

# Novel Organic-Inorganic Hybrid Nanocomposite Coatings by UV-Initiated Sol-Gel Process

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UV-cure coatings have become systems of choice for plastic substrates, primarily due to their low-temperature cure, absence of potentially harmful solvents, and rapid curing. Free-radically curing acrylate resin systems are the ones most commonly used for commercial UV-cure systems for plastics. However, these systems have some inherent limitations—such as oxygen (air) inhibition of curing, poor substrate wetting, shrinkage and odor—that often limit their applications.

A novel chemistry utilizing silane compounds has been explored to study its suitability as an acrylate-free, UV-curable system. We have demonstrated that such systems undergo crosslinking reactions through a UV-initiated, sol-gel process resulting in the formation of hybrid organic-inorganic nanocomposite films. A systematic design of experiment (DOE) methodology has been used to study the effects of various parameters on the properties of the nanocomposite films deposited on polycarbonate substrates.

Organic coatings are widely used for protection and decoration of metals and alloys, plastics, wood and masonry, among others. Using different types of organic polymers and crosslinkers, it is possible to

derive coatings with a wide range of performance required for a myriad of industrial applications. While such organic coatings provide satisfactory protection, many industrial applications require much higher performance—for instance, superior corrosion resistance or high temperature resistance—along with general mechanical and physical properties. In recent years, organic-inorganic hybrid coatings have emerged as promising materials because they combine the properties of inorganic polymers (inertness, chemical and heat resistance, hydrolysis resistance) with properties of organic polymers (flexibility, toughness, compatibility, adhesion, etc.).

Among the number of available processes for deriving organic-inorganic hybrid nanocomposite coatings, the one based on a sol-gel process using silane-functional precursors has been very popular and promising. A traditional approach for sol-gel processing involves dissolving the silanes (or organosilanes) in a water/alcohol/acid mixture and allowing the silane-functional material to hydrolyze into silanols (sol). The resulting sol can then be applied (by dipping or spraying, for example) onto a substrate and baked. Film formation results from the condensation reaction of the silanols to siloxane (gel).<sup>1</sup> Depending

upon the type of silane precursor used, the films formed can be an inorganic or organic-inorganic hybrid type. One of the most interesting features of such systems is that it has potential to form a covalent linkage (Si-O-Metal) with some substrates such as aluminum. This significantly enhances coating adhesion and, hence, a number of performance properties.

While this traditional method of deriving sol-gel coating is very popular, there are a number of limitations. The silane-functional precursors must be soluble in water/alcohol and this limits chemistry of such precursors. Sols have limited stability and can undergo “gel” reaction at room temperature. This restricts their application window. Percent solids of application bath are typically low which restricts dry-film thickness of coatings. Bath solids also affect the application window; the higher the percent solids, the shorter the window. This makes it challenging to apply thick coatings.

In the present work, we have explored the possibility of deriving organic-inorganic hybrid coatings by using an alternate UV radiation-induced, sol-gel reaction that can alleviate most of the limitations of the traditional system stated above. There are number of literature references wherein sol-gel reaction of silanes have been initiated by UV radiation using a photoacid generator (PAG).<sup>2-10</sup> Upon exposure to UV, the PAG generates a super acid in situ that catalyzes the hydrolysis and condensation reactions. Atmospheric moisture has been found to be satisfactory for hydrolysis. This approach eliminates the hydrolysis step prior to application and, hence, significantly extends the application window. The silane precursors do not need to be soluble in water/alcohol, allowing the use of a variety of silane precursors. Also, these systems can be coated at much higher percent

solids, and viscosity can also be more easily controlled.

In the present work, two model urethane-silane oligomers of differing molecular weights have been synthesized from polycarbonate diols and an isocyanate-functional silane. These silane precursors were used to derive organic-inorganic hybrid coatings onto polycarbonate films as substrate, using UV radiation in the presence of a PAG. Compositions with titanium isopropoxide (TIP) as an additive have also been prepared as it is found to significantly improve coating properties, as per an early patent.<sup>6</sup>

The present work investigates the effects on various physical properties of three variables—oligomer molecular weight (MW), presence of TIP and humidity after UV exposure. The experiment was set up using a *Design of Experiment* (DOE) methodology using a 2<sup>3</sup> matrix—each of the three variables is set to a low and high value for a total of eight tests. The DOE method is used to determine which of the three variables, as well as the interactions between them, have a significant effect on the results of a particular test.

## Materials and Methods

Acetone and diacetone alcohol (DAA) were reagent-grade chemicals from Sigma-Aldrich. The TIP was 97% active and was from Sigma-Aldrich. Dibutyltin dilaurate (DBTDL) was 95% active and was from Sigma-Aldrich. The isopropyl-9H-thioxanthene-9-one (ITX), 97% mixture of 2- and 4-isomers was from Sigma-Aldrich. The 3-isocyanatopropyltrimethoxysilane (IPTMS) was a sample from Momentive Performance Materials. The isocyanate (NCO) equivalent weight of the IPTMS was 205 grams/eq. The polycarbonate diols, Desmophen C 1100 (PC-diol-500) hydroxyl equivalent weight was 486 grams/eq, and

Desmophen C 1200 (PC-diol-1000), hydroxyl equivalent weight was 944 grams/eq, were commercial samples from Bayer MaterialScience. The PAG used was Irgacure 250 (Iodonium, (4-methylphenyl) [4-(2-methylpropyl) phenyl]-, hexafluorophosphate, 75% active in propylene carbonate) from BASF. All materials were used as received.

Untreated polycarbonate (PC), 7 mil thickness, T2F OQ from GE was used as the substrate. Using the methods described below, the uncoated PC has a haze of 0.0 and a Taber abrasion of 30.23. The PC is supplied with a protective film on both sides to prevent scratching. One side was peeled off prior to coating. The other side was removed later during haze testing. The fluoropolymer release (FPR) film was R-1655B from Precision Coatings Inc. (Walled Lake, Mich.). This is a white PET film that has been coated with a fluoropolymer to give it nonstick properties.

## Experimental

### Synthesis

Two urethane silane oligomers (silane precursors) have been synthesized under extremely dry reaction conditions, by reaction of PC-diol with IPTMS, using DBTDL (0.02% of total mass) as catalyst in a 500 mL three-neck flask under nitrogen and with continuous stirring. Extra-dry acetone was used as solvent. The reactants were used at NCO:OH ratio of 1.01:1.0 and reaction was carried out at 67-70°C until all the IPTMS was consumed, as indicated by the absence of -NCO absorption peak in the Fourier transform infrared spectroscopy (FTIR) spectrum of the reaction product. The product prepared from PC-diol-500 has been designated as U1 and the one made with PC-diol-1000 as U2. Products were adjusted to %NVM of 76% using acetone and stored in dry, dark bottles.

TABLE 1

## Coating compositions for UV-cure coatings

Component	Weight %			
	F1	F2	F3	F4
Urethane-Silane (U1 or U2)	63.4	62.6	59.9	59.1
Titanium isopropoxide (TIP)	0.0	0.0	2.4	2.4
Diacetone alcohol	34.7	34.7	34.7	34.7
Acetone	0.1	0.9	1.1	1.9
Irgacure-250	1.5	1.5	1.5	1.5
ITX	0.3	0.3	0.3	0.3
<b>Total</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>
<b>Urethane-Silane used</b>	<b>U1</b>	<b>U2</b>	<b>U1</b>	<b>U2</b>

**Coating Application and Curing**

The coatings were applied to the substrates by manual drawdowns with a #22 Mayer rod. The coatings were subjected to a pre-UV heating cycle (five minutes at 120°C), followed by exposure to UV, and finally a post-UV heating cycle (15 minutes at 120°C). A Fusion UV system with a D-bulb was used for UV curing. All UV curing was done at 25 feet/min. Using a compact radiometer from UVPS, the energy density measured was 994.76 mJ/cm<sup>2</sup>. Two ovens were used—a Scientific Products Constant Temperature Oven DK-43 was used for the pre-UV heating cycle and a Tenney Environmental Test Chamber model BTRC with a VersaTenn III controller was used for the post-UV heating cycle (where the humidity needed to be controlled).

**Testing and Evaluation**

A Bruker Tensor 27 FTIR was used to analyze the urethane-silane oligomers that were synthesized. NaCl plates were used. NCO determination by titration was performed according to ASTM D 2572-97.

The physical and mechanical properties of the coatings made from the urethane-silane oligomers

were determined by several different methods. Adhesion was tested by a cross-hatch tape pull (ASTM D 3359-02). Scotch 610 tape was used for all tests. Solvent resistance was determined by MEK double rubs. A Sheen 707KP Pendulum Hardness Rocker was used for all König tests. For all samples tested, at least three readings were taken and averaged. Results are reported in seconds. Coating thickness on polycarbonate was measured with a Heidenhain thickness gauge. Thermal Gravimetric Analysis (TGA) was performed with a TGA Q 500 from TA instruments. The rate of temperature increase was 20°C/min. and the max temperature was 560°C. The onset of degradation (the temperature at which 95% of the original material remains) was recorded, as was the weight of the residue remaining at the end of the test.

Glass Transitions temperatures of the cured films were determined by a Q 2000 Differential Scanning Calorimeter (DSC) from TA instruments. Hermetically sealed T-zero pans were used. The temperature program was as follows: Room temperature to 175°C at a rate of 20°C/min.; 175°C down to -80°C at

a rate of -5°C/min.; -80°C to 175°C at a rate of 20°C/min. Haze was measured with a Hunter Lab Colorquest II Colorimeter—"T-tran" Haze mode was used. Three readings were taken on each sample and the average was reported. Taber abrasion was measured with a Taber abraser model #5130. Each sample tested was subjected to 100 cycles using 500 gram weights. One sample of each variation was tested. The difference in haze before and after abrasion was reported. The contact angle of four solvents (toluene, benzyl alcohol, formamide and water) on the coated films was determined by a FTA200 Dynamic Contact Angle analyzer from First Ten Angstroms. Surface energy was determined by using a Zisman plot.<sup>11</sup> The coated samples were analyzed by Attenuated Total Reflectance IR (ATR-IR); a Bruker Hyperion ATR system was used.

**Coating Composition, Application and Curing**

In order to study the effect of formulation parameters and cure conditions on the film properties, DOE methodology has been adopted. Statgraphics Software (Statgraphics Plus 5.1 from Statistical Graphics Corp.) was used to design the experiment

and to determine which variables had significant effects on each of the physical properties studied. As shown in Table 1, four coating compositions were prepared using U1 and U2 type silane precursors. DAA was used as solvent since it was found to provide good film formation capability and compatibility. The coating formulations F1-F4 used are listed in Table 1 and were made in the following manner—the PAG and ITX were dissolved in diacetone alcohol; then the urethane-silane oligomer (U1 or U2) was added, followed by the acetone and TIP (if applicable); finally, this was hand-stirred for about three minutes.

As per the DOE methodology, an (L-8) experiment—three variables at two levels—was set up. The three variables, varied at 2 levels, are (A) MW of urethane-silane oligomer (-OH equivalent wt. 486 g/eq., and 944 g/eq.); (B) amount of TIP added (0 or 5% by weight based on solids); and (C) relative humidity of post-UV heating cycle (5% RH and 44% RH). Since two of the variables are related to the coating formulation, there are a total of four formulas (designated F1-F4). The DOE matrix is summarized in Table 2. The eight coated samples that result from this matrix are designated S1-S8.

For each of the eight experiments in the series, the coatings were applied to both PC and FPR. All coatings were applied to the substrate with a #22 drawdown application rod and heated for five minutes at 120°C to remove the solvents (this is the pre-UV heating cycle). Then, the coatings were passed through the UV-cure unit five times. Finally, the coatings were heated for 15 minutes at 120°C (this is the post-UV heating cycle). The humidity of the oven was set according to variable C.

The coated samples were then evaluated after 24 hours for appearance, adhesion, König pendulum hardness, thickness, solvent resistance, haze, Taber abrasion, pencil hardness, surface energy, DSC, TGA and ATR-IR. The samples for DSC and TGA were prepared by scraping the coating off the samples coated on the FPR.

## Results and Discussion

### Synthesis of Silane Oligomers (Precursors)

Silane precursors U1 and U2 were synthesized by single-step reaction of PC-diols and ICPTMS using DBTL catalyst. Completion of reaction was confirmed by absence of absorption peak at 2270cm<sup>-1</sup>, corresponding to -NCO group in FT-IR spectrum of

the product.<sup>12</sup> NCO content was also determined by titration method (ASTM D-2572) that showed % NCO 0.01. FT-IR spectra of U1 and U2 silane oligomers are shown in Figure 1. Among other absorption peaks, note the most important one is the peak at 1087cm<sup>-1</sup> (Si-O-C asymmetric stretching)<sup>2,13</sup>. Also, note disappearance of peak at ~3400 corresponding to -OH stretching.

### Application and Properties of Cured Coatings

Among the compositions listed in Table 1, F3 and F4 (containing TIP) showed very short pot life. This can be explained based on the very high hydrolysis/condensation rate of TIP in presence of ambient moisture. The formulas without TIP seemed very stable over a period of a few days.

Coatings were applied in PC and FPR substrates with dry-film thickness of ~10 μ. The coated samples containing TIP were dry-to-touch after the pre-UV baking cycle, while the samples without TIP (F1 and F2) remained distinctly wet. The coated samples of F1 and F2 became dry-to-touch after the first pass of UV curing. The samples with TIP coated on both PC and FPR had a very uniform appearance (coatings were free of orange peel and did not

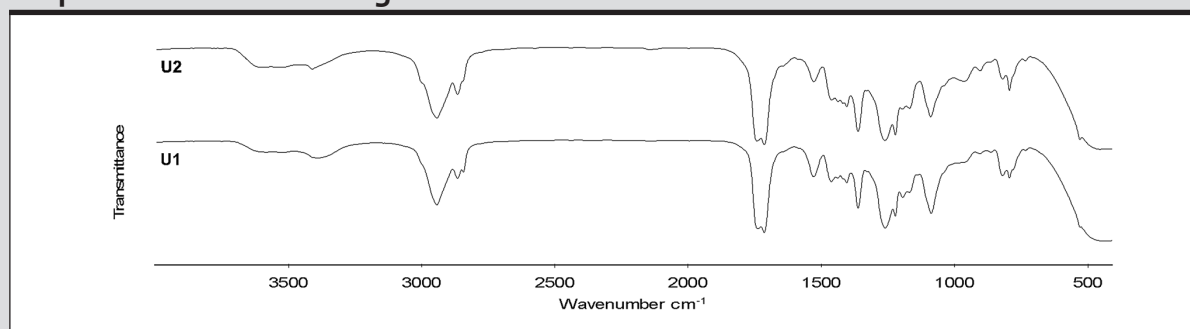
## TABLE 2

### Coating samples—2<sup>3</sup> DOE matrix (three variables at two levels)

Sample	Oligomer MW A	TIP B	Humidity C	Formula
S1	low MW (U1)	0%	low	F1
S2	high MW (U2)	0%	low	F2
S3	low MW (U1)	5%	low	F3
S4	high MW (U2)	5%	low	F4
S5	low MW (U1)	0%	high	F1
S6	high MW (U2)	0%	high	F2
S7	low MW (U1)	5%	high	F3
S8	high MW (U2)	5%	high	F4

## FIGURE 1

## IR spectra of U1 and U2 oligomers



dewet). The coatings on PC without TIP had slight orange peel, while those on FPR completely dewetted—the dewetting occurred during the pre-UV heating cycle. The dewetting was more severe for the coatings made with the lower MW oligomer (U1). Thus, TIP has significant effect on composition stability, appearance and other properties of coatings. It is believed that this effect arises from very rapid hydrolysis/condensation of TIP in presence of ambient moisture, as well as its catalytic activity in

thermal degradation reaction.<sup>17</sup> All of the coatings had a soft, elastomeric (rubbery) feel.

Table 3 shows characteristics of cured coatings on PC substrate. Adhesion of all coatings to PC has been found to be excellent—all samples were rated as 5B with the cross-hatch adhesion test. Surprisingly, the adhesion to the FPR was very good; the coating could not be removed with tape. Solvent resistance, determined by MEK double rubs, was very low (10 double

rubs) for all samples. This could be due to high free volume and high permeability of siloxane for MEK.

The results for the König pendulum, pencil hardness, haze, Taber abrasion, surface energy, TGA and DSC are summarized in Table 3. A representative TGA thermogram (of sample S1) is seen in Figure 2. DSC thermograms of samples S1 and S2 are seen in Figure 3. As can be expected, decreasing the MW of the oligomer increased the König pendulum hardness as well as the Taber abrasion. Decreasing the MW of the oligomer increases both the crosslink density and the urethane group content of cured films. This can be attributed to increase in pendulum (König) hardness, glass transition temperature and abrasion resistance. The increased urethane group content also explains the slight increase seen in surface energy of systems containing low MW oligomers. Increasing the MW of the oligomer seemed to improve wetting, owing to their higher viscosity.

The humidity of the oven during the post-UV heating cycle had no significant effect on any result. We speculated influence of humidity on extent and speed of silane hydrolysis and, hence, on cured film properties. However, based on the results of this study, it appears that most of the hydrolysis takes place upon exposure

## FIGURE 2

**A representative TGA thermogram (sample S1) showing the onset of degradation (i.e., 5% wt. loss), and the percent residue at 560°C**

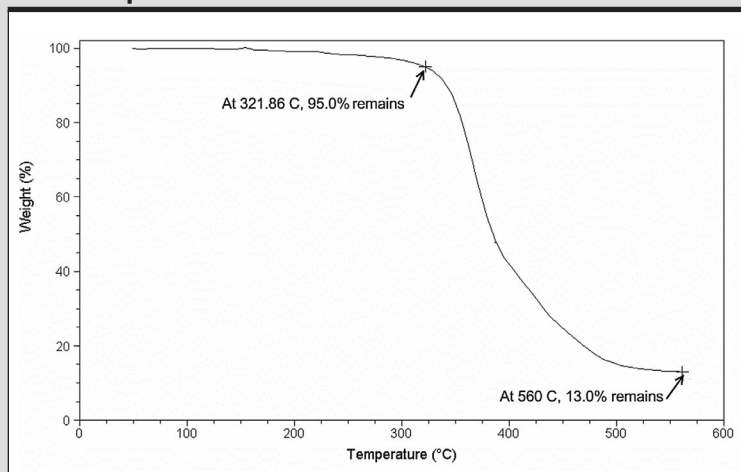


TABLE 3

## Characteristics of UV-cured coatings on PC substrate

Coating Identity	König Hardness, s	Pencil Hardness	Haze	Taber Abrasion Rating	Surface Energy (J/m <sup>2</sup> )	TGA 95%(°C)	TGA Residue (% by wt.)	Tg (°C) (DSC)
S1	134.4	6B	6.1	6.56	27.7	321.9	13.02	-46.41
S2	70	4B	7.07	37.73	23.4	329.5	7.05	-51.39
S3	136.1	2B	0.27	45.8	29.6	206	14.29	-46.93
S4	106.9	2B	0.22	59.95	28.3	214.1	10.92	-53.95
S5	137.5	6B	4.28	3.56	28.9	320.4	12.02	-44.45
S6	87.1	4B	5.04	54.64	22.3	329.2	7.06	-52.46
S7	141.4	2B	0.26	50.72	29.3	219.7	15.74	-48.51
S8	99	2B	0.4	67.39	27.7	227.2	10.37	-53.09

of the wet film to UV, under influence of ambient moisture and in situ generated nascent acid catalyst. If there were still unhydrolyzed methoxy silane groups after UV cure, then exposure to a humid environment would likely help complete hydrolysis.

The presence of TIP had a major effect on many of the coating properties. It completely prevented dewetting on both the PC and FPR. Given that the coatings with TIP become dry-to-touch during the pre-UV heating cycle, it appears that the viscosity is rising fast enough (due to crosslinking) to prevent dewetting.

The effect of TIP on the haze of the coatings was drastic. With TIP, the coatings are extremely clear; without TIP, they are obviously hazy. Coatings containing TIP also showed improved pencil hardness. The slight decrease in abrasion resistance of coatings containing TIP may be due to their thermal degradation for which TIP is a known catalyst.<sup>17</sup> This is also consistent with the observed TGA findings of over 100°C difference in the degradation temperatures of coatings with and without TIP. The exact role TIP plays is not known, but it appears that it influences coating properties

by acting as a crosslinker (by forming Si-O-Ti linkages) as well as thermal degradation catalyst.

One of the most interesting results from these experiments is that *all* formulations had outstanding adhesion to polycarbonate. This can open up many potential applications for such coatings.

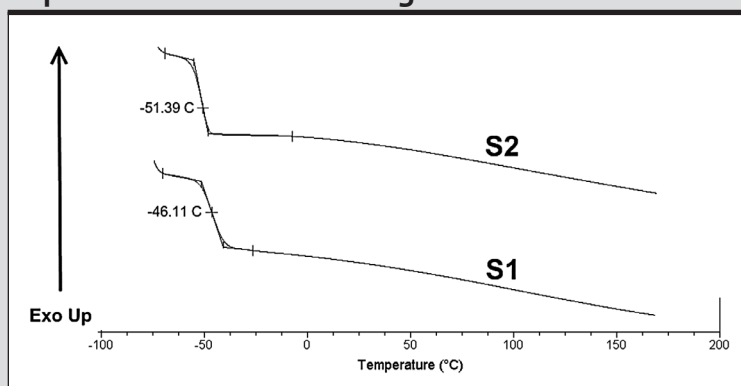
#### Structure of Cured Coatings

The FTIR spectra and DSC study provide useful insight into the structure of the cured coatings.

In comparing the FTIR spectra of the oligomers with their corresponding cured film samples in Figure 4, it is clearly seen that a sharp absorption peak for oligomers at 1087  $cm^{-1}$  (Si-O-C asymmetric stretching)<sup>2,13</sup> is replaced by a broad band at 1,000-1,140 $cm^{-1}$  (Si-O-Si stretching)<sup>14</sup>; indicating formation of siloxane network by sol-gel reaction under UV-cure conditions. Absence of peak at 850  $cm^{-1}$  corresponding to Si-OH indicates complete condensation of silanol groups to siloxane. Thus, as shown in Figure 5, the structure of cured film coating is comprised of nano-silicate inorganic domains formed as a result of sol-gel reaction. The organic component (polycarbonate

FIGURE 3

## Representative DSC thermograms for S1 and S2





chains) serves as a matrix for these inorganic domains that are covalently bonded to the matrix chains.

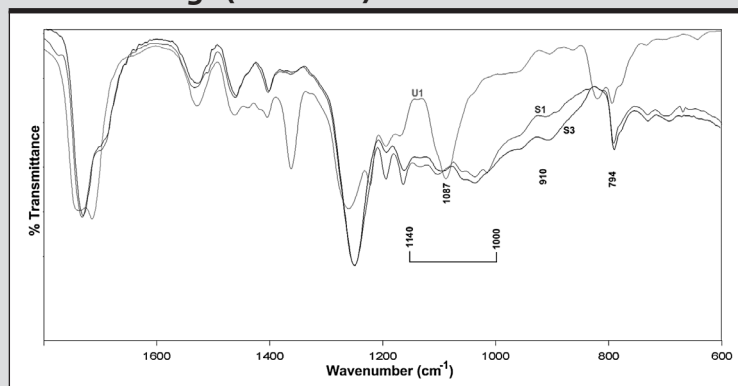
The coated samples containing Ti have a band around  $910\text{cm}^{-1}$  which seems to indicate the formation of Si-O-Ti bonds (this is easily seen in Figure 4). Also, there is not an obvious peak or band around  $600\text{cm}^{-1}$  that would conclusively indicate Ti-O-Ti bond formation. The formation of Si-O-Ti bonds means that the TIP is acting as a crosslinker for the silanes. This would certainly explain the decrease in dry-to-touch time as well as the short pot life. The DSC thermograms for all eight variations (those of S1 and S2 are shown in Figure 3) show a single endothermic baseline shift indicative of a glass transition. No other features are present. The single  $T_g$  suggests that no phase separation occurred during curing.

## Conclusion

Urethane silane oligomers of different molecular weights were prepared to investigate their potential for formation of organic-inorganic hybrid films when exposed to UV radiation in the presence of PAG. The study shows that organic-inorganic hybrid coatings can be efficiently prepared from such oligomers and PAG under normal UV-cure conditions. A crosslinked coating structure, resulting from sol-gel reaction, is affected by number of variables. A systematic DOE study has been carried out to identify the influence of different compositional and process variables. This study reveals that the use of TIP significantly influences appearance and performance properties of such coatings. The study also shows that the molecular weight of telechelic oligomers, more specifically their functional group content, and urethane content significantly affect film properties. The relative humidity

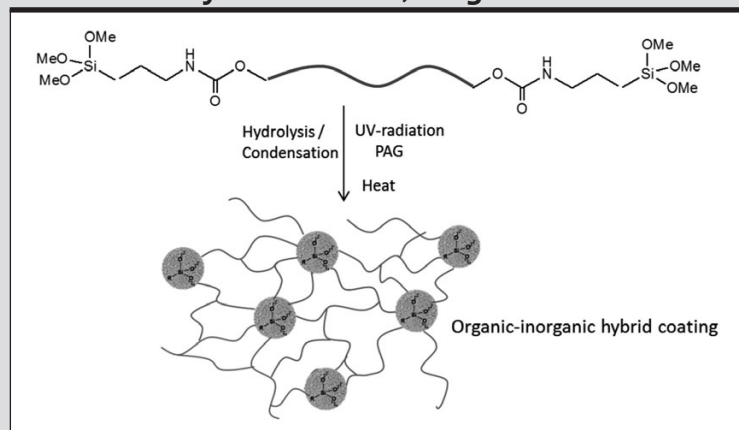
## FIGURE 4

### FTIR spectra of representative oligomer (U1) and cured coatings (S1 and S3)



## FIGURE 5

### Schematic structure of organic-inorganic coatings film formed by UV-initiated, sol-gel reaction



during post UV-cure baking did not affect properties of cured films. The study concludes that silane functional oligomers can successfully be used as components of UV-curable coatings to derive organic-inorganic hybrid coatings for various end-use applications. ▀

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## Technology Update

### Sartomer Hosts UV/EB Technical Seminar

Sartomer USA LLC, a global specialty chemical manufacturer, will hold a free technical seminar on ultraviolet light and electron beam (UV/EB)-curing chemistries on March 26–28, 2013, at the Desmond Hotel in Malvern, Pa., near Philadelphia. The seminar is geared toward industrial chemists and product formulators who want to expand their practical knowledge of current UV/EB chemistries.

The event will feature technology experts from Sartomer and recognized industry leaders from other organizations in the related areas of formulation additives and UV/EB curing equipment. Presentations will

be relevant for attendees with all levels of UV/EB experience. The first two days will focus on chemistry and introductory topics, with more advanced material discussed on day two.

### Participants will receive detailed information about:

- Monomer chemistry
- Oligomer chemistry
- Photoinitiator chemistry
- UV and EB sources
- Additives for UV/EB
- Application-specific products for graphic arts, adhesives, and wood, metal and hard coatings
- Acrylates in non-UV/EB applications
- Advanced adhesion, light and mechanical properties, testing procedures, MVTR, OTR and weathering

Attendance is limited and reservations should be made as soon as possible by contacting James Goodrich, UV/EB technical manager, at [james.goodrich@sartomer.com](mailto:james.goodrich@sartomer.com). A complete seminar agenda is available online at [www.sartomer.com/events.asp](http://www.sartomer.com/events.asp).

Globally based in Exton, Pa., Sartomer USA LLC, is part of Arkema, Inc. Sartomer is a global supplier of specialty chemicals for UV/EB, peroxide and two-part epoxy/amine systems. For more than 50 years, Sartomer has pioneered the development of these advanced technologies, introducing hundreds of products that enhance performance in coatings, inks, adhesives, composites, rubber and other demanding applications. For more information, visit [www.sartomer.com](http://www.sartomer.com). ▀