Technical Paper

EASTMAN FEICA Conference - September 2015
High performance non-phthalate solutions for polysulfide insulating glazing sealants
High performance non-phthalate solutions for polysulfide insulating glazing sealants

Dr Martin Stimpson, Ms Simone Wubbe and Mrs Lorna Williams
FEICA Conference: Vilamoura, Portugal
September 10th 2015
Agenda

- Eastman at a glance
- Introduction to the European sealants market
- Regulatory landscape
- Non-phthalate options in the market
- Introducing Eastman non-phthalate options for sealants
- Eastman VersaBond™ plasticizer for polysulfide sealants
- Summary
Eastman at a Glance

- A global specialty chemical company headquartered in Kingsport, Tennessee
- Approximately 14,000 employees and over 40 manufacturing sites globally
- Serving customers in approximately 100 countries
- 2014 revenue of $9.5 billion
- Eastman first started making plasticisers in 1950
- Eastman is integrated with oxo capability in both NA and Singapore
- Eastman is committed to the global plasticizer market
- Through capacity expansions and 3 acquisitions globally since 2011
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Where are sealants used in Europe?

- 676 KMT demand
- €3 Billion value
- Sealants represented 16% of the volume and 25% of the value of the entire European Adhesives & Sealants market in 2012
- Sealants demand predicted to grow more strongly than the adhesives demand

Source: FEICA – The European Adhesives and Sealants market 2012 -2015, 2012 sealant demand
Polymer resin demand for sealants

- Broad range of polymer types used
- Choice of polymer may be determined by specific end application
- Some polymer types compete

Source: FEICA – The European Adhesives and Sealants market 2012 -2015, 2012 sealant demand
End markets for European sealants

- Four main end-use segments
- Some applications are very cost sensitive
- Some applications are very technically demanding
- Construction applications dominate in terms of sealant volume

Source: FEICA – The European Adhesives and Sealants market 2012-2015, 2012 sealant demand
European construction market—what’s significant about it?

- The construction sector accounts for a significant proportion of sealant end uses.
- Silicone sealants remain the most significant polymer type.
- There are many other polymer types in the construction sector.
- Significant volumes of polyurethane, polysulfide, SMP, and acrylic plasticized sealants are produced.

Source: FEICA – The European Adhesives and Sealants market 2012-2015, 2012 sealant demand
What sealant types can be plasticized?

- 40% of the polymer types used in sealant manufacturing are plasticized
- Resin choice is split fairly evenly
- Complex plasticizer choice

Source: FEICA – The European Adhesives and Sealants market 2012 -2015, 2012 sealant demand
Why plasticize?

Plasticizers are added to commercial sealants for a variety of reasons.

- Modification of the $T_g$
- Viscosity control/increased dispensability/mixing
- Cost
- Improved elasticity
- Improved movement accommodation
- Reduced modulus
- As a carrier for other additives
- Improved resiliency
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History of substituted plasticizers in sealant applications

- There have been a number of plasticizer types used in sealant applications over the last 30 years.
- Some of these have been subject to de-selection.
  - Various ortho-phthalate plasticizers
  - Chlorinated paraffin
REACH Update

- REACH: Registration, Evaluation, Authorization of CHemicals in the EU
- REACH currently has 163 Chemicals identified as SVHC candidates (including 13 phthalates).
  - The next step is to “promote” verified SVHCs as Candidates for Authorization under Annex XIV of REACH (14 on the authorization list so far).
  - Roadmap shows ~440 total SVHC candidates proposed by 2020.
- The “Sunset” date for authorization was February 2015 for four plasticizers (DEHP, DBP, BBP, DBP). After this date, non-authorized uses are banned.
  - Some authorizations for DEHP and DBP were adopted in 2014.
  - The two reviewing committees have recommended authorizations for all of the remaining applications, for a period of 3-4 years.
  - Awaiting European Commission action any time.
### Ortho-Phthalate Esters: Alkyl Group Carbon Number

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<th>C3</th>
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**SVHC** – Substance of Very High Concern

**CoRAP** – COmmunity Rolling Action Plan
California Proposition 65 – DINP listing

- OEHHA agreed in early-December 2013 that there was enough evidence to list DINP as a chemical known to the State of California to cause cancer.
  - Listed 20 December 2013, with one year grace period for labeling.
  - Manufacturers of items had until the end of 2014 to replace DINP in their products for sale in California, **label those products with a Prop 65 Warning label**, or prove that there is no exposure to DINP in their products.
  - OEHHA has proposed a No Significant Risk Level (NSRL) of 146 µg/day, 18x lower than ACC’s proposal of 2664 µg/day.

Examples of Prop 65 labels
California Prop 65 bounty hunters

- Most cases are settled out of court, mainly to avoid the costs associated with litigation (>\$150k).
- The State of California receives 75% of the fines, but the bounty hunters get to claim for their time and testing costs.
- Average settlement costs are ~\$50,000
  - Bounty hunters receive an average of 73% of the settlement money.
- Since Prop 65 arrived in 1986*
  - Between 2000 and 2011 there were 2,381 settlements costing \$178,699,905, exclusive of plaintiffs’ legal costs or court costs.
- 149 Notices of Violation served on DINP products in first 8 months out of 800 notices filed.

*Source: California’s “Prop 65” and US regulatory updates – Nigel Sarginsen Exxon-Mobil presentation at Plasticisers Conference 2014, Brussels
California Prop 65 – 60 day notice of violation

60-DAY NOTICE OF VIOLATION
SENT IN COMPLIANCE WITH CALIFORNIA HEALTH & SAFETY CODE § 25249.6(e)

DATE: January 30, 2015

TO: Thomas J. Falk, Chief Executive Officer – Kimberly-Clark Corporation
   California Attorney General’s Office;
   District Attorney’s Office for 58 Counties; and
   City Attorneys for San Francisco, San Diego, San Jose, Sacramento and Los Angeles

FROM: Anthony E. Held, Ph.D., P.E.

1. INTRODUCTION

My name is Anthony E. Held. I hold a Doctor of Philosophy degree in Environmental Engineering and I am a registered professional engineer in the State of California. I am a citizen of the State of California acting in the interest of the general public. I seek to promote awareness of exposures to toxic chemicals in products sold in California and, if possible, to improve human health by reducing hazardous substances contained in such items. This Notice is provided to the public agencies listed above pursuant to California Health & Safety Code § 25249.6 et seq. (“Proposition 65”). As noted above, notice is also being provided to the alleged violator, Kimberly-Clark Corporation (the “Violator”). The violations covered by this Notice consist of the product exposures, routes of exposure, and type of harm potentially resulting from exposure to the toxic chemical (“listed chemical”) identified below, as follows:

- Product Exposure: See Section VII. Exhibit A
- Listed Chemical: Diisononyl phthalate (“DINP”)
- Routes of Exposure: Ingestion, Dermal
- Types of Harm: Cancer
Excise duty on PVC floor, wall and ceiling coverings

The proposed excise duty on PVC floor, wall and ceiling coverings has been designed to make goods made of PVC that constitute a large part of the surface layer in a dwelling subject to excise duty. Such goods represent a high risk of exposure to hazardous substances in people’s everyday environment. The goods subject to excise duty are defined using the tariff classification in the European Community’s Combined Nomenclature (CN codes).

The excise duty will amount to SEK 10 per kilogram of excisable goods. A deduction of 50 per cent of the excise duty will be allowed for goods that do not contain phthalates included in the candidate list\(^1\). A deduction of 75 per cent of the excise duty will be allowed for goods that do not contain phthalates included in the candidate list, or phthalates that are restricted in toys under the REACH Regulation\(^2\). Since all goods falling within the scope of the excise duty contain more or less hazardous chemicals, it will not be possible to deduct the full tax.

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\(^2\) REACH Regulation.
Retailer initiatives

- Zero discharge of hazardous chemicals
- Looking to develop a Manufacturing Restricted Substance List (MRSL)
- Targeting apparel and footwear products
- MRSL list includes
  - Cleaners and solvents
  - Adhesives
  - Inks, dyes and pigments
  - Waste water treatment
  - Metals
  - Plasticisers
### Ortho-phthalate esters: alkyl group carbon number

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### MRSL instructions

**Group A: Raw material and finished product supplier guidance**
- **No Intentional use**

**Group B: Chemical supplier commercial formulation link**
- **Sum of all phthalates = 250ppm**
Retailer initiatives

Home Depot To Stop Selling Vinyl Flooring with Ortho-Phthalates

Atlanta, GA, April 24, 2015—Facing pressure from consumer groups, Home Depot says it will discontinue use of a potentially harmful chemical in its vinyl flooring by the end of the year, reports the New York Times.

The retailer is asking about a half-dozen suppliers to phase out their use of ortho-phthalates, which will affect about 15% of the company’s vinyl products, according to Stephen Holmes, a spokesman for Home Depot.

Ortho-phthalates are used as plasticizers, meaning that they can make products more flexible. Consumer safety advocates say there is a growing body of evidence linking some phthalates to reproductive and developmental problems, particularly in male babies.
Regulatory issues with chlorinated paraffin

- Chlorinated paraffins are grouped depending on chain length (SCCP C10-13, MCCP C14-17, LCCP C18-30).
- Global focus is questioning the toxicity of these plasticizers.
- Europe has already implemented some restrictions.
- SCCPs are persistent, bioaccumulative, and toxic to aquatic organisms at low concentrations.
- Moving to MCCP or even LCCP may only be a short-term measure.
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There are numerous non-phthalate plasticizers.

But only two non-phthalate alternatives for polysulfide:

- Abietic acid esters
- Adipates
- Azelates
- Citrates
- Chloroparaffins
- Epoxidized Esters
- Glycolates
- Hydrocarbon oils
- Isobutyric acid esters
- Oleates
- Palmitates
- Phosphates
- Polyesters Ricinoleic acid esters
- Sebacates
- Stearates
- Sucrose esters
- Sulfonamides
- Trimellitates

Di-benzoates
Chlorinated Paraffin
What are the non-phthalate options for polysulfide sealants?

- Chlorinated paraffin is a non-phthalate plasticizer but is also subject to de-selection in Western Europe due to eco-toxicity and human toxicity concerns.

- Therefore the only remaining non-phthalate plasticizer for polysulfide sealants are di-benzoate esters.
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Use of plasticizer in sealants

- What is the function of the plasticizer?
- What are the performance requirements?
  - Permanency
  - Compatibility with other materials
  - Formulation stability
  - Reaction/cure time
  - Long-term chemical stability
- What are the final product technical requirements?
  - Performance of the cured/dried product
  - Rheology of the uncured/wet product
  - Mechanical performance
  - Durability
  - Aging
Eastman non-phthalate options for Insulating Glazing Sealants

- No single choice for the best non-phthalate plasticizer
- Benzoate esters have been the main non-phthalate choice for polysulfide since 2001.
- Newly introduced Eastman VersaBond™ plasticizer best for drop-in alternative to ortho-phthalate plasticizers.

<table>
<thead>
<tr>
<th>Polysulfide sealants</th>
<th>Benzoflex™ 50 plasticizer</th>
<th>Benzoflex™ 2088 plasticizer</th>
<th>Benzoflex™ 9-88 plasticizer</th>
<th>Benzoflex™ 9-88SG plasticizer</th>
<th>Eastman Effusion™ plasticizer</th>
<th>Eastman TXIB™ formulation additive</th>
<th>Eastman VersaBond™ plasticizer</th>
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<td>O</td>
<td>Recommended</td>
<td>Suitable in some formulations</td>
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- Recommended
- Suitable in some formulations
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Polysulfide sealants—why plasticize?

- In two-part polysulfide sealants, plasticizers can be utilized in both sides of the formulation.
- Plasticizers allow higher extender loadings (fillers), reduced cost
- Polymer side (A), the plasticizer is used to soften
- On the catalyst side (B), the plasticizer can be used as the dispersion media
- We are talking today about the Part A plasticizer.
Polysulfide sealants—status today

- Large market still dominated by benzyl phthalate plasticizers.
- Reformulation to use Benzoflex in 2001 enabled some manufacturers to replace traditional benzyl phthalate plasticizers.
- A large part of the market however remained on traditional benzyl phthalate plasticizer to avoid extensive reformulation and recertification.
- Eastman VersaBond™ plasticizer was developed as a drop-in replacement for the remaining benzyl phthalate plasticizers.
Market size - Insulating glazing sealants (IG)

- Total market size for all IG sealants is 200KMT.
- Market for polysulfide IG sealants is 130 KMT formulated sealant ~18 KMT plasticizer.
- We are focused on polysulfide sealants using various benzyl phthalate plasticizers ~70 KMT sealant or ~10 KMT plasticizer.

Source: Analysis and assumptions derived from Toray/Akzo Nobel analysis combined with data from IHS Adhesives & Sealants report 2012, FEICA CHEM Research study 2012-2015 & CEH Polysulfide review 2012 with Eastman Customer analysis from sales calls in 2013 & 2014. Derived the entire market and deducted what we knew we sold and what we believed competitors sold. Had some double counting, but the numbers were calculated on the basis of our estimate of the total market, and what we sold Ferro for Santicizer 278, with estimates of how much of that was PS sealant. Numbers. Also used data in The Global Sealants Market By Michael Growney, April 1, 2013 from PCI extract from The Global Sealants Industry, a new study by Steven Nerfl, MineshKusumgar and Michael Growney published on 2012 & “Overview of the European Sealants Market” prepared by IAL Consultants published by Market Publishers Ltd. July 2013 (only had extract as we did not purchase the full market study) & “Requirements on secondary insulating glass sealants” Dr. Dieter Lange, AkzoNobel, Functional Chemicals, Greiz/Germany as a check step for our numbers and assumptions.
Technology requirements

- Small primary* seal - polyisobutylene
- Big secondary seal - 2 K polysulfide sealant
  - Good moisture resistance
  - Resistance to noble gas diffusion
  - Self-healing - cohesive failure due to movement
  - Fast adhesion
  - Backup barrier for primary seal

*Primary = first applied
Eastman VersaBond™ plasticizer
A new tailored solution for polysulfide sealants

- Commercially available in Europe and Canada.

- Non-phthalate plasticizer that complies with existing and known pending regulatory initiatives for plasticizers.

- Represents a direct substitution to industry standard, benzyl phthalate plasticizer, that requires little to no reformulation – saving months of development, approval, and changeover time.

- Outperforms other non-phthalate options.
## Typical formulation

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<tr>
<td>Plasticizer 1</td>
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<td>Hydrocarbon resin</td>
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<table>
<thead>
<tr>
<th>Component B</th>
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<tr>
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### Mixing ratio

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100,0
Increased compatibility

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<td>Benzoflex 9-88</td>
<td>1.97</td>
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A lower RED number means that the plasticizer has more affinity with the polymer

Calculation done with HSPiP version 3.1.26
Improved workability

- Sealants formulated with VersaBond have a long pot life.

- The tack free time is short which enables faster handling.
VersaBond exhibits a similar rheological profile compared to the industry standard, benzyl phthalate plasticizer, enabling drop-in replacement of traditional plasticizers.
Improved cure performance

- Sealants plasticized with VersaBond exhibit rapid development of hardness.
- Early movement of fabricated IG units is “safer” with faster development of cure.
Sealant adhesion to glass can be improved.

- After 24 hours, VersaBond formulation shows very good adhesion to glass compared to other plasticizers.
- Faster adhesion can reduce the risk of problems during transportation of IG units compared to other technologies.
- This trend continues after 1-week cure without loss in elongation at failure.
Stability after cure

- Cured polysulfide sealant formulations plasticized with VersaBond showed no degradation after UVB exposure and water immersion.

- Water uptake of the sealant plasticized with VersaBond was measurably lower.
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- Sealant formulations can be complex.
- Regulatory changes are forcing formulators to rethink their use of traditional benzyl phthalate plasticizers in polysulfide sealants.
- **Polysulfide sealants still** using traditional benzyl phthalate plasticizers can simply be manufactured using Eastman VersaBond™ plasticizer as a drop-in replacement.
- **Polysulfide sealants** can be manufactured using the non-phthalate Benzoflex plasticizers with reformulation.
THANK YOU!

Acknowledgements:
R. van Rossum, C. Hermans, B. Stolte, Eastman Chemical – The Netherlands
S. Boito, Eastman Chemical - USA
See you soon on our websites

www.specialchem.com