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Natural Foams Technology to produce, safer and more environmentally acceptable, flexible PU foams

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Natural Foams Technology to produce, safer and more environmentally acceptable, flexible PU foams

Reporting in the US media has created a surge in consumer concerns over the use and composition of foams used in household products. There are concerns over the odour of new furniture and mattresses as well as the use of flame retardant chemicals in gym mats, play mats, cot mattresses, and all residential furniture and mattresses into which children may come into contact. There is proposed legislation to restrict the sale of children's products containing halogenated flame retardants in the states of California, Connecticut, Maine, Maryland, Massachusetts, New Jersey, Vermont, and Washington. In addition, the state of California is now considering a law to label all PU foam based products as "Contains added FRs" or "No added FRs". The proposed label includes a rider that warns consumers that FRs can migrate into the air and dust where people can become exposed to them. This paper sets out to fully address the issue of migrating materials and build on the unique chemistry developed by Natural Foams Technology (under the Green Urethanes license), using polyols derived from natural oils. Advancements in the chemistry have produced a series of foams that contain 50 wt% bio-renewable materials whose inherent chemistry also results in class-leading low emissions and a new low level of smoke generation.

1. Background

Since 1975, California TB 117 (Cal 117, Cal 117-2013, Cal TB 117-1975, Cal 117-1975) foams have traditionally been made flame resistant by adding large amounts of liquid, non-reactive flame retardants to the chemicals used to make flexible foams. Tests conducted in the USA have concluded that that these non-reactive flame retardants gradually migrate from foam into the living environment where they can impact people's health and the environment.

In response, Californian legislators have introduced a new protocol, California Technical Bulletin 117-2013, which eliminates the need to use flame retardants of any type, by no longer requiring foams to resist an open flame ignition. When the new standard comes into force in January 2015, foams will only be required to pass the smoulder ignition part of the original test. It is hoped that this will eliminate the future use of chemicals that could migrate into the environment. It also means the majority of foams consumed in the US market will easily pass Cal TB 117-2013 without any disruption to the manufacturing process.

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For reasons of customer safety and duty of care responsibilities, it is believed that the polyurethane industry would be in a stronger position with the means to make safe foam materials resistant to cigarette smoulder and open flame ignition sources.

This paper will distinguish between the new and old versions of the California flammability protocol by calling the old test Cal 117-1975 (which includes open flame and cigarette smoulder test criteria) and the new test Cal 117-2013 (cigarette smoulder test criteria only).

The goal of this paper is to demonstrate that a solution exists to this problem which can satisfy environmental concerns while remaining price competitive to pass the more stringent Cal TB 117-1975; and offers a path to reducing the environmental impact across standard foams made outside the Cal TB 117 test criteria.

The paper also highlights the important role of using bio-based polyols and the developments made in recent months by Natural Foams, with 82 % petrochemical polyol being replaced with a natural oil polyol (NOP) based on palm oil sourced from certified plantations. This polyol is a cost competitive replacement to petrochemical materials, with the added advantage of bio-renewability, coupled with excellent physical and performance properties. Several new foams detailed below set new performance standards – notably smoke-generation reductions of up to 80 % compared to all existing Cal 117 compliant technologies.

2. Green Urethanes and Natural Foams Technology

A previous paper [1] from Green Urethanes (GU) demonstrated that the key to eliminating foam emissions is to use reactive instead of non-reactive flame retardants. Trial work was based on using NOPs to produce foam with a NOP replacement value of 55 %. By combining the green chemistry [5, 6] with a reactive flame retardant it was proven possible to produce a wide range of foams that pass Cal 117-1975. The non-migratory nature of the flame retardant was proven by independent laboratory tests. Further, minor changes to the formulation [7, 8] resulted in foams with very low emission properties when measured against the stringent requirements of the CertiPUR-US test and Daimler Chrysler's searching VDA 278 test.

This paper takes three further steps in this development process:

1. Production of a Cal TB 117-1975 foam, where 82 % of the petrochemical polyol is replaced by an NOP
2. Emissions testing of the foam against both VDA 278 VOC and FOG protocols
3. Measurement of heat and smoke emissions from the foam using a cone calorimeter.

Natural Foams' technology (under the Green Urethanes license) is already producing commercial volumes of bio-renewable foams that meet the physical and hardness properties in the popular density range of 1.25–2.0 pcf (20–32 kg/m³). Increasing the NOP content to 82 % of the total polyol component demonstrates that 50 wt% of the foam is bio-renewable through certification by the BioPreferred programme run by the United States Department of Agriculture (USDA).

Additionally, a very high NOP content has been found to give the foam an inherent flame resistant boost. Natural Foams Technology is continuing to explore this new development, coupled with what has already been established [1]: that the amount of reactive flame retardant needed to pass the Cal 117-1975 open flame test is routinely about 80 % less than the amount of non-reactive (emissive) flame retardant that was formerly used.

3. A new class of polyol with unique characteristics

During continuing trials, Natural Foams Technology has produced a new class of polyols for making rigid and flexible foams. These new polyols, whether based on palm, soy, rapeseed or animal oils, are more hydrophobic than standard petrochemical polyols and, as a result, have different solubility and reactivity characteristics to standard polyols. Research has also established that these particular bio-renewable foams produce better humid ageing properties and SAG (support) factors, in addition to the enhanced flammability aspects further outlined below.

Although reactive flame retardants have been on the market for many years, they have proved difficult to work with because of their high reactivity, which tightens the foam, making it unfit for use. Lowering the tin or gelation catalyst seems a logical next step, but foamers have found that even removing all the gelation catalyst from the formula still does not deliver a decent quality, open-cell foam. Using a specialty isocyanate such as toluene diisocyanate (TDI) 65/35 is one alternative, but the cost and availability of the material makes it a difficult choice. Also, foams made using high-water content formulations will still shrink, even when using TDI 65/35.

Previous work in 2013 [1] confirmed that foam emissions could be greatly reduced by using low VOC reactive material such as Degussa's Kosmos EF [8], standard commercially available low emission amine [7], low VOC flame retardant silicones in combination with a reactive flame retardant additive Exolit OP 560 from Clariant Corporation. This is a reactive polyol with a hydroxyl value of around 450, containing a phosphorous group and is halogen-free. Exolit has been available in the North American market for a while, receiving a Program P2 Recognition Project Award in 2009 from the US Environmental Protection Agency. These changes allowed a foam with 55 % of the polyol to be replaced by an NOP to pass Cal TB 117-1975 as well as the VOC part of the Daimler Chrysler VDA 278 emissions test. However, they failed the FOG part of the Daimler test due to the emissions characteristics of the antioxidant package used in the NOP.

Tab. 1: Results of emission tests for standard foams with and without non-reactive flame retardants compared to a foam made with 55 % of the petrochemical polyol replaced by a palm-oil-based NOP against CertiPUR-US specifications of TVOC

		Foam 1	Foam 2	Foam 3
CertiPUR-US results	**Specification**	**Standard**	**Standard**	**New**
	Standard limit	Non Cal 117	Cal TB 117 *	Green Cal 117*
	mg/m³			55 php of NOP
TVOC emissions (base rate)	0.5	0.37	0.434	0.213
Total allowable TVOC emissions	0.8	0.474	0.456	0.255
Exolit OP 560 present	<0.001	Nil by test	Nil by test	Nil by test

*Cal 117-1975
TVOC: CertiPUR-US Total Allowable Volatile Organic Compounds
Foam 1: standard polyether polyol flexible foam without a reactive FR; Foam 2: standard polyether polyol flexible foam with a reactive FR; Foam 3: Green Urethane foam with Exolit OP560

4. Experimental results

4.1 Emission tests

Results were provided by UK-based Hall Analytical Limited, a laboratory certified to carry out CertiPUR-US tests.

Table 1 shows that the total allowable (TVOC) emissions are drastically reduced in the bio-renewable foams compared to the standard foams, regardless of whether they have been made with or without non-reactive flame retardant. Therefore these New Green Cal 117-1975 55 % foams, with their high levels of natural oils, low-emission silicones and catalysts and non-emissive flame retardant OP 560 added, now pass Cal TB 117-1975. The 55 % New Green Cal 117-1975 foam also beats by a margin of 45 % emissions of the standard Cal TB 117-1975 foam, made with a non-reactive flame retardant, standard petrochemical polyols and standard foam additives.

4.2 VDA 278 VOC and FOG results

VDA 278 is a two-part German emissions test originally developed by Daimler Chrysler to safeguard the environment for drivers and passengers in automobiles. VDA 278 is commonly used to test emanations from all car interior furnishings – including fabrics, carpets, leathers, coatings, paints, glues, plastics and, of course, foams. Off-gasses are analysed at 90 °C and 120 °C.

Had standard amine and gelation catalysts been used to make these 55 % and 82 %

natural foams, both would have failed the VOC part of VDA 278, with emission levels estimated to be in excess of 400 ppm **(tab. 2).**

Had a standard non-reactive flame retardant been used to pass Cal TB 117-1975 at the above densities, the 55 % and 82 % foams would have failed the high-temperature FOG test, with emission levels of about 3,500–5,000 ppm compared to the test limit of 250 ppm. Using OP 560 in the 2013 trials **(tab. 2** – Made in 2013) had already solved the problem of flame retardant emissions, alone; but still the foam failed the VDA 278's FOG protocol.

Further trials were therefore needed to Green Cal 117-1975 foam to pass the FOG part of the VDA 278 test. The antioxidant (AO) packages used in the NOP and petrochemical polyol have a considerable bearing on the FOG results. The emissive AO package used in the NOP contributed 659 ppm to the total FOG reading of 767 ppm. The petrochemical polyol part of the foam formulation was chosen with a relatively low amount of antioxidant present, but the presence of anything in the petrochemical polyol that would normally adversely affect the FOG results is mitigated here, because the petrochemical polyol is a relatively small part (18 %) of the total polyol component present in the foam formulation.

Therefore the solution appeared to be in changing the antioxidant package being added to the NOP **(tab. 2** – Made in 2014).

Milliken Chemical [2] was very helpful in technically investigating and recommending that 1,000 ppm of its reactive antioxidant AOX-1 would provide good Oxidation Onset Temperatures (OOT) and reduce discolouration from scorch.

Milliken also commented that the palm-oil polyol showed inherent stability in the OOT tests compared to petrochemical polyols, and appears to resist peroxide formation. The NOP supplier PolyGreen Chemicals reacted positively to these suggestions and changed its antioxidant package to complete this work. The change by PolyGreen will probably be permanent. No AOX-1 was detected in the FOG results from these foams, suggesting the antioxidant material, as we expected, was fully reacted into the polymer [3].

To fully test this antioxidant/FOG relationship theory, the VDA test foam was made using 4,000 ppm of AOX-1 instead of the recommended 1,000 ppm. This was done to settle any doubts about whether the foam's new FOG characteristics may have been caused by the low amount of AO used rather than the type of reactive AO used. The foam in question still easily passed the Daimler Chrysler FOG emissions test (126 ppm versus 767, the upper limit being 250 ppm).

Table 2 also illustrates that the densities of the two foams are different. This would favour the emission results of the lower density Green Cal 117-1975 foam because it has the relatively lower polyol content of the two foams under test. The USDA BioPreferred GU #77-13 foam sample is the higher density material and would normally have a relatively poor FOG result compared to the lower density material made in 2013. In spite of this density penalty, the USDA BioPreferred sample still outshines the previous emission technology used in 2013 to make the Green Cal 117 foam. The improved results come from using Milliken's AOX-1 as the main AO, despite deliberately adding four times more than recommended to the USDA GU #77-13 formulation.

4.3 Emissive flame retardants via solvent extraction

As the OP 560 flame suppressant reacted permanently into the foam matrix, it did not contribute to the TVOC levels found in any of the CertiPUR-US results **(tab. 1)** or the VDA 278 FOG tests **(tab. 2).** Furthermore, a foam sample was recently submitted for sol-

Tab. 2: *Foams made in 2013 with 55 % of the petrochemical polyol replaced by a palm oil-based NOP (New Green Cal 117); compared to foams made in 2014 with 82 % of the petrochemical polyol replaced by a palm oil-based NOP (PolyGreen Chemicals F 6037) and labelled as USDA BioPreferred GU #77-13.*

Foam production date	Made in 2013	Made in 2014
Foam formulation	New Green Cal 117-1975 55 php of NOP / php	USDA BioPreferred GU #77-13 82 php of NOP / php
Certified bio-renewable content / wt%	35	50
Polyol GU 5566V	100	–
Polyol GU 8274V	–	100
Water	3.1	2.85
Low emission amine A	0.3	–
Low emission amine B	–	0.3
Exolit OP560	2.8	2.6
Kosmos EF	0.2	0.2
Flame retardant silicone A	0.9	–
Flame retardant silicone B	–	1.0
TDI 80/20 (index)	103	103
Standard antioxidant Irganox 1135	~4,000 ppm	–
Milliken Chemical Milliguard AOX-1	–	~4,000 ppm
Foam properties		
Foam density pcf / kg/m^3	1.68/27	2.0/32
Foam hardness IFD in lbs / CLD in kPa	18/1.77	50/5.0
Certified results – VDA 278, VOC + FOG		
VDA 278 Daimler Chrysler VOC test standard – limit 100 ppm maximum	48	69
VDA 278 Daimler Chrysler FOG test standard – limit 250 ppm maximum	767*	126**

Polyol GU 5566V – Natural Foams Technology polyol containing 55 % of a well-priced NOP(V)
Polyol GU 8274V – Natural Foams Technology polyol containing 82 % of a well-priced NOP(V)
*includes 659 ppm of various emissive antioxidants contained in the NOP and the petrochemical polyol
**includes 46 ppm of emissive antioxidants contained in the petrochemical polyol

vent extraction and spectrum analysis to further test these findings. As of 12 May 2014, when this paper was submitted, Duke University's Nicholas School of the Environment in Durham, NC, USA, has been unable to find traces of the OP560 flame retardant in foam samples produced according to the USDA BioPreferred formulation shown in **table 2.** The sample sent to Duke University is GU #77-11 containing 50 % bio-renewable material, low emission additives, and reactive flame retardant but containing only the recommended level of 1,000 ppm of Milliguard AOX-1.

4.4 Cone calorimeter – escape time and smoke production results

The cone calorimeter test was originally developed in the USA by the National Institute of Standards and Technology (NIST) and subjects the foam samples to extreme radiant energy – in this case a heat flux of 20 kW/m^2. It monitors off-gases, smoke, temperature, and heat released during the test procedure.

The OP560, 35 % Bio (Green Cal 117) is made with the low emissions package described **(tab. 1** and **2)** using a standard emissive antioxidant at 4,000 ppm. It therefore passes VDA 278 VOC only.

The two foams made with zero bio-renewable contents failed both VOC and FOG parts of the VDA 278 emissions test.

The OP560, 50 % Bio and Zero FR, 50 % Bio are also made with Natural Foams' low emissions package described earlier, including the addition of AOX-1 at the recommended level of 1,000 ppm. Both passed VDA 278 VOC and FOG tests **(tab. 3).**

4.4.1 Escape time

Looking at **figure 1** and **2,** the dark blue solid line, representing foam made with conventional petrochemical polyols without any flame retardant (Zero FR, Zero Bio), reaches its maximum level faster than all the other foams. By adding conventional (non-reactive) flame retardant to the foam, shown in the example HF4, Zero Bio, you see that extra escape time is provided. In this case we are using the non-reactive flame retardant Fyrol HF-4.

Considering heat release (AHRR) statistics alone, the Natural Foams OP560, 35 % Bio with 2.8 php of reactive flame retardant performs similarly to the standard petrochemical foam made with the non-reactive halogen-free flame retardant Fyrol HF-4 (HF4, Zero Bio) but at 12 php added.

Fig. 1: *Heat Release Rate (HRR)*

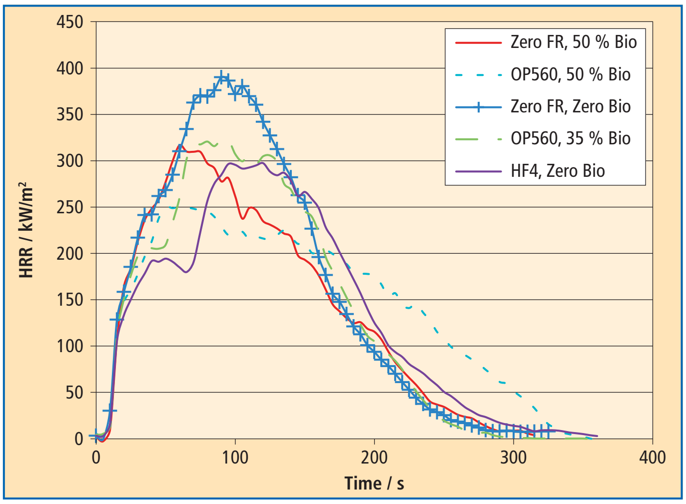


Fig. 2: *Average Rate of Heat Emission (ARHE)*

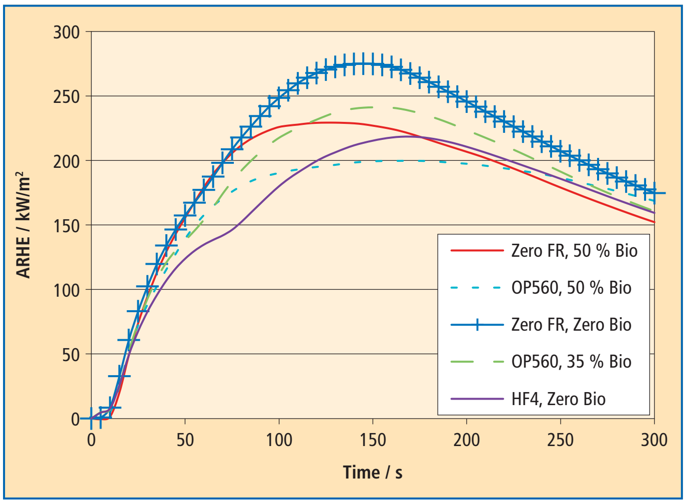


Fig. 3: *Mean Peak Heat Release Rate (PHRR)*

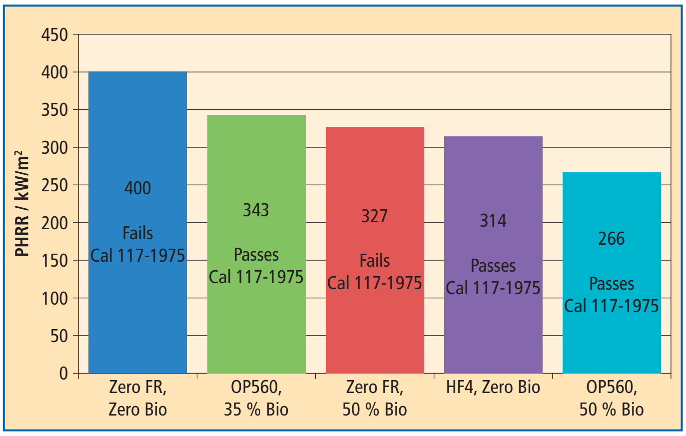
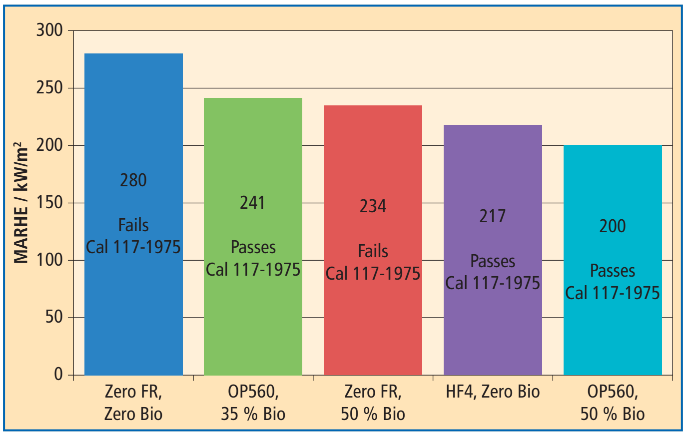


Fig. 4: *Maximum Average Rate of Heat Emission (MARHE)*



The Natural Foams sample OP560, 50 % Bio containing 50 % bio-renewable material with OP 560 present at 2.6 php is the best overall performing foam .

Looking at **figures 3** and **4,** once again, the standard non-flame retardant grade Zero FR, Zero Bio reaches the highest maximum heat emission rate (PHRR) and (MARHE) in a shorter time than the other foams. The Natural Foams OP560, 35 % Bio at 2.8 php of reactive flame retardant shows similar results to the standard petrochemical foam (HF4, Zero Bio) made with Fyrol HF-4 at 12 parts. Interestingly, the 50 % bio-renewable content sample with zero flame retardant (Zero FR, 50 % Bio) performs almost equally to both the OP560, 35 % Bio and the HF4, Zero Bio foams with suppressant added.

The foam with the lowest Rate of Heat Release (PHRR) and Maximum Average Rate of Heat Emission (MARHE) is the 50 % bio-renewable foam containing OP560 (OP560, 50 % Bio).

4.4.2 Escape time and smoke production

Looking at **figures 5** and **6,** the purple line representing the standard petrochemical foam (HF4, Zero Bio) gives the highest smoke production in the quickest time. By comparison, Natural Foams' OP560, 35 % Bio shows a 20 % reduction in smoke, followed by the standard foam (Zero FR, Zero Bio). Both 50 % bio-renewable foams – with and without OP560 – show considerable reductions in smoke formation compared to all the other foam types (results in **tab. 4).**

Figure 6 emphasises the dramatic reduction in smoke generation from tests on foams with 50 % bio-content – with or without OP560 present. The graph indicates a 70 % or more reduction in smoke using OP560, 50 % Bio sample compared to the performance of the standard petrochemical foam with a non-reactive flame retardant (HF4, Zero Bio). The bio-content foam without OP560 (Zero FR, 50 % Bio) shows smoke production is reduced by about 80 %.

These figures will be validated during full production of the 50 % (82 php NOP) foams later this summer. There are also plans to further fire-test these foams under conditions of limited oxygen availability, in contrast to the unlimited oxygen conditions of the cone calorimeter.

Results indicate the best choice may be using foams containing the reactive flame retardant to maintain the initial "escape time" **(fig. 1** and **2),** whilst safeguarding the environment **(tab. 2).**

Disclaimer note: Cal 117, Cal TB 117-1975, Cal TB 117-2013 and the cone calorimeter protocols discussed in this paper are small-scale flammability tests. Any flammability ratings from these tests are not intended to reflect hazards by foam or any other material under actual fire conditions.

The heat and smoke results in **figures 5** and **6** show potential advantages in escape time for occupants, and improved environmental conditions for first responders, such as the fire department or other emergency services. We drew this conclusion from the cone calorimeter results that showed more of these foams remain after the burn tests than is usually the case.

5. Conclusion

Natural Foams' technology reduces the amount of flame retardant required for flexi-

Tab. 3: Results from five sets of foams sent to the Swiss Federal Material Test Laboratory [4] for cone calorimeter analysis all with the same densities of 2.0 pcf (32 kg/m³)

Foam types	Description
Zero FR, Zero Bio	~2.0 pcf density – fails Cal 117-1975 – fails VDA (emissive tin, amine, AO)
HF4, Zero Bio	~2.0 pcf density – passes Cal 117-1975 – fails VDA (emissive FR, tin, amine, AO)
OP560, 35 % Bio	~2.0 pcf density – passes Cal 117-1975 – VDA low emission* VOC pass only
OP560, 50 % Bio	~2.0 pcf density – passes Cal 117-1975 – VDA low emission (also sent to Duke University)
Zero FR, 50 % Bio	~2.0 pcf density – fails Cal 117-1975 – VDA low emission

Fig. 5: Total Smoke Production (TSP)

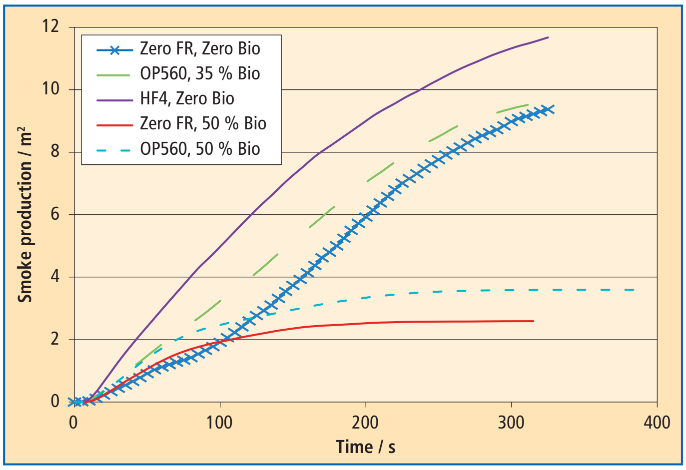
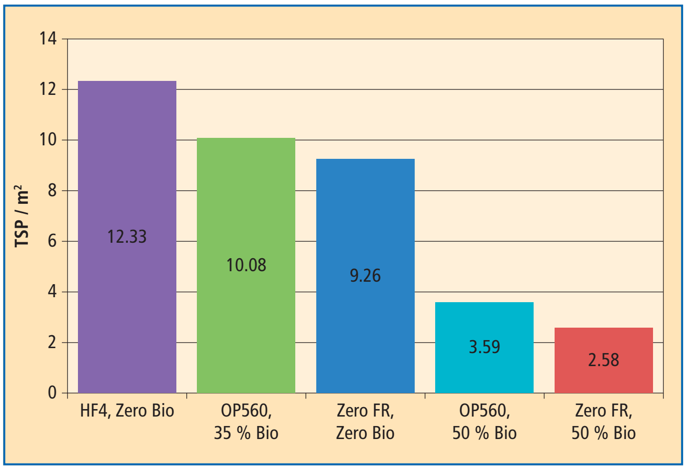


Fig. 6: Total Smoke Production (TSP)



ble foam to pass the smolder and open flame parts of California TB 117-1975 by 80 %. The small amount of a reactive flame retardant needed now becomes fixed into the foam matrix, it can not escape to threaten people's health and the environment.

By making further changes to the choice of tin and amine catalysts, silicone surfactants and antioxidants that can be used to make foam Cal TB 117-1975 and VDA 278 compliant, it is possible to deliver a very clean environment for consumers in terms of odour and off-gassing. The foams demonstrated here have a certified total bio-renewable content of approximately 35 wt% (where 55 % of the petrochemical polyol has been replaced by a NOP), and a certified bio-renewable content of 50 wt% (where 82 % of the petrochemical polyol has been replaced by a NOP).

Developments to produce foam with 50 wt% bio-content for the commercial market are about to enter final machine trials.

Cone calorimeter tests seem to indicate a 70–80 % drop in smoke production from these new 50 % bio-renewable foams.

These developments constitute a new class of foam that has been submitted to the USDA for certification under the BioPreferred programme run jointly by the US Department of Agriculture (USDA) and the American Society for Testing and Materials (ASTM).

The foams are commercially viable, being easy to process on standard equipment using known and plentiful raw materials. Their fire and emissions performances are class-leading when subjected to a wide range of independent, well-respected tests by certified laboratories in the USA and Europe.

The technology offers a more sustainable and environmentally safe route to producing foam that pass Cal 117- 1975 and Cal 117-2013.

These new bio-based foams address key safety issues raised by foams currently dominating the home environment. Measured against a number of standard flammability tests and against standard petrochemical based polyol foams, the new foams offer an equal or stronger degree of flame resistance and greatly reduced smoke levels.

In closing, the technology is a dedicated technical and certified response to legitimate public concern about off-gassing and emissions, which are increasingly affecting all aspects of the chemicals industry.

Foam types	-Delta / %
Zero FR, 50 % Bio versus Zero FR, Zero Bio	73 % less smoke
OP560, 50 % Bio versus HF4, Zero Bio	70 % less smoke
Zero FR, 50 % Bio versus HF4, Zero Bio	80 % less smoke
OP560, 50 % Bio versus Zero FR, Zero Bio	60 % less smoke
Zero FR, 50 % Bio versus OP560, 50 % Bio*	28 % less smoke*
OP560, 35 % Bio versus HF4, Zero Bio	18 % less smoke
Zero FR, Zero Bio versus OP560, 35 % Bio	8 % less smoke
*The TSP smoke performances of the two GU 50 % bio-renewable foams are very similar (3.59 m^3 versus 2.58 m^3); although as a percentage, the gap looks larger	

Tab. 4: Comparison table of foams by Total Smoke Production (TSP)

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