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Formacel 1100: Spray polyurethane foam application development

DuPont is expanding the development of Formacel 1100 (HFO-1336mzz-Z) as a unique zero ODP, low GWP, non-VOC, non-flammable foam expansion agent that provides a balanced option for environmental sustainability, excellent performance and economic conversion. Formacel 1100 is being launched as a liquid foaming agent offering outstanding insulation performance required in building walls, roofs, appliances and refrigerated containers. This paper will discuss continued research efforts and customer developmental activities to explore and understand the key variables involved with delivering optimum performance in spray polyurethane foam applications. A number of advanced analytical tools, such as ^{19}F NMR, were used to couple theoretical models to actual spray foam behavior including k-factor aging and efficiency.

1. Introduction

Energy efficiency continues to be an important global sustainability theme as growing populations and diminishing natural resources drive fuel costs higher. As fossil fuels comprise a significant portion of energy production, the implications of CO_2 emissions and the subsequent impact on climate change play an important role in driving change and improvements in energy efficiency through greenhouse gas reductions [1]. A powerful tool for enabling such reductions involves the insulation of buildings with closed-cell polyurethane spray foam. When properly integrated into high performance building envelope designs, closed-cell spray polyurethane foams can deliver exceptional insulation performance that can translate into significant reductions in energy consumption and subsequent CO_2 emissions over the life of the structure. These foams are especially effective when fluorocarbon gases are used as the foam expansion agents (blowing agents), due to their high relative molecular weight and corresponding insulating properties. However, the predominating fluorocarbon blowing agents, HFC-245fa and HFC-365mfc, face environmental challenges due to their contribution to climate change based on their high global warming potentials (GWPs). Thus a need exists for high performance fluorocarbon gases with low GWP and zero Ozone Depletion Potential (ODP) to support the polyurethane spray foam industry.

In response, the DuPont Company has developed Formacel 1100, a new form of hydrofluorocarbon which contains a double bond that greatly reduces its lifetime in the atmosphere. As a hydrofluoroolefin (HFO), Formacel 1100 provides improved environmental properties (zero ODP and low GWP) while maintaining desired foam expansion agent characteristics for spray foam such as excellent formulation stability, suitable boiling point, low vapor thermal conductivity and non-flammability. In addition, Formacel 1100 is expected to be exempt from Volatile Organic Compound (VOC) regulations. In support of the continued commercial introduction of this product, this paper focuses on general foaming behavior as it pertains to spray foam applications and diffusion behavior once incorporated into a spray foam installation.

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Tab. 1: *Formacel 1100 toxicological assessments*

Test	Results
ALC and LC_{50}	*Very low acute toxicity*
Skin irritation	*Non-irritating*
Mutagenicity Ames	*Non-mutagenic*
Chromosomal aberration	*No genetic material damage when tested in bacterial and mammalian cell cultures*
Cardiac sensitization	*Favorable cardiac sensitization potential profile*
28 day repeated inhalation	*Favorable repeated inhalation profile*
90 day/developmental	*Favorable repeated inhalation profile consistent with 28 day test*

2. Environmental and toxicity properties

As with any component of a domestic or commercial building, the safety and environmental properties of the foam expansion agent is important, both from application and service perspectives. Formacel 1100 ($CF_3CH{=}CHCF_3$) contains no chlorine or bromine atoms and hence is characterized by a zero ODP [2]. Also, with its double bond, it demonstrates a short atmospheric lifetime of 22 days and a low GWP of 2 [3]. The Maximum Incremental Reactivity (MIR) of Formacel 1100 is 0.04, only 14 % of that of ethane [4]. Given such a low reactivity, it is expected that Formacel 1100 will be exempted from VOC regulations and the use of Formacel 1100 will be beneficial in reducing VOC emissions.

Formacel 1100 is characterized by very low acute and chronic inhalation toxicity. No adverse mutagenic or reproductive effects have been observed **(tab. 1).**

As a consequence of its highly favorable toxicity profile, the DuPont Acceptable Exposure Limit (AEL) for Formacel 1100 is 500 ppm (8 h and 12 h time-weighted average, TWA). **Table 2** compares the exposure limit of Formacel 1100 with commercially available foam expansion agents.

3. Physical properties

Spray foam applications require non-flammability due to the relatively large volumes of material that are volatilized in confined spaces. Formacel 1100 is non-flammable based on testing in accordance with ASTM E 681-09 "Standard Test Method for Concentration Limits of Flammability of Chemicals (Vapors and Gases)" carried out at temperatures of 60 °C and 100 °C.

Formacel 1100 is characterized by a boiling point and vapor pressure that are very suitable to polyurethane spray foam processing technology. Formacel 1100 has a vapor pressure very close to that of HCFC-141b and HFC-365mfc, therefore it can be used at the optimal foam expansion agent level in formulations, providing desired foam properties without the concern for high B-side pressure or flammability. Formacel 1100 has low vapor thermal conductivity over a broad temperature range, contributing toward excellent foam insulation performance.

Tab. 2: *Comparison of Formacel 1100 exposure limit with commercial foam expansion agents*

Blowing agent	**TLV, OEL or AEL* / ppm**
Formacel 1100	500
HCFC-141b	500
HFC-245fa	300
HFC-365mfc	1,000
Methyl formate	100

*DuPont Acceptable Exposure Limits (8–12 h TWA)

4. Comparison of Formacel 1100 with other zero ODP foam expansion agent options

Formacel 1100 provides a balanced option for polyurethane spray foam applications. **Table 3** compares the properties of Formacel 1100 to other zero ODP foam expansion agent options. Formacel 1100 is the only foam expansion agent that provides improved environmental properties (zero ODP and low GWP) while maintaining the desired characteristics of HCFC-141b: non-VOC, suitable boiling point, low vapor thermal conductivity and non-flammability.

5. General reaction characteristics study (DoE)

5.1 Rationale

By definition, polyurethane foam chemistry is very complicated involving the interactions of several dependent and independent variables, working in tandem, to deliver a broad range of attributes and performance criteria. Making a substitution of one foam expansion agent for another can impact a number of these variables in unpredictable ways, owing to multiple factors including boiling point, solvent characteristics, heats of evaporation, molecular weight, etc. As a result, we chose to explore the main effects and interactions of the variables via a Design of Experiments (DoE) approach. From this study we could identify the most important variables and their interactions in order to design the most effective optimization strategies.

5.2 Design

The Center for the Polyurethanes Industry (CPI) medium density generic spray formulation was used as the basis for this study and consists of the recipe given in **table 4.**

Based on numerous experiments from the lab and field which indicated a potential for significant impact, six factors were selected as the initial exploration variables for the B-side. In order to identify an area of operability (i. e. avoiding ranges where poor or no foam formation is expected), the ranges shown in **table 5** were selected.

Let X_1 through X_6 denote the pbw of each of the six components (blow catalyst 1, gel catalyst, surfactant, water, Formacel 1100,

Tab. 3: *Comparison of Formacel 1100 with commercially available foam expansion agents*

Property	**Formacel 1100**	**HCFC-141b**	**HFC-245fa**	**HFC-365mfc**	**Methyl formate**
Molecule structure	$CF_3CH=CHCF_3$	CCl_2FCH_3	$CF_3CH_2CHF_2$	$CF_3CH_2CF_2CH_3$	$HCOOCH_3$
Molecular weight	164	117	134	148	60
Boiling point / °C	33	32	15	40	32
ODP	0	0.11	0	0	0
GWP / 100 y ITH AR5	2	782	858	804	<25
VOC	No*	No	No	No	No
Exposure limits / ppm	500**	500	300	1,000	100
Flammability	No	No	No	Yes	Yes
Vapor thermal conductivity (at 25 °C) / mW/(m·K)	10.7	9.7	12.7	10.5	10.7

*Expected based on low MIR value; **DuPont Acceptable Exposure Limits (8–12 h TWA)

Tab. 4: *The Center for the Polyurethanes Industry (CPI) medium density generic spray formulation*

Component	**Amount**
A side	
MDI polyisocyanate	100 %
B side	
Aromatic polyester polyol	52 pbw
Aromatic amino polyether polyol	48 pbw
Blow catalyst 1	1.0 pbw
Blow catalyst 2	0.43 pbw
Gel catalyst	3.70 pbw
Silicone surfactant	1.43 pbw
Flame retardant (TCPP)	22.7 pbw
Water	3 pbw
Formacel 1100	15 pbw

and isocyanate index). The property Y of interest was modelled by second order and interaction terms:

$$\begin{aligned}Y = {} & \beta_0+\beta_1X_1+\beta_2X_2+\beta_3X_3+\beta_4X_4+\beta_5X_5+\beta_6X_6+\\ & \beta_7X_1^2+\beta_8X_2^2+\beta_9X_3^2+\beta_{10}X_4^2+\beta_{11}X_5^2+\beta_{12}X_6^2+\\ & \beta_{13}X_1{\cdot}X_2+\beta_{14}X_1{\cdot}X_3+\beta_{15}X_1{\cdot}X_4+\beta_{16}X_1{\cdot}X_5+\\ & \beta_{17}X_1{\cdot}X_6+\beta_{18}X_2{\cdot}X_3+\beta_{19}X_2{\cdot}X_4+\beta_{20}X_2{\cdot}X_5+\\ & \beta_{21}X_2{\cdot}X_6+\beta_{22}X_3{\cdot}X_4+\beta_{23}X_3{\cdot}X_5+\beta_{24}X_3{\cdot}X_6+\\ & \beta_{25}X_4{\cdot}X_5+\beta_{26}X_4{\cdot}X_6+\beta_{27}X_5{\cdot}X_6\end{aligned}$$

The model has 28 unknown coefficients. To allow for goodness of fit of the model to be assessed, we replicated the centre point five times.

A response surface DoE was generated, however, because of the constraints and large number of factors, a computer program was used in generating an I "optimal" design, so that, with a minimal number of experimental points, the desired amount of information can be extracted from the data. The resultant DoE had 38 runs **(tab. 6)**.

Once the experimental runs are determined, foams are prepared and tested for the properties of interest. This leads to an extensive, experimentally generated dataset of foam compositions and properties. Through statistical regression analysis, mathematical relationships between properties and composition are determined.

5.3 Experimental

A rigorously controlled hand mixed operation was chosen for populating the DoE. The B-side formulation was cooled to 10 °C before premixing with foam expansion agent and A-side (polyisocyanate). The components were mixed at 4,000 min^{-1} for 2 s before addition to suitable vertical moulds. Rise time and tack free time were recorded, and then the foams were allowed to stand for 24 h at room temperature (25 °C/78 °F) before cutting and characterization via the following methods:

Density	ASTM D1622
Thermal conductivity	ASTM C518
Closed cell content	ASTM D6226

Upon cutting into suitable squares for thermal conductivity testing (skinned), the samples were stored under ambient conditions for two months and tested at one month intervals.

5.4 Results and analyses

During the testing regimen, some runs at extreme levels of high and low foaming agents displayed poor physical properties (e. g. sample no. 3). **Table 7** illustrates some of the raw data obtained from these runs.

A backward elimination multiple regression approach was used to establish the mathematical relationships between the amounts of different ingredients and corresponding properties. These equations include the most significant ($p>0.15$) quadratic or interaction terms of the ingredients, maintaining hierarchy in the model. The statistical summary parameters **(tab. 8)** demonstrate that the models describe the data accurately (high R^2_{adj}). For tack free time, one month thermal property at 24 °C (75 °F), lower R^2 was observed due to larger experimental errors associated with these properties.

▼ **Tab. 5:** *Parameter sets for DoE*

Factor	Range
Blow catalyst 1*	0.15–0.34 pbw
Gel catalyst	1.3–2.9 pbw
Silicone surfactant	0.5–3.0 pbw
Water**	0.5–2.5 pbw
Formacel 1100**	6–45 pbw
Isocyanate index	105–130

* *Blow catalyst 2 used in 2.32-fold amount of blow catalyst 1*
** *With constraints to manage densities from 32–64 kg/m³*

▼ **Tab. 6:** *Partial overview of experimental runs*

Sample	Aromatic polyester polyol / pbw	Aromatic amino polyether polyol / pbw	Blow catalyst 2 / pbw	Blow catalyst 1 / pbw	Gel catalyst / pbw	Silicone surfactant / pbw	TCPP / pbw	Water / pbw	Formacel 1100 / pbw	Isocyanate index
1	52	48	0.58	0.25	2.10	1.75	22.7	1.54	26.53	117
2	52	48	0.23	0.10	2.90	3.00	22.7	1.50	6.00	130
3	52	48	0.93	0.40	2.90	1.75	22.7	2.00	45.00	130
4	52	48	0.23	0.10	1.30	0.50	22.7	1.60	6.00	130
⋮										
37	52	48	0.93	0.40	2.90	0.50	22.7	2.30	22.05	104
38	52	48	0.58	0.25	2.10	1.75	22.7	1.54	26.53	117

▼ **Tab. 7:** *Partial overview of experimental run data*

Sample	Cream time / min	Rise time / min	Tack free time / min	Density / kg/m³	Thermal performance (initial) at 24 °C (75 °F) / mW/(m·K)	Thermal performance (initial) at –7 °C (20 °F) / mW/(m·K)	Average closed cell / %	Thermal performance (aged one month) at 24 °C (75 °F) / mW/(m·K)
1	0.02	0.25	0.25	37.8	17.8	17.8	93.70	19.5
2	0.02	0.20	0.20	66.3	21.5	19.1	92.31	23.5
3	0.02	0.22	0.28	30.8	18.6	19.6	Heavy shrinkage	
4	0.02	0.28	0.28	60.9	22.1	19.9	85.43	25.2
⋮								
35	0.02	0.35	0.35	33.6	18.0	17.5	98.93	18.8
36	0.02	0.27	0.27	34.8	17.4	17.2	80.82	18.3
37	0.02	0.13	0.13	34.9	18.7	17.6	94.64	19.5
38	0.02	0.17	0.17	40.2	17.4	17.3	98.53	18.1

5.4.1 Discussion of key output measurements

The following attributes were examined with respect to the six factors: rise time, tack free time, density, initial thermal performance at 24 °C (75 °F) and at –7 °C (20 °F), one month aged thermal performance at 24 °C (75 °F) and at –7 °C (20 °F).

5.4.1.1 Rise time

Figure 1 shows the main effects plot impacting rise time indicating a strong dependence on both catalyst levels with a linear decrease of rise time with the blow catalysts and a decrease with quadratic character for the gel catalyst. This is consistent with the concept that more catalyst should result in faster reaction times, more heat evolution, and subsequently a quicker evaporation and rise of the physical blowing agent, Formacel 1100, with a boiling point of 33 °C. The concentration of the silicone surfactant had a slight impact on the rise time; however the most predominant factor was Formacel 1100 concentration which increased rise time as a function of higher loadings. This is consistent with the fact that physical blowing agents require heat and time to evaporate, thus having a greater mass of Formacel 1100 in the system requires more time to evaporate and rise, all things being equal.

Tab. 8: *Overview of the statistical parameters evaluating the quality of the statistical models*

Parameter	R^2_{adj} / %
Rise time	88.46
Tack free time	80.06
Density	95.89
Initial TP at 24 °C (75 °F)	94.97
One month TP at 24 °C (75 °F)	83.02
Initial TP at –7 °C (20 °F)	75.53
TP = thermal performance	

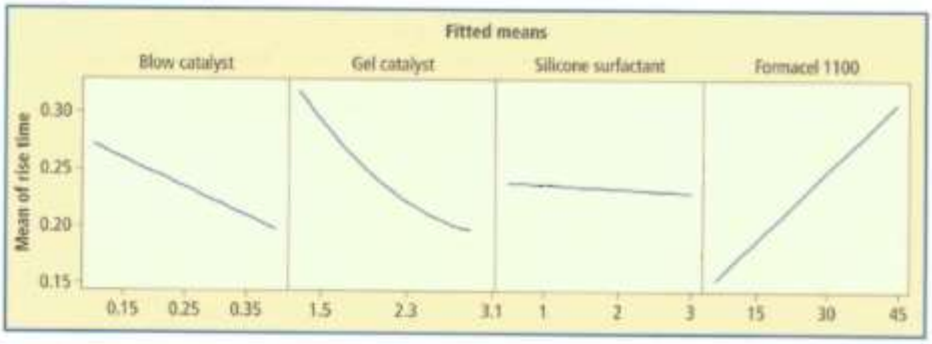


Fig. 1: *Main effects plot for rise time*

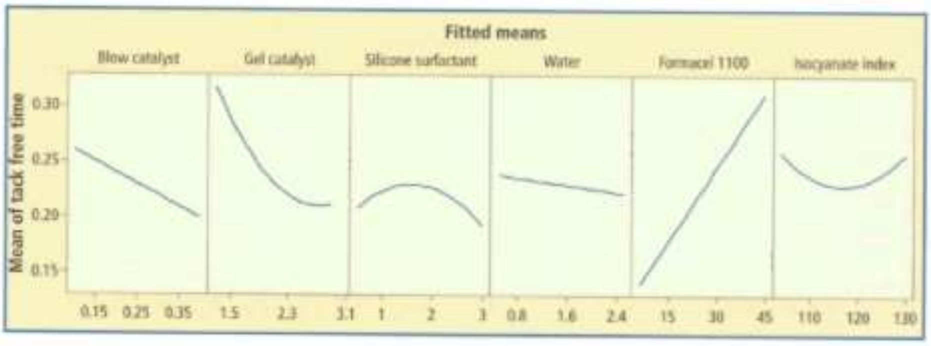


Fig. 2: *Main effects plot for tack free time*

Implications for formulations: as with any physical blowing agent, this data suggests balancing the amounts of Formacel 1100 and catalysts are necessary for controlling desired rise times.

5.4.1.2 Tack free time

As with rise time, the main effects and interaction plots **(fig. 2)** for tack free time illustrated a strong dependence on both catalyst levels and decrease of tack free time with the blow catalysts showing linear behavior whereas gel catalyst has significant quadratic effect. Again this is consistent with the concept that more catalyst should result in faster reaction times with more heat evolution. The concentration of the silicone surfactant had a more pronounced impact on tack free time with a positive quadratic effect. As with rise time, the most predominant factor was Formacel 1100 concentration which increased tack free time with higher loadings, again correlating with the heat and time required to evaporate the physical blowing agent. The isocyanate index also had a negative quadratic effect.

Figure 3 illustrates some important interactions between some of the factors. Water showed an impact on tack free time when interacting with both of the catalysts, Formacel 1100 and isocyanate index. At low catalyst or Formacel 1100 levels, with increase in amount of water, the mean tack free time increased slightly whereas at high catalyst or Formacel 1100 levels, increasing amount of water decreased the tack free time. This may be related to additional CO_2 formation lengthening the expansion phase under low Formacel 1100 conditions and slower expansion times with less catalyst. Conversely, with high amounts of Formacel 1100, the additional water may increase the heat of reaction, thus shortening the expansion phase, as would be the case with higher catalysts loadings. However, at low isocyanate index level, increasing amount of water decreased tack free time and at high isocyanate index level, increasing amount of water increased tack free time.

Implications for formulations: this data suggests balancing the amounts of Formacel 1100, catalysts, water, silicone surfactant, and isocyanate index are critical for controlling desired tack free times.

5.4.1.3 Density

Figure 4 illustrates the main effects and interactions impacting density. The blow catalyst, gel catalyst, and silicone surfactant levels all play a minor role. Both the water level and the Formacel 1100 level showed the strongest impact, as expected being the major means of controlling density through gas expansion. Both, however, illustrated a non-linear plateau effect at highest levels where the limits of density reduction occur due to increased open cell content and dimensional stability problems.

Implications for formulations: this data suggests balancing the amounts of Formacel 1100 and water are critical for controlling desired density.

5.4.1.4 *Initial k-factor measured at 24 °C (75 °F)*

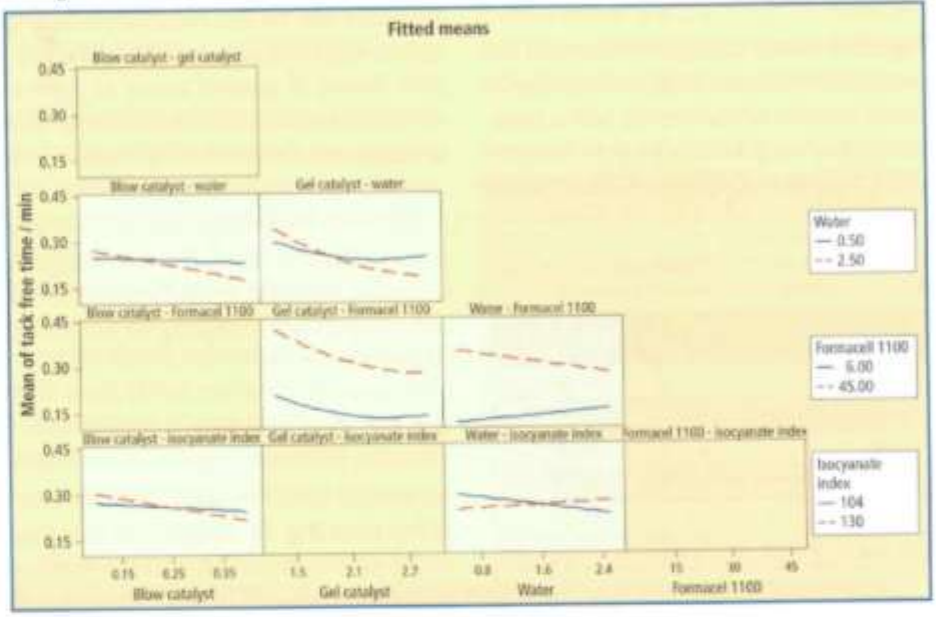
For the main effects and interactions impacting initial thermal conductivity performance measured at 24 °C (75 °F), **figure 5** shows the blow catalyst, gel catalyst, and silicone surfactant levels have small influence. Increasing water levels led to somewhat higher k-factors, potentially due to the higher thermal conductivity of carbon dioxide released during the blow reaction. The most significant impact on thermal conductivity results from Formacel 1100 concentrations with a dramatic decrease in k-factors at increasing levels. This reaches a maximum impact around 38 pbw before beginning to reverse. This is consistent with the superior insulating properties of the Formacel 1100 molecule compared to carbon dioxide and air.

Implications for formulations: this data illustrates the powerful insulating impact of Formacel 1100 as a component in rigid foams, coupled with an optimized level of water.

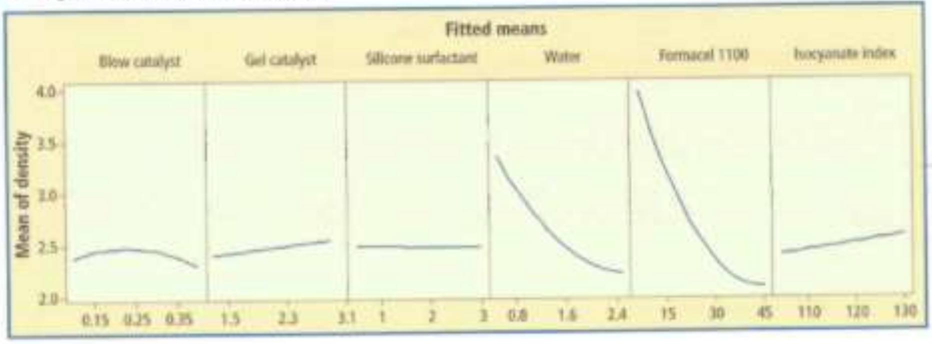
5.4.1.5 *Initial k-factor measured at –7 °C (20 °F)*

In contrast to the 24 °C (75 °F) initial thermal conductivity, **figure 6** illustrates the main effects and interactions impacting initial thermal conductivity performance measured at –7 °C (20 °F) with the blow catalyst, gel catalyst, and silicone surfactant levels having a more pronounced influence. Also in contrast to the 24 °C (75 °F) case, increasing water levels lead to somewhat lower k-factors, possibly from mitigation of condensation of Formacel 1100. The most significant impact on thermal conductivity results from Formacel 1100 concentrations with a dramatic decrease in k-factors at increasing levels until a local minimum around 28 pbw, followed by a rapid increase toward 45 pbw. This is most likely a direct result of the condensation effect of the higher boiling Formacel 1100. This has a detrimental impact on thermal insulation properties as liquid materials tend to conduct heat far more efficiently than gases. Lastly, the isocyanate index also had an impact on thermal conductivity in a positive quadratic response. There were some samples that displayed poor performance at –7 °C (20 °F) as indicated by the last panel collapse at 24 °C (75 °F).

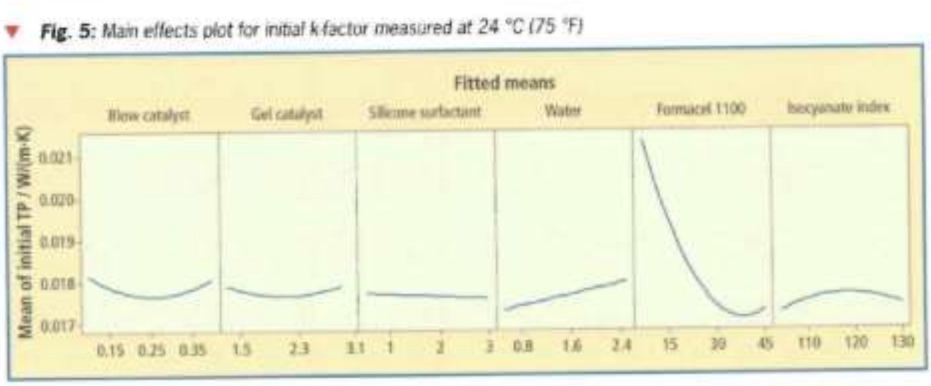
▼ **Fig. 3:** *Interaction plot for tack free time*



▼ **Fig. 4:** *Main effects plot for density*

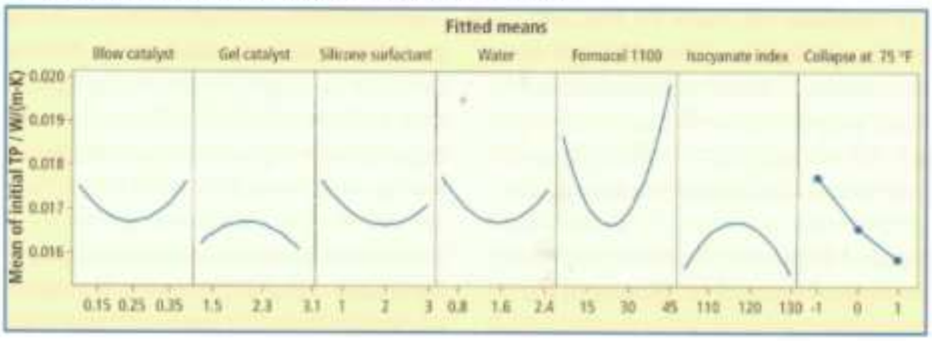


▼ **Fig. 5:** *Main effects plot for initial k-factor measured at 24 °C (75 °F)*

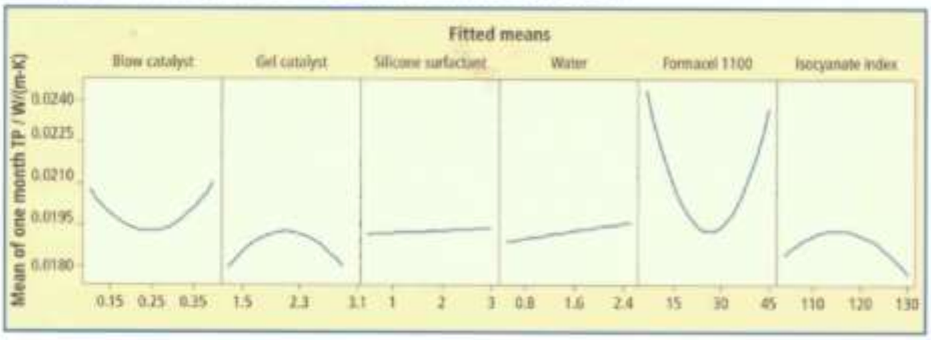


Implications for formulations: this data illustrates the increased importance of optimizing water, catalyst, and surfactant levels in supporting the powerful insulating impact of Formacel 1100 as components in rigid polyurethane foams.

▼ **Fig. 6:** *Main effects plot for initial k-factor measured at −7 °C (20 °F)*



▼ **Fig. 7:** *Main effects plot for one month k-factor measured at 24 °C (75 °F)*

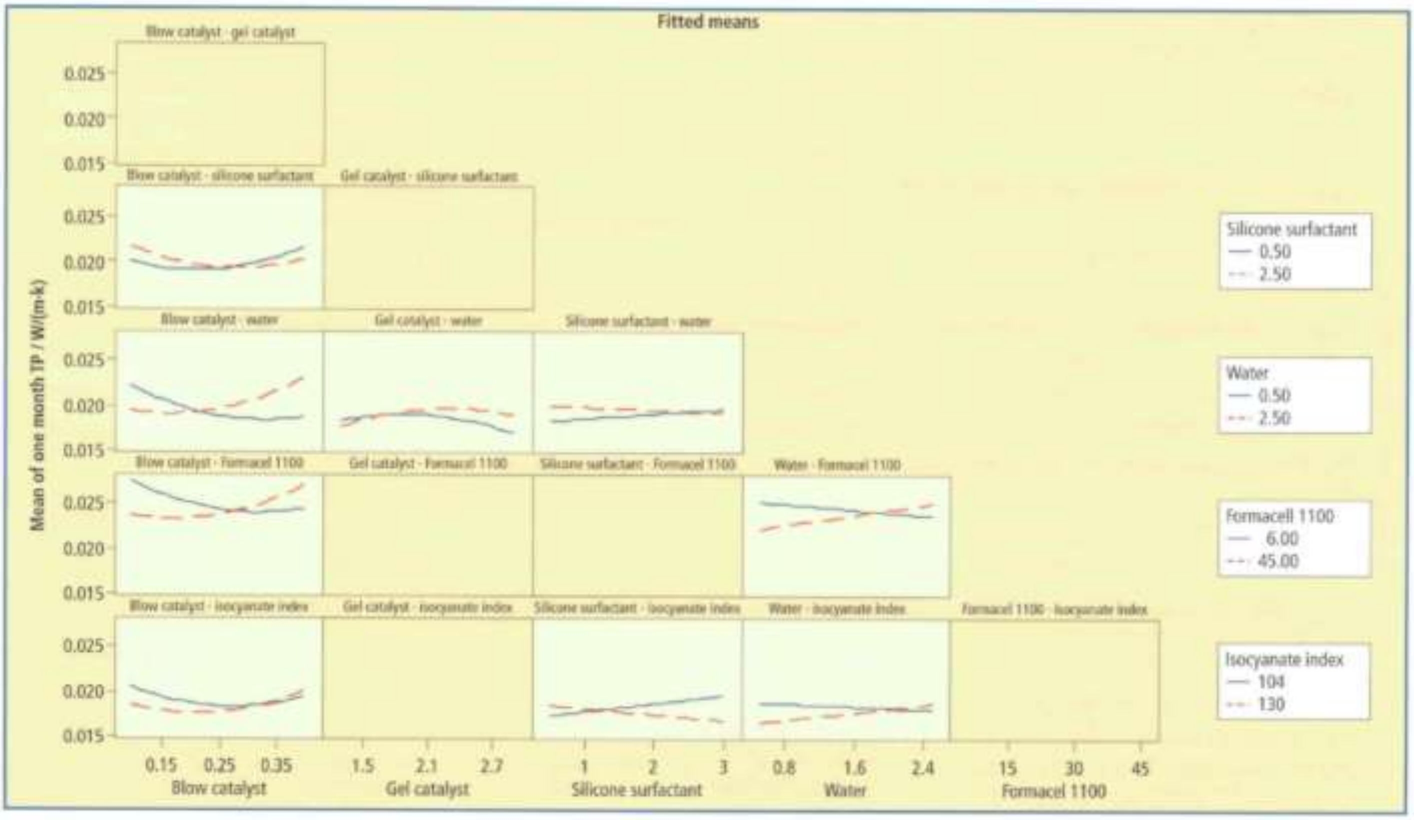


5.4.1.6 One month k-factor measured at 24 °C (75 °F)

In the case of one month room temperature aged foams, **figure 7** illustrates a more dramatic impact of blow catalysts and gel catalyst than the initial numbers with negative and positive quadratic behavior respectively. The silicone surfactant still had little impact. The most significant impact on thermal conductivity results from Formacel 1100 concentrations with a dramatic decrease in k-factors at increasing levels until a local minimum around 28 pbw, followed by a rapid increase toward 45 pbw. This differed from the initial values and resembled the −7 °C (20 °F) case. Lastly, the isocyanate index also had an impact on thermal conductivity in a positive quadratic fashion.

Figure 8 shows the interactions among the factors for one month aged 24 °C (75 °F) thermal performance. Water interacted significantly with both of the catalysts, silicone surfactant, Formacel 1100 and isocyanate index. At low levels of either catalyst or

▼ **Fig. 8:** *Interaction plot for one month k-factor measured at 24 °C (75 °F)*



Formacel 1100 or isocyanate index, with increase in amount of water, the mean thermal performance of the aged foams decreased, whereas at high levels, increasing amount of water increased the mean thermal performance. Clearly water can play multiple roles in these reactions, both positive and negative with impact on thermal performance.

Implications for formulations: As shown in the initial thermal performance examples, this data illustrates the increased importance of optimizing water, catalyst, and surfactant levels in supporting the powerful insulating impact of Formacel 1100 as components in rigid polyurethane foams.

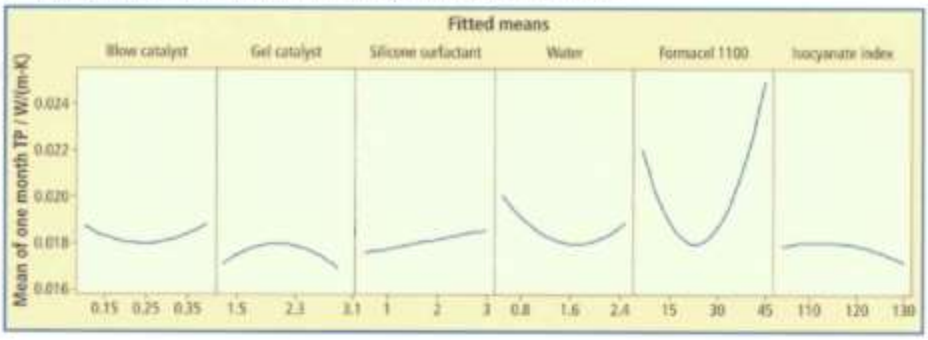
5.4.1.7 One month k-factor measured at –7 °C (20 °F)

In the case of one month room temperature aged foams tested at –7 °C (20 °F) for thermal performance, **figure 9** illustrates the impact of blow catalyst and gel catalyst showing a slight negative and positive quadratic behavior respectively. The silicone surfactant had a slight negative impact at higher levels. The most significant impact on thermal conductivity results from Formacel 1100 concentrations with a dramatic decrease in k-factors at increasing levels until a local minimum around 28 pbw, followed by a rapid increase toward 45 pbw. Water also showed a similar minimum behavior at levels around 1.7 pbw. The isocyanate index also had a slight impact on thermal conductivity in a positive quadratic fashion.

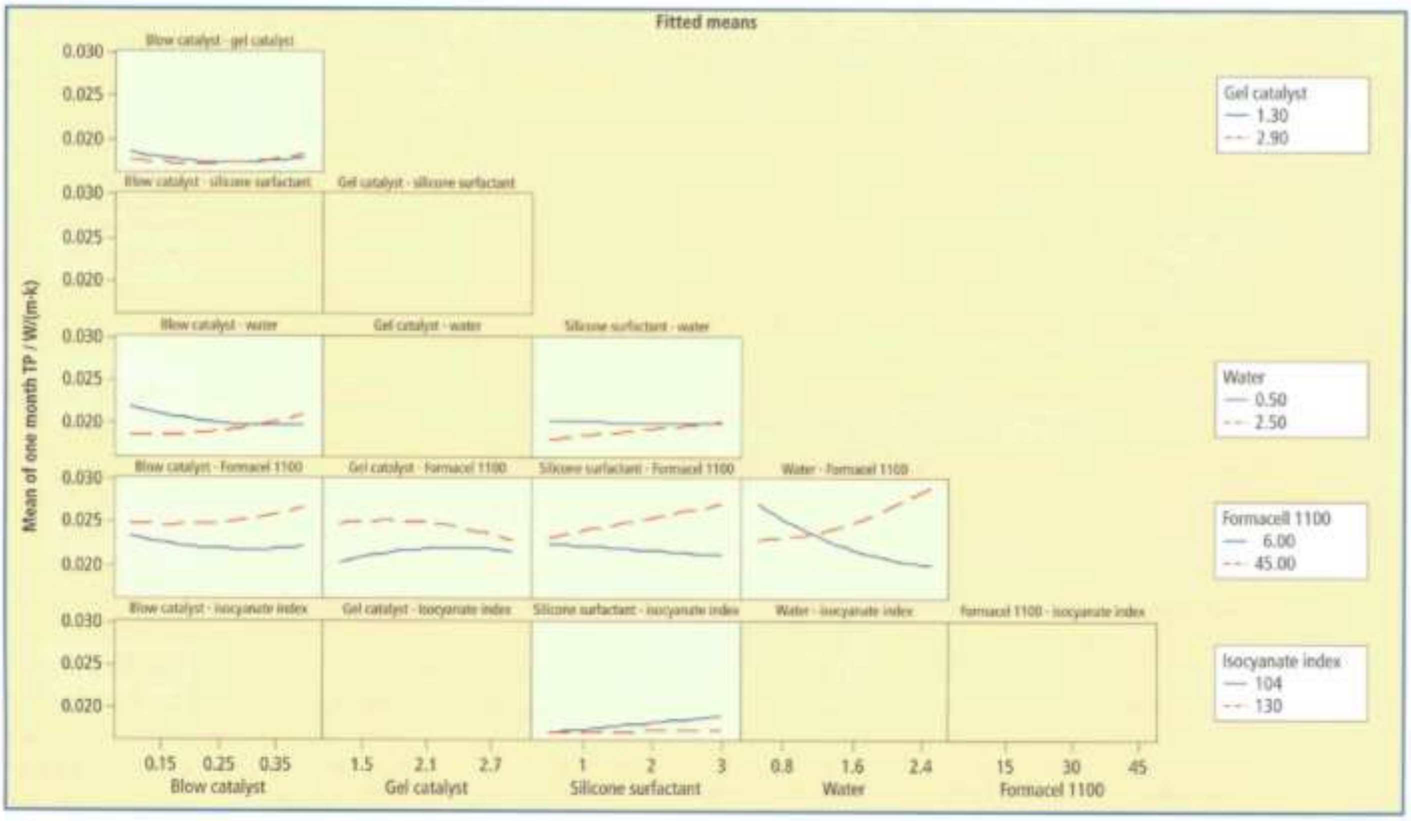
Figure 10 illustrates the interactions among the factors for one month aged –7 °C (20 °F) thermal performance. Water interacted significantly with both of the catalysts, silicone surfactant, and Formacel 1100. At low levels of either catalyst or Formacel 1100, with increase in amount of water, the mean thermal performance of the aged foams decreased, whereas at high levels, increasing amount of water increased the mean thermal performance. Clearly water can play multiple roles in these reactions, both positive and negative with impact on thermal performance.

Implications for formulations: As shown in the initial thermal performance examples, this data illustrates the increased importance

▼ ***Fig. 9:*** *Main effects plot for one month k-factor measured at –7 °C (20 °F)*



▼ ***Fig. 10:*** *Interaction plot for one month k factor measured at –7 °C (20 °F)*



of optimizing water, catalyst, and surfactant levels in supporting the powerful insulating impact of Formacel 1100 as components in rigid polyurethane foams.

5.4.2 Summary of implications for formulation developments

Taken as a whole, the DoE provides a number of insights concerning the most important factors influencing spray polyurethane foam formation and end-use performance. Among these are the Formacel 1100 levels, water levels, and catalyst levels with isocyanate indexes and surfactant levels also playing an important role. The interactions are complex and often drive the desired outputs in opposite directions, however, clear trends emerge which can help guide optimization efforts with this new foam expansion agent. The key learning from the DoE is that simply dropping in one foam expansion agent for another may not offer the best performance in a specific system due to the multiple interactions that can occur from important factors.

Of course, application variables can play a significant role in modifying foam formation and subsequent physical and performance attributes. Selection of the number and thickness of lifts can dramatically influence density, cell structure, cell-gas composition, and diffusion behavior. In addition, other factors not explored in this DoE such as selection of polyols, polyisocyanate, surfactants, catalysts, etc. can impact foam properties in a major fashion. Overall, any reformulation or improvement strategy should involve a holistic approach considering all key aspects of foam production and performance.

6. Cell diffusion behavior

6.1 Background

As noted in the introduction, fluorochemical gases play a critical role in enabling high performance thermal insulation behavior. As such, long term retention of these gases in the insulation matrix is essential for maintaining the value proposition of enduring insulation performance. Historically, the retention behavior of gases is estimated by determining the diffusion constant in the matrix of interest and extrapolating over time. A number of methods have been used and published to obtain such information ranging from direct measurements of diffusion rates across a membrane of polyurethane to destructive cell gas analyses of foam samples over time via infrared and gas chromatography [7–11]. In the case of the membrane method, the nature of the polyurethane may not match well with what is actually created dynamically on a microscale in foams. The cell gas method improves on this, but introduces complexities with sampling and reproducibility. In this section we chose to study the diffusion behavior of the Formacel 1100 in foam samples in situ via a new ^{19}F NMR quantification method. The benefits of such a method include easy sample preparation, analysis and the ability to monitor behavior in situ repeatedly of the same samples.

6.2 Model system

The model system chosen for the initial diffusion studies was based on a modified version of the CPI generic spray foam formulation as presented in chapter 5, the exception being a higher loading of blowing agent that was used to facilitate a stronger response factor for quantification (at 20 % more). Foams were prepared via the same procedure outlined in chapter 5 with the required quantity of blowing agent for the target density of 2.5 pcf (40 kg/m^3).

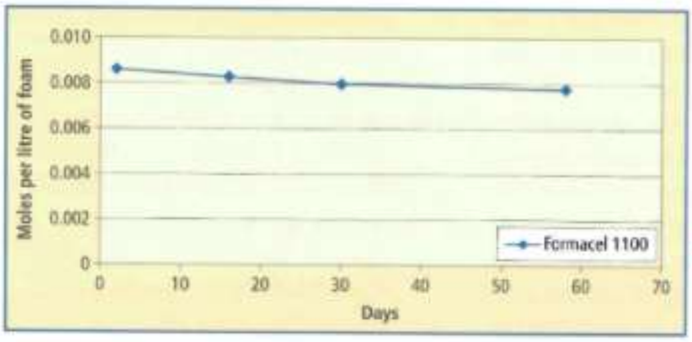


Fig. 11:
^{19}F NMR quantification of Formacel 1100 in foam samples

6.3 ^{19}F NMR introduction

Nuclear magnetic resonance (NMR) spectroscopy is a penetrative, non-destructive analytical technique that can be used to identify and quantify analytes of interest, and also provide information about their physical state [10]. The sensitivity, and to some extent the informativeness, of the technique depends on the observed nucleus. At natural isotopic abundance, the ^{19}F nucleus is detected with sensitivity second only to ^{1}H, and with much greater spectral dispersion, rendering ^{19}F NMR applicable to an especially wide array of systems [11]. It has previously been applied to study fluorinated blowing agents in a polyurethane foam matrix, using "magic angle" spinning to remove broadening from dipolar couplings [12], and it has been used in imaging experiments to determine the diffusion rates of fluorinated blowing agents in polyurethane and polystyrene foams [13]. Here we report the first use of ^{19}F NMR in the study of static foam samples to quantify the blowing agents retained therein, as a function of aging.

6.4 Experimental

Foam samples were prepared by coring approximately 8.9 mm outer diameter (o. d.) cylinders from a larger block. Samples thus prepared were cut to 5.1 cm length and inserted into a cylindrical (10.0 mm o. d.) borosilicate glass NMR tube. NMR spectra were acquired on a Varian 700 MHz VNMRS spectrometer equipped with a 10 mm F, H probe. For the purposes of absolute quantitation, an external standard was prepared containing a known pressure (0.116 bar) of hexafluoroethane. Spectra were acquired of this standard under identical conditions as those used with the foam samples. The pressure of hexafluoroethane was converted to molar concentration by use of the ideal gas law ($n/V=P/RT$).

6.5 Results

The foam samples were stored under ambient conditions without confinement between analyses. Spectra were obtained after 2, 16, 30, and 58 days. Based on the external hexafluoroethane standard method, **figure 11** provides the quantification of the Formacel 1100 concentration as a function of time (in moles per litre of foam).

6.6 Analysis and coefficient calculation

In this work the effective diffusion coefficient was determined for the foam blowing agent in polyurethane foam cells. The fundamental experimental data available are the concentrations of the blowing agent as a function of time. Using this data a flux (J) was determined. This flux was plotted against the concentration gradient, dc/dz to determine the slope, from which the diffusion coefficient as defined by Fick's Law [14] was determined **(eq. 1)**.

$$J = -D\frac{dc}{dz} \tag{1}$$

Where dc is the molar concentration and dz is the distance from the centre of the foam cell to the wall. In this work the diameter of the cell was 300 µm based on SEM determinations.

6.7 Discussion

The calculated diffusion coefficient value of $1.89\cdot10^{9}$ cm²/s at 25 °C compares favorably with the literature values obtained for CFCs, HCFCs and HFCs:

CFC-11:
  $3.94\cdot10^{9}$ cm²/s to $2.43\cdot10^{7}$ cm²/s
  (depending on polyurethane) [8]
HCFC-141b:
  $5.00\cdot10^{9}$ cm²/s to $1.90\cdot10^{8}$ cm²/s
  (different methods) [7, 9, 10]
HFC-245fa:
  $3.90\cdot10^{9}$ cm²/s to $5.44\cdot10^{8}$ cm²/s
  (different methods) [9–11]

As the reference data illustrates, there is a significant impact of the polyurethane composition and preparation on the diffusion rates. In addition, our own studies indicate the diffusion rates change somewhat as a function of the age of the foam, with a significant impact occurring during the first couple of months of aging, possibly due to continued curing, super saturation of foaming agent, and pressure equalization with CO_2/air exchange.

Taken in context of these variables and previous studies, the preliminary assessment of the diffusion behavior of Formacel 1100 would indicate a favorable profile as a long term insulating component in high performance foam applications. The larger molecular cross-section, coupled with a low vapor pressure and moderate solubility in PU matrices would support this observation. A much larger study is underway comparing a number of blowing agents via the same methodology for a more comprehensive and definitive assessment of the diffusion behavior. In a real spray foam application, the influence of skin thickness, number of lifts, and facings will reduce this rate further.

7. Conclusion

Formacel 1100 ($CF_3CH{=}CHCF_3$) represents an attractive next generation foam expansion agent for the polyurethane spray foam market. It is a zero ODP, low GWP foam expansion agent that provides the desired characteristics of current HFCs: non-VOC, suitable boiling point, low vapor thermal conductivity and non-flammability. With respect to spray foam applications, an initial DoE of key formulation factors has illustrated the complexity and strength of interactions for impacting physical and thermal performance attributes. Among these are the Formacel 1100 levels, water levels, and catalyst levels with isocyanate indexes and surfactant levels also playing an important role. Formacel 1100 provides substantial thermal insulation performance when formulated in conjunction with water at low to moderate levels. The interactions are complex and often drive the desired outputs in opposite directions, however, clear trends emerge which can help guide optimization efforts with this new foam expansion agent. The key learning from the DoE is that simply dropping in one foam expansion agent for another may not offer the best performance in a specific system due to the multiple interactions that can occur from important factors.

Concerning long term retention of Formacel 1100, a new NMR methodology has been explored as a means of estimating diffusion coefficients in situ of foam samples. The preliminary assessment of the diffusion behavior of Formacel 1100 would indicate a favorable profile as a long term insulating component in high performance foam applications.

DuPont is committed to providing environmentally sustainable foam expansion agents with good insulation performance and cost effectiveness to the foam industry. DuPont is conducting further studies to maximize the performance of Formacel 1100, and is moving forward with the development and commercialization of Formacel 1100.

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Results of latest field evaluations of Solstice Liquid Blowing Agent spray foam

Solstice Liquid Blowing Agent (LBA) has a GWP of 1 and is non-flammable. It has proven to be an ideal replacement for Enovate 245fa in spray foam applications. A key component for commercializing any blowing agent is not only its laboratory performance but also its field performance. Previous papers have focused on the analysis of small scale field evaluations and the evaluation of initial commercial roof installations. This paper will include a discussion of the latest results on commercial roofing applications but the primary focus will be a discussion of the foam quality in the latest spray polyurethane foam (SPF) wall applications.

1. Introduction

Solstice LBA is an excellent replacement in the spray foam application for Enovate 245fa. This paper will provide a general review of the physical properties of Solstice LBA as well as discuss the commercial availability of the product.

Spray foam is unique in the polyurethane industry because it is manufactured in the field at the job site. Solstice LBA-based spray foam systems are commercially available, being sold and used globally today. The concept of using Solstice LBA as blowing agent has been proven [1–4]. Spray foam wall applications represent over 50 % of the spray foam market in North America. The focus of this paper is twofold: primarily the evaluations of the new North American Solstice LBA wall formulations and secondarily the evaluation of long term field performance of roof and wall product sprayed over one year ago.

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2. Properties

Solstice LBA is (E) 1-chloro-3,3,3-trifluoropropene. It is a physical blowing agent that is suited for use in polyurethane spray foam. Physical properties are one of a mosaic of attributes that must be assessed to determine the suitability of any material as a blowing agent **(tab. 1)**. Solstice LBA is an ideal blowing agent from the environmental perspective since it has an ODP of ~0 and a GWP of 1. These attributes combined with the fact that it is non-flammable and does not have flame limits makes Solstice LBA the ideal replacement for Enovate 245fa in this application.

3. Field performance of Solstice LBA spray foam

3.1 Solstice LBA wall foam

As part of the field evaluation of spray foam systems, on 17 October 2012, a Solstice LBA spray foam system was applied to a small wooden shed located at Honeywell's research facility in Buffalo, NY, USA. At the time of writing this paper the structure has been exposed to 22 months of Western New York weather. The application was conducted by a commercial SPF contracting firm and by an experienced applicator.

Internal wall spray foam application is a common and growing SPF application **(fig. 1)**. There are two types of applications: partial and full wall fill which may result in a single or multiple lift installation. Both of these applications were evaluated in the shed. The partial fill application was simulated with a single lift 2 inch (5 cm) foam application. The full fill application was simulated with a double lift application of 4 inch (10 cm) in total. The foam density was 2.38 lb/ft³ (38 kg/m³). The wall samples for evaluation were harvested from the shed initially and now, 22 months later.

The foam was found to be tightly bonded to the interior wall throughout the 22 month period. It remained bonded even while harvesting by cutting the wall with a power saw. There were no hollow spots detected between the foam and the wall. Both the 2 inch (5 cm) and 4 inch (10 cm) showed no shrinkage throughout the study.

Fig. 1: *Internal wall application (all photos by Honeywell)*

Tab. 1: *Physical properties of blowing agents*

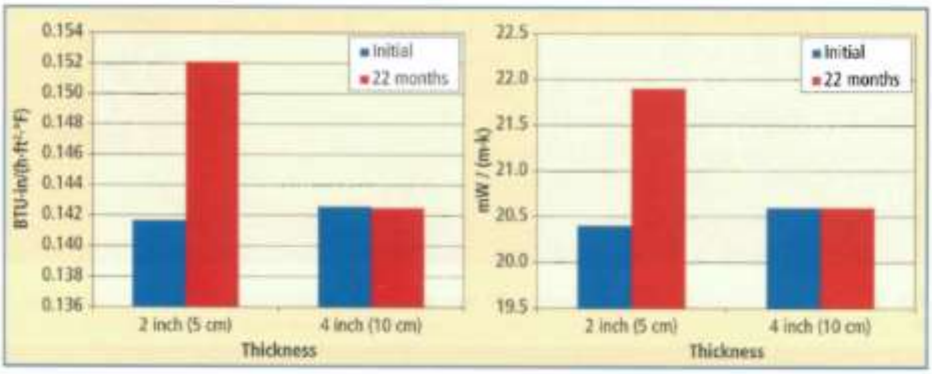
Property	Solstice LBA	Enovate 245fa
Molecular weight	130	134
Flashpoint / °C	None	None
LFL/UFL / vol% in air	None	None
ODP	~0 [5]	~0
GWP / 100 a	1 [7]	858 [5]
OEL (PEL)	800 [6]	300

The interior foam removed from the shed was tested for k-factor. Samples were cut from the middle of the sheet, laterally, as well as being a core-cut vertically. This provides data indicative of real field aging thermal performance of foam. A summary of this data is listed in **table 2** and **figure 2.** The results indicate that the most significant variation is in the 2 inch thick sample. There is a 7.3 % increase in k-factor over the 22 months. The 4 inch thick sample shows less than 0.1 % variation in k-factor over the 22 months.

3.2 Solstice LBA low slope roofing foam

For a new product to be successful in the field it needs to require minimal equipment change, process seamlessly, remain cost effective from an application standpoint and have equivalent field performance over time. There has always been an interest in physical foam performance over time under field versus laboratory conditions. Other than a small adjustment in the processing temperatures and attention to processing conditions, the applicators have found the transition from Enovate 245fa to Solstice LBA SPF system to be seamless. They are using equivalent equipment and the product is being shipped in the same containers and has equivalent shelf life. They are also seeing increased R-value, better yields and higher compressive strengths. The focus of this study was to evaluate "weathering" of a low slope roof that has been in the field for over 12 months. The recent third party inspection of the Solstice LBA-blown SPF roof at the Cleveland Airport shows it achieved an inspection rating of 10 **(fig. 3).**

Fig. 2: *Comparison of k-factor versus time*



Tab. 2: *k-factor of interior wall spray foam tested by ASTM C518 on 1 inch (2.5 cm) core foam at mean temperature of 75 °F (24 °C)*

Thickness	**2 inch**	**5 cm**	**4 inch**	**10 cm**
Initial density	*2.58 lb/ft³*	*41 kg/m³*	*2.38 lb/ft³*	*38 kg/m³*
k-factor				
Initial	*0.1417 BTU-in/(h-ft²-°F)*	*20.4 mW/(m-K)*	*0.1426 BTU-in/(h-ft²-°F)*	*20.6 mW/(m-K)*
After 22 months	*0.1521 BTU-in/(h-ft²-°F)*	*21.9 mW/(m-K)*	*0.1425 BTU-in/(h-ft²-°F)*	*20.6 mW/(m-K)*

Fig. 3: *Summary of inspection of Cleveland airport roof data (Inspection report from PFRI Services Inc.)*

A = total ft² of deviation 0.0 ft²

B = size of roof 14,058 ft²

A/B x 100 = 0 % deviation

Rating = 10

Rating	*% Deviation*
10	*0<0.05*
9	*0.05<0.2*
8	*0.2<0.5*
7	*0.5<1.0*
6	*1.0<3.0*
5	*3.0<6.0*
4	*6.0<10.0*
3	*10.0<15.0*
2	*15.0<50.0*
1	*50.0<100*

After one year of weathering, the only defects noted were interlaminate delamination in one of the four core samples and mechanical damage to the surface by a light. Delamination is often attributed to improper processing conditions. The foam showed no signs of blistering, cracking, or shrinkage. The compressive strength was >60 psi (>0.41 MPa) **(fig. 4).**

The Solstice LBA wall and roof foam that has been applied in the field is performing well. In the samples tested, there are no signs of foam failure due to weathering or exposure.

3.3 Performance of Solstice LBA SPF wall insulation

The next topic is commercial application of Solstice LBA wall foam. There are two North American systems houses which have commercial SPF Solstice LBA wall systems. This paper discusses the properties and full scale application and evaluation of these systems. These systems have been evaluated by third party labs and are in the process of obtaining third party verifications. One system is manufactured by Lapolla Industries Inc. The second is manufactured by a systems house that at this time wishes to be unidentified.

Fig. 4: *Results of inspection of Cleveland airport roof (Data from inspection report from PFRI Services Inc.)*

Avg. foam thickness	*1.7*	inches
Avg. coating thickness	*32*	mils
Avg. compressive strength	*61.7*	psi
Low compressive strength	*0*	% of samples
Average density	*3.79*	pcf
Low density	*0*	% of samples
Number of samples tested	*4*	
Bur adhesion	*0*	% deviation
Bur wet	*0*	% deviation
Foam adhesion	*0*	% deviation
Foam blisters	*0*	% deviation
Foam cracks	*0*	% deviation
Foam exposed	*0*	% deviation
Foam interlaminar adhesion	*0*	% deviation
Foam lifts	*0*	% deviation
Foam off-ration	*0*	% deviation
Foam overcoating	*0*	% deviation
Foam oxidized	*0*	% deviation
Foam spongy	*0*	% deviation
Foam texture	*0*	% deviation
Foam wet	*0*	% deviation

3.3.1 *Vapor pressure*

Since storage and application conditions vary for SPF throughout the application season, it is important to know the vapor pressure of a polyol component (blend) of the system. The vapor pressure of a polyol blend determines the shipping conditions and impacts the processing parameters. **Figure 5** compares the vapor pressure of a commercial polyol blend prepared with equimolar quantities of Enovate 245fa and Solstice LBA at 54 °C. The Solstice LBA polyol blend has a significantly lower vapor pressure than the Enovate 245fa blend does. A 11 % reduction in vapor pressure at 60 °C provides an advantage for shipping, processing and storing of Solstice LBA blends. This reduction in vapor pressure is consistent with what has been seen in commercial SPF roofing systems as well as other formulations.

3.3.2 *Shelf life*

Spray foam systems are used in the field. They are traditionally sold in 250 l metal drums. Because of the variance in job size and location systems are often purchased in large quantities reducing associated shipping costs. This means that systems can be stored in the field for an extended period of time. The applicator requires that the reactivity and processing of the polyol blend remain equivalent during this storage period of time. The shelf life for a SPF system provided by a manufacturer may vary based upon the application area and buying practices of the applicator. Both systems houses report polyol blend shelf life consistent with what is seen with their Enovate 245fa system: Lapolla Industries Inc. reports six months shelf life for both systems, the other systems house nine months.

Tab. 4: *Processing parameters for Foam LOK II-4G (Equipment: Reactor E-30 proportioner and Fusion CS spray gun with 250 ft (76.2 m) hose)*

Processing temperature		
A-side settings	130–134 °F	54–57 °C
B-side settings	130–134 °F	54–57 °C
Hose	120–124 °F	49–51 °C
Processing pressure		
A-Side settings	925–1,000 psi	0.4–6.9 bar
B-Side settings	925–1,000 psi	0.4–6.9 bar

3.3.3 *Foam quality processing and field application*

Each of the systems houses has had their formulations tested by third party labs and have had large scale field applications of their Solstice LBA systems. The summary of this data for each manufacturer follows.

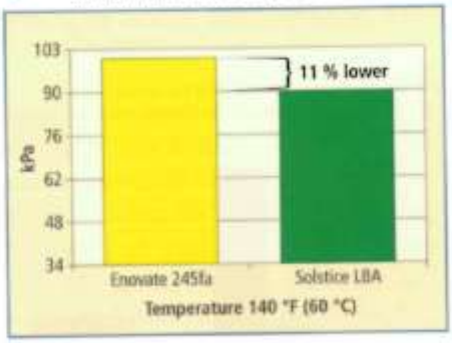
3.3.3.1 *Evaluation I (Lapolla Industries Inc.)*

The third party test results for Foam-LOK II-4G, the Solstice LBA SPF manufactured by Lapolla Industries Inc., are compared with those of their commercial Enovate 245fa system in **table 3.** The Solstice LBA system has a 8 % higher aged R-value and is equivalent in all other test results.

In late July–August 2014, a home at Purdue University in West Lafayette, IN, USA, was sprayed with Foam-Lok II-4G. The exterior siding was removed from the home and the wall, attic and basement were sprayed with the material. Due to weather conditions and the complexity of the job the application took four days and involved two drum sets of material. A picture of the wall application is shown in **figure 6.**

The job was sprayed by a commercial contractor using existing equipment. The application parameters for the job are cited in **table 4.** The contractor reported minimal gun fouling during the application and the foam surface was very smooth. The application temperature and pressure for the new system were optimized.

Fig. 5: *Comparison of vapor pressure of Enovate 245fa and Solstice LBA polyol blends*



Tab. 3: *Comparison of Enovate 245fa and Solstice LBA foam performance (data provided by Lapolla Industries Inc.)*

Property	Test method	Unit	Foam LOK-CC	Foam-LOK II-4G
Blowing agent			Enovate 245fa	Solstice LBA
Aged R-value (after 90 days at 140 °F (60 °C))	ASTM C518	1/inch	6.3	6.8
Aged k-factor (after 90 days at 140 °F (60 °C))		mW/(m·K)	22.9	21.2
Compressive strength	ASTM D1621	psi kPa	25–30 172–207	25 172
Air leakage	ASTM E283	L/s/m²	<0.020 (at 2 inch)	0.008 (at 1 inch)
Closed cell content	ASTM D2856	%	>90	>90
Flammability	ASTM E84		Class 1	Class 1
Dimensional stability (after 28 days at 160 °F (71 °C) / 100 % RH)	ASTM D2126	Vol. change / %	4	4

Tab. 5: *Field performance data*

Day	Application	Surface	Density	Closed cell content / %	Adhesion
1	Internal basement	Concrete	2.08 lb/ft³ (33.3 kg/m³)	>90	+
	Exterior wall	Lath and plaster	2.36 lb/ft³ (37.8 kg/m³)	>90	+
2	Exterior wall	Lath and plaster	2.38 lb/ft³ (38.1 kg/m³)	>90	+
Yield					
Enovate 245fa blown foam			4,200 bdft (9.91 m³)		
Solstice LBA blown foam			4,800 bdft (11.33 m³)		

The challenge with any application of SPF is if the field application will yield equivalent quality product to the laboratory test environment. There are three parameters that an applicator utilizes to assess field performance of a SPF system. These are applied density, open cell content and yield (surface coverage). In addition they look at surface adhesion. The summary of the results from the West Lafayette project are listed in **table 5.**

The application at the West Lafayette, IN home went well. The application data was within acceptable industry tolerances for these applications. There is an increase in density noted between the internal and external application of the product. However, this is not unanticipated due to the wind and temperature differences between these applications. The density was the same on both application days though field conditions were not identical. The biggest improvement is in the yield. Yield represents how much coverage a contractor gets from a system. This is significant to a contractor because the more coverage the less application costs. In essence it is a measure of the efficiency of the system. In this application there is a 14 % increase in yield.

3.3.3.2 Evaluation II (second systems house)

A second systems house has a commercial quality Solstice LBA SPF system. The third party test results for this system are compared with those of their commercial Enovate 245fa system in **table 6.** The Solstice LBA system has a 8 % higher aged R-value and is equivalent in all other test results.

In late July, a garage of a home was sprayed with Solstice LBA blown SPF. A single drum set was used in the application. A picture of the wall application is shown in **figure 7.**

The job was sprayed by a commercial contractor using existing equipment. The application parameters for the job are listed in **table 7.** The contractor reported minimal gun fouling during the application and the foam surface was very smooth. The application temperature and pressure for the new system were optimized.

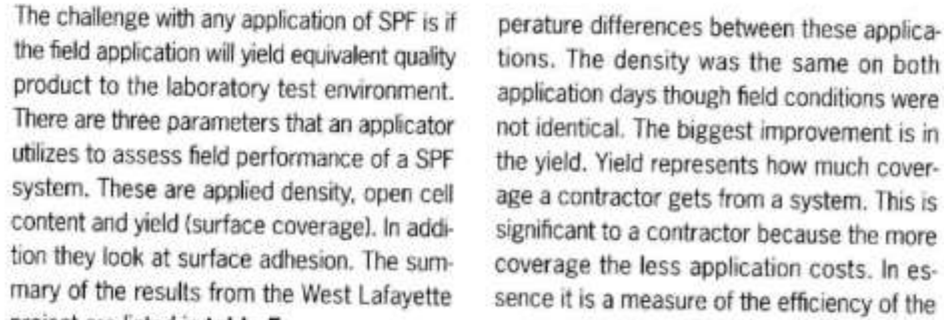
The three parameters that an applicator utilizes to assess field performance of a SPF system are summarized in **table 8** for the Toronto trial.

The application at the home went well. The application data was within acceptable industry tolerances for these applications. After 30 days of field exposure the product looked good. There was no foam blistering, delamination or shrinkage noted at the application site.

4. Personnel exposure testing during application of Solstice LBA spray foam wall insulation

Although the SPF applicator wears respiratory protection during the spray foam application, it is important to assess the potential exposure to Solstice LBA as well. The assay technology ChemExpress Personal Monitor X549AT, a passive personal monitor, has been calibrated for detection of HFC-134a, HFC-245fa and 1233zd(E). The monitor is designed to be worn by an individual to evaluate the exposure to these chemicals. It was used to assess the personal exposure to Solstice LBA. The exposure levels found during Solstice LBA roofing spray foam application at the applicator were well below the PEL for the material **(tab. 9).**

▼ **Fig. 6:** *Pictures of foam application in West Lafayette, IN, USA*



▼ **Fig. 7:** *Pictures of foam application at second application area*

▼ **Tab. 6:** Comparison of Enovate 245fa and Solstice LBA foam performance (data provided by systems house)

Property	Test method	Unit	Enovate 245fa	Solstice LBA
Initial R-value	ASTM C518	1/inch	7.2	7.6
k-factor		mW/(m·K)	20.0	19.0
Density		lb/ft³ kg/m³	2.1 33.6	2.1 33.6
Compressive strength	ASTM D1621	psi kPa	22 152	15–20 103–138
Closed cell content	ASTM D2856	%	>95	>95
Flammability	ASTM E84		Class 1	Class 1
Dimensional stability after seven days at 168 °F (75.6 °C) / 95 % RH	ASTM D2126	Vol. change / %	6	3