Future–Proof Natural Foams for the USDA BioPreferred® Program

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Abstract

Following media reports on the migratory effects of flame retardants in foams, the public are now more aware of emissions and "off-gassing" in their home and office environments. This paper sets out to fully address the issue of migrating materials and build on the unique green chemistry developed by Natural Foams Technology (under the Green Urethanes license). Further advancements have produced a series of foams that contain 50 percent-by-weight biorenewable materials (up from 35 percent previously achieved), resulting in class-leading low emissions and a new low level of smoke generation.

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 routinely adding large amounts of liquid, non-reactive flame retardants to the other chemicals used to make flexible foams. It has now been determined that these non-reactive flame retardant materials gradually migrate out of the foam and can impact people and the environment.

In response, California legislators have introduced a new protocol, California Technical Bulletin 117-2013, which negates the need for 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 remaining smolder part of the original test. The reasoning behind the change is that foam manufacturers will no longer need to add these non-reactive flame retardant materials to the foam, thus eliminating future risk of leakage into the environment. It also means the majority of foams in the US market will easily pass Cal TB 117-2013 without any disruption to the manufacturing process nor the need to add a flame retardant (FR) of any kind. You could arguably conclude from this that there will soon be no fire standard in California governing soft furnishings.

In this paper we 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 smolder test criteria) and the new test Cal 117-2013 (cigarette smolder test criteria only).

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

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

The paper also highlights important bio-content developments, with 82 percent of the petrochemical polyol (82 parts per hundred of total polyol, or php) being replaced with a natural oil polyol (NOP) based on palm oil sourced from certified plantations. NOP 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% percent compared to all existing Cal 117 compliant technologies.

Green 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 retardant. Our trial work was based on using natural oil polyols (NOPs) to produce foam with a NOP replacement value of 55 percent. By combining the green chemistry (5) (6) with a reactive flame retardant we've been able to produce a wide range of foams that pass Cal 117-1975. Furthermore, the flame retardant was permanently incorporated into the foam, as independent laboratory tests have shown. Other precision changes to the ingredients resulted in foams with very low emission properties when measured by the well-known and respected CertiPUR US test and Daimler Chrysler's searching VDA 278 test (described in more detail below).

This paper takes three further steps in this development process:

1) Production of a Cal TB 117-1975 foam, where 82 percent (up from the previous 55 percent) of the petrochemical polyol has been replaced by a natural oil polyol
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 commercially producing bio-renewable foams that meet the physical and hardnesses properties of foams in the most popular density range of between 1.25 and 2.0 pounds per cubic foot (20 to 32 kilos per cubic meter). Advancing the polyol replacement level to 82 percent will let us demonstrate that 50 percent by total weight of the foam is bio-renewable through certification by the BioPreferred® Program run by the United States Department of Agriculture (USDA).

Additionally, we've found that a very high NOP content gives the foam an inherent flame resistant boost. We are continuing to explore this new development, coupled with what we've already 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.

A New Class of Polyol with Unique Characteristics

During continuing trials, Natural Foams 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. We have also established that these particular bio-renewable foams produce better humidity/aging properties and SAG (support) factors, in addition to the 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 doesn't 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 by high-water formulations will still shrink, even when using TDI 65/35.

The previous paper confirmed that foam emissions could be greatly reduced by looking at all the components in the foam formulation, and by exchanging the normal amine catalyst for a low VOC reactive material (7), and the familiar stannous octoate for Degussa's Kosmos EF (8). Most of the silicone surfactants available in the US and Europe are already low VOC, so we used standard commercially available flame retardant silicones.

Natural Foams Technology (NFTech) used the reactive flame retardant additive Exolit OP 560 from Clariant Corporation. This is a reactive polyol with a hydroxyl value of around 450. It contains a phosphorous group and is halogen free. The material 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. Though it's found only limited use in flexible foam production.

The 2013 trials enabled the 55 percent NOP-replacement foams to pass Cal TB 117-1975 as well as the VOC part of the Daimler Chrysler VDA 278 emissions test. But they failed the FOG part of the Daimler test. Poor FOG results were caused by the emissions characteristics of the antioxidant package of the polyol, which we have since solved and discuss later in this paper.

Experimental – Emission Tests

CertiPUR US Results

Presented in the Polyurethane Foamers Association (PFA) paper – May 2013 (1)

Table 1 – Foams made in 2013; 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 (PolyGreen Chemicals F 6037) and containing a reactive flame retardant OP 560.

Table 1		PetroPolyol only	PetroPolyol + HF4	GU Technology + OP560
Hall Analytical (UK)	**Specification**	**Standard**	**Standard**	**New**
CertiPUR US Results	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**
CertiPUR Total Allowable Volatile Organic Compounds (TVOC)

UK-based Hall Analytical limited, are certified to carry out CertiPUR US tests, was given a sample of Exolit OP 560 to typify. We then asked Hall to specifically look for the presence of Exolit OP 560 in the readout of *each* of the randomly marked foam samples we provided for analysis.

Their detection apparatus found no trace (**Table 1**) of the reactive flame retardant polyol OP 560 in the New Green Cal 117-1975 55% foam.

Table 1 also shows that the total allowable (TVOC) emissions are drastically reduced in the biorenewable 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. They also exhibit even lower emissions than the standard petrochemical foams containing zero non-reactive flame retardant (labeled in **Table 1** as the **Standard Non Cal 117-1975** foam).

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). **Table 1** confirms that combining Natural Foams' green chemistry with high-NOP-based foam provides a technical solution that drastically lowers emissions from flame retardants and other additive sources when measured by the CertiPUR US test regime.

<u>VDA 278 VOC and FOG Results</u>

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 and 120 Celsius.

Table 2 -- Foams made in 2013 with 55% (php) of the petrochemical polyol replaced by a palm-oil-based NOP (shown in **Table 1,** above, as **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 labeled as **USDA BioPreferred GU #77-13.**

Table 2	php		php
Foam Formulation	**New Green Cal 117-1975 (made 2013) 55 php of NOP from Table 1**		**USDA BioPreferred GU #77-13 (made 2014) 82 php of NOP**
Certified bio-renewable content % by weight	**35%**		**50%**
Polyol GU **55**66V	100	Polyol GU **82**74V	100
Water	3.1	Water	2.85
Low Emission Amine A	0.3	Low Emission Amine B	0.3
Exolit OP560	2.8	**Exolit OP560**	2.6
Kosmos EF	0.2	Kosmos EF	0.2
Flame Retardant Silicone A	0.9	Flame Retardant Silicone B	1.0
TDI 80/20 (Index)	103	TDI 80/20 (Index)	103
Standard AntiOxidant e.g. Irganox 1135	~4,000 ppm	Milliken Chemicals Milliguard AOX-1	~4,000 ppm

Foam Density PCF/kg per m3	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

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, at levels probably in excess of 400 ppm. (The VOC test limit is 100 ppm.)

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

The unfinished business of the Paper presented to the PFA (**Table 2**) in 2013 is therefore this **Green Cal 117-1975 foam's** failure to pass the FOG part of the VDA 278 test (performed on off-gasses with the foam at 120 Celsius). As you can see, the antioxidant (AO) packages used in the NOP and petrochemical polyol had a considerable bearing on the FOG results. The emissive AO package contributed 659 ppm to the total FOG reading of 767 ppm (FOG spec limit is 250 ppm). We chose a petrochemical polyol 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.

The solution therefore lay in changing the antioxidant package being added to the NOP.

Milliken Chemicals (2) were very helpful in technically investigating and recommending that 1,000 ppm of their *reactive* antioxidant AOX-1 would provide good oxidation onset temperatures (OOTs) and reduce discoloration 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. Thanks then to Milliken for the recommendation, and to our NOP supplier PolyGreen Chemicals for changing their antioxidant package to complete this work. We understand this 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, we made the VDA test foam 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).

You will note in **Table 2** that the densities of the two foams are different. This would favor 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 antioxidant (AO), even when we have deliberately added four times too much of it to the USDA GU #77-13 formulation.

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 (**Table 1)** results or the VDA 278 FOG tests shown in **Table 2.** Furthermore, a foam sample has recently been submitted for solvent extraction and spectrum analysis to further test these findings. As of May 12th, 2014, when this paper was submitted, Duke University's Nicholas School of the Environment in Durham, North Carolina, has been unable to find traces of the OP560 flame retardant in foam samples produced according to the USDA Preferred® formulation shown in **Table 2**. The sample sent to Duke University is GU #77-11 containing 50% biorenewable material, low emission additives, and reactive flame retardant *but* containing only the recommended level of 1,000 ppm of Milliguard AOX-1. Further tests on GU #77-11 foam appears in **Table 3** below, marked for the sake of brevity as ***OP560, 50% Bio***.

Cone Calorimeter – Escape and Smoke Results

Photograph of Cone Calorimeter apparatus at the University of Central Lancashire, UK.

The Cone Calorimeter test was originally developed in the US 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 kilowatts per square meter. It then monitors off-gases, smoke, temperature, and heat released during the test procedure.

Table 3 -- Shows 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 pounds per cubic foot (32 kg/m3).

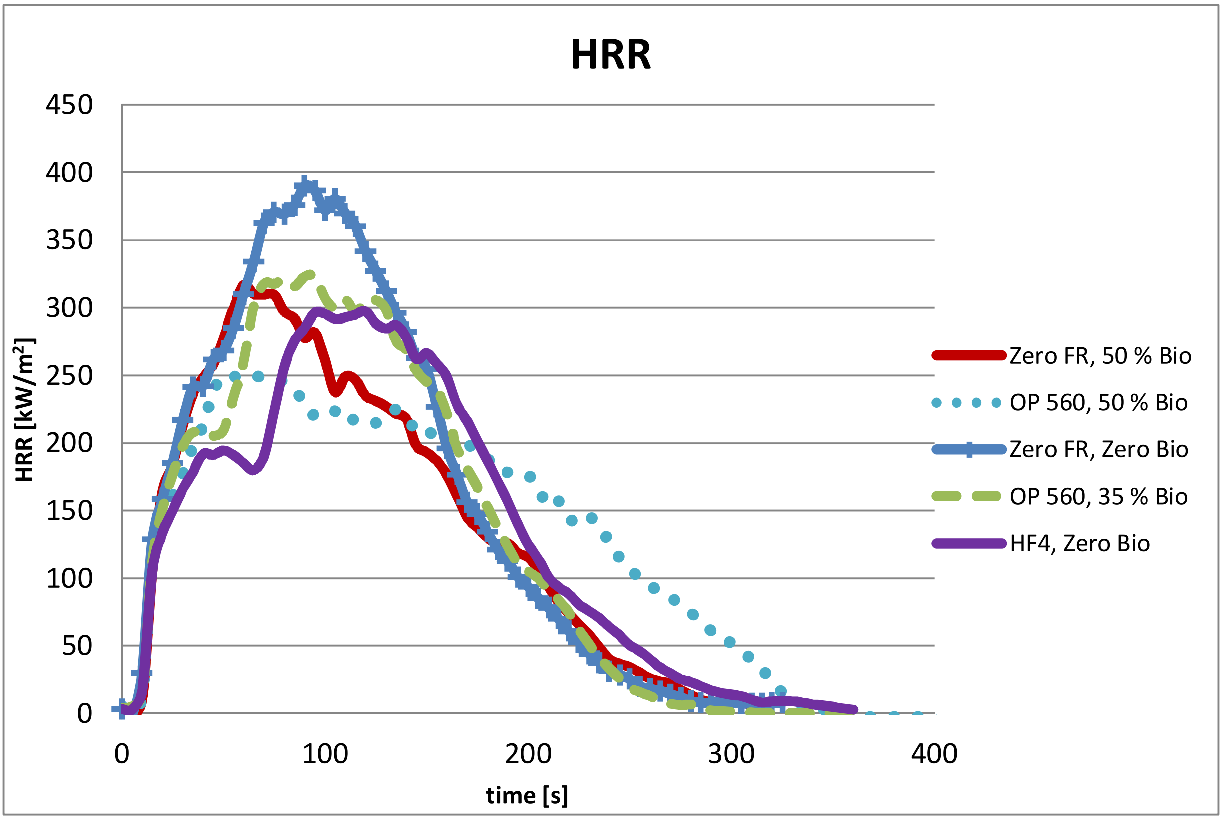
The **OP560, 35% Bio** (our **Green Cal 117**) is made with the low emissions package described in **Tables 1 & 2** using a standard emissive antioxidant at 4,000 ppm. It therefore passes VDA 278 VOC only*.

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

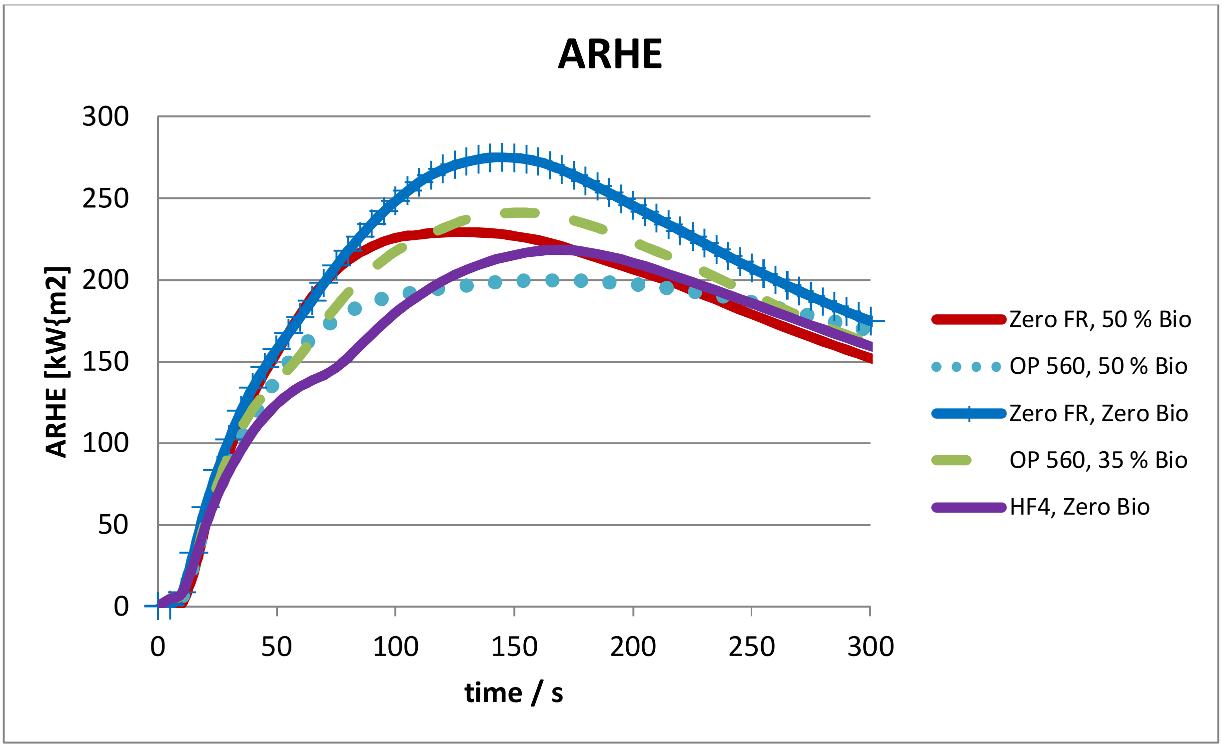
The ***OP560, 50% Bio*** and ***Zero FR, 50% Bio*** are also made with Natural Foams' low-emissions package described earlier, including the addition of AOX-1 at the recommended level of 1,000 ppm. Both passed VDA 278 VOC & FOG tests.

Table 3.

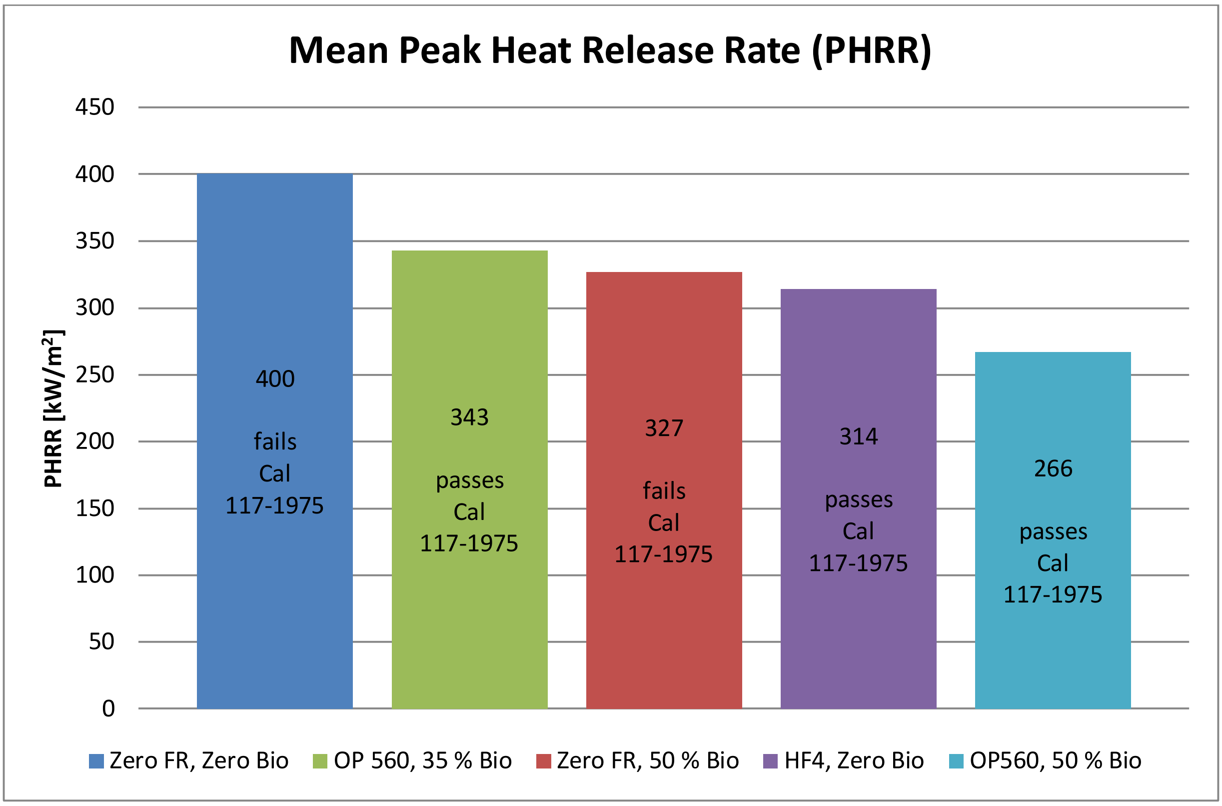
Foam Number #	Description
Zero FR, Zero Bio	~2.0 pcf density - fails Cal117-1975 – fails VDA (emissive tin, amine, AO)
HF4, Zero Bio	~2.0 pcf density - passes Cal117-1975 - fails VDA (emissive FR, tin, amine, AO)
OP560, 35% Bio	~2.0 pcf density – passes Cal117-1975 – VDA low emission* VOC pass only
OP560, 50% Bio	~2.0 pcf density – passes Cal117-1975 - VDA low emission (also sent to Duke University)
Zero FR, 50% Bio	~2.0 pcf density – fails Cal117-1975 – VDA low emission



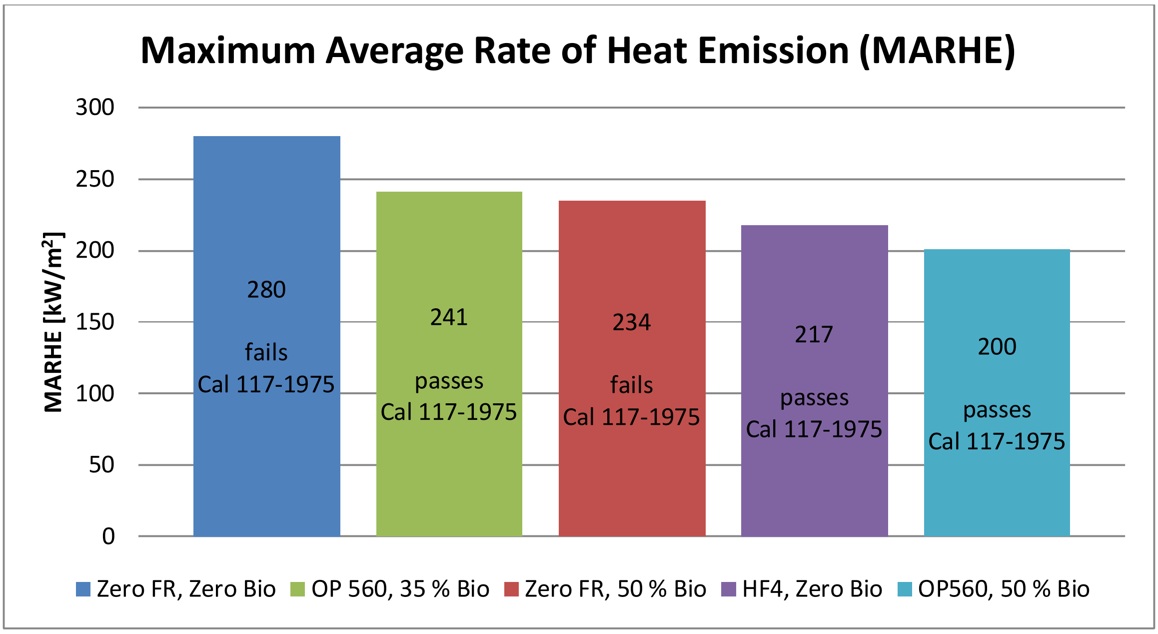
Graph 1: HRR – Heat Release Rate



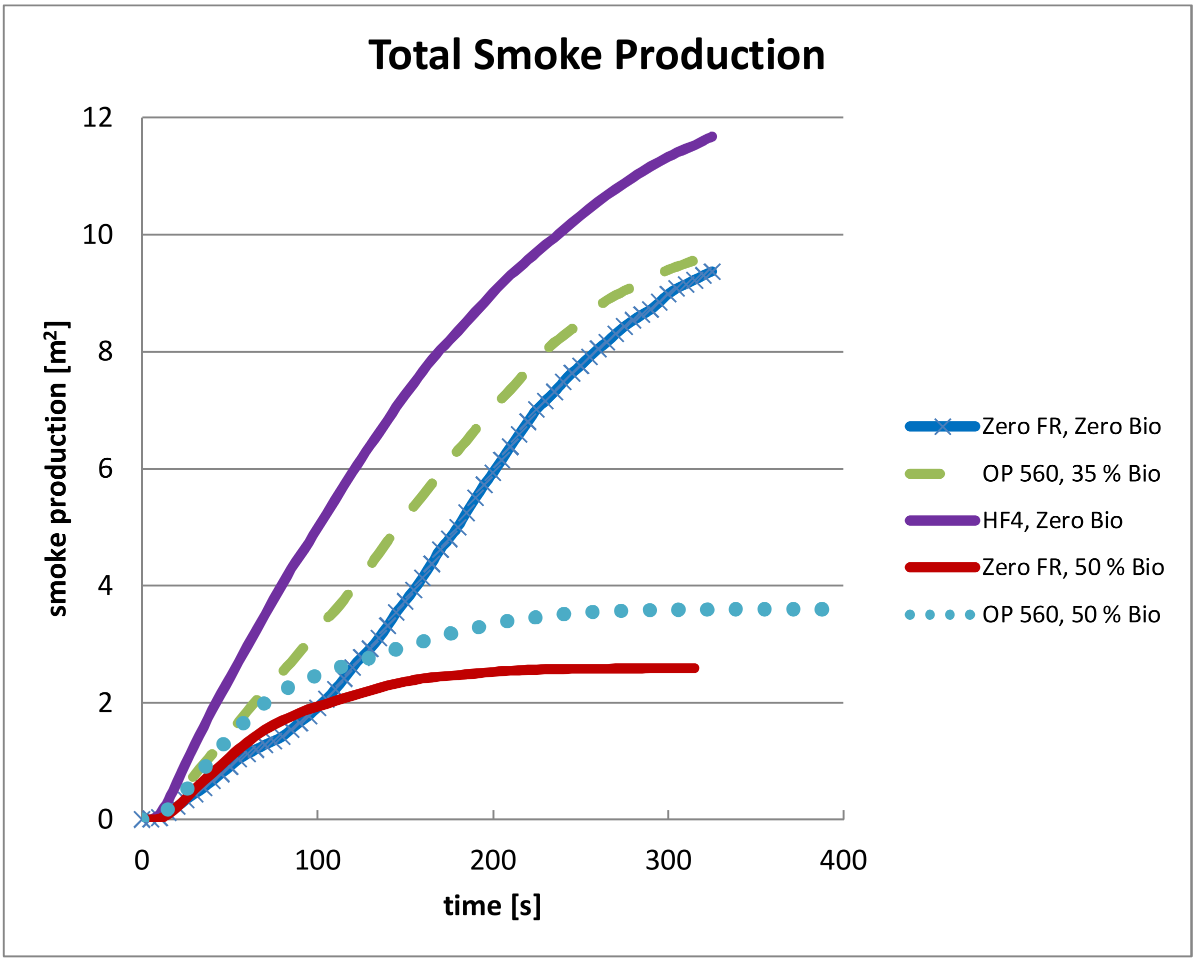
Graph 2: ARHE – Average Rate of Heat Emission



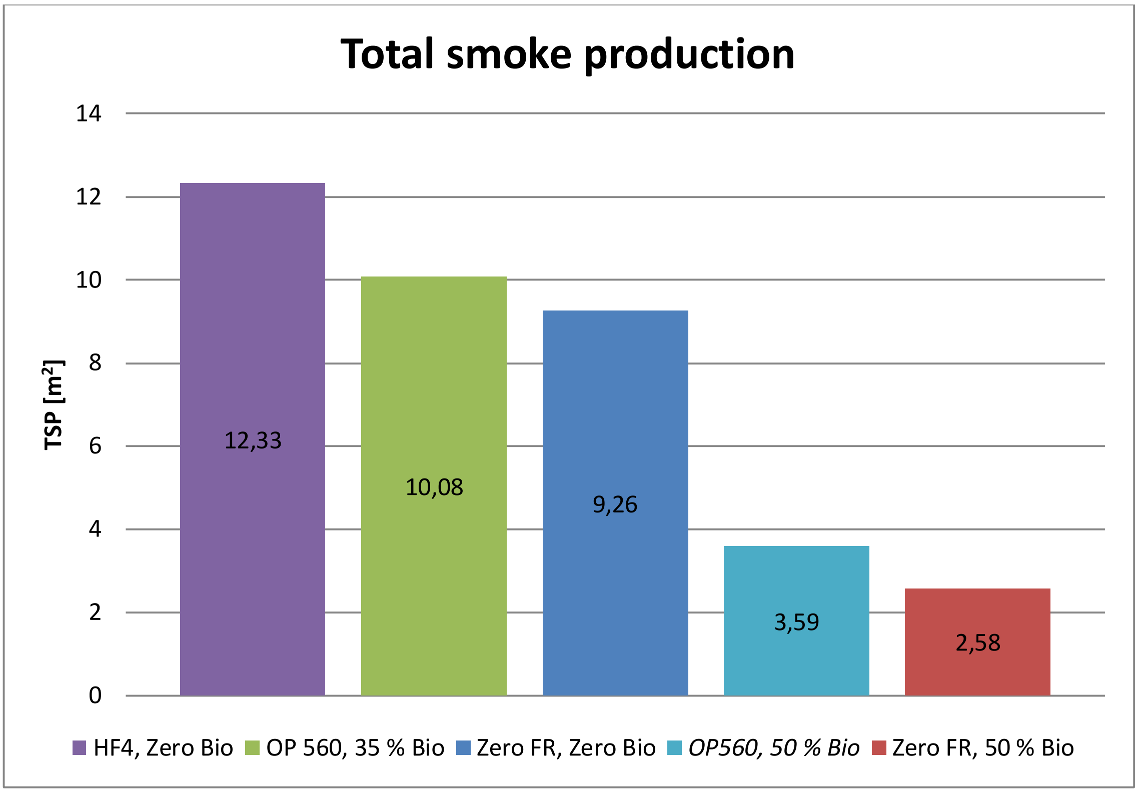
Graph 3: PHRR - Mean Peak Heat Release Rate



Graph 4: MARHE - Maximum Average Rate of Heat Emission



Graph 5: TSP - Total Smoke Production



Graph 6: TSP Total Smoke Production

What the Graphs Show

The best performing foams are those showing long, low, lazy lines with relatively low peaks.

Escape Time

Looking at Graphs 1 & 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.

If you look at 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.

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 (shown in **Graphs 1 and 2)**.

Looking at Graphs 3 & 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***).

Escape & Smoke

Looking at Graphs 5 & 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% biorenewable foams -- with and without OP560 -- show considerable reductions in smoke formation compared to *all* the other foam types (results in **Table 4**).

Graph 6 emphasizes the dramatic reduction in smoke generation from tests on foams with 50 percent bio-content – with or without OP 560 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.

Table 4.

Comparison table of foams by Total Smoke Production – TSP			**~Delta percentage**
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 versus 2.58 m3); although as a percentage, the gap looks larger.

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

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 Graphs 5 & 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.

Future Work

We will continue smoke generation testing on these high bio-content foams using apparatus that restricts oxygen supply to the burn sample. This may complete our insight into the ignition and burning properties of this technology.

We are also developing technology that should reduce the softening suffered by mattresses and furniture seating foams used in humid conditions by up to 70%. We will investigate and confirm these results at the next phase of our development program.

In Conclusion

Natural Foams' technology reduces the amount of flame retardant required for flexible foam to pass the smolder and open flame parts of California TB 117-1975 by 80 percent. The small amount of a reactive flame retardant needed now becomes fixed into the foam matrix and will not escape and threaten people and the environment.

By making further changes to the choice of tin and amine catalysts and silicone 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 biorenewable content of approximately 35% by weight (where 55% of the petrochemical polyol has been replaced by a NOP), and a certified biorenewable content of 50% by weight (where 82% of the petrochemical polyol has been replaced by a NOP).

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

Cone Calorimeter tests seem to indicate a 70 to 80 percent drop in smoke production from these new 50% biorenewable foams.

These developments constitute a new class of foam that has been submitted to the USDA for certification under the BioPreferred® Program run jointly by the United States Department of Agriculture and the American Society for Testing and Materials (USDA & 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 US 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 "future proof" foams address key safety issues raised by foams currently predominating the home environment. Measured against a number of standard flammability tests and against standard petro-polyol foams, the new bio-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.

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Biography

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Jeff Rowlands is a director of Natural Foams Technology, part of Green Urethanes Limited. The UK-based company specializes in developing and licensing natural technologies for the urethane foam industry. Prior to this, Jeff was a director of Interchem International SA in Luxembourg, which developed and licensed the PIPA polyol process for the North American and European flexible slabstock and moulding markets. He was also Technical Director of a foam production facility in the UK, which he designed and commissioned (now part of Recticel NV). He is a Fellow of the Institution of Chemical Engineers, a Chartered Engineer and a registered European Engineer at Fédération Européenne d'Associations Nationales d'Ingénieurs in Paris.