Enhancing the performance of aromatic PU sealant prepolymers using oxazolidine latent hardeners
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case study
Introduction

Whilst there have been a number of technical advances in the resins used in construction sealants, polyurethane chemistry continues to offer the best balance of cost and performance. The chemistry is highly versatile, with aromatic and aliphatic prepolymer offering the formulator a range of properties to meet differing performance requirements.

The main advantage PU offers over other technologies are:

- better mechanical strength
- no toxic by products from the cure mechanism compared to the production of methanol from silane cured systems
- better substrate adhesion
- ease of paintability

The traditional method of curing PU sealants is via the reaction of the terminal isocyanate groups of the polyurethane binder with moisture. This requires a high level of isocyanate content to afford an effective cure which leads to toxicity issues with residual isocyanate monomer. Another problem is the side product, carbon dioxide, produced with this reaction which results in pinholing defects that can then compromise the integrity and aesthetics of the finished coating.

An effective way to eliminate gassing and improve the cure performance of the sealant is to introduce an oxazolidine latent hardener such as Incozol LV or Incozol CF. Using such a hardener type allows the formulator to combine the benefits of the oxazolidine hardener with a prepolymer of much reduced isocyanate content. Typically, the isocyanate level can be reduced by as much as 10-15%.

The benefits of introducing oxazolidine latent hardeners into aromatic PU sealants are:

- Eliminate gassing
- Excellent through cure in thick film applications (compared to moisture cure/aldimine systems)
- Retain good workability of the sealant (skinning control)
- Excellent early Shore hardness development through hardener crosslinking
- At recommended levels of addition, there is no significant impact on mechanical strength or shrinkage through crosslinking
- No detrimental impact on UV resistance
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Study Results

All test data was acquired using the prepolymer with oxazolidine only; no fillers or additives were included in the mixes.

Dealing with moisture ingress

It is vitally important when handling PU prepolymer mixes with oxazolidine latent hardeners that moisture ingress is minimised. Good practice recommends nitrogen purging of all mixing reactors and minimum exposure to atmospheric moisture during handling.

A. Oxazolidines eliminate gassing

Traditionally, PU sealants are cured by the reaction of terminal isocyanate groups with moisture. A side product of this reaction is the generation of carbon dioxide which remains trapped in the cured sealant system. The pictures below illustrate cured PU prepolymer (MDI: Desmoseal M280 & TDI: Desmodur E15) used in sealants mixed with oxazolidine latent hardener (typically 2% w/w) in comparison to the same prepolymer cured solely with moisture.

MDI prepolymer Desmoseal M280 cured without oxazolidine (left), cured with 2% Incozol LV (centre) and cured with 2% Incozol CF (right)

TDI prepolymer Desmodur E15 cured without oxazolidine (left) and cured with 2% Incozol LV (right)
B. Through Cure / Hardness Development

The addition of oxazolidine latent hardener not only eliminates sealant defects created by gassing, but also promotes faster through cure than by moisture alone. This reflects the ability of oxazolidine latent hardeners to hydrolyse quickly and crosslink the PU prepolymer in preference to the water and isocyanate reaction.

The above figures of hardness development show faster cure when oxazolidine latent hardeners are incorporated into the systems. This results in increased concentration of reactive groups.
C. Workability: Effect of the addition of oxazolidine latent hardener on mixing with a PU prepolymer

The addition of oxazolidine latent hardener does not have a significant impact on PU prepolymer viscosity and consequently, does not affect the workability of the sealant. The initial viscosities of the oxazolidine/prepolymer blends shown in the figures below, indicate the influence of the latent hardeners on the systems. Incozol LV either reduces or retains the viscosity whilst Incozol CF increases it slightly. These differences offer options for formulators to retain the desired workability of the system.

**Initial viscosity of oxazolidine/Desmoseal M280 systems**

![Graph showing initial viscosity of oxazolidine/Desmoseal M280 systems.]

**Initial viscosity of oxazolidine/Desmodur E15 systems**

![Graph showing initial viscosity of oxazolidine/Desmodur E15 systems.]

D. Impact on storage stability

The change in viscosity was measured for unopened containers of PU prepolymer (MDI & TDI) mixes with different oxazolidine latent hardeners. Containers were sealed and stored at ambient temperature (23°C).

As expected, TDI based mixes are more stable than MDI systems due to the increased reactivity of MDI prepolymer systems. Mixing with a latent hardener shows an initial viscosity rise where moisture ingress from the mixing/handling is consumed. Excellent stability follows with little viscosity rise whilst the containers are sealed from the atmosphere.

With MDI mixes, the stability is reduced compared to TDI. The data reflects the inclusion of 2% oxazolidine in Desmoseal M280 prepolymer mixes; dilution of the binder and curing agent in fully formulated systems with dry, inert fillers affords improved stability.
E. Impact on tensile strength

The addition of oxazolidine latent hardeners does not significantly alter physical properties such as hardness and tensile strength in the cured PU system. Crosslinking at low levels does not detrimentally alter sealant performance.

In general, Incozol CF slightly increases the hardness and tensile strength of the PU prepolymer whilst Incozol LV slightly reduces the hardness and tensile strength. This is largely due to their respective backbone natures.
Shore A hardness of oxazolidine/Desmodur E15 systems

Tensile properties of oxazolidine/Desmodur E15 systems
Conclusion

The data generated from the study demonstrates the advantages of introducing oxazolidine latent hardeners into aromatic PU sealant formulations.

Firstly, the ability of Incozol latent hardeners to act as moisture scavengers ensures that the reaction between any moisture present and the isocyanate is prevented, thereby preventing the generation of carbon dioxide gas which would result in sealant defects.

Secondly, the addition of an oxazolidine latent hardener into the formulation will promote faster through cure than that achieved by moisture alone. This is due to the ability of the latent hardener to hydrolyse quickly and crosslink the PU prepolymer in preference to the water and isocyanate reaction.

Thirdly, introducing an oxazolidine latent hardener allows the formulator to use a prepolymer with reduced isocyanate content. Typically, the isocyanate level can be reduced by as much as 10-15%.

Oxazolidine latent hardeners, such as Incozol LV and Incozol CF, bring added benefits to the PU sealant formulation but they do this without affecting the physical properties of the system. The data indicates that there is no significant detrimental impact on the workability of the sealant, the tensile strength and the elongation.
Manufacturers of specialist resins & polymers:

- Oxazolidines
- Waterborne epoxy curing agents
- Polyurethane dispersions
- Polyurethane/acrylic hybrid dispersions
- Polyurethane prepolymer