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Terms and definitions

Acronym	**Description**
ARHE	Average rate of heat release
ATH	Aluminium trihydroxide
Δm	Mass increment
E&E	Electric and electronic
FR	Fire retardant
HRR	Heat release rate
LOI	Limited oxygen index
MARHE	Maximum average rate of heat release
MLR	Mass loss rate
PHRR	Peak of heat release rate
SBI	Single burning item
SPR	Smoke production rate
SEA	Specific extinction area
$t_{flameout}$	Time to flameout
t_{ig}	Time to ignition
t_{peak}	Time to peak
THR	Total heat release
TSP	Total smoke production

Executive Summary

This report includes the fire related aspects of foams developed in BreadCell project. The fire behaviour of the foams has been addressed by:

1. Analysis of the potential applications and their fire requirements.
2. Improvement of the fire performance by using acids as fire retardants (FR) in the foams.
3. Improvement of the fire performance of the foams by surface FR coatings or FR papers.

The study of the fire performance has been carried out using a cone calorimeter as the most complete tool for the evaluation of reaction to fire of lab scale samples (10x10 cm) and propagation tests (UL94 and EN 11925-2). We demonstrate that phosphorous-based acids improve the fire performance the most and these results were published in two papers.[1] Specifically, phytic acid showed promising results especially reducing the heat release. Good classification according to Euroclass is expected (B-s1, d0). However, this classification needs to be confirmed by single burning item (SBI, EN13823). In addition to the phytic acid FRs in the foams also FR surface treatments with FR papers used on the foams were tested. These systems improved the ignitability of the surface fulfilling the requirements for insulation materials in building sector (E class) even avoiding the ignition of the surface. According to the results, the treated foams with phytic acid are a good candidate for more restrictive applications (Euroclass B-s1, d0) and the paper protected foams can be candidate for building insulation (Euroclass E classification).

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[1] Orzan, E., Barrio, A., Spirk, S., & Nypelö, T. (2024). Elucidation of cellulose phosphorylation with phytic acid. *Industrial Crops and Products*, *218*, 118858; Orzan, E., Barrio, A., Biegler, V., Schaubeder, J. B., Bismarck, A., Spirk, S., & Nypelö, T. (2025). Foaming and cross-linking of cellulose fibers using phytic acid. *Carbohydrate Polymers*, *347*, 122617.

Project overview

The major challenge to reduce consequences of climate change, microplastic pollution and raw material shortages is to convert our economic system. We need to replace fossil-based resources by renewable ones, replace persistent materials by biodegradables while developing and employing environmentally friendly processing technologies to create safe products with minimum impact on the environment. A real impact on economy, society and ecology is only generated if the materials, processes and products to be replaced are in large scale.

In BreadCell, we develop a radically new technology to produce porous lightweight low-density materials which are currently used in large scale industries and mainly consist of synthetic non-degradable polymers. BreadCell technology comprises a foaming process to create products relying on existing and new raw materials from pulping (cellulose, xylan), and to convert them to high value, lightweight, energy-absorbing and load-transferring composites such as in sports and safety components of cars. The whole process chain will be guided by safe-by-design principles to ensure the production of sustainable and inherently safe products. Today, the innovation capacity of European scientists and industry in the area of renewable materials makes them already the leading global players in the field. We will provide a scalable technology that will further support the European technological leadership in the area by cross-fertilization of different fields (pulp and paper, biotechnology, polymer technology, toxicity) while addressing needs of future materials.

Consortium:

CHALMERS UNIVERSITY OF TECHNOLOGY

TECHNISCHE UNIVERSITÄT GRAZ

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FUNDACION TECNALIA RESEARCH & INNOVATION

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1. Introduction

Fire performance requirements of materials and products depend largely on the application and use. In applications like building or transport, the safety of the occupants and road users is pivotal and specific fire requirements and testing is mandatory. On the other hand, there are applications in which these requirements are not so restrictive or there even may be no fire requirements. Hence, application areas must be selected prior to selecting analysis methods and designing a strategy for the improvement of the fire performance if required.

One of the most restrictive areas in terms of fire is the building sector. Considering limited time to escape, especially in high-rise buildings, building codes are used to define the fire requirements based on the use of the building (residential, public, hospitals, etc). In Europe, the standard for building products is the EN 13501[2], which establishes the requirements for a range of elements of the building (ceilings, walls, floors, structure, etc). As the fire scenarios of these elements are different, the fire tests for classifying them can also vary.

Another sector with complete legislation in terms of fire is transport, especially vehicles from where escaping could take a long time, such as ships and railway. IMO[3] for maritime and EN 45545-2[4] standards regulate and define the testing methods for different elements of ships and railway. There are other products like electronic and electric (E&E) devices where the fire requirements are only focused in preventing the initiation of fires and the fire characterization methods are limited to flammability test (UL94[5], LOI[6], etc).

Considering the flammable nature of cellulose materials, the expectation was that without fire retardants, the target performance cannot be reached. We used fire retardant additives to reduce the flammability of BreadCell foams for desired building and transportation applications. For selecting the fire retardants, sustainability and safety criteria are very topical - especially the use of halogenated compounds, boron salts and Sb should be avoided. Hence in this project cellulose fibers were treated with citric acid and phytic acid (C and P series foams). A fire retardant layer was also applied onto the foams, e.g. by spraying phytic acid onto a foam or by gluing inorganic FR paper (Onyx paper containing aluminium trihydroxide, ATH, as an FR) onto the surface of the foam, in order to hinder heat or fire to penetrate through the foam or sandwich structure.

2. Selection of fire characterization methods and materials

Considering the potential applications of cellulose fiber foams, flammability and parameters related with heat and smoke emissions need to be characterized. Considering the size of the papers and foams, bench scale tests were selected:

1. Flammability test: UL94 was selected for small samples (papers of 250x50 mm) and EN11925-2[7] as the reference standard for building sector (EN13501) for foamed products.

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[2] EN 13501-1. Fire classification of construction products and building elements.

[3] IMO. 2010 FTP Code.

[4] EN 45545-2. European railway standard for fire safety.

[5] UL-94. Tests for Flammability of Plastic Materials for Parts in Devices and Appliances.

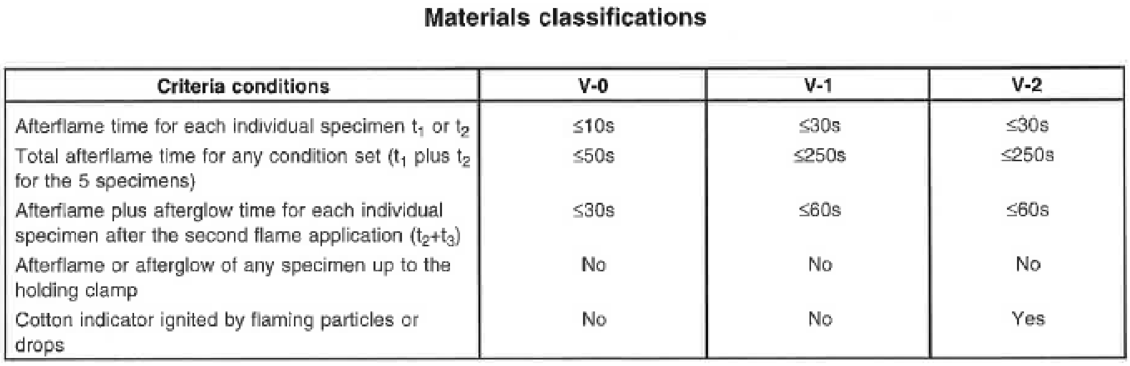
[6] ISO 4589. Plastics — Determination of burning behaviour by oxygen index.

[7] EN ISO 11925-2 Reaction to fire tests — Ignitability of building products subjected to direct impingement of flame — Part 2: Single-flame source test.

2. For assessing heat and smoke production, cone calorimeter (10x10 cm) was selected. This bench scale test allows assessing mass loss, heat release and smoke opacity[8] in accordance to the classification method (EN13501, building products fire reaction characterization, Euroclass according to single burning item (SBI). Hence, with the data generated by cone calorimeter it is possible to predict the behaviour of the materials in large scale test.

2.1. UL94

UL94 (T*ests for Flammability of Plastic Materials for Parts in Devices and Appliances)* specifies flame classifications that were assigned to materials based on the results of small-scale flame test. Burning characteristics after test specimens have been exposed to a specified test flame under controlled laboratory conditions. The test evaluates both the burning and afterglow times and dripping of the burning test specimen. A flame of 50 W (methane) at a distance of 20 mm is applied during 10 s to the bottom edge of the sample. After removal of the ignition source, the self-extinguishing time of the specimen was recorded. It is also tested in UL94 whether the test specimen drips flaming particles that can ignite a cotton indicator located below the specimen. Based on these results, the specimens were classified into three vertical ratings — V-2, V-1 and V-0; classification criteria are reported below:



Materials classifications

Criteria conditions	V-0	V-1	V-2
Afterflame time for each individual specimen t_1 or t_2	≤10s	≤30s	≤30s
Total afterflame time for any condition set (t_1 plus t_2 for the 5 specimens)	≤50s	≤250s	≤250s
Afterflame plus afterglow time for each individual specimen after the second flame application (t_2+t_3)	≤30s	≤60s	≤60s
Afterflame or afterglow of any specimen up to the holding clamp	No	No	No
Cotton indicator ignited by flaming particles or drops	No	No	Yes

2.2. EN11925-2. Reaction to fire tests for building products. Ignitability of building products when subjected to direct impingement of flame. Part 2: Single-flame source test

Foams (250x90x20 mm) were placed on a stainless steel U-shaped double frame hung vertically, in a way that the centre of the bottom edge and the surface of the sample were directly exposed to the flame.

a. The flame is applied on the surface of the foam (surface). A line was drawn on the specimen and parallel to the bottom edge of the specimen; the line was 40 mm away from the bottom edge. The flame was set to 20 mm high and moved to approach to the middle point of the drawn line until flame was 5 mm away from the line. The flame was set to 15 s.
b. The flame is applied on the centre bottom edge of the foam (edge). The nozzle of the burner was 16 mm away from the bottom edge. The flame was set to 20 mm high and moved below the centre of the bottom edge of the specimen. The flame was set to 15 s.

For both tests, a filter paper was placed underneath the testing specimens to test whether any dripping during burning can ignite the filter paper.

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[8] EN 13823. Reaction to fire tests for building products — Building products excluding floorings exposed to the thermal attack by a single burning item.

2.3. Cone calorimeter (ISO 5660-2)

Figure 1. Cone calorimeter.

The cone calorimeter is the most useful equipment for the characterization of fire reaction at bench scale. A cone calorimeter uses the oxygen consumption principle for calculating the heat release in accordance to the classification test (SBI for building materials). The main advantage is the small size of the foams needed for the test (10x10 cm). The key output parameters are: Heat Release Rate (HRR, kW/m^2), Average Rate of Heat Release (ARHE, kW/m^2), Total Heat Release (THR, kW/m^2), Time to Ignition (TTI, s), Mass Loss Rate (MLR, g/s), Specific Extinction Area (SEA, m^2/kg), Smoke Production Rate (SPR, m^2/s) and Smoke Opacity (TSP, m^2).

Another important advantage of a cone calorimeter is that different fire scenarios can be simulated by the choice of heat flux ranges (0-100 kw/m^2). For this study, a flux of 35 kW/m^2 was selected as the fire scenario, representing a medium thermal attack (ISO 5660-3). The separation of the foam specimens from the heat source was 25 mm and the test time was 600 s.

2.4. Selected materials for characterization of their fire performance

Cellulose fiber foams that were investigated were produced using frothing and BreadCell foaming technology. They are foams characterized by density of 80 kg/m^3 and fibrillar network structure. The foams were also incorporated into a sandwich panel. Fire retardants were applied on the foams and on the sandwich panels either 1) in the foam or 2) as a coating on the surface of the foam and sandwich composites. In approach 1, citric acid and phytic acid was mixed in the cellulose foam precursors following by curing at various temperature (Table 1). As the control of the foams containing citric and phytic acids, foams without acids were also subjected to corresponding curing temperature. In approach 2, either the fire retardant (i.e. phytic acid) or fire retardant paper (i.e. Firepli paper with grammage of 200 or 300 gsm, Onyx Paper) was used as coatings on the surface of the foam and sandwich composites (Table 3).

3. Results

3.1. Characterization of foams containing phytic and citric acid

3.1.1. UL94 Results

UL94 is conceptualized for the ignitability of plastic materials that typically are prepared by injection or thermoforming. Since it was difficult to prepare foams with the defined dimensions (125x13 mm) cellulose papers treated with the acids at different curing times were prepared. All the untreated papers ignited quickly and reached the upper edge of the test specimen (Figure 2).

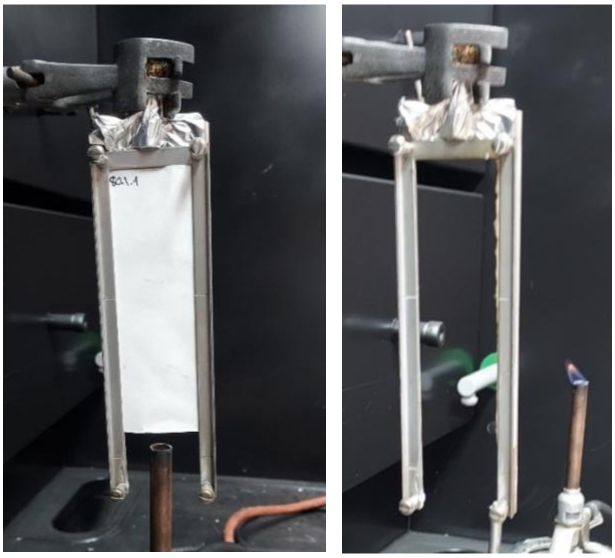

Figure 2. Presentation of a paper before (left) and after (right) UL94 test.

Only in the case of phytic acid treatment (Figure 3) the flame did not reach the top of the frame, giving a preliminary result of the usefulness of phytic acid as a fire-retardant additive for BreadCell foams. However, there was significant deviation between measurements that could not be resolved and hence, we deemed that UL94 was actually not a good method to evaluate the fire propagation in papers.

Figure 3. Paper treated with phytic acid before (left) and after (right) UL94 test.

3.1.2. Cone calorimeter results

Various foams were produced by altering the acid component (phytic or citric acid) and curing temperature that was applied to crosslink the cellulose with the acids (80, 120 and 160 ºC). Details are found in Orzan et al. 2024.[9] Table 1 summarizes the codification of the foams.

Table 1: Foams that were tested included untreated BreadCell foams (CON), foams treated with citric acid (C) and phytic acid (P) fire retardants. Foams were heat treated in respective temperatures for attachment of the fire retardants.

Reference	Description
CON80	Untreated foam 80 ºC curing
CON120	Untreated foam 120 ºC curing
CON160	Untreated foam 160 ºC curing
C80	Foam with citric acid 80 ºC curing
C120	Foam with citric acid 120 ºC curing
C160	Foam with citric acid 160 ºC curing
P80	Foam with phytic acid 80 ºC curing
P120	Foam with phytic acid 120 ºC curing
P160	Foam with phytic acid 160 ºC curing

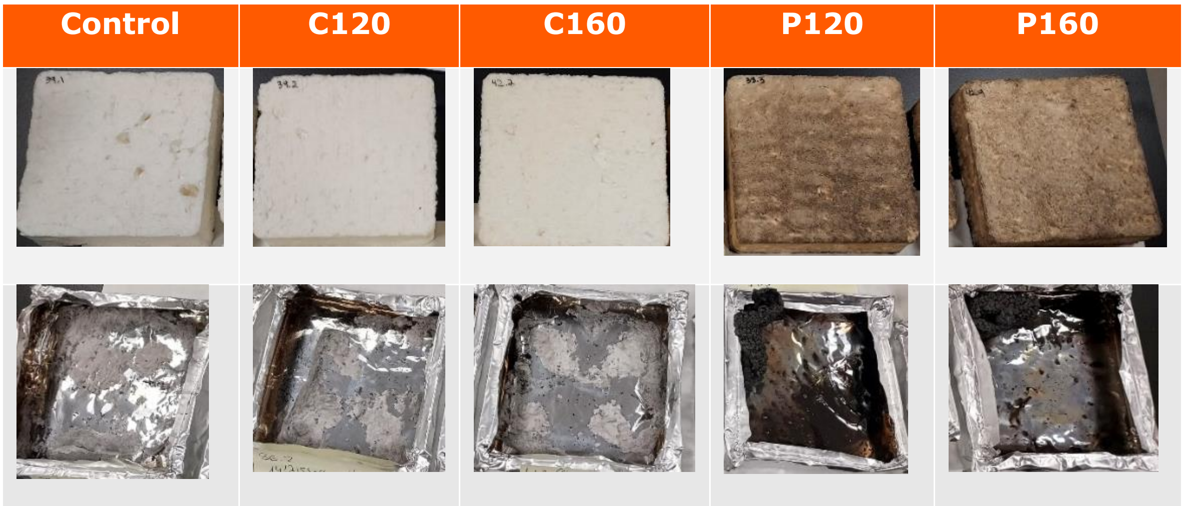

Figure 4. Visual appearance of the foams before (top row) and after (bottom row) the cone calorimeter test.

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[9] Orzan, E., Barrio, A., Spirk, S., & Nypelö, T. (2024). Elucidation of cellulose phosphorylation with phytic acid. *Industrial Crops and Products*, *218*, 118858; Orzan, E., Barrio, A., Biegler, V., Schaubeder, J. B., Bismarck, A., Spirk, S., & Nypelö, T. (2025). Foaming and cross-linking of cellulose fibers using phytic acid. *Carbohydrate Polymers*, *347*, 122617.

Figure 4 shows the visual appearance of selected foams before and after (the fire exposure in the cone calorimeter tests. As can be observed, phytic acid treated foams produce a dark residue (char) while the other foams did not. Table 2 summarizes the results of the main parameters measured in cone calorimeter tests.

Table 2: Cone calorimeter results of foams containing citric acid (C) and phytic acid (P) (Mean values, test done by triplicate).

Ref	**Δm (g)**	**Residue (%)**	**t_{ig} (s)**	**PHRR (kW/m^2)**	**T_{peak} (s)**	**THR (MJ/m^2)**	**MARHE (kW/m^2)**	**TSP (m^2)**	**SEA (m^2/kg)**
CON80	13.26	0.24	9.33	117.67	13.33	19.73	76.80	0.11	5.75
CON120	13.69	0.24	9.33	116.54	103.33	19.87	71.43	0.23	15.21
CON160	12.51	4.70	4.67	119.37	13.33	17.96	79.67	0.14	10.00
C80	14.29	0.23	9.67	132.88	16.00	22.49	79.98	0.13	6.82
C120	14.22	0.23	9.67	123.72	110.67	21.53	75.63	0.22	13.45
C160	14.32	2.98	8.33	115.47	21.33	21.50	69.29	0.19	11.66
P80	13.84	2.98	8.33	98.52	13.33	19.38	70.86	0.03	-0.99
P120	13.28	4.70	4.67	85.33	21.33	18.50	58.81	0.06	1.95
P160	12.65	2.81	3.33	85.52	19.33	17.45	59.34	0.05	2.07

The charring promotion of citric acid was more efficient at 160 ºC (C160) probably due to the crosslinking reaction of the citric acid with the cellulose (residue of 3% for C160 vs 0.2% for C80 and C120). The charring effect of phytic acid was higher (4.7%, P120) but decreased with the temperature (2.8%; P160) probably due to the partial degradation of the cellulose with the acid and temperature (also observed in the images as a dark surface).

In terms of heat release (PHRR and MARHE), foams with phytic acid showed a significant decrease in the heat release as compared to the control group. However, the heat release of BreadCell foam containing phytic acid and cured at 120 °C and 160 °C was similar. On the other hand, foams treated with citric acid showed similar curves and even a higher THR than the untreated foams.

Analyzing the opacity of the smoke (TSP and SEA), phytic acid reduced the foam smoke emission significantly (50% for TSP and 86% for SEA). Curves of the selected parameters (HRR, ARHE, THR, TSP and Mass change as function of time) are included in APPENDIX (Figure A1-A5, correspondingly).

Highlights:

- Citric and phytic acids were tested as fire retardants for cellulose foams
- In selected conditions (35 kW/m^2), the foams ignite and release a significant amount of heat (MARHE= 71.4 kw/m^2).
- The presence of citric acid showed a positive effect promoting charring (160 ºC) but it had a low effect in heat and smoke production.
- Phytic acid significantly reduced the heat and the smoke produced and promote the charring of the foam. The effect of the temperature (160 ºC) seems to affect the formation of the foam by the partial degradation of the cellulose.

3.2. Characterization of foams coated with fire retardant papers and fire retardants

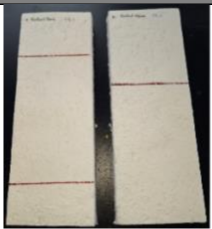
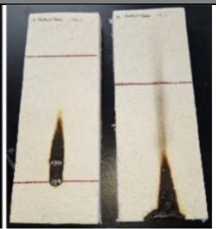
Fire retardant papers and phytic acid were used as surface treatments for the foams. Foams made using two foaming technologies were selected, namely frothed and bioblown foams (Table 3). Frothed foams were also constructed into a sandwich structure and included in the study.

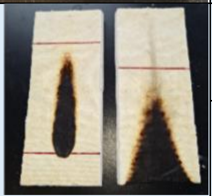
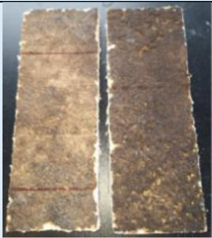
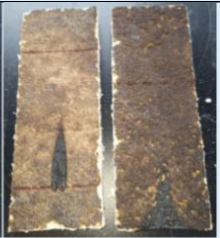
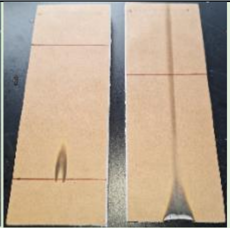
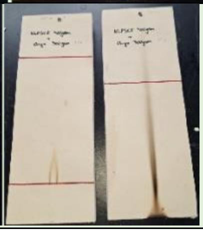
Table 3: Foams surface treated with fire retardant paper (FRpaper, grammage of 200 or 300 gsm) or phytic acid.

Ref.	Description
A	Untreated frothed foam
B	Frothed foam + FRpaper200
C	Frothed foam + FRpaper300
D	Frothed foam + phytic acid in formulation
E	Untreated bioblown foam
F	Bioblown foam + phytic acid coating
G	Sandwich foam
H	Sandwich foam + FRpaper200
I	Sandwich foam + FRpaper300

3.2.1. EN11925-2. Ignitability test

Table 4: Ignitability analysis according to EN 11925-2.

	before	after	Sample ignition		Flame propagation to 150 mm (Fs)	Filter paper ignition
A			Surface:	yes	No	No
			Bottom edge:	yes	No	Yes
B			Surface:	No	No	No
			Bottom edge:	yes	No	Yes

C			Surface:	No	No	No
			Bottom edge:	yes	No	Yes
D			Surface:	yes	No	No
			Bottom edge:	yes	No	Yes
E			Surface:	yes	No	No
			Bottom edge:	yes	No	Yes
F			Surface:	yes	No	No
			Bottom edge:	yes	No	Yes
G			Surface:	yes	No	No
			Bottom edge:	yes	No	Yes
H			Surface:	No	No	No
			Bottom edge:	yes	No	Yes
I			Surface:	No	No	No
			Bottom edge:	yes	No	Yes

Ignitability analysis of the selected foams are summarised in Table 4. All the selected FR treatments reduced the ignitability of the foams reducing the spread of the flame compared with the untreated foams (both frothed and bioblown). Foams with FR paper avoided the ignition of the surface of the foams. No significant differences were observed for FRpapers of difference grammage (200 and 300 gsm). **According to this analysis, these foams can reach Euroclass E classification which is the minimum classification for insulation materials in Building codes (EN 13501).**

3.2.2. Cone calorimetry analysis

Table 5: Cone calorimeter analysis for surface treated foams.

Ref	Δm (g)	Residue (%)	t_{ig} (s)	$t_{flameout}$ (s)	PHRR (kW/m^2)	t_{peak} (s)	MARHE (kW/m^2)	THR (MJ/m^2)	TSP (m^2)
A	16.42	1.18	9	296	99.12	14.50	73.81	19.90	0.09
B	19.50	12.54	29	373	151.23	35.00	60.29	22.35	0.03
C	20.43	15.09	36	444	153.17	42.50	55.34	22.60	0.03
E	14.79	0.68	11	594	114.73	19.00	80.75	19.95	0.02
F	12.53	24.73	59	423	29.75	271.00	14.68	7.20	1.14
G	21.65	8.11	24	379	229.32	32.50	88.53	25.60	0.14
H	25.04	20.09	41	548	219.91	52.50	74.07	24.75	0.14
I	24.62	22.20	48	567	207.54	60.50	66.58	24.05	0.16

Cone calorimetry analysis was performed in duplicate and the results are expressed as mean value (Table 5). Foams and composites containing FR papers (B, C, H and I) had delayed ignitability (t_{ig}) as compared to their corresponding reference foams (A) and composites (G) in line with the results observed for ignitability test; same holds true for phytic acid (F) coated bioblown foam as compared to its benchmark E. However, due to the high thermal attack (compared with the single flame of the propagation test), the peaks of HRR are larger for foams with FR papers B and C, probably due to the combustion of the paper itself. Bioblown foam treated with phytic acid (F) showed a very good result in heat release in consistency with the previous reported results about the study of the fire behaviour of the cross linker and could be a potential candidate for B-s1, d0 classification in building sector (SBI test according to EN13823).

Total heat release is also increased for those foams with FR papers (probably due to the increase in the combustible material, resulting in a doubled mass of the whole specimens) and very low in bioblown foam + phytic acid. Smoke related parameter (i.e. TSP) showed similar results considering the increase in the mass of the foams. The trend of the different parameters is shown in the corresponding curves (See Appendix; HRR, ARHE, THR, TSP and mass responses, Figure A6-A10, correspondingly).

References with FR papers delay the ignition of the foams but release a significant amount of heat (PHRR 150 kW/m^2, B and C, in Table 5). A slight delay in the time to ignition was detected for FR paper 300 compared with FR paper 200 without great impact in the maximum values of HRR. In the case of phytic acid coating, very low HRR were observed (even in one replicate, no ignition was observed).

The effect of the delay in the ignition was also shown from ARHE curve (Figure A7). Due to the delayed ignition, although the Breadcell foams containing FRpaper (B and C) released more heat than the foams without FRpaper (A), such heat was released over a longer period, shown by a lower ARHE of the foams with FRpaper. In THR curves, the increase in the combustion material is observed for foams with FR papers. As expected, based on HRR curves, the THR produced by phytic acid coated foam is reduced considerably.

The smoke produced by the foams is very low considering the low density of the foams. Phytic acid foam releases larger amount of smoke due to the fire retardancy of the foam producing more particles in the effluents due to the incomplete pyrolysis.

4. CONCLUSIONS

Phytic acid was proven to be a good fire retardant especially with regards to heat release. Good classification (B-s1, d0) according to Euroclass (EN 13501) can be expected when it is applied into cellulose fiber foams. Applying fire retardant papers onto foams can reduce the ignitability of the surface fulfilling the requirements for insulation materials in building sector (E class). However, in a more severe fire scenario (heat attack of 35 kW/m^2) the improvement in the fire retardancy of the FRpapers is not enough for the foams to reach better classifications as the heat release is larger than with the untreated foams. Bigger amount of FR (ATH in the case of papers) should be considered for these applications.

The fire classifications of course have to be validated according to regulatory and certification tests. However, we have demonstrated that in laboratory scale the approaches to use phytic acid or FR papers have the potential to transform cellulose fiber foams to comply with fire regulations in sectors such as building and in transport.

5. APPENDIX

Cone calorimeter data for foams including citric and phytic acids

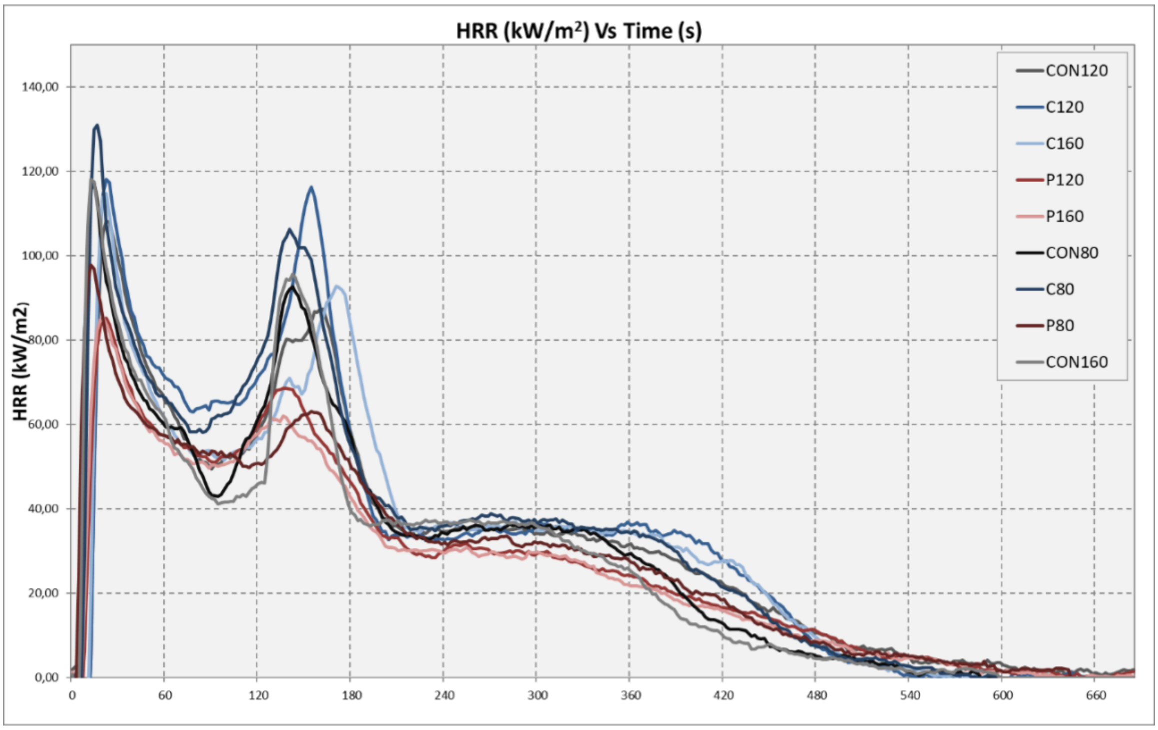


Figure A1. HRR curves of foams containing citric acid (C) and phytic acid (P) (mean).

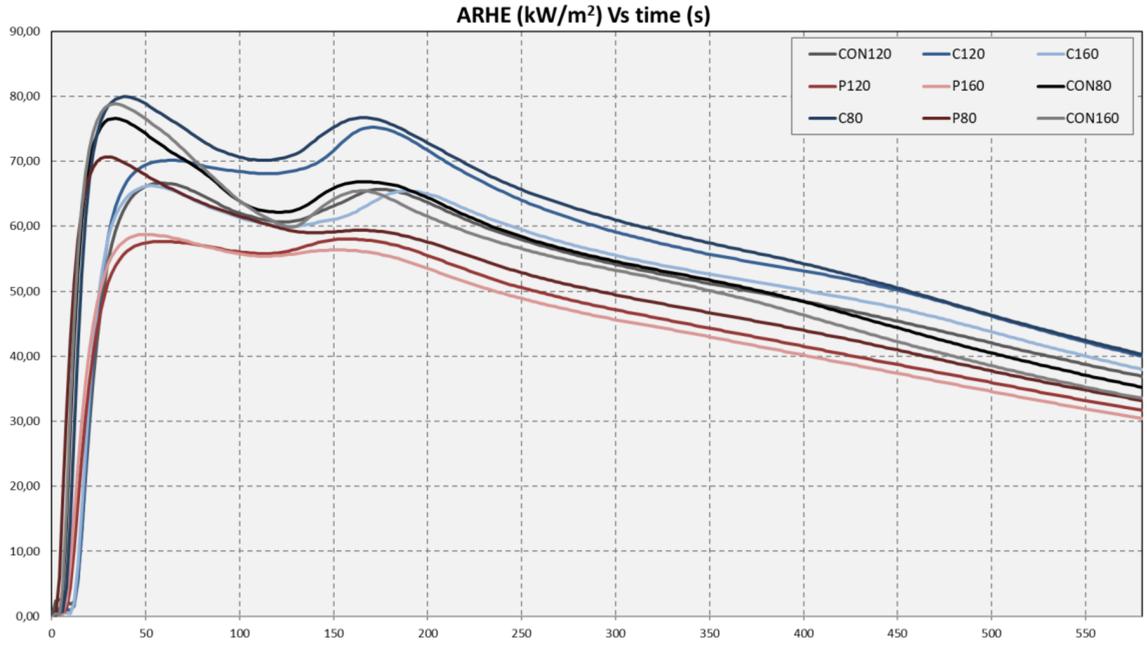


Figure A2. ARHE curves of foams containing citric acid (C) and phytic acid (P) (mean).

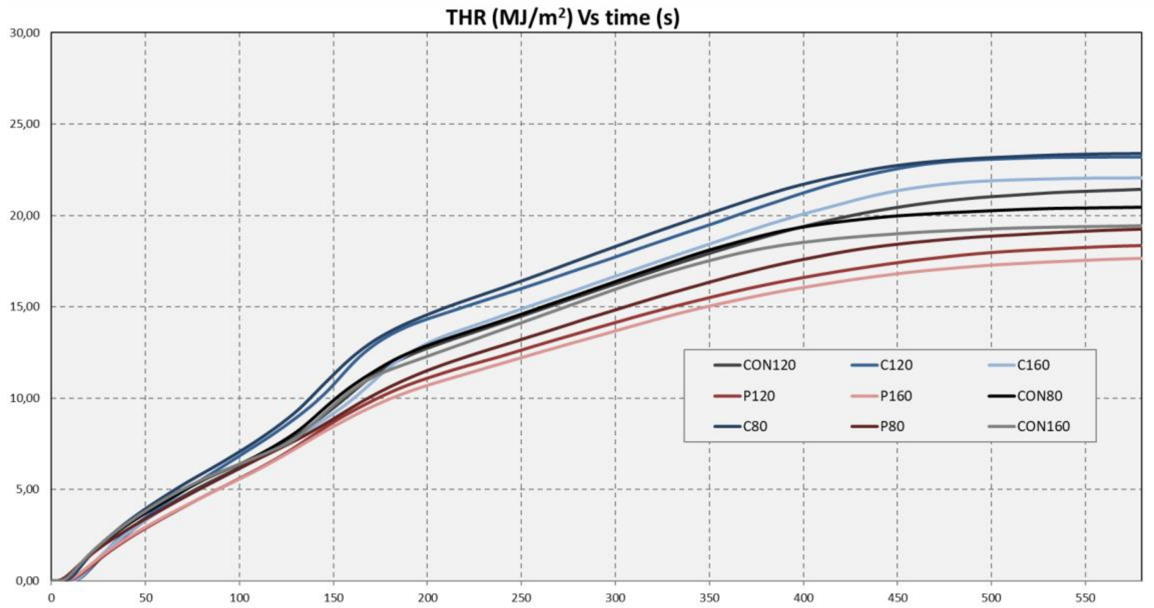


Figure A3. THR curves of foams containing citric acid (C) and phytic acid (P) (mean).

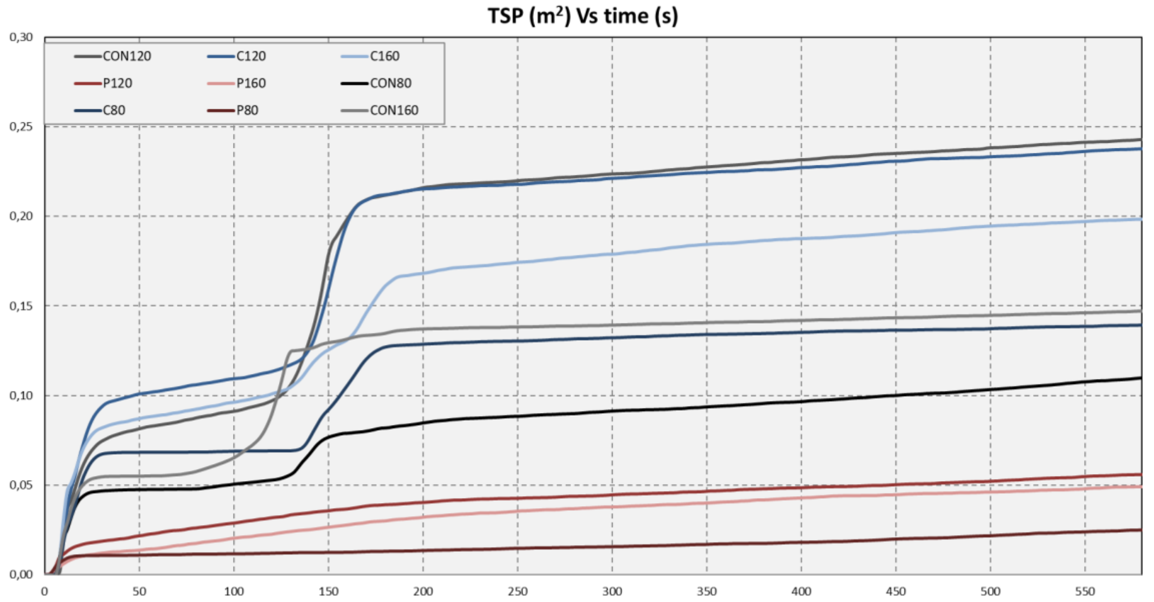


Figure A4. TSP curves of foams containing citric acid (C) and phytic acid (P) (mean).

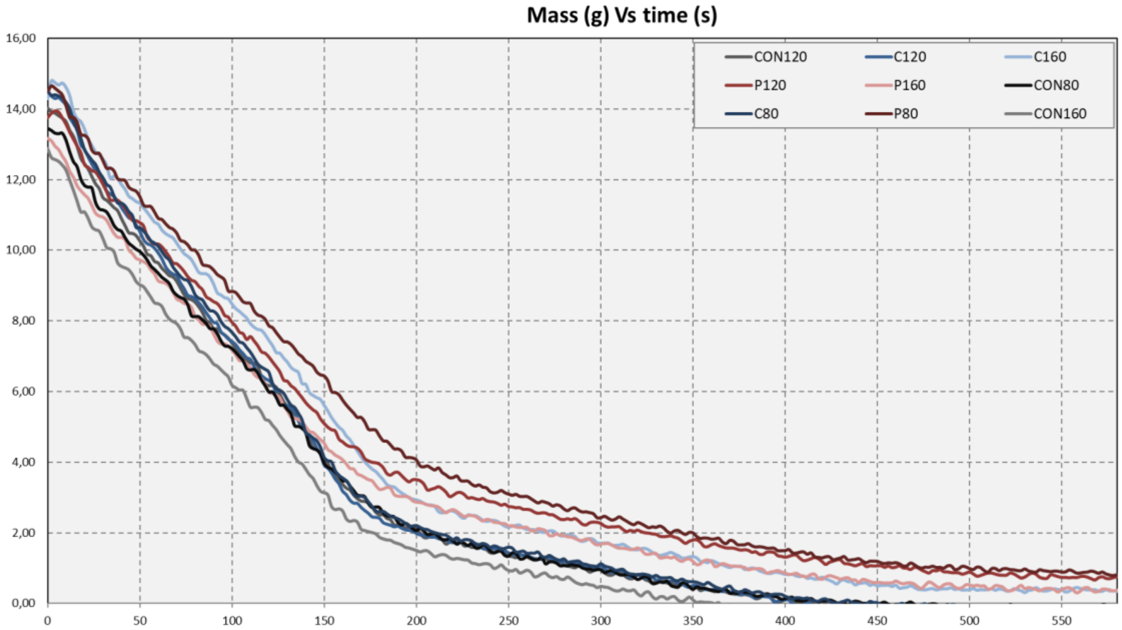


Figure A5. Mass curves of foams containing citric acid (C) and phytic acid (P) (mean).

Cone calorimeter data for foams with surface treatments (phytic acid and FR papers)

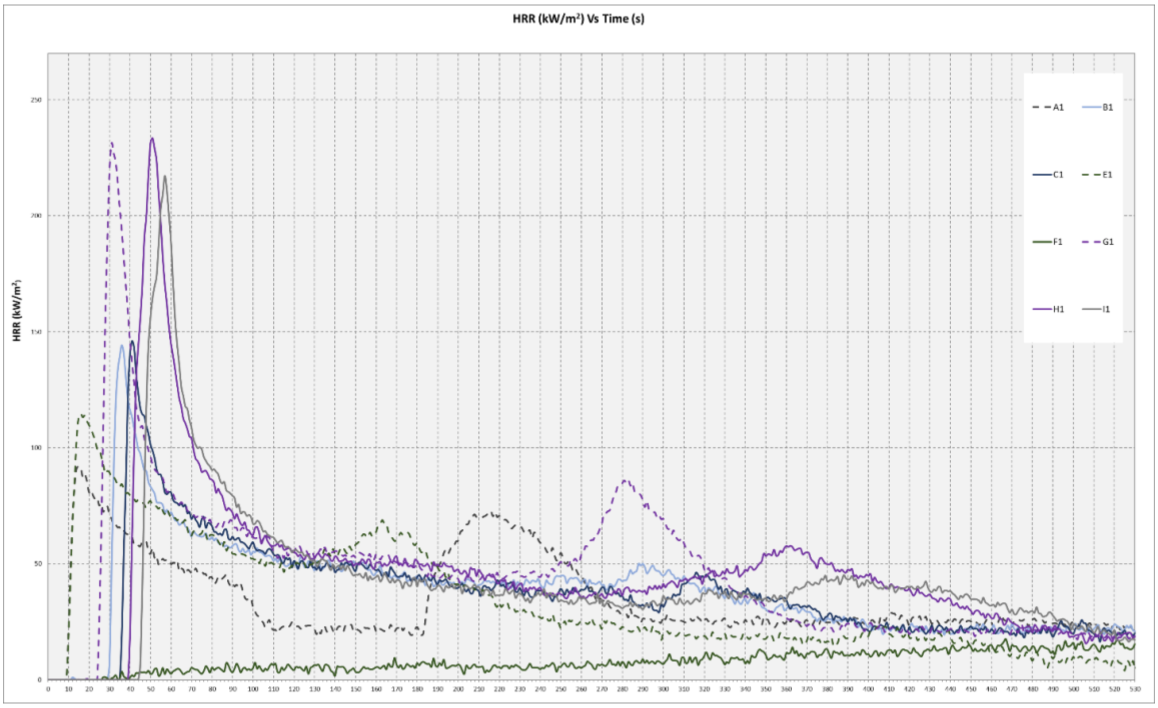


Figure A6. HRR curves of surface treated foams.

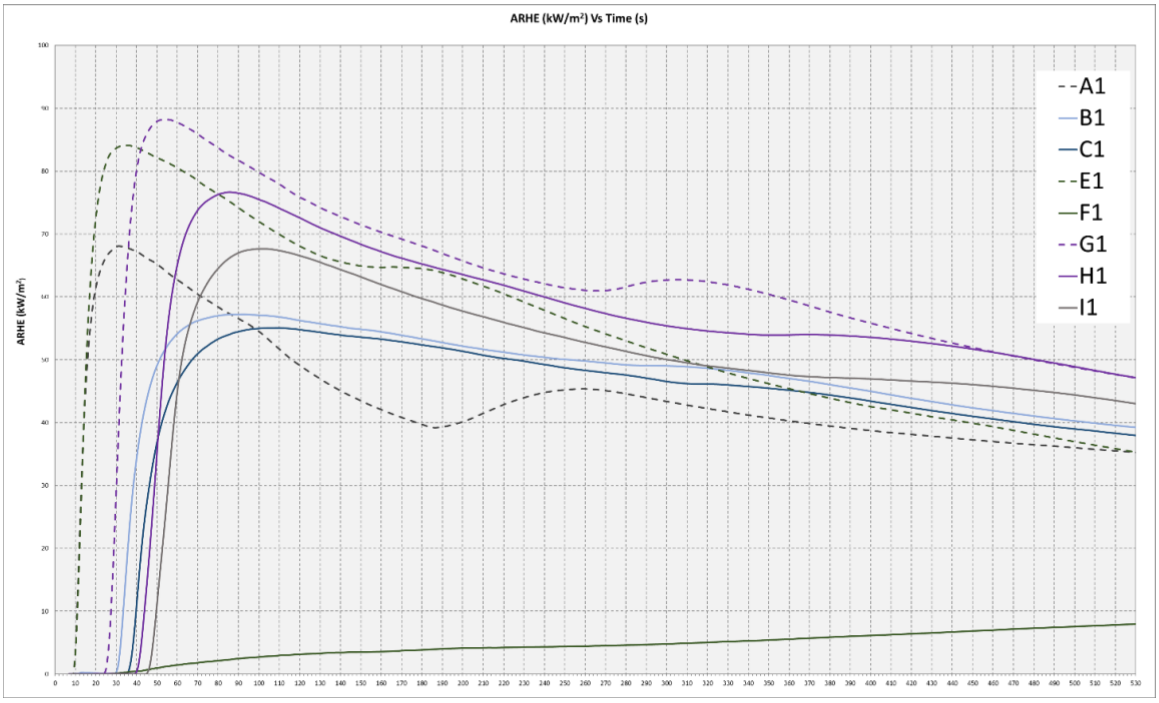


Figure A7. ARHE curves of surface treated foams.

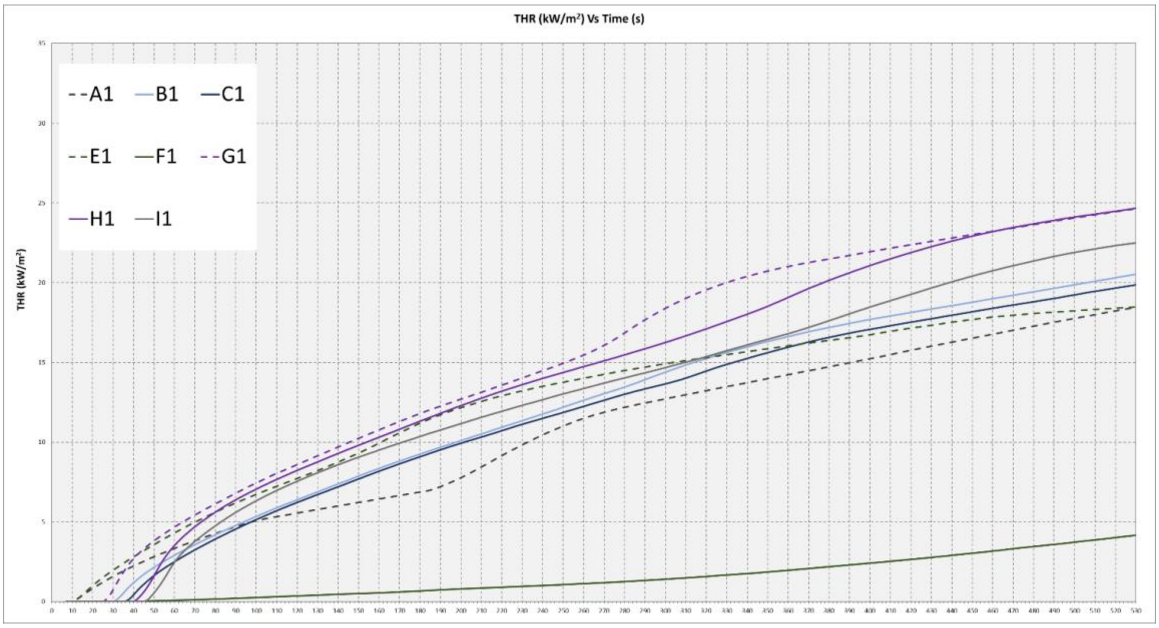


Figure A8. THR curves of surface treated foams (duplicate).

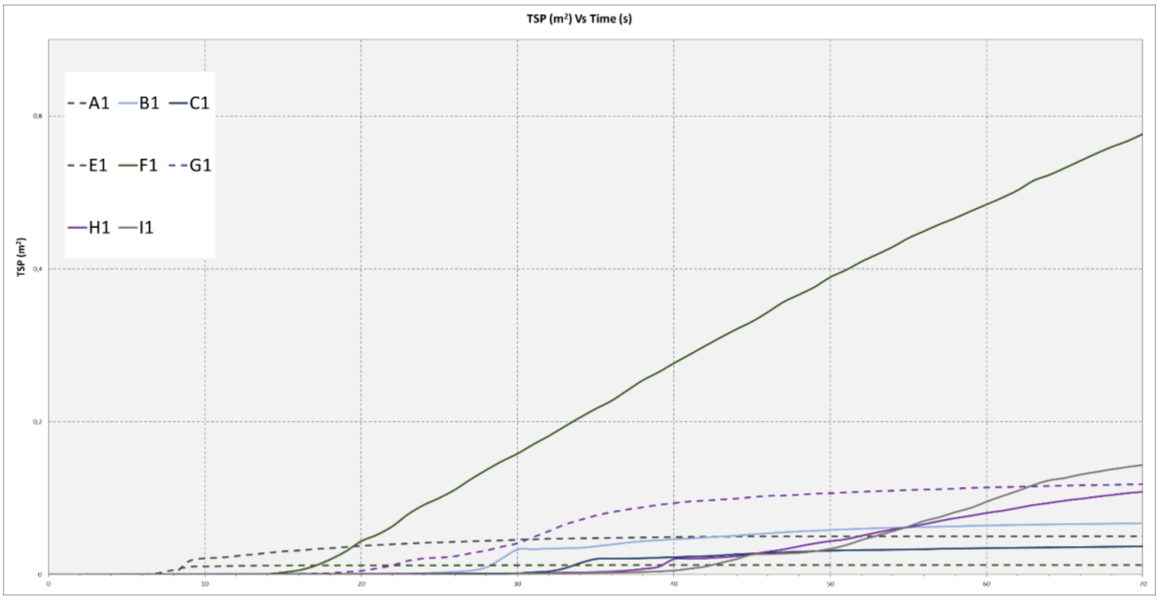


Figure A9. TSP curves of surface treated foams.

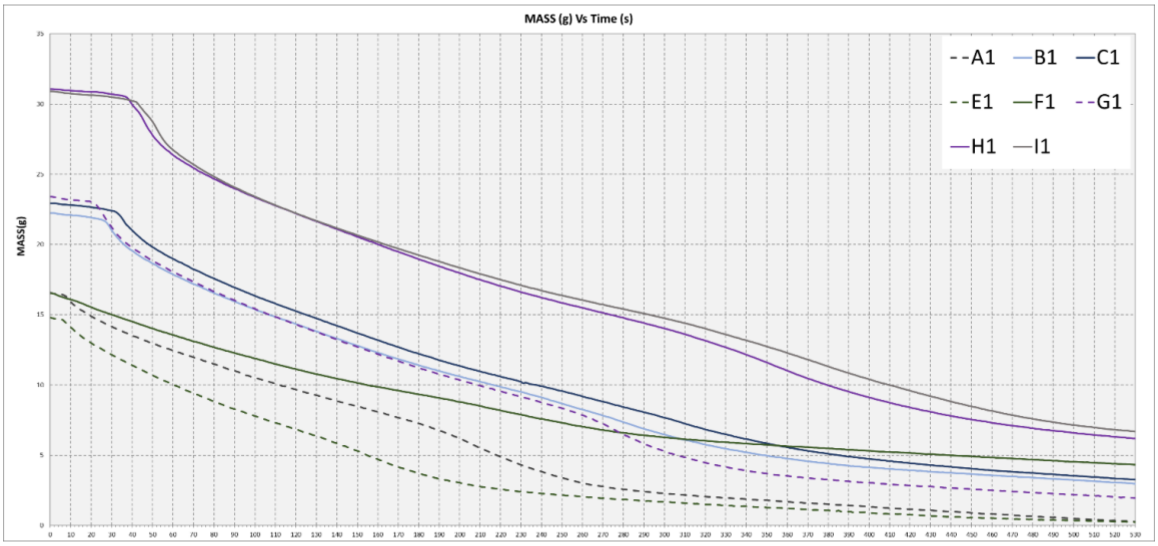


Figure A10. Mass curves of surface treated foams.