Aromatic Triazine Polyol- a Novel Reactive Flame Retardant For Rigid Polyurethane Foam

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ABSTRACT

Rigid foams typically use low-molecular weight multi-functional polyols with polymeric MDI leading to close-meshed, highly cross-linked structures. While aromatic polyester polyols provide thermal insulation and reaction to fire, polyether polyols help achieve the desired mechanical properties. However, the mechanical property improvement generally comes at the expense of diminishing the fire performance.

Resonance™ triazine polyols (RTP) are designed with aromaticity levels as high as 80% and nitrogen levels up to 21%. Owing to their aromaticity, reactivity, and hydroxy equivalent weights, the RTPs can partially replace the high-functional polyether polyols as well as some of the difunctional aromatic polyesters. In addition, the aromatic nature of the nitrogen in RTPs can make them a suitable alternative to halogenated flame retardants. The experimental study demonstrates the effect of incorporating RTP into a PUR formulation. The paper will discuss system properties such as viscosity of the mix and foam reaction profile and rigid foam properties. The study would also include the compatibility with blowing agents and solubility in commonly available polyols, and a review of the key benefits of incorporating the Resonance™ triazine polyols in the rigid foams suitable for various applications such as building industry, appliances, heated pipelines, etc.

INTRODUCTION

Polyols furnish the hydroxyl group (OH group) required for the reaction with isocyanates and, therefore, crucially influence the properties of the final urethane polymer. The most common and commercially relevant sources of hydroxyl groups are polyether polyols or polyetherols (PEP) and polyesters. Among the PEPs, polyols of functionality three and higher are based on initiators such as glycerin, trimethylolpropane, sorbitol, sucrose, etc. PEPs with hydroxyl numbers (OHV) in the range of 350-600 are quite common for rigid foams.[1]

Polyols, one of the major ingredients in a foam, dictate whether the foam will be rigid or flexible, brittle or nonbrittle, and its permeability to gas and moisture. Polyols influence many foam properties, such as processability, dimensional stability, friability, heat resistance, compressive strength, etc. Several published literatures report formulations that contain aromatic polyester polyols and PEPs such as pentaerythritol, sorbitol, sucrose, glycerol etc.[1] However, relatively less is known about the impact of introducing aromatic polyols into PUR and PIR formulations and the effect of their physiochemical attributes on the properties of rigid foams.

Due to their aromatic content, aromatic polyesters are widely used in polyurethane insulation foams to improve reaction to fire.[2] However, they are limited in terms of their functionality, and hence, high-functional polyether polyols are often required to balance properties such as dimensional stability. When PEPs are added to improve mechanical properties, increasing levels of phosphorous or halogen-based flame retardants (FR) are added to the formulation. Halogenated fire retardants are under regulatory scrutiny.

In this paper, we wish to introduce Bakelite's Resonance™ triazine polyols (RTP) into the polyurethane world. These are unique aromatic polyols that combine high aromaticity with nitrogen. The functionality of the RTPs is estimated to be around 4.5. In this study, we wish to introduce two different RTPs that vary in their nitrogen content. TF94-806 has a nitrogen content of ca. 21%, and TF94-2812E has a nitrogen content of ca. 10.5%. Using two times more TF94-2812E than TF94-806 in the formulation, the nitrogen content was nearly the same for the foams made from them. While TF94-806 is manufactured on a large scale, TF94-2812E is an experimental product included primarily for this investigation to observe the effect of these on reactivity and foam properties.

The study illustrates the value of these RTPs when it comes to maximizing flame resistance while maintaining mechanical properties or even improving them in some cases. Since these nitrogen-rich polyols are novel to the PU world, the paper discusses their physiochemical attributes and distinguishing aspects compared to traditional polyols. This paper also shows the solubility of the Resonance™ aromatic triazine polyols in various polyols, reactive diluents and chemical compatibility with hydrochlorofluoroolefins (HCFOs) and hydrofluoroolefins (HFOs).

In previous years, as a part of the Resonance™ series, aromatic polyether polyols such as the PL91-, PM91-, TM91-, and TL91- were shown to effect significant improvements in reaction to fire (RTF) by replacing traditional PEPs and aromatic polyesters in PUR and PIR formulations.[3, 4,5, 6, 8] The aromaticity of these were typically in the range of 33 to

50% and the nitrogen content of the TM91 and TL91- polyether polyols was about 5 to 14%. The aromaticity and the nitrogen content of the RTPs (the two TF94s) in this study are significantly higher.

While the PL-series polyols were liquids, these TF94 RTPs are solids at room temperature. However, due to their excellent miscibility with a variety of common polyols and diluents, they can be delivered as a liquid at room temperature (RT). This paper discusses those aspects in detail as well.

EXPERIMENTAL

Materials & Test Methods

Materials
COIM supplied the aromatic polyester; Monument supplied sucrose polyol; Carpenter supplied PEPs; Chemours and Honeywell supplied HFOs; Evonik supplied surfactants and catalysts; BASF supplied the isocyanate; Sigma-Aldrich supplied TEP and DEG, and Acros Organics supplied n-pentane. Bakelite Synthetics produced the two TF94 polyols.

Dissolution of TF94 in various polyols:
The TF94-806 was melted in a reactor vessel equipped with the capability to agitate, heat and cool the contents. Polyol or polyol blend was added to the molten TF94 under mixing until a homogeneous solution was obtained. The content was cooled to RT, and relevant properties were measured. The concentration of TF94 reported in this study was 10% by weight.

Chemical compatibility with HFOs:
This Bakelite's in-house test is a modified version of the binary compatibility test. As TF94-806 is a solid at room temperature, it was pre-dissolved in methanol at 10-50% concentrations, mainly relevant to their usage level in the foam formulations. With maximum nitrogen content and high reactivity, TF94-806 was chosen for this test as any halide release arising from this interaction is expected to be less for the experimental polyol, TF94-2812E.
Three grams of the TF94 solution was mixed with three grams of HFO in a sealed ACE pressure tube. After the mixture was maintained at 54°C for 24 hours, it was diluted with solvent and filtered. Fluoride and chloride concentrations were measured with Ion Selective Electrode and reported in PPMs.

Foam Preparation and Testing:
Foams were prepared via hand-mixing using a high-torque mixer at 3,000 rpm speed. Polyols and isocyanate of the foam systems were put in a plastic cup and mixed for 10 seconds. Afterward, the mixture was transferred into a paper bag before the cream time. Cream time, gel time, and tack-free time were measured. Formulations were optimized to meet the reference formulation's reactivity and density. Polycat 5, Polycat 8, and Kosmos 33 were used as catalysts. Polycat 8 was adjusted for the RTP-containing foams to match the reactivity of the reference foams. Water and n-pentane were used as the blowing agents. Triethyl phosphate (TEP) was used as FR, and its amount was kept constant in the foams. The iso index was held at 120.

The optimized formulations were then used to prepare free rise 10x10x5 cake box foams for extended testing. In this stage, foams were prepared at a 500-gram total blend size. The foam blocks were cut for testing 5-7 days after preparation. Cut foam samples were conditioned and tested according to proper test methods.

All test specimens were conditioned at 23°C and 50% RH for at least 2 days before the test. The following tests were conducted on the foams:

- Free rise density was measured by dividing mass by volume of cubic foam.
- Closed cell content was obtained from AccuPyc II 1340 Gas Pycnometer according to ASTM D6226 with correction procedure 2. Test specimen was two (2) 25 mm x 25 mm x 25 mm cubes.
- Compressive properties were tested according to ASTM D1621 on an Instron Universal Test Machine equipped with 10 kN load cell at a crosshead speed of 5 mm/min. Test specimen size was 50 mm x 50 mm x 50 mm. Compressive modulus and strength were obtained from the test. The compressive strength was also normalized to a density of 32 kg/m^3. The compressive test was performed on foam directions both parallel and perpendicular to the foam rise direction.

- Dimensional stability was tested according to ASTM D2126 at 70°C and 95% RH for 7 days. Test specimen size was 100 mm x 100 mm x 25 mm. The weight and dimensions were measured before and after the conditioning. The mass and volume change percentages were then calculated.
- Reaction to fire (RTF) test, UL-94V, was performed according to ASTM D3801 in a FTT UL Fire Chamber equipped with a small Bunsen burner and methane gas (purity > 98%). Test specimen size was 125 mm x 13 mm x 13 mm.
- Form k-factor was obtained from FOX314 Heat Flow Meter according to ASTM C518. The test specimen size was 203 mm x 203 mm x 25 mm. The upper and lower plates temperatures were set at 10.0°C and 35.0°C, respectively. The aged k-factor was obtained similarly with pretreatment of test specimen at 70°C for 21 days.

RESULTS and DISCUSSION
Dissolution of TF94-806 in various polyols

Table 1: Solubility of RTP in polyester, polyetherols and sucrose-polyol

TF94-806 at 10% in Polyols	Chemistry	Viscosity, cps @ 25°C
Carpol Triol GP-700	Aliphatic polyether polyol	1,300
Resonance™ PL-203 blend mix	Aromatic polyether polyol	9,250
Resonance™ PL-507	Aromatic polyether polyol	13,570
Isoexter 4404-US	Aromatic polyester	16,000
Poly-G® 74-376	Sucrose (sugar) polyol	19,200

The TF94-806 was dissolved at 10% by weight in concentration in various polyols, and their viscosities were measured (Table 1). In general, these solutions remain dissolved for several weeks. Although the TF solution in GP-700 exhibited cloudiness after ten days, the mixture became clear upon heating. Higher amounts of TF94-806 (> 10%) can be dissolved in many of these polyols, but it will result in a further viscosity increase. Due to TF's excellent solubility in reactive diluents such as polyethylene glycol ethers, these can also be used along with polyols to get the desired viscosity.

Chemical compatibility with HFOs:
Blowing agents (BA) are compounds that can be gas or liquid that provide cellular structure to the foam and a good blowing agent is considered to have considerable solubility in the polyol.[7] In addition to potential environmental concerns, the solubility of the BA in the polyol is an important criterion in selecting an appropriate one for the desired application. The degree of foaming and cellular structure is dictated to a large extent by the solubility of the BA.[8] Of the blowing agents, HCFOs and HFOs are particularly important because of their low global warming potential and low ozone depletion value.[9] In addition, they offer excellent thermal insulation and are non-flammable. Therefore, they are the desired blowing agents in spray foams. However, due to their olefinic nature, these blowing agents have the potential to undergo reactive chemistries in the presence of certain catalysts and polyols. Thus, testing the chemical compatibility of polyols with these blowing agents is critical to the success of the foam formulation and its long-term thermal insulation. A large amount of halide release indicates the destabilization of these BAs. Bakelite's test method is more accurate for fluoride and less reliable for chloride measurement.

Table 2: Binary Compatibility Test

Concentration of TF94	Solstice LBA		Opteon 1100	
	F- conc.	Cl- conc. *	F- conc.	Cl- conc. *
	ppm	ppm	ppm	ppm
10/90 TF94-806/MeOH	0	7		
20/80 TF94-806/MeOH	0	10		
30/70 TF94-806/MeOH	0	4		
40/60 TF94-806/MeOH	0	2		
50/50 TF94-806/MeOH	0	88	1	0

Table 2 shows the binary compatibility test results of TF94-806 with HFO and HCFO. Overall, the TF94 polyol exhibits good compatibility with these blowing agents. With Opteon 1100 (HFO), even at a 50% concentration of TF, there is

no halide release. With Solstice LBA, no significant fluoride or chloride was released up to 40% of TF polyol. However, at 50% concentration, 88 ppm chloride was detected with the Solstice material. The encouraging aspect is that there was no correlation between halide release and TF concentration. Further, since the recommended concentrations of the TF polyols in a foam formulation are below 50%, it is safe to conclude that the TFs exhibit excellent chemical compatibility with the Solstice LBA as well.

Resonance™ aromatic triazine polyols replacing sucrose polyol in PU Rigid Foam

Table 3: Formulation Details

| Materials	OHV	Ref	Mod. A 10% TF94-806	Mod. B 20% TF94-2812E
Polyol Component of PU System				
Isoexter 4404-US f=2	240	55	55	55
Propoxylated sucrose/ glycerol f=5	365	25	22.5	20
TF94-806 f=4.4	590	-	**2.5**	-
TF94-2812E f=4.5	575	-	-	**5**
DC 193		2	2	2
Polycat 5		0.11	0.11	0.10
Polycat 8		0.38	0.20	0.16
Kosmos 33	815	1.50	1.50	1.50
TEP		10	10	10
Water	6233	2.0	2.2	2.2
n-pentane		6.0	7.0	7.0
Total parts		101.99	103.01	102.96
Polyol Component Visc@25C, cps (w/o BA)		632	805	1080
Polyol Component Visc@25C, cps (with BA)		307	445	625
Isocyanate Component of PU system				
Lupranate M20 part	IEW 133.3	111.9	117.7	119.5
Isocyanate Index		**130**	**130**	**130**
Reaction Profile				
Sample scale, g		500	500	500
Dispensed into		10x10x5	10x10x5	10x10x5
Mix time, s		10	10	10
Cream time, s		18	20	20
Gel time, s		48	50	50
Tack-free time, s		95	100	105
Foam Properties				
Free Rise Density, kg/m3		28.4 ± 2.0	26.6 ± 1.0	28.0 ± 0.3
Closed Cell Content, %		86.8 ± 2.7	91.1 ± 0.4	87.7 ± 4.3
k-Factor, mW/(m·K)		27.0	26.8	26.5
k-Factor, aged (24 days @ 70C), mW/(m·K)		31.4	31.0	30.9

The main objective of this study is to replace 10-20% of a sugar-based polyol with two different RTPs and study the effect of introducing the TF94 on the rigid foam properties such as RTF, compressive strength, and dimensional stability. And to achieve this, the PUR reference formulation was kept relatively simple with two polyols- an aromatic polyester and a sucrose/glycerol-based polyol.

Table 3 provides the formulation details that include, in addition to the polyol amounts, the amounts of catalysts, FR, BAs, isocyanate, iso index, reaction profile, density, etc. TF94-806 replaced 10% of the sugar polyol (Mod. A), and TF-2812E (with half the nitrogen) replaced 20% of the sugar polyol (Mod. B). In total, the quantity of TF polyols in the polyol blend was only 3% and 6% in Mod. A and Mod. B respectively. The isocyanate quantity and the iso index were

kept constant for all the foams. In addition, TEP was maintained at the same percentage in the foams across all samples. Polycat 5, Polycat 8, and Kosmos were used as catalysts. Water and n-pentane were used as blowing agents and adjusted for Mod. A and Mod. B to match the density of the reference.

The introduction of RTPs increased the system's reactivity, indicating their catalytic nature. This catalytic activity of RTPs arises primarily due to nitrogen in the backbone. The reduction of catalysts, mainly Polycat 8, brought the reactivity of Resonance™ polyols close to the reference. This can be beneficial as there is a need, particularly in the spray foam area, to reduce the emissions arising from the use of organic amines. Small molecule catalysts such as amines can be volatilized during spray polyurethane installation that can lead to Glaucopsia, fishy odors, and respiratory irritation.[10] Figure 1 shows the impact of both TFs in reducing the amine catalyst. As the TF polyols react into the PU backbone, they do not contribute to volatile emissions and can be considered auto-catalytic. Even though the amount of TF94-2812E is two times the amount of TF94-806, the reactivity of Mod. A and Mod. B is similar. This can be attributed to the same nitrogen content in both cases.

Figure 1: Reduction of Polycat 8 by the introduction of TF94 polyols
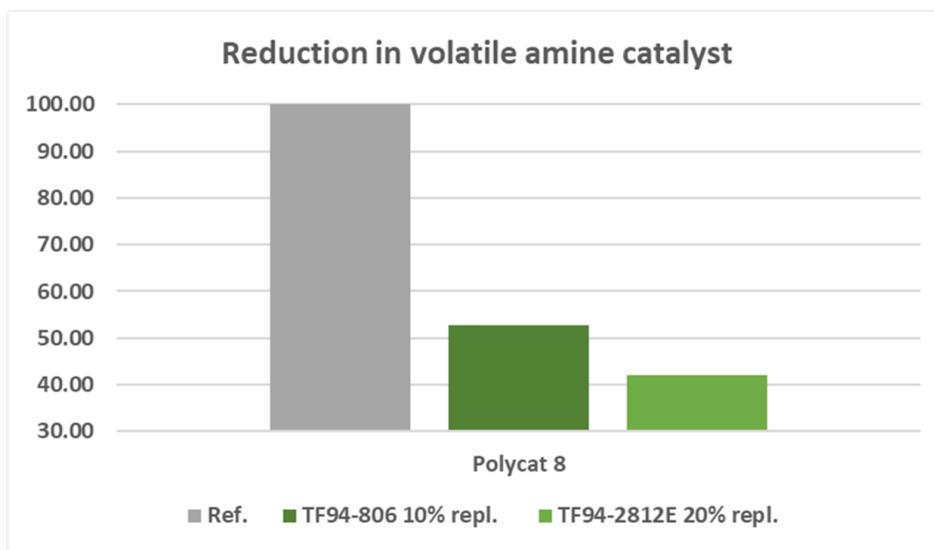
Formulations were thus optimized for target reaction profile (cream time, gel time) and free-rise density to match the reference. The viscosity of Part B blends increased slightly for Mod. A and Mod B. compared to the reference formulation. The viscosity of the test foams is expected to match the reference even closer in a typical spray foam formulation wherein other polyols and ingredients such as HFOs (typically much higher amounts than the amount of pentane used here) are present.

All foams prepared with Resonance™ PL polyols exhibited significantly better RTF than the reference in the UL-94 vertical burn test. Table 4 shows that the reference foam burnt considerably longer than Mod. A and Mod. B foams. Although the t_1 for Reference foam is reported in the table as 4 seconds, there were iterations where it burnt even longer, almost past 10 seconds. Also, the reference foam burnt to the clamp every single time. Per the test criteria, the reference foam failed, whereas Mod A and Mod. B foams achieved the V-0 rating. There was no significant difference between the Resonance™ foams Mod. A and Mod. B.

Figure 2 illustrates the dramatic effect of replacing sugar-based polyol with the two TFs on the RTF of these foams. Compared to Mod. A and Mod. B foams, the reference foam burnt 92-95% longer. The TF-containing foams quit burning as soon as the flame was removed. Figure 3 illustrates the significant difference in mass retention (after the test) between the reference foam vs. Mod. A and B.
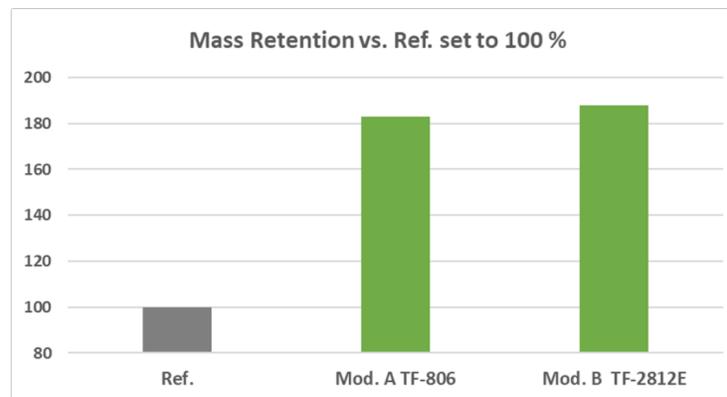
Table 4: Effect of RTP on Reaction to Fire Test

Reaction to fire, UL-94 V	Ref	Mod. A 10% TF94-806	Mod. B 20% TF94-2812E
After Flame Time 1, t_1, s	4 ± 3	0.2 ± 0.4	0.3 ± 0.4
After Flame Time 2, t_2, s	0	0	0
Total Flaming Time, t_f, s	4 ± 3	0.2 ± 0.3	0.3 ± 0.4
Mass Retention %	41±8	75±3	77±3
Burned to Clamp	Yes	No	No
V rating	failed	V-0	V-0

Figure 2: Total flame time of the three foams
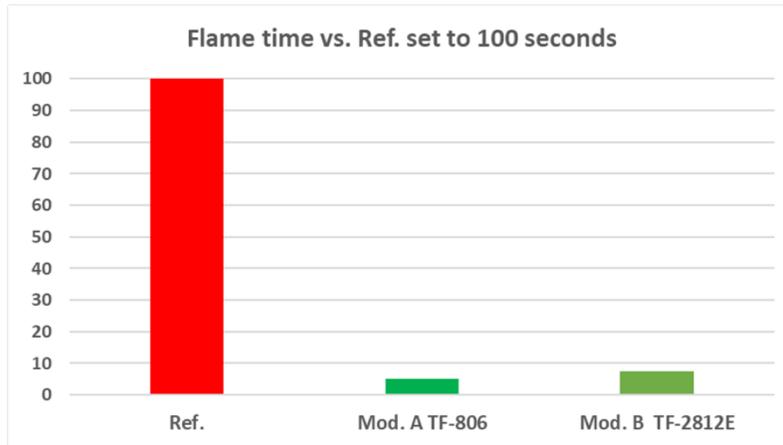
Figure 3: Mass retention of the three foams

No significant difference was observed between Mod. A and Mod B. in terms of flame time and mass retention even though Mod. B replaced twice as much sugar polyol as Mod. A, which also increased its aromatic content by the same amount. Both foams had nearly the same nitrogen content by design. This shows that the aromatic nitrogen content has a more significant effect on the RTF than the overall aromaticity.

Between the two RTPs, TF94-2812E containing Mod. B foam exhibited significantly higher compressive strength (parallel to rise) than Mod. A and the reference foam. This can be attributed to the higher aromatic content of Mod. B due to 20% replacement of sugar polyol (Table 5).

Table 5: Effect of RTP on Compressive Strength

Mechanical Properties	Ref	Mod. A 10% TF94-806	Mod. B 20% TF94-2812E
Parallel			
Compressive Modulus, MPa	5.1 ± 1.3	4.9 ± 1.2	6.0 ± 1.0
Compressive Strength, kPa	132 ± 26	125 ± 27	171 ± 2
Normalized Compressive Strength at 32 Kg/m3, kPa	150 ± 14	153 ± 16	187 ± 12
Perpendicular			
Compressive Strength, kPa	61 ± 2	64 ± 6	55 ± 1
Normalized Compressive Strength at 32 Kg/m3, kPa	70 ± 1	77 ± 8	64 ± 2

The dimensional stability is listed in Table 6. Overall, the test foams, Mod. A and B. were close to the reference foam. All foams meet the requirements of ASTM C1029. Even the small difference in volume change between the reference foam vs. Mod. A for instance can be easily overcome when moving to a more practical spray foam formulation.

Table 6: Dimensional stability at 90% RH at 70°C

Dimensional Stability 95% RH @ 70°C	Ref.	Mod. A 10% TF94-806	Mod. B 20% TF94-2812E
7 days mass change, %	-1.2	-1.4	-1
7 days volume change, %	2.5	5.6	3.8

CONCLUSIONS

This research paper introduces Resonance™ triazine polyols as a novel fire retardant polyol in rigid polyurethane foam. Bakelite's highly aromatic, multi-functional TF94-806 and TF94-2812E were evaluated for their performance in PU Rigid foam systems by replacing 10-20% of sucrose-based polyol. The paper also reported their solubility in various polyols and reactive diluents and their chemical compatibility with HFOs.

Two different RTPs were used for this study. TF94-806 is a commercially available polyol, and the TF94-2812E is an experimental product used primarily to conduct this scientific study. TF94-806 was dissolved in various polyols and diluents, resulting in flowable liquids of varying viscosities, which demonstrates its easy incorporation into PU systems. This characteristic should also remain the same or get better with TF94-2812E. The TF94 polyols were also chemically compatible with HFOs, especially in the concentration range designed to be used in a formulation.

Merely replacing 10% of the sucrose-based polyol with TF94-806 resulted in significant improvement in fire performance, as shown by the UL-94 vertical burn test. Both Mod. A and Mod B. foams containing RTPs achieved a V-0 rating, whereas the reference foam failed to achieve even V-1. The sucrose replacement and aromaticity doubled with Mod. B compared to Mod. A. However, the total flame time and the mass retention remained essentially the same for both. This phenomenon shows that the nitrogen content of these foams dictates the reaction to fire performance more than the percent aromaticity because both these modifications were designed to have similar total nitrogen content.

The introduction of RTPs increased the system's reactivity, which necessitated the reduction in the quantities of catalysts used. Since there is a need, particularly in the spray foam industry, to reduce emissions from volatile amine catalysts, Polycat 8 was adjusted to match the reaction profile of the reference. This resulted in ca. 50% reduction of Polycat 8 with only 3% of TF94-806, and almost 60% reduction with 6% TF94-2812E. Unlike traditional catalysts, the RTPs react with isocyanate and get integrated into the PU backbone and, therefore, do not contribute to emissions. Thus, they help reduce the dependence on the emissive catalyst and improve the catalytic efficiency of the system.

Overall, the mechanical properties such as compressive strength and dimensional stability of the two RTF foams matched the reference. Due to the increase in system aromaticity, the compressive strength of Mod. B containing TF94-2812E foam did increase significantly compared to reference and Mod. A foam. The dimensional stability data met the requirement of the ASTM C1029 standard in the PU industry.

In essence, Resonance™ triazine polyols could unlock simplified and enhanced foam formulations in rigid polyurethanes by reducing the amount of fire retardant and emissive catalysts, and positively influencing other critical properties of the foams in a variety of applications. As these can be an excellent fit in spray foams, the ongoing future work will focus on demonstrating the same effect in a typical spray foam formulation.

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BIOGRAPHIES

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Zhikai holds a master's in material science and a doctoral in polymer physical chemistry. He is now a Sr. Scientist at Bakelite Synthetics, working in the fields of analytical testing and material characterization, structure-property-processing relationship, material failure analysis, and materials' modeling and simulation**.**

Steve Crain
Steve Crain was a Business Development Leader in Bakelite Synthetics. He received his BS degree from Michigan State University in 1983. He has conducted product and process research and development in styrenics and urethanes for over thirty years. Steve has worked in several urethane related markets including thermoplastic polyurethane, isocyanurate board stock and spray polyurethane insulation for the last 23 years.