

Tougher Cycloaliphatic Epoxide Resins

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Introduction

Cycloaliphatic epoxides are used in cationic UV coatings to provide good cure rate and hardness. Polyols can be used in cationic coatings to improve coating flexibility and they help improve the through-cure rate. Higher polyol concentrations generally result in more flexible but softer cationic coatings and less polyol results in more brittle and harder coatings. For some applications preferred coatings would have high flexibility and hardness simultaneously. It would be helpful if cycloaliphatic epoxides that were harder and more flexible were available.

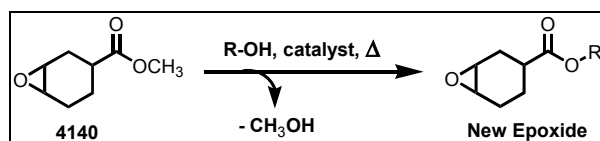
A variety of cycloaliphatic epoxides can be prepared by transesterifying a cycloaliphatic epoxy ester monomer with hydroxyl materials (e.g. polyols) using an appropriate catalyst. This method was used to prepare a series of cycloaliphatic diepoxides containing acyclic backbones having different molecular weights. The series of cycloaliphatic epoxides was tested in UV coatings to determine the effect of molecular weight on coating properties. Cycloaliphatic epoxides containing cyclic (cyclohexylene) groups in their backbones were also prepared to determine if they could provide harder and more flexible UV coatings.

Generally, a benefit of cationic UV compositions is good adhesion. However, improved adhesion to difficult substrates would be helpful in some applications. A new cycloaliphatic epoxide containing a cyclic group in the backbone and polyols containing cyclic and tricyclic groups were tested in UV coatings to determine their effect on adhesion to difficult substrates.

Experimental Procedures

The transesterification reaction between methyl-3,4-epoxycyclohexanecarboxylate (ERL-4140) and polyols, shown generically in Figure 1, was used to prepare new cycloaliphatic epoxides. The transesterification reactions were conducted using 0.1 mol% sodium acetate catalyst (based on 4140) at 130 °C for 3 to 4 hours. Nitrogen sparge and vigorous mixing were used to help remove methanol during the reactions.

Figure 1. Generic Example of the Transesterification Reaction Involving ERL-4140 and Polyol (R-OH)



Epoxide equivalent weight (EEW) values were measured using a titration method. Viscosity values were measured using a Brookfield viscometer after the samples had been equilibrated in a water bath at 25 °C.

Ethylene glycol, 1,3-propanediol, 1,4-butanediol, 1,8-octanediol, 1,4-cyclohexanediol, and *trans*-1,2-cyclohexanediol were purchased from Aldrich. 1,6-Hexanediol and 1,10-decanediol were purchased from Acros. 1,4-Cyclohexanedimethanol (1,4-CHDM) was obtained from Eastman Chemical Company.

ERL-4140, 6110, 6128, polyol 0301, and 6992 were obtained from The Dow Chemical Company. 6110 and 6128 were commercial cycloaliphatic epoxy resins. Polyol 0301 was a commercial triol containing caprolactone and it had a hydroxyl equivalent weight value of 100-g/hydroxyl equivalent. 6992 was a commercial cationic photoinitiator containing mixed triaryl sulfonium hexafluorophosphate salts and propylene carbonate. Liquid CHDM was an isomeric mixture of 1,3/1,4-CHDM and it was a developmental product obtained from Dow. Liquid CHDM was used as a polyol in some of the coatings tested for adhesion.

Tricyclodecanedimethanol (TCDM) was a sample of a commercial product (TCD alcohol DM; CASRN 26896-48-0/26160-83-8) obtained from Celanese Chemicals. It was used as a polyol in some of the coatings tested for adhesion. A commercial silicone surfactant (Silwet L-7604) was obtained from OSi Specialties (GE).

All of the coatings presented contained epoxy resin, polyol 0301, 4 wt% 6992, and 0.5 wt% surfactant unless otherwise noted. The ratio of epoxide to hydroxyl equivalents (*R*-value) was two for the coatings unless otherwise noted. Coatings tested for wetting did not contain surfactant. Epoxy resin E14CHD (Table 1) was a solid at room temperature. E14CHD was melted and then mixed with the other coating ingredients. A coating containing E14CHD and polyol 0301 (*R*-value = 2) was not tested because E14CHD precipitated from solution after the coating mixture cooled to room temperature. A coating mixture containing E14CHD and 6110 at 50/50 w/w and no polyol appeared to remain homogeneous and no precipitate formed at room temperature.

The coatings were applied at a thickness of 4 to 5 microns using a 2.5 wire-wound rod to the substrates indicated. The coatings were cured using an energy density of 150 mJ/cm² unless otherwise indicated. The energy density 150 mJ/cm² was obtained by using a 300 W/in mercury UV lamp (Fusion H) and a conveyor speed of 100 ft/min. The coatings tested for adhesion were cured using an energy density of about 600 mJ/cm², which was obtained using a conveyor speed of 25 ft/min. Energy density was measured using radiometer model IL-390B from International Light Inc. The coatings tested for flexibility and pendulum hardness were heated in an oven at 400 °F (204 °C) for 10 min after UV curing to simulate a food can end manufacturing process.

Coating flexibility was determined using a retort wedge-bend test method. Coated tin-free steel (TFS) panels (100 mm wide) were bent and impacted using a wedge-bend instrument (ATSM D3281). The TFS was obtained from Weirton Steel. The bent panels were placed in the vapor phase of an autoclave and processed at 250 °F (121 °C) for 1-h using deionized water. The length (mm) of the continuous crack visible along the bend after processing was measured and recorded as the crack length. A larger crack length indicated a more brittle coating. Coating hardness was measured using a König pendulum hardness instrument (ASTM D4366).

An experimental thermal cationic catalyst (a trialkyl sulfonium hexafluoroantimonate salt) was added to epoxy resins 6110 and 4360 and the samples were cured in a mold in the shape of plaques (1 mm x 5 mm x 40 mm) by heating the samples in a forced-air oven for 1-h at 204 °C. The plaques were tested using dynamic mechanical analysis. Coating surface-cure rates were measured using a cotton ball method, which has been described previously.¹ The relative humidity was >50% when the coatings were tested for physical properties including surface-cure rate. Coating adhesion was measured immediately after UV cure (600 mJ/cm²) using a crosshatch tape test method (ASTM D3359) and Scotch 610 tape was used to measure adhesion.

The relative wetting performance of coatings was determined by applying coatings to TFS panels and UV curing the samples 10-s later. The time interval was used to give the coatings

the same opportunity to de-wet. The coatings tested for wetting contained epoxy resin, polyol, 4% 6992, and no surfactant. The cured coatings were rated for wetting based on their appearance relative to one another.

Results and Discussion

Preparation of New Epoxides via the Transesterification of ERL-4140

E278, E292, E306, E334, E359, and E391 in Table 1 signify new epoxides prepared using a homologous series of alpha,omega-diols (C2, C3, C4, C6, C8, and C10, respectively). ERLX-4360 signifies the new epoxide prepared using 1,4-CHDM. E14CHD and E-t12CHD signify model epoxides prepared using 1,4-cyclohexanediol and *trans*-1,2-cyclohexanediol, respectively. ERLX-4360, E14CHD, and E-t12CHD were examples of epoxides containing cyclic (cyclohexylene) groups in the backbone. The materials 6110, 6128, polyol 0301, CHDM, and TCDM are also presented in the table. CHDM and TCDM represent diols containing cyclic and tricyclic groups in the backbone, respectively.

Analysis using gas and liquid chromatography indicated that 4360 samples contained diepoxide, mono-epoxy alcohol, oligomers, 4140, and 1,4-CHDM. Mono-epoxy alcohol was the product of the transesterification reaction between 4140 and only one of the hydroxyls of 1,4-CHDM. The concentration of mono-epoxy alcohol was almost 20% in 4360. The samples of the other epoxy resins prepared using 4140 likely contained mono-epoxy alcohol also because they were prepared in manner very similar to 4360. A non-routine analytical technique (mass spectroscopy) was required to confirm the presence of mono-epoxy alcohol. The concentration of mono-epoxy alcohol was therefore not determined in all the samples. The *R*-value of the coatings was calculated using experimental epoxy resin EEW values (see Table 1). The concentration of hydroxyls associated with mono-epoxy alcohol was not taken into consideration when calculating *R*-values. The *R*-value of the coatings containing the experimental epoxides were likely less than two because of the presence of hydroxyls that were not counted.

Performance of the Experimental Epoxides in UV Coatings

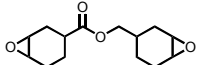
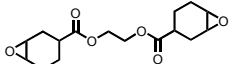
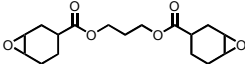
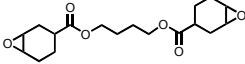
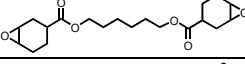
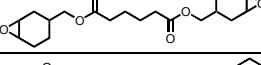
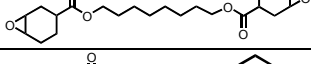
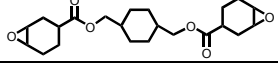
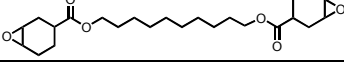
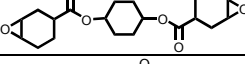
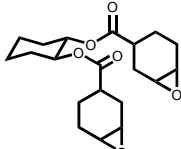
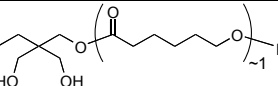
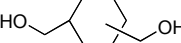
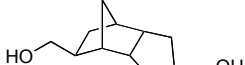
The hardness, flexibility, and surface-cure rate results of coatings containing the homologous series of epoxides containing acyclic backbones (E278, E292, E306, E334, E359, and E391), 4360, and the commercial epoxides (6110 and 6128) were plotted as a function of EEW. These coatings also contained polyol 0301 (*R*-value = 2). These plots highlighted the effect of resin EEW on coating properties.

Hardness decreased with increasing resin EEW (Figure 2) for the coatings containing epoxy resins having acyclic backbones including 6110 and 6128. Three factors likely contributed to the decrease in coating hardness with increasing acyclic chain length of the epoxy resins: (1) increased backbone mobility, (2) lower concentration of cyclic groups associated with the epoxide groups, and (3) lower crosslink density.

The hardness of the coating containing 4360 compared to coatings containing other resins having similar EEW values was anomalous. The relatively high hardness of the 4360 coating can be attributed the cyclohexylene group in its backbone. A cyclohexylene group would be expected to contribute more than an acyclic chain toward coating hardness.

The retort flexibility of the coatings increased with increasing resin EEW (Figure 3). The three factors used to explain lower coating hardness can be used to explain improved flexibility of the coatings containing epoxy resin that contained acyclic backbones.

Table 1. Ideal Chemical Structures of Epoxy Resins and Polyols and Some Properties

Key	Ideal Chemical Structure	Equivalent weight	Viscosity, mPa·s
UVR-6110		137 ^a	400 ^a
E278		163 ^b	285 ^b
E292		167 ^b	175 ^b
E306		175 ^b	155 ^b
E334		191 ^b	225 ^b
UVR-6128		200 ^a	650 ^a
E359		205 ^b	130 ^b
ERLX-4360		206 ^b	1,600 ^b
E391		220 ^b	180 ^b
E14CHD		^c	^d
E-t12CHD		^c	^c
Polyol 0301		100	^c
CHDM		72	^c
TCDM		98	^c

^a Typical values obtained from product literature. ^b Experimental values. ^c Properties were not measured. ^d Sample was a solid at room temperature.

The flexibility of the coating containing 4360 compared to coatings containing other resins having similar EEW values was anomalous. The flexibility test method involved exposing the coated TFS panels to steam (250 °F) for an hour after bending them. The improved crack resistance of the 4360 coating under these conditions may have been the result of better hydrolytic resistance. A cyclohexylene group may contribute more than an acyclic group toward hydrolytic resistance. Coatings containing polyester resins that were prepared using 1,4-CHDM and then cured using a melamine-formaldehyde crosslinker have been shown to have improved hydrolytic resistance.²

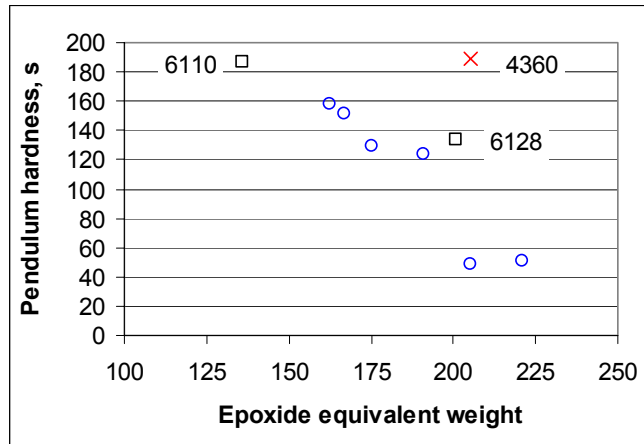


Figure 2. Coating Pendulum Hardness vs. Epoxide Equivalent Weight. The circles (o) signify data for the coatings containing the epoxides of the homologous series (E278, E292, E306, E334, E359, and E391).

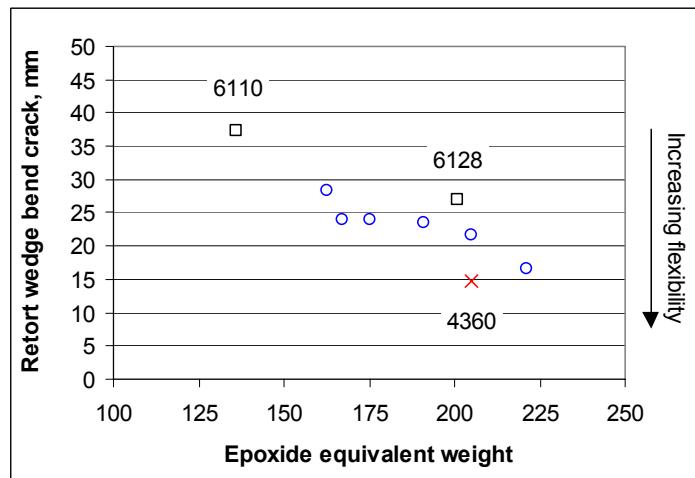


Figure 3. Coating Flexibility vs. Epoxide Equivalent Weight. See Figure 2 for key to symbols.

The correlation between the coating hardness (from Figure 2) and flexibility (from Figure 3) was plotted in Figure 4. Frequently high hardness and flexibility are desirable and mutually exclusive coating properties. The plot of hardness vs. flexibility showed that the coating containing 4360 had the best balance of both properties. Its hardness was similar to the coating containing 6110 and its flexibility under retort conditions was among the best.

Most of the coatings had brown discoloration after retort in the areas along the bend where the coatings were not cracked. The degree of coating discoloration after retort was subjectively ranked after visual inspection and the results are presented in Table 2. Brown discoloration was likely the result of corrosion of the iron in the TFS caused by water that migrated through the coatings during the retort. Discoloration during retort would be an unacceptable outcome for coatings intended for steel food container applications for example. The coating containing 4360 had the least amount of brown discoloration. This suggested the coating containing 4360 had the best water resistance under retort conditions.

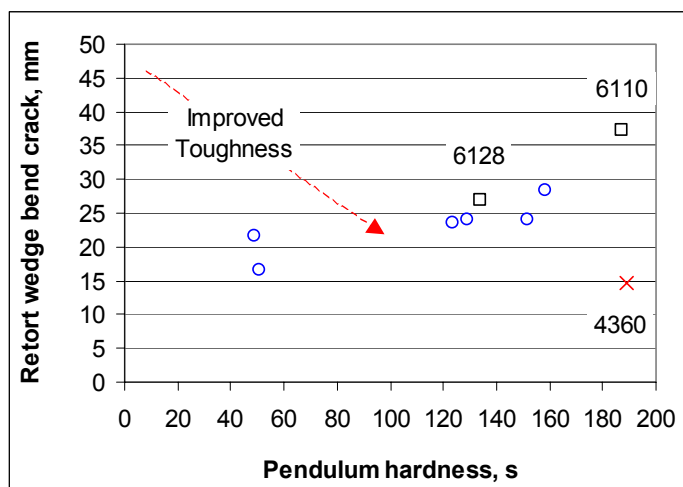


Figure 4. Hardness and Flexibility Properties Plotted to Highlight Coating Toughness. See Figure 2 for key to symbols.

Table 2. Relative Rating of Brown Discoloration Observed on Coated TFS Panels after Retort Processing

Epoxy Resin Used in the Coating	Discoloration Rating*
6110	3
E278	3
E292	3
E306	9
E334	3
6128	9
E359	9
4360	1
E391	3

*1=little or no discoloration; 3=moderate discoloration; 9=much discoloration

The model epoxides E14CHD and E-t12CHD were prepared to further test the effect of the presence of cyclohexylene groups in the epoxy backbone on coating properties. The coatings in Figure 5 contained each of the epoxy resins listed in the figure and 6110 50/50 w/w and no polyol. The retort wedge-bend and pendulum hardness properties of the coatings were plotted in Figure 5. The coatings in containing the epoxy resins containing a cyclohexylene group in the backbone, which included 4360, E14CHD, and E-t12CHD, had the best combination of hardness and flexibility.

The hardness values of the coatings in Figure 5 were similar. This was apparently a result of each of the coatings containing about 50% 6110. The physical properties of the coatings containing 6128 and E334 were very similar because the diepoxide ingredients had similar chemical structures and EEW.

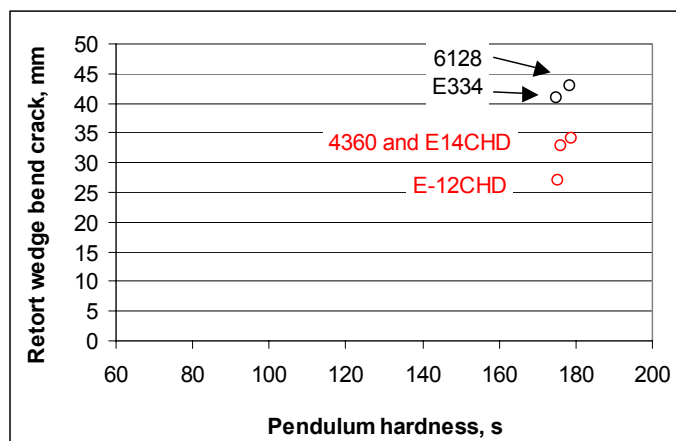


Figure 5. Comparison of Coatings Containing Epoxy Resins and No Polyol. The respective coatings contained the designated epoxy resins and 6110 blended 50/50 by weight, 4 wt% 6992, and 0.5 wt% surfactant.

The individual epoxy resins in Figure 5 with the exception of E14CHD were also blended with polyol 0301 and tested in coatings. The coating hardness and flexibility results are presented in Figure 6. The results confirmed that coatings containing epoxy resins containing cyclohexylene groups in the backbone were the toughest. The coating containing E-t12CHD blistered after retort however.

Analogous to the results in Figure 5, the physical properties of the coatings containing 6128 and E334 in Figure 6 were very similar because the diepoxide ingredients had similar chemical structures and EEW.

The coatings containing E-t12CHD, 4360, 6128, and E334 and polyol presented in Figure 6 were more flexible than the analogous coatings presented in Figure 5 that contained equal parts 6110 and no polyol. 6110 was harder and more brittle than polyol 0301.

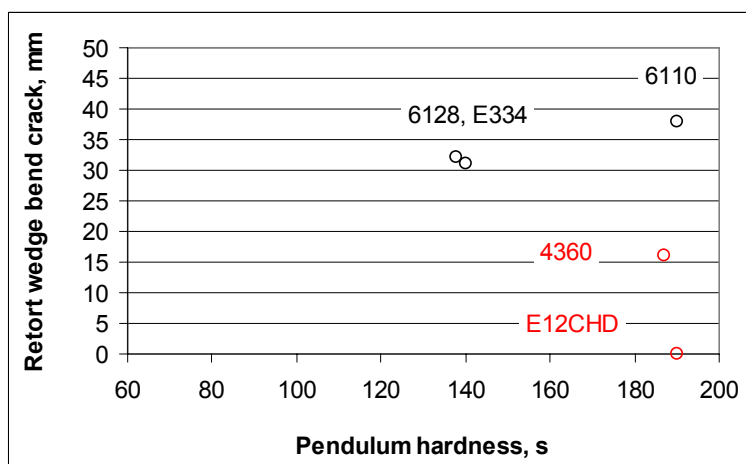


Figure 6. Comparison of Coatings Containing Polyol and the Designated Epoxy Resins. The coatings contained the designated epoxy resins, polyol 0301 (R -values = 2), 4 wt% 6992, and 0.5 wt% surfactant.

Plots of tan delta data for 6110 and 4360 from DMA are shown in Figure 7. The peaks at about 130 and 210 °C for 4360 and 6110, respectively, indicated the glass transition temperatures (T_g) of the respective homopolymers. There were peaks in the tan delta data also at about -60 °C for both samples. Low-temperature transitions are often associated with polymers that have good toughness near room temperature. The stronger the low-temperature transition, the tougher the polymer generally. The low-temperature transition for 4360 was almost twice as intense as the one for 6110, which suggested 4360 could be tougher than 6110. The DMA data appeared to agree with the coatings data in this regard.

The T_g of 6110 homopolymer was slightly greater than the temperature used to cure the samples used for DMA. The same results were obtained when 6110 was cured at higher temperature.

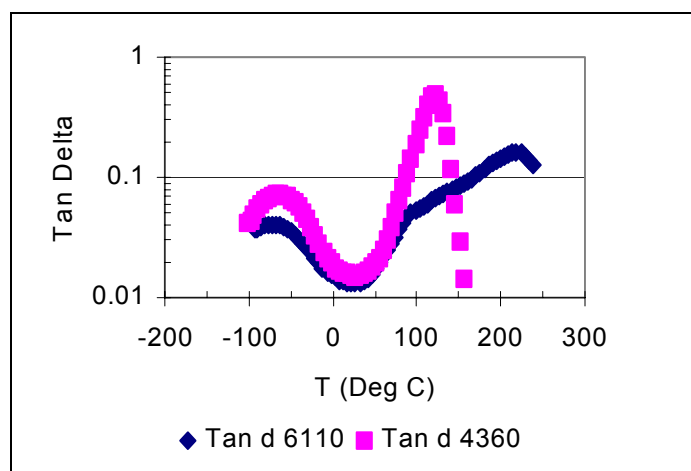


Figure 7. Tan delta data from DMA for cationic thermally cured homopolymers containing 6110 and 4360.

The storage and loss moduli of the cationic thermally cured 6110 homopolymer is shown in Figure 8. The relatively high storage modulus below 200 °C indicated the sample was relatively stiff in this temperature region. A high degree of stiffness would be expected for a highly crosslinked sample. The storage modulus of 6110 decreased near the sample T_g .

The storage and loss moduli of the cationic thermally cured 4360 homopolymer is shown in Figure 9. The storage modulus of 4360 homopolymer (2.66×10^9 Pa) was lower than the storage modulus of 6110 homopolymer (3.31×10^9 Pa) near 25 °C. The storage modulus of 4360 decreased near the sample T_g .

The surface-cure rates of coatings containing the new epoxides were lower than those of coatings containing the commercial epoxides 6110 and 6128 after correcting for differences in resin EEW (see Figure 10). The presence of mono-epoxy alcohol in the experimental epoxy resin samples would have resulted in coating R -values that were less than the R -values of the coatings containing 6110 and 6128. Lower coating R -values typically result in lower surface-cure rates at high relative humidity (>50%) because of the interaction between ambient humidity and polymer chains growing near the coating surface during cure.

The presence of sodium acetate in the resins could potentially behave as a base relative to the acid generated by the photoinitiator, which could slow the cure rate. An aliquot of an epoxy resin that had been prepared by transesterifying 4140 using sodium acetate catalyst was washed using water to remove the sodium acetate and then dried. Coatings containing the

washed and unwashed epoxy resin aliquots had the same surface-cure rates. This demonstrated that sodium acetate did not affect the cure rate under the test conditions.³

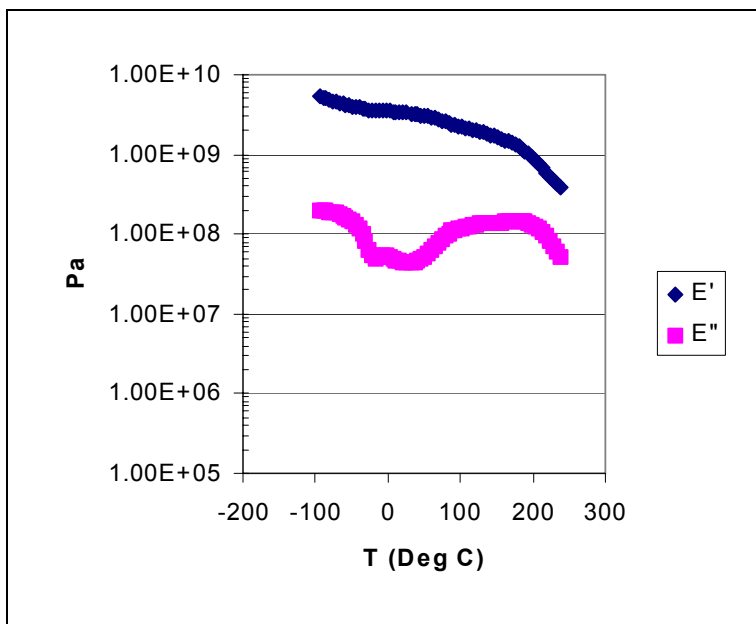


Figure 8. Storage (E') and loss (E'') moduli from DMA for a cationic thermally cured 6110 homopolymer.

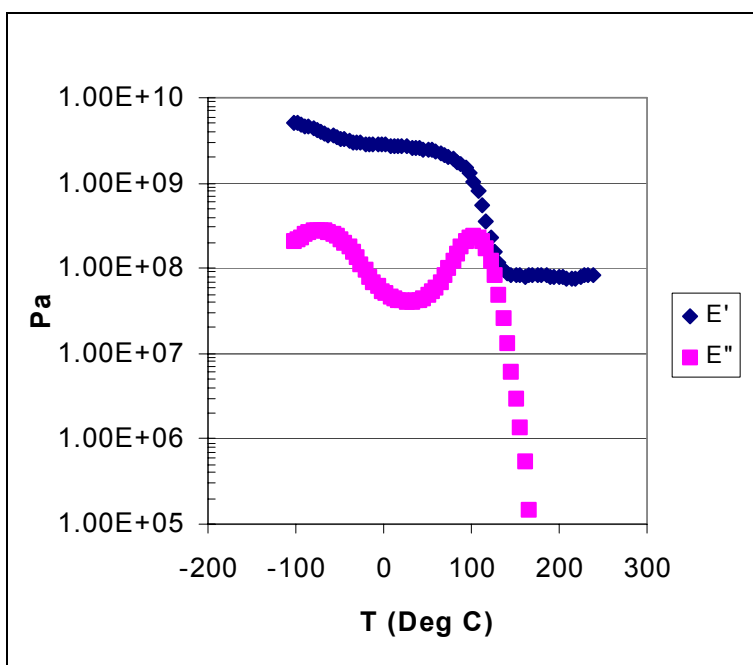


Figure 9. Storage (E') and loss (E'') moduli from DMA for a cationic thermally cured 4360 homopolymer.

Coatings containing 6110 and 4360 were tested for adhesion using different polyols. Polyol 0301 was used as a control. Liquid CHDM and TCDM were tested to determine the effects of

diols containing cyclic and tricyclic groups on adhesion. The coating formulations and the adhesion test results are shown in Tables 3 and 4 for 6110 and 4360, respectively. Each the coatings containing polyol in Tables 3 and 4 had *R*-values of 2 based on experimental EEW values.

Generally speaking, the adhesion of the coatings containing either epoxy resin decreased as follows: TCDM > CHDM > no polyol > polyol 0301. Of particular significance was the finding that a coating containing 6110 and TCDM had good adhesion to TFS after UV cure. TFS is a particularly difficult substrate regarding the adhesion of UV coatings.

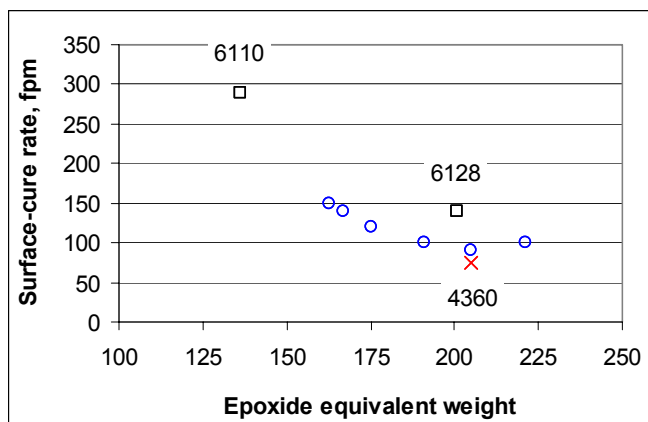


Figure 10. Coating Surface-cure Rate vs. Epoxide Equivalent Weight. See Figure 2 for key to symbols.

Cationic UV coatings containing 6110 and 11 other hydroxyl-containing materials were also tested for adhesion to TFS. The hydroxyl-containing materials were ethylene glycol, propylene glycol, 2-methyl-1,3-propanediol, diethylene glycol, dipropylene glycol, Voranol 234-630, Tone 0305, Desmophen 800, K-Flex XM-A308, K-Flex 148, Desmophen 670, and UVR-6000. All of these coatings had 0% adhesion to TFS after UV cure. Comparing these results with TCDM results further illustrate the unique adhesion benefit of TCDM.

In general, the coatings containing 4360 had better adhesion than the analogous ones containing 6110. The good adhesion of the coating containing 4360 and TCDM to TFS was interesting because coatings intended for food can ends require good toughness and adhesion. The toughness of 4360 under retort conditions was demonstrated (see Figure 4).

Each of the coatings in Table 3 and 4 wetted TFS panels well because they contained a silicone surfactant. Coatings containing epoxy resin, polyol, and 4% 6992 (and no surfactant) were prepared and their ability to wet TFS was compared because good wetting is important for good adhesion. The time between making the draw downs and curing the samples (10-s) provided equal opportunity for the coatings to de-wet the TFS. UV curing the samples essentially froze in place the wetting performance for easy comparison.

The wetting results in Table 5 were based on a visual comparison of the UV cured samples. The coating given the wetting rating of 1, the best rating, had a few spots and streaks that indicated de-wetting. The coatings rated 2 clearly had more spots and streaks of de-wetting than the coating rated 1. The coating rated 3 appeared to have about equal areas of wetting and de-wetting where the coating had been applied. The coatings rated 4, the worst rating, formed small beads like water on a waxed car.

Generally speaking, the wetting of TFS by the coatings containing either epoxy resin decreased as follows: TCDM > CHDM \geq polyol 0301. 4360 was clearly more effective than 6110 for wetting TFS.

Table 3. Adhesion results of coatings containing 6110^a

Ingredients	1	2	3	4
6110	95.5	69.7	75.5	70
polyol 0301	0	25.8	0	0
CHDM	0	0	20	0
TCDM	0	0	0	25.5
6992	4	4	4	4
L7604	0.5	0.5	0.5	0.5
Total	100	100	100	100
<i>R</i> -value ^b	NA ^c	2	2	1.9
Crosshatch adhesion results:				
Tin-free steel	0%	0%	10%	90%
Copper circuit board	0%	0%	70%	95%
Copper 110 alloy	100%	50%	100%	100%
Brass 260 alloy	100%	50%	100%	100%
Polycarbonate	NT	0%	100%	100%
Nylon 66 plaque	NT	0%	100%	100%

^aNT signifies the coating was not tested on the substrate indicated in the table. ^b*R*-value signifies the epoxy/hydroxyl equivalent ratio. ^cNA signifies *R*-value was not calculated because polyol was not added to the coating indicated.

Table 4. Adhesion results of coatings containing 4360^a

Ingredients	5	6	7	8
4360	95.5	64	80	76.7
polyol 0301	0	15.5	0	0
CHDM	0	0	14.6	0
TCDM	0	0	0	18.8
6992	4	4	4	4
L7604	0.5	0.5	0.5	0.5
Total	100	100	100	100
<i>R</i> -value ^b	NA ^c	2	1.9	1.9
Crosshatch adhesion results:				
Tin-free steel	0%	0%	40%	100%
Copper circuit board	NT	0%	100%	100%
Copper alloy 110	100%	50%	100%	100%
Brass alloy 260	100%	80%	100%	100%

^aNT signifies the coating was not tested on the substrate indicated in the table. ^b*R*-value signifies the epoxy/hydroxyl equivalent ratio. ^cNA signifies *R*-value is not calculated because polyol was not added to the coating indicated.

The coating containing 4360, TCDM, and no surfactant had the best wetting (Table 5) and it had the best adhesion when it contained surfactant (Table 4). The coating containing 6110, polyol 0301, and no surfactant had among the worst wetting (Table 5) and its adhesion to various substrates was among the worst when it contained surfactant (Table 3). In these cases, good and poor wetting correlated with good and poor adhesion, respectively.

Table 5. Results of TFS Wetting Experiments^a

Epoxy Resin	4360	4360	4360	6110	6110	6110
Polyol	TCDM	CHDM	Polyol 0301	TCDM	CHDM	Polyol 0301
Wetting Rating ^b	1	2	2	3	4	4

^a The coatings contained the indicated epoxy resins and polyols, 4% 6992, and no surfactant.

^b 1=best wetting and 4=worst wetting of TFS; see text for more details.

Good wetting and adhesion were not always present together however. Each of the coatings containing surfactant in Tables 3 and 4 had good wetting of TFS but some had poor adhesion to TFS. Conversely, the coating containing 6110 and TCDM had relatively poor wetting of TFS when it did not contain surfactant (Table 5) but it had good adhesion to TFS (90%) when it contained surfactant (Table 3).

The presence of cyclic (e.g. 4360 and CHDM) or tricyclic groups (e.g. TCDM) in the backbone of the epoxy resin or the polyol or both was generally associated with improved wetting and adhesion properties. There were however differences in the degree of the effect on wetting and adhesion depending on whether the epoxy resin or the polyol contained the cyclic or tricyclic group.

Coatings containing 4360 had much better wetting than the 6110 coatings. However, the difference in wetting among the samples containing the same epoxy resin and different polyols were not so great. Taken together, these comparisons indicated that the epoxy resin had more affect than the polyol on the wetting results in these tests.

The coatings containing TCDM and CHDM had some adhesion to TFS regardless of the epoxy resin. However, the coating containing 4360 and polyol 0301 in Tables 3 and 4 had no adhesion to TFS in spite of the presence of a cyclohexylene group in the epoxy resin. It seemed the presence of a cyclic or tricyclic group in the polyol was especially important for good adhesion.

Summary

New cycloaliphatic epoxides, including two model compounds, were found to have improved flexibility and hardness properties in cationic UV coatings. The improved hardness was attributed to the presence of cyclohexylene groups in the epoxy resins. The improved flexibility under retort conditions was attributed to a combination of the presence of the cyclohexylene groups in the epoxy resins and resin molecular weight.

Diols containing cyclic and tricyclic groups (CHDM and TCMD, respectively) were found to improve the wetting and adhesion properties of cationic UV coatings. The presence of a cyclic group in one of the new epoxy resins (4360) was also associated with improved wetting and adhesion. The relatively hydrophobic nature of the cyclic groups may be at least partly responsible for the improved wetting and adhesion properties.

Acknowledgement

The authors would like to thank Robert F. Eaton for helpful discussions concerning the DMA and wetting results and for sponsoring the DMA experiments.

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