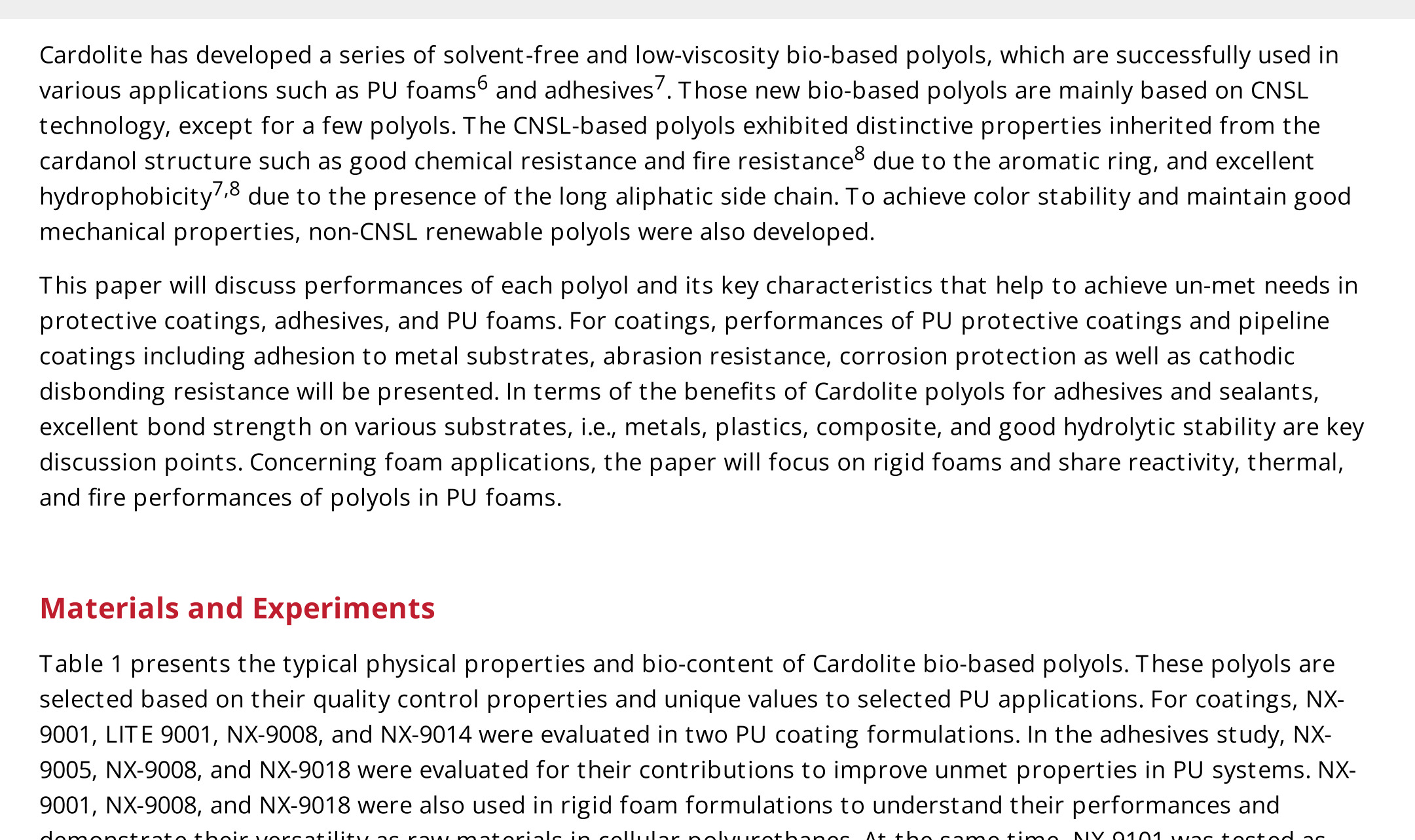
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RENEWABLE, CASHEW NUTSHELL LIQUID-BASED POLYOLS: THEIR BENEFITS IN CASE AND POLYURETHANE FOAMS

Scientists evaluate the performance and key characteristics of CNSL-based polyols in the formulation of various PU systems, including protective coatings, adhesives, and foams.

By Yun Mi Kim, Technical Marketing Director, Cardolite Corp., Bristol, Pennsylvania; Anbu Natesh, Vice President R&D at Cardolite Corp., Bristol, Pennsylvania; Pietro Campaner, R&D Chemist, AEP Polymers Srl, Trieste, Italy; Hong Xu, Technical Service Manager, Cardolite Corp., Bristol, Pennsylvania.

Polyurethanes (PU) have been extensively used in a broad range of industrial applications, such as foams, textiles, composites, adhesives, sealants, and coatings. For example, the polyurethane demand worldwide in 2017 was about 16.9 million tons, and it is forecasted to grow close to 22.5 million tons by 2024¹. In the CASE applications, polyurethane chemistry has become more and more attractive because of its valued properties of superior durability and toughness, excellent abrasion resistance, adhesion strength on various substrates, as well as desired aesthetic finishes². Most of the polyols currently used in polyurethane applications such as foams, coatings, adhesives, and elastomers, derive from petroleum-based feedstock, but the growing interest in materials from renewable sources has led to the development of several bio-based polyols³. There are no significant examples at industrial scale of bio-based polyols with an aromatic backbone, as the majority of bio-based materials currently available are soybean-, palm-, or castor oil-based, which can offer hydrophobicity and good flexibility but lack chemical and thermal performances. The structures and properties of polyols or isocyanates as raw materials can significantly affect the physical, mechanical, and chemical properties of the final polyurethane systems. Therefore, the proper design and modification to the raw materials, especially polyols, is an effective way to enhance the performances of polyurethane systems to meet requirements for various applications. Cashew Nutshell Liquid (CNSL) is a sustainable and non-food-chain biomaterial that is contained in the honeycomb structure of the cashew nutshell⁴⁻⁵. Shown in Figure 1, cardanol is the main component derived from CNSL via decarboxylation and distillation. Cardanol (average structure illustrated in Figure 2) is a penta-decadienyl phenol with a long aliphatic side chain that consists of a mixture of one, two, and three double bonds in a linear chain. Such unique and versatile structure enables this natural oil to become a very important chemical building block for numerous bio-based products including polyols.

[VIEW FIGURE 1](#)[VIEW FIGURE 2](#)

Cardolite has developed a series of solvent-free and low-viscosity bio-based polyols, which are successfully used in various applications such as PU foams⁶ and adhesives⁷. Those new bio-based polyols are mainly based on CNSL technology, except for a few polyols. The CNSL-based polyols exhibited distinctive properties inherited from the cardanol structure such as good chemical resistance and fire resistance⁸ due to the aromatic ring, and excellent hydrophobicity^{7,8} due to the presence of the long aliphatic side chain. To achieve color stability and maintain good mechanical properties, non-CNSL renewable polyols were also developed.

This paper will discuss performances of each polyol and its key characteristics that help to achieve un-met needs in protective coatings, adhesives, and PU foams. For coatings, performances of PU protective coatings and pipeline coatings including adhesion to metal substrates, abrasion resistance, corrosion protection as well as cathodic disbonding resistance will be presented. In terms of the benefits of Cardolite polyols for adhesives and sealants, excellent bond strength on various substrates, i.e., metals, plastics, composite, and good hydrolytic stability are key discussion points. Concerning foam applications, the paper will focus on rigid foams and share reactivity, thermal, and fire performances of polyols in PU foams.

Materials and Experiments

Table 1 presents the typical physical properties and bio-content of Cardolite bio-based polyols. These polyols are selected based on their quality control properties and unique values to selected PU applications. For coatings, NX-9001, LITE 9001, NX-9008, and NX-9014 were evaluated in two PU coating formulations. In the adhesives study, NX-9005, NX-9008, and NX-9018 were evaluated for their contributions to improve unmet properties in PU systems. NX-9001, NX-9008, and NX-9018 were also used in rigid foam formulations to understand their performances and demonstrate their versatility as raw materials in cellular polyurethanes. At the same time, NX-9101 was tested as self-catalytic polyol, benchmarking versus petro-based Mannich.

Viscosity, hydroxyl value, acid value, and water content measurements on the different polyols were performed according to ASTM D4878, ASTM D4274, ASTM D4662, and ASTM D4672, respectively.

GPC analysis for cardanol-based polyols molecular weight characterization and functionality determination was performed on the same HITACHI HPLC-GPC systems, isocratic mode (0.5 ml/min flow rate; wavelength set to 280 nm). Three columns connected in series (Tosoh TSKgel Super H1000, Tosoh TSKgel Super H2000, Tosoh TSKgel Super H3000, 3 µm, 6x150 mm; mobile phase: tetrahydrofuran) or a single column (Tosoh TSKgel G3000, 5 µm, 7.8x330 mm; tetrahydrofuran, buffered with 0.25% TFA and 0.25% TEA) thermostated at 40 °C were used. 11 narrow MW polystyrene standards have been used for the calibration curve.

Commercially available polyols, surfactants, catalysts, and blowing agents were used in the foaming trials. Two types of isocyanates (Table 2) were used as crosslinkers: MDI-based polyisocyanate, referred to as polymeric MDI (pMDI), and hexamethylene-1,6-diisocyanate, referred to as HDI.

[VIEW TABLE 1](#)[VIEW TABLE 2](#)

Test specimens and results are obtained based on the following ASTM methods: Lap shear strength (ASTM D1002) on sandblasted steel, tensile strength/elongation (ASTM D638), Mandrel bend (ASTM D522), direct and reverse impact (ASTM D2794), crosshatch adhesion (ASTM D3359), chemical resistance (ASTM D6943), and shore D/A hardness (ASTM D2240). Cathodic disbonding (CD) was carried out according to ISO 15711-method A. Gel time was measured in 50 grams amounts at 25 °C by using a gel time meter. Linear dry time tests were conducted using an 8-ml wet film thickness drawn down bar according to ASTM D5895-03. Moisture sensitivity of polyols was determined by measuring the amount of CO₂ generated after pouring pre-mixed polyurethane into water and keeping resulting blends at room temperature for 24 h (it is in fact known that isocyanates react with water to generate CO₂ gas, and therefore, the greater the hydrophobicity the lower the CO₂ evolution).

Foam specimens were stored at room temperature for 48 h prior to testing. Density and reactivity, compression strength, water absorption, were measured according to ASTM D7487, ASTM D1621, and ASTM D2842, respectively. Fire tests were determined according to vertical UL-94.

Results and Discussion

Polyurethanes use isocyanates that are moisture sensitive, and the absorbed moisture can induce an unwanted side reaction like generation of CO₂ bubbles. Polyols with excellent hydrophobicity will reduce moisture sensitivity during the blending process of isocyanates and polyols, which leads to lower CO₂ evolution and ultimately extends the service life of the adhesive by preventing failure from hydrolysis. The polyols that are intended to be used in coatings and adhesives have been tested in 2K polyurethane systems using a standard-grade polymeric MDI and compared to a reference castor oil-based polyol characterized by comparable OH value, measuring carbon dioxide evolved after blending 100g of polyurethane premix and 100g of water. As evident from the data reported in Table 3, the amount of CO₂ formed in the NX-9001 and LITE 9001 systems is much lower than in the standard hydrophobic castor oil reference system, indicating superior hydrophobicity of NX-9001 and LITE 9001 followed by NX-9005 and NX-9008.

[VIEW TABLE 3](#)

Basic mechanical properties and Tg data are displayed in Table 4. Cure conditions are varied to achieve full cure of the systems. NX-9001, LITE 9001, NX-9005, and NX-9008 are cured for 7 days at room temperature while NX-9014, NX-9018, and the reference were prepared at 70 °C/4 hrs + 120 °C/2.5 hrs. NX-9005 has the most well-balanced strength and flexibility, while NX-9008 and NX-9018 can offer very high tensile strength and bond strength along with high Tg, with similar properties of an epoxy system. NX-9014 will be featured for its good color stability in PU systems as well as its balanced tensile strength and elongation.

Gel time and linear dry times at various temperatures (25 °C, 5 °C, 0 °C) are shown in Table 5. LITE 9001 is the fastest polyol to cure while the rest of the polyols can also achieve reasonable cure speeds at 25 °C and 5 °C with good pot life.

[VIEW TABLE 4](#)[VIEW TABLE 5](#)

1. Study of PU Coatings

1a. Performance study in low-V.O.C. 2K PU protective coatings

Three 2K low-VOC PU protective coating systems (Table 6) were formulated for air spray application based on LITE 9001, NX-9008, and NX-9014. When combined with polymeric MDI at a 110 NCO index, these three formulations can reach 90% solids by weight with final calculated VOC values of 125.4 g/L, 155.3 g/L, and 159.6 g/L for LITE 9001, NX-9008, and NX-9014 systems, respectively. The three formulations contained about 37-43% inhibitor pigments in total pigment weight and used no catalysts to speed up cure properties.

All test panels were prepared over cold-roll steel substrate by drawing down films of 6 mils wet film thickness; after panels cured at RT for 7 days, Mandrel Bend, impact resistance, and cross-hatch adhesion tests were conducted.

[VIEW TABLE 6](#)[VIEW TABLE 7](#)

Results demonstrate that all three PU coatings exhibited very good Mandrel Bend performance, with the NX-9014 system showing much higher impact resistance than the other systems due to its branched aliphatic structure. Moreover, it can be observed that the NX-9008 system was less flexible in comparison to LITE 9001 and NX-9014, which might relate to its high crosslinking density. Furthermore, one of the big challenges for PU systems is good adhesion to metal substrates. Crosshatch adhesion test results shown in Table 7 suggest that these three new bio-based polyols could provide excellent adhesion to cold-roll steel that could help achieve excellent anti-corrosion performance.

Traditional protective coatings usually consist of an epoxy primer and a PU topcoat to provide the best synergistic performances. For example, the epoxy primer offers excellent adhesion to the metal substrate as well as good barrier property while the PU topcoat delivers toughness and excellent weatherability. However, when a PU coating system is used as a direct-to-metal (DTM) system without an epoxy primer, the long-term corrosion protection becomes a challenge.

In this study, the anti-corrosion performance of LITE 9001, NX-9008, and NX-9014 systems was evaluated by salt spray exposure in a Q-FOG tester chamber (Model SSP600) according to ASTM B117. These three PU coating systems were air sprayed over cold-roll steel substrates (QD-36) to a final dry film thickness of around 90 microns after a 7-day RT cure. After 800 hours of exposure, different performance aspects, such as rust, blisters on field, and creep along the scribe line were checked. The photo images of testing panels after salt spray exposure are shown in Figure 3. It can be seen that there were no blisters or rust on panel films; the maximum creep width was 1.5 mm, 2 mm, and 2 mm for the LITE 9001, NX-9008, and NX-9014 systems, respectively. The salt spray test results suggest that the LITE 9001, NX-9008, and NX-9014 systems could provide very good barrier protection without any blister and delamination issues; the low creep width indicated that the three PU systems could offer good adhesion to metal substrates. Moreover, these results confirm the hydrophobicity derived from CNSL could benefit LITE 9001 and NX-9008 coating systems. The NX-9014 system could also achieve good anti-corrosion performance through its branched structure and higher inhibitor pigment loading.

[VIEW FIGURE 3](#)

It is naturally expected and desirable that PU systems provide excellent UV and weathering stability. As with many natural products, CNSL-based polyols LITE 9001 and NX-9008 tend to not show very good UV resistance. However, NX-9014 was designed for good UV stability while balancing bio-content. In this study, the color stability of NX-9014 was compared to a reference polyol in the formulation in Table 8 using aliphatic HDI. The reference selected as benchmark is considered one of the best polyether/polyester polyols in terms of UV stability.

[VIEW TABLE 8](#)[VIEW FIGURE 4](#)

Based on the test results shown in Figure 4, it is evident that the NX-9014 system exhibited very good UV resistance, similar to the reference system. The excellent color stability of the NX-9014 system suggests it could be a very good PU topcoat system in addition to a good DTM protective coating.

1b. Performance study in SF 2K PU pipeline coating systems

In recent years, the use of polyurethane systems as protective coatings for pipeline applications has increased due to their excellent mechanical performance and good weathering resistance. In this study, LITE 9001, NX-9014, and NX-9001 were formulated as solvent-free 2K PU pipeline coating systems (Table 9), using polymeric MDI (index 110) and without the addition of any catalyst.

These formulations did not use solvents; the final VOC for the LITE 9001, NX-9014, and NX-9001 systems were 8.69 g/L, 7.42 g/L, and 8.46 g/L, respectively, which mainly came from the volatile components in additives. In addition, the three PU pipeline coatings did not contain any corrosion inhibitor pigments; and they had final PVC around 10-11%, which suggested that the barrier protections mainly relied on the PU binder systems.

[VIEW TABLE 9](#)[VIEW FIGURE 5](#)

Pipeline coatings are usually applied and cured on-site, therefore, fast cure speed especially at low temperatures is requested to widen the application window to year-round. The linear dry time data shown in Figure 5 suggests that the LITE 9001, NX-9014, and NX-9001 systems could provide good cure properties. For example, the LITE 9001 system showed dry hard times of 1 hour and 2.25 hours at 25 °C and 10 °C cure conditions, respectively, while the NX-9014 and NX-9001 systems were not as fast as the LITE 9001 system, but still achieved dry hard times of less than 3.5 hours and 10 hours at 25 °C and 10 °C cure conditions, respectively. Since no solvents and catalysts were used in these three pipeline coatings, it can be concluded that the high reactivity of CNSL-based polyols was a major contributor to the fast cure properties of the final PU systems.

[VIEW TABLE 10](#)

In this study, the mechanical properties of three SF PU pipeline coatings were tested over cold roll steel substrate with a wet film thickness of 6 mils. After a 7-day cure at RT, Mandrel Bend, impact resistance, abrasion resistance, and crosshatch adhesion tests were conducted. According to the results reported in Table 10, it can be observed that the LITE 9001 and NX-9014 systems exhibited good Mandrel bend performance, while the NX-9001 system was less flexible. The three SF PU pipeline coating systems showed excellent adhesion to cold-roll steel substrate and achieved very good abrasion resistance properties as indicated by their weight losses after 1000 cycles of 50.8 mg, 26.2 mg, and 33.2 mg for LITE 9001, NX-9014, and NX-9001, respectively. It is also noticeable that the coating films based on LITE 9001 and NX-9001 displayed higher gloss than that of the NX-9014-based coating, which might be due to the lower moisture sensitivity of CNSL-based polyols during the cure process.

[VIEW FIGURE 6](#)

As an additional part of the same study, the anti-corrosion performance of each of the SF PU pipeline coatings was evaluated. The three PU pipeline paint systems were applied over sandblasted steel (SA2.5) via No. 60 RDS lab rod, with a final dry film thickness of about 5 mils after 7 days RT cure. Figure 6 displays the test panel images after 1039 hours of salt spray exposure. Most surface rust stains were removed via slight sanding with 800 grit sandpaper to reveal the details of blisters and creeps. It can be seen that there was almost no creep developed along the scribe line on any of the three PU systems. The LITE 9001 system had more blisters than the NX-9014 and NX-9001 ones, but mainly congregated along the scribe line. The NX-9014 system had almost no blisters, while the NX-9001 system showed some blisters. As noted earlier, no corrosion inhibitor pigments were added in the formulations, thus the corrosion protection of these coatings came from the barrier properties provided by the polyol-isocyanate network. Moreover, test results suggested that both NX-9014 and NX-9001 systems offered better barrier protection than the LITE 9001 system.

For underground pipelines and tanks or buried metal constructions, the major part of the corrosion protection comes from cathodic protection in which the metal substrate becomes the cathode via two methods: one is to connect external anodes to metal substrates; another is to impress necessary currents through external anodes by using a DC power source. In general, the method of applying impressed current systems is more commonly used to protect underground pipelines and tanks due to its better suitability for large size structures and its lower susceptibility to ground environments. Since it is difficult to prevent pipelines from experiencing excessive current at times, coating systems for buried metal constructions should withstand and cathodic disbondment besides providing excellent barrier protection to limit the penetrations of moisture, ions, and oxygen to the metal surface.

[VIEW FIGURE 7](#)

Cathodic disbonding (CD) testing for three SF PU pipeline coatings was carried out according to ISO 15711-method A. The test panels were prepared by brushing the PU paints over sand blasted steel panels to obtain thick coating films. The NX-9014 and NX-9001 systems had dry film thickness of 66 mils and 62 mils, respectively; the LITE 9001 system had a higher film thickness of 87 mils due to its fast viscosity build-up. After 10 days cured at RT, each test panel was drilled to get a Ø 5 mm artificial hole in the center of the panel and glued to an Ø 8 cm plastic tube as the test tank, which was filled with fresh artificial seawater solution. The CD test setup used in this study is shown in Figure 7: graphite electrode was used as anode, copper/copper sulfate electrode was used as reference electrode, and a galvanostat was used to control the test potential.

Figure 8 displayed the panel images of the three SF PU pipeline coating systems after 42 days cathodic disbonding test at RT. It can be seen that the maximum disbonding distances from the artificial holes were 3 mm, 3 mm, and 8 mm for the LITE 9001, NX-9014, and NX-9001 systems, respectively. Furthermore, there were no blisters or rust observed on these three tested PU coatings. The test results suggested that the SF PU coatings based on LITE 9001 and NX-9014 could provide excellent cathodic disbondment resistance. Though not as good as the other systems, the NX-9001 system still showed good cathodic disbondment resistance and could potentially show improved performance in more flexible formulations that compensate for the lower flexibility of NX-9001.

[VIEW FIGURE 8](#)

2. Study of PU Adhesives

Table 4 shared the cohesive strength, flexibility, and Tg of PU systems based on Cardolite's polyols. It is known that good bond strengths can be achieved when the resin systems offer high strength, reasonable flexibility, and good wetting of the substrates. Substrates that are inherently flexible like plastics tend to have good adhesion strength with systems that have good flexibility and good wetting on low-surface-energy substrates, typical characteristics of plastics. Therefore, using PU adhesives especially with NX-9005 is a suitable option for plastics bonding. For metals and composite substrates (carbon fiber reinforced epoxy prepreg), polyols with high tensile strength i.e., NX-9008 and NX-9018, have exhibited higher lap shear (Table 11). All the polyols were tested under the same conditions and cured for 40 °C/16 hr. Metal substrates were sand blasted and alcohol wiped, while plastics and composite substrates were sand paper abraded and alcohol wiped prior to the application of the adhesive.

Hydrolysis of polyurethane adhesives upon exposure to water can result in reduction on adhesive strength and loss of durability. In addition to previous comments (Table 3) on polyols' moisture sensitivity and effect of hydrophobicity, Table 12 shows a change of lap shear strength upon exposure to 80 °C water for 7 days. As expected, polyols with low moisture sensitivity like NX-9001 and NX-9005 have demonstrated the lowest loss of adhesion, due to their excellent hydrophobicity.

[VIEW TABLE 11](#)[VIEW TABLE 12](#)

3. Study of PU Foams

In order to evaluate their versatility and assess their potential applicability in different type of polyurethane systems, some of the grades previously described (NX-9001, NX-9008, and NX-9018) have been tested in rigid polyurethane foams characterized by different reactivity: continuous panels and spray systems. At the same time, NX-9101, a low-viscosity cardanol-based Mannich polyol has been selected as a suitable candidate to be included in the spray foam study, benchmarking versus a petro-based Mannich polyol characterized by comparable hydroxyl value.

As a very first step of the study, the compatibility of the selected candidates and standard petro-based polyols have been verified (Table 13). Several blends (based on combinations of CNSL polyols, petro-based Mannich polyols, aliphatic and aromatic polyester polyols, and polyether polyols) have been prepared and monitored for their stability.

CNSL-based polyols, even if characterized by a high hydrocarbon content, contain enough polar groups (amino, hydroxyl, esters) to guarantee an excellent compatibility with polyether polyols and petro-based Mannichs. On the contrary, as in the case of other NOPs based polyols⁹, CNSL-polyols show limited miscibility with polyester polyols (both aliphatic and aromatic). However, this limitation can be easily overcome by properly tuning the composition of the B-part of the PU formula, for example, using a polyether polyol as a co-polyol, balancing the CNSL-polyol and the polyester polyol ratio, or using non-polyurethane emulsifiers as blowing agents with a solvent effect (e.g. methyl formate).

[VIEW TABLE 13](#)[VIEW TABLE 14](#)

A different aspect that has also been verified is the compatibility of CNSL polyols with standard blowing agents for spray and rigid polyurethane foams (e.g. n-pentane, methyl-formate, HFC, and HFO), as it is a key aspect to guarantee final foam properties and B-part stability. This miscibility study has been accomplished by blending polyols (both CNSL- and petro-based) and blowing agents (60/40 w/w) in tightly closed graduated containers, and recording the appearance of the mixtures after 24 hours at room temperature. In case of phase separation, the miscibility has been determined by measuring the height of the blowing agent phase: since the density is known, the amount (in grams) of the blowing agent dissolved into the polyol-phase can be easily recovered (Table 14). NX-9101, NX-9001, NX-9008, and NX-9018 show excellent miscibility with methyl-formate, n-pentane (same results are obtained with cyclopentane), and HFO (Opteon 1100), better than petro-derived Mannich polyols and polyester polyols (both aromatic and aliphatic ones).

Considering cardanol-based polyols' chemical structure and the contribution of polyols' aromaticity to PU foams fire performances, the aromatic content of cardanol bio-based polyols has been calculated (Table 15), with results being close to aromatic polyester polyols (20-22%) and petro-based Mannich polyols (approx. 17.5-19%)¹⁰.

[VIEW TABLE 15](#)

In particular, among the polyols selected for rigid polyurethane foams, NX-9001 shows the highest aromatic content, due to its peculiar chemical backbone (alkoxylated cardanol novolac), followed by NX-9101 and NX-9008, with NX-9018 being characterized by a limited amount of aromatic rings in its chemical backbone.

In a first round of trials (Table 16), cardanol-polyols have been used in a reference spray-foam closed-cell formula, benchmarking versus petro-based polyols characterized by comparable hydroxyl values. In particular, NX-9101 has been evaluated as potential replacement for a petro-based Mannich polyol (formula PU 2), while NX-9008 as a possible alternative for a sucrose-based polyether polyol (formula PU 3 and PU 4), still having the reference petro-Mannich polyol (formula PU 3) or NX-9101 (formula PU 4) as catalytic polyol. Since the hydroxyl value of NX-9008 is a bit lower than that of the reference sucrose-polyether polyol, a small amount of glycerol has been used to balance overall equivalent ratios.

In general, cardanol-polyol-based systems show faster reactivity than full petro-based reference, with a small increase in final density. All the formulations show average good compression strength values (in both directions): the only exception is formula PU 4, whose compression set is lower than the rest of the systems, most probably due to the higher overall cardanol presence, since the C15 side chain might have a plasticizing effect.

At the same time, thermal stability (measured as weight loss on a thermogravimetric scale set at 150 °C for 30 minutes) of partially modified cardanol formulas show a small improvement over the reference PU 1. A similar trend has been observed for weight variation after immersion in distilled water at 50 °C for 7 days, with PU 2, PU 3, and PU 4 showing low values in average, that are quite close or slightly better than the petro-benchmark. When characterizing for their fire performances on a small scale (vertical UL-94), all the cardanol-based formulas (PU 2, PU 3, and PU 4) exhibit lower after-flame values than the fully petro-based reference.

As an additional part of the study, the same formulations as in Table 16 have been tested for their stability over time. In this case, all the Parts B (polyol blends, additives, catalysts, silicone, and HFO blowing agent) have been stored at 50 °C for 7 days in tightly closed containers. Their reactivity has then been measured again using the same conditions as the fresh formulations (same pMDI, same index).

[VIEW TABLE 16](#)

As evident from the comparative data reported in Figure 9, CNSL-based formulations exhibit better stability after storage at 50 °C than the fully petro-based reference, with percentage variation values for both string time and tack-free time either very low or close to zero.

In order to confirm the versatility of the polyols described earlier for their performances in 2K PU coatings and adhesives, NX-9001, NX-9008, and NX-9018 have been tested in a different system for rigid polyurethane foams, characterized by medium reactivity (Table 17).

[VIEW FIGURE 9](#)[VIEW TABLE 17](#)

In this case, formulations PU 5, PU 6, PU 7, PU 8, and PU 9 are all based on the same sucrose/glycerol polyether polyol (OH 490 mg KOH/g), with cardanol-polyols being used as full replacements for polyether polyols characterized by comparable hydroxyl value. In particular, NX-9001 (formula PU 6) replaces a sorbitol-based polyether polyol (PU 5), while NX-9008 and NX-9018 have been used as potential alternatives to a sucrose-based polyether polyol having a 360 mg KOH/g hydroxyl value. A bio-derived polyester polyol with a similar hydroxyl value (PU 7) has been selected as a commercial bio-based benchmark.

All formulations show quite comparable or slightly faster reactivity than reference PU 5, with no significant difference in density. The lower compression strength values are most probably due to the more flexible structures of the alternatively polyols used in formulations PU 6-PU 9. However, the introduction of PU 5, but also to NX-9008 and NX-9018 significantly reduced the water absorption, not only with respect to reference PU 5, but also to the hydrophobic bio-based polyester benchmark (PU 7). At the same time, the presence of some aromaticity in polyols derived from the cardanol (NX-9001 and NX-9008), as expected, helped to reduce the after-flame values during vertical UL94 test.

Conclusion

Cardanol represents an ideal raw material for applications requiring environmental sustainability and easy processability. Its unique chemical backbone allows the development of polyols that can be successfully used in different polyurethane applications, ranging from coatings to adhesives to rigid foams. These polyols provide very good reactivity, hydrophobicity, chemical and corrosion resistance, adhesion, and mechanical and fire resistance properties. Excellent performances, wide formulation latitude, and low environmental impact are all aspects that qualify CNSL-polyols as valid alternatives to petroleum-based polyols.

Future work will be dedicated to the development of novel structures with increased UV resistance, high bio-content and further improved performances in different types of polyurethane systems (CASE, rigid and flexible PU foams).

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DEVELOPING RESILIENCY AND TARGETING GROWTH OPPORTUNITIES IN THE NEW NORMAL

Today's business landscape offers opportunities for differentiation and growth for organizations that can build on the lessons they have learned over the past several years.

By Daniel Murad, CEO, The ChemQuest Group, Inc.

The CASE industry has done a phenomenal job adjusting to the complexities of the past several years, which included challenges related to numerous force majeure, alternative supply considerations, reformulations, working capital adjustments, and overall business continuity. It now seems that the worst may be over; in the first quarter of 2023, we have seen considerable easing in the supply chain across the industry.

In addition, commodity pricing has started to show declines, and capacity utilization has improved. A recovery is happening, though we are seeing results take shape more quickly in some areas vs. others.

While these trends certainly represent good news for the CASE sector, it is important to come to the realization that the economic and industry conditions that existed prior to the COVID-19 pandemic (i.e., the "old normal") are gone. In addition to numerous concerns, this "new normal" landscape offers opportunities for differentiation and growth for organizations that can build on the lessons they have learned.

Addressing Resilience Factors

Small- and medium-sized enterprises (SMEs) have been particularly hard hit during this tumultuous time. To build resiliency into their short- and long-term strategic planning, SMEs should consider four key factors: supply chain visibility, agility, supplier base diversification, and contingency planning.

Supply Chain Visibility

The crisis of 2020 made clear that the old ways of purchasing are no longer going to produce optimal results. Purchasing cannot remain simply a clerical function that serves only to provide needed materials at the best cost to the production process.

Recent supply chain confusion and disruptions have illuminated — often quite harshly — the importance of visibility. Increased data sharing across the entire supply network (i.e., raw material producers, distributors, coating and adhesive manufacturers, and end users) is absolutely essential.

Agility

Companies must enhance the speed at which their supply network can respond to shifts in the external environment, such as quickly scaling production to easily meet demand or reconfiguring plants and logistics networks, even across different geographies. Opening new demand channels is another strategy to consider, such as shifting from a brick-and-mortar model to e-commerce.

Having a properly functioning chemical logistics system must be a top priority all along a company's supply chain, including its customers. The entire system should be able to react to the smallest changes, delays, or fluctuations in the supply chain, as these issues often lead to production downtime and delivery problems to end-use customers.

Supplier Base Diversification

Just-in-time lean manufacturing processes only work when materials are readily available. However, the global shutdown associated with the pandemic strained supply lines, drove up shipping costs, and extended timelines. To help avoid these problems in the future, companies must evaluate their production footprint and transportation partners to diversify their supplier base.

Companies are increasingly turning away from global-spanning supply chains, which involve producing material in one region and then shipping all over the world from that location, as well as low-cost manufacturing hubs in Asia (e.g., China). The goal is to balance supply across geographies and ensure companies have sufficient capacity for the geographic regions where their plants are located.

This shift toward reshoring for economic stability represents a fundamental change from a business model that had proved popular for the past 30 years. In truth, it could prove a lasting legacy of the COVID-19 pandemic.

Contingency Planning

Tied to diversification is contingency planning, which stresses the ability (coupled with the appropriate tools) to anticipate and respond to disruptions. Along those lines, no organization can really react to changes in the supply chain without creating a risk assessment of its own logistics. This is of primary importance, as it allows for intelligent planning and maximum control.

Perhaps the single most important learning experience from our pandemic life involves the need to develop robust business continuity planning. A business continuity plan (BCP) is a system of prevention and recovery from potential threats. The plan ensures that personnel and assets are protected and able to function quickly in the event of a disaster.

The development of an effective BCP involves defining any and all risks that can affect the company's operations, making it an important part of the organization's risk management strategy. Risks may include natural disasters (e.g., fire, flood, or weather-related events) and cyberattacks.

Once the risks are identified, the BCP should also include steps to:

- Determine how those risks will affect operations
- Implement safeguards and procedures to mitigate the risks
- Test procedures to ensure they work
- Review the process to make sure it is up to date

BCPs are an important part of any business. Threats and disruptions mean a loss of revenue and higher costs, which lead to a drop in profitability.

Focusing on Opportunities for Growth

CASE companies that have successfully navigated the difficult conditions of the past several years should be poised to address numerous growth opportunities. For example, we are seeing signs of a renewed focus on new product development and innovation.

During the last three years, companies struggling with their material supply spent a considerable amount of time on reformulations just so they could survive. One might argue that successful reformulations are a form of innovation. Though not related to new product introduction, the lessons learned are extremely valuable with respect to building resiliency.

This recent focus on development varies a great deal and is somewhat dependent on the size of the organization. SMEs have continued to differentiate through segmentation and sub-segmentation, developing solutions within those segments where they play very well. Many larger, multinational organizations have sought to develop new technologies to tap into a broader range of markets and applications.

For example, in the coatings industry, we are seeing a movement to develop properties that address characteristics beyond traditional protection and decoration. A lot of work is being done to develop functional coating technologies, such as antiviral products to address society's newfound focus on surface cleanliness.

We are also seeing a lot of activity in low-energy systems, which is of particular importance given the Russia-Ukraine conflict and the impact it has had on Europe and beyond. Low-energy systems can help organizations address their high energy costs and improve operational efficiencies (whether in cost savings, labor reduction, reduced utilities and time, reduced waste, reduced layers, higher transfer efficiencies, or improved product performance).

In terms of end-use sectors with bright prospects for CASE growth, we see great potential in infrastructure projects and the increasing adoption of electric vehicles (EVs). Recent legislation in both the United States and Canada is providing needed funds for infrastructure projects. Across the board, our infrastructure is a couple of decades past its original design limits. According to the International Concrete Repair Institute (ICRI), well over 650 bridges in the United States and Canada alone are past their life expectancy and in need of repair or remediation.

EVs are projected to see strong widespread growth, driven by increasing consumer demand and numerous government incentives and initiatives (e.g., California's goal to replace vehicles with internal combustion engines with EVs by 2035). EVs can provide growth prospects in a number of areas, from battery and vehicle assembly to much-needed battery charging infrastructure.

Widespread EV adoption certainly continues to face various challenges. However, the development of the solutions to those problems may very well lie with the CASE industry, providing opportunities for adhesives and coatings producers, as well as the suppliers of materials to those companies.

To learn more, contact the author at dmurad@chemquest.com or visit <https://chemquest.com>.

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COULD MISTLETOE BERRIES HELP PRODUCE A BIOLOGICAL SUPER GLUE?

The abundant, biodegradable, and bio-renewable mistletoe plant holds promising possibilities for adhesives.

A team of researchers from the Max Planck Institute of Colloids and Interfaces in Germany and McGill University in Canada have discovered strong adhesive properties of white-berry mistletoe. The mistletoe berry's flexible fibers adhere to both skin and cartilage as well as to various synthetic materials and could find application in many fields, such as wound sealant in medicine.

Each mistletoe berry can produce up to two meters of a gluey thread called viscin — a natural cellulose adhesive. Viscin allows the seeds of this parasitic plant to stick to and infect host plants. Since ancient times, mistletoe berries have been explored as treatments for everything from infertility and epilepsy to cancer. But, until now, no one has fully investigated the potential medical or technical uses of the glue itself. A recent paper published in *PNAS Nexus* suggests that through simple processing, viscin's ultra-stiff flexible fibers could have a range of applications — both biomedical and beyond.

It is a discovery that came about almost by chance – sparked by the actions of a young girl. “I had never seen mistletoe before living in Germany,” said Matthew Harrington, a senior author on the paper, and an associate professor in the Department of Chemistry at McGill University, and the Tier 2 Canada Research Chair in Green Chemistry. “So, when my daughter was playing with a berry from a mistletoe we bought from a local Christmas market, and it started sticking to everything, I was intrigued.” Harrington’s research focuses on exploring materials and adhesives found in nature and adapting the underlying principles for the development of advanced bio-inspired materials.

For their research, the materials scientists led by Peter Fratzl, the director of the Department of Biomaterials at the Max Planck Institute of Colloids and Interfaces, picked the mistletoe berries from the trees themselves. From his office window, Fratzl can see the many green parasitic plants. "Mistletoe grows in large numbers everywhere, including the Max Planck Campus, and is biodegradable and renewable. For the first time, we are now investigating how to harness its excellent adhesive properties for potentially medical or technical uses," said Fratzl.

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Unusual Qualities of Mistletoe

The researchers discovered that through simple processing when wet, viscin fibers, which stick to themselves as well as to other materials, could be stretched into thin films or assembled into 3D structures. They believe that this means viscin could potentially be used as a wound sealant or skin covering. What makes the flexible viscin fibers so interesting as a material is that their ability to stick to things is fully reversible under humid conditions.

“I wore a thin film of viscin on my skin for three days to observe its adhesive qualities and was able to remove it from my fingers afterwards by simply rubbing them together,” said Nils Horbelt, a recently graduated Ph.D. student at the Max Planck Institute, and the first author on the paper. “But there still remain many questions about this very unusual material.”

“The fact that viscin can adhere to both wood and skin or feathers, may be relevant evolutionarily speaking,” added Harrington. “But it’s harder to explain adherence to various synthetic surfaces, such as plastics, glass and metal alloys, from an adaptive point of view. So viscin may simply represent a highly versatile adhesion chemistry, which is what makes it so interesting to explore what is going on chemically.”

The researchers’ next goals are to gain a better understanding of the chemistry behind this swellable, extremely sticky material so that they can then replicate the process. Given the excellent properties of mistletoe viscin and the fact that mistletoe plants are abundant, and both biodegradable and biorenewable, these findings suggest that this remarkable plant might provide more than holiday ornamentation in the future.

Open image courtesy of Max Planck Institute of Colloids and Interfaces / Nils Horbelt.

MUSSELS-INSPIRED INJECTABLE BIOGLUE SEALS SURGICAL WOUNDS

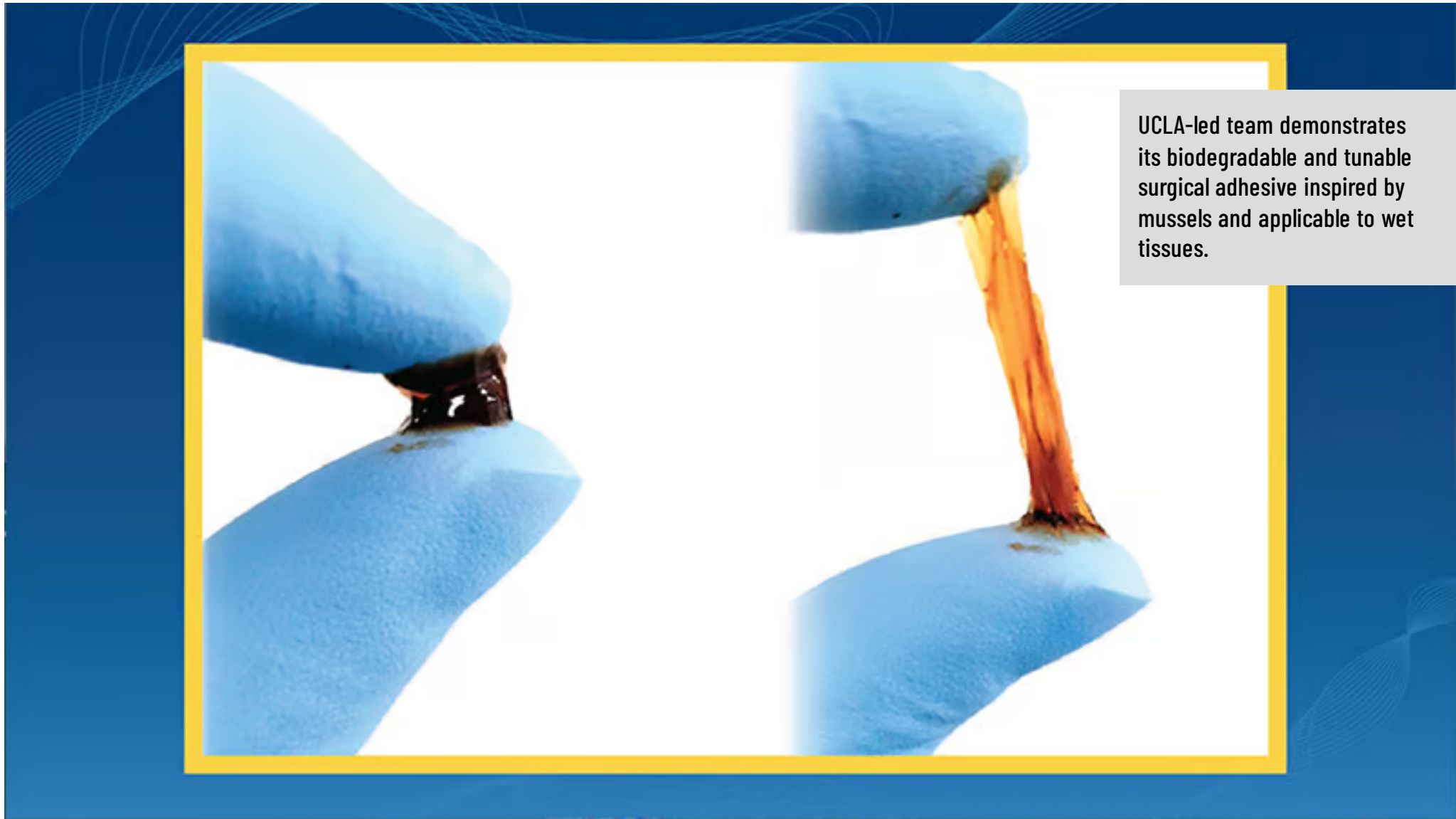
Researchers develop biodegradable hydrogel adhesive for sealing internal soft-tissue incisions using inspiration from nature.

A UCLA-led research team has created a new, biodegradable hydrogel adhesive that is reported to be three-times stronger than comparable bioglues. Injected from a syringe, the bioglue’s honey-like viscosity coupled with its fast-sealing action could improve precision in closing surgical wounds on curved and wet tissues. The new glue, which contains more than 80% water, was inspired by the way that mussels adhere themselves to rocks, pier pilings, and other wet surfaces.

Suturing and stapling are standard techniques to close surgical wounds, but these methods are inefficient for sealing internal soft-tissue incisions such as those on the heart, lung, and bladder tissues. Even the current best surgical adhesives are runny when first applied, making them difficult to control. They also take a long time to set and can cause unwanted adhesion due to dripping.

To address these challenges, a team of researchers looked to mussels commonly found along the coastlines for inspiration. These shellbound molluscs anchor themselves to wet surfaces by secreting highly sticky foot proteins composed of chemical compounds called catechols. These organic compounds enable mussels to stay attached to surfaces in wet conditions, making it a promising material for improved surgical glues.

“Modification of natural biomaterials with catechols has been pursued previously. However, the majority of these modifications were limited by low yield for adequate tissue adhesion,” said study leader Paul Weiss, a distinguished professor of chemistry and biochemistry at UCLA. “In our study, we demonstrated a chemical\modification route that boosts the amount of catechol functionality that one can introduce to natural biomaterials such as gelatin, which is a favorable candidate for us due to its superior biocompatibility and biodegradability.” Weiss also holds faculty appointments in bioengineering, and materials science and engineering at the UCLA Samueli School of Engineering.



The bioglue’s tunable thickness, or viscosity, and rapid solidification make it easier to control for precise injection, allowing it to seal wet wounds and to stop bleeding rapidly. The thermo-sensitive glue can be removed safely, if needed, by reducing its stickiness through controlling the temperature applied to the glue. Made of gelatin and caffeic acid (found in coffee, tea, or wine), the natural glue also demonstrates antioxidant properties, aiding the healing process after surgical procedure.

“While gelatin by itself can work as a biodegradable, nontoxic surgical adhesive, its low viscosity at body temperature means that it can be quite runny, spilling over into places that do not need an adhesive,” said study first author and bioengineering doctoral student Hossein Montazerian. “In this study, we fine-tuned the processing parameters to optimize the amount of catechol — striking the right balance between viscosity and increased adhesion while also satisfying clinical requirements.”

Montazerian is advised by Weiss, who is also a member of the California NanoSystems Institute at UCLA and holds the UC Presidential Chair in Chemistry, and co-corresponding author Nasim Annabi, a chemical and biomolecular engineering assistant professor who also has a faculty appointment in bioengineering. The research was part of a continuing collaboration with co-corresponding author Ali Khademhosseini, CEO of the Terasaki Institute for Biomedical Innovation and a former UCLA professor of bioengineering, chemical and biomolecular engineering, and radiology. Other authors on the paper are Elham Davoodi, Alireza Hassani Najafabadi and Reihaneh Haghniaz of the Terasaki Institute.

A study describing the bioglue was published in the open-access journal *Cell Reports Physical Sciences*.

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GET STARTED

CUSTOM FORMULATING TO MITIGATE PROBLEMS IN RAW MATERIALS SOURCING

Applying the tool of custom formulation of adhesives can help companies address supply chain and raw materials sourcing challenges.

By Brian Flaherty, Managing Director of NextGen Adhesives.

NextGen Adhesives is a manufacturer of a wide range of adhesives serving the electronic, fiber optic, and medical industries. The company has been in business for over 15 years, and in that time, it has seen many changes in both the supplier and competitor landscape. However, the changes that NextGen Adhesives has witnessed since the COVID-19 pandemic have been monumental.

On the supply chain side, raw materials are not only significantly more expensive, but suppliers also require much larger minimum purchases with respect to both a dollar minimum as well as a package size minimum. For example, a raw material previously available in a 5 lb pail must now be purchased in a 500 lb drum.

Lead times for raw materials have also increased. In fact, for a period, in late 2019 and 2020, many raw materials were not available at all due to force majeure. Because of these supply chain issues, some adhesive companies are no longer able to continue to provide the same product offering. Companies are rationing product lines and discontinuing products because they are not able to source the raw materials they need to produce their product.

One way to address the inability to source the correct raw materials is to formulate offsets to discontinued products. One of NextGen’s core capabilities is to custom formulate an adhesive solution for a specific customer application. Creating custom formulations involves gaining an understanding of key properties required in the handling and in the final cured performance of the adhesive. Next, formulating expertise is employed to provide a customized solution for that specific application. This same approach can be used to formulate an offset to a product that has been discontinued.

Recently, NextGen formulated an offset for a room-temperature-curable, optically-clear adhesive for a lens bonding application. The critical properties for the adhesive were the ability to cure without heat, to flow sufficiently to wet a glass substrate (viscosity 500-1000 cps), and to provide a cured hardness of 80D minimum. The hardener of the qualified adhesive had been discontinued and was no longer available. The hardener was critical to achieving all four properties: color, hardness, viscosity, and cure temperature. NextGen formulated three alternatives utilizing different hardener formulation packages utilizing its existing inventory of raw materials. Free evaluation samples were provided to the customer for qualification based on their specific requirements. The customer selected the formulation with the best performance, and NextGen now supplies it, resolving a potentially significant supply chain issue.

NextGen’s ability to provide this service is driven by its combined 75 years of experience in the adhesive industry. The NextGen technical team understands the important adhesive properties for many applications and has developed longstanding relationships with raw material suppliers around the world. The company may have a readily available offset from its existing product line or formulation library. This fast turnaround could be key to ensuring continuous supply and preventing a client from facing a line-down situation.

Supply chain issues have improved in the past 6-8 months; however, issues with respect to lead times, minimums, and availability from suppliers are still prevalent, which ultimately impact product offerings in the marketplace. The past three years have reinforced the need for companies to be flexible and nimble. Many executives have learned that without that flexibility, they will not be successful as new challenges arise. Custom formulating is a critical tool companies can use to create flexibility within their organization, keep production going even when key materials are unavailable, and respond quickly when customer needs change.

To learn more about NextGen Adhesives, visit www.nextgenadhesives.com.

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NextGen Adhesives discusses customized solutions for adhesive formulations in the Bonding with ASI podcast.



PRODUCT FOCUS

ACHIEVING FLEXIBILITY IN ADHESIVE CURING



A line lamp for curing adhesives and other multi-functional polymers offers flexibility while providing high-intensity curing.

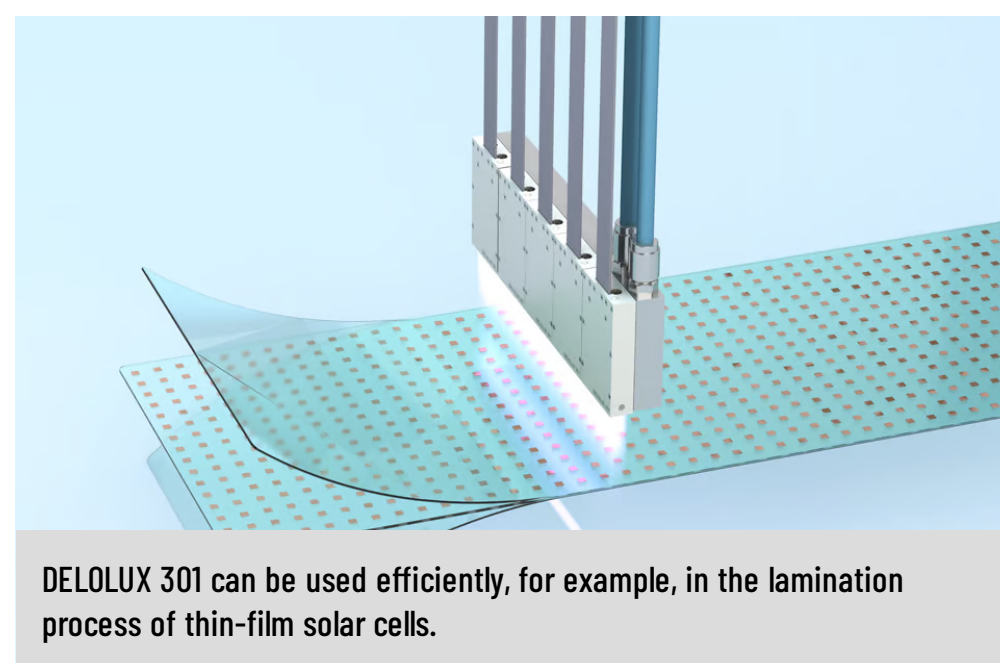
A line lamp for curing adhesives and other multi-functional polymers, DELOLUX 301 from DELO offers high curing speeds and great flexibility for integration into production systems. The lamp is suitable for highly automated processes in small and narrow production lines, like those found in consumer electronics assembly. It can also be used, for example, in the lamination of thin-film solar cells.

Offering very high intensity of up to 30 W/cm^2 , the lamp not only enables maximum speeds for curing adhesives and other multi-functional polymers, but also allows large working distances of up to 100 mm from lamp to components. The lamp's compact lamp head dimensions, measuring 42.7 mm x 13 mm x 67.2 mm, contribute to a high degree of flexibility for integration into production systems as well. It produces a linear irradiated area of 38.7 mm x 8.45 mm.

The lamp can be operated as a single head or be seamlessly combined to form a larger array. When the lamp heads are combined in up to three directions, users can easily implement individual as well as homogeneous exposure geometries. For example, a wide row of lamps can be used for the roll-to-roll lamination process of flexible thin-film solar cells, where adhesives are often needed to protect cells made of organic materials and perovskite from humidity ingress.

In its standard variant, the lamp is available with water cooling with optimized water flow, suitable for clean rooms. It is available with wavelengths of 365 nm, 400 nm and 460 nm, making it suitable for UV-curing as well as light-curing products. It has a service life of more than 20,000 hours.

This plug-and-play lamp head is controlled and supplied with power via the base units of the DELOLUX pilot A series, which are available as touch and PLC-capable models. Both models feature automatic temperature compensation so that the LEDs deliver constant intensity even as they heat up.



Learn more at www.delo-adhesives.com.

Opening images courtesy of DELO.

HEALTHCARE SUPPLY CHAINS

As the healthcare industry supply chain faces more challenges, supporting industries must be proactive, resilient, and innovative.

By Lisa Anderson, Founder and President, LMA Consulting Group

Although the visible bottlenecks in the healthcare supply chains are getting calmer, volatility will remain high. From the impacts of increasing interest rates and bank failures to labor and material shortages to global conflicts, risks in the healthcare supply chain will skyrocket. This situation will create as many opportunities as challenges, and so the proactive, resilient, and innovative companies will thrive while the rest diminish. The key will be deliberately making that choice and ensuring your end-to-end supply chain partners are on that same trajectory.

Current Status

Since the supply chain is an interlinked series of suppliers, manufacturers, and logistics partners, the bottleneck moves from one link to the next as demand and supply are out of alignment. During the pandemic, the visible signs were abundant with ships stacked up at the ports. Fast-forward to post-pandemic and much of China was locked down for almost a year, the Russia-Ukraine region has been in a war, and the Great Resignation has grown across the world (reducing the number of people in the workforce to lower than pre-pandemic levels), severely limiting supply while demand increased as people started to spend money, catch up on medical appointments, and live life again.

Thus, critical shortages and extended lead times remain while there is a glut of inventory of the “wrong” products in the “wrong” place at the “wrong” time, leading to continued disruption, inflation caused by limited supply, and recessionary signals caused by the cost of capital and the oversupply of “wrong” products throughout the supply chain. To make matters more challenging, global tensions are on the rise with China and several other countries that supply essential medical devices, key materials, and active pharmaceutical ingredients, as well as other critical commodities and components.

Added Volatility

Inflation, workforce issues, and labor shortages are further disrupting healthcare supply chains. The country faces a shortage of up to 124,000 physicians by 2034, including 48,000 primary care physicians, according to the Association of American Medical Colleges. Of immediate significance, according to The American Hospital Association, 136 rural hospitals closed from 2010 to 2021 alone. And, according to its chief healthcare executive, the Texas Hospital Association has warned that one in 10 hospitals in that state is at risk of closure, with nearly half of that state's hospitals projecting negative operating margins. As these medical professional shortages persist and closures occur, patients still require attention. Healthcare is on the move, and the supply chains will have to catch up. Thus, more of the “wrong” items will be in the “wrong” places at the “wrong” time, creating additional disruption, inflation to move and/or transfer them (from one owner to the next), and inventory stockpiles in the “wrong” place.

Successful Path Forward

There will be more opportunity than ever before for proactive, resilient, and innovative companies to gain market share during these volatile times. The successful companies will take control. Starting by targeting their ideal customers, they will focus limited resources on what provides the most value to these key customers, including providing value-add services such as vendor-managed inventory so that their customers have the “right” products in the “right” place at the “right” time with minimal resources and risk.

They will go further into their supply chain to assess risk and mitigate shortages of critical components and supplies due to resolvable issues such as delays in transportation and material and labor issues at a third-tier supplier. Reliability will be prioritized over cost, and additional suppliers will be qualified even though the cost and time required is high. Backup suppliers will be scalable to mitigate issues such as those that occurred in the baby formula market. And taking control of essential healthcare supply chains will become a priority as reshoring and nearshoring production gains momentum. For example, Costa Rica and Mexico are building strong medical device manufacturing clusters to support healthcare supply chains. And the successful will deploy technology to support the sustainability and scalability of these initiatives.

It is no longer sufficient to leave manufacturing and supply chain reliability to chance. The proactive, resilient, and innovative will thrive and gain the opportunity to grab market share from those remaining on the roller coaster of volatility. Think ahead, be proactive, and be willing to invest in supply chains of the future to support your ideal customers and be uniquely positioned to grow and thrive.

For more information, contact the author at landerson@lma-consultinggroup.com or visit www.lma-consultinggroup.com.

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EUROPEAN PERSPECTIVES



THE UNIQUE PROPERTIES OF ADHESIVES AND SEALANTS IN ELECTRONICS AND DISASSEMBLY FUNCTIONS

The adhesives industry will continue to develop products for the electronics industry that optimize durability and end-of-life solutions through options such as recycling and repair potential.

By Isabelle Alenus, Senior Communications Manager, FEICA

Considering Europe's future-focused drive towards an increasingly circular economy, FEICA actively supports efforts to improve product efficiency and circularity to help create a more sustainable society. The use of adhesives and sealants in many sectors, including electronics, contributes to improved durability, recycling, CO₂ reduction, reparability, upgradability, and material efficiency of final products.

To FEICA and its members, it is important to raise awareness of the adhesive and sealant industry's contribution to the circular economy, internally and externally, in terms of both activities to improve the sustainability of its own industry and help for customers and end-of-supply-chain industries (recyclers) to operate more sustainably. They therefore support the need for Ecodesign, for example, in order to encourage end-of-life reuse and recycling solutions for electronic products and components.

The aims for reducing waste and ensuring full circularity of the economy go beyond what economic actors can achieve alone. FEICA has already been an active advocate of the Ecodesign legislation proposals for electronics and supports the principle that design for circularity is important.

The value proposition of adhesives and sealants is an integral part of many global value chains. They are an outstanding and versatile fixing solution. For adhesive technologies to make the sustainability benefits of next-generation goods possible, the use of appropriate adhesive bonding solutions to facilitate repair and recycling should be properly planned and engineered during the design phase of a product.

Adhesives in Electronic Device Design

The use of adhesives as a joining technology in electronics allows the extension of the life span of products and components, offering increased durability with waterproof capabilities. These solutions allow designs for lighter, more shock-resistant, and better sealed products. They enhance features such as resource efficiency through increased durability and lifespan of the product, leading to fewer repairs and conservation of resources, as well as convenience and safety for the consumer. Adhesives additionally bear many advantages in the manufacturing processes, such as energy and material efficiency. With regard to the climate goals of the EU Green Deal, the complete life cycle of a product should be taken into account.

The adhesives industry will continue to cooperate by reaching out to relevant sectors or contributing to consultations of Ecodesign studies from the EU Commission to optimize durability and end-of-life solutions through options such as recycling and repair potential. We plan to further engage with relevant experts, as needed, to support a societal and pragmatic shift to a more sustainable future.

Learn more at www.feica.eu.

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