Ref. Ares(2020)1259841 - 28/02/2020

Horizon 2020
European Union Funding
for Research & Innovation

Task 2.4 - EENSULATE Foam material characterisation and durability assessment

WP2

Lead Partner: Selena Labs

Partner Contributors: SELENA

Dissemination Level: Confidential

Deliverable due date: – M 36 (August 2019)

Actual submission date: M 43 (February 2020)

Deliverable Version: V1

Project Acronym	EENSULATE
Project Title	Development of innovative lightweight and highly insulating energy efficient components and associated enabling materials for cost-effective retrofitting and new construction of curtain wall facades
Grant Agreement n°	723868
Funding Scheme	Innovation Action
Call	H2020- EEB-2016
Topic	EEB-01-2016 Highly efficient insulation materials with improved properties
Starting Date	1st August 2016
Duration	42 Months

Executive summary

The present report describes the work undertaken by SELENA to finalise the synthesis and characterisation of a foam with high heat insulating and fire retardant properties to be used for the production of two products: a two-component polyisocyanurate foam (TCF) and a one-component polyurethane foam (OCF), both doped with nanoparticles. The activities described start form the outcomes of Task 2.1 “Synthesis of smart fillers”, Task 2.2 “Surfactants development”, and Task 2.3 “Foam formulation production”. In order to enter the market, an external certification is required. Selena has submitted foam samples to test thermal conductivity (both OCF and TCF) and the reaction to fire (TCF only). The results of the former are satisfactory, while latter test shown that the product is difficult to ignite (C), produces no droplets during burning (d0), but the speed of smoke emission is of high intensity (s3). Final result corresponds to DIN B1 class, equal to current E Euroclass, which is highest possible for polyurethane products; however, our foam contains no toxic chlorinated fire retardants and little TCPP, which makes it more environmentally benign. An outcome of the work, regarding the performance of TCF foam, has been published recently in *Fire Technology* journal.

Table of Contents

List of Figures

List of Tables

Abbreviations and Acronyms

[CC] - Cone calorimeter analysis;

[FP] - Fire performance;

[FR] - Fire retardant;

[LDH] - Layered doubled hydroxides;

[MDI] - Methylene diphenyl isocyanate

[PIR] - Polyisocyanurate insulation;

[PUR] – Polyurethane foam;

[SBI] – Single burning test;

[TGA] - Thermogravimetric analysis;

[ZrP] - Zirconium phosphate;

1 Introduction

Based on the outcomes of Task 2.1, Task 2.2 and Task 2.3, SELENA developed two insulating products: a two-component polyisocyanurate foam (TCF) and a one-component polyurethane foam (OCF), both doped with nanoparticles.

This document presents the work performed by SELENA in the framework of Task 2.4 “EENSULATE Foam material characterisation and durability assessment”. The aim is to characterise the TCF and OCF in order to ensure their compliance with the expected requirements.

The characterisation comprised the assessment of:

a) Insulation properties (initial and aged thermal conductivity measurements of unfilled OCF and filled OCF)

b) Yield (foam volume measurements)

c) Fire resistance (cone calorimeter evaluation)

d) Dimensional stability of OCF under specified temperature and humidity conditions

e) Morphology and porosity (confocal microscopy characterization, X-ray computer assisted microtomography, nitrogen absorption, SAXS-WAXS, SEM-EDX)

f) Aged thermal conductivity measurements of unfilled OCF and filled OCF

g) Thermal properties (TGA-DTA evaluation)

h) Aging of the final product in oven to accelerate the aging process and characterizing the aged product.

Most of the results were already included in D2.3; in the present report the final data are presented.

2 PIR (TCF) and PUR (OCF) further developments

To improve the fire retardancy and fire behaviour of the PIR (TCF) and PUR (OCF) foams, Selena checked the potential synergistic effect between organically modified nanoclay LDH and flame retardants, like expandable graphite. Regarding the flammability of polymer insulation-related flame retardants, the substitution of commonly used halogen-based flame retardants for eco-friendly "greener" ones, such as mineral fillers like Layered Double Hydroxides (LDH) nanocomposites, is currently of great interest for avoiding the release of corrosive and toxic volatile compounds from combustion. In contrast to common halogenated compounds used as flame-retardants, LDH attract significant attention and are explored as second-generation fire-retardant materials to alternatively be used as efficient and more environmental friendly additives to various polymers with the ability to improve both their flame retardancy and thermal stability. Despite their effectiveness, LDHs have until now limited commercial success as fire retardants because of their difficulty to disperse and distribute in polymers, which limits their effectiveness and most available studies concern their incorporation in PU foam. The behaviour of LDH and EG in the foams were checked using scanning electron microscopy, thermogravimetric analysis (TGA), Cone calorimeter (CC) tests, Single Burning Item (SBI) and in Thermal Conductivity Test.

Moreover, PIR foam was produced by using an excess of the methylene diphenyl isocyanate (MDI) component. The resultant PIR product is provided with greater heat stability, increased flame resistance, chemical resistance and dimensional stability, than that of a PUR foam.

The chemical modification generating PIR foam and the possibility to adding traditional flame retardant properties with lamellar filler permit to orient the European classification of flame properties of EENSULATE foam to that of phenolic foam/ PIR block foam (see Table 1) but with thermal insulation typical of PU foam.

Table 2-1 European Fire Resistance classification

PRODUCT	UK Class	LIKELY EUROPEAN CLASSIFICATION						
		A1	A2	B	C	D	E	F
Low density glass wool	NC	A1						
High density glass wool	LC	A1						
Calcium silicate board	LC	A1						
Rockwool + glass tissue face	0	A1						
Rock wool + alu-foil facing	1	A1						
Glass wool + alu-foil facing	0		A2,s1,d0					
Silicate based mineral masonry paint	0		A2,s1,d0					
Cement particle board	0		A2,s1,d0					
Gypsum wallboard – Ordinary	0		A2,s1,d0					
FR Gypsum wallboard	0		A2,s1,d0					
HD mineral wool + 2 coat render	0		A2,s2,d0					
EPS behind plasterboard	0			B,s1,dO				
Duplex embossed wall-covering	0			B,s1,d0				
FR MDF (01)	0			B,s1,d0				
Birch plywood FR0	0			B,s1,d0				
Birch Plywood FR1	1			B,s1,d0				
Gravure printed VINYL wallpaper	0			B,s2,d0				
Vinyl wall-covering fabric backed	0			B,s2,d0				
European redwood FR0 TG Board	0			B,s2,d0				
European redwood FR1 TG Board	1			B,s2,d0				
Unfaced phenolic foam	2			B,s2,d0				
European plywood FR2 TG Board	3				C,s1,d0			
Birch plywood FR2	3				C,s1,d0			
FR MDF (02)	1				C,s2,d0			
PIR block foam	1				C,s2,d0			
Phenolic foam alu-foil for ducts	0				C,s2,d0			
PVC-U cellular cladding system	2Y				C,s3,d2			
PVC-U cellular cladding system	1				C,s3,d2			
Gravure printed vinyl wallcovering with paper backing	0					D,s2,d0		
Expanded polystyrene type A	Unclassif					D,s3,d0		
PIR foam + alufoil facing	0					D,s3,d0		
Extruded polystyrene XPS	Unclassif					D,s3,d1		
Wood fibre medium board – vac. Press	0						E,	
PUR foam + flexible laminate facing	1						E	
PIR Sprayed foam	4						E	
Expanded polystyrene type N	4							F

3 Experimental

In order to investigate the characteristics of the two developed foam formulations, Selena conducted a number of experiments. Tests concerned are:

- ✓ Scanning electron microscopy (SEM);
- ✓ Thermogravimetric analysis (TGA);
- ✓ Cone calorimeter (CC) tests;
- ✓ Single Burning Item (SBI);
- ✓ Thermal Conductivity.

Testing methodologies were extensively described in D2.3, below a short summary is reported in order to facilitate the interpretation of results presented in section 4.

3.1 Test Methods

In order to further investigate the LDH dispersion in the polymer matrix, thermal resistance and insulating properties of the developed two base products, the following analyses were carried out. Tests results are reported in section 4 of the document.

3.1.1 Scanning electron microscopy (SEM)

The Cellular structure of the samples was evaluated by means of **scanning electron microscopy (SEM)**. In particular, field emission scanning electron microscope (FE-SEM) FEG LEO 1525 (Zeiss Oberkochen Germany) equipped with Bruker EDX probe has been used.

The parameters of scanning electron microscope are the following:

- ✓ Accelerating voltage 15 kV;
- ✓ Measurement mode – secondary electrons;
- ✓ FE-SEM micrographs, collected by depositing the samples on a stub holder and after a sputter coating with chromium for 20 s (about 10 nm in thickness).

3.1.2 Thermogravimetric analysis (TGA)

The thermal stability of the samples was evaluated by means of **thermogravimetric analysis (TGA)** under N_2 (inert gas) environment in a Mettler Toledo TGA apparatus. The following parameters were determined:

- ✓ initial degradation temperature, T5% (temperature at 5% weight loss);
- ✓ corresponding maximum temperature, Tmax;
- ✓ weight loss, W, for each degradation step and char residue at 1000°C.

About 10 mg foam sample was placed in an alumina pan with no lid. The heating rate was 20°C/min with a N_2 flow of 150 ml/min.

3.1.3 Cone calorimeter (CC) tests

Cone calorimeter (CC) tests were performed with a Dark Star Research Ltd (UK) apparatus according to the ISO 5660-1 at 50 kW/m^2. The dimensions of the samples are 100mm x 100mm x 24mm and the density of the samples was approximately 50 kg/m^3. Experimental results includes:

- ✓ Mass Loss Rate (MLR);
- ✓ Heat Release Rate (HRR);
- ✓ time to ignition (TTI);
- ✓ Combustion Time (CT);
- ✓ smoke production rate (SPR);
- ✓ smoke;
- ✓ CO yield.

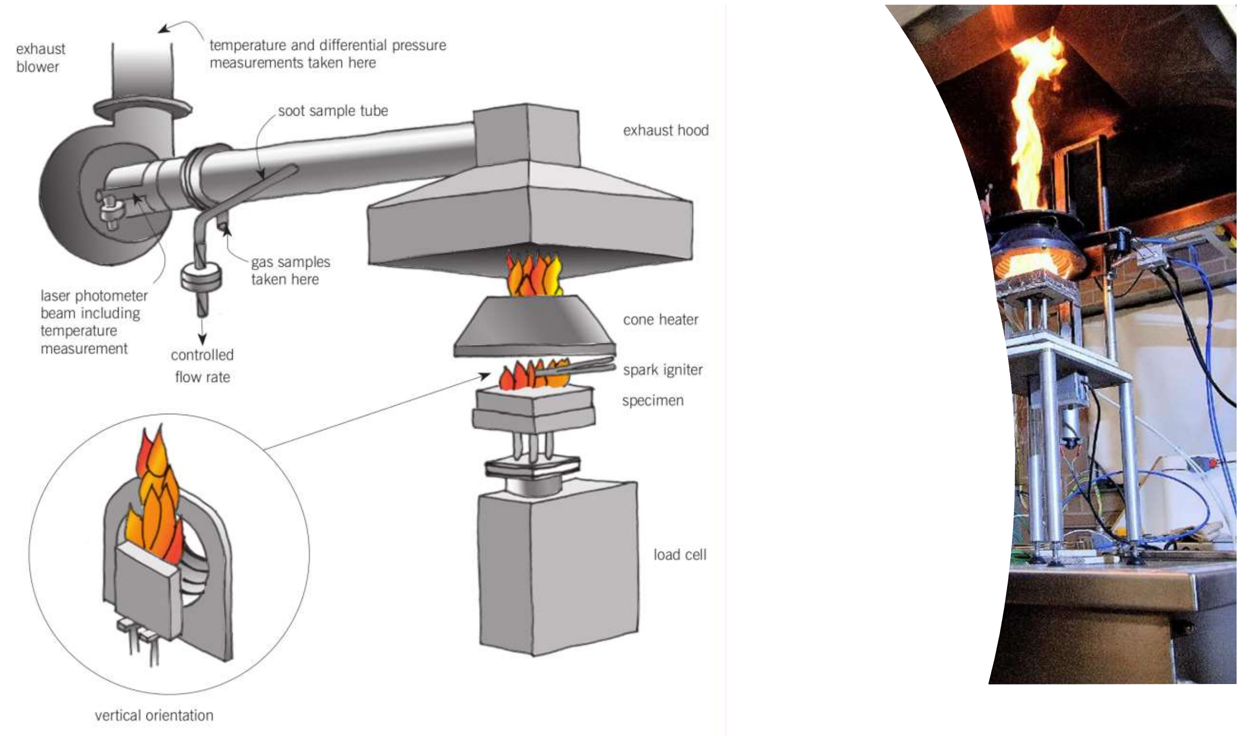


Figure 3-1 Cone calorimeter (CC) tests

3.1.4 Single Burning Item (SBI)

Single Burning Item (SBI) checks the reaction to fire, according to EN ISO 13823.

During each test, Rate of Heat Release (RHR) is determined based on oxygen consumption calorimeter principles. Furthermore, Rate of Smoke Production (RSP) is measured at the exhaust duct based on light attenuation principles. The drop of flaming particles is visually observed during the first **10 minutes** of the entire façade profile and **26 minutes** for the TCF foam alone test. Finally, lateral flame spread, and maximum flame reached during the test is recorded throughout the test.

The classification parameters of the SBI test are

- ✓ Fire Growth Rate (FIGRA), Total Heat Release (THR);
- ✓ Smoke Growth Rate (SMOGRA);
- ✓ Total Smoke Production (TSP);
- ✓ Lateral Flame Spread (LFS);
- ✓ existence of Flame Droplets (FDP);

The foam samples used for the tests were selected by SELENA based on the cone calorimeter and single flame tests.

The preparation of the first SBI test was carried out by FOCCHI and they were tested at ULSTER UNIVERSITY. In total, four samples were tested, including mineral wool and the three foam samples as tested in the cone calorimeter (i.e. 18.3.4_B_Yellow-SBI, 27.3.4_C_Orange-SBI and 27.3.4_D_Black-SBI, as mentioned in chapter 4). The aluminum coated surface face of the specimens was exposed to the heating conditions of the test when the specimens were mounted in the test position. The test specimen comprised two walls mounted into an aperture in a specimen trolley such that they formed a vertical 90° corner (see Figure 3-4).

The second test was prepared by Selene and performed in a certified laboratory the Czech Accreditation Institute -PAVUS. Foam EE 27.3.4 new & new polyol-polyetherol & LDH (modification of the orange formula) were checked.

The test samples were stored in a conditional room prior to the tests. A schematic of the samples is show below.

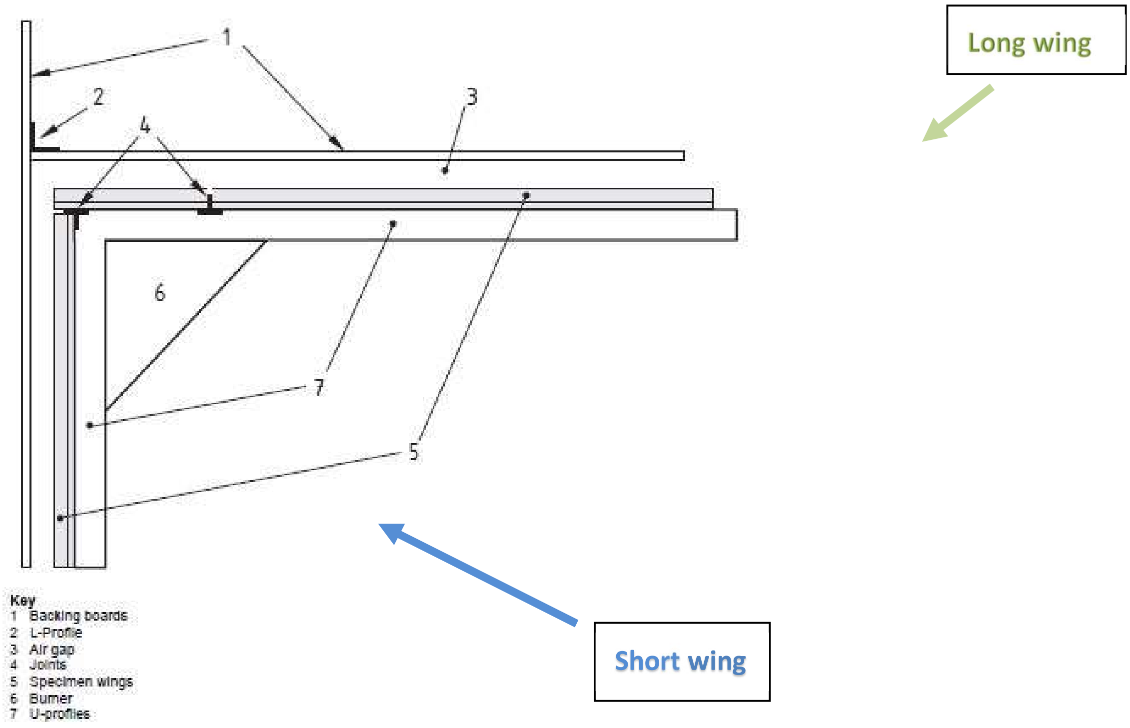


Figure 3-2 Schematic of the SBI test configuration

Figure 3-3 SBI tests performed

3.1.5 Thermal Conductivity

For the measurements of thermal resistance and insulating properties of the developed two products a device for testing the thermal conductivity of matrices insulating: EPS, XPS, PUR, mineral wool, etc. was used. (Figure 3-4).

Figure 3-4 Thermal Conductivity test machine

In a heat flow meter (HFM), the test specimen are placed between two heated plates with controlled temperature, in order to reach a user-defined mean sample temperature. A temperature drop is inducted as well to measure heat flowing through the specimen. The sample thickness (L) corresponds to the actual sample dimension or to match the desired thickness of a compressible sample. The heat flow (Q) through the sample is measured by two calibrated heat flux transducers or covering a large area of both sides of the specimen.

After reaching a thermal equilibrium, the test is considered completed. The heat flux trans- ducer output is calibrated with a standard. For the calculation of the thermal conductivity (X) the average heat flux and the thermal resistance (R) is used,in accordance with Fourier's Law. The thermal transmittance, also known as U-value, is the reciprocal of the total thermal resistance. The lower the U-value, the better the insulating ability.

The samples dimensions were the following**:**

- ✓ Width: min: 300 mm, max: unlimited
- ✓ Length: min: 300 mm, max: unlimited
- ✓ Thickness: min: 3 mm - aerogel, 10 mm - EPS, XPS; max: 60 mm - aerogel, 100 mm - EPS, XPS

Studies of heat transfer properties on a constant level, thermal conductivity and thermal resistance of flat panel specimens in accordance with ISO 8301, EN 12667 and EN 12939 were performed as well.

4 Results

The present section illustrates results obtained from the test performed on the different foam formulations. The table below summarises the final formulations that were investigated.

Table 4-1 performed TCF and OCF formulations with smart filler

#	Name of the developed receipt	Type of Formulation
1	EE18.3.4 yellow	TCF
2	EE27.3.4 black	
3	EE27.3.4 orange	
4	Foam EE 27.3.4 new & black	
5	Foam EE 27.3.4 new & orange	
6	Foam EE 27.3.4 new & new poliolpolieterol & LDH (modification of the orange formula)	
7	WP5-6-2 (spray polyurethane foam)	OCF

4.1 The TCF – PIR foam

4.1.1 Parameters & durability for developed TCF formulations

As illustrated in chapter 4.1 of D2.3, researches has been conducted in order to improve TCF flow dimensional stability, and the formulation for the demonstration buildings was selected (EE 27.3.4 new & new poliolpolyeterol FOAM).

The products are characterized according to specific standards, to enable the comparison with the benchmark solution.

Table 4-2 List of key parameters for TCF a Benchmark - MW/ prepared by foaming machine

Critical parameters	EE 27.3.4 new & new poliolpolyeterol FOAM	Benchmark - mineral wool
Density [kg/m^3]	35-40	70
λ initial [W/m*K] EN ISO 12667	0.032	0.040
Fire properties, SBI test, EN ISO 13823	C-s3-d0	A-s1-d0
Acoustic (reduction in dB) EN ISO 16283	1. 43 with plasterboard 2. 47 without plasterboard	49
Tensile strength (kPa)	140-160	50-100

It is important to emphasize that the foam developed was tested in the reaction to fire test, SBI test, as a separate material (not as an integrated system - curtain wall element), which is vaguely an organic material and it is not possible to obtain a higher class.

Regarding the period of validity for the developed two-component system, part A, polyol may be fit for 3 months from preparation, while part B, PMDI has the suitability of 12 months from the date of manufacture.

Technical and safety data sheets for the developed TCF system have been prepared. The documents are attached to the report as a separate file.

Compared to the previous report (deliverable 2.3), lambda measurements and acoustic tests have been carried out, as shown in Table 4-3. Moreover, external certification of the fire properties has been performed according to EN ISO 13823; the reaction to fire has been classified as C-s3-d0, i.e., the foam examined is combustible materials with limited contribution to fire. Albeit the foam has been found to be difficult to ignite (C), producing no droplets during burning (d0), the speed of smoke emission is of high intensity (s3).

Considering the values of the latter parameter, some measures must be undertaken to reduce smoke production, at least to d2 level – average production of smoke.

4.1.2 Single Burning Item (SBI) analysis for TCF

First SBI test was conducted in FireSERT Lab. University of Ulster have carried out the Single Burning Item test according to the EN ISO 13823. At the end of July 2019, Selena performed the second SBI test, only for selected neat TCF foam, in the accredited Pavus laboratory (Czech Republic). The test method was the same, except that the flame was applied to the PIR foam for 21 minutes, where in Ulster the test lasted 10 minutes and the fire was brought to the aluminum frame constituting the glass façade. Pavus laboratory has carried out the Single Burning Item test according to the EN ISO 13823, using the EE27.3.4 new& new poliolpolyeterol & LDH sample (170418-SBI2- ORANGEFOAM foam after a small modification).

A summary of the results obtained from the SBI test series is presented below:

Table 4-3 Flammability and smoke emission behaviour of EE27.3.4 new& new poliolpolyeterol & LDH

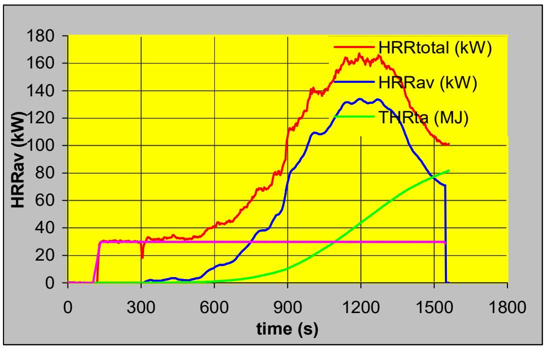
Parameter	Results
	EE27.3.4 new& new poliolpolyeterol & LDH
$FIGRA_{0.3MJ}$ (W/s)	**172**
THR_{600s} (MJ)	**13**
SMOGRA (m^2/s^2)	**92**
TSP_{600s} (m^2)	**1117**
LFS (Y/N)	**Y**
$FDP_{<10s}$ (Y/N)	**Y**

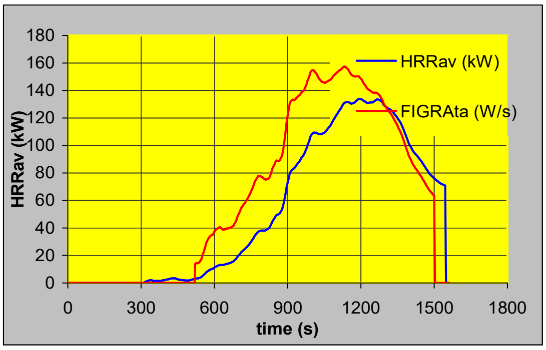
$FDP_{>10s}$ (Y/N)	**Y**

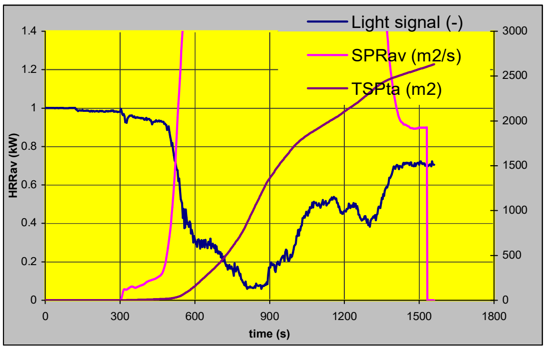
For the developed TCF (EE27.3.4 new& new poliolpolyeterol & LDH) the result of the class reaction to fire is C-s3, d0 (see discussion in Section 4.1.1).

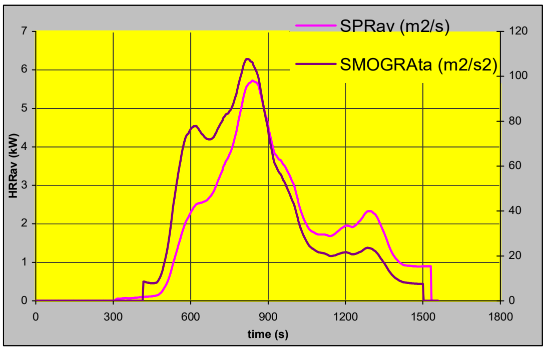
For each of the tests received reports and photographs before and after each SBI test performed are presented in following Figures.

SBI-Test









Date of test: 23 červenec 2019
File name: Z210180481_3.sbi
Operator: Hejná

Specifications

Material:	PIR 150 mm
Mass per unit area (kg/m¸)	0
Thickness (mm)	0

Fire attack

Start of test (s):	300
Burner exposure time (s):	1440
Burner exposure level (kW):	30.172

Test conditions

Mounting:	PAVUS
Substrate:	0
Fixing:	mechanicky
Standard used:	ČSN EN 13823+A1

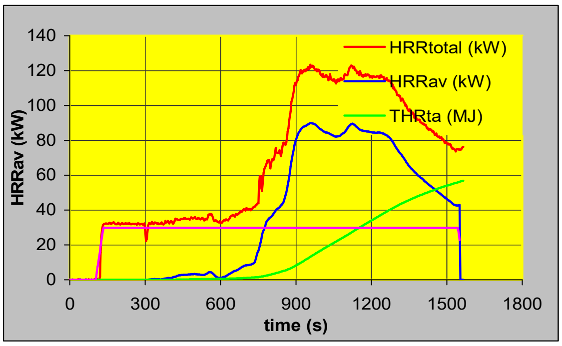
Results

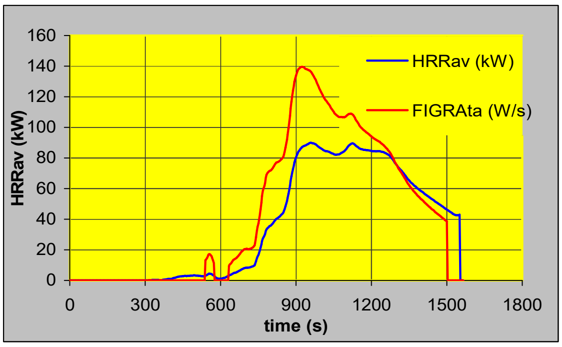
Heat release related		t-t0 (s)	t(s)	
Peak HRRav (t<t0+600s) [kW]:	73.0	600	900	
Peak HRRav (t<t0+900s) [kW]:	133.9	894	1194	
Peak HRRav (t<t0+1200s) [kW]:	133.9	894	1194	
Peak HRRav (t>t0) [kW]:	133.9	894	1194	
THRta (t0_t0+600s) [MJ]:	**10.4**			**C**
THRta (t0_t0+900s) [MJ]:	43.8			
THRta (t0_t0+1200s) [MJ]:	77.4			
Figra_treshold1 [W/s]:	157.28			
Figra_treshold2 [W/s]:	157.28			
Figra [W/s]:	**157.28**			**C**
Corresponding HRRav [kW]:	130.70	831	1131	
t(HRRav >= 3[kW])		120	420	
t(THRta >= 0.2[MJ])		123	423	
t(THRta >= 0.4[MJ])		201	501	
		Estimated class:		**C**

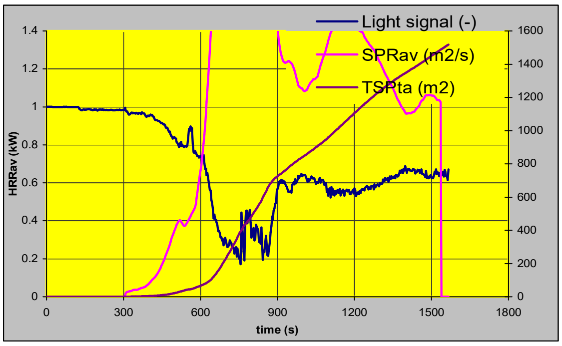
Smoke production related		t-t0 (s)	t(s)	
Peak SPRav (t<t0+600s) [m²/s]:	5.72	540	840	
Peak SPRav (t<t0+900s) [m²/s]:	5.72	540	840	
Peak SPRav (t<t0+1200s) [m²/s]:	5.72	540	840	
Peak SPRav (t>t0) [m²/s]:	5.7	540	840	
TSPta (t0_t0+600s) [m^2]:	**1361.0**			**S3**
TSPta (t0_t0+900s) [m^2]:	2104.9			
TSPta (t0_t0+1200s) [m^2]:	2577.5			
Smogra max [m²/s²]	**107.71**			**S2**
at SPRav [m²/s]	5.60	516	816	
t(SPRav >= 0.1[m²/s])		117	417	
t(TSPta >= 6[m²])		96	396	
		Estimated class:		**S3**

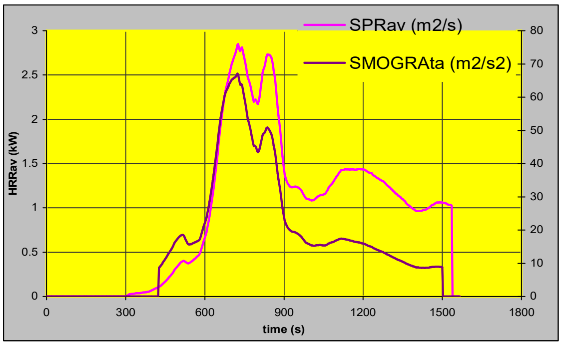
SBI Software V03.2 commissioned by FIRE Ltd. http://www.fire-uk.com

SBI-Test









Date of test: 29 červenec 2019
File name: \SBI\Results\Z210180481_5.sb1
Operator: Hejná

Specifications

Material:	PIR 150 mm
Mass per unit area (kg/m˛)	0
Thickness (mm)	150

Fire attack

Start of test (s):	300
Burner exposure time (s):	1440
Burner exposure level (kW):	32.086

Test conditions

Mounting:	PAVUS
Substrate:	0
Fixing:	mechanicky
Standard used:	ČSN EN 13823+A1

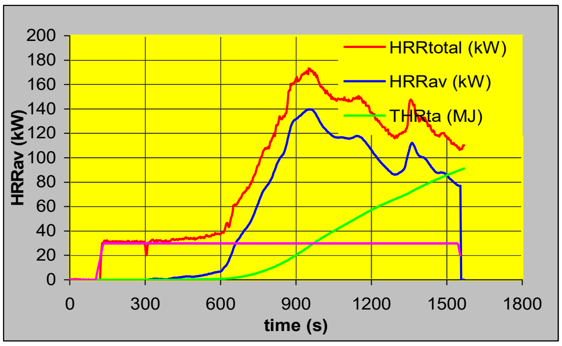
Results

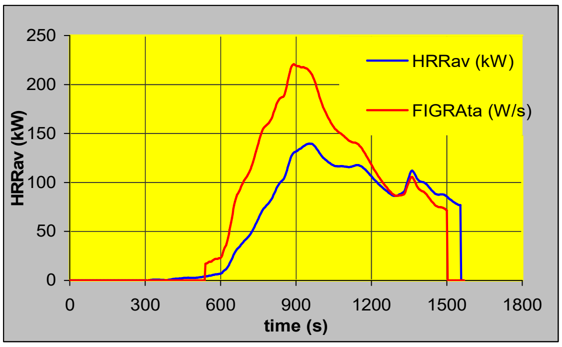
Heat release related		t-t0 (s)	t(s)
Peak HRRav (t<t0+600s) [kW]:	80.3	600	900
Peak HRRav (t<t0+900s) [kW]:	89.9	660	960
Peak HRRav (t<t0+1200s) [kW]:	89.9	660	960
Peak HRRav (t>t0) [kW]:	89.9	660	960
THRta (t0_t0+600s) [MJ]:	**8.2**		**C**
THRta (t0_t0+900s) [MJ]:	34.0		
THRta (t0_t0+1200s) [MJ]:	54.0		
Figra_treshold1 [W/s]:	139.56		
Figra_treshold2 [W/s]:	139.56		
Figra [W/s]:	**139.56**		**C**
Corresponding HRRav [kW]:	87.10	624	924
t(HRRav >= 3[kW])		180	480
t(THRta >= 0.2[MJ])		171	471
t(THRta >= 0.4[MJ])		237	537
		Estimated class:	**C**

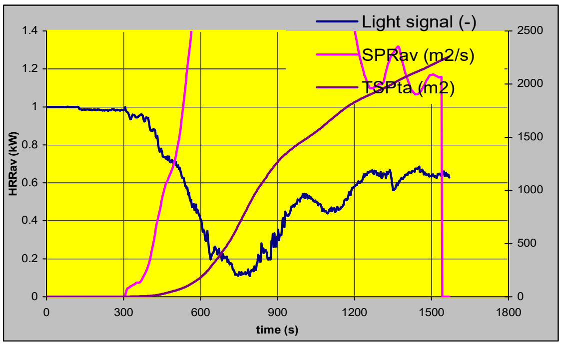
Smoke production related		t-t0 (s)	t(s)
Peak SPRav (t<t0+600s) [m²/s]:	2.85	426	726
Peak SPRav (t<t0+900s) [m²/s]:	2.85	426	726
Peak SPRav (t<t0+1200s) [m²/s]:	2.85	426	726
Peak SPRav (t>t0) [m²/s]:	2.8	426	726
TSPta (t0_t0+600s) [m^2]:	**725.8**		**S3**
TSPta (t0_t0+900s) [m^2]:	1105.0		
TSPta (t0_t0+1200s) [m^2]:	1450.2		
Smogra max [m²/s²]	**67.11**		**S2**
at SPRav [m²/s]	2.80	423	723
t(SPRav >= 0.1[m²/s])		123	423
t(TSPta >= 6[m²])		126	426
		Estimated class:	**S3**

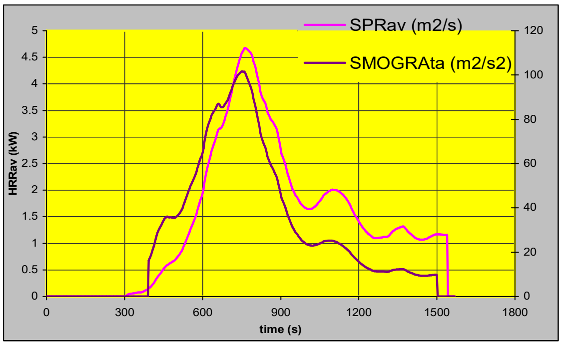
SBI Software V03.2 commissioned by FIRE Ltd. http://www.fire-uk.com

SBI-Test









Date of test: 29 červenec 2019
File name: \SBI\Results\Z210180481_6.sb1
Operator: Hejná

Specifications

Material: PIR 150 mm
Mass per unit area (kg/m¸): 0
Thickness (mm): 150

Fire attack

Start of test (s): 300
Burner exposure time (s): 1440
Burner exposure level (kW): 31.261

Test conditions

Mounting: PAVUS
Substrate: 0
Fixing: mechanicky
Standard used: ČSN EN 13823+A1

Results

Heat release related		t-t0 (s)	t(s)
Peak HRRav (t<t0+600s) [kW]:	131.6	600	900
Peak HRRav (t<t0+900s) [kW]:	139.7	654	954
Peak HRRav (t<t0+1200s) [kW]:	139.7	654	954
Peak HRRav (t>t0) [kW]:	139.7	654	954
THRta (t0_t0+600s) [MJ]:	**20.3**		**D**
THRta (t0_t0+900s) [MJ]:	57.2		
THRta (t0_t0+1200s) [MJ]:	85.7		
Figra_treshold1 [W/s]:	220.73		
Figra_treshold2 [W/s]:	220.73		
Figra [W/s]:	**220.73**		**C**
Corresponding HRRav [kW]:	130.40	591	891
t(HRRav >= 3[kW])		213	513
t(THRta >= 0.2[MJ])		171	471
t(THRta >= 0.4[MJ])		240	540
		Estimated class:	**D**

Smoke production related		t-t0 (s)	t(s)
Peak SPRav (t<t0+600s) [m²/s]:	4.67	462	762
Peak SPRav (t<t0+900s) [m²/s]:	4.67	462	762
Peak SPRav (t<t0+1200s) [m²/s]:	4.67	462	762
Peak SPRav (t>t0) [m²/s]:	4.7	462	762
TSPta (t0_t0+600s) [m²]:	**1265.1**		**S3**
TSPta (t0_t0+900s) [m²]:	1829.0		
TSPta (t0_t0+1200s) [m²]:	2179.7		
Smogra max [m²/s²]	**101.67**		**S2**
at SPRav [m²/s]	4.60	450	750
t(SPRav >= 0.1[m²/s])		78	378
t(TSPta >= 6[m²])		93	393
		Estimated class:	**S3**

SBI Software V03.2 commissioned by FIRE Ltd. http://www.fire-uk.com

Figure 4-1 Results for each of the tests in the reports

Total views to the exposed surfaces of the vertical outer edges of long and short sashes in the SBI test

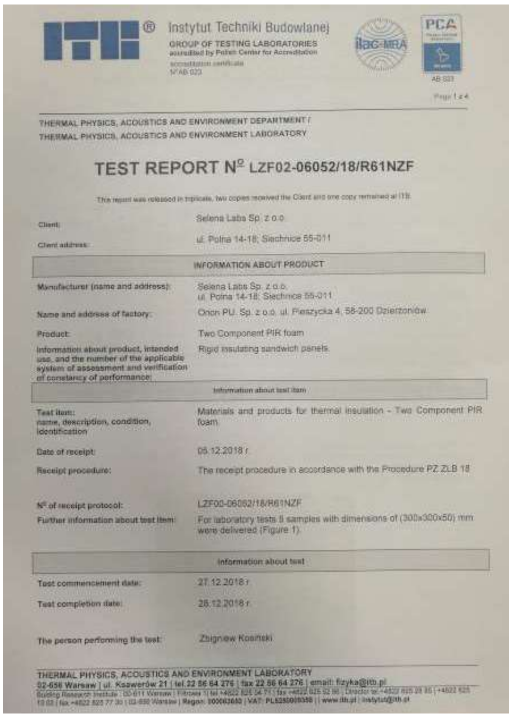
Figure 4-2 Pictures of tested samples

Views to the exposed surfaces of the specimens after the SBI test

Figure 4-3 Pictures of tested samples after exposure to fire

4.1.3 Obtained certificates for TCF

To verify the developed product, Selena performed TCF certification in terms of thermal insulation and fire resistance. Tests was carried out at ITB in Poland (Figure 4-12) and at the Czech Accreditation Institute – PAVUS (Figure 4-13), respectively.


Instytut Techniki Budowlanej
GROUP OF TESTING LABORATORIES
accredited by Polish Center for Accreditation

Page 1 z 4

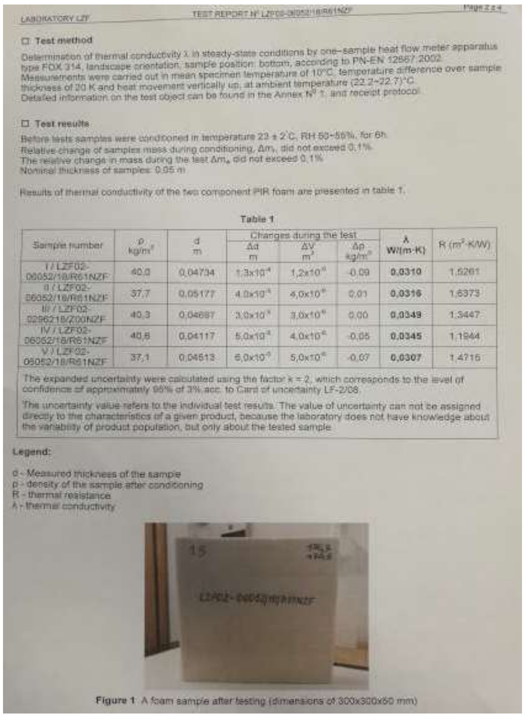
THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT DEPARTMENT / THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT LABORATORY

TEST REPORT N° LZF02-06052/18/R61NZF

This report was released in triplicate, two copies received the Client and one copy remained at ITB.

Client:	Selena Labs Sp. z o.o.
Client address:	ul. Polna 14-18; Siechnice 55-011
INFORMATION ABOUT PRODUCT	
Manufacturer (name and address):	Selena Labs Sp. z o.o. ul. Polna 14-18; Siechnice 55-011
Name and address of factory:	Orion PU. Sp. z o.o. ul. Pieszycka 4, 58-200 Dzierżoniów
Product:	Two Component PIR foam
Information about product, intended use, and the number of the applicable system of assessment and verification of constancy of performance:	Rigid insulating sandwich panels
Information about test item	
Test item: name, description, condition, identification	Materials and products for thermal insulation - Two Component PIR foam
Date of receipt:	05.12.2018 r.
Receipt procedure:	The receipt procedure in accordance with the Procedure PZ ZLB 18
N° of receipt protocol:	LZF00-06052/18/R61NZF
Further information about test item:	For laboratory tests 5 samples with dimensions of (300x300x50) mm were delivered (Figure 1).
Information about test	
Test commencement date:	27.12.2018 r.
Test completion date:	28.12.2018 r.
The person performing the test:	Zbigniew Kosiński

THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT LABORATORY
02-656 Warsaw | ul. Ksawerów 21 | tel. 22 56 64 276 | fax 22 56 64 276 | email: fizyka@itb.pl


LABORATORY LZF — TEST REPORT N° LZF02-06052/18/R61NZF — Page 2 z 4

☐ **Test method**

Determination of thermal conductivity λ in steady-state conditions by one-sample heat flow meter apparatus type FOX 314, landscape orientation, sample position: bottom, according to PN-EN 12667:2002. Measurements were carried out in mean specimen temperature of 10°C, temperature difference over sample thickness of 20 K and heat movement vertically up, at ambient temperature (22,2÷22,7)°C. Detailed information on the test object can be found in the Annex N° 1 and receipt protocol.

☐ **Test results**

Before tests samples were conditioned in temperature 23 ± 2°C, RH 50÷55%, for 6h.
Relative change of samples mass during conditioning, Δm, did not exceed 0,1%.
The relative change in mass during the test Δm, did not exceed 0,1%.
Nominal thickness of samples: 0,05 m

Results of thermal conductivity of the two component PIR foam are presented in table 1.

Table 1

Sample number	ρ kg/m³	d m	Changes during the test: Δd m	ΔV m³	Δρ kg/m³	λ W/(m·K)	R (m²·K/W)
I / LZF02-06052/18/R61NZF	40,0	0,04734	$1,3x10^{-4}$	$1,2x10^{-6}$	-0,09	**0,0310**	1,5261
II / LZF02-06052/18/R61NZF	37,7	0,05177	$4,0x10^{-5}$	$4,0x10^{-6}$	0,01	**0,0316**	1,6373
III / LZF02-0296218/Z00NZF	40,3	0,04687	$3,0x10^{-5}$	$3,0x10^{-6}$	0,00	**0,0349**	1,3447
IV / LZF02-06052/18/R61NZF	40,6	0,04117	$5,0x10^{-5}$	$4,0x10^{-6}$	-0,05	**0,0345**	1,1944
V / LZF02-06052/18/R61NZF	37,1	0,04513	$6,0x10^{-5}$	$5,0x10^{-6}$	-0,07	**0,0307**	1,4716

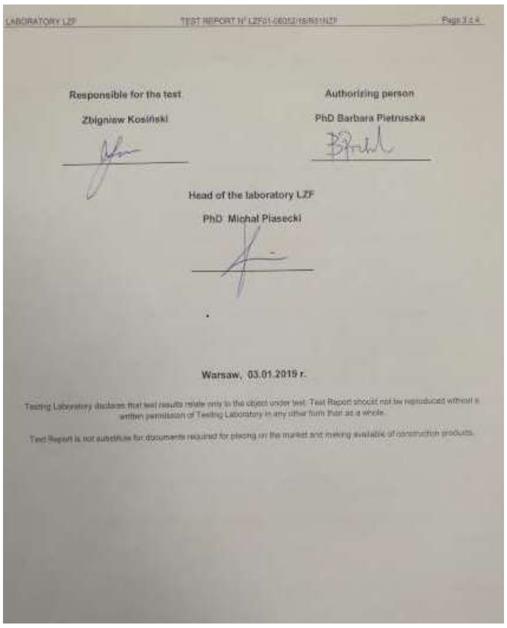
The expanded uncertainty were calculated using the factor k = 2, which corresponds to the level of confidence of approximately 95% of 3% acc. to Card of uncertainty LF-2/08.

The uncertainty value refers to the individual test results. The value of uncertainty can not be assigned directly to the characteristics of a given product, because the laboratory does not have knowledge about the variability of product population, but only about the tested sample.

Legend:

d - Measured thickness of the sample
ρ - density of the sample after conditioning
R - thermal resistance
λ - thermal conductivity

Figure 1 A foam sample after testing (dimensions of 300x300x50 mm)


LABORATORY LZF — TEST REPORT N° LZF01-06052/18/R61NZF — Page 3 z 4

Responsible for the test
Zbigniew Kosiński

Authorizing person
PhD Barbara Pietruszka

Head of the laboratory LZF
PhD Michał Piasecki

Warsaw, 03.01.2019 r.

Testing Laboratory declares that test results relate only to the object under test. Test Report should not be reproduced without a written permission of Testing Laboratory in any other form than as a whole.

Test Report is not substitute for documents required for placing on the market and making available of construction products.

Figure 4-4 Certification of TCF in terms of thermal insulation (ITB).

PAVUS, a.s.
AUTHORIZED BODY 216
NOTIFIED BODY 1391
ACCREDITED CERTIFICATION BODY FOR PRODUCTS N° 3041

Branch: FIRE TESTING LABORATORY VESELÍ NAD LUŽNICÍ
Čtvrť J. Hybeše 879
CZ 391 81 Veselí nad Lužnicí
Czech Republic

Address:
Prosecká 412/74, 190 00 Praha 9 – Prosek
Tel.: 286 019 587 Fax: 286 019 590
E-mail: mail@pavus.cz, http://www.pavus.cz

Phone:+420 381 477 418
Mail to: veseli@pavus.cz

REACTION TO FIRE CLASSIFICATION REPORT

The object of classification:	*Construction products excluding floorings and linear pipe thermal insulation products in accordance with ČSN EN 13501-1:2018, clause 11*
Issue number:	***PK1-01-19-045-E-0***
Product name and type:	*Two-component foam (TCF)*
Sponsor:	*Selena Labs Sp. z o.o.* *Pieszycka 1* *58-200 Dzierżoniów* *Polska*
Issuing organization:	*PAVUS, a.s.* *Authorized Body 216* *Notified Body 1391* *Accredited certification body for products No 3041* *- Accreditation issued by Czech Accreditation Institute, Public Service Company* *- Certificate of Accreditation N° 170/2019* *Prosecká 412/74* *190 00 PRAHA 9* *Czech Republic* *Order no. Z210180481*
Date of issue:	*2019-10-15*
Copies in total:	*4*
Copy number:	*4*
Pages in total:	*4*

1 INTRODUCTION

1.1 This Classification Report specifies classification method for the product *Two-component foam (TCF)* in conformity with the procedures set forth in ČSN EN 13501-1:2018.

1.2 This Classification Report has 4 pages and it can be used or reproduced as a whole only.

2 DETAILED INFORMATION ON THE ELEMENT TO BE CLASSIFIED

2.1 General

The product – *Two-component foam (TCF)* is made by the company Selena Labs Sp. z o.o., Pieszycka 1, 58-200 Dzierżoniów, Polska. It is the highly insulating and flame-retardant foam. It is used as light insulation material.

2.2 Product description

Thickness: 150÷180 mm
Density: 35÷40 kg/m³
Composition: PIR, fire retardants

3 REPORTS AND RESULTS IN SUPPORT OF CLASSIFICATION

3.1 Reports

Name of the Laboratory Address Accreditation number:	Name of sponsor of the Test Report	Report number Date of issue	Test method
PAVUS, a. s. *Veseli nad Luznici* *ATL No. 1026*	*Selena Labs Sp. z o.o.* *Pieszycka 1* *58-200 Dzierżoniów* *Polska*	*Pr-19-1.112* *2019-09-30*	*ČSN EN 13823+A1:2018*
		Pr-19-1.113 *2019-09-30*	*ČSN EN ISO 11925-2:2011*

3.2 Results

Test method	Parameter	Number of tests	Results	
			Continuous parameter-mean	Compliance with parameters
ČSN EN 13823	*$FIGRA_{0.4\,MJ}$ (W/s)* *THR_{600s} (MJ)* *LFS < edge of specimen* *SMOGRA (m^2/s^2) 1)* *TSP_{600s} (m^2) 1)* *non-occurring of flaming droplets/particles*	*3*	*172.5* *13.0* *-* *92.2* *1,117.3* *-*	*≤250 (C)* *≤15.0 (C)* *yes (B)* *≤180 (s2)* *> 200 (s3)* *yes (d0)*
ČSN EN ISO 11925-2 *Surface exposure thickness of 10 mm*	*Fs ≤ 150 mm up to 60 s* *Filter paper non-ignition*	*6*	*-*	*yes (B)* *yes (d0)*
ČSN EN ISO 11925-2 *Edge exposure thickness of 10 mm*	*Fs ≤ 150 mm up to 60 s* *Filter paper non-ignition*	*6*	*-*	*yes (B)* *yes (d0)*
ČSN EN ISO 11925-2 *Surface exposure thickness of 60 mm*	*Fs ≤ 150 mm up to 60 s* *Filter paper non-ignition*	*6*	*-*	*yes (B)* *yes (d0)*
ČSN EN ISO 11925-2 *Edge exposure thickness of 60 mm*	*Fs ≤ 150 mm up to 60 s* *Filter paper non-ignition*	*6*	*-*	*yes (B)* *yes (d0)*

1) Calculation procedure: classical

4 CLASSIFICATION AND FIELD OF APPLICATION

4.1 Classification references

This classification was carried out in conformity with ČSN EN 13501-1:2018.

4.2 Classification

The product - *Two-component foam (TCF)* - in conformity with its behaviour under the reaction-to-fire tests, is classified as follows:

Reaction to fire class: C-s3,d0

4.3 The field of application

This classification applies for the following parameters of the product:

Products from PIR and fire retardants core with thickness of 150 mm and density of 37kg/m³ ± 15 %.

This classification applies for the following end-use applications:

The product *Two-component foam (TCF)* - is intended for using as light insulation material. The core should be covered with metal sheets with thickness minimum of 0.8 mm and melting point equal to or greater than 1,000 °C.

Figure 4-5 Certification of TCF in terms of fire resistance (Pavus).

Thanks to the conducted tests for the integrated curtain wall system and the foam itself, we can compare the results and, above all, confirm the flame class for the developed TCF foam within the EENSULATE project. The certificate from the Czech Accreditation Institute - PAVUS for TCF – PIR foam is under development and will be sent to the EENSULATE project coordinator (due to the tests just carried out, end of July this year).

4.2 The OCF – PU foam

To improve fire behavior of OCF Selena used nanosized inorganic fillers (LDH) and expandable graphite (EG) which ensure high level of fire resistance. The experience from two component foam was transferred to increase the fire properties and removing toxic compound (eg halogen molecules).

Selena Labs has developed an innovative One Component Foam (OCF), WP5_6_2, which is a spray polyurethane foam. To improve fire behaviour and insulating efficiency, Selena Labs used nanosized inorganic fillers and expandable graphite in proven proportions.

4.2.1 Parameters & durability of OCF

Selena as a global leader in the production of OCF has implemented and tested recipes. Thus, the existing recipe of polyurethane foam, benchmark – B1 Straw PU Foam, Tytan, which was modified by replacing the halogen flame retardant with more environmentally friendly. A special polyester polyol was used in foam, which thanks to its chemical structure, works well as a flame retardant in polyurethane foams.

Table 4-4 List of key parameters for developed OCF foam (with <2,5 off LDH1 and <5,5% off EG) and benchmark B1 Straw PU Foam, Tytan

	BENCHMARK **B1 Straw PU Foam, Tytan**	**DEVELOPED OCF - WP5_6_2** <2,5 off LDH1 and <5,5% off EG
TOXIC COMPOUNDS	CONTAINING	NOT PRESENT
NET CONTENT [g]	385	385
FLAMMABILITY CLASS **(DIN 4102)**	B1 *certificate for B s1 d0 (test method EN ISO 11925-2, EN 13823)	B1
YIELD **(linear meter)**	≤ 5	≤ 6
DENSITY [kg/m³]	13 - 15	16 - 18
DIMENSIONAL STABILITY [%] **temperature of the can : application temperature 10 : 30 °C**	≤ 5	≤ 5

Figure 4-6 Developed OCF formulation compared with benchmark, B1– YIELD (left) and FLAMMABILITY CLASS (right)

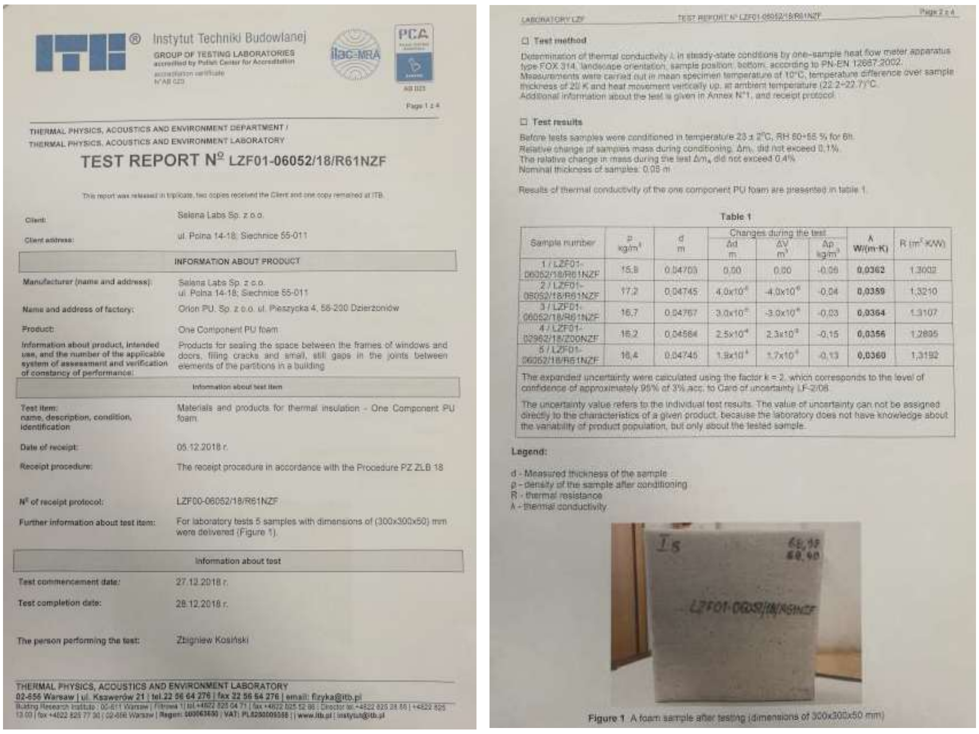
Selena developed a special 1.5 Component system to release solid additives during foam application, solving the problems of sedimentation of solid flame retardants in OCF. An additional cylinder is used, which is embedded in a metal can. Releasing the fillers placed in the additional cylinder is done by unscrewing the bottom of the can and thus the mechanical release of the additives into the foam takes place.

Special system 1,5 Component allows to achieve proper mixing and be stable for a period of 12M from the production.

Figure 4-7 Special system (1,5K) which consist of several elements like specific can with tailored little tube inside

Technical and safety data sheets for the developed OCF system have been prepared. The documents are attached to the report as a separate file.

4.2.2 Certificates obtained for OCF



Instytut Techniki Budowlanej
GROUP OF TESTING LABORATORIES
accredited by Polish Center for Accreditation
accreditation certificate
N° AB 023

Page 1 z 4

THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT DEPARTMENT /
THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT LABORATORY

TEST REPORT Nº LZF01-06052/18/R61NZF

This report was released in triplicate, two copies received the Client and one copy remained at ITB.

Client:	Selena Labs Sp. z o.o.
Client address:	ul. Polna 14-18; Siechnice 55-011
INFORMATION ABOUT PRODUCT	
Manufacturer (name and address):	Selena Labs Sp. z o.o. ul. Polna 14-18; Siechnice 55-011
Name and address of factory:	Orion PU. Sp. z o.o. ul. Pieszycka 4, 58-200 Dzierżoniów
Product:	One Component PU foam
Information about product, intended use, and the number of the applicable system of assessment and verification of constancy of performance:	Products for sealing the space between the frames of windows and doors, filling cracks and small, still gaps in the joints between elements of the partitions in a building
Information about test item	
Test item: name, description, condition, identification	Materials and products for thermal insulation - One Component PU foam
Date of receipt:	05.12.2018 r.
Receipt procedure:	The receipt procedure in accordance with the Procedure PZ ZLB 18
Nº of receipt protocol:	LZF00-06052/18/R61NZF
Further information about test item:	For laboratory tests 5 samples with dimensions of (300x300x50) mm were delivered (Figure 1).
Information about test	
Test commencement date:	27.12.2018 r.
Test completion date:	28.12.2018 r.
The person performing the test:	Zbigniew Kosiński

THERMAL PHYSICS, ACOUSTICS AND ENVIRONMENT LABORATORY
02-656 Warsaw | ul. Ksawerów 21 | tel.22 56 64 276 | fax 22 56 54 276 | email: fizyka@itb.pl
Building Research Institute | 00-611 Warsaw | Filtrowa 1 | www.itb.pl | instytut@itb.pl

LABORATORY LZF — TEST REPORT Nº LZF01-06052/18/R61NZF — Page 2 z 4

☐ **Test method**

Determination of thermal conductivity λ in steady-state conditions by one-sample heat flow meter apparatus type FOX 314, landscape orientation, sample position: bottom, according to PN-EN 12667:2002. Measurements were carried out in mean specimen temperature of 10°C, temperature difference over sample thickness of 20 K and heat movement vertically up, at ambient temperature (22.2÷22.7)°C. Additional information about the test is given in Annex N°1, and receipt protocol.

☐ **Test results**

Before tests samples were conditioned in temperature 23 ± 2°C, RH 60÷65 % for 6h.
Relative change of samples mass during conditioning, Δm_c did not exceed 0,1%.
The relative change in mass during the test Δm_s did not exceed 0,4%.
Nominal thickness of samples: 0,05 m

Results of thermal conductivity of the one component PU foam are presented in table 1.

Table 1

Sample number	ρ kg/m³	d m	Changes during the test: Δd m	ΔV m³	Δρ kg/m³	λ W/(m·K)	R (m²·K/W)
1 / LZF01-06052/18/R61NZF	15,8	0.04703	0.00	0.00	-0.06	**0,0362**	1.3002
2 / LZF01-06052/18/R61NZF	17,2	0.04745	4.0x10^-5	-4.0x10^-6	-0,04	**0,0359**	1,3210
3 / LZF01-06052/18/R61NZF	16,7	0.04767	-3.0x10^-5	-3.0x10^-6	-0,03	**0,0364**	1.3107
4 / LZF01-02962/18/Z00NZF	16,2	0,04584	2.5x10^-4	2.3x10^-5	-0,15	**0,0356**	1,2895
5 / LZF01-06052/18/R61NZF	16,4	0.04745	1.8x10^-4	1.7x10^-5	-0,13	**0,0360**	1,3192

The expanded uncertainty were calculated using the factor k = 2, which corresponds to the level of confidence of approximately 95% of 3% acc. to Card of uncertainty LF-2/06.

The uncertainty value refers to the individual test results. The value of uncertainty can not be assigned directly to the characteristics of a given product, because the laboratory does not have knowledge about the variability of product population, but only about the tested sample.

Legend:

d - Measured thickness of the sample
ρ - density of the sample after conditioning
R - thermal resistance
λ - thermal conductivity

Figure 1 A foam sample after testing (dimensions of 300x300x50 mm)

Figure 4-8 Certification of OCF in terms of thermal insulation

When it comes to fire resistance, the commercially available non-flammable foam - PU foam from B1 in Euroclass B s1 - has been used, which has been modified. The modification involved the use of halogen-free flame retardants, the second generation. Foam development in Ulster tests showed a better flame retardation of the product. For this reason, we have not applied for product certification because it is the second additional product developed as part of the EENSULATE project and the lack of business case for the developed OCF foam.

5 Publication of the results

Recently, paper entitled "Fire Retardant Action of Layered Double Hydroxides and Zirconium Phosphate Nanocomposites Fillers in Polyisocyanurate Foams", by Eleni Asimakopoulou, Jianping Zhang, Maurice Mckee, Kinga Wieczorek, Anna Krawczyk, Michele Andolfo, Marco Scatto, Michele Sisani, Maria Bastianini, Anastasios Karakassides & Pagona Papakonstantinou, has been published in *Fire Technology* (DOI: 10.1007/s10694-020-00953-7; published online on Feb 10th 2020). The title page of the abovementioned paper is shown in Figure 5-1. This study evaluated the fire behaviour of TCF foams, enhanced with lamellar inorganic fillers ($MgAlCO_3$, MgAl Stearate, and Zirconium Phosphate PIR-ZrP octadecylamine), as well as their post-burning characterization and morphological evaluation of residual materials.

Fire Technology

Manufactured in The United States
https://doi.org/10.1007/s10694-020-00953-7

Fire Retardant Action of Layered Double Hydroxides and Zirconium Phosphate Nanocomposites Fillers in Polyisocyanurate Foams

*Eleni Asimakopoulou**, *Jianping Zhang and Maurice Mckee, FireSERT, Belfast School of Architecture and the Built Environment, Ulster University, Newtownabbey BT37 0QB, UK*

Kinga Wieczorek, Anna Krawczyk and Michele Andolfo, SELENA Lab, Ul. Polna 14-18, 55011 Siechnice, Poland

Marco Scatto, Michele Sisani and Maria Bastianini, Prolabin and Tefarm SRL, Via dell'Acciaio 9, 06134 Perugia, Italy

Anastasios Karakassides and Pagona Papakonstantinou, Engineering Research Institute, School of Engineering, Ulster University, Newtownabbey BT37 0QB, UK

Received: 18 June 2019/**Accepted:** 16 January 2020

Abstract. Modern day energy codes are driving the design and multi-layered configuration of exterior wall systems with a significant emphasis on achieving high performance insulation towards improving energy performance of building envelopes. Use of highly insulating polyisocyanurate (PIR) based materials enhanced with eco-friendly lamellar inorganic fillers reinforces energy performance requirements, environmental challenges and cost reduction without compromising the overall building fire safety. The current work assessed the fire behaviour of PIR modified with three layered fillers, namely $MgAlCO_3$ (PIR-LDH1), MgAl Stearate (PIR-LDH2) and Zirconium Phosphate octadecylamine (PIR-ZrP3). For each of the fillers, three loadings (2, 4 and 6% by weight) were used. Optical analysis by X-ray diffraction patterns (XRD), cone calorimeter (CC), thermogravimetric (TGA) analysis, post-burning morphological evaluation using field emission scanning electron microscope (FESEM) and diffuse reflectance infrared spectroscopy (DRIFT) analysis, were performed. The results indicated that fire reaction properties and thermal stability of foam samples were enhanced with all three different lamellar inorganic smart fillers. The initial degradation temperature of PIR-layered filler samples was increased, demonstrating that incorporation of flame retardants decelerated the degradation of the PIR foam and contributed to significant char formation, from 19.5% in pure PIR samples to 33% in PIR-6%LDH1 samples. Increasing the filler content also resulted in improved char properties and decreased peak Heat Release Rates (HRR) in the cone calorime-

*Correspondence should be addressed to: Eleni Asimakopoulou, E-mail: e.asimakopoulou@ulster.ac.uk

Published online: 10 February 2020

Figure 5-1 Title page of recent publication.

6 Conclusion

Selena presents research on highly insulating materials to be a component of curtain wall glass facades, specifically a TCF to be used for the manufacturing of spandrel elements replacing cut-to-measure mineral wool panels (TCF is used either within the manufacturing line) and a OCF to be used as an effective thermal sealant at the interface between curtain wall and sub-structures, to be packaged in a pressurized can of variable size in order to be easily used in construction sites. TCF and OCF formulations with smart filler were prepared in order to evaluate and improve their morphological and mechanical properties, thermal conductivity, yield strength, fire resistance, thermal properties and filler content.

The developed products in the above task were characterized in external accredited units. Thermal insulation efficiency increased by the 20% by means of the TCF with solid additives and density of 35-40 kg/m^3 and of the 10% by means of the OCF with solid additives and density of 16-18 kg/m^3, the lambda parameter of benchmark is 0.04 W/m·K for both products. Two component foam's reaction to fire has been evaluated as C-s3-d0, which is sufficient in terms of fire resistance and droplet formation, but to be improved with respect to smoke generation. Additional activities and tests need to be performed in order to launch the product on the market. In particular, some additional smoke-supressing additives can be examined. The most promising candidates are: zinc aluminate, aluminium trihydrate, magnesium hydroxides, zinc silicate, and pyromellitic anhydride. Moreover, inorganic additives reduce also release of most toxic combustion products, like hydrogen cyanide and carbon monoxide. Selena's experience with aluminium trihydrate is quite positive, as proven in other projects and new products' R&D. Another option is to increase amount of currently used additives, LDHs and/or expanded graphite.

Finally, it has been found that TCF foam exhibits good sound insulating properties, comparable to the benchmark – mineral wool.